N00164.AR.001204
NSA CRANE
5090.3a

SAMPLING AND ANALYSIS PLAN (FIELD SAMPLING PLAN AND QUALITY ASSURANCE PROJECT PLAN) FOR SOLID WASTE MANAGEMENT UNIT 27 ILLUMINANT BUILDING 126 RESOURCE CONSERVATION RECOVERY ACT FACILITY INVESTIGATION NSA CRANE IN
1/1/2011
TETRA TECH NUS INC

Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62470-08-D-1001

Rev. 0
01/11

Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for SWMU 27 – Illuminant Building 126 Resource Conservation and Recovery Act Facility Investigation

Naval Support Activity Crane
Crane, Indiana

Contract Task Order F276

January 2011

NAVFAC
Naval Facilities Engineering Command
Midwest
201 Decatur Avenue
Building 1A, Code EV
Great Lakes, Illinois 60088

TETRA TECH NUS, Inc.

SAP Worksheet No. 1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

SAMPLING AND ANALYSIS PLAN
(FIELD SAMPLING PLAN AND QUALITY ASSURANCE PROJECT PLAN)
January 2011

RESOURCE CONSERVATION AND RECOVERY ACT (RCRA) FACILITY INVESTIGATION (RFI)

SWMU 27 – ILLUMINANT BUILDING 126
NAVAL SUPPORT ACTIVITY (NSA) CRANE
CRANE, INDIANA

Prepared for:
Naval Facilities Engineering Command Midwest
201 Decatur Avenue, Building 1A
Great Lakes, Illinois 60088

Prepared by:
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, Pennsylvania 19406
610-491-9688

Prepared under:
Contract No. N62470-08-D-1001
Contract Task Order F276

Review Signatures:

Richard Barringer, CPG, CHMM, REM /Project Manager/Date 1/25/2011
Tetra Tech NUS, Inc.

Tom Johnston, PhD/CLEAN Quality Assurance Manager/Date
Tetra Tech NUS, Inc.

2/7/11
Thomas J. Brent/NAVFAC PWD Crane ERSM/Date
NSA Crane Environmental Restoration Site Manager

Approval Signatures:

HICKEY.HOWARD.M.1287981070
Digitally signed by HICKEY.HOWARD.M.1287981070
DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=HICKEY.HOWARD.M.1287981070
Date: 2011.03.29 07:33:57 -05'00'
Howard Hickey/Navy RPM/Date

Doug Griffin/RPM/Date
Indiana Department of Environmental Management

BOWERS.KENNETH.A.1230092474
Digitally signed by BOWERS.KENNETH.A.1230092474
DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=BOWERS.KENNETH.A.1230092474
Date: 2011.02.01 10:29:15 -05'00'
Government Chemist/Date
NAVFAC QA Review

This page intentionally left blank.

EXECUTIVE SUMMARY

Tetra Tech NUS, Inc. (Tetra Tech) has prepared this Sampling and Analysis Plan (SAP) for a Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) at Solid Waste Management Unit (SWMU) 27 at Naval Support Activity (NSA) Crane, Indiana. The SAP for the SWMU 27 RFI at NSA Crane was developed under Contract Task Order (CTO) F276, Contract N62470-08-D-1001, Comprehensive Long-Term Environmental Action Navy (CLEAN). **Figure ES-1** shows the locations of the NSA Crane installation and of SWMU 27 within the installation perimeter. SWMU 27 (Illuminant Building 126) is contained within the boundary of NSA Crane and is located in the central-northwest portion of the base. For the purposes of this SAP, SWMU 27 encompasses most of the industrial process area in the general vicinity of Illuminant Building 126, including two discrete Investigation Areas (IAs): IA 1, the Pyrotechnic Production Area (PPA), and IA 2, the former Flare Test Area (see **Figure ES-2**).

The primary purpose of the RFI, is to determine the nature and extent of potential contaminants and impacts associated with industrial activities and operations performed at SWMU 27. The initial field sampling event includes surface and subsurface soil sampling, sampling of catch basin residue, sampling of surface channel material, and minimal groundwater sampling to determine if contamination in site media is present at concentrations above regulatory and risk based screening criteria. If this is the case, additional field events will be completed to determine the extent of contamination in all site media as well as to conduct a baseline risk assessment. The additional field sampling events will be presented in SAP addenda. Due to the industrial nature of SWMU 27, the area is not regarded an ecological habitat, and ecological receptors will not be considered unless contamination has been shown to have migrated beyond the SWMU 27 perimeter.

IA 1 - PPA

Since the early years of World War II, the PPA has produced pyrotechnic devices including signal flares, smoke and dye markers, ground and aircraft signals, submarine signals, and illuminating projectiles such as the 5-inch (star shell) illuminating parachute flare. IA 1 covers approximately 31-acres and is located within restricted access fencing. Although much of NSA Crane is forested, the PPA has been cleared of trees and has been developed as a highly industrialized pyrotechnic production center (see **Figure ES-2**). Building 126 is the primary building in the PPA, and a number of other specialized storage buildings and bunkers, red phosphorus mixing and loading buildings, various curing buildings for pyrotechnic items, and other pyrotechnic production support buildings have been constructed in the PPA as shown on **Figure ES-2**. A metal-plating shop utilizing metals, caustics, acids, and cyanides has been operating on the eastern edge of the PPA (Building 1884).

Drainage swales and ditches convey surface water within the PPA to the south and west (near the point where the railroad track R-1 crosses Highway 99), and throughout the PPA. Additionally, drainage swales adjacent to several concrete basins/pits allowed for collection of surface water overflow if it occurred. In the past, process wastewaters and rinsates from the metal-plating building were discharged to open ditches. Currently, treated wastewater from metal-plating operations is discharged to the sanitary sewer system under a National Pollution Discharge Elimination System (NPDES) permit.

IA 1 - Polychlorinated Biphenyl Transformer Sites

IA 1 RFI sampling will be conducted at the former electrical transformer substation that was located on the western side of the PPA, a replacement transformer located by the north entrance to IA1, and transformers still in use at the SWMU that are labeled as containing PCBs. The transformer located west of Building 124 is co-labeled as containing PCBs and Wecosol®; therefore, area around this transformer will be sampled for both volatile organic compounds (VOCs) and PCBs.

IA 1 - Other Areas of Concern within the PPA

IA 1 RFI sampling will be conducted at locations where fires and explosions have occurred in the past that may have released pyrotechnic compounds to the local environment.

IA 2 - Former Flare Test Area

Little documentation was available regarding the exact location and operation of IA 2. Based on historical photographs and personnel interviews during the September 2009 NSA Crane site visit, three potential flare testing locations north of Highway 5 were identified. This triangular area that contains the three suspected locations covers approximately 7.4-acres (see **Figure ES-2**). Surface water from the former Flare Test Area drains into surface channels along the northern side of Highway 5 or into a culvert north of IA 2.

Purpose and Organization of SWMU 27 UFP-SAP

The RFI includes analysis of environmental samples for pyrotechnic and other related chemicals to determine general risks or hazards posed by these chemicals at SWMU 27. The data, which may be collected through several field events will be presented in the RFI Report and will be used to update and refine the conceptual site model (CSM) as developed and presented in this RFI UFP-SAP. The RFI Report will use the supplemental information to better define and delineate potential contamination source boundaries, to determine the nature of contamination releases to the environment, to describe the pathways/mechanisms for contaminant migration, to identify potential human and ecological receptors (if contamination has migrated off site above Project Screening Levels [PSLs]), and to conclude whether a

determination for No Further Action (NFA), a removal action (RA), or a Corrective Measures Study (CMS) is warranted for SWMU 27.

This RFI Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) was prepared in accordance with the Uniform Federal Policy for Quality Assurance Project Plan (UFP-QAPP) guidelines. There are 37 worksheets required by the guidelines, and all are addressed in some form in this SAP. This document contains the workplan, procedures for field operations and sample analysis, and quality assurance (QA) procedures in one document. The key element involves the planning process. It requires that sponsoring organizations, reviewing/approving organizations, and implementing organizations jointly meet to plan projects and to fully document specific elements of the project planning process. On June 10, 2010, the team met to plan the project. The resulting planning decisions are presented in Worksheet No. 9.

Environmental sampling activities are presented in this RFI UFP-SAP. The RFI UFP-SAP information is presented in the required worksheet format and is focused on presenting the CSMs, documenting the project planning process and development of the data quality objectives (DQOs), and specifying the QA elements built into the data collection and verification/validation process. Some worksheets are supplemented by pages of explanatory text to clearly describe the activities and procedures to be undertaken during this project. In other cases, worksheet text may present introductory discussions on the plan elements as required by the worksheet and reference an appendix of this UFP-SAP for more detailed explanations.

The appendices to this UFP-SAP include the following: Appendix A - Historical SWMU 27 Site Information, Appendix A.3 – June 2010 Site Visit Information, Appendix B - Site-Specific Field Standard Operating Procedures, and Appendix C - Laboratory Standard Operating Procedures.

SAP Worksheets

SAP Worksheet No. 1 -- Title and Approval Page 1
SAP Worksheet No. 2 -- SAP Identifying Information 12
SAP Worksheet No. 3 -- Distribution List 13
SAP Worksheet No. 4 -- Project Personnel Sign-Off Sheet 15
SAP Worksheet No. 5 -- Project Organizational Chart 16
SAP Worksheet No. 6 -- Communication Pathways 17
SAP Worksheet No. 7 -- Personnel Responsibilities and Qualifications Table 20
SAP Worksheet No. 8 -- Special Personnel Training Requirements Table 22
SAP Worksheet No. 9 -- Project Scoping Session Participants Sheet 23
SAP Worksheet No. 10 - Conceptual Site Model 27
SAP Worksheet No. 11 -- Project Quality Objectives/Systematic Planning Process Statements 33
SAP Worksheet No. 12 -- Measurement Performance Criteria Table – Field Quality Control Samples 38
SAP Worksheet No. 13 -- Secondary Data Criteria and Limitations Table 39
SAP Worksheet No. 14 -- Summary of Project Tasks 40
SAP Worksheet No. 15 -- Reference Limits and Evaluation Table 48
SAP Worksheet No. 16 -- Project Schedule / Timeline Table 56
SAP Worksheet No. 17 -- Sampling Design and Rationale 57
SAP Worksheet No. 18 -- Sampling Locations and Methods/SOP Requirements Table 63
SAP Worksheet No. 19 -- Analytical SOP Requirements Table 72
SAP Worksheet No. 20 -- Field Quality Control Sample Summary Table 73
SAP Worksheet No. 21 -- Project Sampling SOP References Table 74
SAP Worksheet No. 22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table 75
SAP Worksheet No. 23 -- Analytical SOP References Table 76
SAP Worksheet No. 24 -- Analytical Instrument Calibration Table 78
SAP Worksheet No. 25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table. 82
SAP Worksheet No. 26 -- Sample Handling System 83
SAP Worksheet No. 27 – Sample Custody Requirements Table 84
SAP Worksheet No. 28 -- Laboratory QC Samples Table 89
SAP Worksheet No. 29 -- Project Documents and Records Table 99
SAP Worksheet No. 30 -- Analytical Services Table 100
SAP Worksheet No. 31 -- Planned Project Assessments Table 101
SAP Worksheet No. 32 -- Assessment Findings and Corrective Action Responses 102
SAP Worksheet No. 33 -- QA Management Reports Table 103
SAP Worksheet No. 34 -- Verification (Step I) Process Table 104
SAP Worksheet No. 35 -- Validation (Steps IIa and IIb) Process Table 105
SAP Worksheet No. 36 -- Analytical Data Validation (Steps IIa and IIb) Summary Table 107
SAP Worksheet No. 37 -- Usability Assessment 108
REFERENCES 111

List of Figures

ES-1 Site Location Map, SWMU 27 - Illuminant Building 126
ES-2 Site Location, IA 1 and IA 2, SWMU 27 - Illuminant Building 126
10-1 Site Location Map, SWMU 27 - Illuminant Building 126
10-2 Site Location, IA 1 and IA 2, SWMU 27 - Illuminant Building 126
10-3 IA 1 - Pyrotechnic Process Area, SWMU 27 - Illuminant Building 126
10-4 IA 1 - Surface Water Flow, SWMU 27 - Illuminant Building 126
10-5 IA 2 - Flare Test Area, SWMU 27 - Illuminant Building 126
10-6 Surface Water Drainage, NSA Crane
10-7 IA 1 - Surface Water Flow, SWMU 27 - Illuminant Building 126
10-8 Conceptual Site Model, Catch Basins, Settling Basins, and Drainageways, SWMU 27 - Illuminant Building 126
10-9 Conceptual Site Model, Flare Testing Activities, SWMU 27 - Illuminant Building 126
10-10 Conceptual Site Model, Polychlorinated Biphenyl (PCB) Transformers, SWMU 27 - Illuminant Building 126
10-11 Conceptual Site Model, Metal-Plating Drainage, SWMU 27 - Illuminant Building 126
17-1 IA 1 - Ditch/Channel/Drainageway Surface Soil Sample Locations, SWMU 27 - Illuminant Building 126
17-2 IA 2 - Ditch/Drainageway Surface Soil Sample Locations, SWMU 27 - Illuminant Building 126
17-3 IA 1 - Flare Test and Fire Incident Area Surface Soil Sample Locations, SWMU 27 - Illuminant Building 126
17-4 IA 2 - Suspected Flare Test Area Proposed Soil Sample Locations, SWMU 27 - Illuminant Building 126
17-5 Pit/Sump/Catch Basin Soil and Residual Sample Locations, SWMU 27 - Illuminant Building 126
17-6 PCB Transformer Surface Soil Sample Locations, SWMU 27 - Illuminant Building 126

Appendices

A – Historical Information
B – Site-Specific Field Standard Operating Procedures
C – Laboratory Standard Operating Procedures

ACRONYMS

ºC	degrees Celsius
µg/L	microgram per liter
A	Analytical
AR	Administrative Record
AWQC	Ambient Water Quality Criterion
BFB	bromofluorobenzene
bgs	below ground surface
BTEX	benzene, toluene, ethylbenzene, and xylenes
CA	Corrective Action
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CMS	Corrective Measures Study
COPC	chemical of potential concern
CSM	conceptual site model
CTO	Contract Task Order
CWAP	Comprehensive Work Approval Process
DCB	decachlorbiphenyl
DCE	dichloroethylene
DL	Detection limit
DO	dissolved oxygen
DoD	Department of Defense
DPT	direct-push technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
Eco-SSL	Ecological Soil Screening Level
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
ERA	Ecological Risk Assessment
ERSM	Environmental Restoration Site Manager
ESL	Ecological Screening Level

EU	Exposure Unit
FID	flame ionization detector
FOL	Field Operations Leader
FTMR	Field Task Modification Request
GC/ECD	Gas Chromotography/Electron Capture Detector
GC/MS	Gas Chromotography/Mass Spectroscopy
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HHRA	human health risk assessment
HI	hazard index
HPLC/ESI/MS	High Performance Liquid Chromotography/Electrospray Ionization/Mass Spectrometry
HSM	Health and Safety Manager
IA	Investigation Area
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICS	Interference Check Standards
ICV	Initial Calibration Verification
ID	identification
I-DCL	Industrial Default Closure Level
IDEM	Indiana Department of Environmental Management
IDW	investigation-derived waste
ILCR	incremental lifetime cancer risk
IAS	Initial Assessment Study
IR	Installation Restoration
IS	Internal Standards
IUPPS	Indiana Underground Plant Protection Services
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LOD	Limit of Detection
LODV	Limit of Detection Verification
LOQ	Limit of Quantitation
MCL	Maximum Contaminant Level
MDL	Minimum Detection Limit
mg/kg	milligram per kilogram
MPC	Measurement Performance Criterion

MS	matrix spike
MSD	matrix spike duplicate
MSDS	Material Data Safety Sheet
msl	mean sea level
NA	Not Applicable
NAVFAC	Naval Facilities Engineering Command
NEESA	Naval Energy and Environmental Support Activity
NELAP	National Environmental Laboratory Accreditation Program
NFA	No Further Action
NIRIS	Naval Installation Restoration Information Solution
NPDES	National Pollution Discharge Elimination System
NRWQC	National Recommended Water Quality Criterion
NSA	Naval Support Activity
NTU	Nephelometric Turbidity Unit
ORNL	Oak Ridge National Laboratory
ORP	oxidation-reduction potential
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
PR	Preliminary Review
PCB	polychlorinated biphenyl
PCE	perchloroethylene
PID	photoionization detector
PM	Project Manager
POC	Point of Contact
PPA	Pyrotechnic Production Area
PPE	personal protective equipment
ppm	part per million
PQLG	Project Quantitation Limit Goal
PQO	project quality objective
PSL	Project Screening Level
PWD	Public Works Department
QA	quality assurance
QAM	Quality Assurance Manager
QAMS	Quality Assurance Management System
QAO	Quality Assurance Officer
QC	quality control
QL	Quantitation Limit

QSM	Quality Systems Manual
%R	percent recovery
RA	Removal Action
RBSSL	Risk-Based Soil Screening Level
RCRA	Resource Conservation and Recovery Act
R-DCL	Residential Default Closure Level
RF	Response Factor
RFI	RCRA Facility Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
R-RSL	USEPA Regions 3, 6, and 9 Residential Regional Screening Level
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RSL	Regional Screening Level
S	Sampling
S&A	Sampling and Analytical
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SPCC	Systems Performance Check Compound
SRCR	Systems Receipt Condition Report
SSO	Site Safety Officer
SWMU	Solid Waste Management Unit
TBD	to be determined
TCE	trichloroethylene
TCMX	Tetra-chloro-meta-xylene
Tetra Tech	Tetra Tech NUS, Inc.
UCL	Upper Confidence Limit
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
UFP-SAP	Uniform Federal Policy for Sampling and Analysis Plan
USCS	Unified Soil Classification System
USEPA	United States Environmental Protection Agency
VC	vinyl chloride
VOA	Volatile Organic Analyte
VOC	volatile organic compound
VSI	Visual Site Inspection

SAP Worksheet No. 2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number:	Naval Support Activity (NSA) Crane
Operable Units:	Solid Waste Management Unit (SWMU) 27
Contractor Name:	Tetra Tech NUS, Inc. (Tetra Tech)
Contract Number:	N62470-08-D-1001
Contract Title:	Comprehensive Long-Term Environmental Action Navy (CLEAN)
Work Assignment Number:	Contract Task Order (CTO) F276

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (USEPA, 2005) and *Guidance for Quality Assurance Project Plans*, United States Environmental Protection Agency (USEPA) QA/G-5, Quality Assurance Management System (QAMS) (USEPA, 2002).

2. Identify regulatory program: Resource Conservation and Recovery Act (RCRA).

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Data Quality Objective (DQO) Meeting	June 10, 2010

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
None	

6. List organizational partners (stakeholders) and connection with lead organization:

Indiana Department of Environmental Management (IDEM) (regulatory oversight), USEPA Region 5 (regulatory oversight), Naval Facilities Engineering Command (NAVFAC) Midwest (property owner), Tetra Tech (Navy contractor)

7. Lead organization: NAVFAC Midwest

8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

   There are no exclusions.

SAP Worksheet No. 3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address	Document Control Number
Howard Hickey	NAVFAC Remedial Project Manager (RPM)/Manager of project activities for NAVFAC	NAVFAC Midwest	847-688-2600 X243	howard.hickey@navy.mil	Not Applicable (NA)
Tom Brent	NAVFAC Public Works Department (PWD) NSA Crane Environmental Restoration Site Manager (ERSM)/Provides support and manages this environmental project as the base Point of Contact (POC)	NAVFAC NSA Crane	812-854-6160	thomas.brent@navy.mil	NA
Jonathan Tucker (electronic upload only)	NAVFAC Project Chemist and Quality Assurance Officer (QAO)/Reviews SAP and quality assurance documentation for NAVFAC	NAVFAC Atlantic	757-322-8288	jonathan.tucker@navy.mil	NA
Bonnie Capito (final cover letter only)	NAVFAC Atlantic Administrative Record (AR) Librarian/Manages ARs for NAVFAC	NAVFAC Atlantic	757-322-4785	bonnie.capito@navy.mil	NA
Doug Griffin	State RPM/Provides state input	IDEM	317-233-2710	dgriffin@idem.in.gov	NA
Peter Ramanauskas	USEPA RPM/provides USEPA overnight	USEPA Region 5	312-866-7890	Ramanauskas.peter@epa.gov	NA
Edward Sedlmyer	Tetra Tech Project Chemist/Conducts data validation and reporting and provides support for laboratory-related issues	Tetra Tech	412-921-8704	edward.sedlmyer@tetratech.com	NA
Ralph Basinski	Crane Activity Coordinator/Coordinates Tetra Tech activities at NSA Crane	Tetra Tech	412-921-8308	ralph.basinski@tetratech.com	NA
John Trepanowski (distribution letter only)	Tetra Tech Program Manager/Manages Navy Initiatives	Tetra Tech	610-382-1532	john.trepanowski@tetratech.com	NA

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address	Document Control Number
Garth Glenn (distribution letter only)	Deputy Program Manager/ Manages Program Activities	Tetra Tech	757-461-3926	garth.glenn@tetratech.com	NA
Rick Barringer, CPG, CHMM, REM	Tetra Tech Project Manager (PM)/Manages project activities on a daily basis	Tetra Tech	412-921-8524	rick.barringer@tetratech.com	NA
To be determined (TBD)	Field Operations Leader (FOL)/Manages Field Operation	Tetra Tech TBD	TBD	TBD	NA
TBD	Site Safety Officer (SSO)/ Oversees site activities to ensure that safety requirements are met	Tetra Tech TBD	TBD	TBD	NA
TBD	Field Operations Technician/Performs Field Sampling Activities	Tetra Tech TBD	TBD	TBD	NA
Kim Kostzer (electronic copy only)	Empirical Laboratories, LLC (Empirical) PM/Representative for laboratory and analytical issues	Empirical	615- 345-1115	kkostzer@empirilabs.com	NA

SAP Worksheet No. 4 -- Project Personnel Sign-Off Sheet

(UFP-QAPP Manual Section 2.3.2)

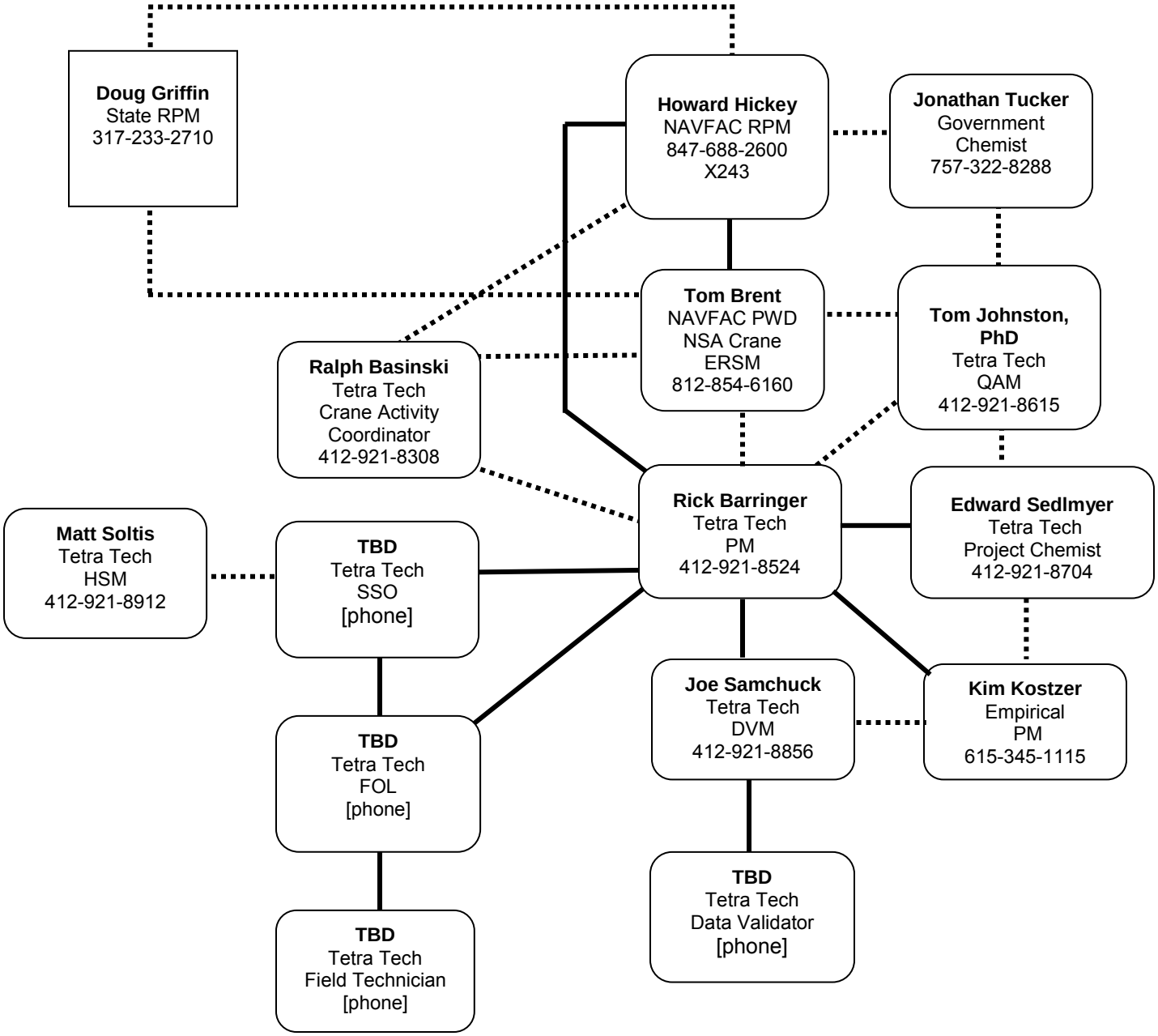
Name[1]	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
TBD	Tetra Tech Field Operations Leader (FOL) and SSO/Manage field operations and oversees site activities to ensure safety requirements are met	TBD		All	
Ralph Basinski	Crane Activity Coordinator/Coordinates Tetra Tech activities at NSA Crane	412-921-8308		All	
Edward Sedlmyer (Electronic copy only)	Tetra Tech Project Chemist/Conducts data validation and reporting and provides support for laboratory-related issues	412-921-8704		All	
Matt Soltis, CIH, CSP (HASP only)	Tetra Tech Project Health and Safety Manager (HSM)/Oversees health and safety activities	412-921-8912		Health and Safety Plan (HASP)	
Joe Samchuck (Electronic copy only)	Tetra Tech Data Validation Manager (DVM)/Oversees data validation activities	412-921-8510		Worksheet Nos. 12, 14, 15, 19, 20, 23-28, 30, and 34-37	
Kim Kostzer	Laboratory PM//Manages project for Empirical	615-345-1115		Worksheet Nos. 6, 12, 14, 15, 19, 20, 23-28, 30, and 34-36	

1 - Persons listed on this worksheet will be responsible for distributing the UFP-SAP to the appropriate people within their organization.

SAP Worksheet No. 5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)

Lines of Authority ——— Lines of Communication



SAP Worksheet No. 6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Driver	Responsible Affiliation	Name	Phone Number and/or E-mail	Procedure
UFP-SAP Amendments/Addenda	Tetra Tech PM Tetra Tech FOL NAVFAC RPM	Rick Barringer TBD Howard Hickey	412-921-8524 TBD 847-688-2600 x243	Tetra Tech FOL will verbally inform Tetra Tech PM within 24 hours of realizing a need for an amendment/addendum. Tetra Tech PM will document the proposed changes via a Field Task Modification Request (FTMR) form within 5 days and send the Navy RPM a concurrence letter within 7 days of identifying the need for change. UFP-SAP amendments will be submitted by Tetra Tech PM to NAVFAC RPM for review and approval. Tetra Tech PM will send scope changes to Project Team via e-mail within 1 business day.
Changes in field work schedule	Tetra Tech PM NAVFAC PWD NSA Crane ERSM NAVFAC RPM Tetra Tech FOL	Rick Barringer Tom Brent Howard Hickey TBD	412-921-8524 812-854-6160 847-688-2600 x243 TBD	Tetra Tech FOL will verbally inform Tetra Tech PM on the day that the issue is discovered. Tetra Tech PM will verbally inform Navy RPM within 1 business day of discovery. Navy RPM will inform IDEM RPM within 1 business day of discovery. Navy RPM will issue scope change (verbally or via e-mail), if warranted; scope change to be implemented before further work is executed. Tetra Tech PM will document the change via a FTMR form within 2 days of identifying the need for change and obtain required approvals within 5 days of initiating the form.

Communication Driver	Responsible Affiliation	Name	Phone Number and/or E-mail	Procedure
Field issues that require changes in field tasks	Tetra Tech FOL Tetra Tech PM NAVFAC PWD NSA Crane ERSM NAVFAC RPM	TBD Rick Barringer Tom Brent Howard Hickey	TBD 412-921-8524 812-854-6160 847-688-2600 x243	Tetra Tech FOL informs PM verbally the day the issue is realized. PM informs the NAVFAC RPM of the issue verbally within 1 day of the FOL's notification. PM also sends a concurrence letter to NAVFAC within 7 days, if project scope is affected. The NAVFAC RPM will sign the letter within 5 days of receipt. Document changes on a FTMR form. Place the form in the project file, with signatures as determined by the PM.
Field issues that require changes in scope of field work	Tetra Tech FOL Tetra Tech PM NAVFAC PWD NSA Crane ERSM NAVFAC RPM	TBD Rick Barringer Tom Brent Howard Hickey	TBD 412-921-8524 812-854-6160 847-688-2600 x243	FOL informs PM verbally the day the issue is realized. PM informs the NAVFAC RPM of the issue verbally within 1 day of the FOL's notification. PM also sends a concurrence letter to the NAVFAC within 7 days, if project scope is affected. The NAVFAC RPM will sign the letter within 5 days of receipt. Document changes on an FTMR form. Place the form in the project file, with signatures as determined by the PM.
Recommendations to stop work and then to initiate work upon corrective action implementation	Tetra Tech FOL Tetra Tech PM NAVFAC RPM NAVFAC PWD NSA Crane ERSM Tetra Tech HSM Tetra Tech QAM Tetra Tech Project Chemist	TBD Rick Barringer Howard Hickey Tom Brent Matt Soltis Tom Johnston, PhD Ed Sedlmyer	TBD 412-921-8524 847-688-2600 x243 812-854-6160 412-921-8912 412-921-8615 412-921-8704	If Tetra Tech is the responsible party for a stop-work command, the Tetra Tech FOL will inform on-site personnel, subcontractor(s), the Navy RPM and base POC, and the identified Project Team members within 1 hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.

Communication Driver	Responsible Affiliation	Name	Phone Number and/or E-mail	Procedure
Field or laboratory data quality issues	Empirical PM Tetra Tech FOL Tetra Tech PM Tetra Tech Project Chemist Tetra Tech DVM	Kim Kostzer TBD Rick Barringer Ed Sedlmyer Joe Samchuck	615-345-1115 TBD 412-921-8524 412-921-8704 412-921-8510	The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within one business day of when an issue related to laboratory data is discovered. The Tetra Tech Project Chemist will notify (verbally or via e-mail) the data validation staff and the Tetra Tech PM within one business day. Tetra Tech DVM or Project Chemist notifies Tetra Tech PM or Tetra Tech FOL verbally or via e-mail within 48 hrs of validation completion that a non-routine and significant laboratory quality deficiency has been detected that could affect this project and/or other projects. The Tetra Tech PM verbally advises the NAVFAC RPM within 24 hours of notification from the project chemist or DVM. The NAVFAC RPM takes corrective action that is appropriate for the identified deficiency. Examples of significant laboratory deficiencies include data reported that has a corresponding failed tune or initial calibration verification. Corrective actions may include a consult with the NAVFAC Navy Chemist.
Corrective action for field program	Tetra Tech QAM Tetra Tech PM	Tom Johnston, PhD Rick Barringer	412-921-8615 412-921-8524	Tetra Tech QAM will notify (verbally or via e-mail) Tetra Tech PM within one business day that the corrective action has been completed. The Tetra Tech PM will then notify (verbally or via e-mail) the Navy RPM within 1 business day.

SAP Worksheet No. 7 -- Personnel Responsibilities and Qualifications Table

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Howard Hickey	NAVFAC RPM/Manages project activities for NAVFAC	NAVFAC Midwest	Oversees project scoping implementation, including data review and evaluation.
Doug Griffin	IDEM RPM/Provides state input	IDEM	Participates in project scoping and implementation, including data review and evaluation, and approves the UFP-SAP.
Tom Brent	NAVFAC PWD NSA Crane ERSM)/Provides support and manages this environmental project as the base POC	NAVFAC NSA Crane	Oversees and coordinates site activities, participates in scoping, data review, and evaluation, and approves the UFP-SAP.
Rick Barringer	Tetra Tech PM/Manages project activities on a daily basis	Tetra Tech	Oversees project, financial, schedule, and technical day-to-day management of the project.
Ralph Basinski	Crane Activity Coordinator/ Coordinates activities at NSA Crane	Tetra Tech	Coordinates Tetra Tech activities at NSA Crane.
TBD	FOL/Manages field operations	Tetra Tech	Supervises, coordinates, and performs field sampling activities.

Name	Title/Role	Organizational Affiliation	Responsibilities
TBD	SSO Oversees site activities to ensure that safety requirements are met	Tetra Tech	Responsible for training and monitoring site conditions. The SSO reports to the HSM and to the Tetra Tech PM. Details of the SSO's responsibilities are presented in the HASP.
Tom Johnston, PhD	QAM/Oversees program and project QA activities	Tetra Tech	Ensures quality aspects of the CLEAN program are implemented, documented, and maintained, oversees preparation of laboratory scope, and conducts data quality reviews. Ensures quality aspects of the CLEAN program.
Joseph Samchuck	DVM/Oversees data validation activities	Tetra Tech	Manages data validation activities within Tetra Tech, including ensuring QA of data validation deliverables, providing technical advice on data usability, and coordinating and maintaining the data validation review schedule.
Matt Soltis	HSM/Oversees health and safety activities	Tetra Tech	Oversees CLEAN Program Health and Safety Program.
Ed Sedlmyer	Project Chemist/ Coordinates laboratory-related functions and conducts data validation and reporting	Tetra Tech	Participates in project scoping, prepares laboratory scopes of work, and coordinates laboratory-related functions with laboratory. Conducts data quality reviews and QA of data validation deliverables.
Kim Kostzer	Laboratory PM/Manages project for Empirical	Empirical	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.

In some cases, one person may be designated responsibilities for more than one position. For example, the FOL may also be responsible for SSO duties. This action will be performed only as credentials, experience, and availability permits.

SAP Worksheet No. 8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

Each site worker will be required to have completed a 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) course (and 8-hour refresher training, if applicable) in health and safety training as described under Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120(e). Safety requirements are addressed in greater detail in the site-specific Tetra Tech HASP (2010).

SAP Worksheet No. 9 -- Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name: NSA Crane SWMU 27 RFI Projected Date(s) of Sampling: March 2011 Project Manager: Rick Barringer	Site Name: SWMU 27 – Illuminant Building 126 Site Location: Crane, Indiana

Date of Session: June 10, 2010
Scoping Session Purpose: DQO Meeting

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Rick Barringer	PM	Tetra Tech	513-557-5057	rick.barringer@tetratech.com	Management
Valerie Plachy	Project Engineer	Tetra Tech	412-921-8389	valerie.plachy@tetratech.com	Management
Tom Brent	NAVFAC PWD NSA Crane ERSM	NAVFAC PWD	812-854-6160	thomas.brent@navy.mil	Management
Doug Griffin	IDEM RPM	IDEM	317-233-2710	dgriffin@idem.in.gov	State RPM
Peggy Churchill	DQO Facilitator	Tetra Tech	321-636-6470 x1300	peggy.churchill@tetratech.com	DQO Facilitation
Ralph Basinski	Crane Activity Coordinator	Tetra Tech	412-921-8308	ralph.basinski@tetratech.com	Management/ Oversight

Comments/Decisions:

Initial data collection efforts occurred during a September 16 to 18, 2009, site visit to NSA Crane. SWMU 27 historical use and available data were discussed. A Trip Report is provided in Appendix A.

The steps for the RFI include multiple field sampling events in accordance with the UFP-SAP format were discussed.

The DQO meeting was conducted on June 10, 2010. The meeting participants developed project quality objectives (PQOs) using USEPA's seven-step DQO process. A site visit/walk was conducted to review proposed sample location. The rationale for the selection of the sample locations and analytical parameters for each location was discussed and consensus was obtained. Tetra Tech was assigned the task of preparing a draft UFP-SAP. Detailed meeting discussion and decision items are presented in Appendix A.

CONSENSUS ITEMS:

1. Analysis for red phosphorus is not required because it will not persist in the environment, and it has been assigned to the unproblematic environmental category (German Environmental Protection Agency) based upon the March 1990 Assessment of Red Phosphorus in the Environment by the US

Army Biomedical Research and Development Laboratory at Fort Detrick. Fredrick, Maryland (Mitchell and Burrows, 1990).

2. The metal-plating process (Building 1884) is of concern regarding historical potential use of metals and chlorinated solvents.

3. SWMU 27 (PPA and suspected flare test area north of Highway 5) does not provide ecological habitat because it is located in an industrial area of the facility.

4. SWMU 27 likely does not impact ecological receptors in Boggs Creek and Lake Gallimore.

5. Detection of off-site contamination during the RFI could require ecological risk screening. If an ecological risk screening is required, it will be addressed in an addendum.

6. The residue material found in the drainageways and channels within SWMU 27 should be referred to as soil not sediment.

7. Drainageway and channel soil/material samples will be collected and composited from the 0 feet below ground surface (bgs) to 1 foot bgs.

8. Phase I samples for soil surrounding the catch basins will be four-point composite samples (one grab from each side) except for Pit 126B.

9. Phase I Pit 126B samples will be two four-point composite samples along each side of the concrete.

10. A residual material four-point composite sample will be collected from the interior of all non-active catch basins. The active catch basins are Pit 133, 122, 130, 2698, 1885, 1886, and Pit 126A.

11. All Phase I catch basin samples will include one basin exterior composite soil sample at the catch basin low point from 1 foot bgs to the depth of the catch basin.

12. Phase I samples for soil surrounding the PCB transformer pads will be four-point composite samples (one grab from each side) except for the transformer west of Building 124 and the former transformer area.

13. Phase I former transformer area samples will be two four-point composite samples along each side of the concrete.

14. The investigation of each suspected flare test area north of Highway 5 will consist of two four-point composite samples if the area is not covered with asphalt.

15. Asphalted suspected flare test areas north of Highway 5 will not be sampled during Phase I unless a direct-push technology (DPT) rig is required for other sample locations.

16. Volatile organic compounds (VOCs) rather than total petroleum hydrocarbons and benzene, toluene, ethylbenzene, and xylenes (BTEX) will be included in the analytical program.

17. Catch basin, residual sump material, and drainageway/channel soil samples will be analyzed for:
    - VOCs
    - Perchlorate
    - Cyanide (Only in The Plating Shop area and down channel of The Plating Shop)
    - Select metals:
      - Aluminum
      - Barium
      - Cadmium
      - Total Chromium
      - Copper
      - Lead
      - Magnesium
      - Manganese
      - Potassium
      - Strontium
      - Sodium
      - Zinc

18. Samples from PCB transformer areas will be analyzed for:
    - PCBs:
      - Aroclor-1016
      - Aroclor-1221
      - Aroclor-1232
      - Aroclor-1242
      - Aroclor-1248
      - Aroclor-1254
      - Aroclor-1260
      - Aroclor-1262
      - Aroclor-1268
      - Total Aroclor

19. Samples from suspected flare test areas and fire incident areas will be analyzed for:
    - Perchlorate
    - Select metals:
      - Aluminum
      - Barium
      - Cadmium
      - Total Chromium
      - Copper

- Lead
- Magnesium
- Manganese
- Potassium
- Strontium
- Sodium
- Zinc

Historical Reports:

PR (Preliminary Review)/VSI (Visual Site Inspection). Naval Weapons Support Center, Crane, Indiana. USEPA ID IN5170023498. Prepared for U.S. Environmental Protection Agency, Region V. Prepared by A.T. Kearney, Inc. USEPA Contract No. 68-01-7038. Work Assignment KOS-02-45. March 1987.

IAS (Initial Assessment Study), 1983. Naval Energy and Environmental Support Activity (NEESA), 1983. Initial Assessment Study of Naval Weapons Support Center Crane. Crane, Indiana. NEESA 13-003. May. (1983)

Action Items: Tetra Tech assigned task of preparing preliminary draft UFP-SAP.

Historical Information:

- Disposition Form SMCCN-IOE (200-1a) for Building 126.
- Disposition Form SMCCN-EDP for PPA.
- Ordnance Briefing Program for Buildings 146 and 126.
- Material Data Safety Sheet (MSDS) for Building 198.
- NSWC Crane 2009 Rule 6 Annual Report.
- PPA historical photographs dated: 1952, 1958, and 1966 and three figures not dated received from Dough Johnson on September 16, 2009.
- PW 3902 – Process Flow Diagram. Plating Shop Waste Treatment System.

Detailed historical Information is presented in Appendix A.

SAP Worksheet No. 10 - Conceptual Site Model

(UFP-QAPP Manual Section 2.5.2)

This worksheet presents general background information about SWMU 27 – Illuminant Building 126 and a conceptual site model (CSM) that describes potential contamination routes and possible exposure pathways. The CSM served as the basis for developing this UFP-SAP.

10.1 PHYSICAL SITE DESCRIPTION

SWMU 27 – Illuminant Building 126 is located in the north-central portion of NSA Crane (see **Figure 10-1**). For the purposes of this UFP-SAP, the Site is an expansion of the Illuminant Building 126 that includes two IAs, IA 1, the PPA, and IA 2, the former Flare Test Area (see **Figure 10-2**).

The PPA (IA 1) is trapezoid shape that is bounded by Highway 5 on the north and Highway 99 on the south. The PPA produces pyrotechnics devices such as flares. IA 1 covers approximately 31-acres and is located within a restricted access fencing (see **Figure 10-3**). While most of NSA Crane is forested, AOC 2 is industrialized. The southern, western, and northwestern portions of IA 1 are grassland (see **Figure 10-2**). IA 1 surface water flows towards Highway 99. The nearest streams are to the west and drain into the Boggs Creek Watershed. Turkey Creek is east of the PPA, but drain swales and local topography convey surface flow in the PPA to the south and west (near the point where the railroad track R-1 crosses Highway 99). Throughout the PPA are various concrete basins/pits (**Figure 10-3**) that previously flowed to surface water drainage changes and have overflowed onto surface soil. The PPA is an active production area with a chain link fence bordering the area. Access is controlled by two locked gates; one on the north and the other on the east portion of the fence. **Figure 10-4** presents the PPA surface water flow.

The former Flare Test Area (IA 2) is represented as a rectangle that is located north of Highway 5. IA 2 covers approximately 2.6-acres and is not a fenced area (See **Figure10-5**). Surface water from the former Flare Test Area drains into channels that parallel the north side of Highway 5 or down the culvert north of IA 2. **Figure 10-7** presents the surface water flow for the former Flare Test Area.

As shown on **Figure 10-4**, highest elevation within the PPA (800 feet above mean sea level [msl]) is the manmade mound for the PPA Wastewater Treatment Plant (Building 3064).

Physical structures at the PPA (see **Figure 10-3**) are presented in Appendix A, **Table A.1-1**. These structures included pyrotechnic production (Buildings 122, 123, 126, 130, and 2697), red phosphorous pressing house (Building 133), laboratory (Building 127), plating and maintenance buildings (Building 1884), phosphorous mixing buildings (Buildings 1885 and 1886), a wastewater treatment plant (Building 3064), numerous storage magazines/buildings, curing buildings, and miscellaneous inactive buildings. Empty containers are stored west, east, and southeast of Buildings 1885 and 1886.

Condensate from steam traps and condensate collection systems flow onto surface soil west of Building 126, north of Building 2697, south of Building 3296, and west of Building 122. Various surface drains with unknown discharge points exist throughout the PPA (north of Building 122, southwest of Building 1884, north of Building 127, southeast of Building 3297, and west of Building 133).

An electrical transformer labeled as containing PCBs is located on a concrete pad north of Building 126; a second electrical transformer with dual labeling as containing PCBs and Wecosol® is located on a concrete pad west of Building 124 (see **Figure 10-3**). A former electric substation that contained PCB transformers was located in the current fenced area across the road west of Building 133 (see **Figure 10-3**). A replacement transformer located near the north entrance to IA 1, is suspected to be a replacement for a transformed that may have contained PCBs (see **Figure 10-3**).

10.2 SITE HISTORY AND BACKGROUND

The PPA consists of a complex of buildings the majority of which were designed and built during World War II for the exclusive purpose of loading, assembling, and packing out star shell ammunition to supply both the Atlantic and Pacific fleets with a variety of pyrotechnic devices including illuminating projectiles, parachute flares, and star shells (see **Figure 10-7**). The star shell, primarily a 5-inch illuminating round with a parachute, required significant support facilities. The star shell production process was as follows:

- Building 121 (second floor) was used for parachute fabrication.
- Buildings 123, 124, and 125 were used for metal part generation (machining and assembly).
- Building 126 (Illuminant Building) was used for preparation of the illumination composition and its pressing into “candles.”
- Building 122 was the final assembly building where the candle (pyrotechnic package) was inserted into the projectile, the parachute was attached and packed, and the whole round was packed out for rail shipment from the plant.
- Several smaller buildings that surround Building 126 were used for chemical storage, candle curing, ready magazines, and inert support warehouses necessary to maintain the process.

Additional buildings were constructed in the PPA after 1942 to support the war effort and later operations, including production of:

- Marker flares, rescue flares, parachute flares
- Illuminating projectiles, incendiaries
- Smoke signals, spotting charges
- Ground and aircraft signals, submarine signals
- Aircraft signal cartridges and depth charge markers

The majority of Naval experimental work associated with pyrotechnic devices was conducted at the NSA Crane PPA.

Illuminant Building 126 is classified as an Installation Restoration (IR) program site by the Navy. According to descriptions of this IR site, red phosphorus was discharged to sump pits where it used to overflow to tributaries of Boggs Creek. Heavy metal contamination from zinc and cadmium has also been identified in open ditches that eventually drain into Boggs Creek. No remedial investigations have been performed for this IR site; therefore, the extent and location of potential contamination have not been delineated. An outdoor burning area was located near Building 126, and another area was located north of Highway 5. Open burning operations at SWMU 27 ceased in 1971.

A metal-plating shop utilizing metals, caustics, acids, and cyanides has been operating on the eastern edge of the PPA (Building 1884). Process wastewaters and rinsates were at one time discharged to open ditches and later to building sumps that may have overflowed into ditches that drain to the south and west into the Boggs Creek watershed. Heavy metal contamination (zinc and cadmium) has been detected in wastewater that was previously discharged into open ditches. Currently, the treated wastewater from metal-plating operations is discharged to the sanitary sewer system under an NPDES permit.

The following summarizes the site-specific information in the Navy's NORM (normalized) database and other relevant SWMU 27 information from NAVFAC:

- Contamination at this site included red phosphorus, chlorate, dyes, oxidizers, and fuels for flares and smoke munitions. Although red phosphorus is probably the most significant contaminant, it is not of concern because red phosphorus does not linger in the environment and is assigned to the unproblematic environmental category.

- Building 126 used sump pits that were pumped out by trucks, and the pumped materials were taken to the burning grounds.

- All sump overflows drained into the Boggs Creek watershed.

- A metal-plating shop that used heavy metals, caustics, acids, and cyanide-based processes is present at the site, and significant heavy metal contamination (zinc and calcium) has been detected in wastewater being discharged into open ditches. However, since November 1992, the plating shop has not used cyanide.

- Two previous burn areas have been identified; one behind Building 126 and one across Highway 5 (PPA). Burning in those areas ceased about 1971.

Historic Fire/Incidents

Building 127 was initially used as a supporting laboratory for the work in Building 126. The northern end of Building 127 once housed a burn chamber where, in the 1960s, experiments were conducted to evaluate the relationship of candle density to illuminating round performance. An explosion occurred when a burn was being conducted with a very low density press loaded candle, and the explosion destroyed the burn chamber.

A Former Curing Building was located north of Building 126 where candles were cooked off and cured. The Former Curing Building was destroyed by a fire in the late 1980s that was traced to a malfunctioning heating unit that overheated. Building 3298 is located in the same general location as the destroyed curing building.

A flash fire occurred in Building 130 (phosphorus weighing building) when an employee attempted to empty red phosphorus from a barrel. The post-incident investigation indicated that although the plastic bag lining the barrel was not conductive, the red phosphorus fines inside the bag generated a spark that ignited the red phosphorus.

A flash fire occurred in Building 1886 during experimental procedures to improve the mixing effort. Red phosphorus, boron, lead dioxide, and a binder material were added into a mixer bowl in an order that differed from the test procedure.

A detailed discussion of the site history and buildings history is provided in Appendix A.

10.3 CONCEPTUAL SITE MODEL

Spills and releases from general site activities (pyrotechnic device production, red phosphorous pressing, and metal plating), sump overflows, flare testing, and PCB transformer maintenance at the PPA are the likely sources of potential contamination. The historical operations at the site and information from

previous site investigations indicate that releases likely occurred directly onto the surface and have potentially migrated into site soil or off site via surface water flow.

PPA Site Activity

The pyrotechnic devices are produced from metal, mixtures of oxidizing agents, fuels, and dyes. Examples of oxidizing agents include chlorates, perchlorates, peroxides, chromates, and nitrates, examples of fuels include aluminum and magnesium powder and their alloys, sulfur, lactose, and other easily oxidizable materials, and examples of dyes include red and yellow dye.

The phosphorus pressing process used red phosphorous, manganese dioxide, magnesium, zinc oxide, and linseed oil.

A CSM schematic for pits, sumps, catch/settling basins, and drainageways is presented as **Figure 10-8**.

PPA Flare Testing Activities

Metals, oxidizing agents, fuels, and dyes are flare constituents. However, with the exception of metals, the majority of these constituents are consumed during the testing process. Additionally, Buildings 124 and 125 maintenance activities, such as parts washing, likely utilized petroleum-based solvents that may have contained VOCs.

A flare testing activities CSM schematic is presented as **Figure 10-9**.

PCB Transformers

No historical information regarding transformer maintenance was available; therefore, potential releases of PCB during transformer maintenance activities could not be eliminated.

Several transformers are PCB-label and one transformer located west of Building 124 is labeled with both PCB and Wecosol®, a chlorinated VOC that is also used in the dry cleaning industry.

A PCB transformers CSM schematic is presented as **Figure 10-10**.

Metal-Plating Operations

The plating operations utilized acid and alkaline cleaning, chrome conversion coating, phosphate coating, and cadmium/zinc cyanide plating on a variety of munitions-related metal parts. The Building 1884 metal finishing operations included the use of solvent for degreasing that may have contained VOCs.

The metal-plating wastewaters and rinsates may have discharged into open ditches near Building 1884 and may have impacted surface soil.

A metal-plating CSM schematic is presented as **Figure 10-11**.

Conclusions

These releases and potential releases to the soil may (1) present complete exposure pathways to human receptors and/or (2) serve as a source of contamination to groundwater and present complete exposure pathways to human receptors through those routes. The PR/VSI Report concluded that there are low release potentials to surface water due to the sanitary sewer system discharge (A.T. Kearny, 1987). Additionally, surface water flow is intermittent; it is highly unlikely that surface water will be present during the sampling events, and it is unlikely that current SWMU 27 activities could be impacting the surface water. Therefore, the impacts of contamination from the Site surface waters reaching Boggs Creek and/or Turkey Creek will not be investigated. However, potential historical releases to site soil prior to connection to the sanitary sewer system may act as contaminant reservoirs for migration to groundwater. Potential human exposure pathways are illustrated on **Figures 10-8 through 10-11**.

Human receptors include persons currently employed at the site or future site construction workers who could interact with contaminated media. The area is rural, and there are no identified residences within 1 mile of the site. However, because future land use is unknown, it is customary to conservatively evaluate future use of a property for residential and recreational purposes. Therefore, potential future receptors include residents and people recreating at the site. Human receptors may be exposed to different media following various pathways based on their specific activities. These media include surface and subsurface soil, groundwater, surface runoff, and sediment within IA1 and IA2. However, just beyond the site boundary there is a potential for ecological receptors. Therefore, potential ecological receptors include terrestrial plants and invertebrates, mammals and birds, and aquatic organisms. Ecological receptors may be exposed to different media following various pathways based on their specific activities. These media include surface soil, surface water, and sediment.

The exposure media for ecological receptors are typically surface soil, sediment, and surface runoff. Because SWMU 27 is largely industrialized, ecological exposure is not considered significant at the site and will not be evaluated. As previously stated, surface water flow is intermittent; it is highly unlikely that surface water will be present during the sampling events, and it is unlikely that current SWMU 27 activities could be impacting the surface water. Therefore, exposure of ecological receptors to surface and subsurface soil, sediment, surface runoff, and groundwater is not anticipated and will not be evaluated.

SAP Worksheet No. 11 -- Project Quality Objectives/Systematic Planning Process Statements

(UFP-QAPP Manual Section 2.6.1)

This section describes the development of PQOs using USEPA's seven-step DQO/Systematic Planning Process.

11.1 PROBLEM DEFINITION

The PPA is currently active and contains multiple warehouses, storage bunkers, manufacturing facilities, and curing buildings. Based on the site history and CSM, it is likely that site-related contaminants are present in environmental media at SWMU 27 at concentrations that could exceed applicable risk-based human health screening values. During the initial RFI field event, site media will be investigated to determine if contamination is identified at levels that exceed risk-based screening criteria in the areas most likely to be contaminated, such as pits, transformer pads, and drainageways. If contamination is identified at concentrations greater than the screening criteria, the project team will return to the site in order to complete delineation of such contamination and to conduct a human health risk assessment (HHRA) for on-site receptors.

Should contamination be present off site (beyond the fenceline) in environmental media at SWMU 27 at concentrations that exceed applicable risk-based ecological screening values, then ecological risks will be evaluated.

The risk assessment and delineation sampling events of the investigation will be presented in an addendum to this SAP.

11.2 INFORMATION INPUTS

The following chemical and physical data are needed to attain project objectives:

1. Chemical Data: Fixed-base laboratory soil and groundwater concentrations of VOCs, perchlorate, metals, PCBs, and cyanide are necessary to determine if contamination is present at the site. The list of target analytes and associated Project Screening Levels (PSLs) for each matrix are presented in Worksheet No. 15. The sampling methods are presented in Worksheet No. 18, and the analytical methods are presented in Worksheet No. 19.

2. PSLs: Chemical data will be collected and compared to risk based and regulatory PSLs. The surface and subsurface soil PSLs are set at the lowest matrix-specific risk-based or regulatory human health criteria appropriate for the site.

The risk-based and regulatory criteria applicable for the PPA and former Flare Test Area include USEPA Regions 3, 6, and 9 Screening Levels for chemical contaminants at Superfund Sites (USEPA, 2010), and IDEM R-DCLs (IDEM, 2009).

Analytical data reported by the laboratory use the following reporting conventions: All results below the Detection Limit (DL) will be considered non-detections; Positive results reported at concentrations between the DL and Limit of Quantitation (LOQ) will be reported with a “J” qualifier; and analytes not found (not detected) in a sample will be reported as the Limit of Detection (LOD) with a "U" qualifier.

To conduct comparisons of site data to screening values for soil and groundwater, the subcontracted laboratory should be able to achieve DLs that are low enough to measure constituent concentrations less than the PSLs. In some cases, this may not be achievable. The rationale for allowing these deviations is described in the footnotes to Worksheet No. 15. The Project Team will accept the laboratory analytical results for these parameters when the PSL is greater than the laboratory’s LOQ. Also, in cases where the PSL is between the laboratory’s Limit of Detection (LOD) and LOQ, the Project Team will accept these analytical results when the results are “J” qualified, that is, when they are qualified as estimated values. When the PSL is less than the LOD for a particular analyte or analytes, an evaluation of DLs and the impact on data usability will be discussed in the RFI Report. Any limitations on the data will be documented at that time and, if significant data gaps remain, additional data may be required during additional field events or in support of future reports, (e.g., Corrective Measure Study [CMS]).

If there are no analyte detections above PSLs and there are only “U” values for a compound with PSLs below laboratory LOD for a given method, no further investigation of that compound will be conducted in subsequent sampling events.

3. Physical parameters for groundwater: Field measurements of water quality parameters including pH, specific conductance, oxidation-reduction potential (ORP), temperature, dissolved oxygen (DO), and turbidity are necessary at all groundwater sample locations. These field measurements (excluding turbidity) will be made using a YSI 600-Series Environmental Monitoring System or equivalent type of instrument. Turbidity measurements will be made using a LaMotte 2020 turbidity meter or equivalent.

4. Background: The background data set for soils at NSA Crane will be used to determine whether concentrations of metals present on site are from naturally occurring sources or are site related. Background data for the various soil types identified at NSA Crane are described in the Final Base-Wide Background Soil Investigation Report for NSWC Crane (Tetra Tech, 2001).

11.3 STUDY AREA BOUNDARIES

The study will address the entire area of SWMU 27 that has been expanded to include IA 1 (PPA) and IA 2 (former Flare Test Area). The following items address the horizontal, vertical, and temporal boundaries of the study:

1. Horizontal: The initial horizontal boundary of the study is defined as the outer perimeter of the area where certain process operations and flare testing took place. These areas have been located based on information from aerial photographs and previous investigations and include the active and inactive catch basins/pits, former Flare Test Area, PCB transformer areas, and drainageway/channel soils (**Figures 10-3 and 10-5**). Lateral expansion of this horizontal study boundary may be required in during subsequent sampling events, if any contamination at levels exceeding human health PSLs is identified during this phase of the investigation.

   If any contamination just inside the site boundary identified during the initial sampling event of the investigation is at levels exceeding the ecological PSLs and are determined to be significant, then expansion of the horizontal study boundary maybe be require during subsequent sampling events.

2. Vertical (Soil): The vertical boundary of the study includes the surface soil interval of interest (0 to 1 foot bgs for ditch, channel, and drainageway locations and 0 to 2 feet bgs for all other surface soil locations). Data from surface soil surrounding the catch basins/pits, former Flare Test Area, and PCB transformer areas are representative of soil that may have been impacted due to activities during normal site operations or potential overflow of the catch basins. Data collected from drainageway/channel soils are representative of surface soil in the 0- to 1- foot bgs interval that may have been impacted by runoff from the active areas of the site. The initial subsurface soil population of interest includes subsurface soil impacted by a potential release from catch basins/pits due to overflow of a pit or a leak in the pit structure. The subsurface soil vertical boundary will extend 2 feet beyond the identified depth of the catch basins/pits (approximately 8 feet bgs). The depth of the sample will be the difference between the internal sidewall height and external sidewall height plus 2 feet, that is, 2 feet below the bottom of the basin.

3. Vertical (groundwater): Groundwater will be investigated in the transformer area adjacent to Buildings 124 and 125, the Wecosol® labeled transformer. Because there are no known monitoring wells in the vicinity, depth to groundwater is currently unknown, but it is presumed to be less than 15 feet bgs. Therefore, the depth of the study will extend to the top of the water table, which is estimated at less than 15 feet bgs at the site.

Groundwater sampling for this transformer will focus on the chlorinated VOCs [PCE (perchloroethylene) and PCE degradation compounds (trichloroethylene {TCE}, cis-1,2-dichloroethylene {DCE}, trans-1,2-DCE, and vinyl chloride {VC})].

4. Temporal: With the exceptions of phosphorus and perchlorate, all target analyte concentrations are anticipated to be relatively unchanged (stable) over the course of time needed to conduct the environmental investigations and into the foreseeable future; therefore, no temporal constraints exist. SWMU 27 RFI field activities are scheduled for early 2011.

If contamination is identified during this investigation, additional field sampling events will be required and an addendum to this UFP-SAP will be submitted identifying the expanded boundaries

11.4 ANALYTICAL APPROACH

Due to limitations in analytical sensitivity, some analytes cannot be detected at their PSL. These analytes are shown as bolded and shaded on Worksheet No. 15. If these analytes are reported as nondetected with U-qualifiers, they will be treated as results below the project action limit for decision making.

The decision statements for the study are as follows:

Determine whether a target analyte concentration in soil or groundwater exceed the applicable PSL for that target analyte and media within and around certain process areas that, based on historical site knowledge and the CSM, are the most likely areas to have been impacted by those target analytes at SWMU 27.

- If all target analyte concentrations in site media are less than PSLs, then the Project Team will recommend no further action (NFA) for the site.

- If any target analyte is detected in site media at a maximum concentration that exceeds the PSL and is greater than the site-specific background concentration (for metals), then the target analyte will be identified as a chemical of potential concern (COPC) and the data will be evaluated by the Project Team to determine the degree to which “step-out” samples (vertical and/or horizontal) are necessary during subsequent sampling events to define the vertical and/or horizontal extent of COPC contamination. Ideally, “step-out” samples will be collected until perimeter sample concentrations are less than PSLs; however, professional judgment will be used to determine if such samples are needed in all cases. Factors considered will include the specific target analyte, magnitude of the exceedance(s), spatial distribution, and overall estimated risk level. The results of initial sampling

and any professional judgments made regarding the need for further investigation to delineate contamination and to collect data for a baseline risk assessment will be reviewed with the Project Team prior to submitting an addendum to this UFP-SAP.

- If the only analytes detected above PSLs are benzene, ethylbenzene, toluene, and xylenes in locations where these detections are likely derived from railroad ties and automotive activities, no further investigation of these compounds will be conducted in subsequent sampling events.

11.5 PERFORMANCE OR ACCEPTANCE CRITERIA

Because the biased sampling locations were strategically selected to locate potential contamination based on former use, probability limits for false positive and false negative decision errors were not established. Simple comparisons of measured concentrations to PSLs are initially being used. The Project Team will use the measured results to determine whether the amount and type of data collected are sufficient to support the attainment of project objectives. This will involve an evaluation of contaminant concentrations and an evaluation of uncertainty for contaminants that have PSLs less than the LODs to ensure that contaminants are likely to have been detected if present. If all data have been collected as planned and no data points are missing or rejected for quality reasons, the sampling event completeness will be considered satisfactory. If any data gaps are identified, including missing or rejected data, the Project Team will assess whether project objectives have been achieved. This assessment will depend on the number and type of identified data gaps; therefore, a more detailed strategy cannot be presented at this time. All Project Team stakeholders will be involved in rendering the final conclusion regarding adequacy of the data.

11.6 DATA COLLECTION PLAN

The sampling design and rationale for the initial sampling event to be collected are provided in Worksheet No. 17. The sampling design and rationale for subsequent field sampling events will be developed, as necessary, based on the results of the previous sampling event and presented in an addenda to this document.

SAP Worksheet No. 12 -- Measurement Performance Criteria Table – Field Quality Control Samples

(UFP-QAPP Manual Section 2.6.2)

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPCs)	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Equipment Rinsate Blank(1)	All fractions	One per 20 field samples per matrix per type of sampling equipment(1)	Bias/Contamination	No analytes >½ LOQ, except common laboratory contaminants, which must be < LOQ.	S & A
Trip Blank	VOCs	One per cooler containing VOC samples shipped to laboratory	Bias/Contamination	No analytes >½ LOQ, except common laboratory contaminants, which must be < LOQ.	S & A
Field Duplicate (FD)	All fractions	One per 20 field samples per matrix	Precision	Values > 5x LOQ: Relative Percent Difference (RPD) ≤30% (aqueous) (2,3) ≤50% (solids) (2,3).	S & A
Cooler Temperature Indicator	All fractions	One per cooler	Representative	Temperature must be less than or equal to 6 degrees Celsius (ºC) (4 ± 2 ºC).	S
Filtered Rinsate Blank4	Dissolved Metals (if necessary due to high turbidity)	One per filter brand	Bias/Contamination	No analytes >½ LOQ, except common laboratory contaminants, which must be < LOQ.	S & A

1 Equipment rinsate blanks will be collected.
2 If duplicate values are < 5x LOQ for aqueous samples, absolute difference should be < 2x LOQ.
3 If duplicate values are < 5x LOQ for solid samples, absolute difference should be < 4x LOQ.
4 A filtered rinsate blank will be collected if filtered samples are collected.

SAP Worksheet No. 13 -- Secondary Data Criteria and Limitations Table

(UFP-QAPP Manual Section 2.7)

Secondary Data	**Data Source** (originating organization, report title and date)	**Data Generator(s)** (originating organization, data types, data generation/ collection dates)	**How Data Will Be Used**	**Limitations on Data Use**
None	NA	NA	NA	No secondary data will be used.

SAP Worksheet No. 14 -- Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

14.1 FIELD INVESTIGATION TASK PLAN

The field tasks are summarized below. A short description of these tasks is also provided.

- Mobilization/demobilization
- Site-Specific Health and Safety Training
- Utility Clearance
- DPT borings
- Monitoring Equipment Calibration
- Surface and Subsurface Soil Sampling
- Groundwater Well Installation and Development*
- Water Level Measurements*
- Groundwater Sampling
- Investigation-Derived Waste (IDW) Management
- Global Positioning System (GPS) Locating (see SOP-01)
- Field Decontamination Procedures
- Field Documentation Procedures
- Sample Custody and Shipment Tasks
- QC Tasks

Mobilization/Demobilization

Mobilization will consist of the delivery of all equipment, materials, and supplies to the site, complete assembly in satisfactory working order of all such equipment at the site, and satisfactory storage at the site of all such materials and supplies. Tetra Tech will coordinate with the Navy to identify appropriate locations for the storage of equipment and supplies. Site-specific health and safety training for field team will be provided as part of site mobilization.

Demobilization will consist of the prompt and timely removal of all equipment, materials, and supplies from the site following completion of the work.

Site-Specific Health and Safety Training

There are no specialized/non-routine project-specific training requirements or certifications needed by personnel to successfully complete the project or tasks. All field personnel will have appropriate training

to conduct the field activities to which they are assigned. Each site worker will be required to have completed the OSHA 40-hour course (and 8-hour refresher, if applicable) in health and safety training. Safety requirements are addressed in greater detail in the site-specific HASP.

Utility Clearance

One week prior to the commencement of any subsurface intrusive activities, the Tetra Tech FOL or designee will contact Indiana Underground Plant Protection Services (IUPPS) to complete a utility clearance ticket for the areas under investigation. Work permits, if required by the facility, will be obtained prior to conducting field activities. The Tetra Tech FOL will be responsible for coordinating these activities.

DPT Boring

DPT (e.g., Geoprobe®) will be used to collect surface and subsurface soil samples from the unconsolidated overburden. A new acetate liner will be used for each 2-foot section of soil core. Each removed soil core will be scanned for VOCs. The soil core will be visually inspected and logged by the field geologist, and the soil texture, grain size (sand, silt, or clay), color (and any unusual discoloration), moisture content, and soil type will be identified by the field geologist based on the Unified Soil Classification System (USCS).

For samples requiring VOC analysis, the VOC sample will be collected from the point along the soil core that had the greatest PID reading using an Encore™ sampler. If no PID readings are greater than background, the VOC sample will be collected from any discolored area of the soil or from the midpoint of the core. For drainageway soil samples:

- From 0 to 1 foot bgs, if neither the PID readings are greater than background nor are visibly discolored, then the VOC sample will be collected at 6 inches from the top of the soil core.

- From 0 to 2-foot bgs, if neither the PID readings are greater than background nor are visibly discolored, then the VOC sample will be collected at a distance that is ¾ from the top of the soil core.

- Greater than 2 feet bgs (e.g., subsurface samples), if neither the PID readings are greater than background nor are visibly discolored, then the VOC sample will be collected at a distance that is ¾ from the top of the soil core.

The Encore™ samples will be collected from each core immediately after PID readings are collected. After the sample for VOC analysis has been collected, remaining aliquot will be placed in a disposable

mixing bowl, rocks, gravel, and other coarse debris will be removed, and the sample will be mixed using a disposable spoon. The appropriate sample jars for remaining analyses will then be filled and properly labeled. The bowl and spoon will be discarded after each sample is collected. Disposable (not stainless steel) bowls and spoons are to be used and appropriately discarded.

Soil borings will be backfilled with the soil cuttings.

When a boring has been sampled and backfilled, it will be identified by a tall wooden lathe driven into the soil near the boring. In addition, a 2- by 2-inch wooden stake will be driven into the center of the backfilled boring. The stake and the lathe will both have brightly colored flagging attached to them to increase visibility (for the survey crew), and both will be labeled using a waterproof marker with a unique soil boring number corresponding to the boring log.

Monitoring Equipment Calibration

Monitoring equipment calibration procedures are described in Worksheet No. 22.

Sample Collection Tasks

Site-specific Standard Operating Procedures (SOPs) have been developed for field activities at NSA Crane, including sample collection tasks (Appendix B). Sample labeling will be in accordance with SOP 02 (Sample Labeling), and the sample numbering scheme will be in accordance with SOP-03 (Sample Identification and Nomenclature). Methods for recording data will be in accordance with SOP-04 (Sample Custody and Documentation of Field Activities, Appendix B), and the selection of sample containers, sample preservation, packaging, and shipping will be in accordance with SOP-05 (Sample Preservation, Packaging, and Shipping).

The sampling and analysis program is outlined in Worksheet No. 18, and the sampling requirements for each type of analysis (i.e., bottleware, preservation, holding time) are listed in Worksheet No. 19. Field and laboratory QC samples will also be collected as outlined in Worksheet No. 20.

Surface and Subsurface Soil Sampling

Soil samples will be collected in accordance with SOP-07 (Soil Coring and Sampling Using Hand Auger Techniques, Appendix B) and SOP-11 (Subsurface Soil Sampling Using DPT). Surface soil samples (from 0 to 2 feet bgs) will be collected with a hand auger, and sample jars will be filled using a dedicated disposable plastic trowel. Soil from drainageways/channels (from 0 to 1 feet bgs) will be collected with a hand auger, and sample jars will be filled using either a decontaminated stainless-steel trowel or

dedicated disposable plastic trowel. Because of the unknown thickness and quality of the suspected gravel layer beneath the resurfaced and asphalt areas in AO 02, a backhoe may be used to excavate the gravel layer and expose the surface soil layer for sampling. Subsurface soil samples will be collected using a DPT, DPT rig with augering capabilities, concrete coring device, backhoe, or stainless- steel hand auger and stainless-steel or disposable trowel. The subsurface soil borings will be described by the Site Geologist in accordance with SOP-08 (Soil Sample Logging) and will be screened for evidence of contamination with a PID. Use of the PID will be in accordance with the manufacturer's instructions. Any qualitative visual signs of potential contamination (such as soil staining) will be noted on the soil boring log.

Groundwater Sampling

The temporary groundwater well installation procedure discussed in SOP-11 (Subsurface Soil and Groundwater Sampling Using Direct-Push Technology) and SOP-12 (Monitoring Well Installation) and the groundwater development procedure discussed in SOP-13 (Monitoring Well Development) will be followed. The groundwater sampling procedures discussed in SOP-14 (Measurement of Water Levels), SOP-15 (Low Flow Well Purging and Stabilization), SOP-16 (Monitoring Well Sampling), and SOP-17 (Calibration and Care of Water Quality Meters) will be followed as necessary.

Investigation-Derived Waste Management

It is not anticipated that significant volumes of solid or semi-solid IDW in the form of soil or sediment will be generated during field activities, including the collection of subsurface samples using DPT or backhoe excavations or the installation of temporary groundwater monitoring wells. At the direction of the Navy POC, excavation soil will be replaced into the excavation from which it was removed.

IDW that is generated, including personal protective equipment (PPE) and decontamination fluids, will be handled in accordance with SOP-09 (Management of Investigation-Derived Waste).

Global Positioning System Locating

A GPS unit will be used to locate all sampling points in accordance with SOP-01 (Global Positioning System Data Collection and Transfer). The GPS equipment will be checked on control monuments before and after each day's use, and these checks will be documented in the field notebook.

Field Decontamination Procedures

Sample containers will be provided certified clean (I-Chem 300 or equivalent) from Empirical. Decontamination of sampling equipment may not be necessary for this project if only dedicated and

disposable hand trowels will be used. However, if decontamination is necessary, the requirements outlined in this section will apply. Decontamination of reusable sampling equipment (e.g., non-disposable hand trowels, hand augers, or DPT or backhoe equipment) will be conducted prior to sampling and between samples at each location. Decontamination of equipment will be conducted according to the sequence established in SOP-06 (Decontamination of Field Sampling Equipment).

If a backhoe is used, decontamination of the excavator bucket will be performed over the completed backfilled excavation using a high-pressure spray washer with water supplied by the base. All decontamination water will be containerized and disposed into the Crane sewer system at a location designated by the NSA Crane ERSM.

Field Documentation Procedures

Field documentation will be performed in accordance with SOP-04 (Sample Custody and Documentation of Field Activity).

Sample Custody and Shipment Tasks

Sample custody and shipment tasks are defined in SOP-05 (Sample Preservation, Packaging, and Shipping) and are discussed in Worksheet No. 27.

Quality Control Tasks

QA/QC samples will be collected at frequencies listed in Worksheet No. 12.

14.2 ADDITIONAL PROJECT-RELATED TASKS

Additional project-related tasks include:

- Analytical tasks
- Data generation procedures
- Data handling and management
- Data tracking and control
- Assessment and oversight
- Data review
- Project reports

Analytical Tasks

Empirical is a current Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) accredited laboratory. A copy of the laboratory certification for Empirical can be found in Appendix C. Analyses will be performed in accordance with the analytical methods identified in Worksheet No. 19. Empirical is expected to meet the PSLs to the extent identified in Worksheet No. 15. Empirical will perform chemical analysis following laboratory-specific SOPs (Worksheet Nos. 19 and 23) developed based on the analytical methods listed in Worksheet Nos. 19 and 30. Copies of the laboratory SOPs are included in Appendix B. All solid results will be reported by the laboratory on a dry-weight basis. Results of percent moisture will be reported in each analytical data package and electronic data files. This information will also be captured in the project database that will eventually be uploaded to the Naval Installation Restoration Information Solution (NIRIS). Percent moisture information will also be included in the RFI Report.

The analytical data packages provided by Empirical will be in a Contract Laboratory Program-like format and will be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method-specific QC (results, recoveries, RPDs, relative standard deviations, and/or percent differences etc.).

Data Generation Procedures

- Project documentation and records include the following:
  - Field sample collection and field measurement records as described in Worksheet Nos. 27 and 29.
  - Laboratory data package deliverables as described in the analytical specifications.
  - Data assessment documents and records as listed in Worksheet No. 29.
- Data recording formats are described in Worksheet No. 27.

Data Handling and Management - After the RFI is completed, the field sampling log sheets will be organized by date and medium and filed in the project files. The field logbooks for this project will be used only for this site and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field sampling activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. The data handling procedures to be followed by Empirical will meet the requirements of the technical specifications. The electronic data results will be automatically downloaded into the Tetra Tech database in accordance with the proprietary Tetra Tech processes.

Data Tracking and Control - The Tetra Tech PM (or designee) is responsible for the overall tracking and control of data generated for the project.

- **Data Tracking**. Data are tracked from generation to archiving in the Tetra Tech project-specific files. The Tetra Tech Project Chemist (or designee) is responsible for tracking the samples collected and shipped to Empirical. Upon receipt of the data packages from Empirical, the Tetra Tech Project Chemist will oversee the data validation effort, which includes verifying that the data packages are complete and that results for all samples have been delivered by Empirical.

- **Data Storage, Archiving, and Retrieval**. The data packages received from Empirical are tracked in the data validation logbook. After the data are validated, the data packages are entered into the Tetra Tech Navy CLEAN file system and archived in secure files. The field records including field logbooks, sample log sheets, chain-of-custody records, and field calibration logs will be submitted by the Tetra Tech FOL to be entered into the Navy CLEAN file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract, the records will be stored by Tetra Tech.

- **Data Security**. Access to Tetra Tech project files is restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Tetra Tech Data Manager maintains the electronic data files, and access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

Assessment and Oversight – Refer to Worksheet No. 32 for assessment findings and corrective actions and to Worksheet No. 33 for QA Management Reports.

Data Review - Data verification is described in Worksheet No. 34, data validation is described in Worksheet Nos. 35 and 36, and the usability assessment is described in Worksheet No. 37.

Project Reports – Draft and final versions of project reports will be prepared and submitted to the Navy and IDEM for review. The reports will include the following sections:

- Executive Summary – will include a brief description of the work conducted and the findings.

- Introduction and Background – will include a description of the history of operations and activities at the site and a summary of any previous investigations and removal actions.

- Description of Field Investigations – will include a summary of the work performed in accordance with the approved UFP-SAP, any approved UFP-SAP addenda, and any field modifications as documented by the Tetra Tech FOL. This section will include maps showing the sampling locations and tables summarizing the data collected.

- Data Quality – will include a summary of quantitative analytical performance indicators such as completeness, precision, bias, and sensitivity and qualitative indicators such as representativeness and comparability. This section will also include a reconciliation of project data with the DQOs and identification of deviations from this UFP-SAP.

  A data usability assessment will be used to identify significant deviations in analytical performance that could affect the ability to meet project objectives. The elements of this review are presented in Worksheet No. 37.

- Nature and Extent of Contamination – will include a discussion of the contamination detected in each medium sampled in relation to the CSM of the site. This section will note the removals previously conducted (if applicable), contamination addressed, and any additional contaminants found during this field effort. Detected contaminant concentrations will be tabulated for each medium and depicted on maps.

- Contaminant Fate and Transport – will include a description of the contaminants detected and their behavior in soil, bedrock, groundwater, surface water, and sediment, particularly with emphasis on the future migration of these contaminants to any possible exposure areas.

- Summary and Conclusions – includes a summary of the findings, conclusions as to whether delineation of contamination is adequate, and recommendations for further investigations, if needed.

The final version of the report will submitted in hardcopy and electronic format to the project stakeholders.

SAP Worksheet No. 15 -- Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soils
Analytical Groups: VOCs, PCBs, Metals, and Miscellaneous Parameters

Analyte	CAS Registry Number	PSL (mg/kg)			PSL References[5]	PQLG[6] (mg/kg)	Empirical		
		HHRA[1,2]	ERA[3,4]	Min			LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
VOCs									
1,1,1-Trichloroethane	71-55-6	1.9	29.8	1.9	IDEM R-DCL/ R5 ESL	0.6	0.005	0.0025	0.0013
1,1,2,2-Tetrachloroethane	**79-34-5**	**0.00052**	**0.127**	**0.00052**	**USEPA RBSSL/ R5 ESL**	**0.0002**	**0.005**	**0.0025**	**0.0013**
1,1,2-Trichlorotrifluoroethane	76-13-1	3000	---	3000	USEPA RBSSL	1000	0.005	0.0025	0.0013
1,1,2-Trichloroethane	**79-00-5**	**0.00156**	**28.6**	**0.00156**	**USEPA RBSSL/ R5 ESL**	**0.0005**	**0.005**	**0.0025**	**0.0013**
1,1-Dichloroethane	75-34-3	0.0138	20.1	0.0138	USEPA RBSSL/ R5 ESL	0.005	0.005	0.0025	0.0013
1,1-Dichloroethene	75-35-4	0.058	8.28	0.058	IDEM R-DCL/ R5 ESL	0.02	0.005	0.0025	0.0013
1,2,4-Trichlorobenzene	120-82-1	0.136	11.1	0.136	USEPA RBSSL/ R5 ESL	0.05	0.005	0.0025	0.0013
1,2-Dibromo-3-chloropropane	**96-12-8**	**2.8E-06**	**0.035**	**2.8E-06**	**USEPA RBSSL/ R5 ESL**	**0.0000009**	**0.005**	**0.0025**	**0.0013**
1,2-Dibromoethane	**106-93-4**	**0.000036**	**1.23**	**0.000036**	**USEPA RBSSL/ R5 ESL**	**0.00001**	**0.005**	**0.0025**	**0.0013**
1,2-Dichlorobenzene	95-50-1	7.2	2.96	2.96	USEPA RBSSL/ R5 ESL	1	0.005	0.0025	0.0013
1,2-Dichloroethane	**107-06-2**	**0.00084**	**21.2**	**0.00084**	**USEPA RBSSL/ R5 ESL**	**0.0003**	**0.005**	**0.0025**	**0.0013**
1,2-Dichloropropane	**78-87-5**	**0.0026**	**32.7**	**0.0026**	**USEPA RBSSL/ R5 ESL**	**0.0009**	**0.005**	**0.0025**	**0.0013**
1,3-Dichlorobenzene	541-73-1	2.3	37.7	2.3	IDEM R-DCL/ R5 ESL	0.8	0.005	0.0025	0.0013
1,4-Dichlorobenzene	106-46-7	0.0082	0.546	0.0082	USEPA RBSSL/ R5 ESL	0.003	0.005	0.0025	0.0013
2-Butanone	78-93-3	30	89.6	30	USEPA RBSSL/ R5 ESL	10	0.01	0.005	0.0025
2-Hexanone	591-78-6	0.22	12.6	0.22	USEPA RBSSL/ R5 ESL	0.07	0.01	0.005	0.0025

Analyte	CAS Registry Number	PSL (mg/kg)			PSL References[5]	PQLG[6] (mg/kg)	Empirical		
		HHRA[1,2]	ERA[3,4]	Min			LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
4-Methyl-2-pentanone	108-10-1	9	443	9	USEPA RBSSL/ R5 ESL	3	0.01	0.005	0.0025
Acetone	67-64-1	28	2.5	2.5	IDEM R-DCL/ R5 ESL	0.8	0.01	0.005	0.0025
Benzene	**71-43-2**	**0.0042**	**0.255**	**0.0042**	**USEPA RBSSL/ R5 ESL**	**0.001**	**0.005**	**0.0025**	**0.0013**
Bromodichloromethane	**75-27-4**	**0.00064**	**0.54**	**0.00064**	**USEPA RBSSL/ R5 ESL**	**0.0002**	**0.005**	**0.0025**	**0.0013**
Bromoform	75-25-2	0.046	15.9	0.046	USEPA RBSSL/ R5 ESL	0.02	0.005	0.0025	0.0013
Bromomethane	74-83-9	0.044	0.235	0.044	USEPA RBSSL/ R5 ESL	0.01	0.01	0.005	0.0025
Carbon disulfide	75-15-0	6.2	0.094	0.0941	USEPA RBSSL/ R5 ESL	0.03	0.005	0.0025	0.0013
Carbon tetrachloride	**56-23-5**	**0.0034**	**2.98**	**0.0034**	**USEPA RBSSL/ R5 ESL**	**0.001**	**0.005**	**0.0025**	**0.0013**
Chlorobenzene	108-90-7	1.24	13.1	1.24	USEPA RBSSL/ R5 ESL	0.4	0.005	0.0025	0.0013
Chloroethane	75-00-3	0.65	---	0.65	IDEM R-DCL	0.2	0.01	0.005	0.0025
Chloroform	**67-66-3**	**0.00106**	**1.19**	**0.00106**	**USEPA RBSSL/ R5 ESL**	**0.0004**	**0.005**	**0.0025**	**0.0013**
Chloromethane	74-87-3	0.98	10.4	0.98	USEPA RBSSL/ R5 ESL	0.3	0.01	0.005	0.0025
cis-1,2-Dichloroethene	156-59-2	0.4	0.784	0.4	IDEM R-DCL/ R5 ESL	0.1	0.005	0.0025	0.0013
cis-1,3-Dichloropropene	**10061-01-5**	**0.003**	**0.398**	**0.003**	**USEPA RBSSL/ R5 ESL**	**0.001**	**0.005**	**0.0025**	**0.0013**
Cyclohexane	110-82-7	69	---	69	IDEM R-DCL	23	0.005	0.0025	0.0013
Dibromochloromethane	**124-48-1**	**0.00078**	**2.05**	**0.00078**	**USEPA RBSSL/ R5 ESL**	**0.0003**	**0.005**	**0.0025**	**0.0013**
Dichlorodifluoromethane	75-71-8	12.2	39.5	12.2	USEPA RBSSL/ R5 ESL	4	0.01	0.005	0.0025
Ethylbenzene	100-41-4	0.034	5.16	0.034	USEPA RBSSL/ R5 ESL	0.01	0.005	0.0025	0.0013
Isopropylbenzene	98-82-8	11	---	11	IDEM R-DCL	4	0.005	0.0025	0.0013
Methyl acetate	79-20-9	150	---	150	USEPA RBSSL	50	0.01	0.005	0.0025
Methyl tert-butyl ether	1634-04-4	0.056	---	0.056	USEPA RBSSL	0.02	0.005	0.0025	0.0013

Analyte	CAS Registry Number	PSL (mg/kg)			PSL References[5]	PQLG[6] (mg/kg)	Empirical		
		HHRA[1,2]	ERA[3,4]	Min			LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Methylcyclohexane	108-87-2	---	---	---	---	---	0.005	0.0025	0.0013
Methylene chloride	75-09-2	0.023	4.05	0.023	IDEM R-DCL/ R5 ESL	0.008	0.01	0.005	0.0025
Styrene	100-42-5	3.5	4.69	3.5	IDEM R-DCL/ R5 ESL	1	0.005	0.0025	0.0013
Tetrachloroethene	**127-18-4**	**0.00098**	**9.92**	**0.00098**	**USEPA RBSSL/ R5 ESL**	**0.0003**	**0.005**	**0.0025**	**0.0013**
Toluene	108-88-3	12	5.45	5.45	IDEM R-DCL/ R5 ESL	2	0.005	0.0025	0.0013
trans-1,2-Dichloroethene	156-60-5	0.62	0.784	0.62	USEPA RBSSL/ R5 ESL	0.2	0.005	0.0025	0.0013
trans-1,3-Dichloropropene	**10061-02-6**	**0.003**	**0.398**	**0.003**	**USEPA RBSSL/ R5 ESL**	**0.001**	**0.005**	**0.0025**	**0.0013**
Trichloroethene	79-01-6	0.0144	12.4	0.0144	USEPA RBSSL/ R5 ESL	0.005	0.005	0.0025	0.0013
Trichlorofluoromethane	75-69-4	16.6	16.4	16.4	USEPA RBSSL/ R5 ESL	5	0.01	0.005	0.0025
Vinyl chloride	**75-01-4**	**0.000112**	**0.646**	**0.000112**	**USEPA RBSSL/ R5 ESL**	**0.00004**	**0.01**	**0.005**	**0.0025**
Xylenes (Total)	1330-20-7	4	10	4	USEPA RBSSL/ R5 ESL	1	0.015	0.0075	0.0038
PCBs									
Aroclor-1016	**12674-11-2**	**0.39**	**0.0003**	**0.000332**	**USEPA RSL/ R5 ESL**	**0.0001**	**0.007**	**0.0033**	**0.002**
Aroclor-1221	**11104-28-2**	**0.0024**	**0.0003**	**0.000332**	**USEPA RBSSL/ R5 ESL**	**0.0001**	**0.007**	**0.0033**	**0.002**
Aroclor-1232	**11141-16-5**	**0.0024**	**0.0003**	**0.000332**	**USEPA RBSSL/ R5 ESL**	**0.0001**	**0.007**	**0.0033**	**0.002**
Aroclor-1242	**53469-21-9**	**0.106**	**0.0003**	**0.000332**	**USEPA RBSSL/ R5 ESL**	**0.0001**	**0.007**	**0.0033**	**0.002**
Aroclor-1248	**12672-29-6**	**0.104**	**0.0003**	**0.000332**	**USEPA RBSSL/ R5 ESL**	**0.0001**	**0.007**	**0.0033**	**0.002**
Aroclor-1254	**11097-69-1**	**0.176**	**0.0003**	**0.000332**	**USEPA RBSSL/ R5 ESL**	**0.0001**	**0.007**	**0.0033**	**0.002**
Aroclor-1260	**11096-82-5**	**0.22**	**0.0003**	**0.000332**	**USEPA R-RSL/ R5 ESL**	**0.0001**	**0.007**	**0.0033**	**0.002**
Aroclor-1262	**37324-23-5**	**0.22**	**0.0003**	**0.000332**	**USEPA R-RSL/ R5 ESL**	**0.0001**	**0.007**	**0.0033**	**0.002**

Analyte	CAS Registry Number	PSL (mg/kg)			PSL References[5]	PQLG[6] (mg/kg)	Empirical		
		HHRA[1,2]	ERA[3,4]	Min			LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aroclor-1268	**11100-14-4**	**0.22**	**0.0003**	**0.000332**	**USEPA R-RSL/ R5 ESL**	**0.0001**	**0.007**	**0.0033**	**0.002**
Metals									
Aluminum	7429-90-5	7700	50	50	USEPA RSL/ EPA OAKRIDGE	16.7	10	5	2.5
Barium	7440-39-3	1500	1.04	1.04	USEPA R-RSL/ R5 ESL	0.35	0.25	0.1	0.05
Cadmium	7440-43-9	7	0.36	0.36	USEPA R-RSL/ Eco SSL	0.12	0.25	0.1	0.05
Chromium	**7440-47-3**	**0.017**	**26**	**0.017**	**USEPA RBSSL/ Eco SSL**	**0.0057**	**0.25**	**0.2**	**0.1**
Copper	7440-50-8	310	28	28	USEPA R-RSL/ Eco ESL	9.3	0.5	0.4	0.25
Lead	7439-92-1	400	11	11	USEPA R-RSL/ Eco SSL	3.7	0.15	0.15	0.075
Manganese	7439-96-5	180	220	180	USEPA R-RSL/ Eco SSL	60.0	0.75	0.3	0.15
Strontium	7440-24-6	4700	---	4700	USEPA R-RSL	1570	1.2	0.6	0.3
Zinc	7440-66-6	2300	46	46	USEPA R-RSL/ Eco SSL	15.3	1	0.5	0.25
Miscellaneous Parameters									
Perchlorate	14797-73-0	55	---	55	USEPA/R-RSL/R5 RSL	18.3	0.02	0.01	0.005
Cyanide	57-12-5	0.94	1.3	0.94	IDEM R-DCL/ R5 RSL	0.31	0.5	0.25	0.125

Acronyms
CAS - Chemical Abstracts Service.
Min - minimum.

1 HHRA Project Screening Levels apply to soils and catch basin residues within the SWMU 27 fenceline.
2 The lowest of the human health screening criteria of USEPA Regional Screening Level for residential direct contact with soil and risk-based soil screening level for protection of groundwater, (May 2010) or IDEM Residential Default Closure Level (2009) was selected as the PSL for human health exposure.
3 ERA Project Screening Levels apply to soils beyond the SWMU 27 fenceline.

4 The ecological PSL is based on the following hierarchy: USEPA Ecological Soil Screening Levels (Eco-SSL) (2005-2007), then USEPA Region 5 Ecological Screening Level (R5 ESL) (2003), then Oak Ridge National Laboratory (ORNL) (1998) screening levels. No criteria are listed when Eco-SSL, R5 ESL, or ORNL criteria are not available.

5 Surface soil screening level references: R-RSL and RBSSL – USEPA Regional Screening Level (RSL) Table (May 2010); IDEM R-DCL – IDEM Default Closure Level Table (2009); R5 ESL – USEPA Region 5 Ecological Screening Level (2005-2007); Eco SSL – USEPA's Eco–SSLs (2008a).

6 Project Quantitation Limit Goal (PQLG) is one-third of the minimum PSL.

Bolding indicates that the minimum PSL is greater than the laboratory LOD but less than the laboratory LOQ.
Bolding and shading indicates that the PSL is less than the laboratory LOD.

All results will be reported to DLs and any limitations on data use that result from having DLs greater than PSLs will be described in the RFI Report.

Matrix: Water
Analytical Groups: VOCs, PCBs, Metals, and Miscellaneous Parameters

Analyte	CAS Registry Number	PSL (µg/L)			PSL References[5]	PQLG[(6)] (µg/L)	Empirical		
		HHRA[(1,2)]	ERA[(3,4)]	Min			LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
VOCs									
1,1,1-Trichloroethane	71-55-6	200	76	76	USEPA MCL/R5 ESL	25	1	0.5	0.25
1,1,2,2-Tetrachloroethane	**79-34-5**	**0.067**	**380**	**0.067**	**USEPA TAP/**R5 ESL	**0.022**	**1**	**0.5**	**0.25**
1,1,2-Trichlorotrifluoroethane	76-13-1	140	---	140	USEPA VAPOR/R5 ESL	47	1	0.5	0.25
1,1,2-Trichloroethane	**79-00-5**	**0.24**	**500**	**0.24**	**USEPA TAP/**R5 ESL	**0.08**	**1**	**0.5**	**0.25**
1,1-Dichloroethane	75-34-3	2.4	47	2.4	USEPA TAP/R5 ESL	0.8	1	0.5	0.25
1,1-Dichloroethene	75-35-4	7	65	7	USEPA MCL/R5 ESL	2	1	0.5	0.25
1,2,4-Trichlorobenzene	**120-82-1**	**0.41**	**30**	**0.41**	**USEPA TAP/**R5 ESL	**0.14**	**1**	**0.5**	**0.25**
1,2-Dibromo-3-chloropropane	**96-12-8**	**0.00032**	**---**	**0.00032**	**USEPA TAP**	**0.0001**	**2**	**1**	**0.5**
1,2-Dibromoethane	**106-93-4**	**0.0065**	**---**	**0.0065**	**USEPA TAP**	**0.002**	**1**	**0.5**	**0.25**
1,2-Dichlorobenzene	95-50-1	37	14	14	USEPA TAP/R5 ESL	5	1	0.5	0.25
1,2-Dichloroethane	**107-06-2**	**0.15**	**910**	**0.15**	**USEPA TAP/**R5 ESL	**0.05**	**1**	**0.5**	**0.25**
1,2-Dichloropropane	**78-87-5**	**0.39**	**360**	**0.39**	**USEPA TAP/**R5 ESL	**0.13**	**1**	**0.5**	**0.25**
1,3-Dichlorobenzene	541-73-1	80	38	38	IDEM R-DCL/R5 ESL	13	1	0.5	0.25
1,4-Dichlorobenzene	**106-46-7**	**0.43**	**9.4**	**0.43**	**USEPA TAP/**R5 ESL	**0.1**	**1**	**0.5**	**0.25**
2-Butanone	78-93-3	710	220	220	USEPA TAP/R5 ESL	73.33	10	5	2.5

Analyte	CAS Registry Number	PSL (µg/L)			PSL References[5]	PQLG[(6)] (µg/L)	Empirical		
		HHRA[(1,2)]	ERA[(3,4)]	Min			LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
2-Hexanone	**591-78-6**	**4.7**	**99**	**4.7**	**USEPA TAP/**R5 ESL	**1.6**	**5**	**2.5**	**1.5**
4-Methyl-2-pentanone	108-10-1	200	170	170	USEPA TAP/R5 ESL	57	10	5	2.5
Acetone	67-64-1	2200	1700	1700	USEPA TAP/R5 ESL	567	10	5	2.5
Benzene	**71-43-2**	**0.41**	**114**	**0.41**	**USEPA TAP/**R5 ESL	**0.14**	**1**	**0.5**	**0.25**
Bromodichloromethane	**75-27-4**	**0.12**	**---**	**0.12**	**USEPA TAP/**R5 ESL	**0.04**	**1**	**0.5**	**0.25**
Bromoform	75-25-2	8.5	230	8.5	USEPA TAP/R5 ESL	2.8	2	1	0.5
Bromomethane	**74-83-9**	**0.87**	**16**	**0.87**	**USEPA TAP/**R5 ESL	**0.29**	**1**	**0.5**	**0.25**
Carbon disulfide	75-15-0	100	15	15	USEPA TAP/R5 ESL	5	1	0.5	0.25
Carbon tetrachloride	**56-23-5**	**0.4**	**240**	**0.4**	**USEPA VAPOR/**R5 ESL	**0.1**	**1**	**0.5**	**0.25**
Chlorobenzene	108-90-7	9.1	47	9.1	USEPA TAP/R5 ESL	3.0	1	0.5	0.25
Chloroethane	75-00-3	62	---	62	IDEM R-DCL/R5 ESL	21	1	0.5	0.25
Chloroform	**67-66-3**	**0.19**	**140**	**0.19**	**USEPA TAP/**R5 ESL	**0.06**	**1**	**0.5**	**0.25**
Chloromethane	74-87-3	19	---	19	USEPA TAP	6	1	0.5	0.25
cis-1,2-Dichloroethene	156-59-2	37	970	37	USEPA TAP/R5 ESL	12	1	0.5	0.25
cis-1,3-Dichloropropene	**10061-01-5**	**0.43**	**---**	**0.43**	**USEPA TAP**	**0.14**	**1**	**0.5**	**0.25**
Cyclohexane	110-82-7	100	---	100	USEPA VAPOR	33	1	0.5	0.25
Dibromochloromethane	**124-48-1**	**0.15**	**---**	**0.15**	**USEPA TAP**	**0.05**	**1**	**0.5**	**0.25**
Dichlorodifluoromethane	75-71-8	1.5	---	1.5	USEPA VAPOR	0.5	1	0.5	0.25
Ethylbenzene	100-41-4	1.5	14	1.5	USEPA TAP/R5 ESL	0.5	1	0.5	0.25
Isopropylbenzene	98-82-8	68	---	68	USEPA TAP	23	1	0.5	0.25
Methyl acetate	79-20-9	3700	---	3700	USEPA TAP	1233	2	1	0.5
Methyl tert-butyl ether	1634-04-4	12	---	12	USEPA TAP	4	1	0.5	0.25
Methylcyclohexane	108-87-2	---	---	---	---	---	1	0.5	0.25
Methylene chloride	75-09-2	4.8	---	4.8	USEPA TAP	1.6	4	2	1
Styrene	100-42-5	100	32	32	USEPA MCL/R5 ESL	11	1	0.5	0.25
Tetrachloroethene	**127-18-4**	**0.11**	**45**	**0.11**	**USEPA TAP/**R5 ESL	**0.04**	**1**	**0.5**	**0.25**
Toluene	108-88-3	230	253	230	USEPA TAP/R5 ESL	77	1	0.5	0.25
trans-1,2-Dichloroethene	156-60-5	11	970	11	USEPA TAP/R5 ESL	4	1	0.5	0.25
trans-1,3-Dichloropropene	10061-02-6	---	---	---	---	---	1	0.5	0.25
Trichloroethene	79-01-6	2	47	2	USEPA TAP/R5 ESL	0.7	1	0.5	0.25
Trichlorofluoromethane	75-69-4	18	---	18	USEPA VAPOR	6	1	0.5	0.25

Analyte	CAS Registry Number	PSL (µg/L)			PSL References[5]	PQLG[(6)] (µg/L)	Empirical		
		HHRA[(1,2)]	ERA[(3,4)]	Min			LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Vinyl chloride	**75-01-4**	**0.016**	**930**	**0.016**	**USEPA TAP/R5 ESL**	**0.01**	**1**	**0.5**	**0.25**
Xylenes (Total)	1330-20-7	20	27	20	USEPA TAP/R5 ESL	7	4	2	1
PCBs									
Aroclor-1016	**12674-11-2**	**0.26**	**0.00012**	**0.00012**	**USEPA TAP/R5 ESL**	**0.00004**	**0.5**	**0.25**	**0.125**
Aroclor-1221	**11104-28-2**	**0.0068**	**0.00012**	**0.00012**	**USEPA TAP/R5 ESL**	**0.00004**	**0.5**	**0.25**	**0.125**
Aroclor-1232	**11141-16-5**	**0.0068**	**0.00012**	**0.00012**	**USEPA TAP/R5 ESL**	**0.00004**	**0.5**	**0.25**	**0.125**
Aroclor-1242	**53469-21-9**	**0.034**	**0.00012**	**0.00012**	**USEPA TAP/R5 ESL**	**0.00004**	**0.5**	**0.25**	**0.125**
Aroclor-1248	**12672-29-6**	**0.034**	**0.00012**	**0.00012**	**USEPA TAP/R5 ESL**	**0.00004**	**0.5**	**0.25**	**0.125**
Aroclor-1254	**11097-69-1**	**0.034**	**0.00012**	**0.00012**	**USEPA TAP/R5 ESL**	**0.00004**	**0.5**	**0.25**	**0.125**
Aroclor-1260	**11096-82-5**	**0.034**	**0.00012**	**0.00012**	**USEPA TAP/R5 ESL**	**0.00004**	**0.5**	**0.25**	**0.125**
Metals									
Aluminum	**7429-90-5**	**3700**	**87**	**87**	**USEPA TAP/USEPA NRWQC**	**29**	**200**	**100**	**50**
Barium	7440-39-3	730	220	220	USEPA TAP/R5 ESL	73	40	20	10
Cadmium	**7440-43-9**	**1.8**	**0.25**	**0.25**	**USEPA TAP/USEPA NRWQC**	**0.1**	**1.5**	**0.5**	**0.25**
Chromium	**7440-47-3**	**0.043**	**11**	**0.043**	**USEPA TAP/USEPA NRWQC**	**0.01**	**1.25**	**1**	**0.5**
Copper	7440-50-8	150	9	9	USEPA TAP/USEPA NRWQC	3	1.25	8	10
Lead	**7439-92-1**	**15**	**2.5**	**2.5**	**USEPA MCL/USEPA NRWQC**	**0.8**	**3**	**3**	**1.5**
Manganese	7439-96-5	88	---	88	USEPA TAP	29	15	10	5
Strontium	7440-24-6	2,200	---	2200	USEPA TAP	733	6	3	1.5
Zinc	7440-66-6	1100	120	120	USEPA TAP/USEPA NRWQC	40	20	10	5
Miscellaneous Parameters									
Perchlorate	14797-73-0	2.6	---	2.6	USEPA TAP	0.9	1	0.5	0.2
Cyanide	57-12-5	73	5.2	5.2	USEPA TAP/R5 ESL	1.7	0.02	0.01	0.005

µg/L - Milligrams per kilogram.
NRWQC - National Recommended Water Quality Criterion.

1 HHRA Project Screening Levels apply waters within the SWMU 27 fenceline.
2 The lowest of the human health screening criteria of the USEPA Maximum Contaminant Level, USEPA Regional Screening Level for tap water, or USEPA VAPOR was selected as the PSL for human health exposures.
3 ERA PSLs apply to waters beyond the SWMU 27 fenceline.
4 The ecological PSL is based on the hierarchy: USEPA NRWQCs (formerly, USEPA AWQCs) then USEPA R5 ESLs. NC (no criterion) is listed when a USEPA NRWQC or USEPA R5 ESL is not available.
5 Groundwater screening levels references: USEPA MCL - 2009 Edition of the Drinking Water Standards and Health Advisories (2009); USEPA TAP – USEPA Regional Screening Level Table (May 2010); USEPA VAPOR – OSWER Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air from Groundwater and Soils (Subsurface Vapor Intrusion Guidance) (2002); USEPA NRWQC – National Recommended Water Quality Criteria (2009); R5 ESL – USEPA Region 5 Ecological Screening Level (2005-2007).
6 PQLG is one-third of the minimum PSL.

Bolding indicates that the minimum PSL is greater than the laboratory LOD but less than the laboratory LOQ.
Bolding and shading indicates that the PSL is less than the laboratory LOD.

All results will be reported to DLs and any limitations on data use that result from having DLs that are greater than PSLs will be described in the RFI Report.

SAP Worksheet No. 16 -- Project Schedule / Timeline Table

(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Developing the UFP-SAP	Tetra Tech	06/01/2010	03/01/2011	UFP-SAP	10/22/2010 (draft) 01/19/2011 (draft final) 03/01/2011 (final)
HASP	Tetra Tech	10/18/2011	12/01/2010	HASP	09/27/2010 (draft) 12/01/2010 (final)
Environmental Sampling	Tetra Tech	03/15/2011	08/24/2011	SAP Addenda	05/01/2011 (Supplemental Sampling Addendum A) 06/01/2011 (Supplemental Sampling Addendum B)
RFI Report	Tetra Tech	06/01/2011	05/27/2012	RFI Report, SWMU 27	11/17/2011 (draft) 02/20/2012 (draft final) 05/27/2012 (final)

SAP Worksheet No. 17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

Samples will be collected at SWMU 27 to determine whether target analyte concentrations in Site media (surface and subsurface soil and groundwater) exceed the most conservative (lowest) applicable risk-based human health screening values. If these screening values are exceeded, the Project Team will evaluate human health risks and delineate the nature and extent of contamination at the Site during subsequent sampling events which will be presented in an addendum to this SAP.

SURFACE SOIL

Surface soil sample locations have been selected to provide representative coverage of surface soil that may have been impacted by PPA operations and maintenance activities and pit/sump/catch basin overflows.

Up to 55 soil samples (plus duplicate samples for QC purposes) from 55 locations will be collected in IA 1 and IA 2. These samples are categorized as surface ditches/channels/drainageways samples, flare test/fire incident areas samples, pits/sumps/catch basin samples, and PCB transformer area samples, as described below.

Surface Ditches/Channels/Drainageways

Up to 10 surface soil samples from 10 locations within surface ditches, channels, and drainageways will be collected in IA 1 and IA 2.

These samples will be analyzed for VOCs, perchlorate, metals, and, in the plating shop area, cyanide.

IA 1 PPA

Up to eight surface soil samples (27SS001G0002 to 27SS008G0002) from eight locations will be collected as shown on **Figure 17-1**.

IA 2 Flare Test Area

Up to two surface soil samples will be collected, one east of Building 3395 (27SS009G0001) and one sample west of Building 3395 (27SS058G0001) will be collected as shown on **Figure 17-2**.

No upgradient surface ditches/channels/drainageways soil samples will be collected for IA 1 or IA 2.

Flare Test/Fire Incident Areas

Up to nine surface soil samples will be collected from nine locations in former and suspected flare test areas and historical fire incident areas. Surface soil samples will be analyzed for perchlorate and metals.

IA 1 PPA

Up to one composite sample (27SS010C0002) will be collected at the laboratory flare test pad located north of Building 127. The composite sample will be comprised of three aliquots collected from the northern, western, and eastern sides of the pad. Two discrete samples (27SS011G0002 and 27SS012G0002) will be collected north of Building 3297 in the area where a fire incident occurred. These sample locations are presented on **Figure 17-3**.

IA 2 Flare Test Area

As shown on **Figure 17-4**, up to two discrete surface soil samples (27SS013G0002 and 27SS014G0002) will be collected from two locations northeast of Building 3395, two discrete surface soil samples (27SS015G0002 and 27SS016G0002) will be collected from two discrete locations north of Building 3395, and two surface soil samples (27SS017G0002 and 27SS018G0002) will be collected from two locations in the parking lot southwest of Building 3395.

Pits/Sumps/Catch Basins

Up to 19 surface soil samples will be collected in and around the pits/sumps/catch basins from former and suspected historical locations were overflows are suspected to have occurred from 19 locations. Except as noted below, four-point composite samples will be collected from each side of the pits/sumps/catch basins. For inactive pits/sumps, up to a six- point composite sample (i.e., a composite sample collected as grab samples from residual material in each pit/sump chamber) will be collected. A sketch of a typical pit/sump, which contains 6 chambers, is presented in Appendix A (P.W. Sketch No. 1896). Surface soil samples will be analyzed for VOCs, perchlorate, metals, and, in the plating shop area, cyanide.

IA 1 PPA

Up to 19 pit/sump/catch basin surface soil sample locations are presented on **Figure 17-5**. Detailed sample locations are presented on **Figure 17-5a** for Building 2698 Pit A, Building 126 Pit A, Building 126 Pit B, and Building 133 Pit A. Detailed sample locations are presented on **Figure 17-5b** for Building 122 Pit A, Building 130 Pit A, Building 1885 Pit A, and Building 1886 Pit A.

Up to one composite surface soil samples will be collected around Building 130 Pit A (27SS019C0002), Building 133 Pit A (27SS020C0002), Building 1885 Pit A (27SS021C0002), and Building 1886 Pit A (27SS022C0002). Each composite sample will be comprised of four aliquots collected from the northern, southern, eastern, and western side of these pits.

Up to two composite surface soil samples will be collected around Building 2698 Pit A (27SS023C0002) and Building 122 Pit A (27SS024C0002) (analyses will include cyanide). Each composite sample will be comprised of three aliquots collected from the northern, eastern, and western side of these pits.

Up to four composite surface soil samples (27SS025C0002 through 27SS028C0002) will be collected around Building 126 Pit B. Each composite sample, comprised of four aliquots, will be collected from the northern, southern, eastern, and western sides of the pit.

Up to seven composite samples will be collected from the chambers in Building 122 Pit A (27SS029C0002) (including cyanide), Building 126 Pit A (27SS03C00002), Building 130 Pit A (27SS031C0002), Building 133 Pit A (27SS032C0002), Building 1885 Pit A (27SS033C0002), Building 1886 Pit A (27SS034C0002), and Building 2698 Pit A (27SS035C0002). Each composite sample will be comprised of six aliquots (one from each chamber within the pit).

Up to two grab samples will be collected from the interior of the two small sumps south of Building 1885 (27SS056G0002) and Building 1886 (27SS057G0002).

IA 2 Former Flare Test Area

No samples will be collected in IA 2 because the existing pits/sumps/catch basins are for storm water collection/routing only and were installed after activities in the former Flare Test Area stopped.

PCB Transformer Areas

Up to 17 surface soil samples will be collected around the former PCB transformer and PCB-labeled transformer areas. Except as noted below, each composite sample will be comprised of four aliquots (one from each side of the transformer area). All transformer-related surface soil samples will be analyzed for PCBs. Additionally, the surface soil samples associated with the transformer west of Building 126 will be analyzed for VOCs.

IA 1 PPA

Up to seventeen PCB samples and three VOC samples will be collected from 17 locations as shown on **Figure 17-6**.

Up to six surface soil composite samples (27SS043C0002, 27SS044C0002, 27SS045C0002, 27SS046C0002, 27SS047C0002, and 27SS048C0002) will be collected from the exterior of the fenced area that formerly contained PCB transformers. This former transformer storage area is located west and across the road from Building 133. Two composite samples will be collected from the northern side (northwest [27SS044C0002] and northeast side [27SS043C0002]), two composite samples will be collected from the southern side (southwest [27SS046C0002] and southeast side [27SS047C0002]), one composite sample will be collected from the eastern side (27SS045C0002), and one composite sample will be collected from the western side of the fenced area (27SS048C0002). Each composite sample location will be comprised of four aliquots.

Up to four composite surface soil samples will be collected from the area around the new transformer at the western side of the main entrance to IA 1 (27SS059C0002 from the north side, 27SS060C0002 from the south side, 27SS061C0002 from the west side, and 27SS062C0002 from the east side of the transformer area). Each composite sample location will be comprised of four aliquots.

Up to four composite samples (27SS049C0002, 27SS050C0002, 27SS051C0002, and 27SS052C0002) will be collected from the PCB-labeled transformer located east of Building 3298 and north of Building 126. One composite sample will be collected from the north side (27SS049C0002), one composite sample will be collected from the south side (27SS051C0002), one composite sample will be collected from the east side (27SS050C0002), and one composite sample will be collected from the west side (27SS052C0002). Each location will be comprised of four aliquots.

Up to three discrete PCB and chlorinated VOCs (PCE and degradation compounds) samples will be taken from the north, south, and west sides of the PCB-labeled and Wecosol®-labeled transformer west of Building 124 (27SS053G0002, 27SS054G0002, and 27SS055G0002, respectively).

Additionally, one groundwater chlorinated VOCs (PCE and degradation compounds) sample (27TW001) will be collected from one of the borings, which will be installed as a temporary groundwater well. This groundwater sample location will be determined in the field based on the locations of infrastructure such as utility lines and curbing. The groundwater well will be installed in a practical location. If the groundwater sample location is in the proximity of a soil sample location, the soil sample location proximity will be identified in the field log book.

IA 2 Former Flare Test Area

No PCB transformer sampling will be conducted in IA 2 because there are currently no transformers in the area labeled as containing PCBs, and the review of historical photographs and drawings did not indicate any PCB transformers previously located in the area.

SUBSURFACE SOIL

Subsurface soil sample locations have been selected to provide representative coverage of the subsurface soil that may have been impacted by PPA pit/sump/catch basin overflow or by potential integrity issues at the pits/sumps/catch basins.

Up to 11 subsurface soil samples (plus duplicate samples for QC purposes) from nine locations will be collected in IA 1 (**Figures 17-5, 17-5a, and 17-5b**) and IA 2 (**Figure 17-4**).

Flare Test/Fire Incident Areas

Up to four subsurface soil samples will be collected from two locations in former and suspected flare test areas and historical fire incident areas. Subsurface soil samples will be analyzed for perchlorate and metals.

IA 2 Flare Test Area

As shown on **Figure 17-4**, up to four discrete subsurface soil samples (27SB017G0204, 27SB017G04XX, 27SB018G0204, and 27SB018G04XX) will be collected from two locations in the parking lot southwest of Building 3395.

Pits/Sumps/Catch Basins

Up to seven subsurface soil samples will be collected from seven areas near the lowest corners of the pits/sumps/catch basins at locations were overflows are suspected and to verify the integrity of the concrete basins. Subsurface soil samples will be analyzed for VOCs, perchlorate, metals, and, in the plating shop related pit area, cyanide.

IA 1 PPA

Figures 17-5, 17-5a, and 17-5b present the pit/sump/catch basin sample locations.

Up to seven discrete subsurface soil samples will be collected at a vertical boundary that extends 2 feet below the identified depth of the sumps/pits/catch basins (e.g., structure) (to be determined in the field). The depth of the sample will be the difference between the internal sidewall height and external sidewall height plus 2 feet, that is, 2 feet below the bottom of the basin. The sample locations and numbers are: Building 122 Pit A (27SB036GXXXX) (including cyanide), Building 126 Pit B (27SB037GXXXX), Building 130 Pit A (27SB038GXXXX), Building 133 Pit A (27SB0390GXXXX), Building 1885 Pit A (27SB040GXXXX), Building 1886 Pit A (27SB041GXXXX), and Building 2698 Pit A (27SB042GXXXX). Due to a frequency of 1 duplicate per 20 samples, QC samples will not be collected at every location.

IA 2 Former Flare Test Area

No pits/sumps/catch basins will be sampled in IA 2 because the existing pits/sumps/catch basins were not in operation during the former flare testing activities.

GROUNDWATER

A groundwater sample (27TW001) will be collected southwest of the transformer pad located west of Building 126. This sample location will be determined in the field based on the locations of infrastructure such as utility lines and curbing. The well will be installed in a practical location. The associated surface soil sample for the groundwater sample location will be identified in the field log book.

Field QC Samples

Groundwater samples (plus duplicate samples for QC purposes) will be collected and analyzed for the VOCs. Groundwater samples will also be analyzed for water quality parameters including pH, specific conductivity, turbidity, temperature, ORP, and DO to support field sampling decisions and site-specific risk calculations. Worksheet No. 20 presents the field QC sample summary.

SAP Worksheet No. 18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

SURFACE DITCHES, CHANNELS, AND DRAINAGEWAYS
SURFACE SOIL SAMPLES

Sample Location[1]	Sample ID[2,3]	Sample Location Description	NUMBER OF SAMPLES			
			SW-846 8260B VOCs	SW-846 6850 Perchlorate	SW-846 9012A Cyanide	SW-846 6010C (Metals)[4]
IA 1 Surface Ditches, Channels, and Drainageways[5]						
27SB001	27SS001G0001	North of SWMU 27 Fence	1	1		1
27SB002	27SS002G0001	Southwestern Corner of Building 1884	1	1	1	1
27SB003	27SS003G0001	West of Building 133 Outside of SWMU 27 Fenceline	1	1		1
27SB004	27SS004G0001	Northwest of Building 134	1	1		1
27SB005	27SS005G0001	Southwest of Building 134	1	1		1
27SB006	27SS006G0001	Northeast of Building 135	1	1		1
27SB007	27SS007G0001	SWMU 27 South Drainage Channel	1	1		1
27SB008	27SS008G0001	SWMU 27 South Drainage Channel Outside of SWMU 27 Fenceline	1	1		1
IA 2 Surface Ditches, Channels, and Drainageways[6]						
27SB009	27SS009G0001	East of Building 3395 at Crest of Drainageway	1	1		1
27SB058	27SS058G0001	West of Building 3395 at Crest of Drainageway	1	1		1
Total Samples			**10**	**10**	**1**	**10**

1 SB = Soil boring
2 SS = Surface soil; G = Grab; Last four digits of sample identification (ID) indicate depth below ground surface in feet.
3 Surface soil for surface ditches, channels, and drainageways is defined as 0 to 1 foot bgs.
4 Metals include aluminum, barium, cadmium, total chromium, copper, lead, manganese, strontium, and zinc.
5 Locations presented on **Figure 17-1**
6 Locations presented on **Figure 17-2**

FLARE TEST AND FIRE INCIDENT AREAS
SOIL SAMPLES

Sample Location[1]	Sample ID[2]	Sample Location Description	NUMBER OF SAMPLES	
			SW-846 6850 Perchlorate	SW-846 6010C (Metals)[3]
IA 1 Flare Test and Fire Incident Areas[4]				
27SB010	27SS010C0002[5]	North of Building 127	1	1
27SB011	27SS011G0002	~45 feet North of Building 3297	1	1
27SB012	27SS012G0002	~20 feet North of Building 3297	1	1
IA 2 Suspected Flare Test Area[6]				
27SB013	27SS013G0002	Northeast of Building 3395	1	1
27SB014	27SS014G0002	Northeast of Building 3395	1	1
27SB015	27SS015G0002	North of Building 3395	1	1
27SB016	27SS016G0002	North of Building 3395	1	1
27SB017	27SS017G0002	Southwest of Building 3395 in Parking Lot	1	1
	27SB017G0204		1	1
	27SB017G04XX[7]		1	1
27SB018	27SS018G0002	Southwest of Building 3395 in Parking Lot	1	1
	27SB018G0204		1	1
	27SB018G04XX[7]		1	1
		Total Samples	**13**	**13**

1 SB = Soil boring
2 SS = Surface soil; SB= Subsurface soil; G = Grab. Last four digits of sample identification (ID) indicate depth below ground surface in feet.
3 Metals include aluminum, barium, cadmium, total chromium, copper, lead, manganese, strontium, and zinc.
4 Locations presented on **Figure 17-3**
5 Four aliquots per composite surface soil sample.
6 Locations presented on **Figure 17-4**
7 "XX" indicates depth to be determined in the field.

IA 1 – PITS, SUMPS, AND CATCH BASINS
SOIL SAMPLES

Sample Location[1,2]	Sample ID[3]	Sample Location Description	NUMBER OF SAMPLES			
			SW-846 8260B VOCs	SW-846 9012A Cyanide	SW-846 6850 Perchlorate	SW-846 6010C (Metals)[4]
27SB019	27SS019C0002[5]	Building 130 Pit A	1		1	1
27SB020	27SS020C0002 [5]	Building 133 Pit A	1		1	1
27SB021	27SS021C0002[5]	Building 1885 Pit A	1		1	1
27SB022	27SS022C0002[5]	Building 1886 Pit A	1		1	1
27SB023	27SS023C0002[6]	Building 2698 Pit A	1		1	1
27SB024	27SS024C0002 [6]	Building 122 Pit A	1	1	1	1
27SB025	27SS025C0002[5]	North of Building 126 Pit B	1		1	1
27SB026	27SS026C0002[5]	East of Building 126 Pit B	1		1	1
27SB027	27SS027C0002[5]	South of Building 126 Pit B	1		1	1
27SB028	27SS028C0002[5]	West of Building 126 Pit B	1		1	1
27SB029	27SS029C0002[7]	Building 122 Pit A Interior Chambers	1	1	1	1
27SB030	27SS030C0002 [7]	Building 126 Pit A Interior Chambers	1		1	1

IA 1 – PITS, SUMPS, AND CATCH BASINS
SOIL SAMPLES

Sample Location[1,2]	Sample ID[3]	Sample Location Description	NUMBER OF SAMPLES			
			SW-846 8260B VOCs	SW-846 9012A Cyanide	SW-846 6850 Perchlorate	SW-846 6010C (Metals)[4]
27SB031	27SS031C0002[7]	Building 130 Pit A Interior Chambers	1		1	1
27SB032	27SS032C0002 [7]	Building 133 Pit A Interior Chambers	1		1	1
27SB033	27SS033C0002[7]	Building 1885 Pit A Interior Chambers	1		1	1
27SB034	27SS034C0002[7]	Building 1886 Pit A Interior Chambers	1		1	1
27SB035	27SS035C0002[7]	Building 2698 Pit A Interior Chambers	1		1	1
27SB036	27SB036GXXXX[3,8]	Exterior at the Lowest Corner of Building 122 Pit A	1	1	1	1
27SB037	27SB037GXXXX[3,8]	Exterior at the Lowest Corner of Building 126 Pit B	1		1	1
27SB038	27SB038GXXXX[3,8]	Exterior at the Lowest Corner of Building 130 Pit A	1		1	1

IA 1 – PITS, SUMPS, AND CATCH BASINS
SOIL SAMPLES

Sample Location[1,2]	Sample ID[3]	Sample Location Description	NUMBER OF SAMPLES			
			SW-846 8260B VOCs	SW-846 9012A Cyanide	SW-846 6850 Perchlorate	SW-846 6010C (Metals)[4]
27SB039	27SB039GXXXX[3,8]	Exterior at the Lowest Corner of Building 133 Pit A	1		1	1
27SB040	27SB040GXXXX[3,8]	Exterior at the Lowest Corner of Building 1885 Pit A	1		1	1
27SB041	27SB041GXXXX[3,8]	Exterior at the Lowest Corner of Building 1886 Pit A	1		1	1
27SB042	27SB042GXXXX[3,8]	Exterior at the Lowest Corner of Building 2698 Pit A	1		1	1
27SB056	27SS056G0002[9]	Small Pit south of Building 1885	1		1	1
27SB057	27SS057G0002[9]	Small Pit south of Building 1886	1		1	1
Total Samples			**26**	**3**	**26**	**26**

1 SB = Soil boring
2 See **Figures 17-5, 17-5A, and 17-5B**.
3 C = Composite; G = Grab; SS = Surface soil; SB = Subsurface soil; Last four digits of sample identification (ID) indicate depth below ground surface in feet. "XX" indicates depth to be determined in the field.
4 Metals include aluminum, barium, cadmium, total chromium, copper, lead, manganese, strontium, and zinc.
5 Four aliquots per composite surface soil sample.
6 Three aliquots per composite surface soil sample.
7 Catch basin residue sample is to be a composite sample with an aliquot from each chamber/bay of the catch basin. Typically, there are six chambers/bays in a catch basin. Typically, a six-aliquot composite sample will be collected per basin (i.e., one aliquot per chamber/bay).

8 The depth of the sample will be the difference between the internal sidewall height and external sidewall height plus 2 feet, that is, 2 feet below the bottom of the basin.
9 Small suspected collection/catch basin with steel cover plate located south of the building.

Note: Soil samples will be collected in accordance with SOP-09 (Appendix B). Field duplicate and matrix spike (MS)/matrix spike duplicate (MSD) samples will be collected at a frequency of 1 per 20 samples per medium per analyte for fixed-base laboratory samples. Therefore, field QC samples will not be collected at every site.

PCB TRANSFORMER AREAS SURFACE SOIL SAMPLES

Sample Location[1,2]	Sample ID[3]	Sample Location Description	SW-846 8260B Chlorinated Solvents[4]	SW-846 8082A PCBs
27SB043	27SS043C0002[5]	Northeast side of Former Transformer Fenced area		1
27SB044	27SS044C0002[5]	Northwest side of Former Transformer Fenced area		1
27SB045	27SS045C0002[5]	East side of Former Transformer Fenced area		1
27SB046	27SS046C0002[5]	Southwest side of Former Transformer Fenced area		1
27SB047	27SS047C0002[5]	Southeast side of Former Transformer Fenced area		1
27SB048	27SS048C0002[5]	West side of Former Transformer Fenced area		1
27SB049	27SS049G0002[5]	North side of Transformer Pad North of Building 126		1
27SB050	27SS050C0002[4]	East side of Building 126 Transformer Pad		1
27SB051	27SS051G0002[5]	South side of Transformer Pad North of Building 126		1
27SB052	27SS052C0002[5]	West side of Transformer Pad North of Building 126		1
27SB053	27SS053G0002	North side of Transformer Pad Located West of Building 124	1	1
27SB054	27SS054G0002	South side of Transformer Pad Located West of Building 124	1	1
27SB055	27SS055G0002	West side of Transformer Pad Located West of Building 124	1	1
27SB059	27SS059C0002[5]	North Side of Transformer by IA 1 Highway 5 entrance		1

PCB TRANSFORMER AREAS SURFACE SOIL SAMPLES

Sample Location[1,2]	Sample ID[3]	Sample Location Description	SW-846 8260B Chlorinated Solvents[4]	SW-846 8082A PCBs
27SB060	27SS060C0002[5]	South Side Transformer by IA 1 Highway 5 entrance		1
27SB061	27SS061C0002[5]	West Side Transformer by IA 1 Highway 5 entrance		1
27SB062	27SS062C0002[5]	East Side Transformer by IA 1 Highway 5 entrance		1
Total Samples			**3**	**17**

1 SB = Soil boring
2 See **Figure 17-6** for sample locations.
3 SS = Surface soil; Last four digits of sample identification (ID) indicate depth below ground surface in feet.
4 Chlorinated solvents include PCE and PCE degradation products.
5 Four aliquots per composite sample.

Note: Soil samples will be collected in accordance with SOP-09 (Appendix B). Field duplicate and MS/MSD samples will be collected at a frequency of 1 per 20 samples per medium per analyte for fixed-base laboratory samples. Therefore, field QC samples will not be collected at every site.

PCB TRANSFORMER AREAS GROUNDWATER SAMPLES			
Sample Location(1)	**Sample ID**	**Sample Location Description**	**SW-846 8260B Chlorinated Solvents(2)**
27TW001	27TW00101	Field Located at or near the West Side of Transformer Pad West of Building 124	1
		Total Samples	**1**

1 The groundwater sample location will be determined in the field based on the locations of infrastructure such as utility lines and curbing at a practical location. If the groundwater sample location is in the proximity of a soil sample location, the soil sample location proximity will be identified in the field log book.
2 Chlorinated solvents include PCE and PCE degradation products.

SAP Worksheet No. 19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

MATRIX	ANALYTICAL GROUP	ANALYTICAL AND PREPARATION METHOD/SOP REFERENCE(1)	CONTAINERS (number, size, and type)	SAMPLE VOLUME (units)	PRESERVATION REQUIREMENTS (chemical, temperature, light protected)	MAXIMUM HOLDING TIME (preparation/ analysis)
Groundwater and aqueous QC samples	VOCs	SW-846 5030/8260B, Empirical SOP-202	Three 40-milliliter (mL) glass vials	5 mL	Hydrochloric acid to pH<2; Cool to ≤ 6 °C; no headspace	14 days to analysis
Soil	VOCs	SW-846 5035/8260B, Empirical SOP-202/225	Three 5-gram (g) Encore samplers or terracores	5 g	Sodium bisulfate ≤ 6 °C; or in water and freeze to < -10 °C	48 hours from sampling to preparation, 14 days to analysis
Groundwater and aqueous QC samples	PCBs	SW-846 3510C/3520/ /8082A, Empirical SOP-211/302	Two 1-liter (L) glass amber bottles	1,000 mL	Cool to ≤ 6 °C	7 days until extraction, 40 days to analysis
Soil	PCBs	SW-846 3540/3545/ 3550/8082A, Empirical SOP-211/343	One 4-ounce (oz.) glass jar	30 g	Cool to ≤ 6 °C	14 days until extraction, 40 days to analysis
Groundwater and aqueous QC samples	Metals	SW-846 3010A/ 6010C, Empirical SOP-100/105	One 500-mL plastic bottle	50 mL	Nitric acid to pH <2; Cool to ≤ 6 °C	180 days to analysis except mercury, 28 days for mercury
Soil	Metals	SW-846 3050B/ 6010C, Empirical SOP-100/105	One 4-oz glass jar	1 to 2 g	Cool to ≤ 6 °C	180 days to analysis except mercury, 28 days for mercury
Groundwater and aqueous QC samples	Cyanide	SW-846 9012A, Empirical SOP-164/175	One 250-mL plastic bottle	100 mL	Sodium hydroxide to a pH > 12; Cool to ≤ 6 °C	14 days to analysis
Soil	Cyanide	SW-846 9012A, Empirical SOP-164/175	One 4-oz glass jar	5 g	Cool to ≤ 6 °C	14 days to analysis
Groundwater and aqueous QC samples	Perchlorate	SW-846 6850 Empirical SOP-239	500 mL plastic	100 mL	Cool to ≤ 6 °C	28 days to analysis
Soil	Perchlorate	SW-846 6850 Empirical SOP-239	One 4-oz glass jar	5 g	Cool to ≤ 6 °C	28 days to analysis

1 Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet No. 23).

SAP Worksheet No. 20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates(1)	No. of MS/MSDs(2)	No. of VOA Trip Blanks(3)	Total No. of Samples to Laboratory
Soil	VOCs	43	2	2	5	45
Soil	Cyanide	5	1	1	NA	6
Soil	Perchlorate	48	3	3	NA	52
Soil	Metals	48	3	3	NA	52
Soil	PCBs	13	1	1	NA	15
Groundwater	VOCs(4)	1	1	1	1	1

1 Field duplication sample will be collected at a frequency of 1 per 20 field samples.
2 MS/MSD samples will be collected at a frequency of 1 per 20 field samples. Although the MS/MSD is not typically considered a field QC, it is included here because location determination is often established in the field. MS/MSD samples are not included in the total number of samples sent to the laboratory.
3 One volatile organic analyte (VOA) trip blank per cooler that contains samples for VOC analysis. Field sample collections will determine the number of trip blanks and coolers. The five cooler estimate is based on the site being an active production area and previous sample collection experience at NSA Crane.
4 PCE and PCE degradation compounds.

SAP Worksheet No. 21 -- Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP-01	Global Positioning System	Tetra Tech	GPS unit	Y	
SOP-02	Sample Labeling	Tetra Tech	NA	Y	
SOP-03	Sample Identification Nomenclature	Tetra Tech	NA	Y	Project-specific nomenclature
SOP-04	Sample Custody and Documentation of Field Activities	Tetra Tech	Field logbook, sample log sheets, boring logs	Y	
SOP-05	Sample Preservation, Packaging, and Shipping	Tetra Tech	NA	Y	
SOP-06	Decontamination of Field Sampling Equipment	Tetra Tech	Decontamination equipment, scrub brushes, 5-gallon buckets, spray bottles, phosphate free detergent, deionized water	Y	
SOP-07	Soil Coring and Sampling Using Hand Auger Techniques	Tetra Tech	Stainless steel auger bucket, extension rods, and T-handle	Y	
SOP-08	Soil Sample Logging	Tetra Tech	NA	Y	
SOP-09	Management of Investigation-Derived Waste	Tetra Tech	NA	Y	
SOP-10	Calibration and Use of Photoionization Detector	Tetra Tech	Photoionization air meter	Y	
SOP-11	Subsurface Soil and Groundwater sampling using DPT	Tetra Tech	NA	Y	
SOP-12	Monitoring Well Installation	Tetra Tech	NA	Y	
SOP-13	Monitoring Well Development	Tetra Tech	NA	Y	
SOP-14	Measurement of Water Levels	Tetra Tech	NA	Y	
SOP-15	Low-Flow Well Purging and Stabilization	Tetra Tech	NA	Y	
SOP-16	Monitoring Well Sampling	Tetra Tech	NA	Y	
SOP-17	Calibration and Care of Water Quality Meters	Tetra Tech	Multi-parameter water quality meter, such as a Horiba U-22	Y	

SAP Worksheet No. 22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity[1]	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[2]	Comments
FID	Calibration and Visual Inspection	Daily	Manufacturer's guidance	Replace	FOL or designee	Manufacturer's guidance	To be used to determine the subsurface soil depth that is most impacted for biased sample collection.
PID	Calibration and Visual Inspection	Daily	Manufacturer's guidance	Replace	FOL or designee	SOP-11	To be used to determine the subsurface soil depth that is most impacted for biased sample collection.
Multi-Parameter Water Quality Meter	Visual Inspection, Calibration	Daily	Manufacturer's guidance	Replace	FOL or designee	SOP-17	
Turbidity Meter	Visual Inspection, Calibration	Daily	Manufacturer's guidance; calibrations must bracket expected values. Initial Calibration Verification (ICV) must be <10 Nephelometric Turbidity Units (NTU).	Replace	FOL or designee	SOP-17	

1 Activities may include calibration, verification, testing, maintenance, and/or inspection.
2 Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet No. 21).

SAP Worksheet No. 23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

LAB SOP NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX AND ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)
Empirical SOP-100	Metals Digestion/ Preparation, Methods 3005A/ USEPA CLP ILMO 4.1 Aqueous, 3010A, 3030C, 3050B, USEPA CLP ILMO 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C (Revision 20, 04/27/10)	Definitive	Soil, groundwater, and aqueous QC samples/ Metals Digestion	None/Preparation	Empirical	N
Empirical SOP-105	Metals by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) Technique, SW-846 Methods 6010B, 6010C, USEPA Method 200.7, Standard Methods 19th Edition 2340B, USEPA CLP ILMO 4.1 (Revision 16, 04/11/10)	Definitive	Soil, groundwater, and aqueous QC samples/ Metals	ICP-AES	Empirical	N
Empirical SOP-164	Distillation of Aqueous/Solid Samples for Cyanide, Total and Amenable, SW846 Method 9012A, USEPA Methods 335.1, 335.4, Standard Methods SM4500-CN C, G, 18th and 19th ED/ USEPA CLP ILMO 4.1 (Revision 15, 03/25/10)	Definitive	Soil, groundwater, and aqueous QC samples/ Cyanide preparation	None/ Distillation	Empirical	N
Empirical SOP-175	Post-Distillation Analysis for Cyanide by Lachat Flow Injection Analyzer, Methods 335.4, SW846 9012A, USEPA-CLP 4.1; Addendum for USEPA CLP ILM 05.2 Aqueous/Soil/ Sediment (Revision 11, 09/07-/10)	Definitive	Soil, groundwater, and aqueous QC samples/ Cyanide	Automated Ion Analyzer	Empirical	N
Empirical SOP-202	GC/MS Volatiles using USEPA Method 624 and SW846 Method 8260B, Including Appendix IX Compounds (Revision 23, 09/09/10)	Definitive	Soil, groundwater, and aqueous QC samples/ VOCs	Gas Chromatography/Mass Spectroscopy (GC/MS)	Empirical	N

LAB SOP NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX AND ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)
Empirical SOP-211	Gas Chromatography/ Electron Capture Detector (GC/ECD) Organochlorine Pesticides/ PCBs using USEPA Method 608/608.2 or SW846 Method 8081A/8082 or 8081B/8082A (Revision 22, 07/07/10)	Definitive	Soil, groundwater, and aqueous QC samples/ PCBs	GC/ECD	Empirical	N
Empirical SOP-225	GC/MS Volatile Non-Aqueous Matrix Extraction using SW-846 Method 5035 for 8260B Analysis (Revision 9, 9/07/10)	Definitive	Soil / VOCs Extraction	Purge and Trap instrumentation coupled with GC/MS	Empirical	N
Empirical SOP-239	Perchlorate in Water, Soil, and Solid Waste Using High Performance Liquid Chromatography/ Electrospray Ionization/ Mass Spectrometry by SW846 Method 6850 (Revision 04, 09/07/10)	Definitive	Soil, groundwater, and aqueous QC samples / Perchlorate	High Performance Liquid Chromatography/ Electrospray Ionization/ Mass Spectrometry (HPLC/ESI/MS)	Empirical	N
Empirical SOP-302	Pesticide/PCBs, Aqueous Matrix Extraction for USEPA 608/608.2 and SW846 Method 8081A/8082 Using SW846 Method 3510C (Revision 17, 04/26/10)	Definitive	Groundwater and aqueous QC samples/ PCBs Extraction	None/ Extraction	Empirical	N
Empirical SOP-343	BNA, Pesticide/PCB, and TPH Non-Aqueous Matrix (Microwave Extraction) Using SW-846 Method 3546 (Revision 1, 09/09/10)	Definitive	Soil / PCBs Extraction	Microwave Extraction	Empirical	N
Empirical SOP-404	Laboratory Sample Receiving, Log-in and Storage SOPs (Revision 13, 6/29/09)	Definitive	Log-in	None/ Log-in	Empirical	N
Empirical SOP-405	Analytical Laboratory Waste Disposal (Revision 5, 6/23/09)	Definitive	Disposal	None/ Disposal	Empirical	N
Empirical SOP-410	SOP for Laboratory Sample Storage, Secure Areas, and Sample Custody (Revision 7, 6/23/09)	Definitive	Log-in	None/ Log-in	Empirical	N

Copies of laboratory SOPs listed are included in Appendix C.

SAP Worksheet No. 24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS VOCs	Bromofluoro-benzene (BFB) Tune	Prior to each Initial Calibration (ICAL) and at the beginning of each 12-hour period.	Must meet the ion abundance criteria required by the method (SW-846 8260B; Section 7.3.1; Table 4).	Retune and/or clean or replace source. Samples will not be analyzed without a valid tune.	Analyst/ Supervisor	Empirical SOP-202
	ICAL – a minimum of a five-point calibration is prepared for all target analytes	Upon instrument receipt, for major instrument changes, or when continuing calibration verification (CCV) does not meet criteria.	The average response factor (RF) for System Performance Check Compound (SPCC) must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. The percent relative standard deviation (RSD) for RFs for calibration check compounds (CCCs) must be ≤ 30%; and %RSD for each target analyte must be ≤ 15%, or the linear regression correlation coefficient (r) must be ≥ 0.995; or the coefficient of determination (r^2) must be ≥ 0.99 (six points are required for second order).	Correct problem then repeat ICAL. Samples cannot be analyzed until ICAL has passed.	Analyst/ Supervisor	
	Retention Time (RT) Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	None	Analyst / Supervisor	
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target analyte must be within ± 0.006 RRT units.	Correct problem, then rerun ICAL.	Analyst / Supervisor	
	ICV – Second Source	Once after each ICAL, prior to beginning a sample run.	The percent recovery (%R) for all target analytes must be within 80-120%.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. Samples cannot be analyzed until the ICV has been verified.	Analyst/ Supervisor	
	CCV	Perform one per 12-hour analysis period after tune and before sample analysis.	The minimum RF for SPCCs must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. The percent difference or percent drift (%D) for all target analytes and surrogates must be ≤ 20%.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst/ Supervisor	Empirical SOP-202

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/ECD PCBs	ICAL - A minimum of a five-point calibration of Aroclor-1660 (1016/1260 mixture) is prepared	Upon instrument receipt, major instrument change, when CCV does not meet criteria.	Option 1: %RSD must ≤20% for Aroclor 1016/1260. If not met, Option 2: r must be ≥ 0.995; or Option 3: r^2 must be ≥ 0.99 for 6-point calibration. Mid-point calibration of other Aroclors – if an Aroclor is detected in a sample, a minimum of five-point ICAL must be performed and meet the above criteria.	Correct problem then repeat ICAL. Samples cannot be analyzed until ICAL has passed.	Analyst/ Supervisor	Empirical SOP-211
	ICV – Second Source	Once after each ICAL prior to sample analysis.	%R must be between 80% and 120% for all analytes.	Evaluate, repeat, if still failing, recalibrate.	Analyst/ Supervisor	
	CCV	Analyze standard at the beginning and end of sequence and after every 10 samples.	The %D of all target analytes must be ≤20%.	If %D is high and sample result is not detected, qualify/narrate with project approval. If %D is low or project approval not received, reanalyze all samples since the last successful CCV.	Analyst/ Supervisor	
ICP-AES Metals	ICAL - a 1-point calibration per manufacturer's guidelines is prepared for all target analytes	At the beginning of each day, or if the QC is out of criteria, prior to sample analysis.	None; only one high standard and a calibration blank must be analyzed. If more than one calibration standard is used, r must be ≥ 0.995.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	Empirical SOP-105
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R must be within 90-110%.	Investigate reasons for failure, reanalyze once. If still unacceptable, correct problem and repeat ICAL.	Analyst/ Supervisor	
	CCV	At the beginning and end of the sequence and after every 10 samples.	The %R must be within 90-110%.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze all affected samples.	Analyst/ Supervisor	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No analytes detected > than the LOD.	Correct the problem, then reprepare and reanalyze.	Analyst / Supervisor	
	Continuing Calibration Blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence.	No analytes detected > than the LOD.	Correct the problem, then reprepare and reanalyze calibration blank and all affected samples.	Analyst / Supervisor	Empirical SOP-105

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
ICP-AES Metals (continued)	Low-Level Check Standard (if using one-point ICAL)	Daily after 1-point ICAL and before samples.	The %R must be within 80-120.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst / Supervisor	
	Interference Check Standards (ICS – ICS A and ICS B)	At the beginning of an analytical run.	ICS A %Rs must be within the absolute value of the LOD; and ICS B %Rs must be within 80-120 %.	Terminate analysis; locate and correct problem; reanalyze ICS.	Analyst / Supervisor	
Lachat Cyanide	ICAL– A minimum of a six-point calibration is prepared	Perform daily prior to sample analysis.	r must be ≥ 0.995. All calibration standards must be distilled if samples are expected to contain sulfides.	Correct problem then repeat ICAL. No samples can be analyzed until ICAL has passed.	Analyst/ Supervisor	Empirical SOP-164/175
	ICV – Second Source	At the start of every sequence prepared fresh daily (undistilled), prior to sample analysis.	The %R must be within 85-115%.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples can be analyzed until ICV has been verified.	Analyst/ Supervisor	
	ICB	After the ICV (undistilled).	Must be < LOQ for the target analyte.	Determine source of contamination, correct problem, and restart sequence.	Analyst/ Supervisor	
	CCV	Every 10 samples (undistilled).	The %R must be within 85-115%.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst/ Supervisor	Empirical SOP-164/175
	ICV (distilled, high and low)	Once per ICAL.	The %R must be within 85-115%.	Determine cause for failure, correct problem, and redistill standards.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
HPLC/ESI/ /MS Perchlorate	Tune	Prior to ICAL and after any mass calibration or maintenance is performed.	Tuning standards must contain the analytes of interest and meet acceptance criteria outlined in the laboratory SOP.	Re-tune instrument. If the tuning will not meet acceptable criteria, an instrument mass calibration must be performed and the tuning redone. Sample analysis shall not proceed without acceptable tuning.	Analyst/ Supervisor	Empirical SOP-239
	ICAL – A minimum of a five-point calibration is prepared.	Upon instrument receipt, major instrument change, or when the CCV does not meet criteria.	The %RSD for RFs must be ≤ 20% or r must be ≥ 0.995. The concentration corresponding to the absolute value of the Y-intercept of the calibration curve must be ≤ LOD.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	
	ICV – Second Source	Once after each ICAL, prior to the analysis of samples.	The %R must be within 85-115%.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples can be analyzed until ICV has been verified.	Analyst/ Supervisor	
	CCV	Before sample analysis and after every 10 field samples, and at the end of the sequence.	Low-range standard: Perchlorate must be within 50-150 %R. Mid-range standard: Perchlorate must be within 85-115 %R.	Correct problem and rerun CCV. If that fails, repeat ICAL and re-analyze all samples analyzed since the last successful CCV.	Analyst/ Supervisor	
HPLC/ESI/ /MS Perchlorate (continued)	Limit of Detection Verification (LODV) (per batch)	Prior to sample analysis and at the end of the sequence. It can be analyzed after every 10 samples to reduce the re-analysis rate.	The %R must be within 70-130%. Per the DoD Quality Systems Manual (QSM) for Environmental Laboratories, Version 4.1, the perchlorate spike concentration must be approximately 2 times the LOD.	Correct problem, rerun LODV and all samples since last successful LODV. If a sample with perchlorate concentration at or between the LOD and LOQ is bracketed by a failing LODV, it must be re-analyzed. A sample with a concentration above the LOQ can be reported. Results cannot be reported without a valid LODV.	Analyst/ Supervisor	Empirical SOP-239

SAP Worksheet No. 25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.2.3)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference[1]
GC/MS	Check pressure and gas supply daily. Bake out trap and column, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-201/202
GC/ECD	Check pressure and gas supply daily. Bake out column, change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in Equipment Maintenance SOP.	PCBs	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	If % D > 20% and samples are < LOD, narrate. If %D > 20% only on one column, narrate. If % D > 20% for closing CCV, and is likely due to matrix interference, narrate. Otherwise, reanalyze all samples since the last acceptable CCV.	Analyst/ Supervisor	Empirical SOP-211/343
ICP-AES	Clean the torch assembly and the spray chamber when they become discolored or when degradation in data quality is observed. Clean the nebulizer, and check the argon supply. Replace the peristaltic pump tubing as needed.	Metals	Inspect the torch, nebulizer chamber, pump, and tubing	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-100/105
Lachat	Check and clean segments weekly, clean reagent tubes monthly. Change lamp, change diluent and wash tubes, change mixing paddles and syringes, and change dispensing needle, all as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Cyanide	Tubing and rollers.	Prior to ICAL or as necessary.	Acceptable ICAL and CCV.	Recalibrate and/or perform necessary equipment maintenance. Reanalyze samples not bracketed by passing CCV.	Analyst/ Supervisor	Empirical SOP-164/175
HPLC/ESI/MS	Clean the source and capillary needle. Change analytical column as needed, change mobile phase when insufficient for run or contamination, change inlet filters as needed for contamination.	Perchlorate	Check pump pressure, check for leaks, check for adequate mobile phase.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-239

1 Reference SOPs are provided in Appendix C.

SAP Worksheet No. 26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

SAMPLE HANDLING SYSTEM
SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL or designee/ Tetra Tech
Sample Packaging (Personnel/Organization): FOL or designee/ Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL or designee/ Tetra Tech
Type of Shipment/Carrier: Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians/ Empirical
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Empirical
Sample Preparation (Personnel/Organization): Extraction Laboratory, Metals Preparation Laboratory / Empirical
Sample Determinative Analysis (Personnel/Organization): GC Laboratory, GC/MS Laboratory, Metals Laboratory / Empirical
Sample Archiving
Field Sample Storage (Number of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (number of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (Number of days from sample collection): None
Sample Disposal
Personnel/Organization): Sample Custodians/ Empirical

SAP Worksheet No. 27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

27.1 SAMPLE NOMENCLATURE, SAMPLE COLLECTION DOCUMENTATION, HANDLING, TRACKING, AND CUSTODY PROCEDURES

The following sections outline the procedures that will be used to document project activities and sample collection, handling, tracking, and custody procedures during the investigation. All forms must be completed as fully as possible.

27.1.1 Sample Identification

Refer to Worksheet No. 18 for how the samples will be labeled, and refer to Worksheet No. 20 for how the field QA/QC samples will be labeled.

27.1.2 Sample Collection Documentation

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, VOC screening logs, and monitoring well construction logs. Field logbooks utilized on this project will be bound and water resistant. The pages of the logbook will be numbered sequentially, and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details, and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration. Example field forms are included in Appendix B.

For sampling and field activities, the following types of information will be recorded in the field logbook as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities involved with sampling
- Subcontractor activity summary
- Site observations including site entry and exit times

- Site sketches made on site
- Visitor names, affiliations, and arrival and departure times

Health and safety issues including PPE

27.1.3 Sample Handling and Tracking System

Following sample collection into the appropriate bottleware, samples will be immediately placed on ice in a cooler. Glass sample containers will be enclosed in bubble wrap to protect the bottleware during shipment. The cooler will be secured using strapping tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to Empirical for analysis. Samples will be preserved as appropriate based on the analytical method. The laboratory will provide pre-preserved sample containers for sample collection. Samples will be maintained at 0 to 6 °C until delivery to the laboratory. Proper custody procedures will be followed throughout all sampling events including collection and handling.

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., FedEx). Chain-of-custody forms will document the date and time of sample collection, the sampler's name, and the names of others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the chain-of-custody form. Tetra Tech Field Documentation [SOP-02 (Sample Labeling), SOP-03 (Sample Identification Nomenclature), SOP-04 (Sample Custody and Documentation of Field Activities), SOP-08 (Soil Sample Logging), SOP-10 (Calibration and Use of Photoionization Detector), and SOP-17 (Calibration and Care of Water Quality Meters)] provides further details on the chain-of-custody procedure. Field SOPs are provided in Appendix B.

These subsections outline the procedures that will be used by field and laboratory personnel to document project activities and sample collection procedures during this RFI. Each form must be filled in as completely as possible.

27.1.4 Sample Handling

Sample handling requirements are described in Worksheet No. 26. Tetra Tech personnel will collect the samples. The samplers will take care not to contaminate samples through improper handling. Samples will be sealed in appropriate containers, packaged by Tetra Tech personnel, and placed into sealed coolers under chain of custody in accordance with SOP-04 (see Worksheet No. 21). Samples to be analyzed for VOCs will be accompanied by a VOC trip blank. All coolers will contain a temperature blank. Samples will be transferred under chain of custody to a courier as described below. Once received by the laboratory, receipt will be documented on the chain-of-custody form, and the samples will be checked

in. The samples will remain under chain of custody throughout the analysis period to ensure that their integrity is preserved. Details are provided below.

27.1.5 Sample Delivery

Samples will typically be delivered to the laboratory via a public courier (i.e., FedEx). After samples have been collected, they will be sent to the laboratory within 24 hours. Under no circumstances will sample holding times be exceeded.

27.1.6 Sample Custody

Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples will be packaged and shipped to the laboratory in accordance with Tetra Tech SOP-05 (see Appendix B).

A sample is under custody if:

- The sample is in the physical possession of an authorized person.
- The sample is in view of an authorized person after being in his/her possession.
- The sample is placed in a secure area by an authorized person after being in his/her possession.
- The sample is in a secure area, restricted to authorized personnel only.

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used, with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the form each time a sample transfer occurs.

Integrity of the samples collected during the site investigation will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report. Sample custody is described in Worksheet No. 27.

The Tetra Tech FOL or designee is responsible for the care and custody of the samples collected until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs included in Appendix C.

27.1.7 Laboratory Custody

This section documents specific laboratory custody procedures utilized by Empirical. Custody seals are supplied with all bottle orders and are affixed to the cooler after sampling. The presence or absence of custody seals is noted on the Sample Receipt Condition Report (SRCR).

Upon receipt of samples from the field, the laboratory sample management personnel will sign off on the chain-of-custody form, open the sample cooler(s), verify sample integrity, and compare received samples to samples listed on the chain-of-custody form. If there is a discrepancy or problem (i.e., broken sample containers), the laboratory will contact the FOL or other qualified personnel and resolve the issue. Additionally, the laboratory completes a SRCR, which documents visual inspection of the samples and specific parameters such as cooler temperature, holding times, and preservation. Discrepancies or changes will be documented on the SRCR.

The laboratory sample management personnel assigns a unique laboratory work order number for the entire sample set listed on the chain-of-custody form. The samples are then logged into the laboratory information management system, and a Login Chain-of-Custody Report is generated. Each sample within a work order is labeled numerically. Each container of a particular sample is uniquely identified by adding an alphabetical suffix to the sample number. The laboratory labels each sample container with a Laboratory Custody Label that will remain on the sample bottle for the duration of laboratory sample storage. The laboratory also initiates the appropriate Internal Custody Record for the sample set. Personnel fill out the Internal Custody Records to document sample removal from and return to sample storage.

A laboratory data file is also initiated for the work order. This file includes the Login Chain-of-Custody Report, chain-of-custody form, and SRCR. The folder also includes a Login File Sheet that summarizes the analyses for which the work order has been logged. This sheet is used to track data completion.

Samples for a project may be batched or grouped together by the laboratory. A series of batched work orders is referred to as a Sample Delivery Group (SDG). The SDG includes those samples received one chain-of-custody form, duplicate samples, and field QA/QC samples and can include samples of different media. QA/QC samples will be run at the frequencies specified in the analytical methods. The SDG is given a specific identification number.

Samples are stored at the laboratory in refrigerators prior to, during, and after analysis. Refrigerators at the laboratory are constantly monitored for temperature. Proper temperatures and lighting are maintained in the refrigerators to ensure sample integrity and preservation. Samples are retained by the laboratory

for a period of 90 days after the data report is mailed to the client, unless otherwise specified in a client contract. The laboratory then disposes of non-hazardous samples following certified disposal practices. Hazardous samples are either returned to the client or disposed of through a licensed broker. Documentation of disposal is maintained by the laboratory.

Chain-of-custody requirements are also documented with instructions contained in each shipment from the laboratory (Empirical SOP-404 [Laboratory Sample Receiving Log-In and Storage], provided in Appendix C).

SAP Worksheet No. 28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**VOCs**
Analytical Method/ SOP Reference	**SW-846 8260B/ Empirical SOP-202**

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples	All target analytes must be ≤ ½ LOQ, except common lab contaminants, which must be < LOQ.	Investigate source of contamination and rerun method blank prior to analysis of samples, if possible. Evaluate the samples and associated QC, if blank results are above LOQ, then report sample results that are <LOQ or >10X the blank concentration. Re-prepare and reanalyze blank and those samples that were >LOQ and <10X the blank.	Analyst, Supervisor, Data Validator	Bias/Contamination	Same as QC Acceptance Limits
Laboratory Control Sample (LCS)	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12-hour clock and is acceptable, then narrate. If the LCS recoveries are high, but the sample results are <LOQ, then narrate. Otherwise, reprepare and reanalyze the LCS and associated samples.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**VOCs**
Analytical Method/ SOP Reference	**SW-846 8260B/ Empirical SOP-202**

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD Quality Systems Manual (QSM) Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be ≤ 30%.	CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met unless RPDs indicate obvious extraction/ analysis difficulties, then re-prepare and reanalyze MS/MSD.	Analyst, Supervisor, Data Validator	Accuracy/Bias/ Precision	Same as QC Acceptance Limit

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**VOCs**
Analytical Method/ SOP Reference	**SW-846 8260B/ Empirical SOP-202**

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards (ISs)	Every field sample, standard, and QC sample - three per sample- Fluorobenzene Chlorobenzene-d5 1,4-dichlorobezene-d4	RTs must be within ± 30 seconds and the response areas must be within -50% to +100% of the ICAL midpoint standard for each IS.	Inspect mass spectrometer and gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits
Surrogates	All field and QC samples - four per sample- Dibromofluoromethane 1,2-dichloroethane-d4 Toluene-d8 BFB	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	If sample volume is available, then reprepare and reanalyze sample for confirmation of matrix interference when appropriate.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
Results between DL and LOQ	NA	Apply “J” qualifier to results detected between DL and LOQ.	None	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**PCBs**
Analytical Method/SOP Reference	**SW-846 8082A/ Empirical SOP-211**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix	All target analytes must be ≤ ½ LOQ.	Re-analyze to confirm the analyte detections. Re-prepare the samples associated with the contaminated Blank. Prepare a non-compliance report to identify the non-compliance to the PM.	Analyst, Supervisor, Data Validator	Bias/Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix PCB: Spike with Aroclor 1016/1260 mix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	If an MS/MSD was performed and is acceptable, then narrate. If a LCS/ Laboratory Control Sample Duplicate (LCSD) were performed and only one of the set was unacceptable, then narrate. If the LCS recovery is high, but the sample results are <LOQ, then narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix (spike same as LCS)	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be ≤ 30%.	Evaluate the samples and associated QC and if the LCS results are acceptable, then narrate. If both the LCS and MS/MSD are unacceptable, then reprepare the samples and QC.	Analyst, Supervisor, Data Validator	Accuracy/Bias/ Precision	Same as QC Acceptance Limits

Matrix	**Soil, Groundwater, and Aqueous QC Samples**
Analytical Group	**PCBs**
Analytical Method/SOP Reference	**SW-846 8082A/ Empirical SOP-211**

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogates	All field and QC samples - two per sample Decachlorbiphenyl (DCB) Tetra-chloro-meta-xylene (TCMX)	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	No corrective will be taken when one surrogate is within criteria. If surrogates recoveries are high and sample is <LOQ, then no CA is taken. If surrogates recoveries are low, then the affected samples are re-extracted and reanalyzed.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD ≤ 40%. Report the higher of the two concentrations, unless there is interference.	None. Apply "J" flag if RPD >40% and discuss in the case narrative.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	None	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits

Matrix	**Soil, , Groundwater, and Aqueous QC Samples**
Analytical Group	**Metals**
Analytical Method/SOP Reference	**SW-946 6010C/ Empirical SOP-103, 105**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix	All target analytes must be ≤ ½ LOQ.	If the blank value > LOQ, then report sample results. If the blank value < LOQ or > 10x the blank value, then redigest. If blank value is less than negative LOQ, then report sample results. If > 10x the absolute value of the blank result, then redigest and reanalyze.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix	%R must be within 80-120%.	Evaluate and reanalyze, if possible. If the LCS recoveries are high, but the sample results are < LOQ, then narrate. Otherwise, redigest and reanalyze all associated samples for failed target analyte(s).	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
MS	One per preparatory batch of 20 or fewer samples of similar matrix	%R should be within 80-120% (if sample is < 4x spike added).	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
Sample Duplicate	One per preparatory batch of 20 or fewer samples of similar matrix	The RPD should be ≤ 20% for duplicate samples for both water and soils.	Narrate any results that are outside control limits.	Analyst, Supervisor, and Data Validator	Precision	Same as QC Acceptance Limits
Serial Dilution	One per preparatory batch with sample concentration(s) >50x LOD	The 5-fold dilution result must agree within ±10%D of the original sample result if result is >50x LOD.	Perform post-spike addition.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits

Matrix	**Soil, , Groundwater, and Aqueous QC Samples**
Analytical Group	**Metals**
Analytical Method/SOP Reference	**SW-946 6010C/ Empirical SOP-103, 105**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Post Spike	One is performed when serial dilution fails or target analyte concentration(s) in all samples are < 50x LOD	The %R must be within 75-125% of expected value to verify the absence of an interference. Spike addition should produce a concentration of 10-100x LOQ.	Flag results for affected analytes for all associated samples with “J”.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
Results between DL and LOQ	Not known at this time	Apply “J” qualifier to results between DL and LOQ.	None	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits

Matrix	**Soil, Groundwater and Aqueous QC Samples**
Analytical Group	**Cyanide**
Analytical Method / SOP Reference	**SW-946 9012A/ Empirical SOP-164, 175**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix	Cyanide must be ≤ ½ LOQ.	Stop analysis, correct problem, and recalibrate.	Analyst, Supervisor, Data Validator	Bias/Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Re-prepare and reanalyze the LCS and all associated samples.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
MS	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Flag data with an "N", unless recovery is > 4x the spike added; If the sample results exceed 4x the spike added, then spike the un-spiked aliquot of the sample at 2x the indigenous level or 2x the LOQ.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
Sample Duplicate	One per preparatory batch of 20 or fewer samples of similar matrix	RPD should be ≤ 20%, if concentration is > 5x LOQ; or within ± the LOQ, if the concentration is < 5x LOQ.	Flag data for associated samples with an "*."	Analyst, Supervisor, and Data Validator	Precision	Same as QC Acceptance Limits

Matrix	**Soil, Groundwater and Aqueous Field QC Samples**
Analytical Group	**Perchlorate**
Analytical Method/ SOP Reference	**SW-846 6850 Empirical SOP-239**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Isotope Ratio $^{35}Cl/^{37}Cl$	Every sample, batch QC sample, and standard.	Monitor for the daughter ion at masses 83/85. Theoretical ratio ~ 3.06. Ratio must be within 2.3 to 3.8.	If criteria are not met, the sample must be rerun. If the sample was not pretreated, the sample should be re-extracted using cleanup procedures. If, after cleanup, the ratio still fails, use alternative techniques to confirm presence of perchlorate.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
IS	Every sample, batch QC sample, standard, instrument blank, and method blank.	Measured ^{18}O IS area must be within 50-150% of the average IS area counts of the ICAL. RRT of the perchlorate ion must be within 0.98-1.02.	Rerun the sample at increasing dilutions until the 50-150% acceptance criteria is met. If criteria cannot be met with dilution, interference is suspected - use additional pretreatment steps.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Interference Check Sample (ICS)	One per batch to verify method performance at the matrix conductivity threshold (MCT). At least one ICS must be analyzed daily.	The %R must be within 70-130% of true value.	Correct problem and re-analyze all samples in that batch. Replace cleanup filters or column if necessary. No samples may be reported that are associated with a failing ICS.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Reagent Blank	Prior to calibration, after over-range samples, and at the end of the analytical sequence.	No perchlorate detected > ½ LOQ.	Re-analyze reagent blank (until no carryover is observed) and all samples processed since the contaminated blank. Results may not be reported without a valid reagent blank.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.

Matrix	**Soil, Groundwater and Aqueous Field QC Samples**
Analytical Group	**Perchlorate**
Analytical Method/ SOP Reference	**SW-846 6850 Empirical SOP-239**

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	One per preparatory batch of 20 or fewer samples.	Perchlorate must be ≤ ½ LOQ.	Investigate source of contamination and evaluate the samples and associated QC: i.e. If the blank results are above ½ LOQ, then report sample results which are non-detect. Otherwise, re-prepare blank and associated samples.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
LCS (LCSD not required)	One per preparatory batch of 20 or fewer samples. LCS must be spiked at the LOQ.	%Rs must be between 80-120. RPD should be ≤ 15%, if LCSD is analyzed.	If the %R of a target analyte in the LCS is greater than the upper control limit, and there are no positive findings for that compound, no further action is taken. Otherwise, re-analyze the LCS and affected samples or flag the results. If the %R of any target analyte is below the lower control limit, re-analyze the LCS and affected samples.	Analyst, Supervisor, Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch of 20 or fewer samples per matrix. The MS and MSD must be spiked at the LOQ.	%Rs should be between 80-120. RPD should be ≤ 15%	Flag the parent sample for failed analytes, which exceed the acceptance criteria.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet No. 29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

DOCUMENT	LOCATION MAINTAINED
Sample Collection Documents and Records: • Field logbook (and sampling notes) • Field sample forms (e.g., boring logs, sample log sheets, drilling logs, etc.) • Chain-of-custody records • Sample shipment airbills • Equipment calibration logs • Photographs • Field Task Modification Request forms • SAP • Field Sampling SOPs	Tetra Tech project file; results will be discussed in subject document
Laboratory Documents and Records in the form of analytical data package: • Sample receipt/login form • Sample storage records • Sample preparation logs • Equipment calibration logs • Sample analysis run logs • Reported results for standards, QC checks, and QC samples • Data completeness checklists • Telephone logs • Extraction/clean-up records • Raw data • Electronic Data Deliverables (EDDs)	Tetra Tech project file; long-term data package storage at third-party commercial document storage firm.
Other Documents • HASP • All letter and e-mail correspondence with regulatory agencies, including approvals and comments • Telephone logs • Field investigation data packages • All versions of project reports	Tetra Tech project file.

SAP Worksheet No. 30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

MATRIX	ANALYTICAL GROUP	SAMPLE LOCATIONS/ IDENTIFICATION NUMBERS	ANALYTICAL METHOD	DATA PACKAGE TURNAROUND TIME	LABORATORY / ORGANIZATION (name and address, contact person and telephone number)	BACKUP LABORATORY/ ORGANIZATION (name and address, contact person, and telephone number)
Soil, Groundwater, and Aqueous QC samples	VOCs	See Worksheet No. 18	SW-846 8260B	21 calendar days	Kim Kostzer kkostzer@empirlabs.com Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270 Nashville, TN 37228 (615) 345-1115	None
	PCBs		SW-846 8082A			
	Perchlorate		SW-846 6850			
	Metals		SW-846 6010C			
	Cyanide		SW-846 9012A			

SAP Worksheet No. 31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

ASSESSMENT TYPE	FREQUENCY	INTERNAL OR EXTERNAL	ORGANIZATION PERFORMING ASSESSMENT	PERSON(S) RESPONSIBLE FOR PERFORMING ASSESSMENT (title and organizational affiliation)	PERSON(S) RESPONSIBLE FOR RESPONDING TO ASSESSMENT FINDINGS (title and organizational affiliation)	PERSON(S) RESPONSIBLE FOR IDENTIFYING AND IMPLEMENTING CA (title and organizational affiliation)	PERSON(S) RESPONSIBLE FOR MONITORING EFFECTIVENESS OF CA (title and organizational affiliation)
Laboratory System Audit(1)	Every two years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QAM or Laboratory Manager, Empirical	Laboratory QAM or Laboratory Manager, Empirical	Laboratory QAM or Laboratory Manager, Empirical

1 Empirical is DoD ELAP accredited. The DoD ELAP accreditation letter is included in Appendix C.

SAP Worksheet No. 32 -- Assessment Findings and Corrective Action Responses

(UFP-QAPP Manual Section 4.1.2)

ASSESSMENT TYPE	NATURE OF DEFICIENCIES DOCUMENTATION	INDIVIDUAL(S) NOTIFIED OF FINDINGS (name, title, organization)	TIMEFRAME OF NOTIFICATION	NATURE OF CA RESPONSE DOCUMENTATION	INDIVIDUAL(S) RECEIVING CA RESPONSE (name, title, organization)	TIMEFRAME FOR RESPONSE
Laboratory System Audit	Written audit report	Randy Ward, Laboratory QAM, Empirical	Specified by DoD ELAP Accrediting Body	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

SAP Worksheet No. 33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

TYPE OF REPORT	FREQUENCY (daily, weekly monthly, quarterly, annually, etc.)	PROJECTED DELIVERY DATE(S)	PERSON(S) RESPONSIBLE FOR REPORT PREPARATION (title and organizational affiliation)	REPORT RECIPIENT(S) (title and organizational affiliation)
Data validation report	Per SDG	Within 3 weeks of receipt of laboratory data	DVM or designee, Tetra Tech	PM and project file, Tetra Tech
Major analysis problem identification (Internal Tetra Tech Memorandum)	When persistent analysis problems are detected by Tetra Tech that may impact data usability	Immediately upon detection of problem (on the same day)	CLEAN QAM, Tetra Tech	PM, CLEAN QAM, Program Manager, and project file, Tetra Tech
Project monthly progress report	Monthly for duration of the project	Monthly	PM, Tetra Tech	Navy RPM, Navy; CLEAN QAM, Program Manager, and project file, Tetra Tech
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, Empirical	PM and project file, Tetra Tech
Data validation report	Per SDG	Within 3 weeks of receipt of laboratory data	DVM or designee, Tetra Tech	PM and project file, Tetra Tech

SAP Worksheet No. 34 -- Verification (Step I) Process Table

(UFP-QAPP Manual Section 5.2.1)

VERIFICATION INPUT	DESCRIPTION	INTERNAL/ EXTERNAL	RESPONSIBLE FOR VERIFICATION (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that the samples listed are included in the shipment to the laboratory and that the sample information is accurate. The forms will be signed by the sampler, and a copy will be retained for the project file, Tetra Tech PM, and Tetra Tech Data Validators.	Internal	Sampler and FOL, Tetra Tech
	The Empirical Laboratory Sample Custodian will review the sample shipment for completeness and integrity and will sign accepting the shipment. The Tetra Tech Data Validators will check that the chain-of-custody form was signed and dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory Sample Custodian, Empirical 2 - Data Validators, Tetra Tech
UFP-SAP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed samples listed in the UFP-SAP tables have been collected.	Internal	FOL or designee, Tetra Tech
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL or designee, Tetra Tech
UFP-SAP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain of custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	PM or designee, Tetra Tech
UFP-SAP/ Analytical SOPs/ Analytical Data Packages	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Internal	Laboratory QAM, Empirical
UFP-SAP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the Laboratory QAM will contact the Tetra Tech PM verbally or via e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Empirical
UFP-SAP/ Chain-of-Custody Forms	Check that field QC samples listed in Worksheet No. 20 were collected as required.	Internal	FOL or designee, Tetra Tech
EDDs/Analytical Data Packages	Each EDD will be verified against the chain-of-custody form and hard copy data package for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the DL and the LOQ will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifier.	External	Data Validators, Tetra Tech

Notes: Verification includes field data verification and laboratory data verification. Verification inputs as per Worksheet No. 34 will be checked.

SAP Worksheet No. 35 -- Validation (Steps IIa and IIb) Process Table

(UFP-QAPP Manual Section 5.2.2) (Figure 37 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	UFP-SAP/ Sample Log Sheets	Sample Coordinates - Ensure that sample locations are correct and in accordance with the UFP-SAP proposed locations. Document any discrepancies in the final report.	PM, FOL, or designee, Tetra Tech
IIa	Chain-of-Custody Forms	Custody - Ensure that the custody and integrity of the samples were maintained from collection to analysis, that custody records are complete, and that any deviations are recorded. Review that the samples were shipped and store at the required temperature and that sample pH values for chemically preserved samples met the requirements listed in Worksheet No. 19. Ensure that the analyses were performed within the holding times listed in Worksheet No. 19.	Project Chemist or Data Validators, Tetra Tech
IIa/IIb	UFP-SAP/ Laboratory Data Packages/ EDDs	Accuracy - Ensure that the laboratory QC samples listed in Worksheet No. 28 were analyzed and that the MPCs listed in Worksheet No. 12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Project Chemist or Data Validators, Tetra Tech
		Precision - Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSD, if available. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet No. 12.	
		Representativeness - Check that the laboratory recorded the temperature at sample receipt and the pH values of chemically preserved samples to ensure sample integrity from sample collection to analysis.	
		Completeness - Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet No. 36. Check that all data have been transferred correctly and completely to the final Structured Query Language database.	

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIb	UFP-SAP/ Laboratory Data Packages/ EDDs	Sensitivity - Ensure that the project LOQs listed in Worksheet No. 15 were achieved.	Project Chemist or Data Validators, Tetra Tech
		PSLs - Discuss the impact on reported DLs due to matrix interferences or sample dilutions performed because of the high concentration of one or more other contaminants, on the other target compounds reported as non-detected. Document this usability issue and inform the Tetra Tech PM. Review and add PSLs to the laboratory EDDs. Flag samples and notify the Tetra Tech PM of samples that exceed PSLs listed in Worksheet No. 15.	
		QA/QC - Ensure that all QC samples specified in the UFP-SAP were collected and analyzed and that the associated results were within prescribed UFP-SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the Laboratory QAM shall have contacted the Tetra Tech PM.	
		Deviations - Summarize deviations from methods, procedures, or contracts in the Data Validation Report. Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results. Qualify data results based on method or QC deviation, and explain all the data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifier codes that summarize the reasons for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	

SAP Worksheet No. 36 -- Analytical Data Validation (Steps IIa and IIb) Summary Table

(UFP-QAPP Manual Section 5.2.2.1)

Step **IIa / IIb**	MATRIX	ANALYTICAL GROUP	VALIDATION CRITERIA	DATA VALIDATOR (title and organizational affiliation)
IIa and IIb	Soil, Groundwater, and Aqueous QC Samples	VOCs, PCBs, and Perchlorate	Data validation will be performed using criteria for SW-846 Methods 8260B, 8082A, and 6850 listed in Worksheets Nos. 12, 15, 24, and 28 and the current DoD QSM. If not included in the aforementioned, then the logic outlined in the USEPA CLP National Functional Guidelines for Organic Data Review USEPA-540/R-99-008 (October 1999) will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Soil, Groundwater, and Aqueous QC Samples	Metals and Cyanide	Data validation will be performed using criteria for SW-846 Methods 6010C and 9012A listed in Worksheet Nos. 12, 15, 24, and 28 and the current DoD QSM. If not included in and the aforementioned, then the logic outlined in the USEPA CLP National Functional Guidelines for Inorganic Data Review, USEPA 540-R-04-004 (October 2004) will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech

SAP Worksheet No. 37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment

The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the assessors determine that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics:

Completeness

- For each matrix that was scheduled to be sampled, the FOL (or other person designated by the Tetra Tech PM) acting on behalf of the Project Team will prepare a table listing planned samples/analyses compared to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified, the Tetra Tech PM and risk assessor will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech PM will consult with the Navy RPM and other Project Team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Precision

- The Project Chemist acting on behalf of the Project Team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheet Nos. 12 and 28. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or if data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

Accuracy

- The Project Chemist acting on behalf of the Project Team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet No. 28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability and analyte recoveries for surrogates, MS samples, and LCSs. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness

- A Project Scientist identified by the Tetra Tech PM and acting on behalf of the Project Team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with this UFP-SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

Comparability

- The Project Chemist acting on behalf of the Project Team will determine whether the data generated under this project are sufficiently comparable to historical site data generated by different methods and for samples collected using different procedures and under different site conditions. This will be accomplished by comparing overall precision and bias among data sets for

Data Usability Assessment

each matrix and analytical fraction. This will not require detailed quantitative comparisons unless the Project Chemist indicates that such detailed analysis is required.

Sensitivity

- The Project Chemist acting on behalf of the project team will determine whether project sensitivity goals listed in Worksheet No. 15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Project Chemist will enlist the help of the Project Risk Assessor to evaluate deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

- The Tetra Tech PM and designated team members will evaluate whether project assumptions are valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested. Quantitative assumptions include assumptions related to data distributions (e.g., normal versus log normal) and estimates of data variability. Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. Potential outliers will be removed if a review of the associated data indicates that the results have an assignable cause the renders them inconsistent with the rest of the data. During this evaluation, the team will consider whether outliers could be indications of unanticipated site conditions. Consideration will be given to whether outliers represent an unanticipated site condition.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

After the completion of data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include calculation of simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples with non-detected results, number of samples with detectable results, and the proportion of samples with detected and non-detected results. The Project Team members identified by the PM will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated for by other data. Although rejected data will generally not be used, there may be reason to use them in a weight-of-evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to the sample-specific limit of detection. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations; however, the average of the original and duplicate concentrations will be used to represent the concentration at a particular sampled location.

Identify the personnel responsible for performing the usability assessment:

The Tetra Tech PM, Project Chemist, FOL, and Project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM, Tetra Tech PM, and IDEM PM. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face-to-face meeting or a teleconference, depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

Data Usability Assessment
Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest resampling or other corrective actions, if necessary.

REFERENCES

A.T.Kearney, 1987. Preliminary Review/Visual Site Inspection Report of Naval Weapons Support Center. Crane, Indiana. USEPA ID IN5170023498. Prepared for U.S. Environmental Protection Agency Region V by A. T. Kearney, Inc. March.

IDEM (Indiana Department of Environmental Management), 2009. Risk Integrated System of Closure Users Technical Resource Guidance Document. Indiana Department of Environmental Management, Revised May 1, 2009.

Mitchell, Wayne R. and Elizabeth P. Burrows, 1990. Assessment of Red Phosphorus in the Environment. US Army Biomedical Research and Development Laboratory. Fort Detrick, Frederick, Maryland. Technical Report AD-A221 704. March.

NEESA (Naval Energy and Environmental Support Activity), 1983. Initial Assessment Study of Naval Weapons Support Center Crane. Crane, Indiana. NEESA 13-003. May.

ORNL (Oak Ridge National Laboratory), 1998. Empirical Model for the Uptake of Inorganic Chemicals from Soil by Plants. BJC/OR-133 (for aluminum in soil). September.

Tetra Tech (Tetra Tech NUS, Inc.), 2001. Final Basewide Background Soil Investigation Report, Naval Surface Warfare Center Crane, Crane, Indiana. January.

Tetra Tech, 2010. Health and Safety Plan. Resource Conservation and Recovery Act Facility Investigation. SWMU 27 – Illuminant Building 126. Naval Support Activity (NSA) Crane. Crane, Indiana. August.

USEPA (United States Environmental Protection Agency),1986. Test Methods for Evaluating Solid Waste; Physical/Chemical Methods (SW-846), 3rd Edition (including Update III). Office of Solid Waste and Emergency Response, Washington, DC.

USEPA, 2002. Office of Solid Waste and Emergency Response (OSWER) Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance), Publication EPA530-D-02-052, November.

USEPA, 2002. Guidance for Quality Assurance Project Plans (EPA QA/G-5). USEPA/240/R-02/009. USEPA Office of Environmental Information, Washington DC. December.

USEPA, 2003. Ecological Screening Levels (ESLs) (for water and soil). USEPA Region 5 (http://www.epa.gov/reg5rcra/ca/edql.htm). August.

USEPA, 2005. Ecological Soil Screening Level for Cadmium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-65. March.

USEPA, 2005. Ecological Soil Screening Level for Lead, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-70. March.

USEPA, 2005. Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP), Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs (Final Version 1), EPA-505-B-04-900A. March.

USEPA, 2006. National Recommended Water Quality Criteria: 2006 (for copper). Office of Water.

USEPA, 2007. Ecological Soil Screening Level for Copper, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-68. February.

USEPA, 2007. Ecological Soil Screening Level for Manganese, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-71. April.

USEPA, 2007. Ecological Soil Screening Level for Zinc, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-73. November.

USEPA, 2009. National Recommended Water Quality Criteria: 2009. Office of Water.

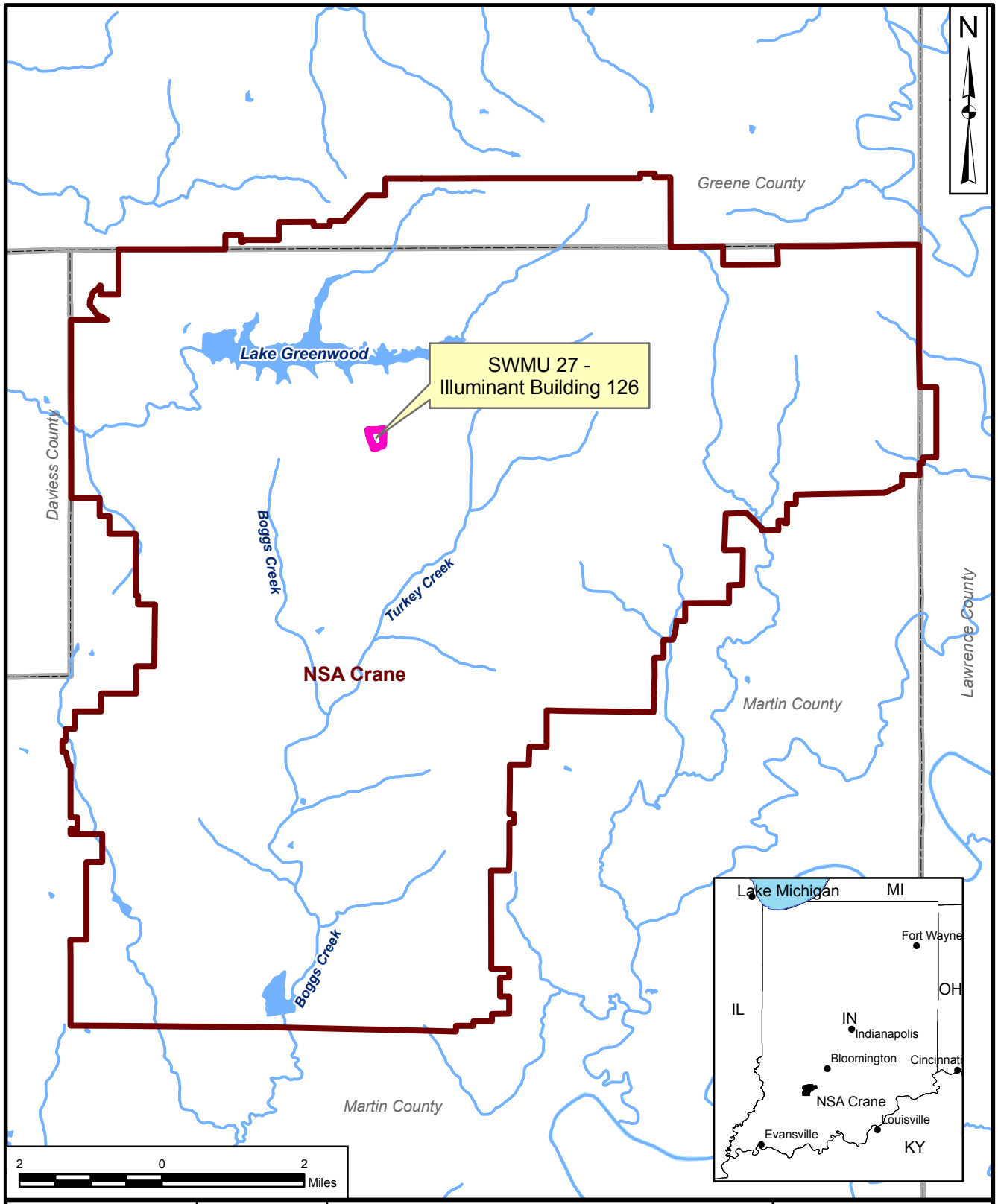
USEPA, 2009. 2009 Edition of the Drinking Water Standards and Health Advisories. Office of Water, Washington, D.C. EPA 822-R-09-011. October.

USEPA, 2009. National Primary Drinking Water Regulations. December.

USEPA, 2010. Regions 3, 6, and 9 Regional Screening Levels for Chemical Contaminants at Superfund Sites. RSL Table Update. May 15.

USEPA, 2010. Regional Screening Levels for Chemical Contaminants at Superfund Sites, prepared by Oak Ridge National Laboratory. http://epa-prgs.ornl.gov/chemicals/index.shtml. May.

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU27_SITE_LOCATION_LARGE_SCALE.MXD 04/01/10 TW



DRAWN BY J. ENGLISH	DATE 09/30/09	SITE LOCATION MAP SWMU 27 - ILLUMINANT BUILDING 126 NSA CRANE CRANE. INDIANA	CONTRACT NUMBER CTO F276
CHECKED BY V. PLACHY	DATE 04/01/10		APPROVED BY ___ DATE ___
REVISED BY T. WHEATON	DATE 04/01/10		APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE ES-1 / REV 0

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU_27_SITE_MAP.MXD 10/20/10 JEE

CONTRACT NUMBER
CTO F276

APPROVED BY — DATE —

APPROVED BY — DATE —

FIGURE NO. FIGURE ES-2

REV 0

SITE LOCATION
IA 1 AND IA 2
SWMU 27 - ILLUMINANT BUILDING 126
NSA CRANE
CRANE, INDIANA

NAVFAC

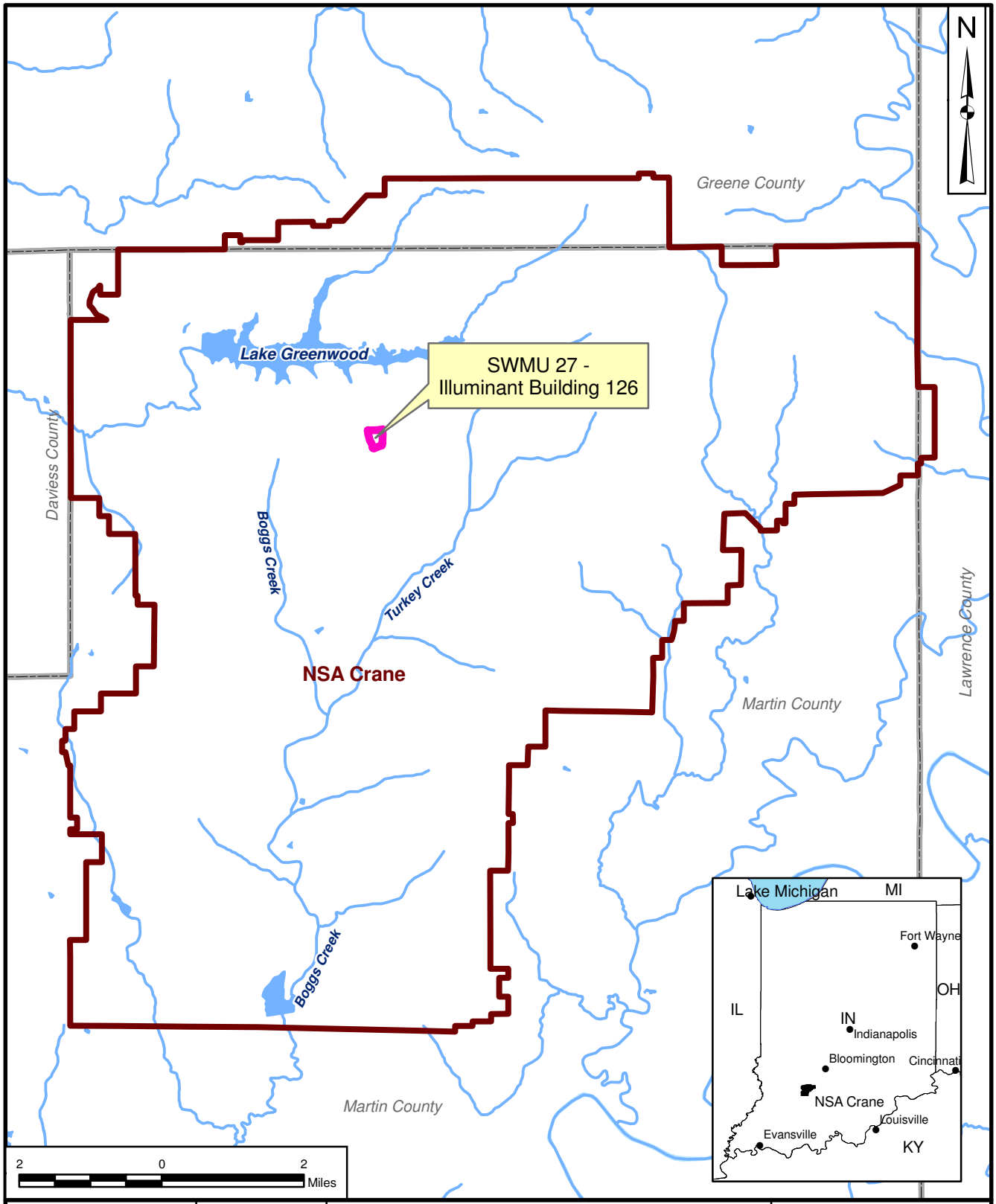
DRAWN BY T. WHEATON DATE 10/16/09

CHECKED BY V. PLACHY DATE 10/20/10

COST/SCHEDULE-AREA

SCALE AS NOTED

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU27_SITE_LOCATION_LARGE_SCALE.MXD 04/01/10 TW



DRAWN BY J. ENGLISH	DATE 09/30/09	SITE LOCATION MAP SWMU 27 - ILLUMINANT BUILDING 126 NSA CRANE CRANE. INDIANA	CONTRACT NUMBER CTO F276
CHECKED BY V. PLACHY	DATE 04/01/10		APPROVED BY ___ DATE ___
REVISED BY T. WHEATON	DATE 04/01/10		APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 10-1 / REV 0

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU_27_SITE_MAP.MXD 10/20/10 JEE

CONTRACT NUMBER
CTO F276

APPROVED BY — DATE —

APPROVED BY — DATE —

FIGURE NO. FIGURE 10-2

REV 0

SITE LOCATION
IA 1 AND IA 2
SWMU 27 - ILLUMINANT BUILDING 126
NSA CRANE
CRANE, INDIANA

NAVFAC

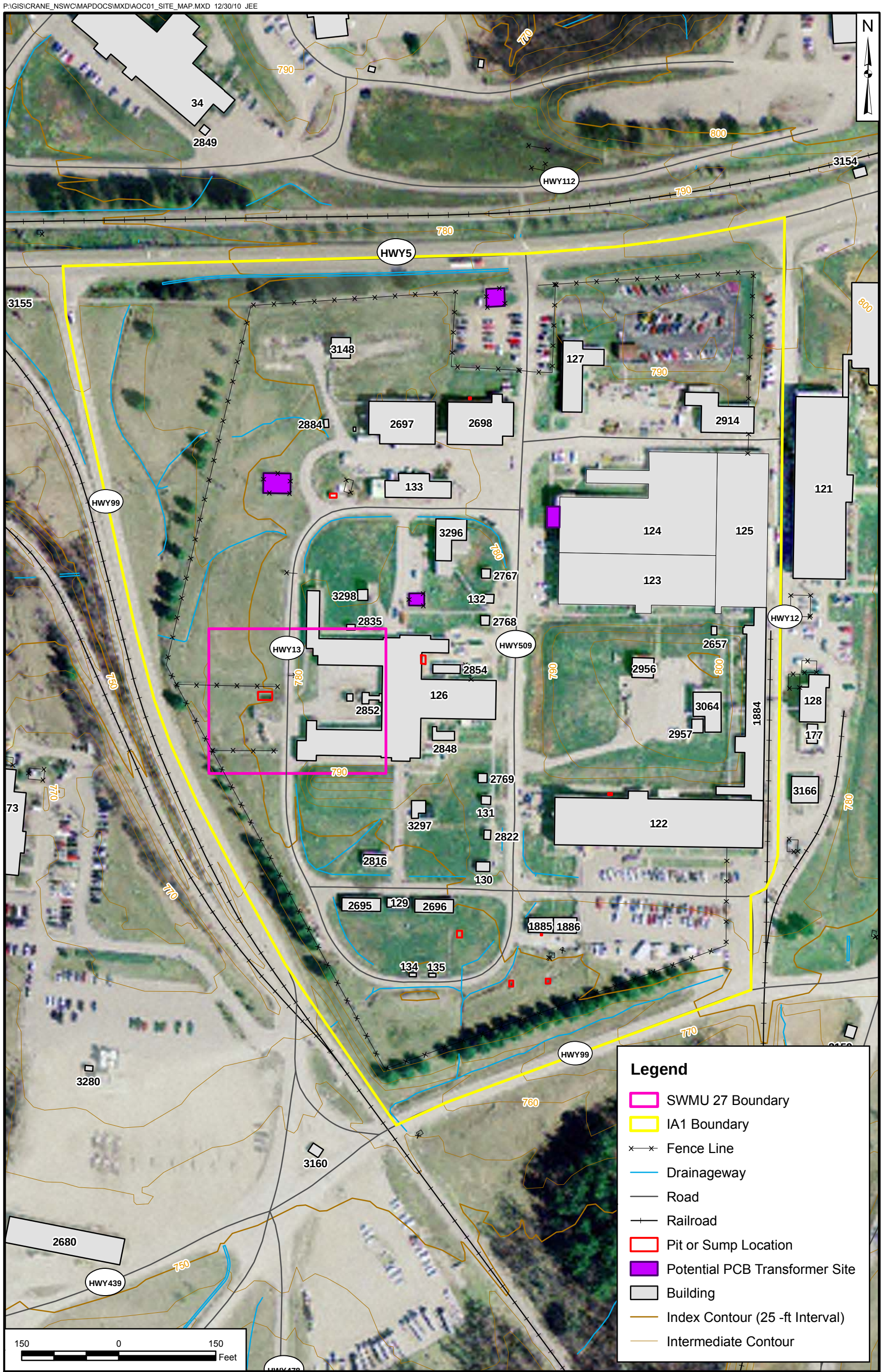
DRAWN BY T. WHEATON DATE 10/16/09

CHECKED BY V. PLACHY DATE 10/20/10

COST/SCHEDULE-AREA

SCALE AS NOTED

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\AOC01_SITE_MAP.MXD 12/30/10 JEE



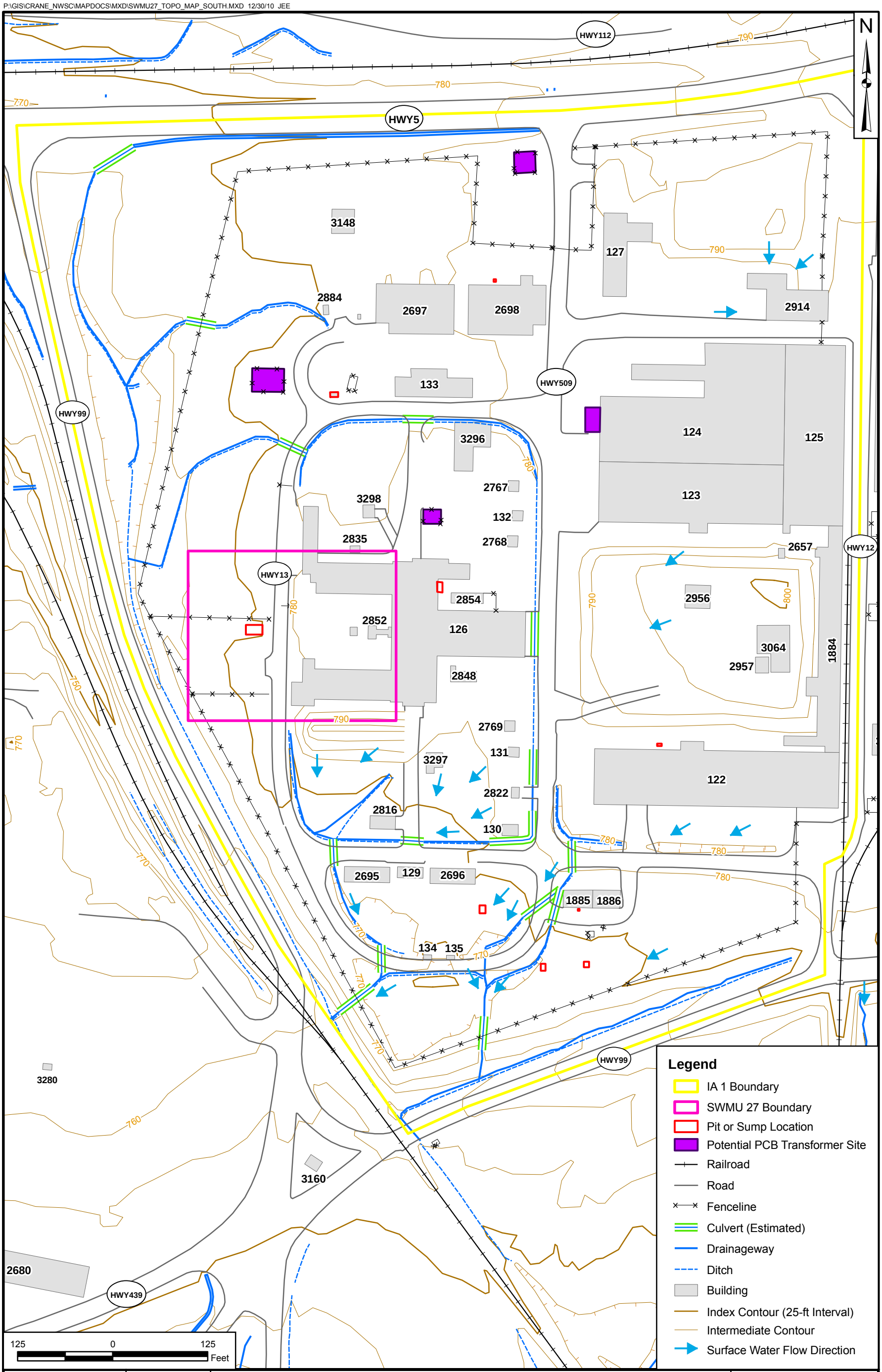
DRAWN BY	DATE
T. WHEATON	10/16/09
CHECKED BY	DATE
V. PLACHY	12/30/10
REVISED BY	DATE
—	—
SCALE	
AS NOTED	

NAVFAC

IA 1 - PYROTECHNIC PROCESS AREA
SWMU 27 - ILLUMINANT BUILDING 126
NSA CRANE
CRANE, INDIANA

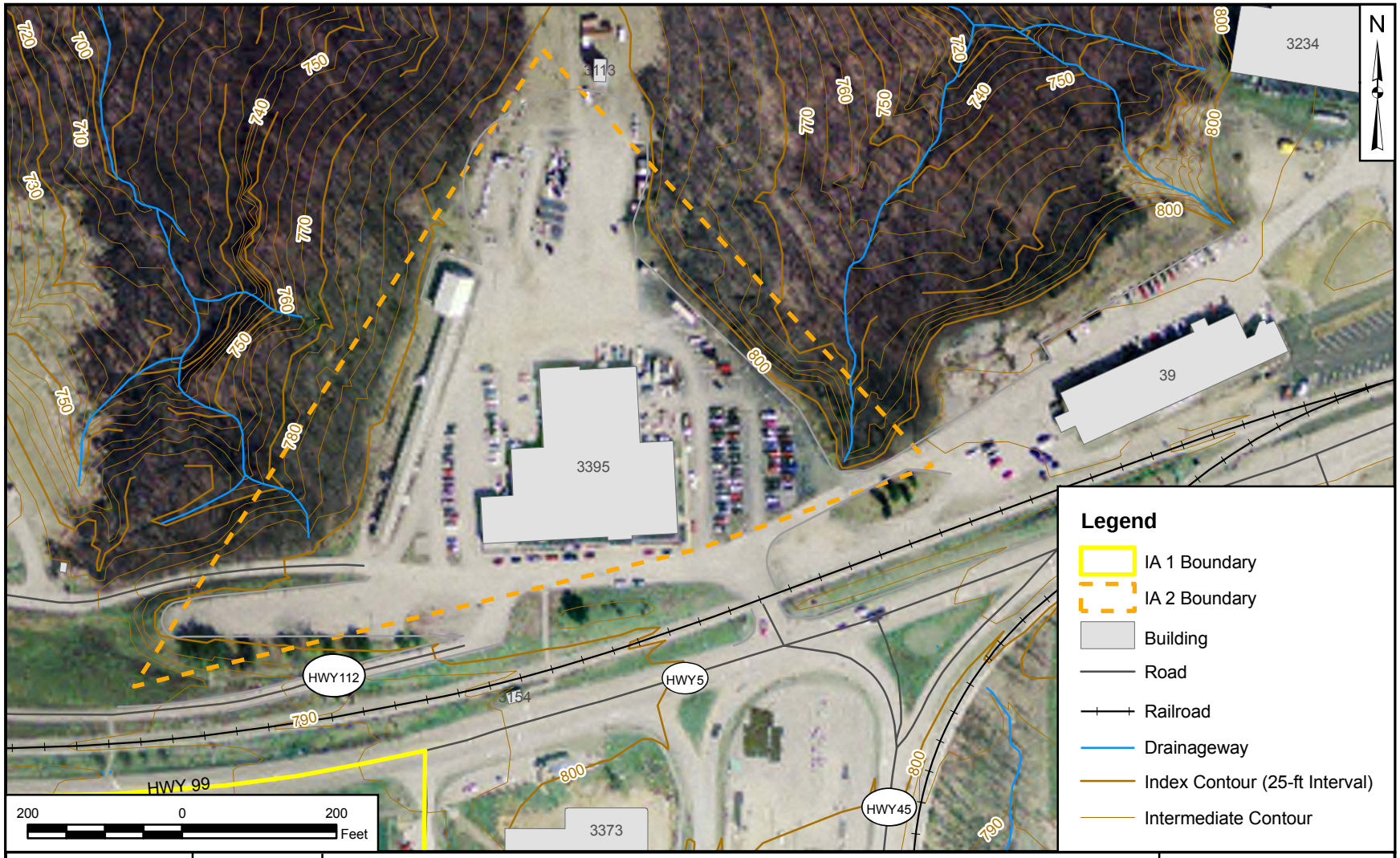
CONTRACT NUMBER	
F276	
APPROVED BY	DATE
—	—
APPROVED BY	DATE
—	—
FIGURE NO.	REV
FIGURE 10-3	0

P:\GIS\CRANE_NWSC\MAPDOCS\MXD\SWMU27_TOPO_MAP_SOUTH.MXD 12/30/10 JEE



DRAWN BY J. ENGLISH	DATE 09/29/09	SWMU 27 - ILLUMINANT BUILDING 126 IA 1 SURFACE WATER FLOW NSA CRANE CRANE, INDIANA	CONTRACT NUMBER CTO F276
CHECKED BY V. PLACHY	DATE 12/30/10		APPROVED BY ___ DATE ___
REVISED BY J. ENGLISH	DATE 12/30/10		APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 10-4 / REV 0

P:\GIS\CRANE\NSWC\MAPDOCS\MXD\SWMU27_NORTH.MXD 12/30/10 JEE



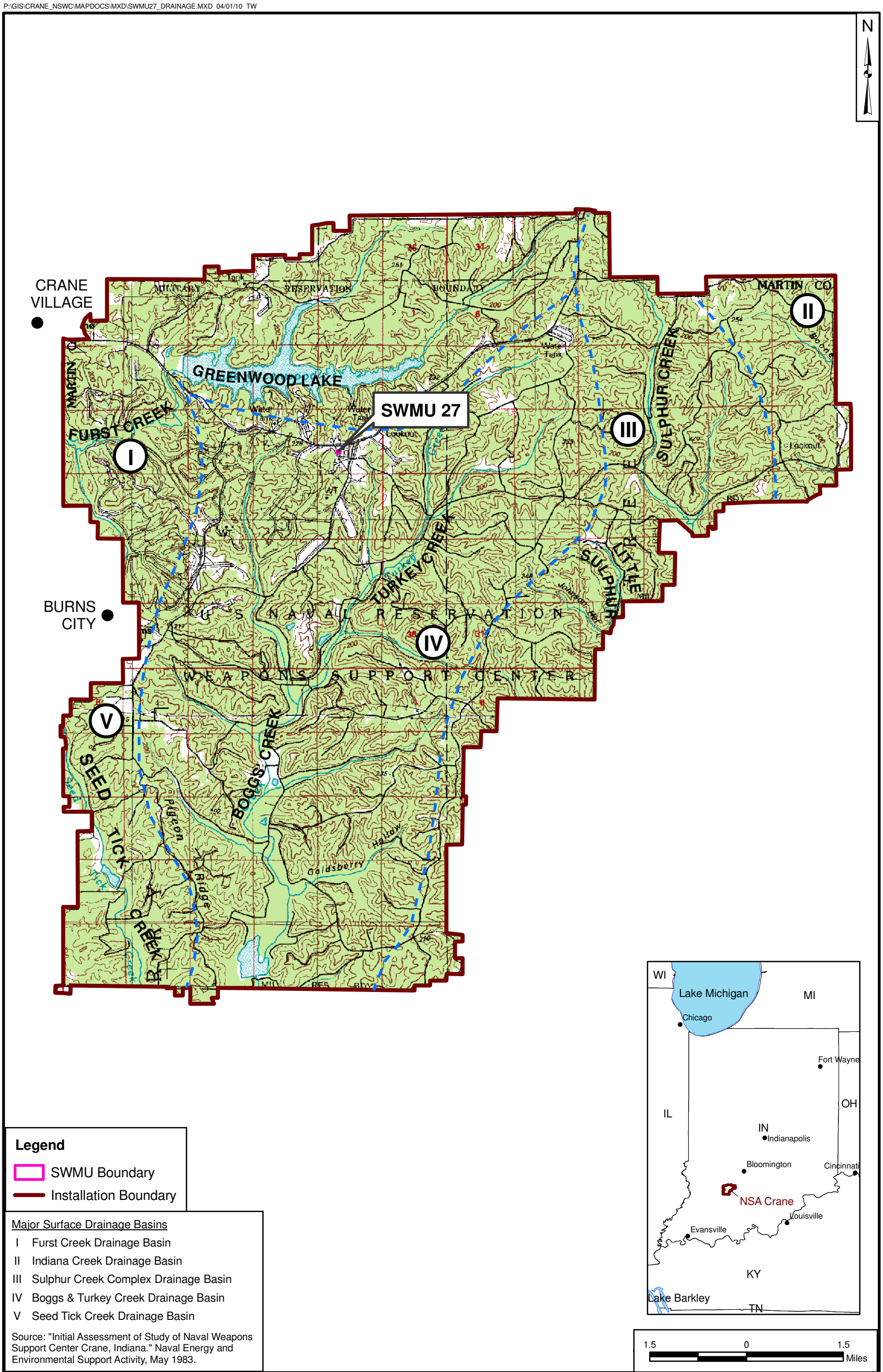
DRAWN BY	DATE
J. ENGLISH	09/28/09
CHECKED BY	DATE
V. PLACHY	12/30/10
REVISED BY	DATE
J. ENGLISH	12/30/10
SCALE	
AS NOTED	

NAVFAC

SWMU 27 - ILLUMINANT BUILDING 126
IA 2 - FLARE TEST AREA
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER	
CTO F276	
APPROVED BY	DATE
___	___
APPROVED BY	DATE
___	___
FIGURE NO.	REV
FIGURE 10-5	0

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU27_DRAINAGE.MXD 04/01/10 TW



Legend

- SWMU Boundary
- Installation Boundary

Major Surface Drainage Basins

- I Furst Creek Drainage Basin
- II Indiana Creek Drainage Basin
- III Sulphur Creek Complex Drainage Basin
- IV Boggs & Turkey Creek Drainage Basin
- V Seed Tick Creek Drainage Basin

Source: "Initial Assessment of Study of Naval Weapons Support Center Crane, Indiana." Naval Energy and Environmental Support Activity, May 1983.

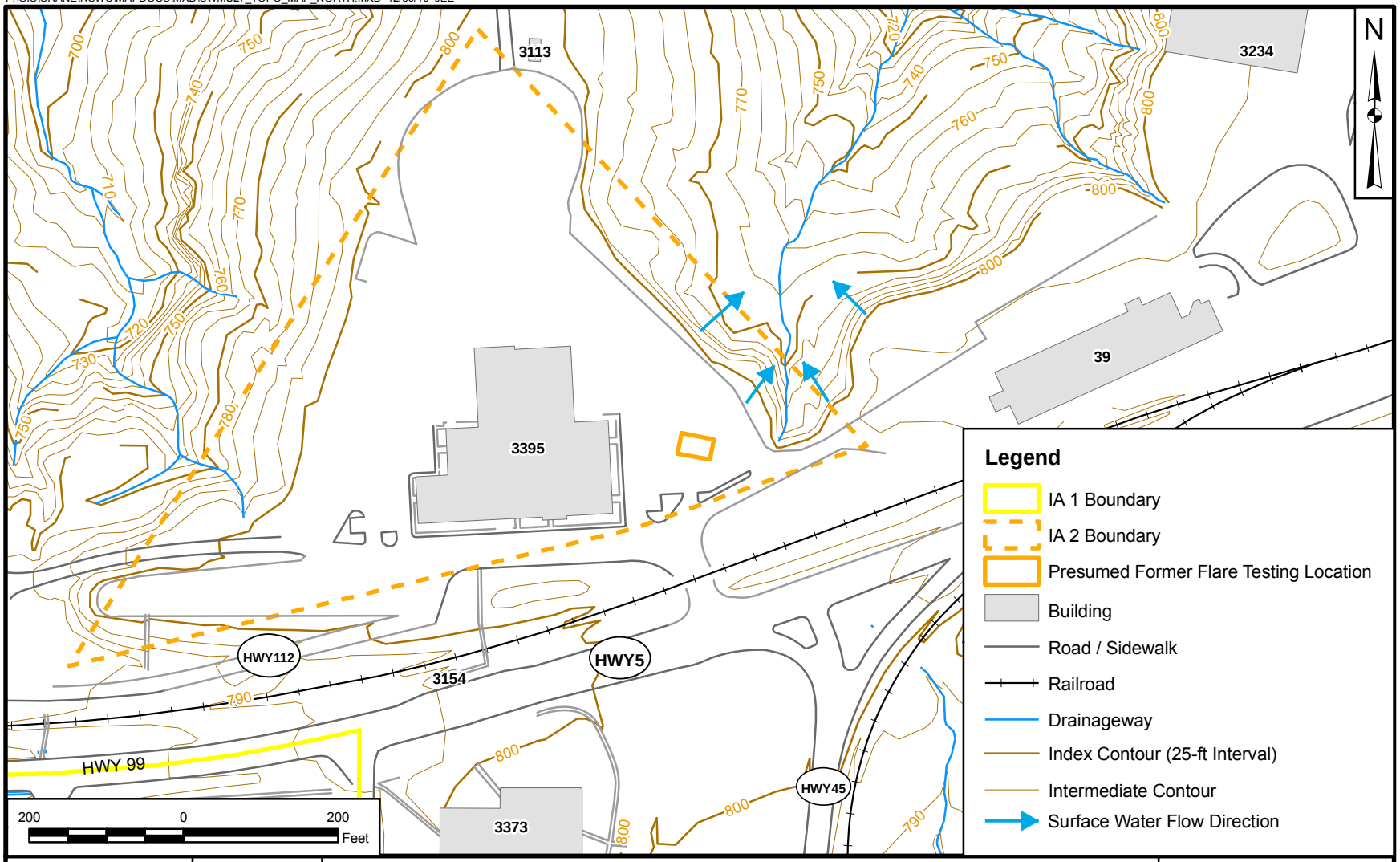
DRAWN BY	DATE
T. WHEATON	04/01/10
CHECKED BY	DATE
V. PLACHY	04/01/10
REVISED BY	DATE
___	___
SCALE	
AS NOTED	

NAVFAC

SWMU 27 - ILLUMINANT BUILDING 126
SURFACE WATER DRAINAGE
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER	
CTO F276	
APPROVED BY	DATE
___	___
APPROVED BY	DATE
___	___
FIGURE NO.	REV
FIGURE 10-6	0

P:\GIS\CRANE\NSWC\MAPDOCS\MXD\SWMU27_TOPO_MAP_NORTH.MXD 12/30/10 JEE

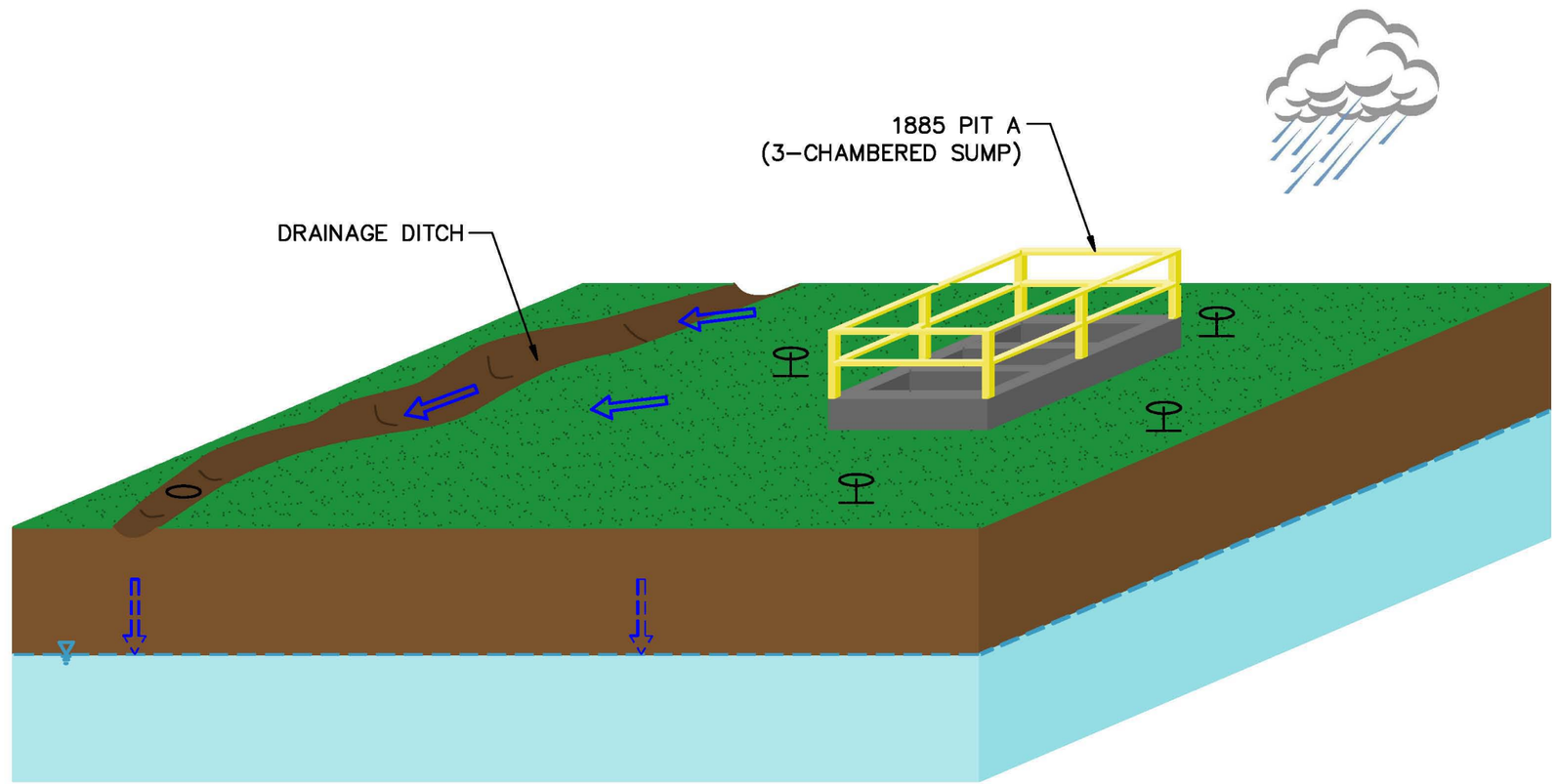


DRAWN BY	DATE
J. ENGLISH	09/28/09
CHECKED BY	DATE
V. PLACHY	12/30/10
REVISED BY	DATE
J. ENGLISH	12/30/10
SCALE	
AS NOTED	

NAVFAC

SWMU 27 - ILLUMINANT BUILDING 126
IA 1 - SURFACE WATER FLOW
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER	
CTO F276	
APPROVED BY	DATE
___	___
APPROVED BY	DATE
___	___
FIGURE NO.	REV
FIGURE 10-7	0

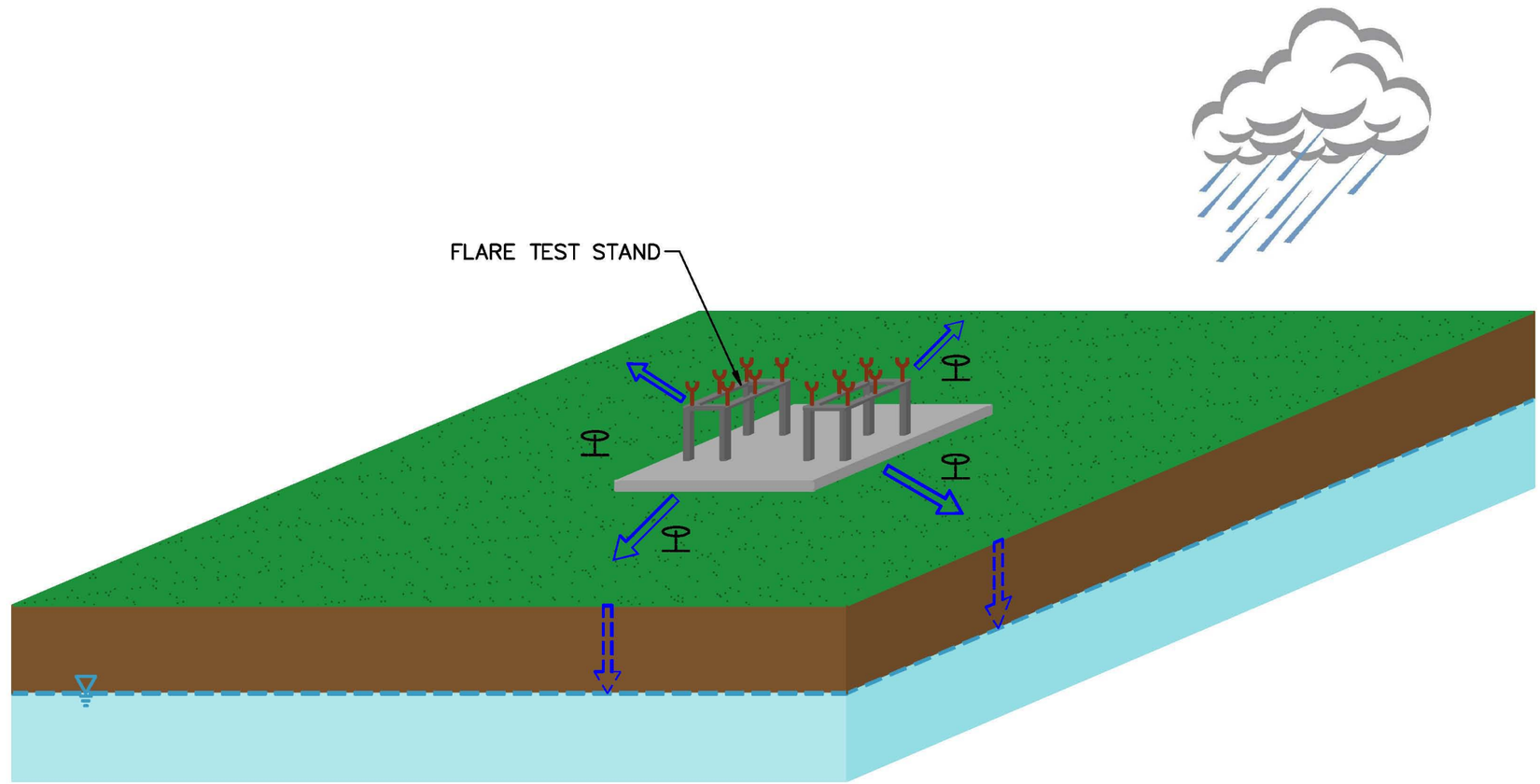


LEGEND

- WATER TABLE ELEVATION (ESTIMATED)
- SURFACE RUNOFF FLOW DIRECTION
- INFILTRATION
- 0-2 FT BGS

DRAWN BY CK	DATE 4/07/10	CONCEPTUAL SITE MODEL CATCH BASINS, SETTLING BASINS, AND DRAINAGWAYS NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY	DATE		OWNER NO.
REVISED BY	DATE		APPROVED BY / DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10-8 / REV. 0

R:\2126\CSM\2126CSM01.dwg PIT CARLY.KRAMER 5/19/2010 8:19:57 AM



LEGEND

SURFACE RUNOFF FLOW DIRECTION

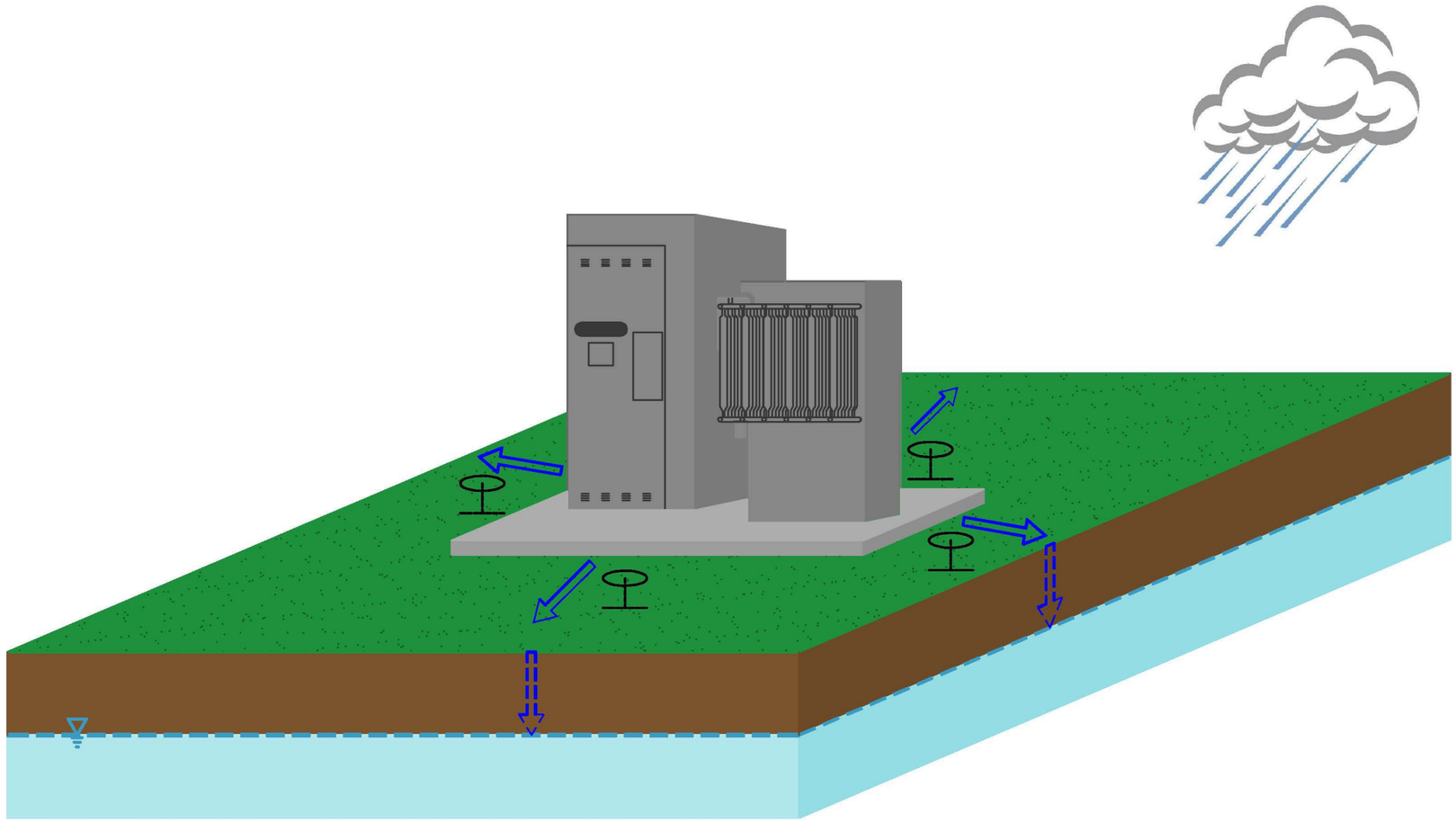
INFILTRATION

WATER TABLE ELEVATION (ESTIMATED)

0–2 FT BGS

DRAWN BY CK	DATE 4/07/10	CONCEPTUAL SITE MODEL FLARE TESTING ACTIVITIES NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY	DATE		OWNER NO.
REVISED BY	DATE		APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10–9 REV. 0

R:\2126\CSM\2126CSM02.dwg PIT CARLY.KRAMER 5/19/2010 8:37:18 AM



LEGEND

SURFACE RUNOFF FLOW DIRECTION

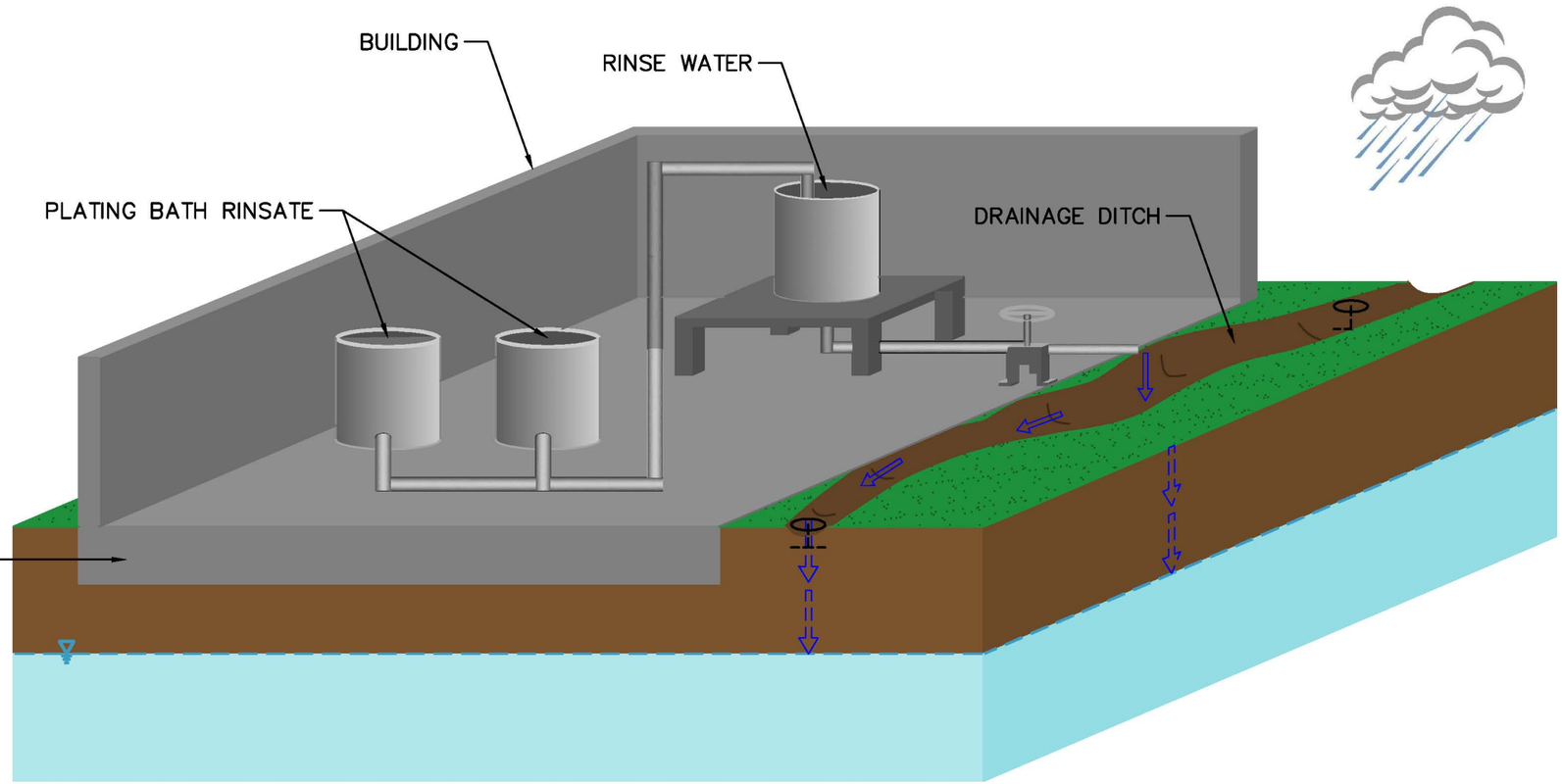
INFILTRATION

WATER TABLE ELEVATION (ESTIMATED)

0–2 FT BGS

DRAWN BY CK	DATE 4/07/10	NAVFAC	CONCEPTUAL SITE MODEL POLYCHLORINATED BIPHENYLS (PCBs) TRANSFORMERS NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY	DATE			OWNER NO.
REVISED BY	DATE			APPROVED BY / DATE
SCALE NOT TO SCALE				DRAWING NO. FIGURE 10–10 / REV. 0

R:\2126\CSM\2126CSM03.dwg PIT CARLY.KRAMER 5/19/2010 8:40:28 AM



LEGEND

SURFACE RUNOFF FLOW DIRECTION

INFILTRATION

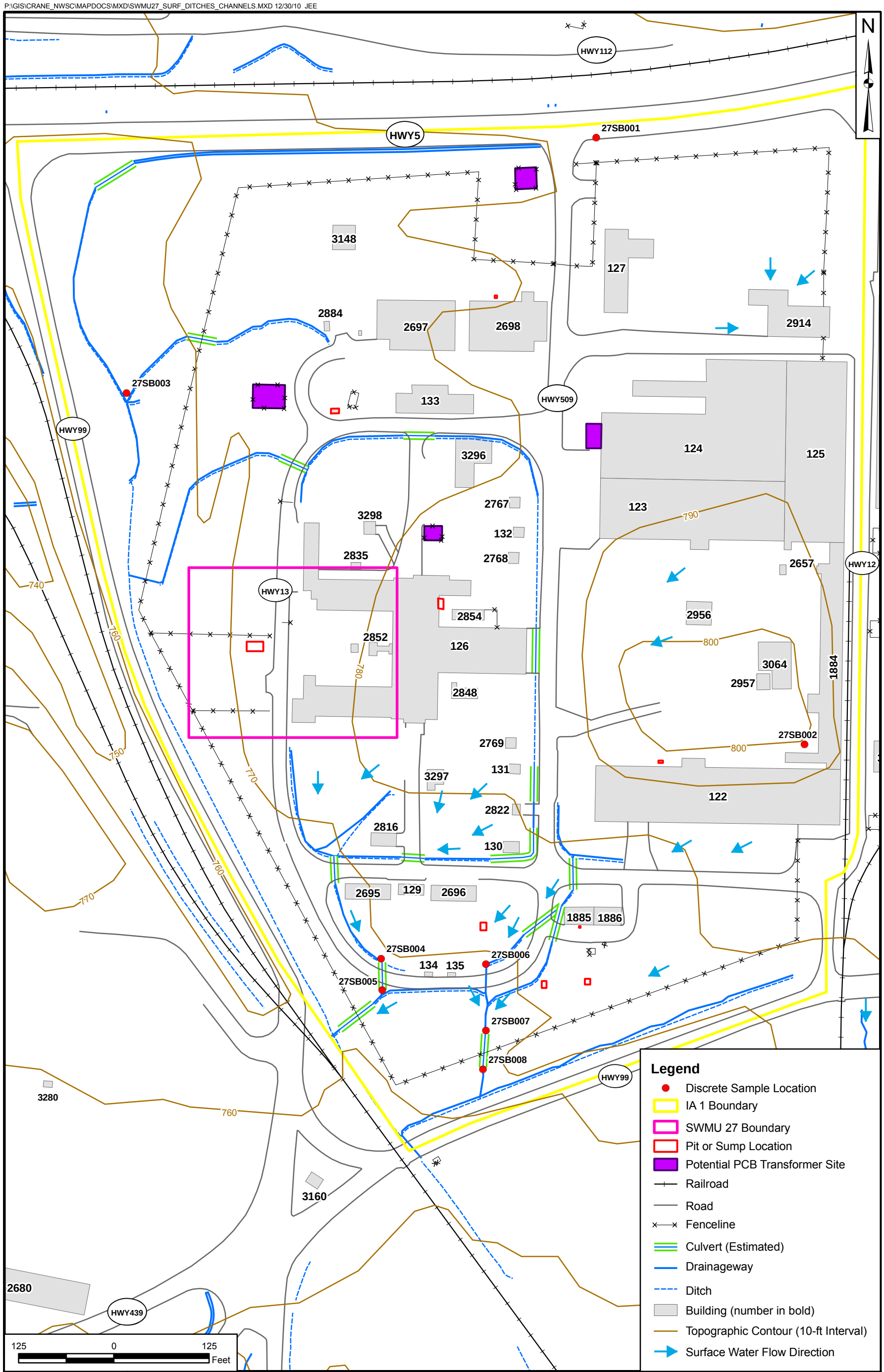
WATER TABLE ELEVATION (ESTIMATED)

0–1 FT BGS

DRAWN BY CK DATE 4/07/10	NAVFAC	CONCEPTUAL SITE MODEL METAL PLATING DRAINAGE NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY DATE			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10–11 / REV. 0

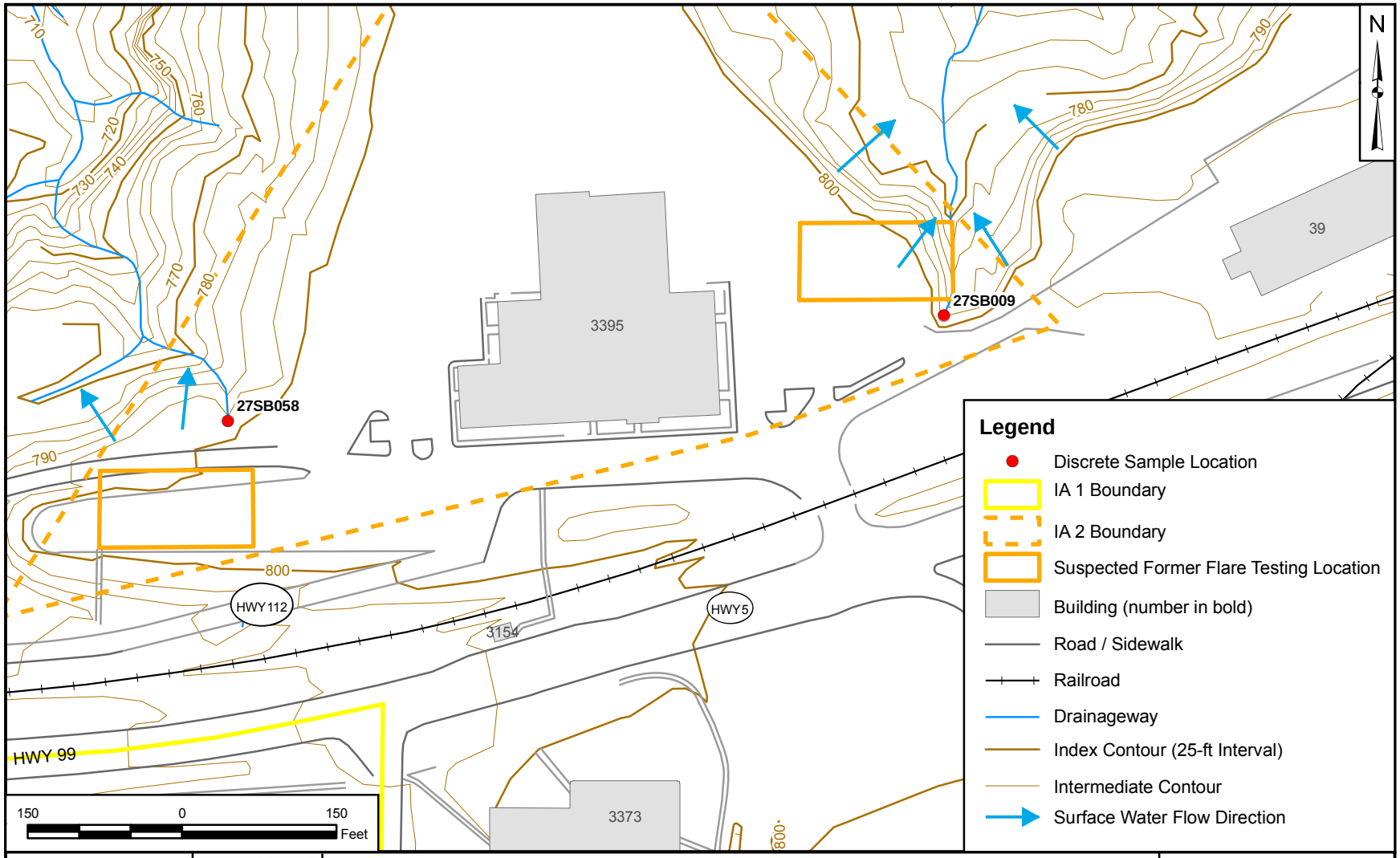
R:\2126 – NSA Crane\CSM\2126CSM04.dwg PIT CARLY.KRAMER 10/20/2010 11:44:29 AM

P:\GIS\CRANE_NWSC\MAPDOCS\MXD\SWMU27_SURF_DITCHES_CHANNELS.MXD 12/30/10 JEE



DRAWN BY S. STROZ	DATE 06/29/10	IA 1 DITCHES/CHANNELS/DRAINAGEWAYS SWMU 27 - ILLUMINANT BUILDING 126 SURFACE SOIL SAMPLE LOCATIONS NSA CRANE CRANE, INDIANA	CONTRACT NUMBER CTO F276
CHECKED BY V. PLACHY	DATE 12/30/10		APPROVED BY ___ DATE ___
REVISED BY J. ENGLISH	DATE 12/30/10		APPROVED BY ___ DATE ___
SCALE AS NOTED	NAVFAC		FIGURE NO. FIGURE 17-1 / REV 0

P:\GIS\CRANE\NSWC\MAPDOCS\MXD\SWMU27_IA2_SURF_DITCHES.MXD 12/30/10 JEE



DRAWN BY	DATE
T. WHEATON	10/06/10
CHECKED BY	DATE
V. PLACHY	12/30/10
REVISED BY	DATE
—	
SCALE	
AS NOTED	

NAVFAC

IA 2 DITCHES/DRAINAGEWAYS SURFACE SOIL SAMPLE LOCATIONS

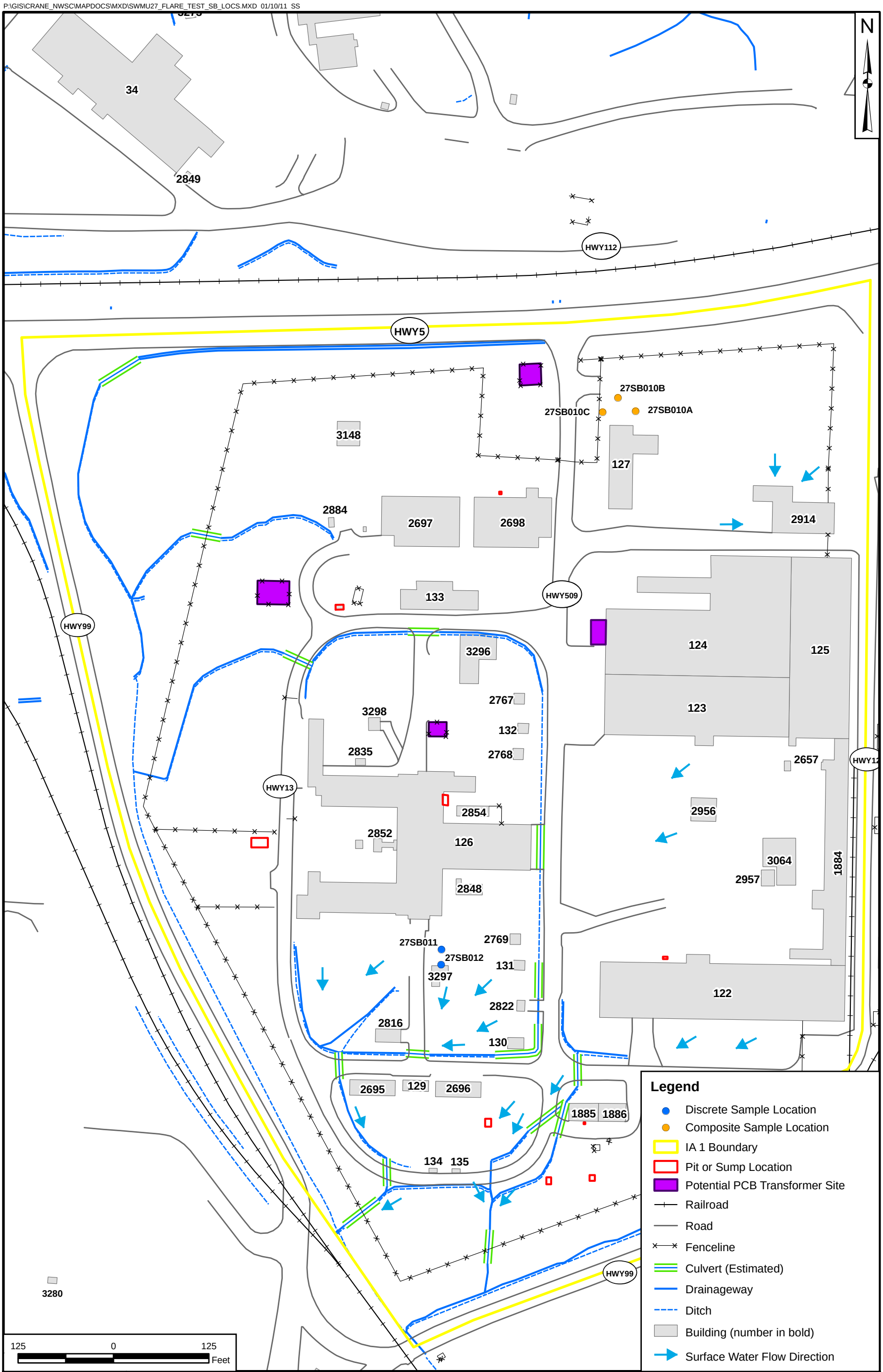
SWMU 27 - ILLUMINANT BUILDING 126

NSA CRANE

CRANE, INDIANA

CONTRACT NUMBER	
CTO F276	
APPROVED BY	DATE
—	—
APPROVED BY	DATE
—	—
FIGURE NO.	REV
FIGURE 17-2	0

P:\GIS\CRANE_NWSC\MAPDOCS\MXD\SWMU27_FLARE_TEST_SB_LOCS.MXD 01/10/11 SS



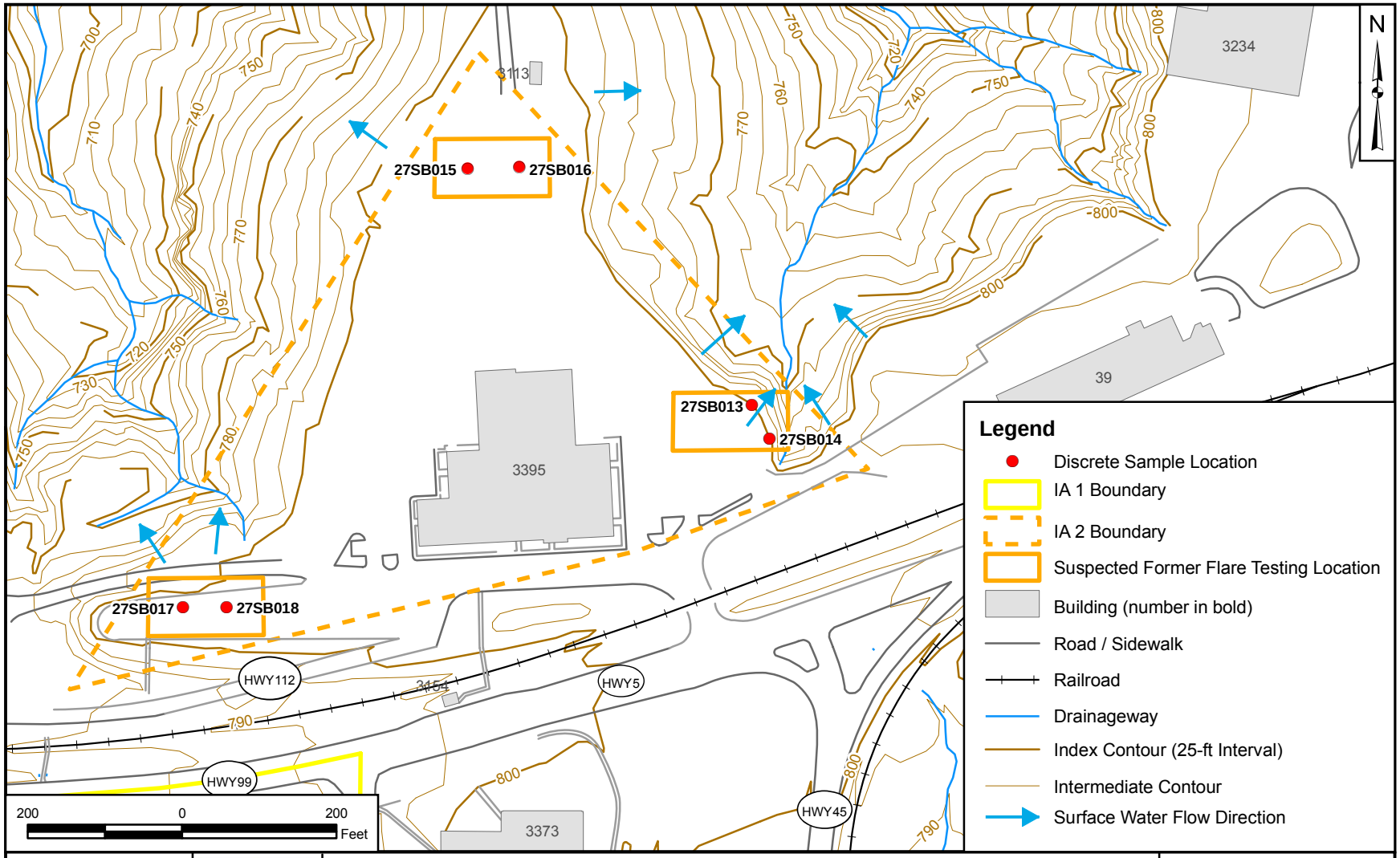
DRAWN BY	DATE
S. STROZ	06/29/10
CHECKED BY	DATE
V. PLACHY	01/10/11
REVISED BY	DATE
S. STROZ	01/10/11
SCALE	
AS NOTED	

NAVFAC

IA 1 FLARE TEST AND FIRE INCIDENT AREAS
SWMU 27 - ILLUMINANT BUILDING 126
SURFACE SOIL SAMPLE LOCATIONS
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER	
CTO F276	
APPROVED BY	DATE
___	___
APPROVED BY	DATE
___	___
FIGURE NO.	REV
FIGURE 17-3	0

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU27_IA2_SUSP_FLARE_TEST.MXD 12/30/10 JEE



DRAWN BY	DATE
T. WHEATON	10/06/10
CHECKED BY	DATE
V. PLACHY	12/30/10
REVISED BY	DATE
—	—
SCALE	
AS NOTED	

NAVFAC

IA 2 SUSPECTED FLARE TEST AREA PROPOSED SOIL SAMPLE LOCATIONS

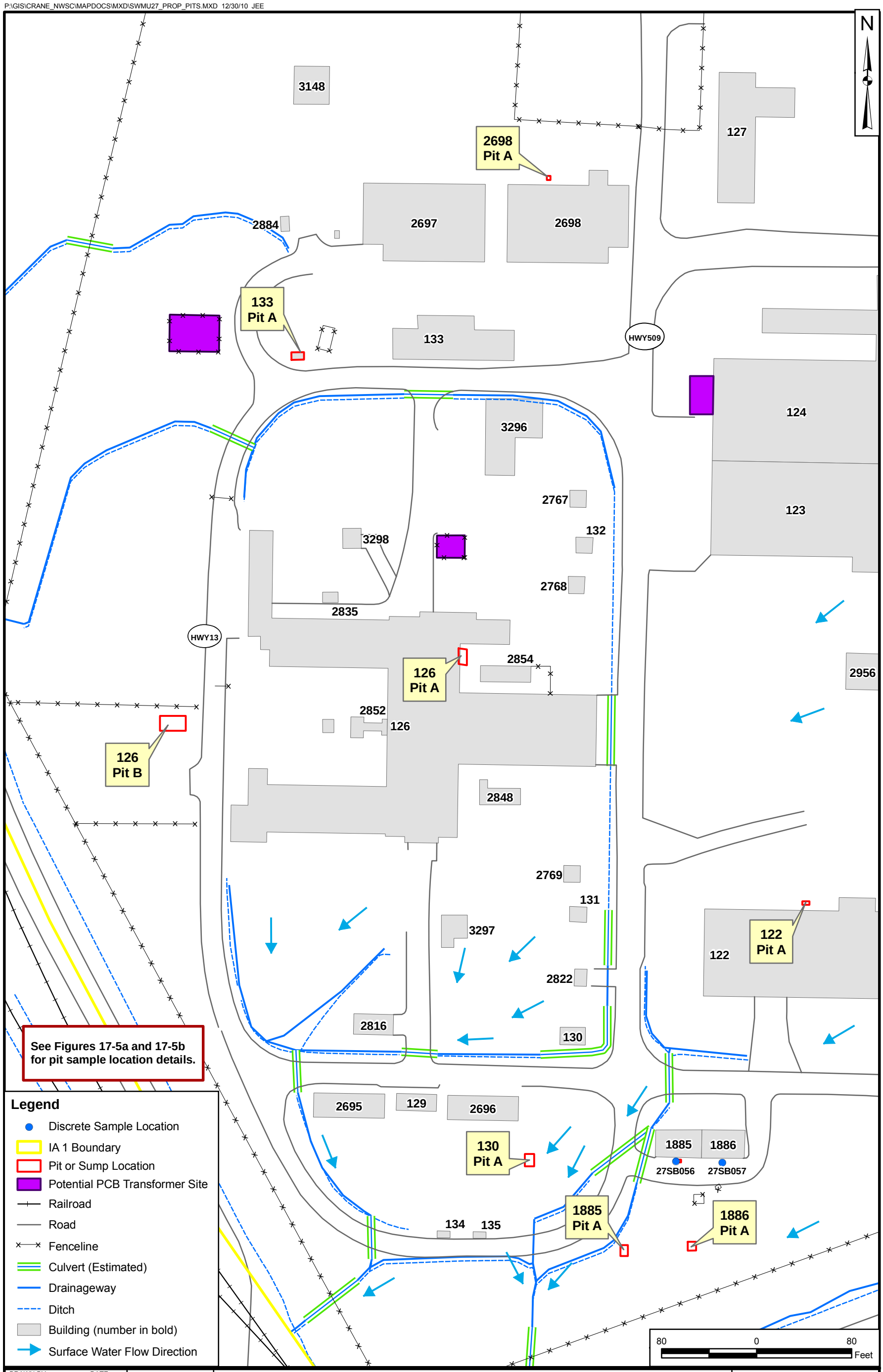
SWMU 27 - ILLUMINANT BUILDING 126

NSA CRANE

CRANE, INDIANA

CONTRACT NUMBER	
CTO F276	
APPROVED BY	DATE
—	—
APPROVED BY	DATE
—	—
FIGURE NO.	REV
FIGURE 17-4	0

P:\GIS\CRANE_NWSC\MAPDOCS\MXD\SWMU27_PROP_PITS.MXD 12/30/10 JEE



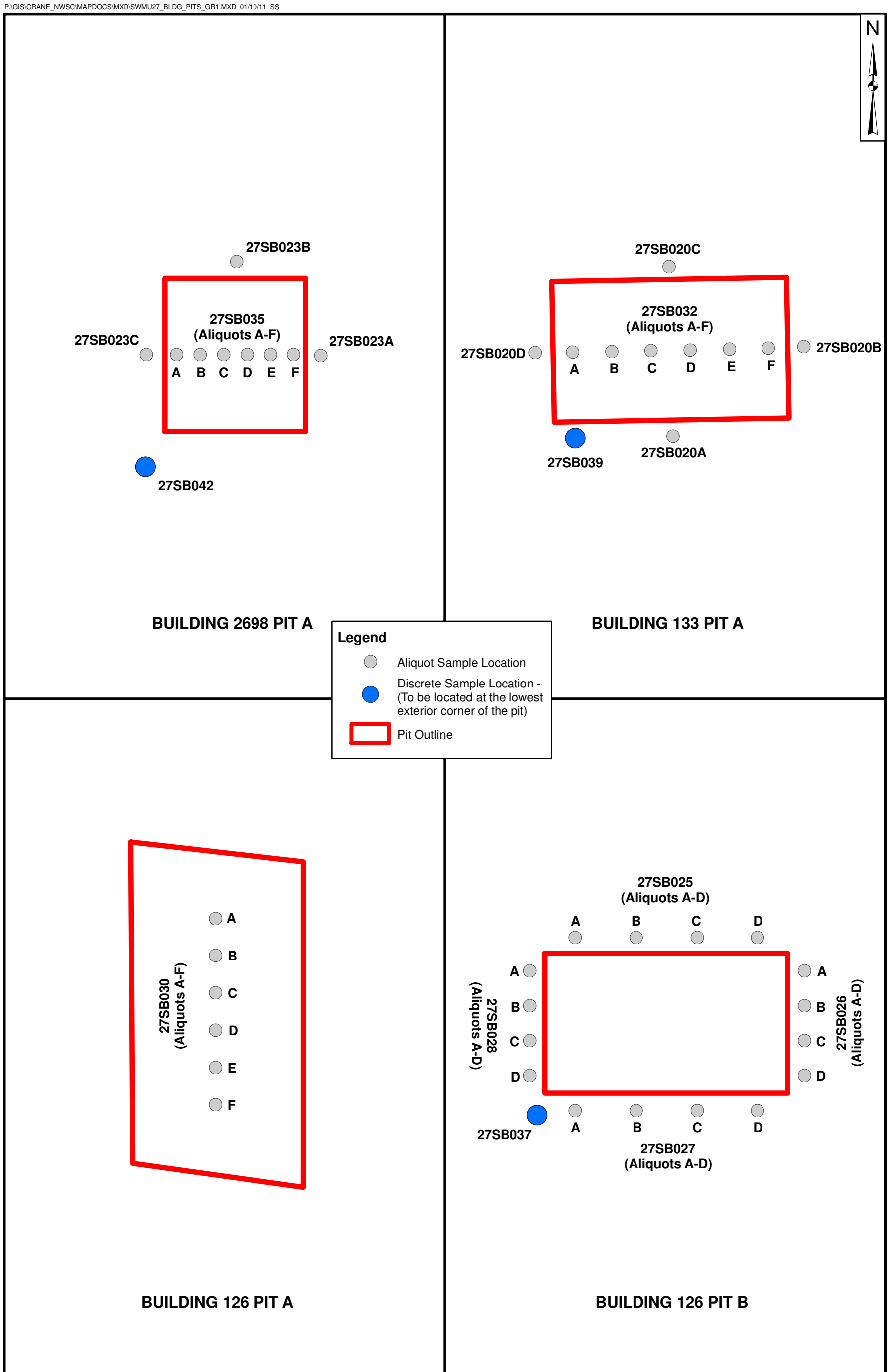
DRAWN BY	DATE
S. STROZ	06/29/10
CHECKED BY	DATE
V. PLACHY	12/30/10
REVISED BY	DATE
J. ENGLISH	12/30/10
SCALE	
AS NOTED	

NAVFAC

PITS/SUMPS/CATCH BASINS
SWMU 27 - ILLUMINANT BUILDING 126
SOIL AND RESIDUAL SAMPLE LOCATIONS
NSA CRANE
CRANE, INDIANA

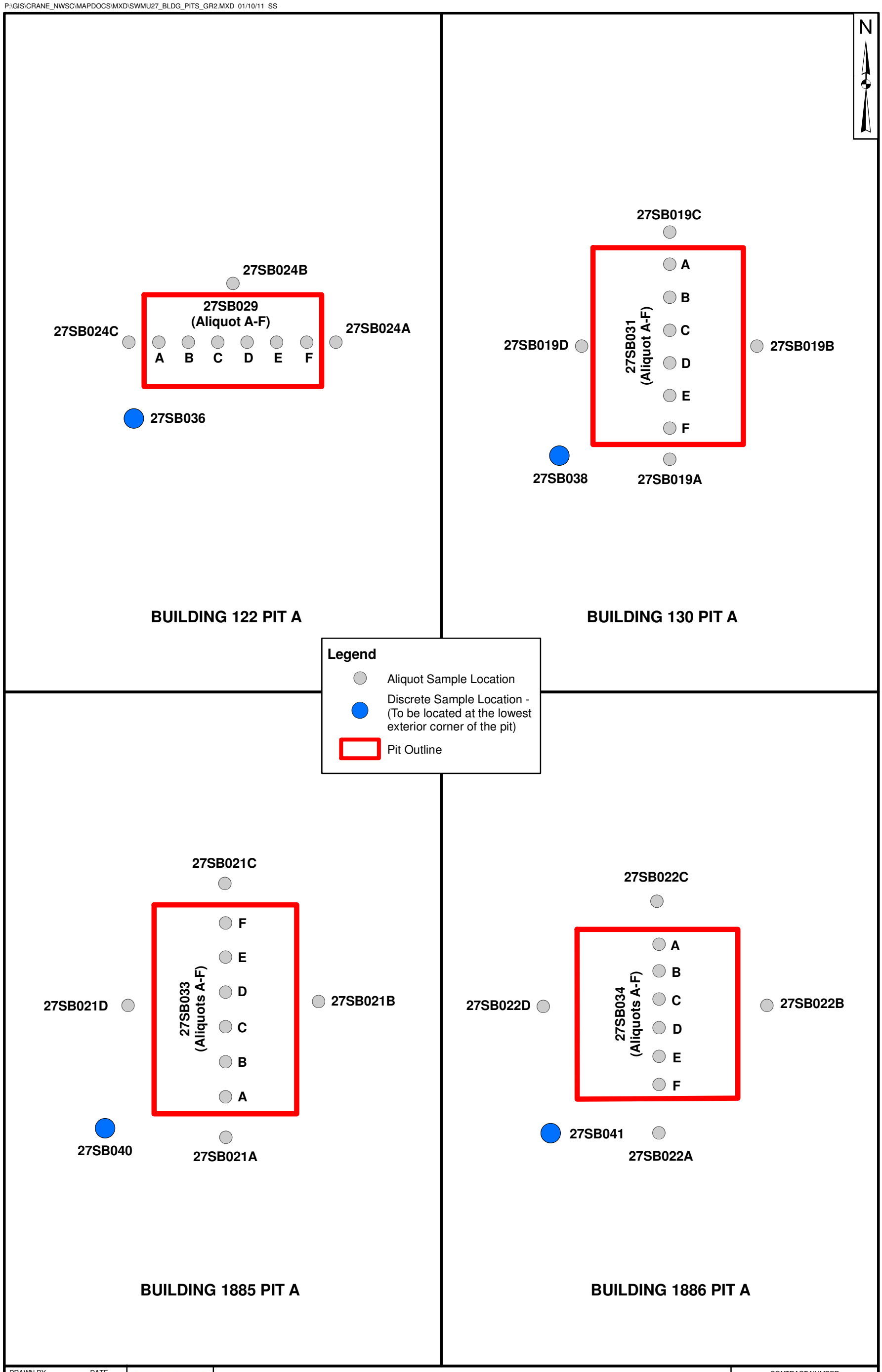
CONTRACT NUMBER	
CTO F276	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO.	REV
FIGURE 17-5	0

P:\GIS\CRANE_NWSC\MAPDOCS\MXD\SWMU27_BLDG_PITS_GR1.MXD 01/10/11 SS



DRAWN BY M. MAGUIRE	DATE 09/02/10	BUILDING 2698 PIT A, BUILDING 133 PIT A, BUILDING 126 PIT A, BUILDING 126 PIT B SWMU 27 - ILLUMINANT BUILDING 126 SOIL AND RESIDUAL SAMPLE LOCATIONS NSA CRANE CRANE, INDIANA	CONTRACT NUMBER CTO F276
CHECKED BY V. PLACHY	DATE 01/10/11		APPROVED BY ___ DATE ___
REVISED BY	DATE		APPROVED BY ___ DATE ___
SCALE NOT TO SCALE			FIGURE NO. FIGURE 17-5a / REV 0

P:\GIS\CRANE_NWSC\MAPDOCS\MXD\SWMU27_BLDG_PITS_GR2.MXD 01/10/11 SS



DRAWN BY	DATE			CONTRACT NUMBER
M. MAGUIRE	09/02/10	NAVFAC	BUILDING 122 PIT A, BUILDING 130 PIT A, BUILDING 1885 PIT A, BUILDING 1886 PIT A SWMU 27 - ILLUMINANT BUILDING 126 SOIL AND RESIDUAL SAMPLE LOCATIONS NSA CRANE CRANE, INDIANA	CTO F276
CHECKED BY	DATE			APPROVED BY / DATE
V. PLACHY	01/10/11			APPROVED BY / DATE
REVISED BY	DATE			FIGURE NO. / REV
SCALE				FIGURE 17-5b / 0
NOT TO SCALE				

APPENDIX A

SITE INFORMATION

A.1 Detailed Site and Process Description

A.2 September 2009 Site Visit Information

A.3 June 2010 Site Visit Information

A.4 Red Phosphorus Technical Report

A.5 Historical Aerial Photographs

A.1

DETAILED SITE AND PROCESS DESCRIPTION

APPENDIX A
DETAILED SITE DESCRIPTION

This appendix presents detailed background information about SWMU 27 – Illuminant Building 126.

A.1.1 PHYSICAL SITE DESCRIPTION

SWMU 27 – Illuminant Building 126 is located in the north-central portion of NSWC Crane. The Site is contained within the boundary of NSA Crane and is located in the central-northwest portion of the Base. For the purposes of this UFP-SAP, the Site is an expansion of the Illuminant Building 126 that includes 2 Investigation Areas (IA): IA 1, the Pyrotechnic Production Area (PPA), and IA 2, the former Flare Test Area.

The PPA (IA 1) is trapezoid shape that is bounded by Highway 5 on the north and Highway 99 on the south. The PPA produces pyrotechnics devices such as flares. IA 1 covers approximately 31-acres and is located within a restricted access fencing. While most of NSA Crane is forested, IA 2 is industrialized. The southern, western, and northwestern portions of IA 1 are grassland. IA 1 surface water from areas generally north and west of Building 126 flow into surface water channels running off site towards the Highway 99; surface water from areas east and south of flow into surface water channels that also run off the Site towards Highway 99. The nearest streams are to the west and drain into the Boggs Creek Watershed. Turkey Creek is east of the PPA, but drain swales and local topography convey surface flow in the PPA to the south and west (near the point where the railroad track R-1 crosses Highway 99). Throughout the PPA are various concrete basins/pits that previously flowed to surface water drainage changes and have overflowed onto surface soil. The PPA is an active production area with a chain link fence boarding the area. Access is controlled by two locked gates; one on the north and the other on the east portion of the fence.

The former Flare Test Area (IA 2) is represented as an ellipse whose location has been determined to be north of Highway 5. IA 2 covers approximately 31-acres and has an open access area within NSA Crane. Surface water from the former Flare Test Area drains into channels that parallel the north side of Highway 5 or down the culvert north of IA 2.

The PPA highest elevation (800 feet above mean sea level (msl)) is the manmade mound for the PPA Wastewater Treatment Plant (Building 3064). Surface water, from areas northwest of Building 3064, drain to channels that exit the PPA to the south drainage channel along Highway 99. Surface water, from areas southwest of Building 3064, drain to channels along the western and southern boundaries of the PPA and exit the Site into drainage channels along Highway 99.

Physical structures at the PPA are presented in Table A.1-1. These structures included pyrotechnic production (Buildings 122, 123, 126, 130, and 2697), red phosphorous pressing house (Building 133), laboratory (Building 127), plating and maintenance buildings (Buildings 1884), phosphorous mixing buildings (Buildings 1884 and 1885), a wastewater treatment plant (Building 3064), numerous storage magazines/buildings, curing buildings, and miscellaneous inactive buildings. Empty contains are stored west, east, and southeast of Building 1885/1886.

Condensate from steam traps and condensate collection systems flow onto surface soil west of Building 126, north of Building 2697, south of Building 3296, and west of Building 122. Various surface drains with unknown discharge points exist throughout the PPA (north of Building 122, southwest of Building 1884, north of Building 127, southeast of Building 3297, and west of Building 133).

An electrical transformer labeled as containing polychlorinated biphenyls (PCBs) is located on concrete pad north of Building 126; a second electrical transformer also labeled as containing PCBs is located on a concrete pad west of Building 124. Former electric substation that contained PCB transformers was located in the current fenced area across the road west of Building 133.

A.1.2 SITE HISTORY AND BACKGROUND

Primarily, the Site was used for industrial activity such as loading, modifying, and renovating of pyrotechnic items and compounds. However, testing of pyrotechnic devices, that is flare testing, also occurred at the Site with the majority of the flare testing occurring at IA 2 and limited flare testing at IA 1 on a concrete pad north of Building 127 that is still visible.

A.2.1 PPA (IA 1)

During World War II (WWII), the Crane pyrotechnics plant was the largest and best equipped plant of its kind in the United States (U.S.) and supplied both the Atlantic and Pacific fleets with a variety of pyrotechnic devices including illuminating projectiles, parachute flares, and star shells.

In November 1942, the first production item, loading of the Mark 4 aircraft parachute flares, begun at the PPA. In the early part of December 1942, the installation of production machines in the Machine Shop (Buildings 124 and 125) was completed. By February 1943, the assembly line was full operation.

TABLE A.1-1
PPA BUILDING SUMMARY
PYROTECHNIC PRODUCTION AREA
NSA CRANE
CRANE, INDIANA

Building Number / Structure ID	Historical Information	Use / Comments	Potential Contaminants	Building Size (sq ft)
122	5"/54 parachute flares were placed in the projectiles at Building 122. Asbestos lines were cut to length and tied to the parachute rig. Excess asbestos line was discarded in the regular trash because it was non-friable, that is, it did not pose a health hazard. A section of Building 122 was used for the recovery of magnesium and sodium nitrate from the MK45 and MK24 flare candles. During operations in 1974 and 1975, 2,000 gallons of a mixed methylene chloride and methanol was recovered in Building 122. Building 122 also included a paint booth and a polyurethane foam machine for the MK58 marine marker. Prior to 1979, out-of-specification foam, about six 55-gallon drums, was sent to the ammunitions burning grounds annually. Prior to the mid 1970s, paint sludge was sent: to the ordnance burning ground.	Flare Assembly Completed: April 15, 1942	methylene chloride methanol paint (VOCs, metals) asbestos polyurethane foam oxidizing agents (chlorates, perchlorates, peroxides, chromates, and nitrates) fuels (aluminum and magnesium powder, their alloys, sulfur, lactose, and other easily oxidizable materials) polyurethane foam	30,638
123	Use for loading of miscellaneous projectiles such as non-fragmenting, illuminating, and window projectiles contain pellets or charges of black powder for expelling and/or igniting color burst units or other types of loads.	Black Powder Projectile Assembly Building Completed: February 15, 1942		67,519
124	Detailed operational information was not available; however, there is the potential for the use parts washers in this machine shop.	Ordnance Machine Shop (metals fabrication division)	chromium	Included in Building 122
125	Detailed operational information was not available; however, there is the potential for the use parts washers in this machine shop.	Ordnance Machine Shop	chromium	Included in Building 122

Building Number / Structure ID	Historical Information	Use / Comments	Potential Contaminants	Building Size (sq ft)
126	The main operating building for pelleting presses used 200 pounds per day of boron-potassium nitrate for igniters. The building is composed of many separate rooms connected to a main interior corridor. Each room has an exit to the outside. The various compositions are prepared here by drying, mixing, blending, and weighing, and are pressed into "candles," which are the flares or smoke generating ordnance items, at a rate of 1,000 per day. Each of the rooms has a floor drain that allows each room to be flushed with water that drains to a common collector under the building. The collector empties into a large outside sump. In 1972 the sump overflow was connected to the sewage treatment plant. Prior to that time, the sump water drained to surface channels and the solids / sluges were collected about once a year for disposal at the ammunitions burning grounds. Current operations included pumping the contaminated pyrotechnic sludge about every six months for disposal at the ordnance burning grounds. Building 126 also has a dry dust collection system run by a wet vacuum pump that are emptied periodically for solids disposal at the ordnance burning grounds.	Illuminant Building Completed: June 15, 1942	Paints (VOCs, metals); chemicals acetone chromium explosives	37,081
127	Flare testing occurred north of Building 127 on a concrete pad.	Lunch &. Locker / Magnaflux Inspection Former Laboratory	metals	3,938
129	No historical information was found for this building.	Chemical Storage Building	asphaltum (petroleum asphalt and mineral spirits) hexamine hexamethylenetetramine), sugar TNC (tetranitrocarbozole) zinc dust boron	435
130	Inactive (phosphorus screening)	Flare Fuse Assembly Magazine Completed: November 15, 1942	phosphorus	267
131	Contents are unknown	Small Arms Magazine	no information	155
132	Contents are unknown	Small Arms Magazine	no information	157

Building Number / Structure ID	Historical Information	Use / Comments	Potential Contaminants	Building Size (sq ft)
133	Building 133 uses a composition of red phosphorous, manganese dioxide, magnesium, zinc oxide, and linseed oil, which was a wet operation that required wet floors, wet walls, etc. Water from Building 133 flowed to a sump behind the building. Prior to tie-in to the sewer system, sump overflow went to surface water.	Red Phosphorus Pressing House	nitrates hydraulic oil red phosphorus manganese dioxide magnesium zinc oxide linseed oil	2,784
1884	The metal finishing operations include: cleaning, grinding, brazing, solvent degreasing, assembling, testing, machining, welding, soldering, hot dip coating, and calibration. There were chemical storage tanks were contained within earthen dikes.	Plating & Maintenance	dyes cadmium cyanide anodizing tank acid metals	4,800
1885	Building 1885, which was used for phosphorus mixing, were added after 1942.	Phosphorus Mixing Building	Explosives	424
1886	Building 1886, which was used for phosphorus mixing, were added after 1942.	Phosphorus Mixing Building	Explosives WW Effluent tank (east of building)	424
2657	No historical information was found for this building.	Magazine Ready		54
2695	No historical information was found for this building.	Storage Building Chemical	magnesium carbonate magnesium cellulose acetate camphor chlorinated rubber PVC potassium sulfate sodium oxalate shellac yellow dye red dye manganese dioxide zinc oxide lead peroxide	1,200
2696	No historical information was found for this building.	Storage Building Chemical A sump for the red phosphorous buildings is located between the chemical storage, Building 2696, and the ready magazine, Building 135.		1,200
2697	No historical information was found for this building.	Pyro Production "Building 2697 in the pyro area is operated the same as others in the area." (IAS, 1983)		5,125

Building Number / Structure ID	Historical Information	Use / Comments	Potential Contaminants	Building Size (sq ft)
2698	Building 2698 was operated by the Applied Science Department and housed a foam machine and other process equipment. Oxalic acid was used in Building 2698. The TMAE (tetrakis diaminoethylene) is chemilumninescent compound that was used in Building 2698.	Applied Science Facility (Thermal Baters Building)	Solvent VOC paints oxalic acid red phosphorus explosive TMAE (tetrakis diaminoethylene)	5,125
2767	No historical information was found for this building.	Chemical Storage Building ??		196
2768	No historical information was found for this building.	Paint Storage Locker		196
2769	No historical information was found for this building.	Paint Storage Locker		196
2815	No historical information was found for this building.	Cold Storage Warehouse		458
2816	No historical information was found for this building.	Dehumidified Warehouse		642
2822	No historical information was found for this building.	Covered Storage		135
2835	No historical information was found for this building.	Pump House		123
2836	No historical information was found for this building.	General Warehouse Navy		272
2848	No historical information was found for this building.	Pump House		504
2852	No historical information was found for this building.	Paint Storage		144
2854	No historical information was found for this building.	Pump House		504
2855	No historical information was found for this building.	Raw material receiving building		
2856	No historical information was found for this building.	Storage magazine		
2857	No historical information was found for this building.	Drying Building		
2858	No historical information was found for this building.	Drying Building		
2859	No historical information was found for this building.	Screening Building		
2860	No historical information was found for this building.	Dry Magazine Storage Building		
2861	No historical information was found for this building.	Dry Magazine Storage Building		
2862	No historical information was found for this building.	Screening Building		
2863	No historical information was found for this building.	final weighing and blending operation before the actual loading into the ordnance item in Building 136		
2882	No historical information was found for this building.	Caged storage area		
2883	No historical information was found for this building.	West of Building 126		
2884	No historical information was found for this building.	Caged storage area		
2914	No historical information was found for this building.			
2939	Part of Building 125	Potentially used for metals fabrication		
2956	No historical information was found for this building.	Wet Storage Building		152
2957	No historical information was found for this building.	Drying Building		269
2958	No historical information was found for this building.	Drying Building		269

Building Number / Structure ID	Historical Information	Use / Comments	Potential Contaminants	Building Size (sq ft)
3064	Wastewater Treatment Plant	Recovers plating rinse waters from Building 1884		
3148	No historical information was found for this building.			
3296	No historical information was found for this building.	Pyrotechnic component facility		
3297	curing / storage building			
3298	Cold Storage (refrigeration) Curing / storage building		Organic peroxides	

The majority of naval experimental work associated with pyrotechnic devices was conducted at the NSA Crane PPA. To aid in experimental work and production control, a complete laboratory for chemical testing was set up in the pyrotechnics area (Building 127). Three areas are known to have been used for pyrotechnic testing at the Site. Two of these three testing areas are still evident (a concrete pad located north of Building 127 and two concrete basins located west of Building 126). The third flare testing area location, the former Flare Test Area (IA 2) was described as being located north and east of former Building 37.

The concrete pad testing area north of Building 127 is approximately 0.002-acre test area and was used to test pyrotechnics. Additional pyrotechnic testing area occurred in two open square concrete testing basins west of Building 126. These two concrete basins were included in the MRP SI scope (CTO F-272) and will not be addressed in this document. Former Flare Test Area (IA 2) is approximately 7.4-acres north of Highway 5 and east of Building 3395.

The peak PPA production of projectiles and aircraft parachute flares (e.g., pyrotechnic devices) occurred in 1945. Types of pyrotechnic devices produced at the PPA include flares, illuminating projectiles, smoke signals, spotting charges, ground and aircraft signals, and incendiaries. Later developments include chemical delay powders, self-releasing bouyant submarine signals, rescue flares, depth charge markers, aircraft signal cartridges, and parachute flares. During this peak product, 5"/38 Illuminating projectiles, 5"/38 Illuminating projectiles, 5"/25 Illuminating projectiles. 5"/38 Window projectiles, 6"/47 Illuminating projectiles, Mark 6 Mod. 4 Aircraft parachute flares, Mark 6 Mod. 4 Aircraft parachute flares, Mark 8 Mod. I Aircraft parachute flares, and Mark 8 Mod. 2 Aircraft parachute flares were manufactured.

Pyrotechnics devices such as flares typically contain mixtures of an oxidizing agent and a fuel that produces an exothermic self-containing reaction when heated to the ignition temperature. Examples of oxidizing agents include chlorates, perchlorates, peroxides, chromates, and nitrates; examples of fuels include aluminum and magnesium powder, their alloys, sulfur, lactose, and other easily oxidizable materials.

Initially, the PPA production line consisted of Building 122, flare assembly; Building 123, projectile assembly; Buildings 124 and 125, machine shops; and Building 126, the illuminant building.

The buildings in the PPA use sump pits to collect wastewater and washdown water. The water in these sumps were pumped out periodically; however, prior to being connected into the sewer system in the late 1970s, or perhaps as late as the early 1980s, overflow from these sumps flowed onto surface soils and then into surface water drainageways that drain into the Boggs Creek watershed. It is believed that significant heavy metal contamination (zinc and cadmium) has occurred from wastewater overflow from these sumps.

Building 122 was transferred to the Navy from the Army in the 1990's after being renovated to support the 16"/50 caliber projectile rework program that was no longer needed when the Iowa Class Battleships were decommissioned. Building 122 was used for the production of 5"/54 parachute flares. The production process involved placing the projectiles that were tied to the parachute rig into asbestos lines that were cut to the appropriate length. Excess asbestos line was discarded in the regular trash because it was the non-friable type that is not a health hazard. A section of Building 122 was used for the recovery of magnesium and sodium nitrate from the MK45 and MK24 flare candles. The plant operated in 1974 and 1975 and used a mixed methylene chloride and methanol, which was recovered. Building 122 also housed a paint booth and a polyurethane foam machine for the MK58 marine marker. Prior to 1979, the out-of-specification foam was sent to the Ammunition Burning Grounds (ABG). Prior to the mid-1970s, paint sludge was sent: to the ordnance burning ground. Wastewater from Building 122 is still collected in a concrete sump along the north wall of the building (Photograph A-1). The flow from this sump prior to being tie into the sewer system is not known and is considered a potential release to surface soil during overflow conditions.

Photograph A-1
Building 122, Flare Assembly Building Concrete Sump

Building 123 was used for the assembly of black powder projectiles such as non-fragmenting, illuminating, and window projectiles that contained pellets or charges of black powder for expelling and/or igniting color burst units or other types of loads. In this type of black powder operations, the black powder that falls to the floor is typically swept for disposal at the ABG. Little or no process water is associated with this type of operation. Consequently, the potential for contamination from this activity is minimal.

Metal finishing activities were conducted in Buildings 123, 124, and 125 machine shops. These buildings occupy approximately 65,000 square feet of floor space. Complex machining, welding, and heat treating operations were performed in support of PPA production and tooling requirements. Wastewaters from the machine shops consist of cooling waters (from welding and heat treating operations), deburring machine waste, and water-soluble machining coolants. In 1984, wastewater generated from machine shop operations was discharged to the sanitary sewer. Prior to 1984, where the wastewater discharged from the machine shop operations is not known.

Because Buildings 124 and 125 were placed into service in 1945, it is likely that petroleum-based solvents were used for parts washing and that the wastewater associated with these buildings may have contained metals such as arsenic, barium, cadmium, chromium, lead, mercury, selenium, or silver and volatile organic compounds (VOCs) such as benzene, carbon tetrachloride, chlorobenzene, chloroform, 1,2 dichloroethane, 1,1-dichloroethylene, methyl ethyl ketone, tetrachloroethylene, trichloroethylene, or vinyl chloride.

Building 126 is the main operating building where pelleting presses used boron-potassium nitrate for igniters. Building 126 is composed of many separate rooms connected to a main interior corridor. Each room has an exit door to the outside (See Photograph A-4).

Photograph A-4
Building 126 Individual Room Exit Doors to Outside

The various compositions are prepared here by drying, mixing, blending, and weighing, and are pressed into "candles," which are the flares or smoke generating ordnance items. Each room in Building 126 has a drain that allows for the room to be flushed with water that flows into a common collector under the building. The collector empties into a large sump (126 Pit A) outside (See Photograph A-5).

Photograph A-5
Building 126 Pit A

In 1972 the Building 126 Pit A overflow was connected to the sewage treatment plant. Prior to 1972, the sump water drained to surface channels and the solids / sludges were collected approximately once per year for disposal at the ABG. Current Building 126 Pit A sump operations included pumping the contaminated pyrotechnic sludge approximately twice per year, every six months, for disposal at the ordnance burning grounds. Building 126 also has a dry dust collection system run by a wet vacuum

pump (see Photograph A-6). These collectors are emptied periodically, and the solids are taken to the ordnance burning grounds.

Photograph A-6
Exhaust vents on the South Side of Building 126

Building 133, the Red Phosphorus Pressing House, is used for red phosphorus candle pressing. Industrial presses are used in the manufacturing process. Other materials used in Building 133 include manganese dioxide, magnesium, zinc oxide, and linseed oil. This is a wet operation, requiring wet floors, wet walls, etc. Wastewater from Building 133 is collected in a sump located behind the building (133 Pit A) (see Photographs 10-2 and 10-3). The sludges and solids are removed from this basin and sent to the ABG for processing.

Photograph A-2
Building 133 Pit A Sump and Covered Concrete Trench

Photograph A-3
Building 133 Pit A Sump Interior

Building 2698 in the pyro area is operated by the Applied Science Department. Building 2698 houses a foam machine and other process equipment. Oxalic acid and TMAE (tetrakis diaminoethylene), a chemluminescent compound, are used in Building 2698.

A series of buildings formerly located in the general area of Building 3148 was used for candle drying / curing. Candle curing and storage is currently conducted in Buildings 3297 and 3298. No know releases are associated with these curing / drying activities.

Various buildings and magazines located throughout the PPA were or are currently being used for chemical storage. These buildings and magazines are listed in Table 10-1. No know releases occurred from these buildings and magazines were documented during the September site visit. However, should a release have occurred, it is anticipated that these releases would have been to surface soil and flow through surface water drainageways towards Boggs Creek.

Building 1884, the plating shop, utilized acid and alkaline cleaning, chrome conversion coating, phosphate coating, and cadmium/zinc cyanide plating on a variety of munitions-related metal parts in support of the PPA industrial machine shop. A schematic of metal finishing operations, believed to have been used, is presented in Figure A-1.

The main source of Building 1884 wastewater was the continuous rinsing that follows the acid/alkaline cleaning, chrome/phosphate coating, and cadmium/zinc cyanide plating operations. Spent concentrated process plating solutions were disposed using a hazardous waste contractor.

Wastewaters from Building 1884 are segregated into three independent collection systems (acid/alkali, chromium and cyanide collection systems) before being pumped to the plating shop wastewater pretreatment facility (PSWPF) in Building 3064. Wastewater treatment in Building 3064 consists of neutralization and treatment to destroy cyanide and removal of solids. Building 3064, the PSWPF, began

operations in September 1977. The Metal Plating Shop discharge (Discharge Point 005) is permitted under NSA Crane National Pollution Discharge Elimination System (NPDES) Permit No. IN-0035157. Prior to start up of the PSWPF, historical wastewater discharges from the Metal Plating Shop indicated periodic failure to fully comply with discharge limits for pH, Total Suspended Solids (TSS), chromium, copper, cyanide, iron, lead, and zinc.

Cadmium and zinc cyanide wastewaters were collected in an underground sump located outside of Bldg 1884. Wastewater was pumped from the sump to a cyanide destruction process (two-stage alkaline chlorination) before mixing with other waste for subsequent heavy metals removal.

Buildings 1885 and 1886 (See Photograph A-4) was used phosphorus mixing. These buildings are no longer in service and have been emptied. Miscellaneous empty containers are stored southwest external of Building 1886. These containers have magnesium powder labels. Water from Buildings 1885 and 1886 flowed into two sump pits (1885 Pit A and 1886 Pit B) (see Photograph A-5). A suspected floor drain in Building 1885 mixing room appears to drain into a suspected sump south of the building (see Photographs 10-6 and 10-7). Surface waters from Building 122 parking area and the grassy areas surrounding Buildings 1885 and 1886 appear to flow into 1886 Pit A (see Photographs 10-8 and 10-9). The condensate collection system southwest of Building 122 overflows into a surface water drainageways that flows into 1886 Pit A. Overflow from these pits traveled over the ground surface to a stormwater drainageways that discharges from the south-central portion of the PPA (see Photograph A-10).

Photograph A-4
Buildings 1884 and 1885 Exterior

Photograph A-5
1884 Pit A and 1885 Pit A

Photograph A-6
Suspected Building 1885 Floor Drain

Photograph A-7
Suspected Building 1885 Sump

Photograph A-8
Surface Water Flow From Building 122 Parking Area into 1886 Pit A

Photograph A-9
Surface Water Flow into 1886 Pit A

Photograph A-10
Condensate Collection System Southwest of Building 122

Another sump for the Red Phosphorous Buildings (Building 130) is located between the chemical storage: Building 2696, and the ready magazine, Building 135 (See Photograph A-11).

Photograph A-11
Building 130 Sump between Buildings 135 and 2696

The nearest streams are to the west and drain into the Boggs Creek Watershed. Turkey Creek is east of the PPA, but drain swales and local topography convey surface flow in the PPA to the south and west (near the point where the railroad track R-1 crosses Highway 99).

During the September 16 through 18, 2009 site walk, two transformers with labels indicating a PCB containing oil were observed (see Photographs 10-12 through 10-15). A third former transformer believed to have contained PCB transformers was located in the current fenced area across the road west of Building 133 (see Photograph A-16).

Photograph A-12
Transformer West of Building 124

Photograph A-13
Transformer West of Building 124 Label

Photograph A-14
Transformer North of Building 126

Photograph A-15
Transformer North of Building 126 Label

Photograph A-16
Former Electrical Substation West of 133 Pit A

A.2.2 Former Flare Test Are (IA 2)

IA 2 covers approximately 7.4-acres and has an open access area within NSA Crane.

Little documentation was available regarding the exact location and operation of IA 2. The location of IA 2 has been gleamed from historical photographs and the September 16-18, 2009 NSA Crane site visit during personnel interviews.

A.3.1 PREVIOUS ENVIRONMENTAL INVESTIGATIONS AND ACTIONS

Previous environmental inspections and investigations conducted at the site include a Preliminary Review/Visual Site Inspection in 1987 (PA, 1987) and the Initial Assessment Study in 1983 (IAS, 1983).

A.2.2 Preliminary Review/Visual Site Inspection Report

In March 1987, A.T. Kearny issued a Preliminary Review/Visual Site Inspection Report that identified three areas within IA 1 [Units 22 (Building 126 Sump – SWMU 22), 24 (Scrap Storage Area Behind Building 126), and 26 (Building 133 Sump)].

IA 1 (PPA)

Unit 22 (Building 126 Sump)
Unit 22 was described as a below-grade concrete sump that receives rinse waters from Building 126 production rooms via individual drains and a central connector. The rinse water is generated from the washing of grit screens and empty paint barrels. Visual observations included cracks in the tank and deterioration of the retaining wall. The sump rinse waters contain phosphorous, manganese, and paint residues and discharged directly to the sanitary sewer after solids settle to the bottom. Solids are removed and taken to the Unit 46 [CSF – Cast High Explosives Fill (Building 146)].
Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is moderate due to the relatively poor visual appearance of the sump.

The report concluded that there are low release potentials for surface water due to the sanitary sewer system discharge, air to due to the dilute nature of the wastes; and subsurface gas due to the nature of the wastes managed and the open design of the unit (A.T. Kearny, 1987).

The report recommendations included taking soil samples near the sump walls to determine if any contamination has occurred due to the cracks in the sump walls.

Unit 24 (Scrap Storage Area Behind Building 126)
Unit 24 was described as a concrete pad with a metal canopy and no perimeter containment that was used for temporary storage of waste paint cans, starter mix, fuel oil, and explosive contaminated metal parts. Visual observations indicated that the wastes contained were in small containers or jugs.

The report concluded that are low release potentials for surface water, air, and subsurface gas due to the containerization of wastes and the unit's distance from any local surface drainage.

The report recommendations no further action (NFA) for this unit.

Unit 26 (Building 133 Sump)
Unit 24 was described as this sump as receiving wastewater from the red phosphorous candle pressing house that uses red phosphorous, manganese dioxide, magnesium, zinc oxide, and linseed oil within a totally wet operation. The sump consists of three separate concrete chambers that are approximately 6 ft. (long) x 2 ft. (wide) x 3 ft (deep).

Rinsate water contaminated with red phosphorous is allowed to settle in the sump prior to being pumped the Sanitary Sewer System. Sludge is accumulated and then transported to the Sludge Dewatering Units at the Ammunitions Burning Grounds.

The report concluded that there are low release potentials for soil/groundwater due to apparent good condition of the concrete sump, surface water because solids in the rinse water are allowed to settle before the water phase is discharged to the sanitary sewer system discharge, air to due to the nature of the wastes; and subsurface gas due to the open design of the unit and nature of the wastes handled.

The report recommendations NFA for this unit.

IA 2 (Former Flare Test Area)

A.2.2 PREVIOUS ENVIRONMENTAL INVESTIGATIONS AND ACTIONS

IA 2 is not addressed in the Preliminary Review/Visual Site Inspection report.

A.2.2 Initial Assessment Study

IA 1 (PPA)
Buildings 1885 and 1886 were used for phosphorus mixing.

In Building 126, the main operating building, pelleting presses used boron-potassium nitrate for igniters. Building 126 is composed of many separate rooms connected to a main interior corridor. Each room has an exit to the outside and each has a drain for water flushing that flows to a common collector under the building. The collector empties into a large sump outside the building.

In 1972 the Building 126 sump overflow was connected to the sewage treatment plant. Prior to that time, the sump was drained about once a year, and the contents were taken to the burning grounds. Contaminated pyrotechnic sludge is pumped about every six months and taken to the ordnance burning grounds. Building 126 also has a dry dust collection system run by a wet vacuum pump. These collectors are emptied periodically, and the solids are taken to the ordnance burning grounds. Another sump for the red phosphorous buildings is located between the chemical storage (Building 2696) and the ready magazine (Building 135).

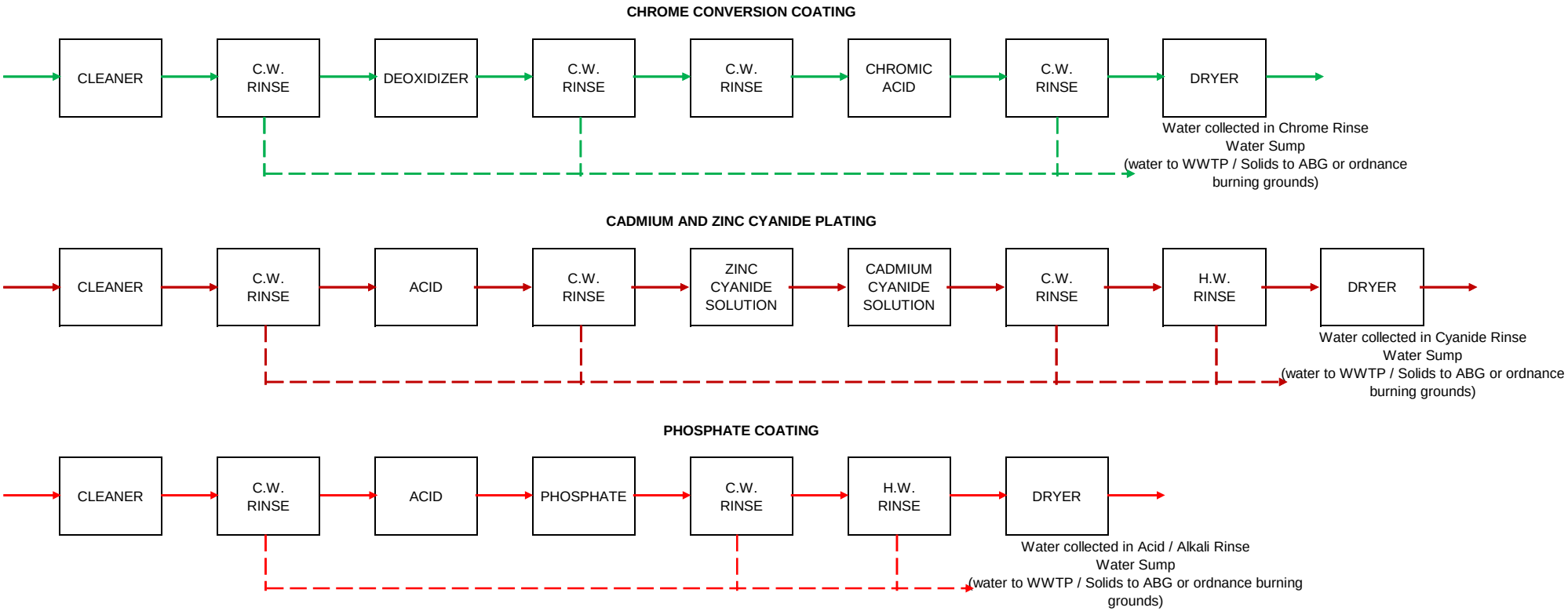
One section of Building 122 (the Flare Assembly Building) is used for the recovery of magnesium and sodium nitrate from the MK45 and MK24 flare candles. Building 122 housed a paint booth and a polyurethane foam machine for the MK58 marine marker. Prior to the mid-1970s, paint sludge from Building 122 was sent to the ordnance burning ground.

Building 133, the phosphorus pressing house, uses a composition of red phosphorous, manganese dioxide, magnesium, zinc oxide, and linseed oil in a wet operation that requires wet floors, wet walls, etc. The wastewater from this operation was sent to a sump behind Building 133.

Building 2698 was operated by the Applied Science Department housed a foam machine and other process equipment that used oxalic acid and TMAE (tetrakis diaminoethylene), a chemilumninescent compound.

Prior to 1981, Building 136 wastewaters from leads azide and lead styphinate operations were rendered insensitive, poured into a pond, and periodically pumped to an unknown location. The pond solid material was taken to the burning grounds for disposal. In 1981, after the liquid and sediment in the pond was analyzed, the contaminated pond material was removed and hauled off site to an approved landfill site by a state certified waste hauler. Since 1981, the Building 136 wastewaters have been treated in the WWTP (Building 3064).

FIGURE A-1
BUILDING 1884 (PLATING SHOP) METAL FINISHING OPERATION PROCESS FLOW DIAGRAM
SWMU 27 - ILLUMINANT BUILDING 126
NSA CRANE
CRANE, INDIANA



PROCESS FLOW
WASTEWATER FLOW
C.W. - COLD WATER
H.W. - HOT WATER
WWTP - Wastewater Treatment Plant (Building 3064)

Source: Figure E-1 US Army Water Quality Engineering Study No. 32-24-0575-85

A.2

SEPTEMBER 2009 SITE VISIT INFORMATION

DISPOSITION FORM

For use of this form, see AR 340-15; the proponent agency is TAGO.

REFERENCE OR OFFICE SYMBOL	SUBJECT
SMCCN-IOE (200-1a)	Grinding Ferric Acetylacetonate (FeAA)

TO SMCCN-IOA FROM SMCCN-IOE DATE 14 April 1988 CMT1
Mr. Burcham/cs/3660

1. Request the supervisor of building 126 use the Micro-Pulverizer from NWSC-50 to pulverize 25 pounds of FeAA. The finished material will be used during testing, at building 200, of PBXN-106 mixing and loading operations.

2. During development of the grinding process, request all material ground be ROTAP'd with the following stack of sieves, 100, 200 and 325 mesh. Only material passing through the 325 mesh sieve is acceptable. Acceptable material will be containerized at 100 grams per container. Mark container with date, name of contents, stock number, weight of material and the sieve size the material passed through.

3. Equipment and tooling cleaning can be accomplished with water. Proper personnel protective equipment must be worn when working with FeAA. A copy of the material safety data sheet is provided as enclosure 1. Personnel working with this project will be charged to 701PBX. This work should be accomplished prior to 9 May 1988.

4. The material is located in building 334 in 100 pound drums. The stock number is 6810-00-X85-2390.

5. The point of contact for this project is Randall Burcham, SMCCN-IOE, extension 3660.

D. C. Rumble
D. C. RUMBLE
Chief, Engineering Division

Encl
as

CF:
NWSC-04
NWSC-12C
NWSC-0924

0924 SMH
0924 PAK

U.S. DEPARTMENT OF LABOR
Occupational Safety and Health Administration

Form Approved
OMB No. 44-R1387

MATERIAL SAFETY DATA SHEET

Required under USDL Safety and Health Regulations for Ship Repairing, Shipbuilding, and Shipbreaking (29 CFR 1915, 1916, 1917)

Reviewed July 1982

H 2
F 4
R 0

PPE - X

Style NC-L503X

SECTION I

MANUFACTURER'S NAME: The Shepherd Chemical Company	EMERGENCY TELEPHONE NO.: 513-731-1110
ADDRESS (Number, Street, City, State, and ZIP Code): 4900 Beech Street, Cincinnati, Ohio 45212	
CHEMICAL NAME AND SYNONYMS: Ferric Acetylacetonate Fe.AA	TRADE NAME AND SYNONYMS: Ferric Acetylacetonate
CHEMICAL FAMILY: metallic salt of 2,4 pentanedione	FORMULA: $Fe(C_5H_7O_2)_3$ C.A.S. 14024-18-1

SECTION II - HAZARDOUS INGREDIENTS

PAINTS, PRESERVATIVES, & SOLVENTS	%	TLV (Units)	ALLOYS AND METALLIC COATINGS	%	TLV (Units)
PIGMENTS			BASE METAL		
CATALYST			ALLOYS		
VEHICLE			METALLIC COATINGS		
SOLVENTS			FILLER METAL PLUS COATING OR CORE FLUX		
ADDITIVES			OTHERS		
OTHERS					

HAZARDOUS MIXTURES OF OTHER LIQUIDS, SOLIDS, OR GASES	%	TLV (Units)

SECTION III - PHYSICAL DATA

BOILING POINT (°F.)	400-450	SPECIFIC GRAVITY (H_2O=1)	1.42
VAPOR PRESSURE (mm Hg.)	N.A.	PERCENT, VOLATILE BY VOLUME (%)	N.A.
VAPOR DENSITY (AIR=1)	N.A.	EVAPORATION RATE (_____ =1)	N.A.
SOLUBILITY IN WATER	moderate		

APPEARANCE AND ODOR: bright red crystalline powder, slight 2,4 pentanedione odor

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (Method used): dispersed dust will ignite	FLAMMABLE LIMITS: N.A.	Lel	Uel

EXTINGUISHING MEDIA: foam CO_2 Dry Chemical

SPECIAL FIRE FIGHTING PROCEDURES

UNUSUAL FIRE AND EXPLOSION HAZARDS: Moderate when exposed to extreme heat and flame.

(Continued on reverse side)

Form OSHA-20
Rev. May 72

ENCL 1

SECTION V - HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE
not established orl LD 50 rat 2.14 gm/kg

EFFECTS OF OVEREXPOSURE
Dust is definite eye hazard. Contact with eyes may produce iritis, conjunctivitis, and ulceration of eyeball.

EMERGENCY AND FIRST AID PROCEDURES
Eyes - immediately rinse with copious amounts of water. Consult physician immediately. Skin - wash with soap and water. Ingestion - give water. Induce vomiting. Consult physician.

SECTION VI - REACTIVITY DATA

STABILITY			CONDITIONS TO AVOID
	UNSTABLE		
	STABLE	X	

INCOMPATABILITY *(Materials to avoid)*
strong oxidizing agents

HAZARDOUS DECOMPOSITION PRODUCTS
Thermal decomposition may produce CO.

HAZARDOUS POLYMERIZATION			CONDITIONS TO AVOID
	MAY OCCUR		
	WILL NOT OCCUR	X	

SECTION VII - SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED
Sweep or shovel to recover if not contaminated. Flush into waste treatment unit with water.

WASTE DISPOSAL METHOD
Dispose as water soluble product in accordance with local procedures and regulations.

SECTION VIII - SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION *(Specify type)*
NIOSH/MESA approved such as TC-21C or TC-23C

VENTILATION	LOCAL EXHAUST	SPECIAL
	yes - if dusting procedures used.	
	MECHANICAL *(General)*	OTHER

PROTECTIVE GLOVES	EYE PROTECTION
yes	yes - monogoggles

OTHER PROTECTIVE EQUIPMENT
eyewash station

SECTION IX - SPECIAL PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING
Store in closed fiber containers under normal conditions.

OTHER PRECAUTIONS
Good housekeeping procedures should be followed to avoid generation of dust during processing.

CC 7/2/86

MEMORANDUM TO FILE

FILE # B-126 Facility

SUBJECT: Chemical Analysis Large Sump B-126 - Empty Large Sump

ORIGINATOR: Jim Hunsicker

DATE OF RECORD: 6/30/86

Gave Jim Norris go ahead to drain sump + residue off of bottom into sanitary sewer. This decision was based on the attached analytical results.

Jim's Copy

ANALYTICAL REPORT

ENVIRONMENTAL RESEARCH GROUP, INC.

117 N. FIRST
ANN ARBOR, MICHIGAN 48104 (313) 662-3104

Project: A70-5.2A
Report Date: 06-19-86

Client P.O. N62472-85-C-7073
Report: 19579

Samples Recvd: 05-15-86
Refer Questions To:
LINDA DUNLAP

Client:
OFFICER IN CHARGE OF CONSTRUCTION
CRANE N.W.S.C.
CODE 09246
BUILDING 2516
CRANE, INDIANA 47522
Attention: JO BELCHER

Approved: Art Gabenuk

***
Residual Samples Will Be Held
TWO WEEKS
***

Client I.D.: B126-05136
ERG Sample No.: 05/152089
Matrix: SLUDGE
Date Sampled: 05-13-86

Parameter	Result	Units
EP-LEACHATE	DONE	

Client I.D.: LEACHATE OF B126-05136
ERG Sample No.: 05/152090
Matrix: EP-TOX LEACHATE

Parameter	Result	Units
ARSENIC, TOTAL	0.003	mg/L
AVERAGE OF DUPLICATE RUNS		
BARIUM, TOTAL	16	mg/L
CADMIUM, TOTAL	0.03	mg/L
CHROMIUM, TOTAL	0.04	mg/L
LEAD, TOTAL	0.39	mg/L
MERCURY	0.0005	mg/L
SELENIUM, TOTAL	<0.01	mg/L
AVERAGE OF DUPLICATE RUNS HIGHER DETECTION LIMIT DUE TO MATRIX INTERFERENCE.		
SILVER, TOTAL	0.008	mg/L

Client I.D.: T2801-05136
ERG Sample No.: 05/152091
Matrix: OIL
Date Sampled: 05-13-86

Parameter	Result	Units
EP-LEACHATE	DONE	
PCB		
PCB, TOTAL	1.3	mg/Kg
PCB 1242	ND (0.50)	mg/Kg
PCB 1248	ND (0.50)	mg/Kg
PCB 1254	ND (0.50)	mg/Kg
PCB 1260	1.3	mg/Kg

 See last page for explanation of symbols.

CCA 5/5/86

MEMORANDUM TO FILE

FILE # B-126 Facility File

SUBJECT: Sump - Analytical Results - large outside sump needs drained + cleaned

ORIGINATOR: Jim Hensicker 09245

DATE OF RECORD: 5/5/86

==========

After reviewing the attached results I informed Jim Norris that we couldn't drain + clean sump until we had collected new samples + had new results in hand.

The analytical results on the sludge sample raises some questions regarding what's going into sump and possible validity of results. I questioned Jim some on where we would be picking up lead, Cd, Cr + Ba; because not a part of formulations found in some units they produce.

Jim also mentioned the laboratory that was built/added on back of B-126 this past year. They could possibly be dumping things into sump since they are connected to it.

Told Jim we would collect new samples and as soon as results available I would get back with. I indicated to Jim follow-up would be forthcoming to find out what's being done in lab process control lab at B-126.

MEMORANDUM TO FILE

09246 JB
09245 JMH
file

FILE # B 126

SUBJECT: Samples from sump at B126

ORIGINATOR: J. Belcher

DATE OF RECORD: 5-5-86

These are the results I received over the phone from Louis Swank, B2707, which I verified with Bill Brand.

results in mg/l	Pb	Cd	Cr	Ba
B126-2-04036 water sample	.35	<.05	<.05	.48
B126-1-04036 sludge	400	10	90	530

CCA

MEMORANDUM TO FILE

FILE # Building 126 Facility File

SUBJECT: Dumping large outside sump (19,200 gal.) which collects wastewater from clean-up of mix rooms etc.

ORIGINATOR: Jim Hunsicker Code 09245

DATE OF RECORD: 12/6/85

==========

On 12/9/85 plan to give Jim Norris [redacted] go ahead to drain large sump/holding tank into Sanitary Sewer System. This decision was based on analysis we had run on sampled water from the sump (see attachment) and having Phil Keith review to be sure none of the levels of metals found exceeded or would cause us to exceeded NPDES standards/limitations set for STP outfall.

12/9/85

Gave Jim Norris go ahead to ~~pump~~ drain pit into sewer –

Project: A70-10.5A
Report Date: 11-27-85

Client I.D.: P126-10295 SUMP
ERG Sample No.: 11/140185
Matrix: IND. EFFLUENT
Date Sampled: 10-29-85

Parameter	Result	Units
ARSENIC, TOTAL	<0.006	mg/L
HIGHER DETECTION LIMIT DUE TO MATRIX INTERFERENCE.		
BARIUM, TOTAL	0.48	mg/L
CADMIUM, TOTAL	<0.01	mg/L
CHROMIUM, TOTAL	0.04	mg/L
LEAD, TOTAL	<0.05	mg/L
MERCURY	0.0004	mg/L
PHOSPHOROUS, TOTAL	0.07	mg/L
SELENIUM, TOTAL	ND (0.005)	mg/L
SILVER, TOTAL	ND (0.02)	mg/L

Client I.D.: H34-10305
ERG Sample No.: 11/140186
Matrix: IND. EFFLUENT

Parameter	Result	Units
PCB		
PCB, TOTAL	ND (0.50)	mg/Kg
PCB 1242	ND (0.50)	mg/Kg
PCB 1248	ND (0.50)	mg/Kg
PCB 1254	ND (0.50)	mg/Kg
PCB 1260	ND (0.50)	mg/Kg

Client I.D.: ABG-8-10025
ERG Sample No.: 11/140187
Matrix: SEDIMENT
Date Sampled: 10-02-85

Parameter	Result	Units
EP-LEACHATE Red phosphorus Ash	DONE	

Client I.D.: LEACHATE OF ABG-8-10025
ERG Sample No.: 11/140188
Matrix: EP-TOX LEACHATE
Date Sampled: 10-02-85

Parameter	Result	Units
ARSENIC, TOTAL	0.008	mg/L
BARIUM, TOTAL	ND (0.5)	mg/L
HIGHER DETECTION LIMIT DUE TO MATRIX INTERFERENCE.		
CADMIUM, TOTAL	0.12	mg/L
CHROMIUM, TOTAL	0.04	mg/L
LEAD, TOTAL	<0.05	mg/L

MEMO from Jim H.

TO B-126 file DATE 12/1/83 TIME

SUBJECT Analysis of wastewater from B-126 sump

After discussing analysis with Cathy gave Bill Hubble the go ahead to dump the sump at B-126 into the sanitary sewer.

If possible sump should be emptied prior to cleaning up B-126, this way new wastewater could be collected - suspended solids allowed to settle and new analysis done.

Jim Hunsicker

☐ REPLY ☐ INITIAL AND RETURN ☐ SEE ME

Form MGL © The Drawing Board Inc., Box 505 Dallas, Texas

Sample B126S-13103

EP Tox Metals

Mg/L

Arsenic	less than	0.005
Barium	"	4
Selenium	"	0.0009
Silver	"	0.004
Mercury		0.0009
Cadmium		0.006
Lead		0.42
Chromium		0.015

Magnesium		6.4
Aluminum		2.0
Zinc		1.0
Nitrates		0.08
Nitrites	less than	0.01
TOC		19
Total Chloride		6
Copper		0.60
TOH	organic chlorine	0.01

Bromine	ND	0.002
Iodine		

Reactivity		Sulfide		Cyanide
	less than	0.010	less than	0.010
PH	9.3			

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Rags, Gloves, with Phosph. & Spent Scrap of Phosph. in Fuel Oil	Metal	Fuel Oil	126/Pyro/IDA	8 lb.
Electric Squib w/ Starter Strip in Water	Metal	Water	126/Pyro/IDA	8 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
[signature]	3026	IDC	4/26/2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
[signature]	DOD-B		4/26/2000
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**		**DATE**
[signature]	ABC		4-26-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
4FF. in acetone	Metal	Acetone	126/Pyro/~~IDA~~	40 lb
Alum. Comp in Acetone	Metal	Acetone		40 lb
Alum. Comp in fuel Oil		Fuel Oil		25 lb
Scrap Binder, Prop Strips Hardwy in water		Water		15 lb
Sodium Nitr in Fuel Oil		Fuel Oil		20 lb
PBXN 106, HBX Acetone & Toluene	Metal	Acetone & Toluene	126/Pyro/~~IDA~~	2 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
Melvin Jackson	X3026	IDC	4/25/00
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
Jim Rouck	DOD-B		4/25/2000
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**		**DATE**
[illegible]	ABG		4-25-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Reject 60mm	METAL	Reject Candles	126/RRO/IOA	603 ea. (1 pal.)
ILLUM CANISTER				
ASSY. F/TEST.				
NOTE TEST BATCHES:				
ODC-B00005				
ODC-B00006				
ODC-B00007				

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
[illegible]	X3026	IOC	4/13/2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
MB Clark	CAAA 10A		04/13/00
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**		**DATE**
Bobby Talbert	ABG		4-13-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Binder Containers in Water	Metal	Water	126/Pyro/IDA	25 lb
IR Comp in Fuel Oil	Metal	Fuel Oil		(3) @ 60 lb ea
TNC Contaminated Rags, Gloves + Plastic Bags in Fuel Oil	Metal	Fuel Oil		25 lb
Pyro Contaminated Filters in Fuel Oil	Metal	Fuel Oil		50 lb
235 Starter Mix in Fuel Oil	Metal	Fuel Oil		15 lb
Oil Dry Contaminated w/ Oil	Metal	Oil		2 @ 20 lb ea.
AAA Ignition Mix in Acetone	Metal	Acetone	126/Pyro/IDA	2 @ 10 lb ea.

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
Melanie [illegible]	X3026	IDC	4/12/2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
Cletus [illegible]	ABG		4/13/00
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**		**DATE**
Cletus [illegible]	ABG		4-13-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCG 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Yellow First Fire in Fuel Oil	Metal	Fuel Oil	126/IOA	3@ 45 lb ea.
IR Comp in Fuel Oil	Metal	Fuel Oil	126/IOA	2@ 50 lb ea
Yellow First Fire in Fuel Oil	Metal	Fuel Oil	126/IOA	1@ 35 lb
Contaminated Rags Filters + Gloves in Fuel Oil	Metal	Fuel Oil	126/IOA	50 lb
Reject 60 mm IR Candles	Metal Ammo Box	IR Comp	126/IOA	3 ea.
Potassium Nitrate in Fuel Oil	Metal	Fuel Oil	126/IOA	7@ 25 lb ea.
68 Starter Comp in Fuel Oil	Metal	Fuel Oil	126/IOA	3@ 45 lb ea.
Potassium Nitrate in Fuel Oil	Metal	Fuel Oil	126/IOA	5@ 25 lb ea.
Mag. Powder in Fuel Oil	Metal	Fuel Oil	126/IOA	15 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
Melanie D. Durkee	X3026	IOC	4/5/2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
[illegible]	DOD-B		4/6/2000
SIGNATURE (Operator of Treatment Facility)	**TREATMENT FACILITY**		**DATE**
[illegible]	ABG		4-6-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
IR Comp in Fuel Oil	Metal	Fuel Oil	B126/IDA	65 lb
IR Comp in Acetone		Acetone		50 lb
Vapor Ignition Mix in Acetone		Acetone		2@ 20 lb ea.
PBXN 106 Acetone Binder in Fuel Oil	Metal	Acetone & Fuel Oil	B126/IDA	7 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)	CODE	DATE
[signature] 3026	IDC	3/30/2000

SIGNATURE (Transporter)	ORGANIZATION	DATE
[signature]	DOD-B	3/30/2000

SIGNATURE (Operator at Treatment Facility)	TREATMENT FACILITY	DATE
[signature]	ABG	3-30-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Reconditioned Oil	Metal	Fuel Oil	126/IDA	15 lb
Rags, Oil Dry & Brushes Contaminated w/ VAAR	Metal	VAAR (Vinyl Alcohol Acetate Resin)		25 lb
Scrap Illum Comp in Acetone		Acetone		40 lb
Illum Comp in Fuel Oil		Fuel Oil		40 lb
Contaminated Rags, Filters & Brushes in Fuel Oil		Fuel Oil		58 lb
Binder, Rags & Sticks Cont. & Cups in Water		Binder / Water		60 lb
Yellow First Fire in Fuel Oil	Metal	Fuel Oil	126/IDA	180 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
[signature]	3026	ZOC	3/21/2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
[signature]	DOD-B		3/21/2000
SIGNATURE (Operator of Treatment Facility)	**TREATMENT FACILITY**		**DATE**
[signature]	ABC		3-22-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
VAAR Ignition Mix in Acetone	Metal	Acetone	B-126/IDA	20 lb
Epoxy Cups + Paper in Water	Metal	Water/Epoxy		15 lb
IR Comp in Acetone	Metal	Acetone		50 lb
IR Comp in Fuel Oil	Metal	Fuel Oil	B126/IDA	50 lb
T-50 Catalyst F-100 Resin Epoxy in cup w/water	Metal	Water	B-126/IDA	8 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE *(Generator or Facility Supervisor-Extension No.)*		CODE	DATE
[signature]	3026	IDC	3/14/2000
SIGNATURE *(Transporter)*	**ORGANIZATION**		**DATE**
[signature]	DOD-B		3/14/2000
SIGNATURE *(Operator at Treatment Facility)*	**TREATMENT FACILITY**		**DATE**
[signature]	ABG		3-14-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
60, 81 ψ /20mm Yellow Comp in Acetone	Metal	Acetone	B/126/IDA	40 lb
Yellow Comp in Fuel Oil	Metal	Fuel Oil		20 lb
(2) Reject 60mm Yellow.	Metal	Yellow Comp.	B-126/IDA	4 lb.
Yellow 1st fire in Acetone	Metal	Acetone	B-126/IDA	65 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
Melvin Dave Dexter	X3026	IOC	3/9/2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
Jim Roark	DOD-B		3/9/00
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**		**DATE**
[illegible]	ASC		3-9-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Eng. Mixer in Fuel Oil	Metal	Fuel Oil	IOA/126	25 lb
Alum. Comp in fuel Oil	Metal	Fuel Oil	IOA/126	50 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
[signature]	X3026	IOC	3/6/2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
[signature]	DOP-B		3/6/2000
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**		**DATE**
[signature]	ABG		3-6-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Zinc Dust in Fuel Oil	Metal	Fuel Oil	126/IOA	8@50 lbs ea.
Hexane	Metal	Hexane		25 lbs
Zinc Dust con w/ Hole in fuel Oil	Metal	Fuel Oil		50 lbs
Zyco Cont Resp Filter in fuel Oil	Metal	Fuel Oil		40 lbs
Magnesium Scrap in fuel Oil	Metal	Fuel Oil		20 lbs
PAR Ignition Wire in Ethyl Alcohol	Metal	Ethyl Alcohol	126/IOA	2@8 lbs ea.

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE *(Generator or Facility Supervisor-Extension No.)*		CODE	DATE
[signature]	X 3026	IOC	2-14-2000
SIGNATURE *(Transporter)*	**ORGANIZATION**		**DATE**
[signature]	DOD-B		2-14-2000
SIGNATURE *(Operator at Treatment Facility)*	**TREATMENT FACILITY**		**DATE**
[signature]	ABG		2-14-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Ignition Mix in Fuel Oil	Metal	Fuel Oil	B126/IOA	25 lb
Scrap Binder Nitrocellulose + acetone	Metal	Acetone		
Scrap IR Mag Teflon in Acetone	Metal	Acetone		
MK13 Fire + Fire in Fuel Oil	Metal	Fuel Oil		
Hexane	Metal	Hexane	B126/IOA	25 lb
Zirconium Wire in Fuel Oil	Metal	Fuel Oil	126/IOA	4 @ 50 lb ea

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)	CODE	DATE
[illegible] x3026	IOC	2/9/2000
SIGNATURE (Transporter)	**ORGANIZATION**	**DATE**
[illegible]	DOD-B	2/9/2000
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**	**DATE**
[illegible]	ABG	2-10-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 0280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Cesium Nitrate in Fuel Oil	Metal	Fuel Oil	B-126/IOA	30 lb
White Smoke Scrap in Fuel Oil	Metal	Fuel Oil		40 lb
Pyro Cont. Rags, Filters etc. in Fuel Oil	Metal	Fuel Oil	B-126/IOA	45 lb
White Smoke Scrap in fuel Oil	Metal	Fuel Oil	B126/IOA	100 lb
Potassium Nitrate in fuel Oil	Metal	Fuel Oil	B126/IOA	30 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
Melanie D. Wilkerson	X3026	IOC	2/8/2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
Jim Le Roach	DOD-B		2/8/2000
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**		**DATE**
Clettus Fudgit	ABG		2-10-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION*	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Pyro Cont. Rags & Filters in fuel Oil	Metal	fuel Oil	126/IOA	2 @ 50 lb ea
			126/IOA	1 @ 8 lb ea.
Manganese Dioxide Rags in fuel Oil	Metal	fuel Oil		

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
[signature]	X3026	IOC	2/1/2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
[signature]	DOD-B		2/1/2000
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**		**DATE**
[signature]	ABG		2-2-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION*	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Pyro Cont. Rags Filters, Gloves etc TNG Fuel Oil	Metal	Fuel Oil	126/IDA	45 lbs
Pyro Cont. Rags Filters, Gloves etc TNG Fuel Oil	Metal	Fuel Oil	126/IDA	35 lbs

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE *(Generator or Facility Supervisor-Extension No.)*		CODE	DATE
Melanie Henderson	X3026	IOC	1/24/2000
SIGNATURE *(Transporter)*	**ORGANIZATION**		**DATE**
M. Rond	DOP-B		1/24/2000
SIGNATURE *(Operator at Treatment Facility)*	**TREATMENT FACILITY**		**DATE**
Carlos Padgett	ABG		1-25-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION*	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
White Smoke Comp in fuel Oil	Metal	Fuel Oil	126/~~FOA~~	2 @ 55 lb ea
2 yrs Cont. Rag + filters in fuel oil	Metal	Fuel Oil	126/~~FOA~~	3 @ 40 lb ea.

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor-Extension No.)		CODE	DATE
Melanie Weckard	x3026	IOC	1/20/2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
Jim L. Road	DOD-B		1/20/2000
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**		**DATE**
Chris Cady	ABC		1-20-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION*	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
AAR Ignition Mix in Acetone	Metal	Acetone	126/~~10A~~	25 lb
Illum Comp in Acetone	Metal	Acetone		45 lb
Zirc Dust in Fuel Oil	Metal	Fuel Oil	126/~~10A~~	30 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE *(Generator or Facility Supervisor-Extension No.)*		CODE	DATE
[illegible]	X3026	IBC	1-13-2000
SIGNATURE *(Transporter)*	**ORGANIZATION**		**DATE**
[illegible] Roark	DOP-B		1-13-2000
SIGNATURE *(Operator at Treatment Facility)*	**TREATMENT FACILITY**		**DATE**
[illegible]	ABG		1-13-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION*	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
yellow fire in Fuel Oil	Metal	Fuel Oil	126/IDA	3 @ 60 lb ea.
pyro contaminated oil filter in fuel oil	Metal	Fuel Oil	126/IDA	3 @ 65 lb ea.
yellow fire fire in Fuel Oil	Metal	Fuel Oil	126/IDA	1 @ 20 lb ea.

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE (Generator or Facility Supervisor Extension No.)		CODE	DATE
[signature]	X3026	IOC	1-10-2000
SIGNATURE (Transporter)	**ORGANIZATION**		**DATE**
[signature]	DOD B		1-10-2000
SIGNATURE (Operator at Treatment Facility)	**TREATMENT FACILITY**		**DATE**
[signature]	ABG		1-11-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

CONTAMINATED SCRAP MANIFEST
NWSCC 6280/4 (REV. 6-84)

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION*	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)
Potassium Nitrate in Fuel Oil	Metal	Fuel Oil	126/IOA	16 lb
Contaminated Filters & Bags in Fuel Oil	Metal	Fuel Oil	126/IOA	60 lb

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE *(Generator or Facility Supervisor-Extension No.)*		CODE	DATE
Melanie Duckard	X3026	IOC	1-3-2000
SIGNATURE *(Transporter)*	**ORGANIZATION**		**DATE**
[illegible]	DOD-B		1-3-2000
SIGNATURE *(Operator at Treatment Facility)*	**TREATMENT FACILITY**		**DATE**
[illegible]	ABG		1-5-00

DISTRIBUTION

The following copies shall be withdrawn by the respective personnel and sent to Code 0924, Building 2516, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

NOTE: Generator - retains Pink; Operator of Treatment Facility retains Green

ORDNANCE BRIEFING PROGRAM FOR EXPLOSIVES SAFETY PERSONNEL
18 AUGUST 2008 SESSION

General. The following is presented for the purpose of transferring historical and pertinent timely information to selected explosives safety personnel.

1. Building 146 Area. Building 146 is one of three buildings that made up the 40mm loading and assembly plant constructed during WWII. Building 146 was used to load the 40mm projectiles, Building 145 was used to load the cartridge cases and to handle the final assembly of the projectile to the cartridge case as well as for pack-out of the finished round. Building 148 was an inert materials support building for the area.

Building 146 has been used through the years for many different types of ordnance work. Beginning in the early 1960's the south end of the building was used for the demilitarization of small arms and 20mm ammunition. Three rotary furnaces, similar to the APE 1236 furnace design, were installed and operated. The process involved breaking down the rounds to remove propellant and then introducing the projectile to one furnace while the empty but still primed case was introduced to another much smaller furnace. The scrap was then sold and the money reinvested for future use in funding other demil processes. During the 1980's other demilitarization efforts were undertaken in the building such as 5" projectile explosive removal by dry methods and explosive removal using high pressure water. In addition, a demilitarization line was installed for the sole purpose of breaking down white phosphorus rounds to permit their introduction into the white phosphorus to phosphoric acid conversion plant at Building 69. Currently, a conversion process for illuminating composition is being installed in bays 1, 2 and 3. The future capabilities of Building 146, regarding work with Class 1 Division 1 materials, are controlled by the environmental protection office, Building 3260.

Building 145 has been used through the years for processes involving propellant loading. In the early 1980's an automated bag charge loading line was installed for testing. It was being tested in preparation for installation at the Indiana Army Ammunition Plant, Charlestown Indiana. It however, failed to work and was removed. No major long term work was accomplished in this building until the early 1990's when a final assembly building was required to support infrared decoy flare production. That process has been up and running since that time. Building 145 is now heavily constrained by Highway H-45 that will continue to be a Public Traffic Route. The latest feasibility study of CAAA buildings indicates that the IR decoy flare production will most likely shift to Building 2520 and not remain in Building 145. Like Building 146, Building 145 has an WWII primary lightning protection design that would need to be upgraded if the building was to be site approved in the future. Unlike Building 146, Building 145 has been formally site approved. However, at the time of the review, the

actual design of the primary lightning protection system was not an issue requiring the submission of the design documents for approval.

Building 148 was reworked in the 1990's to remove and replace the gable roof with the flat roof now visible. It continues to be used in a support capacity but not directly associated with the work accomplished in either Building 145 or Building 146. By strict interpretations of safety rules, the functions now on-going should be out of Building 148 and placed in a building at inhabited building criteria distance from ordnance areas if the entire Building 146 site was to be further developed and officially approved.

2. Pyrotechnic Production Area (Building 126 Complex). The complex of buildings was designed and built during WWII for the exclusive purpose of loading, assembling and packing out star shell ammunition. The star shell, primarily a 5" illuminating round with a parachute, required a good deal of support facilities. The process flowed as follows:

a. Building 121 second floor was used for the fabrication of the parachutes.
b. Buildings123, 124 and 125 were used for the generation of metal parts.
c. Building 126 was used for the preparation of the illumination composition and its pressing into candles.
d. Building 122 was the final assembly building where the candle was inserted into the projectile, the parachute was attached and packed and the whole round packed-out for rail shipment from the plant.
e. The host of smaller buildings that surround Building 126 housed chemical storage, candle curing, ready magazines and inert support warehouses that were necessary to maintain the process.

Building 126 was designed with many separate work cells where ingredients were prepared, mixed and then pressed into a final candle configuration.
Because of the unique multi-cell design, Building 126 became attractive as a general purpose pyrotechnic loading building. Through the 1950's and 1960's, the building supported a number of illuminating candle processes. The Mk 24 and Mk 45 illuminating candles were produced in the building using an automated pressing line. From that time until now, the building continues to be used for supporting pyrotechnic illuminating rounds, smokes and signals.

Building 122 was transferred to the Navy from the Army in the 1990's after being renovated to support the 16"/50 caliber projectile rework program that was no longer needed when the Iowa Class Battleships were decommissioned.

Buildings 2697 and 2698 have been used to support the Mk 25 and Mk 58 marine location marker production efforts. A new site plan is being prepared to site approve these buildings for supporting a number of pyrotechnic production efforts. Building 133 and Building 3148, were also used to support the marker program which now is not work loaded at Crane. These two buildings remain dormant. Buildings 1885 and 1886 were used for mixing the red phosphorus composition that was used for the markers. These two buildings are no longer in good condition and need to be demolished. They are heavily contaminated with red phosphorus which is a fire hazard.

There are four stories of particular interest pertaining to the Building 126 Complex. The first involves Building 127 now used as a break area for the complex. This building was first used a supporting laboratory for the work on-going in Building 126. An attachment to the west of Building 127 housed a burn chamber similar to that near Building 190. During experiments in the 1960's involving the relationship of candle density to illuminating round performance, an explosion occurred while a burn was being conducted with a very low density press loaded candle. The explosion destroyed the burn chamber and displaced a door to the control room, striking an engineer killing him instantly. After the explosion, the burn chamber was relocated to the Building 190 area where it was installed as a portion of an employee access tunnel between Building 2540 and Building 198.

The second involved the destruction by fire of a curing building located to the south of Building 126 where a new curing hut now stands. The incident occurred on a Sunday morning in the late 1980's. The building was totally destroyed and the fire was traced to an overheating condition where the unit producing the heat needed for curing candles failed to be controlled, thus, cooking off the candles.

The third involved Building 130 that was being used for weighing out red phosphorus. A flash fire occurred when an employee attempted to empty the last remaining red phosphorus from a barrel. The investigation revealed that the plastic bag that was the liner of the barrel was not conductive as thought, and as the red phosphorus was falling through space inside the bag, a spark ignited the finely ground red phosphorus. The slow evolving fire did not result in injury because the employee reacted quickly and jumped into the horse trough that was placed at the door for fire suppression purposes. That incident highlighted the nature of finely ground powders to generate sufficient electrical charge while falling in space to result in a spark discharge.

The fourth story involves an R&D effort that was being conducted in Building 1886 in the 1980's. The Navy R&D folks employed CAAA production personnel in a joint effort to experiment with new marker compositions. The experiments involved red phosphorus, boron, lead dioxide and a binder material. The process involved mixing the three dry ingredients with the binder liquid. The test procedure provided sensitivity data for all of the ingredients separately and the final mix. During the loading effort, a flash fire occurred that seriously burned an employee. The investigation revealed that the ingredients were added into the mixer bowl in a different manner than that called for by the test procedure in an effort to improve the mixing effort. Analysis of the ingredients, as they were actually placed in the mixer bowl, revealed a sensitivity to impact that was the most severe ever recorded in the industry. The lesson learned was that knowing the sensitivity of individual ingredients and the final product may not be good enough to outline the true hazards of that process. More information on the sensitivity of ingredients during the various process steps (especially for pyrotechnic compositions) may be necessary to obtain.

3. Explosive Actuating Devices Area. This area was first designed and constructed to load 5" rockets during WWII. Building 138 was the pressing building for the warheads, building 136 was used for the propellant portion and Building 2520 as the final assembly building. A conveyor tunnel connects Building 136 and 2520 in support of this process. In the early 1960's a detonator loading line was established in Building 136 where lead

B-198 Dye Test Area

John Laswell & Dave McNulty are proposing to set off a M18 Smoke Hand Grenade simultaneous with the dye test units for comparision of smoke/dye cloud produced.

Proposing to test one M18 per dye test unit with a maximum of 10 units of each per day being tested.

7/7/83.

Discussed proposal with Cathy and what effects this new testing could have on area. Cathy was in agreement to go ahead with testing and that I should be on-site when initial testing starts to see how far smoke drifts.

Contacted Dave McNulty asking him to go ahead on prepare test procedure for test smoke grenade simultaneous with dye unit. Also ask Dave to look at wind factor when testing smoke grenade.

DEPARTMENT OF THE ARMY
U. S. ARMY ENVIRONMENTAL HYGIENE AGENCY
ABERDEEN PROVING GROUND, MARYLAND 21010-5422

Mr. Lancellotti/dlc/AUTOVON
584-3816

REPLY TO
ATTENTION OF

17 APR 1985

HSHB-EW-S/WP

SUBJECT: Water Quality Engineering Study No. 32-24-0575-85, Metal Finishing Operation Compliance, Crane Army Ammunition Activity, Crane Indiana, 9-13 September 1984

Commander
US Army Materiel Command
ATTN: AMCSG
5001 Eisenhower Avenue
Alexandria, VA 22333-0001

1. Twenty copies of the subject report with Executive Summary are enclosed.

2. Additional copies of this report are enclosed for mailing to Commander, US Army Corps of Engineers (DAEN-ZCF-U/DAEN-ZCE) and Commandant, Academy of Health Sciences (HSHA-IPM).

FOR THE COMMANDER:

Encl

KARL J. DAUBEL
Colonel, MS
Director, Environmental Quality

CF:
HQDA(DASG-PSP) (wo/encl)
Cdr, AMCCOM (AMSMC-SG/AMSMC-ISE) (w/encl)
Cdr, HSC (HSCL-P) (w/encl)
Cdr, Crane AAA (2 cy) (w/encl)
Cdr, WRAMC (PVNTMED Svc) (w/encl)
Cdr, MEDDAC, Ft Knox (PVNTMED Svc) (2 cy) (w/encl)
C, USAEHA-Rgn Div North (w/encl)

DEPARTMENT OF THE ARMY
U. S. ARMY ENVIRONMENTAL HYGIENE AGENCY
ABERDEEN PROVING GROUND, MARYLAND 21010-5422

REPLY TO
ATTENTION OF

HSHB-EW-S/WP

EXECUTIVE SUMMARY
WATER QUALITY ENGINEERING STUDY NO. 32-24-0575-85
METAL FINISHING OPERATION COMPLIANCE
CRANE ARMY AMMUNITION ACITIVITY
CRANE, INDIANA
9-13 SEPTEMBER 1984

1. PURPOSE. To determine whether Crane Army Ammunition Activity's (AAA) metal finishing operations were complying with the recently promulgated metal finishing categorical effluent limitation guidelines and to estimate waste-water characteristics and regulatory compliance under mobilization conditions.

2. ESSENTIAL FINDINGS.

a. Production during the study was approximately 55 percent of mobilization capacity.

b. The plating shop wastewater pretreatment facility complied with the metal finishing BAT/BCT (best available technology economically achievable/ best conventional pollutant control technology) effluent limitations.

c. Machine coolants, which are eventually discharged to the sanitary sewer, contain high levels of metal finishing BAT/BCT pollutants.

c. [redacted] which are eventually discharged to the sanitary sewer, [redacted]

d. Wastewater samples from the chrome rinse water sump contained cyanide.

e. Crane AAA should be able to comply with metal finishing BAT/BCT effluent limitations at mobilization.

3. MAJOR RECOMMENDATIONS.

a. Dispose of spent machine coolant fluids through a hazardous waste contractor.

b. Request that the permitting authority certify compliance with the total toxic organics standards.

b. Request [redacted] the [redacted]

c. Investigate plating shop operations in order to determine if plating shop personnel are conforming to standard operating procedures with respect to the cyanide plating operation.

DEPARTMENT OF THE ARMY
U. S. ARMY ENVIRONMENTAL HYGIENE AGENCY
ABERDEEN PROVING GROUND, MARYLAND 21010-5422

REPLY TO
ATTENTION OF

HSHB-EW-S/WP

WATER QUALITY ENGINEERING STUDY NO. 32-24-0575-85
METAL FINISHING OPERATION COMPLIANCE
CRANE ARMY AMMUNITION ACTIVITY
CRANE, INDIANA
9-13 SEPTEMBER 1984

1. AUTHORITY. Letter, HQ AMCCOM, DRSMC-ISE(R), 5 July 1983, subject: Proposed Effluent Regulations, Metal Finishing Point Source Category - US Army Environmental Hygiene Agency (USAEHA) Assistance, with 1st Ind, HQ DARCOM, DRCSG-S, 12 September 1983.

2. REFERENCES. A list of references used is included as Appendix A.

3. PURPOSE. The purpose of this study was to determine whether Crane Army Ammunition Activity's (AAA) metal finishing operations were complying with the recently promugated metal finishing categorical effluent limitation guidelines (reference 2) and to estimate wastewater characteristics and regulatory compliance under mobilization conditions.

4. GENERAL.

a. Personnel Contacted. A list of personnel contacted during this study is included as Appendix B.

b. Project Execution. A summary of the sampling methods, analytical schedule and analytical methodology is included as Appendix C.

5. BACKGROUND.

a. Mission. Crane AAA, a US Army Materiel Command activity under the intermediate command of the US Army Armament, Munitions and Chemical Command, produces and renovates conventional ammunition and ammunition-related components. It performs manufacturing (industrial) engineering and product assurance in support of production. It also stores, ships, and/or demilitarizes and disposes of conventional ammunition and related items.

b. Regulatory Requirements. Crane AAA was required to comply with the metal finishing BAT/BCT (best available technology economically achievable/ best conventional pollutant control technology) limits as soon as possible but not later than 1 July 1984 (reference 2). Metal finishing BAT/BCT limits are shown in Table 1. Total toxic organics (TTO) consist of the sum of those organic compounds listed in Appendix D detected at a concentration greater than 10.0 micrograms per liter (µg/L). These limits are applicable to wastewater discharges from any of the operations listed in Table 2, if the industrial activity performs at least one of the following metal finishing

TABLE 1. METAL FINISHING CATEGORY, BAT/BCT EFFLUENT LIMITATIONS

Pollutant or Pollutant Property (all units mg/L unless stated otherwise)	Maximum for any 1 Day	Average of Daily Values of 30 Consecutive Days Shall Not Exceed
Cadmium	0.69	0.26
Chromium	2.77	1.71
Copper	3.38	2.07
Lead	0.69	0.43
Nickel	3.98	2.38
Silver	0.43	0.24
Zinc	2.61	1.48
Cyanide, Total	1.20	0.65
Cyanide, Amenable*	0.86	0.32
Total Toxic Organics	2.13	-----
Oil and Grease	52	26
Total Suspended Solids	60	31
pH	between 6.0 and 9.0 standard units at all times	

* May be substituted for cyanide, total limitation where cyanide treatment systems exist.

TABLE 2. METAL FINISHING CATEGORICAL OPERATIONS

Cleaning*	Machining*
Grinding*	Polishing
Tumbling	Pressure Deformation
Impact Deformation	Heat Treating*
Shearing	Welding*
Thermal Cutting	Soldering*
Brazing*	Sandblasting
Flame Spraying	Electric Discharge Machining
Other Abrasive Jet Machining	Electron Beam Machining
Electrochemical Machining	Plasma Arc Machining
Laser Beam Machining	Sintering
Ultrasonic Machining	Hot Dip Coating*
Laminating	Vapor Plating
Sputtering	Salt Bath Descaling
Thermal Infusion	Paint Stripping
Solvent Degreasing*	Electrosatic Painting
Painting	Vaccum Metalizing
Electropainting	Calibration*
Assembling*	Mechanical Plating
Testing*	

* In operation at Crane AAA

operations on any basis metal: electroplating, electroless plating, anodizing, coating (chromating, phosphating, and/or coloring), chemical etching and milling, or printed circuit board manufacturing. Electroplating and coating operations are a part of the manufacturing facilities at Crane AAA. Regulated operations at Crane AAA are noted in Table 2. Metal finishing BAT/BCT limitations have been incorporated into Crane AAA's National Pollutant Discharge Elimination System (NPDES) Permit.

c. Industrial Operations at Crane AAA.

(1) Industrial Machine Shop.

(a) Metal Finishing Activity. The machine shop occupies approximately 65,000 square feet of floor space in Bldg 123, 124, and 125. Complex machining, welding, and heat treating operations were being performed for production and tooling requirements.

(b) Wastewater Sources. Wastewaters consist of cooling waters (from welding and heat treating operations), deburring machine waste, and water-soluble machining coolants.

(b) Wastewater Sources. Wastewaters consist of cooling water (from welding and heat treating operations), [redacted] waste, and [redacted] coolants.

(c) Wastewater Treatment. Wastewater generated from machine shop operations was being discharged to the sanitary sewer.

(2) Metal Plating Shop.

(a) Metal Finishing Activity. The plating shop in Bldg 1884 performed acid and alkaline cleaning, chrome conversion coating, phosphate coating, and cadmium/zinc cyanide plating on a variety of munitions-related metal parts in support of the adjacent industrial machine shop. A schematic of metal finishing operations is included as Figure E-1, Appendix E.

(b) Wastewater Sources. The main source of plating shop wastewater was from continuous rinses following acid/alkaline cleaning, chrome/phosphate coating, and cadmium/zinc cyanide plating operations. Spent concentrated process plating solutions were disposed of through a hazardous waste contractor.

(c) Wastewater Treatment. Wastewaters were segregated into three independent collection systems, to facilitate treatment, before being pumped to the plating shop wastewater pretreatment facility (PSWPF) in Bldg ~~2957~~ 3064 (acid/alkali, chromium and cyanide collection systems). A schematic of the wastewater collection and treatment system is included as Figure E-2, Appendix E.

- Cadmium and zinc cyanide wastewaters were collected in an underground sump located outside of Bldg 1884. Wastewater was pumped from the sump to a cyanide destruction process (two-stage alkaline chlorination) before mixing with other waste for subsequent heavy metals removal.

- Chrome-bearing wastewater was collected in a sump in Bldg 1884 and pumped to a holding tank in Bldg ~~2957.~~ 3064 Since most of the chrome was in the hexavalent state, it is subjected to chemical reduction to the trivalent state (with sulfuric acid and sodium metabisulfite) before mixing with other waste for subsequent precipitation and filtration.

- Wastewater from the acid/alkaline cleaning processing line was pumped directly to the pretreatment holding tank and mixed with the pretreated (oxidized) cyanide and chrome (reduced) wastewaters.

- The combined wastewater in the pretreatment holding tank (oxidized cyanide wastewater, reduced chrome wastewater, and acid/alkali wastewater) was pumped to a two-stage precipitation reaction process to render metals insoluble. Sulfuric acid and lime were added to effect precipitation of metal hydroxide at low and high pH levels (8 and 10, respectively). The effluent from the secondary precipitation reactor was pumped through a diatomaceous earth filter. Effluent from the filter was directed to a final pH adjustment tank and then discharged to the sanitary sewer. This discharge was designated as outfall 101 on Crane AAA's NPDES permit. Discharge limitations are included in Appendix F.

6. FINDINGS.

a. Production. During the study period a variety of small ammunition-related metal parts were processed through the machine and plating shops (bomb saddles, bands, smoke canisters, etc.) The total number of production pieces processed during the study is summarized in Table 3. Crane AAA's personnel estimated that this level of production was approximately 55 percent of mobilization capacity.

TABLE 3. STUDY PRODUCTION LEVELS, CRANE AAA, 10-12 SEPTEMBER 1984

Date	Metal Parts Processed
10 Sep 1984	2136
11 Sep 1984	4339
12 Sep 1984	2933
Total	9408

b. BAT/BCT Effluent Limitations Compliance.

(1) PSWPF. The Crane AAA PSWPF was in compliance with metal finishing BAT/BCT standards during the study. As can be seen from the PSWPF effluent summary shown in Table 4, the maximum and average effluent concentrations during the study were well below the BAT/BCT limits. Complete analytical results for the PSWPF effluent are included in Tables G-1 through G-3, Appendix G.

TABLE 4. PSWF EFFLUENT SUMMARY, CRANE AAA, 10-12 SEPTEMBER 1984

Parameter (units mg/L unless stated otherwise)	Effluent Concentration Maximum	Effluent Concentration Average*	Metal Finishing BAT/BCT Daily Max	Metal Finishing BAT/BCT 30-day Average
Cadmium	0.045	0.040	0.69	0.26
Chromium	<0.025	<0.025	2.77	1.71
Copper	<0.025	<0.025	3.38	2.07
Lead	<0.020	<0.20	0.69	0.43
Nickel	<0.10	<0.10	3.98	2.38
Silver	<0.010	<0.010	0.43	0.24
Zinc	<0.015	<0.015	2.61	1.48
Cyanide, Total	0.03	<0.017	1.20	0.65
Cyanide, Amenable	<0.01	<0.01	0.86	0.32
Total Toxic Organics†	0.770	0.377	2.13	-----
Oil and Grease	3.5	2.8	52	26
Total Suspended Solids	5.1	3.1	60	31
Flow (gallons)	9,440	7,737	-----	-----

* Three-day average
† See Appendix D for a breakdown of individual compounds in TTO category

(2) Machine Coolants. Machine coolants (friction-reducing lubricant) were being used in a number of machining operations at the Crane AAA machine shop (Bldgs 123 through 125). Machines using these coolants had built-in reservoirs which recirculated the coolant while the machines were in operation. Machine shop personnel indicated that coolants break down after prolonged use and have to be replaced with fresh coolant fluids (coolants last 3-6 months) and that spent coolant fluids are discharged to the sanitary sewer after floating oils have been removed. These discharges are related to Crane AAA's metal finishing operation and are, therefore, regulated by metal finishing BAT/BCT effluent limitations. During the study period grab samples were taken from coolant reservoirs at machines No. 176 and 215 and analyzed for metal finishing BAT/BCT pollutants. Analyses of the samples taken are presented in Table 5. A comparison of sample concentrations with allowable BAT/BCT daily maximum effluent limitations indicates that a number of these limits would have been exceeded had the coolants been discharged to the sewer at the time of this survey. In the future, spent coolant fluids should be disposed of through a hazardous waste contractor instead of being discharged to the sanitary sewer.

c. Certification. The metal finishing BAT/BCT effluent limitation regulations allow Crane AAA to "certify" its compliance with the TTO standard in place of regular TTO analytical monitoring. This was done because the sample collection procedure and analyses for TTO can be prohibitively expensive. The substitution of certification for analysis can be done only with the permission of the permitting authority. In light of the low levels of TTO's detected at the PSWPF effluent (outfall 101) during this study and the low potential for the discharge to exceed the TTO standard, Crane AAA should request permission to certify compliance with the TTO standard.

TABLE 5. ANALYTICAL RESULTS, SAMPLE POINTS 5 AND 6, MACHINE COOLANT, MACHINE SHOP, CRANE AAA, 10 SEPTEMBER 1984

Parameter (units mg/L unless stated otherwise)	Machine No. 176 (sample point 5)	Machine No. 215 (sample point 6)	Metal Finishing BAT/BCT Daily Maximum
Cadmium	0.213	0.222	0.69
Chromium	[redacted]	0.235	2.77
[redacted]	[redacted]	[redacted]	3.38
[redacted]	0.36	[redacted]	0.69
Nickel	0.22	<0.10	3.98
Silver	<0.10	<0.010	0.43
Zinc	3.73*	1.265	2.61
[redacted]	[redacted]	0.520	2.13
Oil and Grease	8185.0*§	88.0*	52.0
[redacted]	[redacted]	[redacted]	[redacted]

* Exceeds metal finishing BAT/BCT daily maximum effluent limitation.
† See Tables G-6 and G-7, Appendix G, for a breakdown of compounds analyzed and their detection limits.
‡ Only the volatile organic fraction of TTO compounds was analyzed.
§ Oil phase was not removed before analysis.

d. Process Rinse Water Sumps.

(1) Analytical Data. Grab samples were taken from the chrome, cyanide, and acid/alkali rinse water sumps on three consecutive days. Analytical data for the cyanide and acid/alkali sump samples are given as Tables G-4 and G-5, Appendix G. Analytical data for the chrome rinse water sump are discussed in the following paragraph.

(2) Cyanide-contaminated Chrome Rinse Water. Analyses of chrome rinse water sump samples [redacted] operations were draining into the chrome rinse water [redacted] in order to ensure that plating shop personnel are processing metals parts requiring cyanide plating through designated cyanide process tanks (including rinse water tanks) and that spillage from these tanks is being kept to a minimum. Conformance with the above standard operating procedures should eliminate cyanide contamination of the chrome rinse water sump.

TABLE 6. ANALYTICAL RESULTS, METALS AND NONMETALS, SAMPLE POINT 2, CHROME RINSE WATER SUMP, METAL PLATING SHOP, CRANE AAA, 10-12 SEPTEMBER 1984

Parameter (units mg/L)	10 Sep 84	11 Sep 84	12 Sep 84	Mean
[redacted]mium	[redacted]	2.69	12.5	[redacted]
[redacted]omium	[redacted]	21.4	24.5	[redacted]
Copper	29.1	3.54	5.92	12.85
Lead	1.06	<0.20	0.29	<0.52
Nickel	0.73	<0.10	0.15	<0.33
Silver	0.035	<0.010	0.011	<0.019
Zinc	38.2	1.42	6.31	15.31
Cyanide, Total	[redacted]	0.77	1.2	[redacted]

e. Mobilization.

(1) Production. Crane AAA personnel did not have a definitive production schedule for full mobilization. Production at the time of this study was estimated to be at approximately 55 percent of full mobilization (one 10-hour shift). The following assumptions were used to estimate the mobilization wastewater flow rates and wastewater characteristics.

(a) Mobilization is considered to be two 10-hour production shifts, 6 days per week (500-hour production month).

(b) The existing manufacturing equipment will remain the same without any major changes, i.e., equipment moderization.

(c) Current manufacturing compounds/solutions will remain the same.

(d) No additional water conservation techniques will be instituted.

(e) Only wastewater being discharged from the plating shop will require treatment.

[redacted]

(g) The three process rinse water streams (chrome, cyanide, and acid/alkali) are assumed to be discharging equal amounts of wastewater to the PSWPF during a production shift.

(h) Estimated wastewater flow to the PSWPF will be based on measured PSWPF effluent data obtained during the study period.

(2) Wastewater Flow. Installation personnel estimated that at full mobilization, wastewater flow to the PSWPF during the first shift will increase by 25 percent of the present flow. Wastewater flow during the second shift will be at approximately the same level that is currently being discharged to the PSWPF. Estimated wastewater flow to the PSWPF during the first shift (maximum discharge period) is presented in Table 7.

TABLE 7. ESTIMATED MOBILIZATION WASTEWATER FLOW, PSWPF INFLUENT, CRANE AAA

Date	Total Flow (gal)	Time of Flow (hour)	Average Flow (gpm)
Study Period Measured PSWPF Effluent Wastewater Flow			
10 Sep 84	7,110	8.0	14.8
11 Sep 84	9,440	10.5	15.0
12 Sep 84	6,660	8.0	13.9
Average Flow			14.6
First Shift Estimated Mobilization Wastewater Flow			
Total Estimated PSWPF Influent Flow (1.25 x 14.6 gpm)			18.3
Estimated chromium reduction system influent flow			6.0
Estimated cyanide oxidation system influent flow			6.0
Estimated precipitation/neutralization system influent flow			18.3

(3) Wastewater Characteristics. Given the assumptions outlined above, the wastewater characteristics at mobilization should be equivalent to the current wastewater characteristics.

(4) Wastewater Treatment. [redacted] tions, Crane AAA [redacted] As can be see from Table 8, at mobilization flow rates, the PSWPF unit processes are within the recommended minimums.

TABLE 8. MOBILIZATION OPERATION ANALYSIS, PSWPF, CRANE AAA

Treatment Tank	Tank No.	Tank Volume (gal)	Mobilization Detention Time (min)	Recommended Detention Time (min)
Chromium Reduction	T-6	216	36	>5*
Cyanide Oxidation				
Stage I (cyanide to cyanate)	T-8	306	51	>10*
Stage II (cyanate to CO_2 & N_2)	T-9	535	89	>20*
Metals Precipitation				
Stage I (pH 7-9)	T-16	1490	81	>20*
Stage II (pH 9-10)	T-17	535	29	>20*
Neutralization (pH 6-9)	T-22	476	26	> 3†

* Extracted from reference 3
† Extracted from reference 4

7. CONCLUSIONS.

a. Production during the study was approximately 55 percent of mobilization capacity.

b. The PSWPF complied with the metal finishing BAT/BCT effluent limitations.

c. Machine coolants, which are eventually discharged to the sanitary sewer, contain high levels of metal finishing BAT/BCT pollutants.

[redacted]

d. Wastewater samples from the chrome rinse water sump contained cyanide.

e. Mobilization wastewater flows and characteristics have been estimated.

f. Crane AAA should be able to comply with metal finishing BAT/BCT effluent limitations at mobilization.

8. RECOMMENDATIONS. The following recommendations are based on good environmental engineering practices.

a. Disposal of spent machine coolant fluids through a hazardous waste contractor [see paragraph 6b(2), this report.

a. [redacted]

b. Request that the permitting authority certify compliance with the TTO standard (see paragraph 6c, this report).

b. [redacted]

c. Investigate plating shop operations in order to determine if plating shop personnel are conforming to standard operating procedures with respect to the cyanide plating operation (see paragraph 6d, this report).

9. TECHNICAL ASSISTANCE. Requests for services should be directed through appropriate command channels of the requesting activity to Commander, US Army Environmental Hygiene Agency, ATTN: HSHB-EW, Aberdeen Proving Ground, MD 21010-5422, with an information copy furnished to the Commander, US Army Health Services Command, ATTN: HSCL-P, Fort Sam Houston, TX 78234-6000.

for KENNETH A. LANCELLOTTI
Chemical Engineer
Water Quality Engineering Division

APPROVED:

JAMES M. STRATTA
LTC, MS
Chief, Water Quality Engineering Division

12

APPENDIX A

REFERENCES

1. Public Law (PL) 92-500, Federal Water Pollution Control Act Amendments of 1972, 18 October 1972, as amended by PL 95-217, Clean Water Act of 1977, 27 December 1977, and PL 95-576, Amendments to the Clean Water Act, 14 October 1978.

2. Title 40, Code of Federal Regulations (CFR), 1984 rev, Part 433, Metal Finishing Point Source Category.

3. Metal Finishing Guidebook, Directory Issue 84, Metal Parts and Plastics Publications Inc., January 1984.

4. Vincent Cavaseno and the Staff of Chemical Engineering Magazine, Industrial Wastewater and Solid Waste Engineering, McGraw-Hill Publications Co., 1980.

5. Letter, USAEHA, HSHB-EW/WP, 19 October 1985, subject: Preliminary Report, Water Quality Engineering Study No. 32-24-0575-85, Metal Finishing Operation Compliance, Crane Army Ammunition Activity, Crane, Indiana, 9-13 September 1984.

APPENDIX B

PERSONNEL CONTACTED

MAJ Bohdan Dombchewskyj, Acting Commander, Crane AAA.

Mr. Bill Hubble, Environmental Coordinator, Project Engineering Division, Engineering Directorate, Crane AAA.

Mr. Virgil Davis, Chief, Industrial Shop Division, Industrial Operations Directorate, Crane AAA.

Mrs. Patty Roberts, Engineering Technician, Project Engineering Division, Engineering Directorate, Crane AAA.

Mr. Phillip A. Keiths, Environmental Specialist, Naval Weapons Support Center, Crane.

APPENDIX C

PROJECT EXECUTION

1. SAMPLE TYPES AND LOCATIONS. Sample types and locations are summarized in Table C-1. The project analytical schedule is included as Table C-2. Sample locations are illustrated on the Figure. The analytical methodology used is summarized in Table C-3.

2. SAMPLE PRESERVATION. Analyses were performed at this Agency. All samples were preserved according to the US Environmental Protection Agency's preservation guidelines and transported by Federal Express to this Agency. All analyses were conducted within the prescribed holding time.

TABLE C-1. SAMPLE TYPE AND LOCATION, CRANE AAA

Sample Point	Location	Sample Type
1	Waste Treatment Facility Outfall	Continuous flow, flow composite, grab
2	Chrome Rinse Sump	grab
3	Cyanide Rinse Sump	grab
4	Acid-Alkali Rinse Sump	grab
5	Machine Coolant Waste	grab
6	Machine Coolant Waste	grab

TABLE C-2. ANALYTICAL SCHEDULE, CRANE AAA

Parameter	Composite Samples (1/day/3 days) Sample Point	Grab Samples (1/day/3 days) Sample Point			Grab Samples (1/3 days) Sample Point	
	1	2	3	4	5	6
Cadmium	X	X	X	X	X	X
Chromium	X	X	X	X	X	X
Copper	X	X	X	X	X	X
Lead	X	X	X	X	X	X
Nickel	X	X	X	X	X	X
Silver	X	X	X	X	X	X
Zinc	X	X	X	X	X	X
Cyanide, Total	X	X	X	X		
Cyanide, Amenable	X					
Oil and Grease	X				X	X
Total Suspended Solids	X				X	X
Volatile Organics	X				X	X
Acid Extractable Organics	X				X	X
Base Neutral Extractable Organics	X				X	X
Pesticides/PCBs	*					

* Sample taken on one day.

16

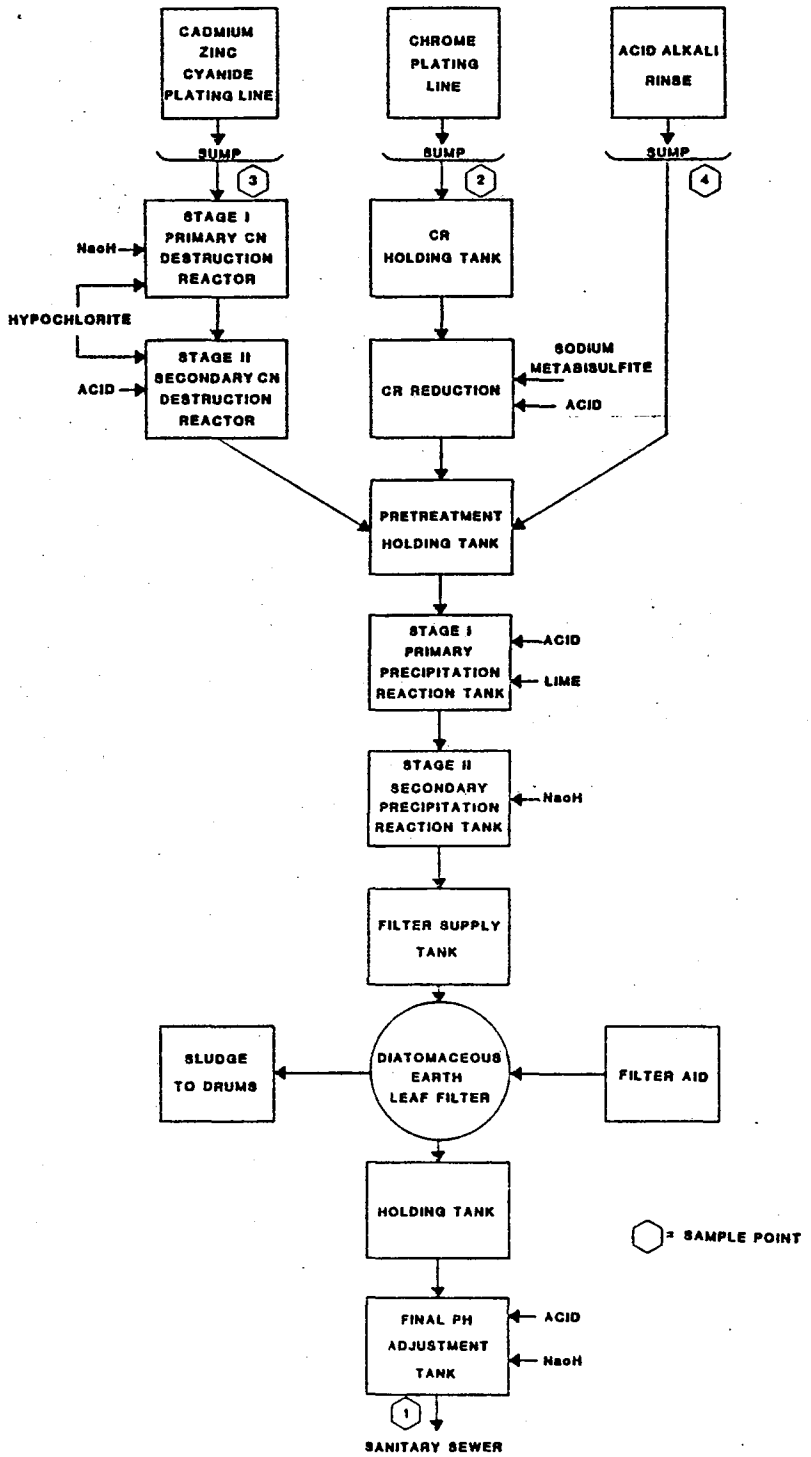


FIGURE SAMPLE POINT LOCATIONS, PSWPF, CRANE AAA, SEPT 84

TABLE C-3. ANALYTICAL METHODOLOGY

Parameter	Reference	Description
Cyanide, Total	EPA 335.2*	Spectrophotometric, manual distillation
Cadmium	EPA*	Flame Atomic Absorbtion
Chromium, Total	EPA*	Inductively Coupled Argon Plasma
Copper	EPA*	Inductively Coupled Argon Plasma
Lead	EPA*	Flame Atomic Absorbtion
Nickel	EPA*	Inductively Coupled Argon Plasma
Silver	EPA*	Inductively Coupled Argon Plasma
Zinc	EPA*	Inductively Coupled Argon Plasma
Oil and Grease	EPA 413.1*	Liquid-Liquid Extraction, gravimetric
Volatile Organics	EPA 624†	Purge and Trap, GC/MS
Base/Neutral Extractables	EPA 625†	Methylene Chloride Extraction, GC/MS
Acid Extractables	EPA 625†	Methylene Chloride Extraction, GC/MS
Pesticides/PCBs	EPA 625†	Methylene Chloride Extraction, GC/MS
pH	EPA 150.1*	Electrochemical
Total Suspended Solids	EPA	Gravimetric, dried at 103-105°C

* EPA Methods for Chemical Analysis of Water and Wastes, EPA 600-4-79-020, Environmental Monitoring and Support Laboratory, US Environmental Protection Agency, Cincinnati, OH 45268, March 1983.
† Final Rule and Interim Final Rule, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, 49 FR 43234, 26 October 1984.

18

APPENDIX D

TOTAL TOXIC ORGANICS

Acrolein
Acrylonitrile
Benzene
Toluene
Ethylbenzene
Carbon tetrachloride
Chlorobenzene
1,2-Dichloroethane
1,1,1-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethylene
1,1,2-Trichloroethane
1,1,2,2-Tetrachloroethane
Chloroethane
2-Chloroethyl vinyl ether
Chloroform
1,2-Dichloropropane
1,3-Dichloropropene
Methylene Chloride
Methyl chloride
Methylbromide
Bromoform
Dichlorobromomethane
Chlorodibromomethane
Tetrachloroethylene
Trichloroethylene
Vinyl chloride
1,2-trans-Dichloroethylene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Hexachloroethane
Hexachlorobutadiene
Hexachlorobenzene
1,2,4-Trichlorobenzene
bis(2-Chloroethoxy)methane
Naphthalene
2-Chloronaphthalene
Isophorone
Nitrobenzene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
4-Bromophenyl phenyl ether
bis(2-Ethylhexyl)phthalate
Di-n-octyl phthalate
Dimethyl phthalate
Diethyl phthalate
Di-n-butyl phthalate
Acenaphthylene
Acenaphthene
Butyl benzyl phthalate
Fluorene
Fluoranthene
Chrysene
Pyrene
Phenanthrene
Anthracene
Benzo(a)anthracene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-c,d)pyrene
Dibenzo(a,h)anthracene
Benzo(g,h,i)perylene
4-Chlorophenyl phenyl ether
3,3-Dichlorobenzidine
Benzidine
bis(2-Chloroethyl)ether
1,2-Diphenylhydrazine
Hexachlorocyclopentadiene
N-Nitrosodiphenylamine
N-Nitrosodimethylamine
N-Nitrosodi-n-propylamine
bis(2-Chloroisopropyl)ether
Phenol
2-Nitrophenol
4-Nitrophenol
2,4-Dinitrophenol
4,6-Dinitro-o-cresol
Pentachlorophenol
p-Chloro-m-cresol
2-Chlorophenol
2,4-Dichlorophenol
2,4,6-Trichlorophenol
2,4-Dimethylphenol
α-Endosulfan
β-Endosulfan
Endosulfan sulfate
α-BHC
β-BHC
δ-BHC
γ-BHC
Aldrin
Dieldrin
4,4'-DDE
4,4'-DDD
4,4'-DDT
Endrin
Endrin aldehyde
Heptachlor
Heptachlor epoxide
Chlordane
Toxaphene
Aroclor 1016
Aroclor 1221
Aroclor 1232
Aroclor 1242
Aroclor 1248
Aroclor 1254
Aroclor 1260
2,3,7,8-Tetrachlorodibenzo-dioxin (TCDD)

APPENDIX E

MANUFACTURING AND WASTEWATER TREATMENT SCHEMATICS

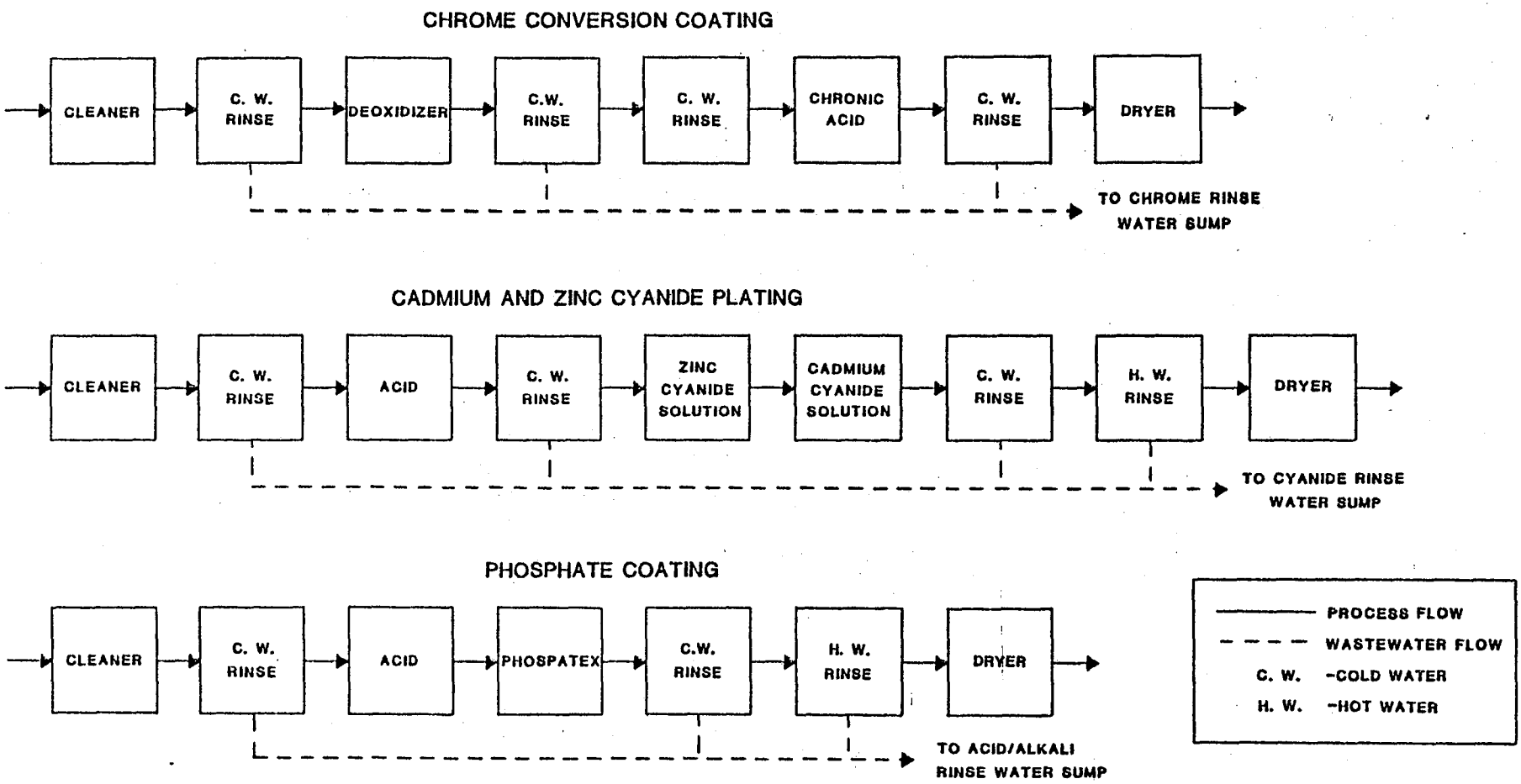


FIGURE E-1 PLATING SHOP METAL FINISHING OPERATIONS, CRANE AAA, SEPT, 1984

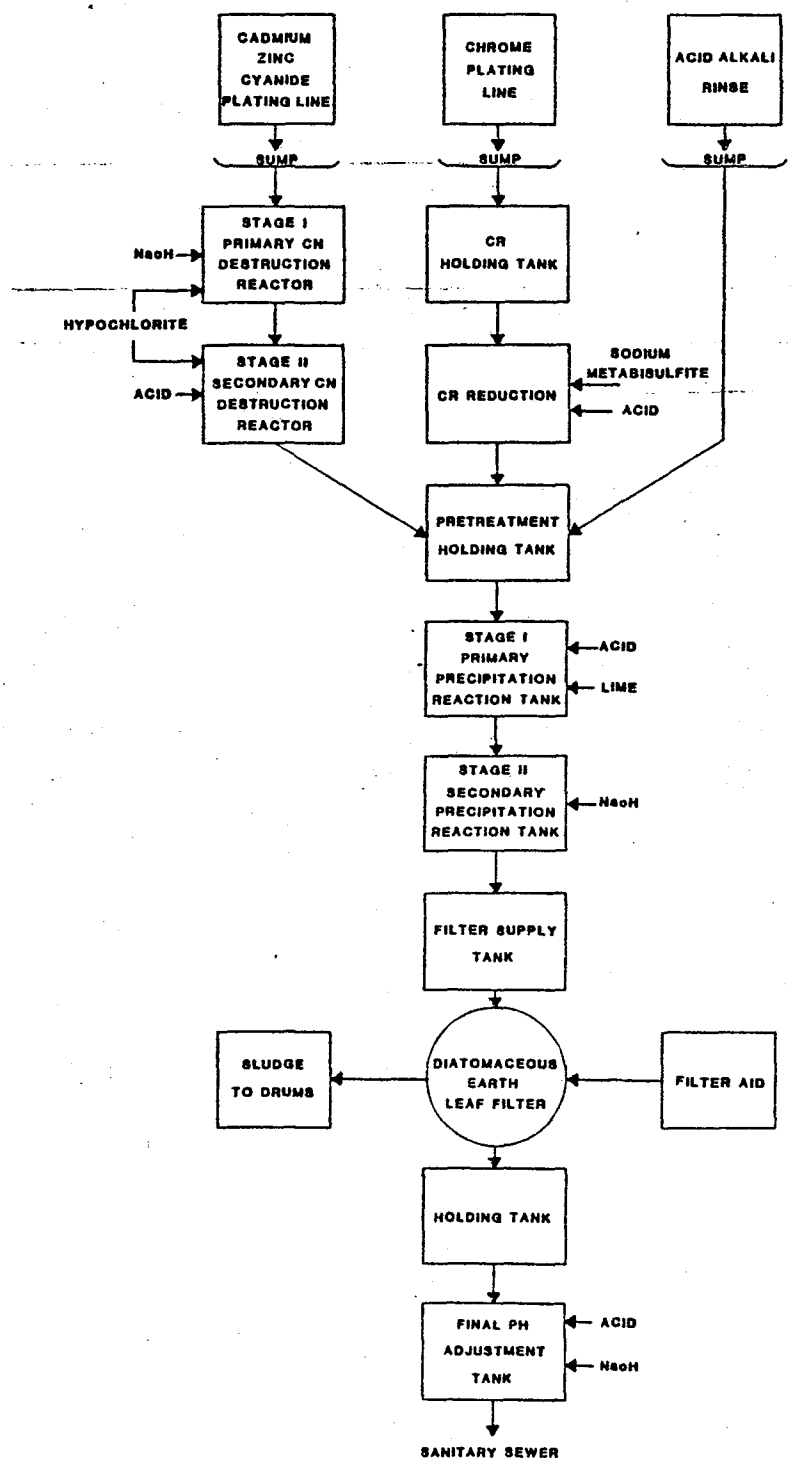


FIGURE E-2 PLATING SHOP WASTEWATER PRETREATMENT FACILITY, CRANE AAA, SEPT 84

22

APPENDIX F

NPDES PERMIT EFFLUENT LIMITATIONS
CRANE NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA
OUTFALL 101
PLATING SHOP WASTEWATER PRETREATMENT FACILITY

Effluent Parameter	Discharge Limitations Daily Average		Daily Maximum	
	lbs/day	(mg/L)	lbs/day	(mg/L)
Oil and Grease	----	(26.0)	----	(52.0)
Total Cadmium	0.05	(0.26)	0.13	(0.69)
Total Zinc	0.28	(1.48)	0.50	(2.61)
Total Cyanide	0.12	(0.65)	0.23	(1.20)
Hexavalent Chromium	0.02	(0.09)	0.05	(0.25)
Total Chromium*	----		0.53	(2.77)
Total Copper*	----		0.38	(2.00)
Total Toxic Organics†	----		0.41	(2.13)
pH (standard units)	in the range of 5 to 11			

* Six-month sampling is required of these parameters. If, after the required sampling period is complete, any of these parameters are demonstrated not to be present in significant amounts, they may be deleted from the permit without Public Notice or opportunity for hearing.
† The limitation for TTO applies to the summation of all quantifiable values greater than 0.01 mg/L for all toxic organics listed under 40 CFR 433.11(e) which are reasonably expected to be present.

APPENDIX G

ANALYTICAL RESULTS

TABLE G-1. ANALYTICAL RESULTS, METALS AND NONMETALS, SAMPLE POINT 1, PLATING SHOP WASTEWATER PRETREATMENT FACILITY EFFLUENT, CRANE AAA, 10-12 SEPTEMBER 1984

Parameter (units in mg/L unless stated otherwise)	10 Sep 84	11 Sep 84	12 Sep 84	Mean
Cadmium	0.045	0.037	0.038	0.040
Chromium	<0.025	<0.025	<0.025	<0.025
Copper	<0.025	<0.025	<0.025	<0.025
Lead	<0.20	<0.20	<0.20	<0.20
Nickel	<0.10	<0.10	<0.10	<0.10
Silver	<0.010	<0.010	<0.010	<0.010
Zinc	<0.015	<0.015	<0.015	<0.015
Cyanide, Total	<0.01	0.03	0.01	<0.017
Cyanide, Amenable	-----	<0.01	-----	<0.01
Total Toxic Organics*†	0.770	0.080	0.280	0.377
Oil and Grease	1.4	3.5	3.5	2.8
Total Suspended Solids	5.1	2.1	2.2	3.1
pH (standard units)	6.9	7.7	7.3	7.3
Flow (gallons)	7,110	9,440	6,660	7,737

* See Appendix D for a breakdown of individual compounds in TTO category.
† See Tables G-2 and G-3 for detection limits of compounds analyzed.

25

TABLE G-2. ANALYTICAL RESULTS, EXTRACTABLE ORGANICS, SAMPLE POINT 1, PLATING SHOP WASTEWATER PRETREATMENT FACILITY EFFLUENT, CRANE AAA, 10-12 SEPTEMBER 1984

Compound (units µg/L)	Detection Limit	10 Sep 84	11 Sep 84	12 Sep 84
Acenaphthene	10	ND	ND	ND
Acenaphthylene	10	ND	ND	ND
Anthracene	10	ND	ND	ND
Benzo(a)anthracene	10	ND	ND	ND
Benzo(a)pyrene	10	ND	ND	ND
Benzo(b)fluoranthene	10	ND	ND	ND
Benzo(ghi)perylene	25	ND	ND	ND
Benzo(k)fluoroanthene	10	ND	ND	ND
Chrysene	10	ND	ND	ND
Dibenzo(a,h)anthracene	25	ND	ND	ND
Fuoranthene	10	ND	ND	ND
Fluoroene	10	ND	ND	ND
Indeno (1, 2, 3-c, d) pyrene	25	ND	ND	ND
Naphthalene	10	ND	ND	ND
Phenanthrene	10	ND	ND	ND
Pyrene	10	ND	ND	ND
Hexachlorocyclopentadiene	10	ND	ND	ND
Hexachlorobenzene	10	ND	ND	ND
Hexachlorobutadiene	10	ND	ND	ND
Hexachloroethane	10	ND	ND	ND
1,2-Dichlorobenzene	10	ND	ND	ND
1,2,4-Trichlorobenzene	10	ND	ND	ND
1,3-Dichlorobenzene	10	ND	ND	ND
1,4-Dichlorobenzene	10	ND	ND	ND
2-Chloronaphthalene	10	ND	ND	ND
Benzidine	10	ND	ND	ND
3,3'-dichlorobenzidine	10	ND	ND	ND
Benzyl butylphthalate	10	ND	ND	ND
Bis(2-ethylhexyl)phthalate	10	500	ND	TR
Di-n-butyl phthalate	10	ND	ND	TR
Di-n-octyl phthalate	10	ND	ND	ND
Diethyl phthalate	10	ND	ND	TR
Dimethyl phthalate	10	ND	ND	ND
N-nitrosodimetylamine	10	ND	ND	ND
N-nitrosodiphenylamine	10	ND	ND	ND
N-nitrosodi-n-propylamine	10	ND	ND	ND
Isophorone	10	ND	ND	ND
Nitrobenzene	10	ND	ND	ND
2,4-Dinitrotoluene	10	ND	ND	ND
2,6-Dinitrotoluene	10	ND	ND	ND
1,2-Diphenylhydrazine	10	ND	ND	ND
Bis(2-chloroetyl) ether	10	ND	ND	ND
Bis(2-chloroethoxy) methane	10	ND	ND	ND
Bis(2-chloroisopropyl) ether	10	ND	ND	ND
4-Bromophenyl phenyl ether	10	ND	ND	ND
4-Chlorophenyl phenyl ether	10	ND	ND	ND

See footnotes, page G-4

Compound (units µg/L)	Detection Limit	10 Sep 84	11 Sep 84	12 Sep 84
4-Chloro-3-methylphenol	25	ND	ND	ND
2-Chlorophenol	25	ND	ND	ND
2,4-Dichlorophenol	25	ND	ND	ND
2,4-Dimethylphenol	25	ND	ND	ND
2,4-Dinitrophenol	250	ND	ND	ND
2-Methyl-4,6-dinitrophenol	250	ND	ND	ND
2-Nitrophenol	25	ND	ND	ND
4-Nitrophenol	25	ND	ND	ND
Pentachlorophenol	25	ND	ND	ND
Phenol	25	ND	ND	ND
2,4,6-trichlorophenol	25	ND	ND	ND
BHC (ALPHA)	20	ND	ND	ND
BHC (BETA)	20	ND	ND	ND
BHC (DELTA)	20	ND	ND	ND
Heptachlor	20	ND	ND	ND
Aldrin	20	ND	ND	ND
Heptachlor Epoxide	20	ND	ND	ND
4,4'-DDE	20	ND	ND	ND
Dieldrin	20	ND	ND	ND
Endrin	20	ND	ND	ND
4,4'-DDD	20	ND	ND	ND
4,4'-DDT	20	ND	ND	ND
Endosulfan sulfate	20	ND	ND	ND
Endosulfan I	20	ND	ND	ND
Endosulfan II	20	ND	ND	ND
Chlordane	20	ND	ND	ND
Toxaphene	500	ND	ND	ND
Endrine Aldehyde	20	ND	ND	ND
PCB 1016	50	ND	ND	ND
PCB 1221	50	ND	ND	ND
PCB 1232	50	ND	ND	ND
PCB 1242	50	ND	ND	ND
PCB 1248	50	ND	ND	ND
PCB 1254	50	ND	ND	ND
PCB 1260	50	ND	ND	ND
Butoxy Ethoxy Ethanol*	--	700-1600	400-1600	500-1500
Hexanedioic Acid-Dioctyl Ester*	--	500-2000	--	--

* Estimated concentration ranges, compounds not TTO compounds.
ND - None detected.
TR - Below detection limit listed, but trace quantity detected.

27

TABLE G-3. ANALYTICAL RESULTS, VOLATILE ORGANICS, SAMPLE POINT 1, PLATING SHOP WASTEWATER PRETREATMENT FACILITY EFFLUENT, CRANE AAA, 10-12 SEPTEMBER 1984

Compound (units µg/L)	Detection Limit	10 Sep 84	11 Sep 84	12 Sep 84
Benzene	3	ND	ND	ND
Bromomethane	3	ND	ND	ND
Bromodichloromethane	3	ND	ND	ND
Bromoform	3	ND	ND	ND
Carbon Tetrachloride	3	ND	ND	ND
Chlorobenzene	3	ND	ND	ND
Chloroethane	3	ND	ND	ND
2-Chloroethylvinyl ether	3	ND	ND	ND
Chloroform	3	270	80	280
Chloromethane	3	ND	ND	ND
Dibromochloromethane	3	ND	ND	ND
1,1-Dichloroethane	3	ND	ND	ND
1,2-Dichloroethane	3	ND	ND	ND
1,1-Dichloroethene	3	ND	ND	ND
1,2-Dichloroethene (Trans)	3	ND	ND	ND
1,2-Dichloropropane	3	ND	ND	ND
1,3-Dichloropropene (Cis)	3	ND	ND	ND
1,3-Dichloropropene (Trans)	3	ND	ND	ND
Ethyl Benzene	3	ND	ND	ND
Methylene Chloride	3	ND	ND	ND
1,1,2,2-Tetrachloroethane	3	ND	ND	ND
Tetrachloroethylene	3	ND	ND	ND
1,1,1-Trichloroethane	3	ND	ND	ND
1,1,2-Trichloroethane	3	ND	ND	ND
Trichloroethylene	3	ND	ND	ND
Trichlorofluoromethane	3	ND	ND	ND
Toluene	3	ND	ND	ND
Vinyl Chloride	3	ND	ND	ND
Acetone*	3	70-200	40-120	40-100

* Estimated concentration range, compounds not TTO compounds.
ND - None detected.

TABLE G-4. ANALYTICAL RESULTS, METALS AND NONMETALS, SAMPLE POINT 3, CYANIDE RINSE WATER SUMP, METAL PLATING SHOP, CRANE AAA, 10-12 SEPTEMBER 1984

Parameter (units in mg/L unless stated otherwise)	10 Sep 84	11 Sep 84	12 Sep 84	Mean
Cadmium	17.1	32.6	24.6	24.77
Chromium	<0.025	<0.025	0.030	<0.027
Copper	0.422	0.753	0.546	0.574
Lead	<0.20	<0.20	<0.20	<0.20
Nickel	<0.10	0.15	<0.10	<0.12
Silver	<0.010	<0.010	<0.020	<0.010
Zinc	0.132	0.313	0.221	0.219
Cyanide, Total	14.0	45.0	42.0	33.7

TABLE G-5. ANALYTICAL RESULTS, METALS AND NONMETALS, SAMPLE POINT 4, ACID/ALKALI RINSE WATER SUMP, METAL PLATING SHOP, CRANE AAA, 10-12 SEPTEMBER 1984

Parameter (units in mg/L unless stated otherwise)	10 Sep 84	11 Sep 84	12 Sep 84	Mean
Cadmium	0.853	1.12	0.142	0.705
Chromium	0.202	0.144	<0.025	<0.124
Copper	0.593	0.277	0.066	0.312
Lead	<0.20	<0.20	<0.20	<0.20
Nickel	<0.10	<0.10	<0.10	<0.10
Silver	<0.010	<0.010	<0.010	<0.010
Zinc	39.9	9.34	6.31	18.52
Cyanide, Total	0.03	0.03	0.01	0.02

TABLE G-6. ANALYTICAL RESULTS, EXTRACTABLE ORGANICS, SAMPLE POINT 6, MACHINE COOLANT, MACHINE SHOP, CRANE AAA, 10-12 SEPTEMBER 1984

Compound (units µg/L)	Detection Limit	Machine No. 215 (sample point 6)
Acenaphthene	10	ND
Acenaphthylene	10	ND
Anthracene	10	ND
Benzo(a)anthracene	10	ND
Benzo(a)pyrene	10	ND
Benzo(b)fluoranthene	10	ND
Benzo(ghi)perylene	25	ND
Benzo(k)fluoroanthene	10	ND
Chrysene	10	ND
Dibenzo(a,h)anthracene	25	ND
Fuoranthene	10	ND
Fluoroene	10	ND
Indeno (1, 2, 3-c, d) pyrene	25	ND
Naphthalene	10	ND
Phenanthrene	10	ND
Pyrene	10	ND
Hexachlorocyclopentadiene	10	ND
Hexachlorobenzene	10	ND
Hexachlorobutadiene	10	ND
Hexachloroethane	10	ND
1,2-Dichlorobenzene	10	ND
1,2,4-Trichlorobenzene	10	ND
1,3-Dichlorobenzene	10	ND
1,4-Dichlorobenzene	10	ND
2-Chloronaphthalene	10	ND
Benzidine	10	ND
3,3'-dichlorobenzidine	10	ND
Benzyl butyl phthalate	10	300
Bis(2-ethylhexyl) phthalate	10	140
Di-n-butyl phthalate	10	ND
Di-n-octyl phthalate	10	ND
Diethyl phthalate	10	10
Dimethyl phthalate	10	ND
N-nitrosodimetylamine	10	ND
N-nitrosodiphenylamine	10	ND
N-nitrosodi-n-propylamine	10	ND
Isophorone	10	ND
Nitrobenzene	10	ND
2,4-Dinitrotoluene	10	ND
2,6-Dinitrotoluene	10	ND
1,2-Diphenylhydrazine	10	ND
Bis(2-chloroethyl) ether	10	ND
Bis(2-chloroethoxy) methane	10	ND
Bis(2-chloroisopropyl) ether	10	ND
4-Bromophenyl phenyl ether	10	ND
4-Chlorophenyl phenyl ether	10	ND

See footnotes, page G-8

Compound (units µg/L)	Detection Limit	Machine No. 215 (sample point 6)
4-Chloro-3-methylphenol	25	ND
2-Chlorophenol	25	ND
2,4-Dichlorophenol	25	ND
2,4-Dimethylphenol	25	ND
2,4-Dinitrophenol	250	ND
2-Methyl-4,6-dinitrophenol	250	ND
2-Nitrophenol	25	ND
4-Nitrophenol	25	ND
Pentachlorophenol	25	ND
Phenol	25	ND
2,4,6-trichlorophenol	25	ND
BHC (ALPHA)	20	ND
BHC (BETA)	20	ND
BHC (GAMMA)	20	ND
BHC (DELTA)	20	ND
Heptachlor	20	ND
Aldrin	20	ND
Heptachlor Epoxide	20	ND
4,4'-DDE	20	ND
Dieldrin	20	ND
Endrin	20	ND
4,4'-DDD	20	ND
4,4'-DDT	20	ND
Endosulfan sulfate	20	ND
Endosulfan I	20	ND
Endosulfan II	20	ND
Chlordane	20	ND
Toxaphene	500	ND
Endrine Aldehyde	20	ND
PCB 1016	50	ND
PCB 1221	50	ND
PCB 1232	50	ND
PCB 1242	50	ND
PCB 1248	50	ND
PCB 1254	50	ND
PCB 1260	50	ND
Methyl 2,4-Pentanediol*	--	25,000-50,000
2-Propanol, 1-(2-<2-Methoxy-1-Methylethoxy)-1-Methylethoxy>*	--	20,000-40,000
Unidentified Compounds*	--	10,000-30,000
Hydrocarbons (C - C)*	--	1,000-3,000

* Estimated concentration range, compounds not TTO compounds.
ND - None detected.

31

TABLE G-7. ANALYTICAL RESULTS, VOLATILE ORGANICS, SAMPLE POINTS 5 AND 6, MACHINE COOLANT, MACHINE SHOP, CRANE AAA, 10 SEPTEMBER 1984

Compound (units µg/L)	Detection* Limit	Machine No. 176 (sample point 5)	Machine No. 215 (sample point 6)
Benzene	30	ND	ND
Bromomethane	30	ND	ND
Bromodichloromethane	30	ND	ND
Bromoform	30	ND	ND
Carbon Tetrachloride	30	ND	ND
Chlorobenzene	30	ND	ND
Chloroethane	30	ND	ND
2-Chloroethylvinyl ether	30	ND	ND
Chloroform	30	160	70
Chloromethane	30	ND	ND
Dibromochloromethane	30	ND	ND
1,1-Dichloroethane	30	ND	ND
1,2-Dichloroethane	30	ND	ND
1,1-Dichloroethene	30	ND	ND
1,2-Dichloroethene (Trans)	30	ND	ND
1,2-Dichloropropane	30	ND	ND
1,3-Dichloropropene (Cis)	30	ND	ND
1,3-Dichloropropene (Trans)	30	ND	ND
Ethyl Benzene	30	ND	ND
Methylene Chloride	30	ND	ND
1,1,2,2-Tetrachloroethane	30	ND	ND
Tetrachloroethylene	30	ND	ND
1,1,1-Trichloroethane	30	2500	ND
1,1,2-Trichloroethane	30	ND	ND
Trichloroethylene	30	ND	ND
Trichlorofluoromethane	30	ND	ND
Toluene	30	360	ND
Vinyl Chloride	30	ND	ND
Acetone†	30	400-600	----
Dimethyl hexane†	30	200-400	----
Methyl methylethenylcyclohexene	30	200-400	----
1-Propoxy-2-propanol†	30	3000-6000	----

* Detection limit increased from 3 to 30; sample had to be diluted 1.10.
ND - None detected.
† Estimated concentration range, compounds not TTO compounds.

B-1884 0924

0924-CCA:ae
11346

4 JUN 1984

From: Commanding Officer, Naval Weapons Support Center
To: Commander, Crane Army Ammunition Activity (SMCCN-IOS)

Subj: Disposal of Electroplating Baths

Encl: (1) Pending Permit Limitations for Site 101-Pretreatment Facility (Building 1884)

1. All chemical bath solutions used in electroplating operations shall be handled as hazardous waste and properly containerized and disposed via 0924. No concentrated chemical bath solution is to be batch treated at the pretreatment facility in Building 3064. Only finewater is to be channeled there for processing. This treatment facility was originally designed to handle wastewater not concentrated chemical solutions. There is an acute overload on the system which must be remedied immediately in order to be in compliance with new limitations (enclosure (1)) established for our discharge from that plant and to prolong the life of this facility.

2. In order to comply with Hazardous Waste and Clean Water Act regulations, 0924 must be kept informed of all new chemicals used in the plating or cleaning process. Chemical data sheets and/or information on composition of all new chemical formulations proposed for use in the operations at 1884 shall be provided 0924 prior to their use. All operating procedures shall be changed accordingly and submitted for review.

3. NAVWPNSUPPCEN Crane point of contact is Mrs. Cathy Andrews, 0924, extension 3114/1132.

J. D. FARIS
By direction

Copy to:
SMCCN-EDP

Blind copy to:
Jim Market
Ben Mol Corp. (Ben Molayem)

DEPARTMENT OF THE NAVY
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA 47522

IN REPLY REFER TO:
0924-CCA:ae
11346

4 JUN 1984

From: Commanding Officer, Naval Weapons Support Center
To: Commander, Crane Army Ammunition Activity (SMCCN-IOS)

Subj: Disposal of Electroplating Baths

Encl: (1) Pending Permit Limitations for Site 101-Pretreatment Facility (Building 1884)

1. All chemical bath solutions used in electroplating operations shall be handled as hazardous waste and properly containerized and disposed via 0924. No concentrated chemical bath solution is to be batch treated at the pretreatment facility in Building 3064. Only rinsewater is to be channeled there for processing. This treatment facility was originally designed to handle wastewater not concentrated chemical solutions. There is an acute overload on the system which must be remedied immediately in order to be in compliance with new limitations (enclosure (1)) established for our discharge from that plant and to prolong the life of this facility.

2. In order to comply with Hazardous Waste and Clean Water Act regulations, 0924 must be kept informed of all new chemicals used in the plating or cleaning process. Chemical data sheets and/or information on composition of all new chemical formulations proposed for use in the operations at 1884 shall be provided 0924 prior to their use. All operating procedures shall be changed accordingly and submitted for review.

3. NAVWPNSUPPCEN Crane point of contact is Mrs. Cathy Andrews, 0924, extension 3114/1132.

J. D. Faris

J. D. FARIS
By direction

Copy to:
SMCCN-EDP

REPRODUCED AT GOVERNMENT EXPENSE

2. During the period beginning on the effective date of this permit and lasting until the expiration date, the permittee is authorized to discharge from outfall(s) 101 - Pretreatment Facility (Building 1884). Such discharge shall be limited and monitored by the permittee as specified below:

Discharge Limitations

Effluent Characteristic	kg/day (lbs/day) Daily Average	Daily Maximum	Other Limitations Daily Average	Daily Maximum	Monitoring Requirement Measurement Frequency	Sample Type
Flow (MGD)	--	--	--	--	Measure When Sampling	
Oil & Grease	--	--	26 mg/l	52 mg/l	2 X Weekly	Grab
T. Cadmium	.07 (.15)	.18 (.40)	.26 mg/l	.69 mg/l	2 X Weekly	24 Hr. Comp.
T. Zinc	.39 (.85)	.68 (1.5)	1.48 mg/l	2.61 mgl	Weekly	24 Hr. Comp.
T. Cyanide	.17 (.37)	.31 (.69)	.65 mg/l	1.2 mg/l	2 X Weekly	24 Hr. Comp.
Hex Chromium	.02 (.05)	.07 (.14)	.09 mg/l	.25 mg/l	Weekly	24 Hr. Comp.
T. Chromium*	--	.73 (1.60)	--	2.77 mg/l	Monthly	24 Hr. Comp.
T. Copper*	--	.17 (.38)	--	2.0 mg/l	2 X Monthly	24 Hr. Comp.
Total Toxic Organics**	--	.56 (1.23)	--	2.13 mg/l	Monthly***	Grab

*The above noted parameter(s) are to be monitored for 6 months to determine whether or not they are present in significant quantities. At the end of this sampling period, the permittee may request, in writing, a review of these requirements. Upon review by the ISPCB, monitoring requirements may be deleted, if appropriate, without public notice or opportunity of hearing. Alternatively, the permit may be modified, after public notice and opportunity for hearing, to include appropriate effluent limitations.

**The limitation for TTO applies to the summation of all quantifiable values greater than 0.01 mg/l for all toxic organics listed under 40 CFR 433.11(e) which are reasonably expected to be present.

***In lieu of monthly monitoring for TTO, the party responsible for signing the monthly discharge monitoring report (DMR) forms may make the following statement, as part of the DMR; "Based on my inquiry of the persons directly responsible for managing compliance with the permit limitations for TTO, I certify that, to the best of my knowledge and belief, no dumping of concentrated toxic organics into the wastewaters has occurred since filing of the last discharge monitoring report. I further certify that this facility is implementing the solvent management plan submitted to the Indiana Stream Pollution Control Board, as required by this Permit." See Additional Requirements, Part I.C. of this Permit.

REPRODUCED AT GOVERNMENT EXPENSE

a. The pH shall not be less than 5.0 nor greater than 11.0. The pH shall be monitored as follows: by a weekly grab sample.

b. Samples taken in compliance with the monitoring requirements above shall be taken at a point representative of the discharge but prior to entry into the sanitary sewer.

REPRODUCED AT GOVERNMENT EXPENSE

OFFICIAL USE ONLY

Return By_____

Inspector_____

STATE OF INDIANA
AIR POLLUTION CONTROL BOARD
OPERATION PERMIT APPLICATION

General Information

Company Name Naval Weapons Support Center

Plant Location Bldg 1884 | Crane | Martin | 47522
Street Address | City | County | Zip Code

Mailing Address for plant if Different than Above:

Bldg 2516, Code 092 | Crane | Indiana | 47522
Street Address | City | State | Zip Code

Person to Contact on Matters of Air Pollution Control:

Name J. D. FARIS Title Manager, Engr Div Phone (812) 854-1480

If you have changed company name or location in the past 2 years, please list the old name(s) below:

Name_____

Location_____

Standard Industrial Classification Plating Code 3479, Degreasing Code 3479
(If you don't know this number, give a short description of your business.)

Type of Permit Requested:

_____Renewal X Operation _____Change of Ownership _____Relocation

If Renewal, Has There been Any Change? _____Yes _____No

____Private ____Utility Government (Local)_____ State____ Federal X)

Year for which information is submitted 19 79 .

I hereby certify that the information submitted this____day of _____19___ is true and accurate to the best of my knowledge.

Signature J K Swistock

Title J. R. SWISTOCK
~~CDR. CEC. USN~~
Public Works Officer

OFFICIAL USE ONLY

Return By_____

Inspector_____

STATE OF INDIANA
AIR POLLUTION CONTROL BOARD
OPERATION PERMIT APPLICATION

General Information

Company Name Naval Weapons Support Center

Plant Location

Street Address	City	County	Zip Code
Bldg 1884	Crane	Martin	47522

Mailing Address for plant if Different than Above:

Street Address	City	State	Zip Code
Bldg 2516, Code 092	Crane	Indiana	47522

Person to Contact on Matters of Air Pollution Control:

Name J. D. FARIS Title Manager, Engr Div Phone (812) 854-1480

If you have changed company name or location in the past 2 years, please list the old name(s) below:

Name_____

Location_____

Standard Industrial Classification Plating Code 3479, Degreasing Code 3479
(If you don't know this number, give a short description of your business.)

Type of Permit Requested:

_____Renewal X Operation _____Change of Ownership _____Relocation

If Renewal, Has There been Any Change? _____Yes _____No

____Private ____Utility Government (Local)_____ State____ Federal X)

Year for which information is submitted 19 79 .

I hereby certify that the information submitted this____day of _____19___
is true and accurate to the best of my knowledge.

Signature_____

Title_____

STATE OF INDIANA
Air Pollution Control Board

E

Process Information

Company Name Naval Weapons Support Center, Crane

Products produced Small metal parts are electroplated with zink and cadmium. Some are iridite coated.

Raw Materials (list each type) lbs/hr under normal operation

Small Metal Parts - 120 lbs/hr HCL - 17.35 lbs/hr

Nitric Acid - 1.51 lbs/hr

Sodium Hydroxide - 3.85 lbs/hr

Chromic Acid - .34 lbs/hr

Zink / Cadmium — Sodium Cyanide - 2.16 lbs/hr

Operating schedule

8 **hours/day** 5 **days/week** 52 **weeks/year**

Control Equipment

	STACK #1*	STACK #2*
Type of control	Exhaust	Exhaust
Efficiency %	-	-
Stack height (feet above ground)	19' 10"	19' 10"
Stack Diameter (inches)	24"	20"
Exit temperature °F	70° or Ambient	70° or Ambient
Gas flow rate (cfm)	6,600 CFM	4,400 CFM

Process Weight Rate _____ **lb/hr** _____ **tons/yr**

*Stacks are numbered north to south

STATE OF INDIANA
Air Pollution Control Board

E

Process Information

Company Name Naval Weapons Support Center, Crane

Products produced Same as Stacks #1 and #2

Raw Materials (list each type) lbs/hr under normal operation

Same as Stacks #1 and #2

Operating schedule

8 **hours/day** 5 **days/week** 52 **weeks/year**

Control Equipment

	STACK #3*	STACK #4*
Type of control	Exhaust	Exhaust
Efficiency %	-	-
Stack height (feet above ground)	19' 10"	19' 10"
Stack Diameter (inches)	30"	24"
Exit temperature °F	70° or Ambient	70° or Ambient
Gas flow rate (cfm)	8,850 CFM	6,720 CFM

Process Weight Rate _____ **lb/hr** _____ **tons/yr**

*Stacks are numbered north to south

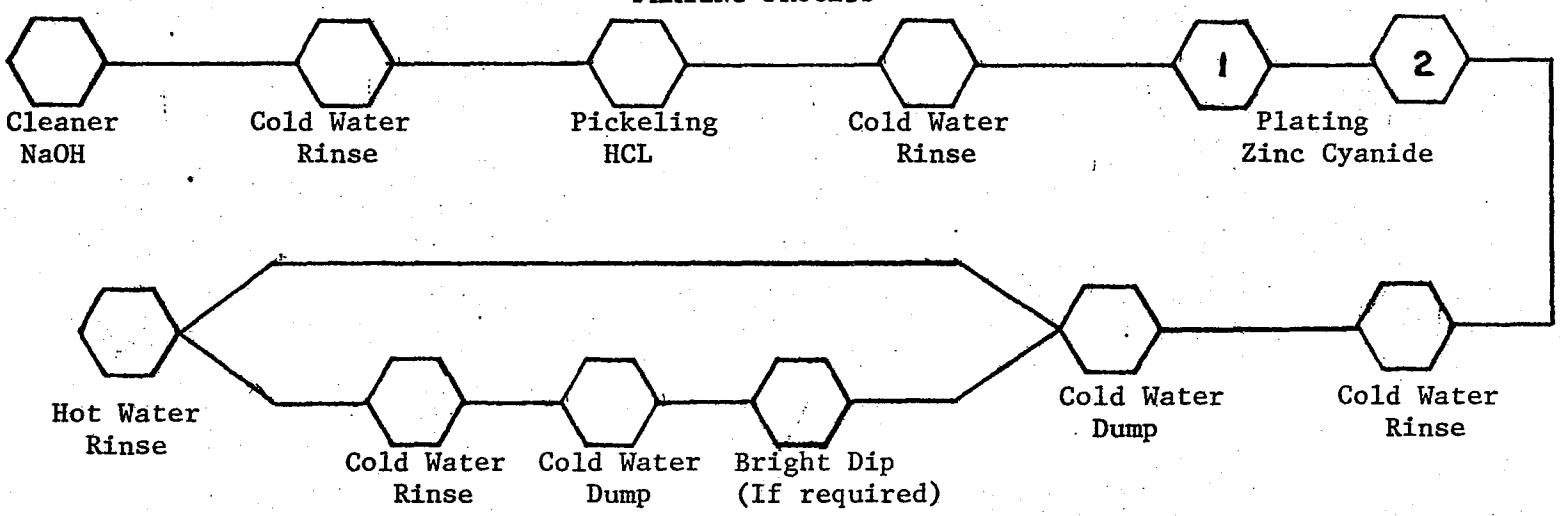
STATE OF INDIANA
Air Pollution Control Board

Company Name Naval Weapons Support Center, Crane

Flow Diagram

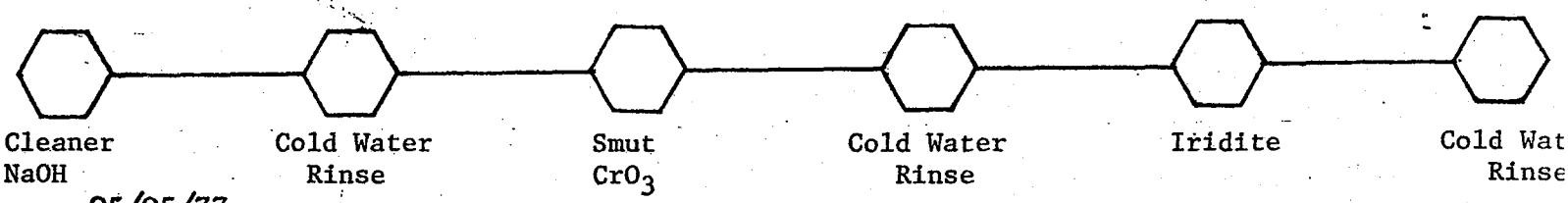
Show a simple sketch of your operation from raw materials coming in to finished products. Show points of emissions including stacks. If there is any air pollution control equipment, show its locations, what process it is controlling, control equipment code, and removal efficiency. Include an hourly rate of material flow throughout the process.

PLATING PROCESS



Cadmium Plating Process is identical to the above process except that only one tank is required for the plating step.

IRIDITE PROCESS



05/05/77

L

STATE OF INDIANA
Air Pollution Control Board

Degreasing

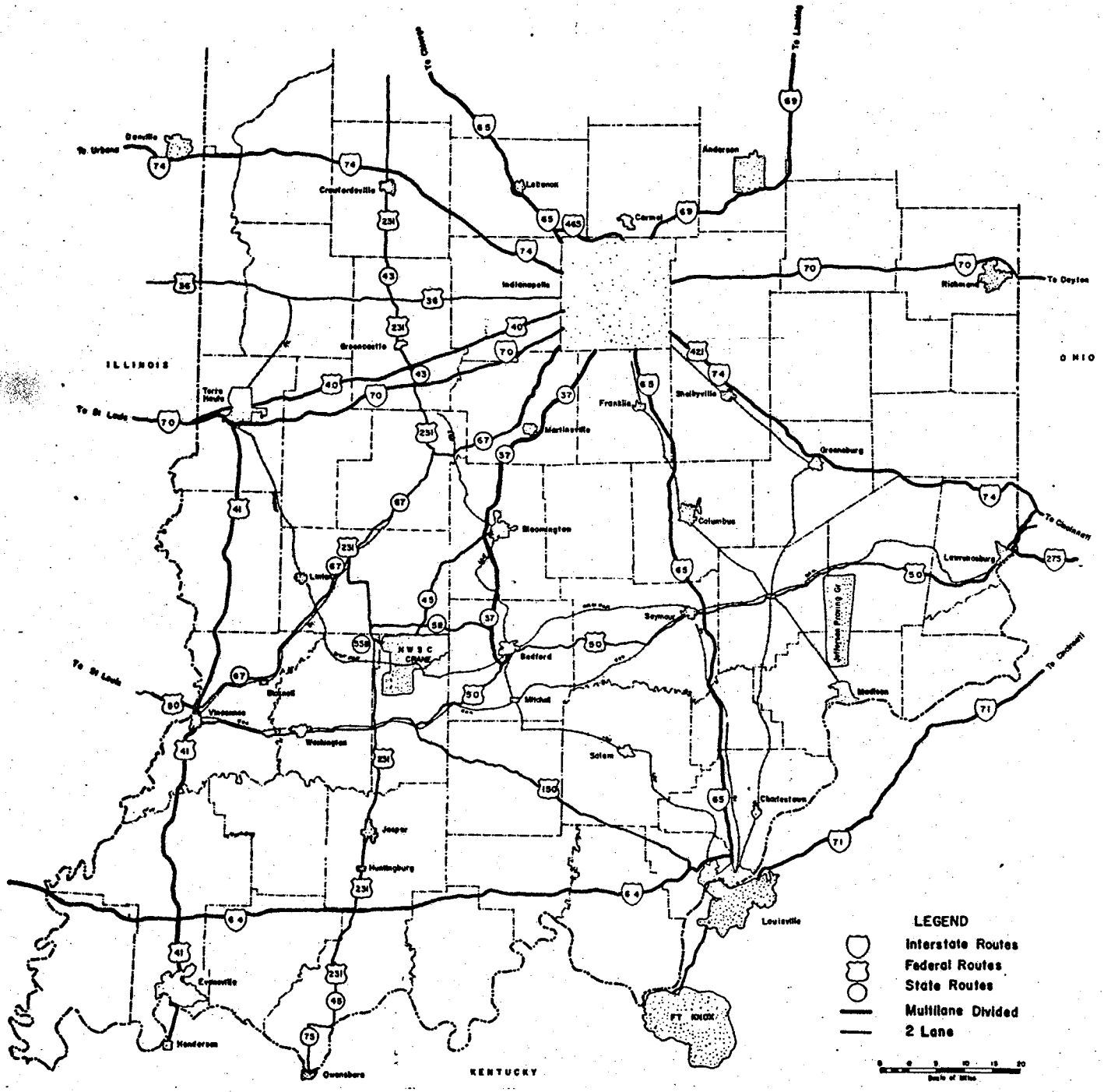
Company Name Naval Weapons Support Center, Crane, Indiana

Solvent used	Density (lb/gal)	Usage gal/hr	Usage gal/yr	Control Device & Efficiency	Amount Sold or Disposed of gal/yr
Chlorothene VG	12 lb/gal	1.25	2600	Exhaust	-

Degreasing schedule 8 hours/day 5 days/week 52 weeks/year

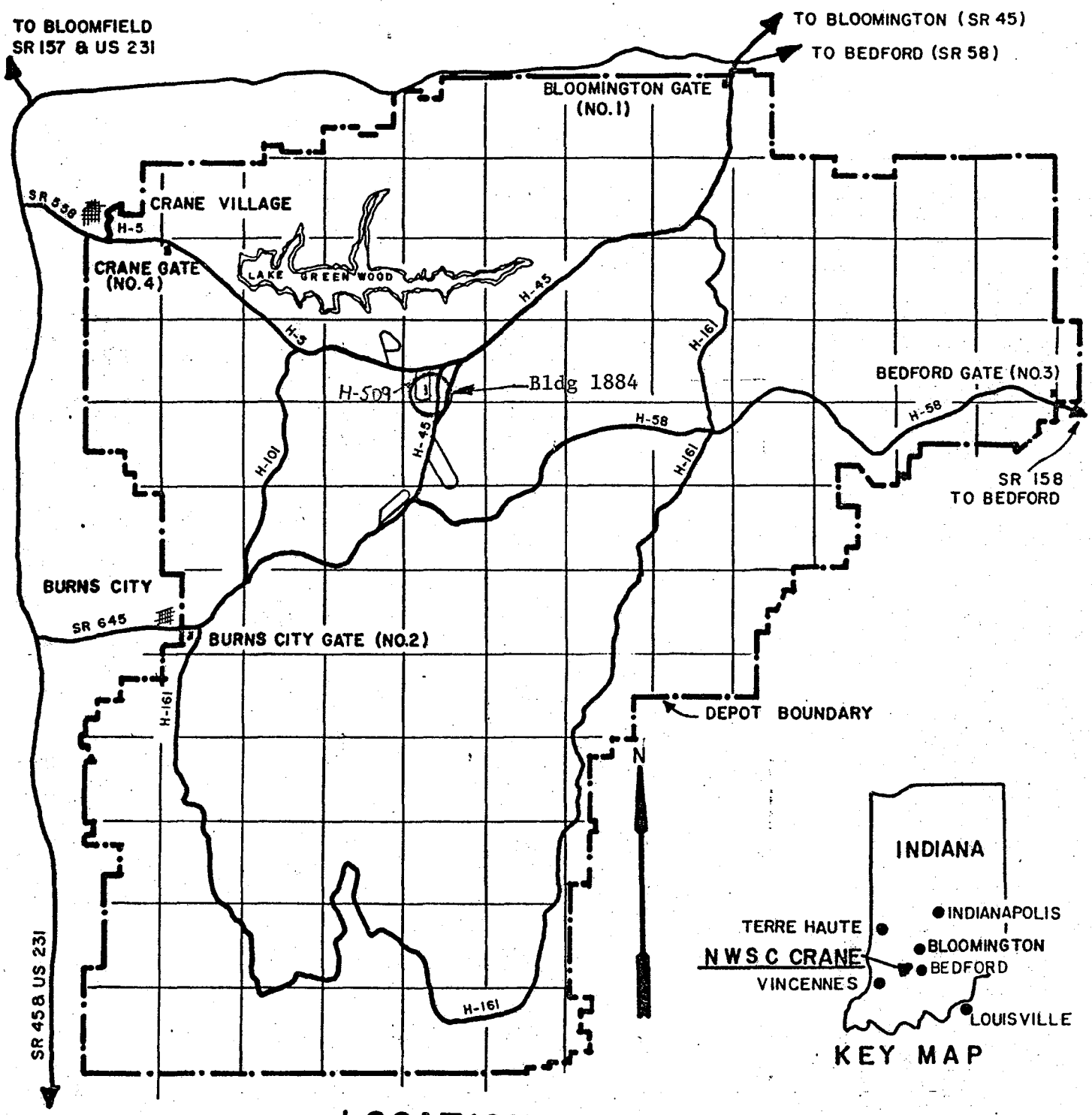
Composition (by chemical names not brand names) of the solvent(s) by volume percent is required. Attach separate sheet(s) with this breakdown. If you don't have this information contact your supplier.

STATE OF INDIANA
AIR POLLUTION CONTROL BOARD



VICINITY MAP
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA

STATE OF INDIANA
AIR POLLUTION CONTROL BOARD



LOCATION PLAN

SCALE = GRID LINES FORM MILE SQUARES

Please file in
Let me know
where your
filed [illegible]

SARCN-EN (18 Sep 81) 1st Ind Mr. Martis/art/AUTOVON 482-1402
SUBJECT: Survey of DARCOM Installations Engaged in Metal Plating Operations

DA, Crane Army Ammunition Activity, Crane, IN 47522 3 November 1981

TO: Commander, US Army Armament Materiel Readiness Command, ATTN: DRSAR-ISE, Rock Island, Illinois 61299

Survey response requested is forwarded.

FOR THE COMMANDER:

ORIGINAL SIGNED

1 Incl
as

JOHN G. MARTIS
Chief Engineer

CF:
DRDME-GS (ATTN: G. Chesler)
NWSC-092V
SARCN-RMB (w/o Incl)

PLATING WASTES SURVEY

Return to: Commander
USAMERADCOM
DRDME-GS (G. Chesler)
Ft. Belvoir, VA 22060

Please return by 8 October 1981.

1.0 PLATING ESTABLISHMENT DATA

Installation CRANE ARMY AMMUNITION ACTIVITY

Address Crane, IN 47522

Plating Facility Supervisor Mr. Kenneth Hack

Telephone: AV 482-1677

Commercial (812) 854-1677

Industrial Wastewater Treatment Facility Supervisor Mr. David Turner

Telephone: AV CONTRACTOR ("Turner Technology")

Commercial (219) 267-3305

Environmental Officer Ms. Catherine C. Andrews

Telephone: AV 482-3114

Commercial (812) 854-3114

Attach copy of the plant's current effluent requirements document. Also indicate whether city ___, town ___, county ___, state X, federal ___. Do these requirements apply to industrial wastewater treatment facility only X or to effluent when combined with domestic wastewater treatment facility effluent ___? SEE ATTACHMENT C.

Total number of plating lines in facility 3

Types of parts and/or products plated Electronic chasis and pyrotechnic components, small parts up to 2 foot square steel, stainless steel and aluminum.

Incl 1

Principal raw materials used by the plating shop (metals, proprietary solutions, etc.). List approximate quantities in amounts per year.

Sodium Cyanide	- 4,500 lbs	Hydrochloric Acid	- 35,000 lbs
Nitric Acid	- 3,000 lbs	Sodium Hydroxide	- 8,000 lbs
Zinc	- 4,500 lbs	Cadmium	- 4,500 lbs
Chromic Acid	- 700 lbs		
Alkali Cleaners	- Small Metal Parts	- 240,000 lbs	

2.0 SPECIFIC PLATING PROCESSES EMPLOYED

Check ( ) processes employed in the plant

Electroplating:

1. ___ Copper
2. ___ Nickel
3. ___ Chromium
4. X Zinc
5. X Cadmium
6. ___ Tin
7. ___ Solder
8. ___ Silver
9. ___ Gold
10. ___ Platinum
11. ___ Iron
12. ___ Anodizing
13. X Etching
14. ___ Milling
15. X Chromating
16. X Phosphating
17. ___ Immersion Plating

Electroless plating on plastics:

18. ___ Copper
19. ___ Nickel
20. ___ Gold
21. ___ Other, specify _____

Electroless plating on metals (indicate metal plated and base metal):

22.	Steel	Zinc	24.	Stainless Steel	Phosphating
23.	Steel	Cadmium	25.	Aluminum	Iriditing

Others:

26. ___ Electrochemical machining
27. X Polishing (electro or other)
28. ___ Electropainting
29. ___ Nonaqueous plating
30. X Coloring
31. ___ Other electrochemical processes
32. ___ Lead electroplating
33. X Bright dip
34. ___ Printed circuits

How was your work spread out among the categories of the above processes last year? (For example, "90% of the work done in this shop was zinc electroplating, 5% was cadmium electroplating and the rest was electroless nickel plating.") 45% Cadmium, 25% Zinc, 10% Chromating of Aluminum, 10% Bright Dipping, 10% Phosphating.

3.0 COMPLEXING AGENTS

Complexing agents are used in many electroless plating processes. Examples of these agents include EDTA, NTA, lactic acid and thiourea. Are you using complexing agents? NO If yes, what is the effect of complexing agents on your waste treatment process?

4.0 WASTEWATER TREATMENT

4.1 Water Usage in Plating Shop

State the average gal/day used during plating plant operation

tap water 4,000, recycled water 40,000,

and total water 44,000.

4.2 Waste Streams from the Plating Process

Waste Streams from the Plating Process	Average gal/day
Cyanide bearing wastewater	14,500
Chromium bearing wastewater	7,200
All other wastewater (other rinses, etc.)	22,300
Total wastewater flow from the plating shop into the industrial waste treatment facility	44,000

Describe your method of dealing with concentrated batch dumps. For example, how are baths dumped when necessary? Pumped to holding tank and slowly bled into continuous system.

Are your chromium and cyanide waste streams segregated from other waste streams as listed above? Describe. Cyanide baths and cyanide rinse tanks have curbs to contain volume of all tanks. Separate rinse and floor spill wastewater segregation of cyanide, chromium, and acid/alkali wastes exist.

4.3 In addition to plating shop wastewaters, are there other streams which contribute to the total volume of industrial wastewater requiring treatment? NO. If yes, please list contributing industrial operations and approximate volumes, if known.

4.4 Is the industrial wastewater treatment system working satisfactorily? Yes or no. YES If not, please describe any problems.

4.5 What changes, if any, are planned for your industrial wastewater treatment system? None, at present. Possible construction of holding tank before discharge into sanitary sewer is being considered.

4.6 Comment on Page A which follows regarding plating shop operations which have impact on the wastewater treatment process. For example, what rinsewater flow controls are installed? Do they accomplish their mission? Any other comments regarding this area of concern are welcome.

4.7 Waste Treatment Cost Information (conventional treatment)

(designations A, B, and C are used below in case you have more than one treatment system)

	Treatment System Name	Date Installed	Capital Costs	Operating Costs ($/yr)	Raw Waste Streams Treated (see par. 4.2)	Design Flow Level (gal/day)
A	B-3064 Plating Shop Waste Treatment	11/79	$400,000	$110,000	(4.2)	36,000/24 hours
B						
C						

4.8 Waste Treatment Cost Information (for any recycle streams in the plating shop)

	Treatment System Name	Date Installed	Capital Costs	Operating Costs ($/yr)	Raw Waste Streams Treated (see par. 4.2)	Design Flow Level (gal/day)
A	Included in design of above system.					
B						

4.9 Include below any other cost information applicable to this survey.

N/A

Comments (4.6) about plating shop operations which have impact on wastewater treatment.

A number of plating shop modifications were implemented with the waste treatment system to minimize both waste volume and loadings. They are summarized as follows:

1. Counterflow rinses were installed where possible, i.e., acid rinses, counterflow to cleaner rinses and two stage rinses after plating baths.

2. Cyanide, chromium and acid/alkali wastes are segregated in rinses, floor spills and batch dumps.

3. Non-chelated soak and electrocleaners are used in all lines.

4. Zinc cyanide bath formulation was changed to a "low to mid range" cyanide concentration.

5. All lines are "hand-operated". The operators have been instructed on drip techniques to minimize drag-out.

6. A large portion (approaching 90%) of the treated wastewater is reused in the plating shop in "non-critical" rinses, i.e., acid and cleaner rinses.

7. All rinse tanks have flow control valves to limit the maximum flow of water.

8. All fresh water rinses use conductivity controllers to regulate the supply of fresh water to the rinse tanks.

9. All reuse water rinses are manually turned off/on as needed in the process.

See Attachment A "Plating Process Schematic".

4.10 From information available to you, please furnish average chemical analyses of effluents from (a) plating shop, (b) industrial wastewater treatment plant, (c) installation sewer discharge. This description should be in terms of 1)the plating metals, 2)flow, 3)pH, 4)temperature, 5)any other parameters listed in your effluent requirements. Please attach a description or write one on page B which follows.

5.0 SCHEMATIC OF PLATING OR COMBINED INDUSTRIAL WASTE TREATMENT SYSTEM

Draw your schematic using descriptors listed below or others which describe processes used in your system. Use page C for the schematic.

General description

01 Batch process
02 Continuous process
03 Integrated process
04 Other _____ (specify)

Sludge Dewatering

40 Thickening
41 Centrifugation
42 Vacuum Filtration
43 Pressure Filtration
44 Other _____ (specify)

Sludge Disposal

50 Lagooning
51 Land Fill
52 Incineration
53 Contractor Removal
54 Other _____ (specify)

In-Plant Processes

10 Reverse Osmosis
11 Ion Exchange
12 Evaporation
13 Electrodialysis
14 Pfaudler
15 Lancey
16 Innova
17 Wastesaver
18 Ultrafiltration
19 Other _____ (specify)

End-of-Pipe Processes

20 Screening
21 Emulsion Breaking
22 Skimming
23 Chemical Oxidation (incl cyanide)
24 Chemical Reduction (incl chromium)
25 Neutralization
26 Chemical Precipitation
27 Flotation
28 Lagooning (for solids settling)
29 Clarification (with addition of coagulants)
30 Filtration
31 Ion Exchange
32 Reverse Osmosis
33 Adsorption
34 Evaporation (distillation)
35 Chlorination
36 Lagooning (for biological decomposition)
37 Other _____ (specify)

Oil Disposal

60 Incineration/Combustion
61 Processing for Reuse
62 Contractor Removal
63 Other _____ (specify)

Water Discharge

70 Sanitary Sewer
71 Stream/River
72 Lake/Pond
73 Deep Well
74 Leach Field
75 Processing for Reuse
76 Other _____ (specify)

note: Circle if the following are discharged into your industrial waste treatment facility: 80 Sanitary Wastes, 81 Noncontact cooling water, 82 Boiler Blowdown

Comments (4.10) which furnish effluent data on the plating shop, the industrial wastewater treatment system, the sewer discharge.

See Attachment B, "Sept 1981 Monthly Monitoring Report for NPDES Permit IN 0021539 Outfall 101".

See Attachment C, "Permit No. IN 0021539".

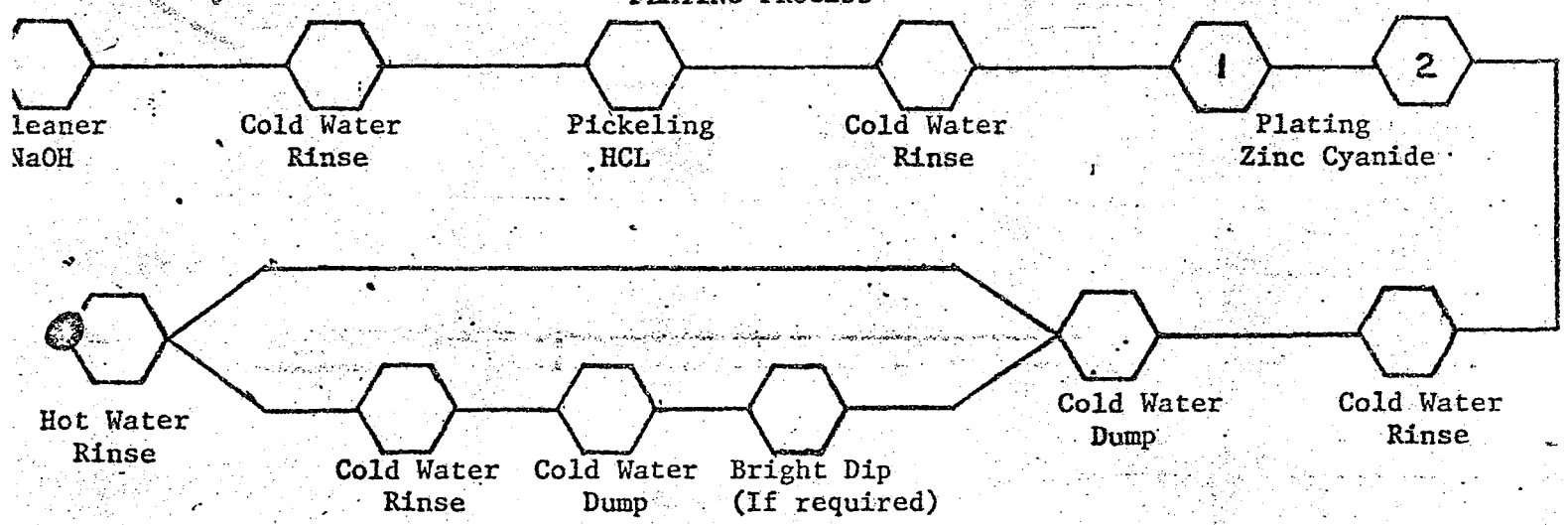
Comments (5.0) Industrial Wastewater Treatment System Schematic

See Attachment D, "Operation of Chemical Treatment Plant", partial Section 00005.

See Attachment E, Photo, Schematic of Treatment Facility.

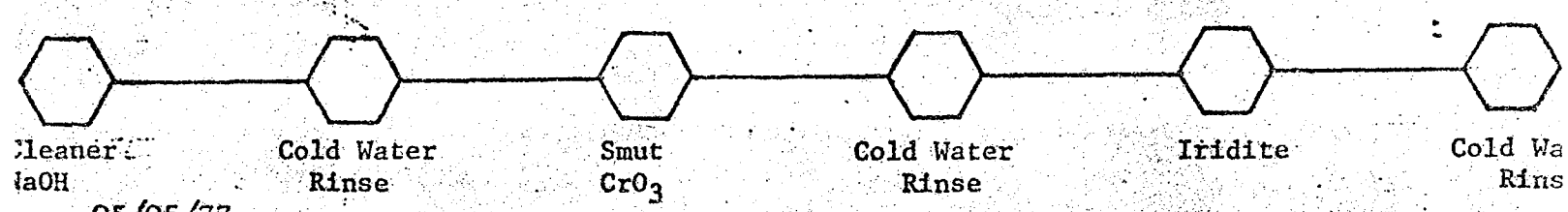
ATTACHMENT A

PLATING PROCESS



admium Plating Process is identical to the above process except that only one tank is required or the plating step.

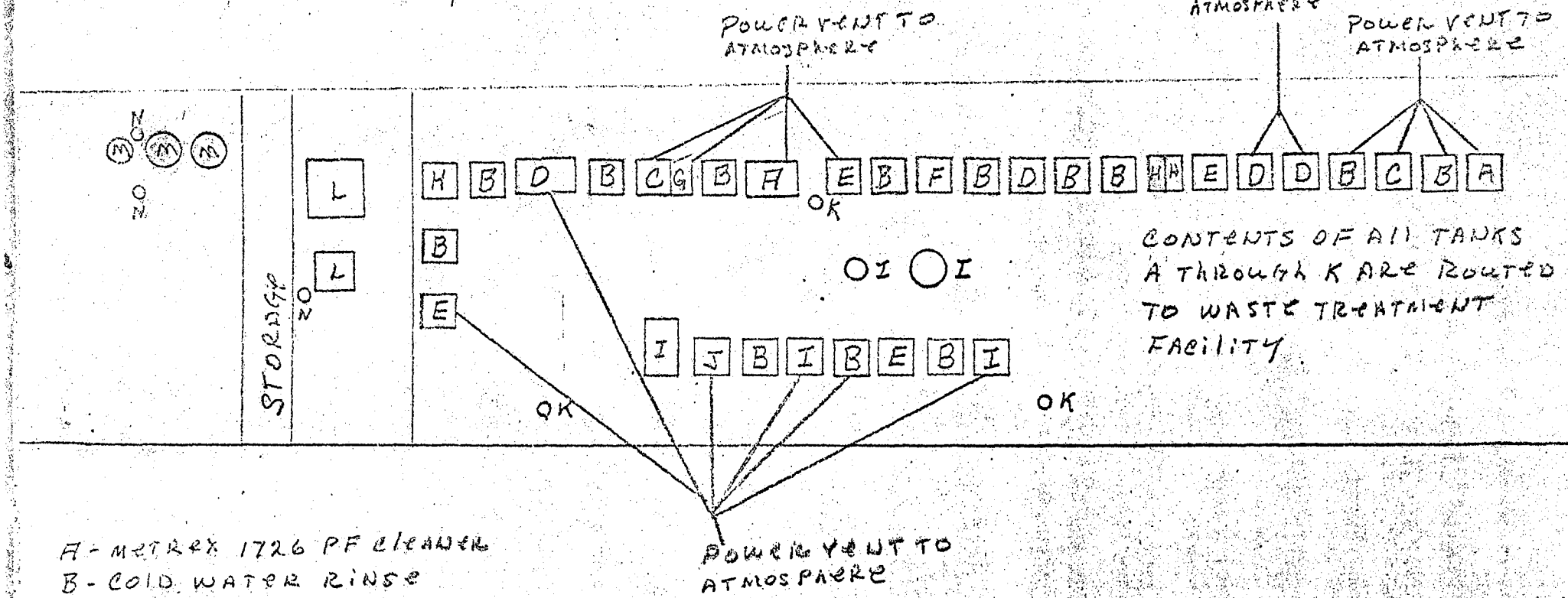
IRIDITE PROCESS



05/05/77

REF: SOP # SOREN PPR-1 (Aug. 197

LAYOUT OF BUILDING 1884 PLATING SHOP



A - METREX 1726 PF CLEANER
B - COLD WATER RINSE
C - HYDROCHLORIC ACID
D - SODIUM CYANIDE - CAUSTIC SODA
E - HOT WATER RINSE
F - PHOSPATEX
G - NITRIC ACID
H - NOT USED
I - CHROMIC ACID
J - FIRALINE CLEANER
K - EYE WASH/SAFETY SHOWER
L - DE-GREASER TRICHLOROETHANE " " "
M - VIBRATORY MILL
N - FLOOR DRAIN

DEPARTMENT OF THE NAVY
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA 47522

IN REPLY REFER TO:

3025-SR/se
8000/2
3 Mar 1981

MEMORANDUM

From: 3025
To: 092 (C. Andrews)

Subj: Plating Shop Effluent Trail Analysis

1. Grab samples identified as #1--Storm sewer outfall, Pyro Area; #2--stream across railroad-1; and #3--Building #1884 condensate were received on 19, 20, 23 and 24 February 1981. It was requested that pH, cyanide, cadmium and zinc analyses be performed on each sample.

2. Cyanide and pH analysis results (using Electrode methods) plus cadmium and zinc analysis results (as determined by atomic absorption spectrophotometry) are listed below:

Sample Number	Date Received	pH @ 20°C	mg/l Cyanide	mg/l Cadmium	mg/l Zinc
1	2-19-81	7.5	3.60	0.14	1.78
2	2-19-81	7.1	1.00	<0.1	0.42
3	2-19-81	7.6	0.04	<0.1	<0.1
1	2-20-81	6.8	1.90	0.42	1.07
2	2-20-81	6.8	0.26	0.42	0.56
3	2-20-81	7.4	0.04	<0.1	<0.1
1	2-23-81	6.8	1.60	<0.1	0.72
2	2-23-81	7.0	0.30	<0.1	0.15
3	2-23-81	8.2	0.04	<0.1	<0.1
1	2-24-81	6.9	1.00	0.12	0.53
2	2-24-81	7.0	0.18	<0.1	0.14
3	2-24-81	8.5	0.04	<0.1	<0.1

3. The Environmental Protection Agency has recommended maximum holding time prior to cyanide analysis is 24 hours. Due to work load complications, cyanide analysis on all the above listed samples was performed on 26 February 1981.

4. Further communications regarding these samples should reference Explosives Analysis Branch Numbers H-911, H-912, H-913 and H-918.

R. E. Klausmeier

R. E. KLAUSMEIER

DEPARTMENT OF THE NAVY
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA 47522

IN REPLY REFER TO:
3025-SR/se
8000/2
24 Feb 1981

MEMORANDUM

From: 3025
To: 092 (C. Andrews)

Subj: Pyro Area and Stream at R-1 Water Analysis

1. One grab sample from the pyro area outfall storm sewer and one from the stream at R-1 were received on 2-18-81. It was requested that analysis for pH, cyanide, cadmium and zinc be performed on each sample.

2. The pH and cyanide results as determined by electrode methods plus cadmium and zinc concentrations as determined by atomic absorption spectrophotometry are listed below:

Sample #	Sample Identification	pH @ 20°C	Cyanide mg/l	Cadmium mg/l	Zinc mg/l
1	outfall storm sewer pyro area	6.3	2.3	0.75	2.82
2	stream at R-1	6.7	0.4	<0.10	0.65

3. Further communications regarding these samples should reference Explosives Analysis Branch Number H-908.

R. E. Klausmeier

R. E. KLAUSMEIER

092V

DEPARTMENT OF THE NAVY
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA 47522

IN REPLY REFER TO:
092V-CA:dpt
11346-4084E/150
8 MAY 1981

From: Commanding Officer, Naval Weapons Support Center (Code 09), Crane, Indiana 47522
To: Commander, Crane Army Ammunition Activity (SARCN-10), Crane, Indiana 47522

Subj: Plating Shop Operation Corrections

Encl: (1) Service Contract Managers Report
(2) Laboratory Services' Analysis Report.

1. It has been brought to the attention of Code 092V, that there have been recent violations of cyanide effluent limitations from the Plating Shop Treatment Facility, Building 3064. Enclosures (1) and (2) indicate that the problem may lie in the improper segregation of chemical waste in the Plating Shop, Building 1884. This results in cyanide entering the treatment facility through the wrong channels thus leading to improper treatment and an unacceptable discharge of cyanide into the environment. It is noted in enclosure (2) that any cyanide which does not enter the treatment system via the correct lines will not be treated before it is discharged. Therefore, it is required that all chemicals be properly segregated and channeled to the appropriate lines for treatment. Failure to correct the discharge problem could lead to fines levied against the Commanding Officer and Naval Weapons Support Center (NAVWPNSUPPCEN) Crane by the Environmental Protection Agency with possible subsequent internal actions. Due to the nature of the discharge, and the fact that Plating Shop operators have been previously advised of the problem, it is imperative that actions to correct the mishandling of Plating Shop waste be initiated immediately.

2. Code 092V, Cathy Andrews, extension 3114, can be contacted to answer questions or provide assistance if needed.

J. D. Faris
J. D. FARIS
By direction

Copy to:
SARCN-EDP2
AROICC (Dave Turner) — THIS SHOULD
AROICC (Mark Bault) NOT BE ADDRESSED THIS WAY

DAVE SHOULD RECEIVE A Blind copy.

DEPARTMENT OF THE NAVY
OFFICER IN CHARGE OF CONSTRUCTION
BUILDING 2516
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA 47522

OICC-MB:tlh
4330
10 April 1981

From: OICC
To: 092V

Subj: Contract N62472-81-C-6394, "Operation of Chemical Treatment Plant"

1. Confirming our conversation of this date, the contractor operating the plating shop water treatment facility has reported that the effluent from that facility has contained an unacceptable level of cyanide on two different occasions in the past month. The reason for this is that the plating shop operation has recently been changed so that cyanide is being introduced into a part of the system where the waste water is not treated for cyanide contaminants. When this occurs the operator has no way of knowing that cyanide is in that part of the system. Therefore, the cyanide is not neutralized.

2. Please review this problem and advise what action needs to be taken to correct it.

Mark Bault
MARK BAULT
Service Contract Manager

Enclosure (1)

TURNER
INCORPORATED
P.O. Box 1096
Warsaw, IN 46580
(219) 267-3305

RECEIVED
OICC/ROICC
APR 16 1 21 PM '81
N.W.S.C.
CRANE, IN.

April 14, 1981

Mr. Mark Bault
Building 2516
Naval Weapons Support Center
Crane, Indiana 47522

Ref: Contract N62472-81-C-6394

Dear Mr. Bault:

Enclosed please find the results of recent testing of the plating shop wastes and of selected points within the treatment plant. Also, included are copies of similar test results dated January 5, 1979, February 12, 1979 and March 7, 1979.

The primary significance of this data is that there is Cyanide in the Chrome and Acid-Alkali wastes water and Hexavalent Chromium in the Acid-Alkali wastewater. These materials will not be treated unless they are properly segregated in the plating shop and thus enter the treatment system at the proper place.

The March NPDES reports show two days of high cyanide effluent concentrations. These are most likely due to the above mentioned reasons.

Thank you for the opportunity to be of service.

Sincerely:

David M. Turner, CMfgE
President

DMT/cjt

Enclosure (2)

3/14/81

Laboratory Results for NWSC-Crane

Sample Number	13024	13081	13025	13080	13030	13029	13082
I.D.	Chrome inc	Chrome inc	Cyanide inc	Cyanide inc	A-A inc	A-A inc	A-A inc
Date	3/12/81	3/25/81	3/12/81	3/25/81	2/28/81	3/12/81	3/25/81
Cadmium	7.6 mg/l	58 mg/l	38 mg/l	23 mg/l	0.41 mg/l	21 mg/l	8.0 mg/l
Chromium Total	11.0	193	0.69	*0.01	27	0.69	*0.01
Chromium Hexavalent	11.0	175	*0.01	*0.01	19	*0.01	*0.01
Copper	*0.01	1.0	0.75	0.75	1.3	4.9	0.10
Zinc	*0.01	2.0	0.10	5.0	0.69	0.69	2.0
Cyanide total	0.07	0.50	440	110	1.3	1.7	0.36
Suspended Solids	15	158	168	32	57	12	11
pH	6.2 units	3.7 units	11.4 units	11.3 units	6.0 units	2.2 units	2.3 units

Sample Number	13027	13028	13026	13079
I.D.	T-17	T-18	Effluent	Effluent
Date	3/12/81	3/12/81	3/12/81	3/25/81
Cadmium	40 mg/l	11 mg/l	*0.01 mg/l	*0.01 mg/l
Chromium Total	8.2	0.69	*0.01	*0.01
Chromium Hexavalent	*0.01	*0.01	*0.01	*0.01
Copper	1.6	0.81	0.31	
Zinc	4.0	4.0	*0.01	*0.01
Cyanide Total	1.2	1.2	1.7	0.24
Suspended Solids	456		9	
Insoluble Solids		365		
pH	11.4 units	11.3units	11.3 units	10.5 units

* - indicates less than
Note: The effluent is actually t-20 (before final neutralization)

All Tests were conducted according to "Standard Methods".

SHEET 1 OF 2

Commanding Officer
Naval Weapons Support Center
Code 092
Crane, Indiana 47522

PLEASE COMPLETE AND SUBMIT ONE COPY AT THE END OF EACH MONTH. THE REPORT MUST REACH THE STREAM POLLUTION CONTROL BOARD OFFICE BY THE 28TH OF THE FOLLOWING MONTH.

IN 0 0 2 1 1 5 3 9 (1-7) PERMIT NUMBER

1 0 1 (8-10) OUTFALL/ FAC. NO.

0 3 8 1 (11-12)(13-14) MO. YR.

ENTER: 1 for NPDES, 2 for SPC-15: 1 (15)

ENTER: 1 - INDUSTRY, 4 - FEDERAL, 5 - WATER SUPPLY: 4 (68)

EFFLUENT CHARACTERISTICS		FLOW	pH		Cyanide Tot		Zinc Total		Chromium Hex	
EFFLUENT NO. (16-21)		Q50050	C00400		C00720	C00720	01092	01092	01033	01033
SAMPLE TYPE (22-23)	Permit Cond.	NA	GR		24	24	24	24	24	24
	Monitored	NA	GR		24	24	24	24	24	24
FREQUENCY (24-28)	Permit Cond.	5/7	5/7		1/7	1/7	1/7	1/7	1/7	1/7
	Monitored	5/7	5/7		1/7	1/7	1/7	1/7	1/7	1/7
EFFLUENT LIMITATIONS	Daily Avg. (24-36)		5.0-		0.7	0.029		1.14	0.09	0.054
	Daily Max. (37-44)		11.0		2.0	0.063	20	2.5	0.25	1.14
DATE / UNITS		MGD	HI	LOW	mg/l	lb/day	mg/l	lb/day		
1										
2		0.008	7.4		2.4	0.16	*0.01	0	*0.01	0
3										
4		0.006	8.4							
5		0.010	7.2							
6										
7										
8										
9		0.002	8.2							
10		0.003	8.6		0.48	0.012	*0.01	0	*0.01	0
11		0.005	8.4							
12		0.006	8.6							
13		0.005	8.6							
14		0.005	8.[illegible]							
15										
16		0.002	8.6		1.7	0.028	*0.01	0	*0.01	0
17		0.006	8.6							
18		0.009	8.8							
19										
20		0.008	8.9							
21										
22										
23		0.003	8.6							
24		0.003	7.4							
25		0.004	8.4		0.24	0.008	*0.01	0	*0.01	0
26		0.007	7.6							
27										
28										
29										
30		0.005	7.6							
31		0.004	8.2							
AVERAGE (29-36)		0.005	X		1.2	0.05	*0.01	0	*0.01	0
HIGHEST VALUE OF MONTH (37-44)		0.010	8.9	X	2.4	0.16	*0.01	0	*0.01	0
LOWEST VALUE OF MONTH (45-52)		0.002	X	7.2	0.24	0.008	*0.01	0	*0.01	0
NO. OF TIMES MAX. EFFLUENT LIMITATIONS EXCEEDED (53-54)		0	0		2	2	0	0	0	0

*indicates less than

SIGNATURE OF CERTIFIED OPERATOR (55)

(60) (SIGNATURE OF PRINCIPAL EXECUTIVE OFFICER OR AUTHORIZED AGENT)

219/267-3305 743376 4-9-87

MONTHLY MONITORING (OPEN FORM)

REPORT FOR SPC-15 OR NPDES DISCHARGE PERMITS

SHEET 2 OF 2

Officer
Weapons Support Center
092
, Indiana 47522

PLEASE COMPLETE AND SUBMIT ONE COPY AT THE END OF EACH MONTH. THIS REPORT MUST REACH THE STREAM POLLUTION CONTROL BOARD OFFICE BY THE 28TH OF THE FOLLOWING MONTH.

PERMIT NUMBER (1-7)	OUTFALL/ FAC. NO. (8-10)	MO. YR. (11-12) (13-14)
2015319	101	03/81

ENTER: 1 for NPDES, 2 for SPC-15: 1 (15)

ENTER: 1 - INDUSTRY, 4 - FEDERAL, 5 - WATER SUPPLY: 4 (66)

EFFLUENT CHARACTERISTICS		Cadmium							
EFFLUENT NO. (16-21)		c0102	0102	c	0	c	0	c	0
SAMPLE TYPE (22-23)	Permit Cond.	24	24						
	Monitored	24	24						
FREQUENCY (24-28)	Permit Cond.	1/7	1/7						
	Monitored	1/7	1/7						
EFFLUENT LIMITATIONS	Daily Avg. (29-36)	0.5							
	Daily Avg. (37-44)	1.0							
UNITS		mg/l	lb/day	mg/l	lb/day	mg/l	lb/day	mg/l	lb/day
DATE									
1									
2		*0.01	0						
3									
4									
5									
6									
7									
8									
9									
10		*0.01	0						
11									
12									
13									
14									
15									
16		*0.01	0						
17									
18									
19									
20									
21									
22									
23									
24									
25		*0.01	0						
26									
27									
28									
29									
30									
31									
AVERAGE (29-36)		*0.01	0						
HIGHEST VALUE OF MONTH (37-44)		*0.01	0						
LOWEST VALUE OF MONTH (45-52)		*0.01	0						
NO. OF TIMES MAX. EFFLUENT LIMITATIONS EXCEEDED (53-54)		0	0						

January 5, 1979

Able Associated Enterprises, Inc.
North & East Streets
Montozuma, Indiana 47862

Attention: Mr. E.C. Market

Dear Gene:

Attached please find the December results of the laboratory testing and the completed November and December monthly reports for the Navy.

Dick and I are getting together to talk about the needed changes on the control panel.

Sincerely:

David M. Turner
President

DMT/cjt

1/3/79

Laboratory Results for NWSC-Crane

parameter	EFF	EFF	EFF	EFF	Chrome	Cyanide	Acid-alkali	Sludge
sample no.	098	099	030	031	034	033	035	032 Leachate
Date	12/8	12/14	12/21	12/27	12/27	12/27	12/27	12/21
Cadmium	*0.1 mg/l	*0.1 mg/l	2.2 mg/l	5.1 mg/l	*0.1 mg/l	15.0 mg/l	4.2 mg/l	*0.1 mg/l
Chromium(Tot)	80.1	00.1	0.4	4.8	18.2	*0.1	2.6	*0.1
Copper	*0.1	*0.1	*0.1	*0.1	*0.1	*0.1	*0.1	*0.1
Iron	*0.1	*0.1	*0.1	*0.1	*0.1	1.0	2.0	0.2
Lead	*0.1	*0.1	*0.1	*0.1	*0.1	*0.1	*0.1	*0.1
Cyanide(tot)	0.02	*0.01	*0.01	*0.01	*0.01	360	*0.01	*0.01
Oil & Grease	*1	*1	*1	*6n	*1	*1	21	-
Chromium (Hex)	*0.01	*0.01	*0.01	0.10	18	*0.01	*0.01	*0.01
Suspended Solids	4	3	4	3	-	-	-	-
pH	7.6	8.1	6.7	7.8	6.7	12.4	7.3	-
% Solids	-	-	-	-	-	-	-	33.4%

*-indicated less than

All tests were conducted according to "Standard Methods" or regulatory agency approved techniques.

February 12, 1979

Able Associated Enterprises, Inc.
North & East Streets
Montezuma, Indiana 47862

Attention: Mr. E.C. Market

Dear Gene:

Following are the results of the influent testing for NWSC-Crane for January 1979:

Sample Number	055	056	057	058
Date	1/4	1/24	1/24	1/24
Type	Sludge	Acid-Alkali	Cyanide	Chromium
% solids	60.8 %			
Suspended Solids	leachate	2 mg/l	84 mg/l	2 mg/l
Cyanide (tot)	*0.01 mg/l	0.74	220	0.06
Oil & Grease		12	*1	*1
Cadmium	0.3	0.8	15.5	7.2
Chromium (tot)	*0.1	*0.1	*0.1	54
Chromium (hex)	*0.01	*0.01	*0.01	52
Zinc	*0.1	7.8	29	1.2
Iron	*0.1	11	1.0	0.5
Lead	*0.1	*0.1	*0.1	*0.1
Copper	*0.1	0.2	*0.1	*0.1
pH	-	2.2 units	11.8 units	6.8 units

* - indicates less than

All tests were conducted according to the latest edition of "Standard Methods".

Sincerely

David M. Turner
President

DMT/ojt

March 7, 1979

Able Associated Enterprises, Inc.
North & East Streets
Montezuma, Indiana 47862

Attention: Mr. E.C. Market

Dear Gene:

Following are the results of the February testing at Crane:

Date	2/9	2/14	2/22
Suspended Sol.	6 mg/l	6 mg/l	5 mg/l
Cyanide(tot)	0.32	0.38	0.28
Oil&Grease	*1	*1	*1
Cadmium	0.4	*0.1	*0.1
Chromium(tot)	*0.1	*0.1	*0.1
Zinc	*0.1	*0.1	*0.1
Iron	*0.1	*0.1	*0.1
Copper	*0.1	*0.1	*0.1
Chromium(Hex)	*0.01	*0.01	*0.01
pH	7.4	8.9	8.9

Date	2/14	2/14	2/14	2/14	2/14
Type	sludge	A-A inc	Cyanideinc	Chrome inc.	
% Solids	50.9%				
Susp. Solids		8 mg/l	93 mg/l	2 mg/l	
	Leachate				
Cyanide(tot)	*0.01mg/l	*0.01	156	*0.01	
Oil& Grease	*1	22	8	*1	
Cadmium	0.2	0.9	24	5.4	
Chromium(tot)	*0.1	*0.1	*0.1	72	
Chromium(hex)	*0.01	*0.01	*0.01	70	
Zinc.	*0.1	15	22	3.5	
Iron	*0.1	11	0.9	0.2	
Lead	*0.1	*0.1	*0.1	*0.1	
Copper	*0.1	*0.1	*0.1	*0.1	
pH	-	3.4	11.7	4.5	

* - indicates less than

All tests were conducted according to the latest edition of "Standard Methods".

Sincerely:
David M. Turner

DMT/cjt

INTRODUCTION

The Crane Plating Shop Waste Treatment Facility Post Occupancy Evaluation is unique in that the construction contractor made major changes in the design resulting in a highly successful operation. The construction contractor later was contracted to operate the plant. At the time of evaluation, the plant had operated successfully for 18 months. Although improvements could be made, no action items for construction changes are necessary. The contractor was awarded a "Commander's Certificate of Commendation" for his outstanding performance.

In this case the "lessons learned" are perhaps better defined as guidelines based on demonstrated performance rather than malfunctions. This report describes the changes made and methods used to effect the changes.

DESCRIPTION OF THE FACILITY

HISTORY

The construction contract was awarded to low bidder, Able Associates, Inc., for $394,300.00. Soon after award, the contractor identified problems with the specified effluent limitations and deficiencies in the collection system design. Modifications which would improve plant operation were also suggested. The contractor was encouraged to develop a revised design and submit it for evaluation.

The contractor submitted 23 revised drawings and related specification changes which with minor changes were approved as a no cost change order. The modifications included a revised collection system; increased retention capability, a more efficient sludge de-watering system, a tank exhaust system, semi-automatic control (sequential processor and flow proportional control valves), elimination of chemical hand-mixing, and changing the type of building construction.

During operator training it became evident that the Navy would require two Class III certified operators to meet Indiana State requirements. It was then decided to contract the operation and maintenance functions. Able Associates, Inc., was the successful bidder on the one-year service contract.

In summary, the procedures followed for the Crane operation has proven very successful. However, its success was dependent upon the availability of a construction contractor with skilled knowledge of the process, and highly qualified operators. Each project of this type should be evaluated and the contractural method selected that will produce desired results.

Able Associates, Inc., has been recognized for their commendable performance, but pertinent Crane PWC, ROICC, and Northern Division personnel contributing to this success should also be recognized.

04247

09247-PK:cjs
11346/Fac. File B-1884
06 FEB 1985

From: Commanding Officer, Naval Weapons Support Center
To: Commander, Crane Army Ammunition Activity

Subj: Pollution Control Measures at Building 1884

Encl: (1) Toxic Organic Pollutant Management Plan

1. To satisfy the requirements of National Pollutant Discharge Elimination System (NPDES) Permit IN0021539, issued to Naval Weapons Support Center (NAVWPNSUPPCEN) Crane on 7 September 1984, a Toxic Organic Pollutant Management Plan, enclosure (1), has been written and submitted to the Indiana State Board of Health. The plan includes measures to prevent water pollution caused by toxic organic compounds used in the plating operations at Building 1884.

2. One such toxic compound, 1,1,1-trichloroethane (Chlorothene), is used in the vapor degreasers at Building 1884. It is listed in 40 CFR 433.11(e) and is therefore considered a toxic organic pollutant if it should be introduced into wastewaters or surface waters, by itself or in combination with other toxic organic compounds, at concentrations exceeding limitations (2.13 mg/l, daily maximum) specified in this regulation.

3. The Toxic Organic Pollutant Management Plan written for Building 1884 requires the following measures to be taken to prevent water pollution caused by 1,1,1-trichloroethane in the event of an accidental discharge:

a. Floor drains in the vapor degreasing room shall be plugged when drums of Chlorothene are in the area for degreaser replenishing and when spent solvent is pumped out of the degreasers.

b. Absorbent material shall be readily available to spread on any spill of Chlorothene. The contaminated absorbent shall be handled as a hazardous waste and disposed via 0924.

c. Pumps which may be used for transferring Chlorothene shall be flushed out and rinsed off before being used to pump any other solutions. Water which is used for flushing and rinsing should be containerized for disposal as a hazardous waste via 0924.

4. All operators at the Building 1884 plating shop shall be informed of these new practices so that they may include them as part of their routine procedures.

5. Plugs are being ordered for floor drains, and 0924 will distribute them when they are received. If new or additional absorbent material is needed, contact Code 0924.

09247-PK:cjs
11346
06 FEB 1985

6. NAVWPNSUPPCEN Crane point of contact is Mrs. Cathy Andrews, 0924, extension 3114/1132.

J. D. FARIS
By direction

Copy to:
SMCCN-IOS
SMCCN-EDP (Bill Hubble)
SMCCN-EDN

TOXIC ORGANIC POLLUTANT

MANAGEMENT PLAN

NAVAL WEAPONS SUPPORT CENTER

CRANE, INDIANA

19 NOVEMBER 1984

Signature of Principal Executive Officer

J. A. Faris, P.E.

Enclosure (2)

TOXIC ORGANIC POLLUTANT MANAGEMENT PLAN

I. Outfall 101

A. Wastewater Discharges

Outfall 101 is the final discharge of pretreated wastewaters which are a result of metal finishing operations.

A plating shop is located in Building 1884. Wastewaters produced in the plating shop are pumped to Building 3064 where they are pretreated before being discharged to the sanitary sewer system.

Wastewaters which are pumped to the pretreatment facility include rinsewaters from cadmium and zinc plating operations, acid-alkali rinsewaters, and rinsewaters from chromating, phosphating and coloring operations.

Process operations associated with the metal finishing operations at Building 1884 include deburring and vapor degreasing. Neither of these operations discharges wastewater through outfall 101.

B. Toxic Organic Pollutants Identified

None of the compounds listed in 40CFR433.11(e) have been identified as occurring in the makeup of any of the solutions currently used in the metal finishing operations which discharge wastewaters through outfall 101.

On 26 October 1983 the discharge at outfall 101 was sampled. The sample was analyzed for toxic organic compounds. Results of the analysis were recorded on EPA Form 2C and submitted as part of the application for the current NPDES permit. The analysis indicated concentrations of 0.01, 0.83, and 0.14 mg/l for the toxic organic compounds chloroform, methylene chloride, and toluene, respectively. These compounds are not used in the plating shop. Their occurrence in the sampled wastewaters is believed to be from glues used for installing/repairing plastic piping or from epoxy coating solvents.

One toxic organic compound was identified as being in use in the vapor degreasing operation. The solvent used in the vapor degreasers is 1,1,1-trichloroethane. The trichloroethane is not released to any wastewater collection system. When it is necessary to dispose of the material, it is pumped into 55-gallon drums and handled as a hazardous waste. A disposal contract for hazardous waste hauling and chemical disposal is in effect with a permitted waste hauler.

C. Measures Taken to Prevent Pollution

Although the trichloroethane used in the vapor degreasers should not routinely spill or leak into the wastewater collection system, surface water or groundwater, it is conceivable that an unanticipated spill or leak could

occur during vapor degreaser replenishing or when removing the used solvent. Floor drains in the degreasing room would be most apt to receive such spillage. The floor drains drain to a storm sewer, not to outfall 101.

Measures will be taken to prevent pollution by the trichloroethane. These include plugging the floor drains in the degreasing room and storing absorbent material close to the area to contain spills. Additionally, if a pump is used to remove or add trichloroethane to the degreaser, it will be decontaminated before it is used for any other pumping operation. The water used for flushing the pump will be containerized and handled as a hazardous waste.

II. Outfall 401

A. Wastewater Discharge

Outfall 401 is the discharge from a pipe located in a manhole outside of a laboratory plating shop at Building 38. Plating operations are performed on printed circuit boards which are manufactured at Building 38. The wastewater which flows through outfall 401 consists of rinsewaters from black oxide coating and from nickel-gold, copper, and tin-lead plating lines. Also included is rinsewater from a circuit board etching machine and wastewater from two sinks. There is no pretreatment of the wastewaters. At the manhole the water enters the Center's sanitary sewer system.

Process operations associated with the printed circuit board manufacturing include cleaning, etching, machining, shearing, soldering, deburring, laminating, and assembly. The etching operation is the only process operation which contributes wastewater to outfall 401.

B. Toxic Organic Pollutants Identified

None of the toxic organic compounds listed in 40CFR433.11(e) have been identified as occurring in the makeup of any of the solutions currently used in the plating of circuit boards or in the associated process operations.

C. Measures Taken to Prevent Pollution

Since no toxic organic compounds have been identified, no special measures beyond normal spill control and cleanup procedures have been taken to prevent pollution by the compounds.

III. Outfall 501

A. Wastewater Discharge

Outfall 501 is the discharge from a pipe in a manhole outside of a small arms coating operation at Building 2521. The wastewater which flows through outfall 501 consists of rinsewaters from phosphate and black oxide coating lines. At the manhole the water enters the Center's sanitary sewer system without pretreatment.

Process operations associated with the small arms coating operation include cleaning, machining, sandblasting and vapor degreasing. None of the process operations contribute wastewater to outfall 501.

B. Toxic Organic Pollutants Identified

None of the toxic organic compounds listed in 40CFR433.11(e) have been identified as occurring in the makeup of any of the solutions currently used in the coating operations which discharge wastewaters through outfall 501.

In the process operations only one toxic organic compound listed in 40CFR433.11(e) has been identified as being used. The solvent trichloroethylene is used in the vapor degreaser.

C. Measures Taken to Prevent Pollution

The trichloroethylene used should not routinely spill or leak into the wastewater collection system, surface water or groundwater.

Should a spill of trichloroethylene occur, it would be contained by absorbent material stored in the area of the degreasing operation. There is but a slight chance that spilled solvent would reach either of the two floor drain areas which are over 30 feet away from the degreaser. Spent trichloroethylene is drained into 55-gallon drums which are transported off the Center by the contracted hauler.

IV. Handling of Containerized Wastes and Spill Response

Once the trichloroethane and trichloroethylene wastes have been containerized, they are stored (for less than ninety days) outside of the buildings at which they were generated. When the storage areas become full, the Center's hazardous waste handlers are notified. The waste handlers transport the drums to a central hazardous waste storage area (currently under interim status). At the central storage area the drums are loaded for removal to an off-Center permitted facility.

The response action to a spill or leak in the drums which are being held for shipment is covered in the Center's Spill Contingency Plan.

B-2698

6810E/63C

ENVIRONMENTAL PROTECTION FACILITY INSPECTION REPORT

TABLE OF CONTENTS

		Page
I.	General Information	1
II.	Operations Data	1
III.	Air Pollution Control	2
IV.	Wastewater Discharge Management	3
V.	Solid Waste and HW Waste Management	4
VI.	PCB Management	6
VII.	Spill Contingency	7
VIII.	Special Treatment Facility	8

6810E/63C

ENVIRONMENTAL PROTECTION FACILITY INSPECTION REPORT

I. General Information

A. Building Number/Facility/Location: B 2698 pyro

B. Date: 6-17-86

C. Operator/Facility Supervisor: Bill Long, Technican: Tom Clements
Lester Hannon

D. Code: CAAA

E. Extension:

F. Process(es): Load & Assembly of MK 58 Candles.

II. Operations Data

A. General description of environmentally related operations (wastewater, air emissions, solid waste, HW production, storage, disposal) At the last phase of the assembly operation, a foam is injected into the canister. This foam is composed of Component A Isocyanate and B polyol (polyol) urethane foam. This mixture then hardens in the canister. This part of the operation is the only part that really contributes any Hazardous Waste, as there is some excess Isocyanate that is generated and is then picked up as HW by Code 0924 personnel. There is also a stenciling line for the MK 58 Candles. Stenciling Ink and Naphtha are used in this operation, but this does not generate any HW.

— There are (2) one gal pails (open) that have Isocyanate in one and B polyol in the other. These are on floor along wall by foam operation. I told Lester Hannon to dispose of this.

Check if the following are applicable; include sizes, no., & vol.:

1. Waste Containers		8. Plating	
2. Tanks or Sumps		9. Maintenance and Repair	
3. Surface Impoundments		10. Testing	✓ *
4. Waste Pile		11. Thermal Treatment	
5. Incinerators/ Component Parts		12. Chemical, Physical and Biological Treatment	
6. Painting (stenciling)	✓	13. Other	
7. Degreasing			

* each Candle is submerged in Water to test for any leaks.

III. Air Pollution Control

A. Applicable equipment (degreaser, paint booth, boiler, etc.):

NA

(If applicable, indicate primary/secondary fuels used in the system:

NA)

1. Model Number(s): NA

2. Manufacturer(s): NA

3. Is operation exhausted to atmosphere? yes

4. Describe pollution control or exhaust equipment involved:

Small Air Vent is over part of operation where the foam is injected into canester

5. Exhaust flow information (CFM rates):

6. Exhaust system stack diameter/height:

B. Chemicals used in process

1. Nomenclature: Isocyanate, Polyeurathane foam, Naphtha (Methylene Chloride used to clean spray nozzles when operation halts) (Polyol) only

2. Volumes used per unit time: Naphtha approx - 1 pint a day. Isocyanate and Polyeurathane foam. approx 1 qt per unit.

C. Are process procedures readily available and updated (SOP's, M.A., etc.)? operation not in process therefore I'm not sure if SOPs updated. NA

D. Is the operator familiar with the procedures? yes

E. Is asbestos insulation used for piping, ductwork, etc.? no

IV. Wastewater Discharges

A. Describe discharge point (sanitary sewer, environmental):

drains go to Sanitary sewer - drains are located in Area of stenciling operation and are approx 5-0ft from loading operation

Are discharges covered by permit? NA

Are permit requirements being met? NA ;

Note problems: NA

;

Note corrective measures:

B. Sumps/Tanks No Sumps or Tanks

1. Are sump/tank overflows covered under permit?

2. Are inspection forms being completed properly?

a. Is the operator indicating on the inspection form any deficiencies or malfunctions and the action taken to correct problem?

Has he/she notified 0924 and made a record of such?

b. Is the operator submitting a copy of the completed inspection reports (as applicable) by the 28th of each month to Code 0924 for review and retention?

c. Does the operator have copies of all completed inspection logs (last 3 years) on file in his office?

3. Are overflows from tanks/sumps obvious?

a. Do tanks and sumps have sufficient freeboard?

b. Do overflows discharge to the environment or sewage system?

c. Is discharge alarm/control equipment operational?

d. Is the construction of the tank/sump and/or related confinement area faulty? _____

e. Note the type and size of containment structure vs. the volume of the tank or sump system:

_____

f. Do tanks/sumps have covers? _____

g. Are they properly secured? _____

h. Are tanks/sumps labeled as to contents? _____

C. Are cooling waters directly discharged to the environment? NA

1. Are these waters contaminated with oil/antifreeze or other chemicals? _____

V. Waste Management

A. Type of Waste(s) Generated: (Note if it is sanitary solid waste or hazardous waste) container description (dumpster, drums, sump, sludge, etc.)

Isocyanate is cleaned out of spray nozzles by spraying Methylene Chloride through the nozzle.

B. Amount Generated per Unit Time: approx a qt per unit

C. Storage/Method of Disposal: Waste is placed in 55 gal drum and placed on pallets outside of Bldg (this is dangerous - a covered container is needed) discussed this with Bill Long. Bill to call in TC for Trails

D. Is the operator familiar with containerization requirements? Yes

1. Is HW material, as applicable, placed in used containers)? Yes

2. Are wastes being mixed without approval of 0924? no

3. Are empty drums handled properly? Yes

E. Labeling (H.W.)

1. Have the following items been filled out on label for H.W.? (This includes scrap explosives/pyrotechnics):

   a. Contents yes
   b. Serial Number (Date and Code) yes
   c. Contact yes
   d. Extension yes
   e. First Aid no
   f. Spill Treatment no
   g. Date when first hazardous waste placed in drum yes

2. Has permanent marker been used to fill out label? yes

3. Has the waste been stored properly in the hazardous waste storage shelters (if available)? None available

4. Do laboratory wastes have labels per each category? NA

   Have they been placed in trays? NA

F. Waste generation projected for future or future volume changes (e.g., solvents, waste oil, plating wastes, etc.)

Operation not in process, unable to get parts at this time

G. Waste Transfer Operations

1. How is waste collected (skimming, drained, transferred, (e.g., sumps, tanks, etc.))?

   Collected in small pails from nozzles

2. How is it transferred into large containers (pump, pouring)?

   poured from small pails to 55 gal drum located on pallet outside.

3. Are materials/equipment used for transfer decontaminated or, if not, secured and labeled as to use? no.

4. Are fuel oil tanks diked? (This is applicable to facilities which are located next to bodies of water or tanks over 500 gallons.) NA

5. Are there containment areas around the operation? If so, what type and size?

No

6. General comments (How could it be improved, equipment needed, etc.).

Operation was not in process while I was there, but everything seems to be in order and safe with HW generation at a minimum. There were 2 open pails containing isocyanate and polyol. Lester to dispose of this.

H. Training

1. Have all persons involved in handling, supervising or transporting hazardous waste attended an annual training course for hazardous waste handlers and transporters? yes, only Bill Long (Ernie Wyttenbach needs to receive training)

2. Has this been recorded in their personnel files? unknown, as the files are kept at B2532 not at B2698.

I. Facility Standards

1. Are there appropriate warning signs posted at an HW facility? _____

2. How is the facility secured? _____

3. What emergency communication equipment is available in case of accident or spill?

Radio ____ Telephone ____ Alarm System ____

VI. PCB Review/Control NA

A. Heat Transfer Systems/Hydraulics

1. Labeled (PCB, non-PCB): _____

2a. When sampled/resampled: _____;

2b. Analysis results noted: _____

3. Labels/records updated: _____

4. Leaks noted/corrections or containment: _____

5. Comments _____

B. Items (transformers/capacitors, etc.)

1. Are items turned in immediately or stored indefinitely in area?

2. Are leaks controlled? _____

3. Are 1348-1's filled out properly? _____

4. Are items turned in to 0924? _____

VII. Contingency Response and Emergency Procedures

A. Do personnel know who to contact in case of a spill or related emergency? yes, supervisor

B. Do they know what action to take if there is a spill (e.g., plugging drains, evacuation, etc.)? yes

C. Are there emergency/spill control equipment/supplies (absorbent, empty drums for waste) near areas of potential spills? absorbent, drums

1. Are containers and/or areas used for storage of equipment and spill control supplies labeled clearly as to what they contain? yes

2. Are personnel familiar with their location? yes

3. If equipment and supplies are available, what are they? (Should be at least one compatible pump available)

absorbent

4. What areas of the facility are rated as high risk areas for potential spills?

There is a seperate room which can only be entered from outside of the Bldg, which houses an electronic operated unit for pumping the Isocyanate and polyurethane (polyol) foam through hoses which lead to the spray nozzles in the foam injecting operation. There are no drains in Bldg. If spill occurs, absorbent is used to collect waste. (appropriate breathing apparatus is used along w/other gear)

5. What chemicals are involved? Isocyanate, polyurethane foam (polyol)

D. What emergency equipment is available, (fire extinguisher, eyewashes, safety showers, etc.)?

Fire extinguisher, safety shower

1. Is/are the operator/personnel familiar with the safety equipment and where it is located? yes

2. Is the equipment routinely checked for usability? yes

   What frequency? _____

3. Is/are the operator/personnel familiar with emergency shutdown operations? they check w/supervisor

4. Is an alarm system, radio, telephone available in the immediate area? yes

VIII. Special Treatment Facilities

A. Plating Shop Pretreatment Facility
B. Building 36 Acid Neutralization System
C. Rockeye/B-160 Carbon Facilities
D. ABG/Demolition Area/Old Rifle Range/B-2167/OTA
E. Surface Impoundments
F. Ash Pile
G. Demil Incinerators

A. PRETREATMENT FACILITY OF PLATING SHOP (BUILDING 3064)

1. Is the filter press keeping water content of the sludge low enough? _____

2. Is filter leaking? _____

3. Where is leaking water channeled? _____

4. Is the treated discharge water meeting the requirements/discharge limits set forth in the Center's NPDES permit? _____

5. Is the operator familiar with procedures for packaging, storing and disposing of the sludge from the pretreatment process? _____

6. Are Assoc. NPDES log forms being completed properly? _____

B. ACID NEUTRALIZATION SUMP (BUILDING 36)

1. Is pH being adjusted before discharging to sewer? _____

2. Is the pH in the sump being checked by sewage treatment plant personnel before discharging? _____

3. Are records being kept of the pH checks? _____

4. Are they complete? _____

5. Condition of the sump and associated equipment and lines:

_____

_____

C. CARBON ADSORPTION SYSTEMS (ROCKEYE TREATMENT B-3044) AND TREATMENT FACILITY AT B-160)

1. Is operator aware/familiar with cleaning carbon adsorption system? _____

2. Is operator aware/familiar with procedure for labeling, storing, and disposing of spent carbon/ sludges? _____

3. Check alarm and wastewater cut-off system and determine if any problem with sump capacity.

_____

4. Is there a current SOP for plant operation/ cleaning and disposing of spent carbon and sludge from the carbon adsorption system? _____

5. Is the treated discharge water meeting the requirements/discharge limit? _____

6. Is carbon being changed frequently enough? _____

7. Are operation logs complete? _____

8. Is oil contamination of system obvious? _____

D. THERMAL TREATMENT - ABG/DEMOLITION AREA/RIFLE RANGE/B-2167/OTA

1. Is the burning/detonation/testing being done in compliance with State and Federal regulations and variances? _____

2. Are necessary records and reports required by the variances being kept and filled out properly? (Meeting parameters at both the Center and state levels, e.g., logs, meteorological reports) _____

   a. Are ash removal operations and storage accomplished in accordance with regulations? _____

   b. How are contaminated liquids handled (stored/treated/tested/disposed)?

   _____

   _____

   c. Are scrap manifest forms done properly and on file at facility? _____

3. Describe how access to the site is controlled:

   _____

   _____

4. Is there any evidence of environmental spills (oil, solvent, dyes, etc.)? _____

5. Is the burning/testing/demo being done in properly assigned areas? _____

6. Are the areas being maintained? _____

7. Is the waste ash container lid being kept closed/covered? _____

8. Is the container properly labeled? _____

9. Has ash been magnetized to remove as much metal as possible before being placed in container? _____

10. Is there fire truck on standby or is a hydrant available with hose connected? _____

11. Does it appear that other than contaminated dunnage is burned at ABG? (check scrap manifest to verify) _____

12. Are current records kept on containers (tanks, etc.) at ABG being flashed? (Check scrap manifest to verify) _____

13. Is excess fuel used for test/burn operations? _____

14. Are only explosive/pyrotechnic-contaminated chemical wastes burned at the ABG? (Check manifest) _____

15. Are SOP's current for all applicable operations at ABG/Demo/Old Rifle Range/2167? _____

16. Are TP's being followed at OTA? _____

17. Are ordnance items at Demo labeled/tagged/logged as to disposition? _____

18. Are fuel oil storage tanks leaking? _____

19. Is a contingency plan available at the ABG/Demo sites? _____

E. SURFACE IMPOUNDMENTS - ABG

1. Do surface impoundments have at least two feet of freeboard? _____

2. Are the surface impoundments and area around them kept free/clean of any wood or metal objects which could puncture or rip liner? _____

3. Are the surface impoundments being inspected weekly and after storms to determine if:

   a. Severe erosion of dikes or containment areas has occurred? _____

   b. Liner has been exposed in any areas of the surface impoundment? _____

   c. Covers for impoundments are still intact and in place? _____

   d. Sand used for filtering TNT- and phosphorus-contaminated water or sludges is in good condition and is replaced as necesary? _____

   e. Plugging of filter media or piping is occurring? _____

   f. Determine inspection frequency _____

4. How waste levels are being measured in tank? _____

5. How leaks are determined? _____

6. Are current SOP's available for impoundments? _____

7. Are tanks labeled properly? _____

F. WASTE ASH PILE

1. Is erosion evident? _____

2. Is runoff/run-on control maintained? _____

3. Is liner surfacing? _____

4. Are inspection reports completed routinely for the pile? _____

G. DEMIL INCINERATORS

1. Is the demil furnace brought to a steady state before the addition of waste? (Check logs) _____

2. Is a steady state maintained or correction attempted? (Check logs) _____

3. Is the airflow and temperature monitored and recorded in the log? _____

4. Is the equipment being inspected daily for leaks, spills and fugitive emissions, etc.? _____

5. Is stack plume observed at least hourly for normal color and opacity? _____

6. What type of emergency alarms and shutdown controls does the equipment have and is it being inspected on a daily basis as noted in inspection reports for proper operation?

_____

7. Are copies of all associated reports/logs submitted to Code 0924 by the 28th of the month? _____

8. Are they complete? _____

9. Are copies of military specifications, for any waste being incinerated, on file at the site? _____

10. Is this information included on reports/in logs? _____

11. Are records being kept on the types of ammunition incinerated and the amount per shift? _____

12. What is the general condition/appearance of the incineration equipment?

_____

_____

13. Are only compatible materials incinerated together? _____

14. Who determines compatibility? _____

15. Is an evacuation/contingency plan available? _____

16. Is safety equipment checked routinely? _____

17. Is any waste ash evident in the area? _____

18. Are fuel oil tanks in a containment area? _____

19. Are oil spills obvious in area? _____

V. Code 0924 must be notified in writing of the proposed corrective measures within 15 days of its receipt. Action must be initiated within 30 days of the inspection to correct any deficiency noted in the report.

Date: 6-17-86

Code 0924 Representative: Jo Belcher Eng Tech.
Signature/Title

Responsible Party: Bill Long Expl Oper Fam
Signature/Title

TETRA TECH

PPA INTERVIEW QUESTIONNAIRE

Individual Interviewed: Doug Johnson Project: CTO F276

Association (Military/Civilian): Project No.: 112G02126

Telephone Number: (812) 854-1481 Email Address: DougJohnson1@us.army.mil

Tetra Tech Interviewer: VJPlachy/RABarringer Date: 9/16/2009

1. **Your job background / history:**
   Approximately, when and how long did you work at Pyrotechnics Production Area (PPA)?
   What was /is your job description / function at PPA?
   In which area/building(s) did you work?
   - B-3148 – Curing Candles transferred from B-126
   - B-2854 – pumphouse; any cuttings or vacuum (e.g., candle wastes) collected in the accumulator then pumped (powder or dusts)
   - B-767, 132, 768, … chemical storage
   - B-1885/1886 – mixing flow to sump/settling basins
   - B-3296 – cooler
   - B-133 – assembling, red phosphorus (Mk58, Mk32, Mk38); pressing (floor was kept wet & ran to sump) This sump was refurbished but remained in the same location
   - B-1884 (metal plating) – degreasers [TPR or TCE (trichloroethylene)?]; switched from cyanide to cadmium then cadmium to zinc
   - B-122 – power coating, former flare assembly building
   - B-123 – machine shop; quench bath/solvent/degreasers (unknown type/oils)/oils
   - B-2697/2698 (flare assembly) – B-2697 paint booths?; B-2698 TDI (toluene diisocyanate) or MDI [di(phenylisocyanate) de methylene] based polymer system (toluene, diisocyanate; die casting)

2. **PPA history:**
   a) What operations / processes have occurred / are occurring at PPA?

   b) What materials / wastes were handled / managed in and around PPA [Buildings 122, 125 (includes 123 and 124), 126, 130, 133, 1884, 1885, 1886, and 2698]?

   c) What materials were handled / transferred at the pumphouse northwest of Building 126 (Building 2854)?

   d) What operations / processes occurred at the seasoning houses northwest of Building 2692?

3. **What were the disposal practices for processes at PPA?**

4. **Location of current / former underground process or waste (Figure Doug Johnson):**
   a) Piping (wastewater, storm water, process, etc.),

PAA Interview Questionnaire (cont.)
Associate: Date:

5. **Location of current / former underground process or waste (Figure Doug Johnson):**
   Sumps (vats, catch basins, separators, trenches, USTs, etc.)
   - Underground steel sump (settling basin) on the Building 122 North side adjacent to Paint Spray Machine.
   - Tanks between Buildings 122 & 123 (constructed ~1971)
   - Ditch along south and northwest corner of Building 1886
   - Ditch ~8'-0" from north wall of Building 126
   - USTs south of Building 1886 may have collected floor drains from building
   - USTs southeast of Building 2696 collected floor drains from Building 130

   b) Floor drains,
   - Building 1884 (exit under south-central wall)
     - -2698 had TDI waste materials
   - Building 125 (exit under east-central wall by "new degreaser" / "new metal platform"
   - Location of Storm Sewer that connects to Building 1884 drains
   - Location of downspout that connects to Building 126 drains
     - -2698 had TDI waste materials

   c) Lagoon / pits
   -

   d) Spills / emissions / discharges / releases (date, location, material)
   -

   e) Pyrotechnic test area burn area
   -

   f) Environmental work (spill responses, discharge cleanups, scrap cleanouts, UST removals, etc.)
   -

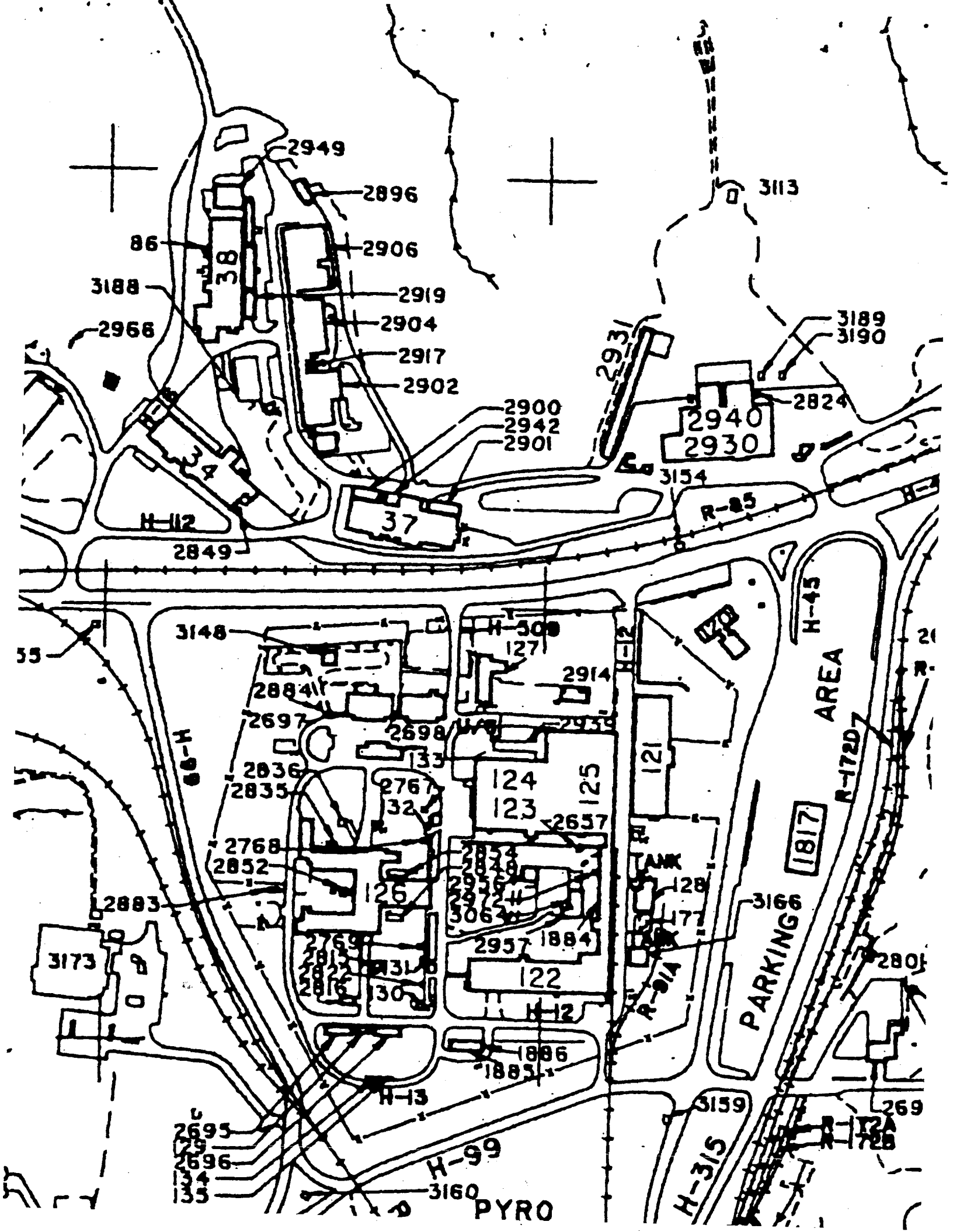
6. **Location of current / former process boilers (Figure 1-_____):**
   a) Building 123

7. **Location of current / former process cooling towers (Figure 1-_____):**
   a) Building 124

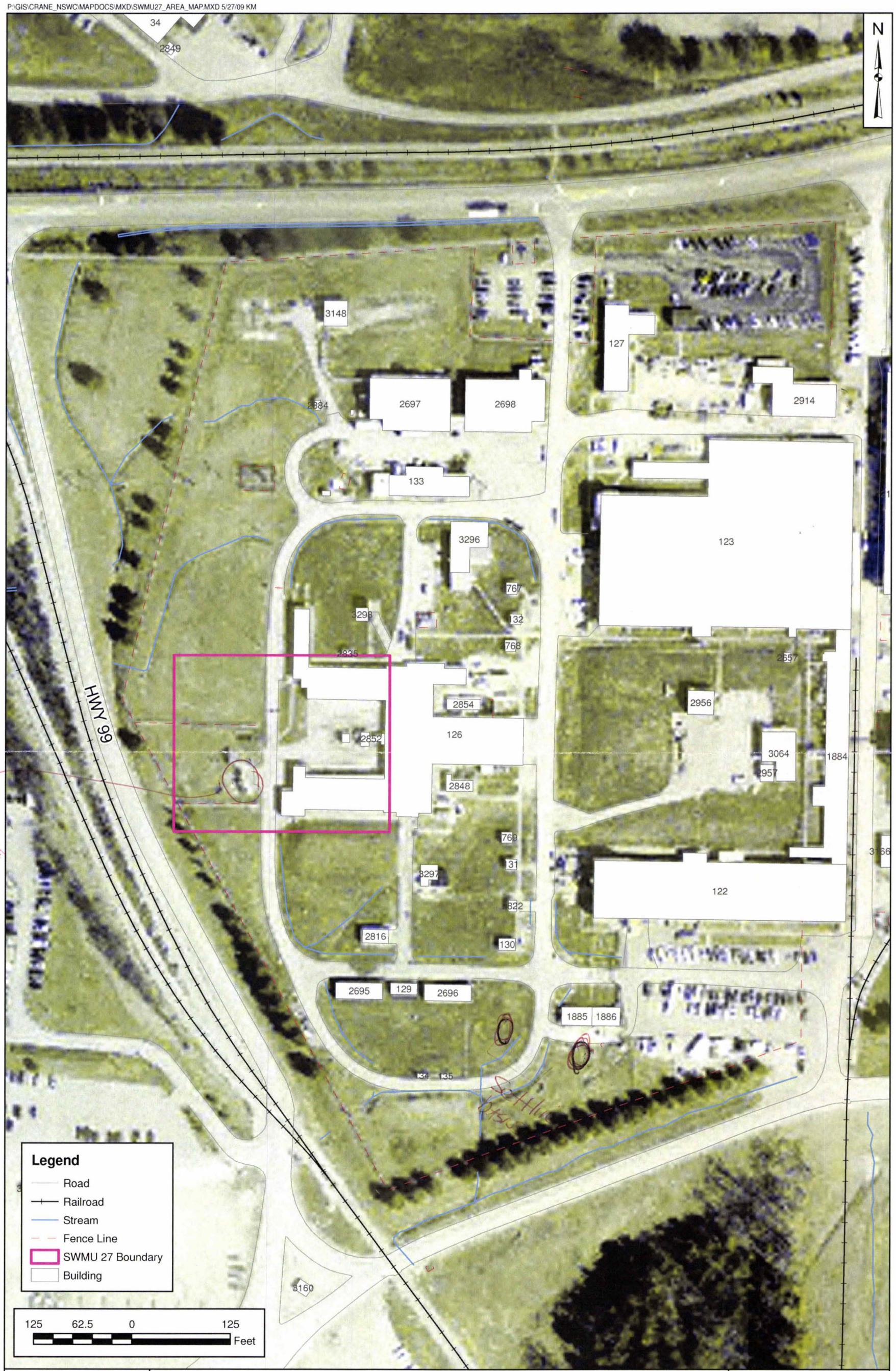
8. **Do you know the current / former types of waste generated?**
   a) Type of wastes generated
   - B-2698 had TDI waste materials

   b) PCB / oil transformers (sampling records / documentation regarding transformers removal)
   - Northwest corner of Building 124 (replaced/closed in 1981)
   - West of Building 133 roadway
   - Northeast of Building 126 north-central corner (Due south of Building 133)
     - Greater than or equal to 50 parts per million (ppm) PCBs (TSCA Regulated)
     - Less than 50 ppm (not generally TSCA regulated, although some requirements apply)

9. **Additional Comments / Notes:**


2949
2896
86
2906
3188
2919
2904
2966
2917
2902
2900
2942
2901
2931
3113
3189
3190
2824
2940
2930
3154
R-85
34
37
H-112
2849
3148
H-309
127
2914
2884
2697
2698
2939
133
2836
2835
2767
32
124
123
125
121
2657
2768
2852
2883
126
2834
2848
2936
2972
3063
128
177
3166
2769
2813
2822
2816
131
30
2957
1884
122
3173
H-12
R-914
1886
1885
H-13
3159
2695
29
2696
134
135
3160
H-99
PYRO
H-315
H-45
AREA
R-172D
1817
PARKING
2801
269
R-172A
R-172B

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU27_AREA_MAP.MXD 5/27/09 KM



Scrap pad (A2 waste storage) #3 TO take on back side

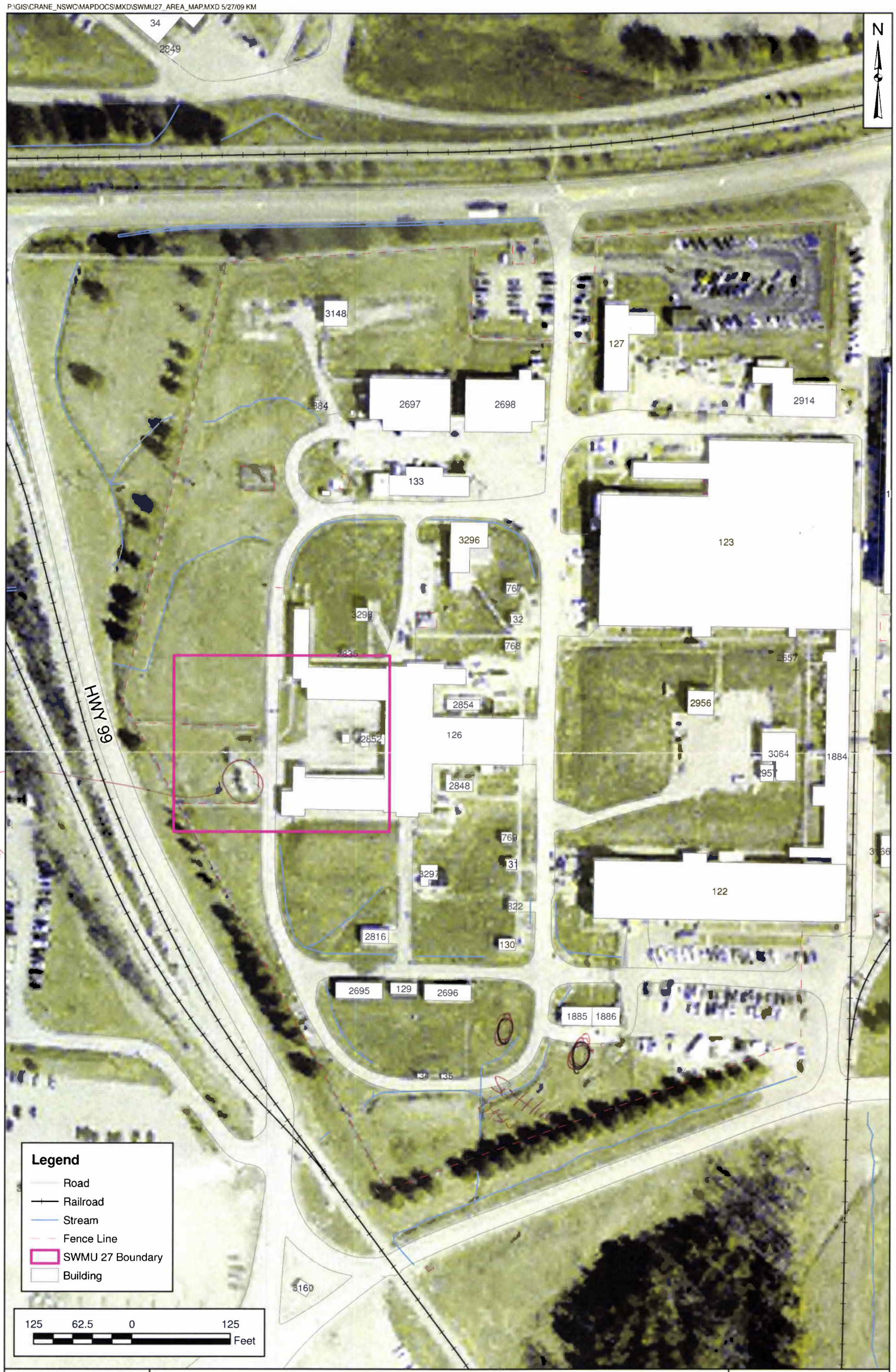
DRAWN BY K. MOORE	DATE 5/28/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO F276	
CHECKED BY R. BARRINGER	DATE 5/28/09	SWMU 27 - ILLUMINANT BUILDING 126 NSWC CRANE CRANE, INDIANA	APPROVED BY —	DATE —
REVISED BY —	DATE —		APPROVED BY —	DATE —
SCALE AS NOTED			FIGURE NO. FIGURE	REV 0

Doug Johnson

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU27_AREA_MAP.MXD 5/27/09 KM

DRAWN BY K. MOORE	DATE 5/28/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO F276
CHECKED BY R. BARRINGER	DATE 5/28/09	SWMU 27 - ILLUMINANT BUILDING 126 NSWC CRANE CRANE, INDIANA	APPROVED BY — DATE —
REVISED BY —	DATE —		APPROVED BY — DATE —
SCALE AS NOTED			FIGURE NO. FIGURE / REV 0

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU27_AREA_MAP.MXD 5/27/09 KM



Scrap pad (A2 waste storage) #3 FO take on back side

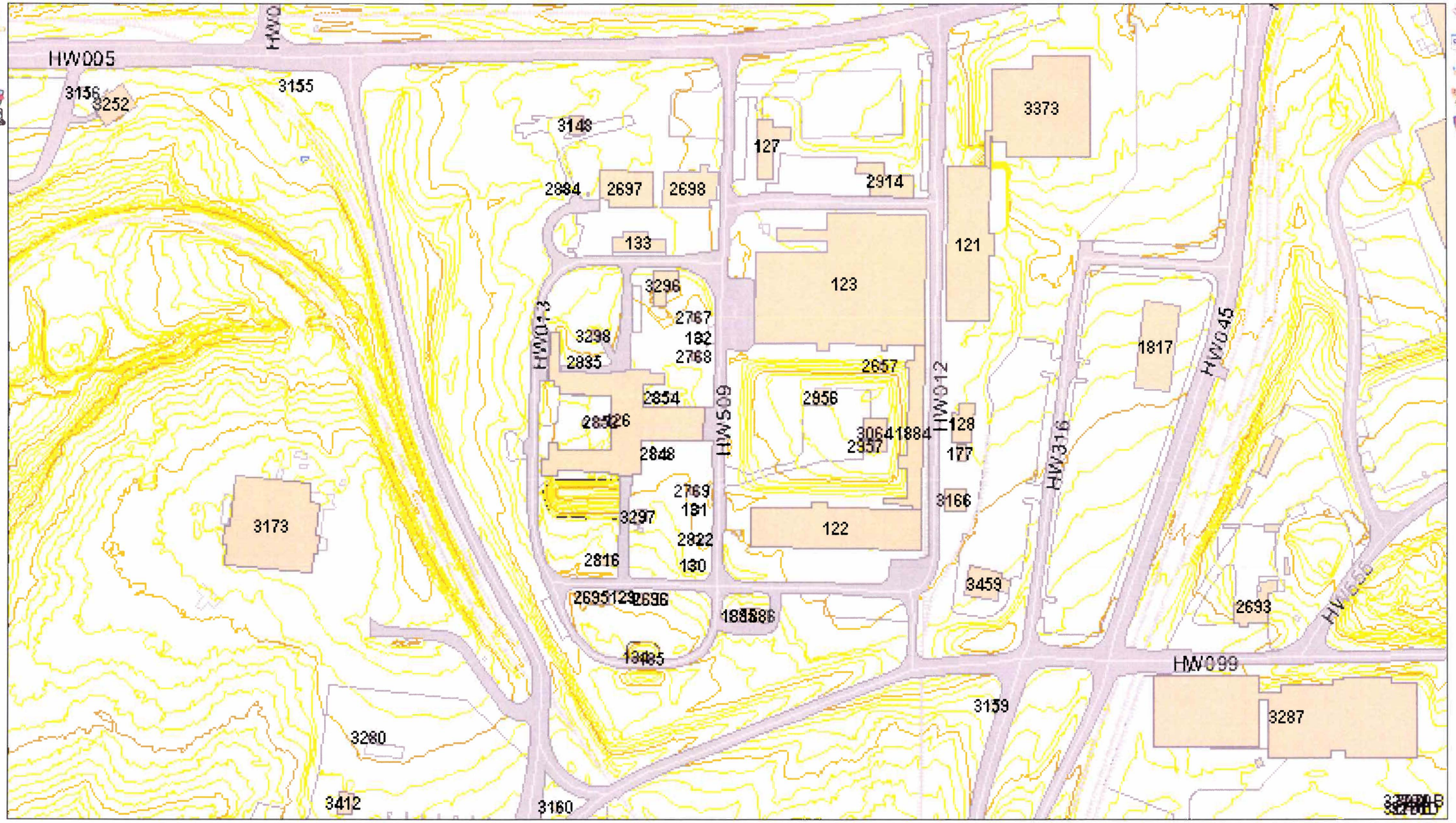
DRAWN BY K. MOORE	DATE 5/28/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO F276
CHECKED BY R. BARRINGER	DATE 5/28/09	SWMU 27 - ILLUMINANT BUILDING 126 NSWC CRANE CRANE, INDIANA	APPROVED BY — DATE —
REVISED BY —	DATE —		APPROVED BY — DATE —
SCALE AS NOTED			FIGURE NO. FIGURE / REV 0

Doug Johnson

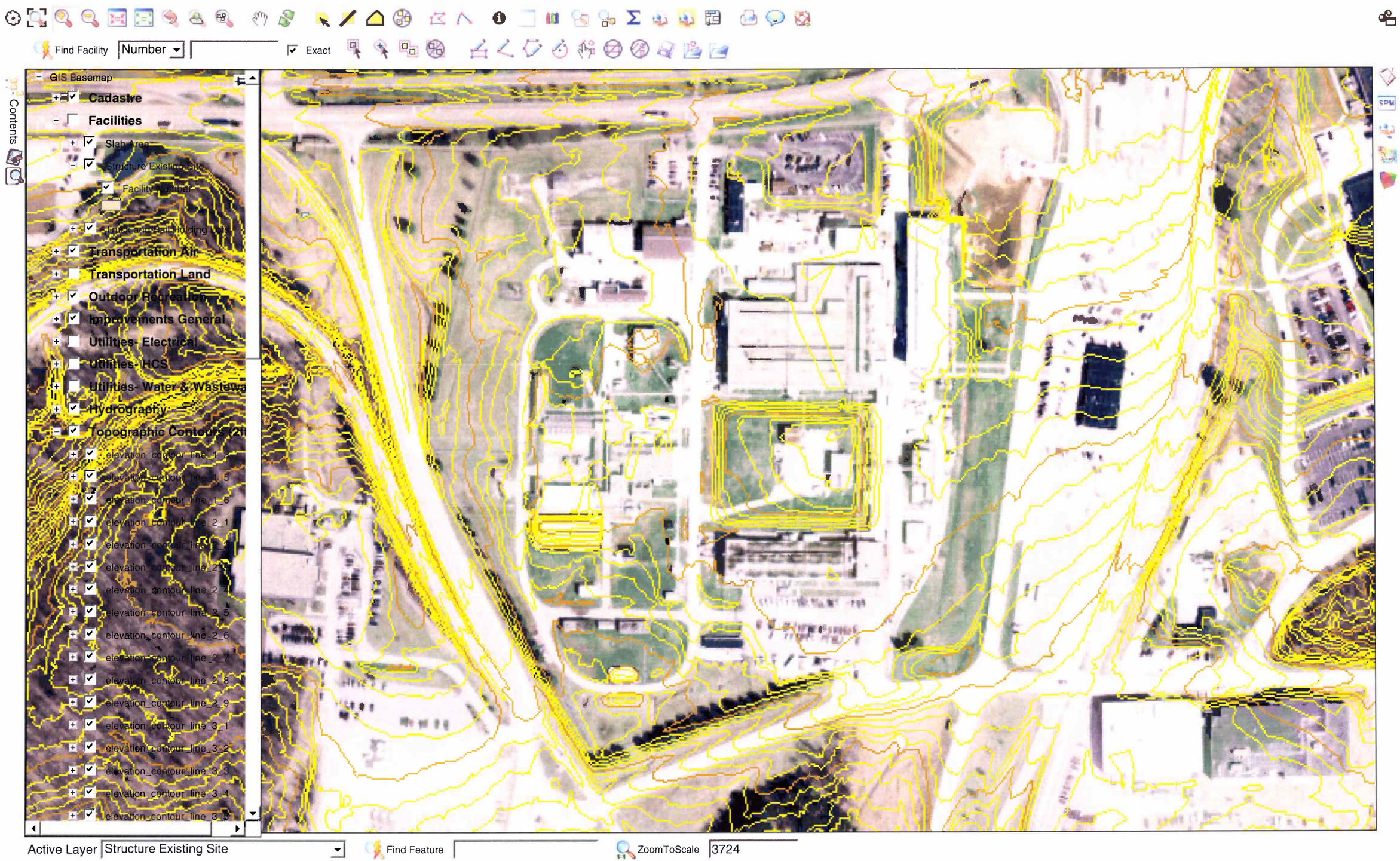
P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU27_AREA_MAP.MXD 5/27/09 KM

DRAWN BY K. MOORE	DATE 5/28/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO F276
CHECKED BY R. BARRINGER	DATE 5/28/09	SWMU 27 - ILLUMINANT BUILDING 126 NSWC CRANE CRANE, INDIANA	APPROVED BY — DATE —
REVISED BY —	DATE —		APPROVED BY — DATE —
SCALE AS NOTED			FIGURE NO. FIGURE / REV 0

Find Facility Number Exact



Active Layer Structure Existing Site Find Feature ZoomToScale 3724


Find Facility
Number
Exact
GIS Basemap
Cadastre
Facilities
Slab Area
Structure Existing Site
Facility Number
Transportation Air
Transportation Land
Outdoor Recreation
Improvements General
Utilities- Electrical
Utilities- HCS
Utilities- Water & Wastewa
Hydrography
Topographic Contours (2
elevation_contour_line_1_5
elevation_contour_line_1_6
elevation_contour_line_2_1
elevation_contour_line_2_2
elevation_contour_line_2_3
elevation_contour_line_2_4
elevation_contour_line_2_5
elevation_contour_line_2_6
elevation_contour_line_2_7
elevation_contour_line_2_8
elevation_contour_line_2_9
elevation_contour_line_3_1
elevation_contour_line_3_2
elevation_contour_line_3_3
elevation_contour_line_3_4
elevation_contour_line_3_5
Contents
Active Layer
Structure Existing Site
Find Feature
ZoomToScale
3724

Find Facility Number 1966 Exact

Contents

Active Layer Structure Existing Site Find Feature ZoomToScale 3724

ATK 1987

The following is a summary of the SWMUs and other areas of concerns identified during the PR and VSI. Units not observed during the visual site inspection are noted by an asterisk.

1. Old Burning Pit
2. McComish Gorge Dump
3. FS Smoke Storage Facility
4. Rockeye Loading Area Sumps
5. Rockeye Percolation Site
6. Rockeye Loading Area Wastewater Treatment Unit - Building 3004
7. Waste Carbon Container Storage Area Outside of Building 3004
8. Paint Waste Container Storage Area at the Rockeye Loading Area
9. Active Solid Fill Site
10. Old Solid Fill Site
11. Above-ground Waste Oil Storage Tank - Building 2801
12. Classified Papers Incinerator - Building 45

*13. Building 136 Sump

*14. Wastewater Treatment Unit (lead) - Building 136

15. Wastewater Treatment Unit - Plating Shop-Building 3064
16. Drum Storage Area behind Building 3064
17. Battery Shop Dump Building 36
18. Acid Neutralization Pit

*19. PCB Burial Area - (Pole Yard)

20. Roads and Grounds Dump Area

21. PCP Dip Tank

22. Building 126 Dump

*23. Dust Collectors - Building 126

24. Scrap Storage Area Behind Building 126

*25. Red Phosphorous Building Sump - Building 1886

26. Building 133 Sump

27. Railroad Maintenance Shop Wash Area Building 7

28. Railroad Maintenance Shop Oil/Water Separator

29. Auto Maintenance Shop - Building 1820

30. Heavy Equipment Maintenance Shop - Building 1818

31. Truck Wash Area at the Heavy Equipment Maintenance Building

32. Oil/Water Separator at the Heavy Equipment Maintenance Building

33. Outside Truck Wash Rack Adjacent to Building 1818

34. Roll - Off Boxes Outside Building 1820

35. CONEX Hazardous Waste Transfer Containers behind Building 1820

36. Oil Pan Wash Out/Disposal Rack Adjacent to Building 1820

37. Underground Waste Oil Storage Tank -- Building 1818

38. Underground Waste Oil Storage Tank -- Building 1820

*39. Mechancial Maintenance Shop -- Building 56

*40. 400 Gallon Solvent Storage Tank (Building 56 Paint Shop)

*41. Red Phosphorous Mixing Emergency Sump

*42. Booster Area - Lead Azide Sumps - Building 106 and 107

43. Wastewater Treatment Unit Building 3074

44. Lead Azide Loading Pond

45. Open Storage Area Outside Building 2801

*46. Cast High Explosives Fill (Building 146)

47. Central Storage Facility

48. APE 1236 Incinerators Building 146

49. Prototype Incinerator Building 146

50. Defense Reutilization and Marketing Office (DRMO) Hazardous Waste Storage Area

51. DRMO Storage Lot

*52. Paint Shop Building 2889

*53. Load and Fill Area -- Buildings 105, 198 and 200

*54. Wastewater Treatment Unit Building 160

55. Wastewater Treatment Unit - Sewage Plant - Building 3049

56. Old Sludge Drying Beds

57. Sewage Sludge Vacuum Truck and and Discharge Pad

58. Demolition Range

59. Waste Explosive Storage Area at the Demolition Range

60. Pest Control Area Building 2189

61. Former site of Waste Oil Underground Storage Tank near the Pest Control Area

62. Drum Storage Area Adjacent to SWMU #61

63. Rifle Range

64. Sedimentation Pond #1

65. Mine Fill A

66. Mine Fill B

*67. Wastewater Treatment Unit - Building 104

68. Explosive D Wash Areas

*69. Load and Fill Area Sumps Building 104

*70. Steam Out Unit - Building 160

71. Highway 58 Dump Site A

*72. Storage Tanks - Building 160

*73. Bomb Proof Group

74. DRMO Salvage Yard

*75. Heavy Equipment Storage Area - Building 2189

76. Sanitary Sewer System

*77. Turkey Creek Quarry

78. Highway 58 Dump Site B

79. Ammunition Burning Grounds (ABG)

80. Sludge Dewatering Units (ABG)

81. Underground Storage Tanks (ABG)

82. Ash Pile (ABG)

83. Ash Roll-off Boxes (ABG)

84. Open Burning Area (ABG)

85. Dye Burial Grounds

86. Sanitary Landfill

87. Sanitary Landfill Leachate Collection Ponds

88. Sewage Sludge Land Application Area (Landfarm)

89. PCB Storage Facility

90. Sedimentation Ponds 2, 3, and 4

91. Pyro Test Area (Ordnance Testing Area)

*92. Test Track

*93. Composition Testing Facility - Decontamination Building 181

94. Rocket Range

95. Restored Mustard Gas Burial Area

96. Temporary Flammable

*97. Metal Shavings Collection Area

*98. 500 Gallon Tank Wagon

*99. 3-Ton Pump Truck

100. Settling Pond behind Building 106

OTHER AREAS OF CONCERN

A. Underground Diesel Fule/Gasoline Tanks
B. Stream East of McComish Gorge
C. White Phosphorous to Phosphoric Acid Production Building

*Units not observed during the VSI

22. UNIT NAME: Building 126 Sump (Ref. 3, p.6--3)

Unit Description: This unit is a below-grade concrete sump that receives rinse waters from production rooms in Building 126 via individual drains and a central connector. This rinse water is generated from the washing of grit screens and empty paint barrels (Ref. 20). During the VSI, cracks were observed in the tank, and the retaining wall had deteriorated.

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: The unit received rinse water containing phosphorous, manganese, and paint residues (Ref. 20).

Release Controls: The unit discharges directly to the sanitary sewer after solids settle to the bottom. Solids are removed and taken to the CSF (Unit 46).

Release History: Unknown.

UNIT 22. (Continued)

Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is moderate due to the relatively poor visual appearance of the sump.

Surface Water: The potential for release to surface water is low; the unit discharges to the sanitary sewer system.

Air: The potential for release to air is low due to the dilute nature of the wastes.

Subsurface Gas: The potential for generation of subsurface gas is low due to the nature of the wastes managed and the open design of the unit.

Suggested Further Action: Soil samples should be taken near the walls of the sump to determine if any contamination has occurred due to the cracks in the sump.

*23. UNIT NAME: Dust Collectors - Building 126 (Ref. 3, p.6-3)

Unit Description: This unit collected explosive dust produced in Building 126. The unit used a wet vacuum pump. The collectors were emptied periodically and taken to the Ammunition Burning Grounds (Unit 79) (Ref. 3, p.6-3).

Date of Start-Up: Unknown.

Date of Closure: Unknown.

Waste Managed: The unit collected explosive dust that was trapped using water vapor.

Release Controls: Unknown.

Release History: Unknown.

UNIT 23. (Continued)

Conclusions:	This unit was not observed during the VSI. Insufficient information was available to develop conclusions regarding release potentials to the various pathways.
Suggested Further Action:	Obtain information on the age, design, and operation of this unit and on the disposition of any wastewater.

24. UNIT NAME: Scrap Storage Area Behind Building 126

Unit Description: This unit consists of a concrete pad with metal canopy. It is used for temporary storage of waste starter mix, fuel oil, and explosive contaminated metal parts. The wastes were observed to be in small containers or jugs during the VSI.

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: Various scrap products and salvageable parts are placed on the pad for disposal or renovation. The wastes include waste starter mix, fuel oil, explosive contaminated metal parts, and paint residues. During the VSI, three drums of unlabeled wastes (in good condition), three waste paint cans, and a 15-gallon above-ground fuel oil tank were observed.

Release Controls: The unit consists of a concrete pad protected by a metal overhang. There is no perimeter containment.

Release History: Unknown.

UNIT 24. (Continued)

Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is low due to the containerization of wastes and release controls employed at the unit.

Surface Water: The potential for release to surface water is low due to the containerization of wastes and the unit's distance from any local surface drainage.

Air: The potential for release to air is low due to the containerization of wastes.

Subsurface Gas: There is no potential for the generation of subsurface gas due to the design of the unit.

Suggested Further Action: No further action is suggested for this unit at this time.

UNIT 25. (Continued)

Conclusions:	This unit was not observed during the VSI. Insufficient information was available to develop conclusions regarding release potentials to the various pathways.
Suggested Further Action:	Obtain information on the operation, period/frequency of use, and integrity of the unit.

26. UNIT NAME: Building 133 Sump (Ref. 3, p.6-8)

Unit Description: This sump receives wastewater from the red phosphorous candle pressing house (Ref. 20). The operation uses red phosphorous, manganese dioxide, magnesium, zinc oxide, and linseed oil within a totally wet operation. The sump consists of three separate concrete chambers that are approximately 6 ft. (l) x 2 ft. (w) x 3 ft. (d).

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: Rinsate water contaminated with red phosphorous is allowed to settle in the sumps prior to pumping the supernatant into the Sanitary Sewer System.

Release Controls: Accumulated sludge is hauled to the Sludge Dewatering Units (Unit 80) at the ABG (Unit 79).

Release History: Unknown.

UNIT 26. (Continued)

Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is low due to the apparent good condition of the concrete sumps.

Surface Water: The potential for release to surface water is low because rinse water is allowed to settle prior to discharge to the Sanitary Sewer System.

Air: The potential for release to air is low due to the nature of the wastes.

Subsurface Gas: The potential for generation of subsurface gas is low due to the open design of the unit and the nature of the wastes handled.

Suggested Further Action: No further action is suggested for this unit at this time.

DISPOSITION FORM

For use of this form, see AR 340-15; the proponent agency is TAGO.

REFERENCE OR OFFICE SYMBOL	SUBJECT
SARCN-EDP	Procedures for Cleaning/Pumping of Hazardous Wastes in Sumps in Pyro Production Area

TO NWSC 097 FROM SARCN-ED DATE 15 June 1982 CMT 1

Mr. Hubble/mjh/1353

To avoid confusion and potential safety/enviromental problems, the following procedures are requested to be followed in reference to transfer of the hazardous wastes from the sumps in the pyro production area to the ammunition burning grounds:

a. Only the pyro production area supervisor will initiate arrangement with code 097 to have the transfer made on a specified day.

b. Upon arrival at the pyro production area, code 097 will report to the pyro office trailer before initiating the transfer itself.

c. The pyro production area supervisor will notify code 0924 and the Ammunition Burning Grounds of the pending transfer of the hazardous waste.

STAN ARMSTRONG
Acting Director, Engineering Directorate

CF:
SARCN-IO
NWSC 0924
SARCN-IOA-G (R. Bechtel)
SARCN-MMD (R. Bland)

DISPOSITION FORM

file – Pyro Produc. area

For use of this form, see AR 340-15; the proponent agency is TAGO.

REFERENCE OR OFFICE SYMBOL
NWSC-041

SUBJECT
Contamination of Buildings 1885 and 1886 by Compositions Containing Red Phosphorus

TO THRU NWSC 104 concur 10 Oct 1985 FROM NWSC 041 DATE 9 October 1985 CMT 1
Mr. Hampton/mm/1667

TO SMCCN-IO

1. On 4 October 1985, Public Works Department personnel were performing repair work on building 1886. The work consisted of removal of exterior siding from the building and replacement of siding and studs in portions of a wall. During removal of the siding, a fire was initiated in the wall of the building. The personnel at the scene when the fire was initiated in the wall were unsuccessful in their efforts to extinguish the fire using a locally available water hose because the fire was located in a confined space within the wall and they could not get a sufficient volume of water directly on the burning surfaces. The fire was extinguished by Fire Department personnel who were equipped to apply a sufficient quantity of water. Damage to the structure was minor, the fire burned in the composition that was deposited on the surface of the wood frame construction.

2. During the investigations it was found that there is a significant amount of contamination with pyro composition containing red phosphorus in buildings 1885 and 1886. Loose powder has settled on surfaces inside the mixer rooms, on the exhaust fans and ducts and in the door frames and composition has infiltrated cracks in the interior walls so that the interior of the frame construction walls is contaminated. The nature of the composition is such that it is easily ignited by friction or hot particles. The deposited composition is a loose powder that is easily ignited and burns rapidly when ignited. When the loose powder is ignited fire rapidly evolves from an initial stage of smoldering with no visible flame to visible flame that spreads rapidly through the material. Incidents of ignition in the areas where the composition is handled are relatively frequent and usually from various mechanical effects such as friction and impact. This incident occurred when an employee pulled a board loose from the frame of the building. A piece of wood fell into the space within the wall and the employee noticed smoke rising from the space. Flames evolved very rapidly. Incidents are common when cutting candles to length during production of candles containing the composition and fires are common during pressing of candles from friction or impact of scoops used to transfer the loose material to presses, or from impact of containers against tables during other operations.

3. Observation of the degree of contamination, the combustible construction materials, and the operations performed in buildings 1885 and 1886, indicates that a high probability exists of igniting the composition contaminating the inside surfaces of the buildings, ceilings, top surfaces of pipes and the interior of the frame construction walls and that if ignited, the composition is sufficiently hidden in the interior of the walls and above the ceiling that the existing fire suppression equipment would not be effective in extinguishing the burning composition. Hazard from fire to personnel performing operations in buildings 1885 and 1886 is significant. The combination of the degree of contamination, the small size of the rooms, the quantity of material contained in the mixers, the ease of ignition of the composition and the rapid spread of the fire once ignited could rapidly lead to total involvement of a mixer room if a fire occurred. One characteristic of the composition

contaminating the buildings is that it tends to progress relatively slowly from initial ignition to rapidly spreading flames. But this characteristic is negated in these buildings by the loose, powdery nature of the composition that contaminates the building. The material becomes involved rapidly and flame spreads quickly in the loose composition. The fire suppression system reacts slowly and may not reach areas where the composition rests on pipes or has migrated into the interior of the walls. Personnel handling containers or moving other items in the mixer rooms in buildings 1885 and 1886 could strike a contaminated surface and start a fire. A blow against the door frame while carrying material in or out of the room could ignite material contaminating the door area or impact against a wall, pipe or conduit could initiate a fire.

4. The condition of the building is in violation of explosive safety standards. OP 5, VOL 1, 3.1.1.5.a requires good housekeeping with elimination of dusts, and cleaning of working surfaces and areas to eliminate fire hazards. OP 5, VOL 1, 6.3.1.2 requires that the interior surface finish of walls in process buildings be smooth and free of cracks and crevices with joints sealed to prevent intrusion of hazardous materials. The interior finish is supposed to be of a type that will minimize the impregnation of construction materials by hazardous material used in the processes. OP 5, VOL 1, 3.1.1.5.e states that "explosive dust or other hazardous material shall not be allowed to accumulate on structural members or other surfaces, including radiators, heating coils, ventilating ducts, steam and hot water pipes, or electrical fixtures. Spilled explosives must be removed promptly. Cracks or crevices where explosives may accumulate must be eliminated. Window ledges should be sloped where practicable to reduce the accumulation of explosive dust." DARCOM 385-100, chapter 5, has essentially identical guidance. The fire suppression system in the buildings is antiquated and does not comply with MIL-HDBK-1008, 4.4.2. The system does not provide rapid response personnel protection from thermal effects.

5. It is recommended that buildings 1885 and 1886 not be used for operations until the combustible construction has been replaced with non-contaminated construction so arranged that effective periodic clean up of the phosphorus composition can be performed and the migration of the composition into hidden non-accessible areas of the structure prevented. The hazard from the combination of poor structural integrity and degree of contamination in these areas is such that continued operation in these areas presents a high probability of facility damage/loss and a somewhat lower probability of personnel injury.

D. L. Groh

D. L. GROH
Safety Director

CF: SMCCN-CO, SMCCN-ED, SMCCN-SF

A.3

JUNE 2010 SITE VISIT INFORMATION

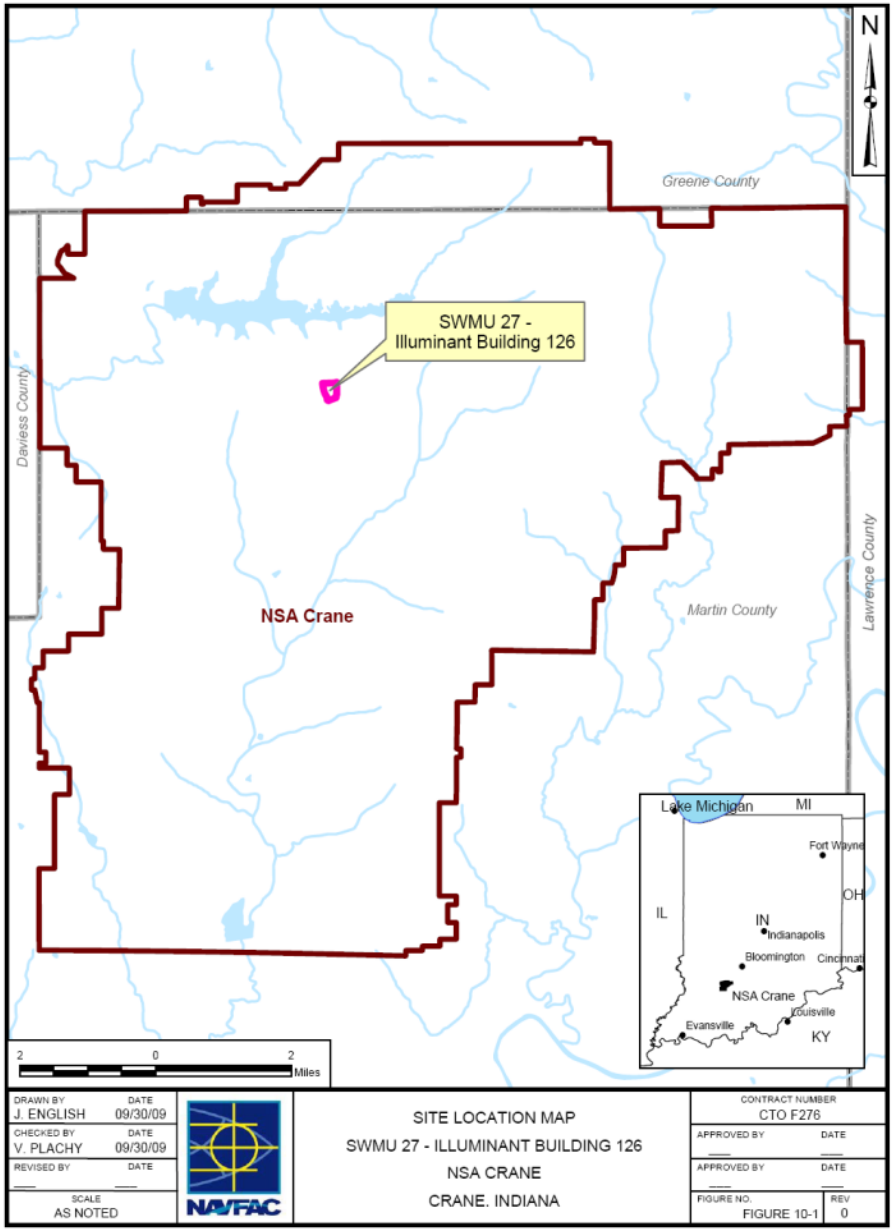
Solid Waste Management Unit 27 Pyrotechnics Production Area (PPA) RCRA Facility Investigation Project Data Needs and Objectives Planning

Naval Support Activity (NSA) Crane, Crane, Indiana

Introductory Presentation/Planning Session Part 1

June 2010

Naval Support Activity Crane Installation Map



SWMU 27 – Pyrotechnic Production Area (PPA) Building 126 – Illuminant Building

- The Illuminant Building 126 is classified as an IR site by the Navy.
- According to descriptions of this IR site, red phosphorus was discharged to sump pits where it used to overflow to tributaries of Boggs Creek.
- There has also been heavy metal contamination from zinc and cadmium identified in open ditches that eventually drain into Boggs Creek.
- No remedial investigation studies have been performed for this IR site; therefore, it is difficult to know the extent and location of potential contamination.
- An outdoor burning area was located near Building 126 and another area north of Highway 5. Open burning operations ceased in 1971.

Project Background for CTO F276 – RFI for SWMU 27

Site History From the Navy's NORM (normalized) database (an internal environmental tracking database of contamination problems at Navy installations):

- Contamination at this site included red phosphorus, chlorate, dyes, oxidizers, and fuels for flares and smoke munitions.
- The building used sump pits that were pumped out by trucks and taken to the burning grounds.
- All sump overflows drained into the Boggs Creek watershed.
- A metals plating shop that has utilized heavy metals, caustics, acids, and cyanide-based processes is present at the site.
- Significant heavy metal contamination (zinc and cadmium) has been experienced from wastewater being discharged into open ditches.
- There are also burn areas; one behind Building 126 and one across Highway 5.

Project Background for CTO F276 – RFI for SWMU 27

Site History from NAVFAC:

- Red Phosphorous is the most significant contaminant with lesser chlorates, dyes, oxidizers and fuels for flares and smoke munitions.
- The building (Building 126) used sump pits that were pumped out by vacuum trucks and taken to the burning grounds.
- All sump overflows drained into the Boggs Creek watershed.
- A metal plating shop that has utilized metals, caustics, acids, and cyanides is also present and significant heavy metal contamination (zinc and cadmium) has been experienced from wastewater being discharged into open ditches.
- In the late 1970's, a pre-treatment facility was constructed for the aforementioned plating shop. It is currently in use and treated wastewater is discharged to the sanitary sewer system under an NPDES permit.
- There are also burn areas that were used years ago; one behind Building 126 and one across Highway 5. Burning at those locations ceased in 1971.

Previous Environmental Inspections and Investigations - NSA Crane PPA IAS Report - 1983

- Initial Assessment Study (IAS) of Naval Weapons Support Center Crane, Indiana (Report #NEESA 13-003) prepared by the Naval Energy and Environmental Support Activity (NEESA), Port Hueneme, California, May 1983.

- The IAS report described the development of the Pyrotechnics Plant operations and facilities during the early 1940s and identified specific buildings, activities, and pyrotechnic production operations that included flares, illuminating projectiles, smoke signals, spotting charges, ground and aircraft signals, and incendiaries.

- Later PPA developments included chemical delay powders, self-releasing submarine signals, rescue flares, depth charge markers, aircraft signal cartridges, and parachute flares.

Previous Environmental Inspections and Investigations - NSA Crane PPA PR/VSI Report - 1987

- Preliminary Review / Visual Site Inspection (PR/VSI) report of the Naval Weapons Support Center Crane, Indiana (EPA ID IN5170023498) prepared by A.T. Kearney, Inc. for the U.S. Environmental Protection Agency Region V, March 1987.

- The PR/VSI report identified several elements within the PPA facility where pyrotechnic process rinse waters and sludges were managed (primarily in sumps and the sanitary sewer system) and other locations where pyrotechnic process wastes and residual materials were managed (scrap storage areas, explosive dust collectors, waste water treatment unit [Building 3074], etc.).

- PR/VSI report recommendations were to collect samples as necessary to determine if contamination releases had occurred.

Historical PPA Operations at NSA Crane

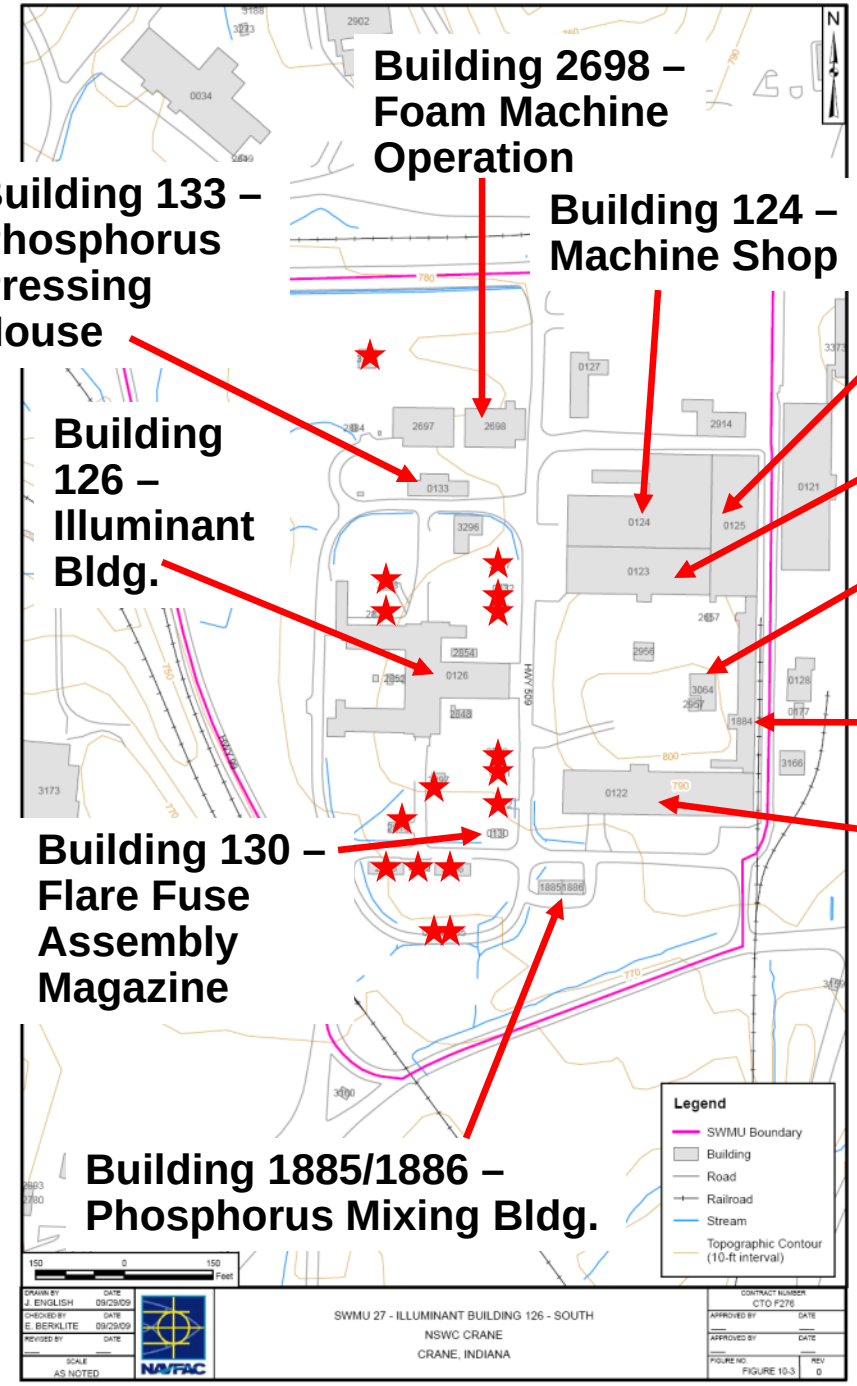
- The complex of buildings was designed and built during WWII for the exclusive purpose of loading, assembling and packing out star shell ammunition. The star shell, primarily a 5-inch illuminating round with a parachute, required a good deal of support facilities. The process flowed as follows:
  - Building 122 (second floor) was used for parachute fabrication.
  - Buildings 123, 124, and 125 were used for metal part generation (machining and assembly).
  - Building 126 (Illuminant Building) was used for the preparation of the illumination composition and its pressing into “candles”
  - Building 122 was the final assembly building where the candle (pyrotechnic package) was inserted into the projectile, the parachute was attached and packed and the whole round packed-out for rail shipment from the plant.
  - The host of smaller buildings that surround Building 126 housed chemical storage, candle curing, ready magazines and inert support warehouses that were necessary to maintain the process.

SWMU 27 – Pyrotechnic Production Area (PPA)

Chronology of PPA Activities at NSA Crane

- PPA Construction by U.S. Navy (1941-1942) included:
  - Building 130 – Flare Fuse Assembly Magazine,
  - Building 124 – Projectile Assembly and Machine Shop,
  - Building 122 – Flare Assembly and Storage Plant,
  - Building 126 – Illuminant Building
- Additional buildings were constructed in the PPA to support the war effort and later operations, including production of:
  - marker flares, rescue flares, parachute flares,
  - illuminating projectiles, incendiaries,
  - smoke signals, spotting charges,
  - ground and aircraft signals, submarine signals,
  - aircraft signal cartridges and depth charge markers

SWMU 27 – Pyrotechnic Production Area (PPA) Buildings



Building 125 – Machine Shop

Building 123 – Former Projectile Assembly

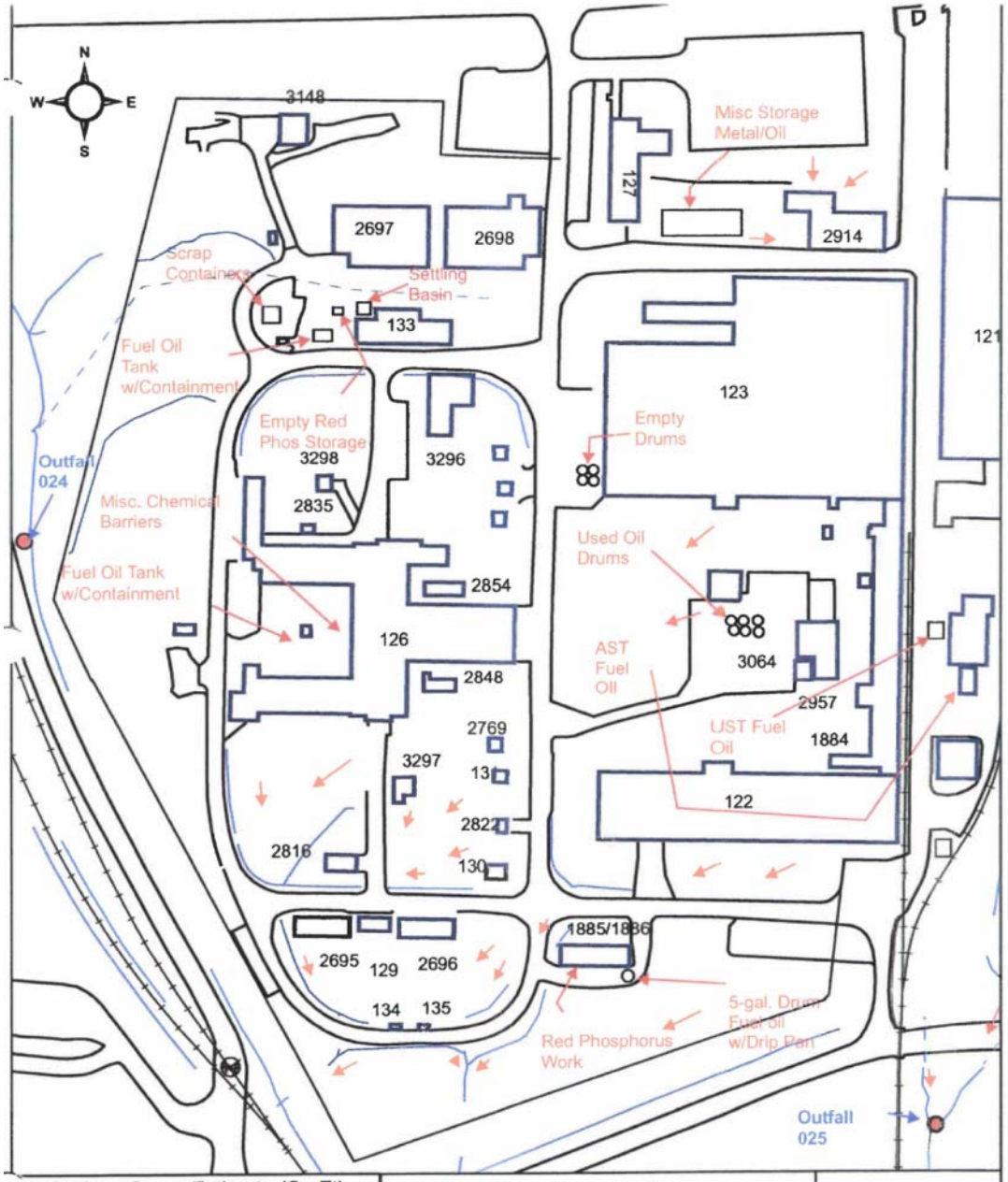
Building 3064/2957 – Wastewater Treatment Plant (includes Metal Plating Facility rinse waters).

Building 1884 – Metal Plating Operations

Building 122 – Former Flare Assembly Bldg. and Paint Booth / Urethane Foam Operation

Many smaller buildings in the PPA are locked storage warehouses and bunkers where raw materials used in pyrotechnic production were stored, mixed, or where pyrotechnic items were permitted to cure. These locations are noted on this map with a★.

Surface Water Flow and Drainages in SWMU 27



Surface Cover Estimate (Sq Ft)		
	Impervious	Pervious
024	146,362	341,512
025	148,105	148,105

Naval Support Activity
Crane, IN

PYRO AREA

0 100 200 400 Feet

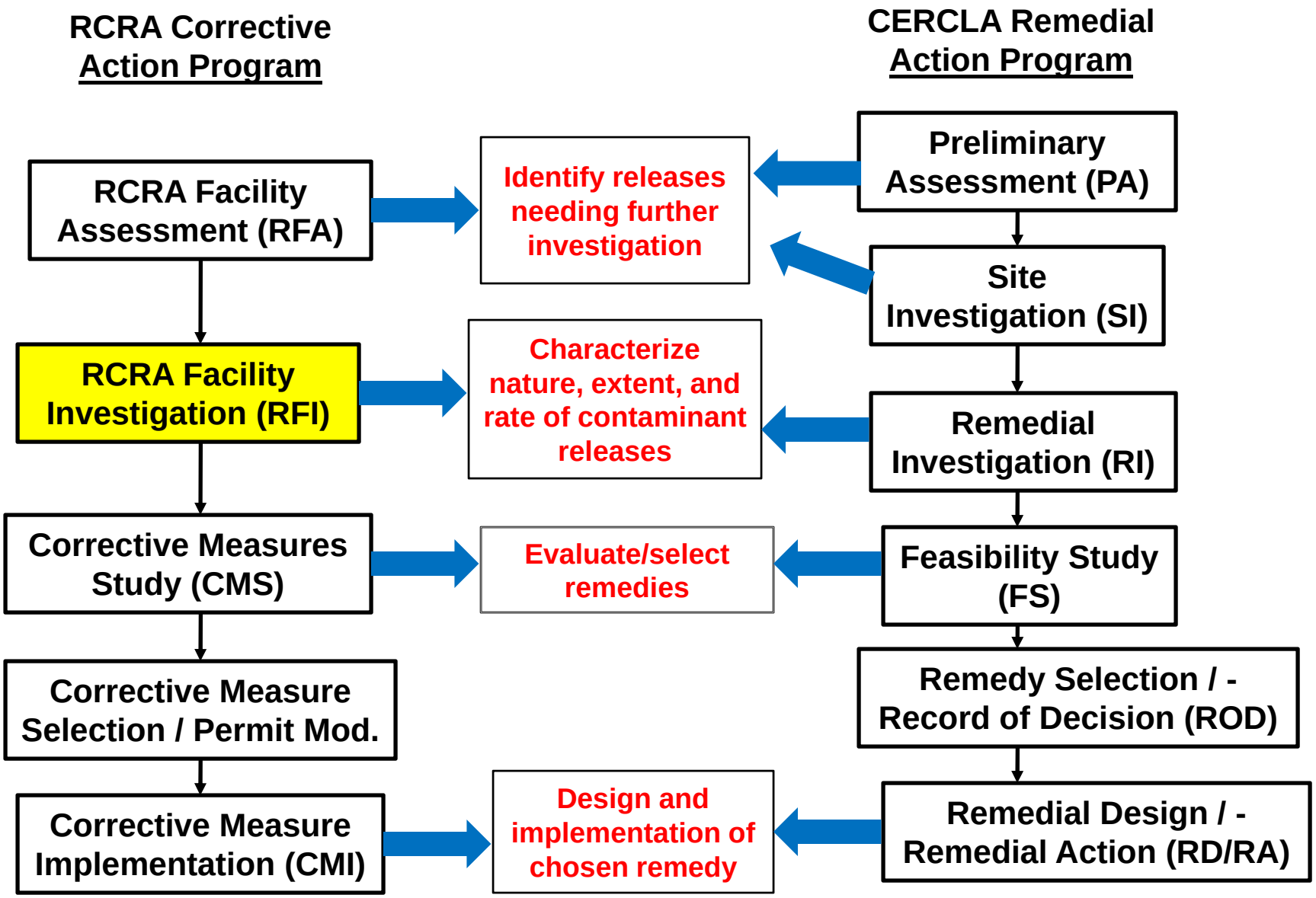
FIGURE
2.2.36

- Pyrotechnic production processes in the PPA included red phosphorus, chlorates, dyes, oxidizers, and fuels for flares and smoke munitions. Process wastewaters and rinsates were at one time discharged to open ditches and later to sumps that may have overflowed.
- The soil in areas adjacent to process sumps and sump pits that may have been impacted by sump overflows.
- The PPA drains to the south and west into the Boggs Creek watershed.

RCRA Corrective Action Process – RFI Work Plan

- The RCRA Facility Investigation is the second component of the overall site evaluation following an initial assessment.
- Builds upon information presented in the Initial Assessment Document, PR/VSI report, or RCRA Facility Assessment Report by collecting site-specific environmental data.
- The RFI work plan provides the basis for the entire investigation:
  - Work plan requirements are clearly specified
  - Establishes current site conditions at facility
  - Identifies procedures for:
    - Establishing environmental characterization
    - Monitoring and data / sample collection
    - Quality assurance / quality control
    - Data management procedures
    - Identification of potential receptors
    - Other information as needed.

Comparison of RCRA vs. CERCLA Processes



Objectives of the RFI Work Plan and RFI

- **Verify** any contamination from suspected releases
- **Characterize** the sources of releases and wastes associated with these sources
- **Gather** information on the environment at the facility affecting the migration and fate of releases
- **Assess** the nature, rate, and extent of releases
- **Determine** the need for interim and final corrective measures

Purpose of RFI for PPA at NSA Crane

- Build on information presented in the Initial Assessment Document and Preliminary Review / Visual Site Inspection Report by collecting site-specific environmental data.
- Perform field reconnaissance and surveys according to RFI Work Plan (UFP-SAP/QAPP).
- Outline potential sources (discharge areas, sump overflows, release areas, testing/operations areas, burning areas, etc.).
- Determine more accurate site boundaries for activities associated with the SWMU and delineate potential contaminant releases and migration pathways.
- Develop or expand on a Conceptual Site Model (CSM) using field reconnaissance/survey data and evaluate initial human health exposure hazards and risk screening results.
- Summarize information and recommend future site actions.

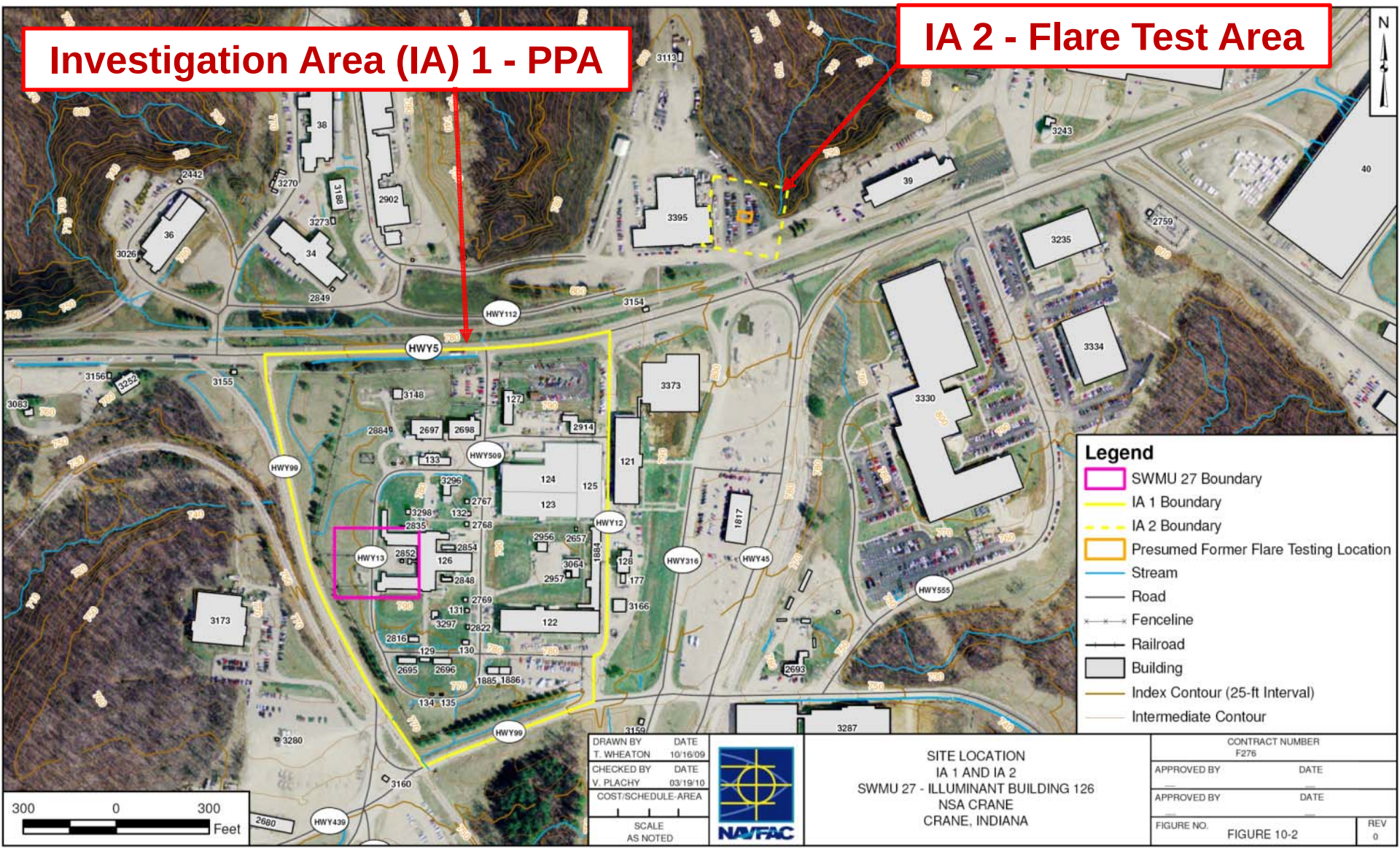
Solid Waste Management Unit 27 Pyrotechnics Production Area (PPA) RCRA Facility Investigation Project Data Needs and Objectives Planning

Naval Support Activity (NSA) Crane, Crane, Indiana

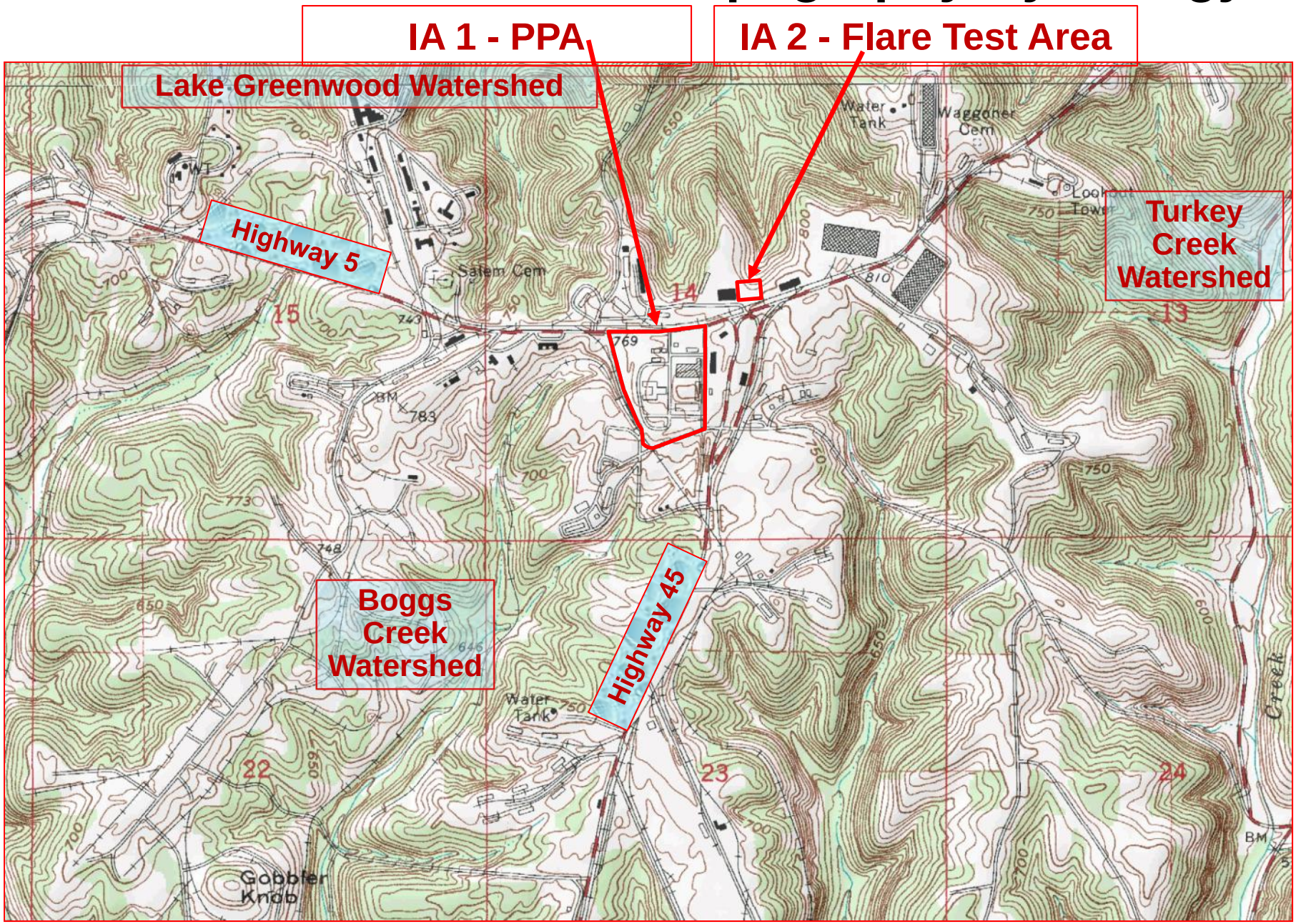
Introductory Presentation/Planning Session Part 2

June 2010

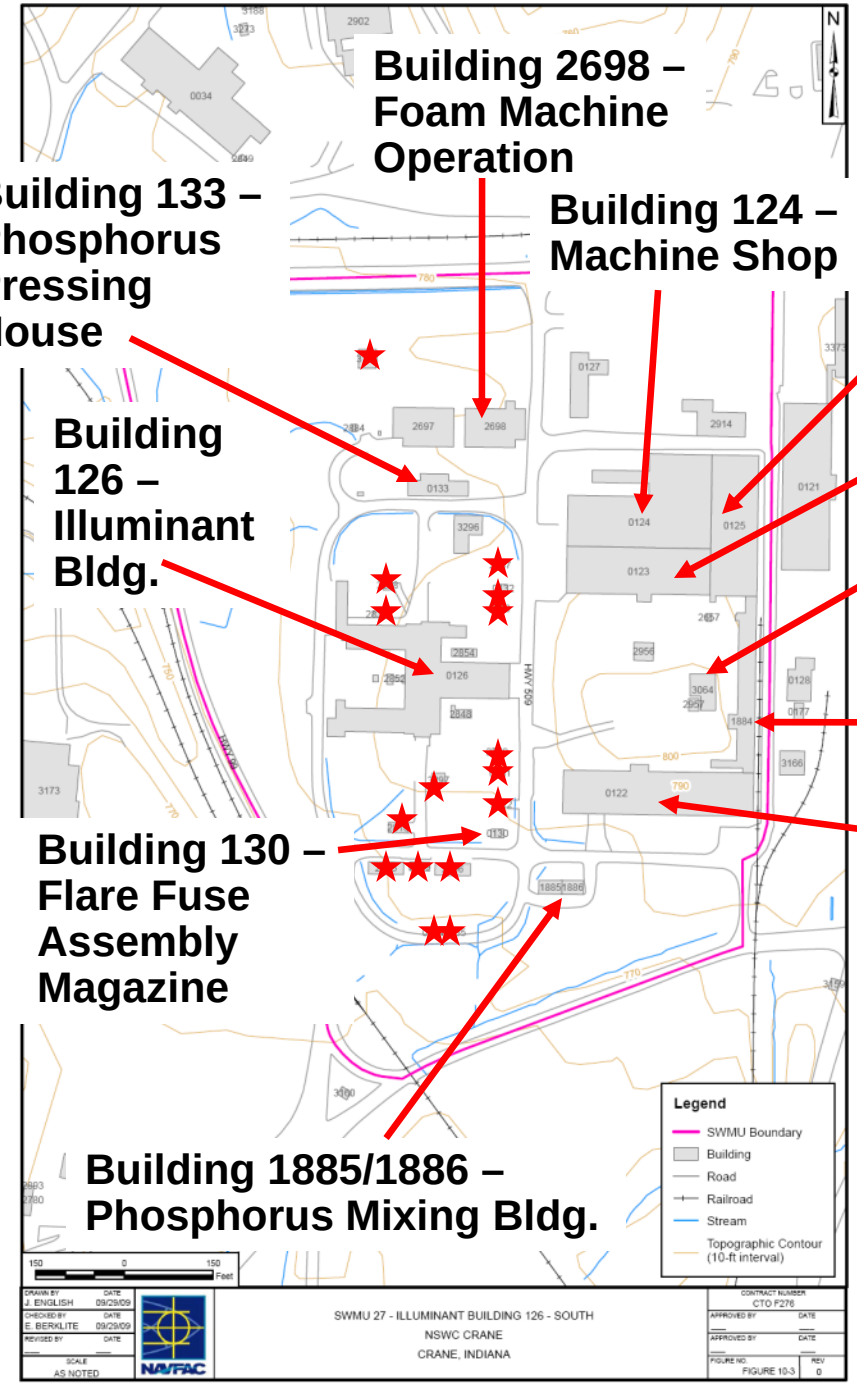
Crane SWMU 27 PPA RFI – Areas to be Investigated



NSA Crane PPA – Local Topography/Hydrology



SWMU 27 – Pyrotechnic Production Area (PPA) Buildings



Building 125 – Machine Shop

Building 123 – Former Projectile Assembly

Building 3064/2957 – Wastewater Treatment Plant (includes Metal Plating Facility rinse waters).

Building 1884 – Metal Plating Operations

Building 122 – Former Flare Assembly Bldg. and Paint Booth / Urethane Foam Operation

Many smaller buildings in the PPA are locked storage warehouses and bunkers where raw materials used in pyrotechnic production were stored, mixed, or where pyrotechnic items were permitted to cure. These locations are noted on this map with a★.

SWMU 27 – PPA storage warehouses and bunkers

Many smaller buildings in the PPA are locked storage cages, storage lockers, warehouses, and bunkers where raw materials used in pyro-technic production are/were stored, mixed, or where finshed items were permitted to cure.

Summary of Approach for CTO F276 – RFI for SWMU 27 (PPA) – Sumps and Ditches

- Pyrotechnic production processes in the PPA included red phosphorus, chlorates, dyes, oxidizers, and fuels for flares and smoke munitions. Process wastewaters and rinsates were at one time discharged to open ditches and later to sumps that may have overflowed.
- The soil in areas adjacent to process sumps and sump pits that may have been impacted by sump overflows will be evaluated to determine presence or absence and possible degree of potential releases. The PPA drains to the south and west into the Boggs Creek watershed.

Building 126 (Illuminant Building)

- Building 126 was designed with many separate work cells (↓) where the pyrotechnic ingredients were prepared, mixed, and then pressed into a final candle configuration. Because of the unique multi-cell design, Building 126 became attractive as a general purpose pyrotechnic loading building.
- Through the 1950's and 1960's, the building supported a number of illuminating candle processes. The Mk 24 and Mk 45 illuminating candles were produced in the building using an automated pressing line. From that time until now, the building continues to be used for supporting pyrotechnic illuminating rounds, smokes and signals.

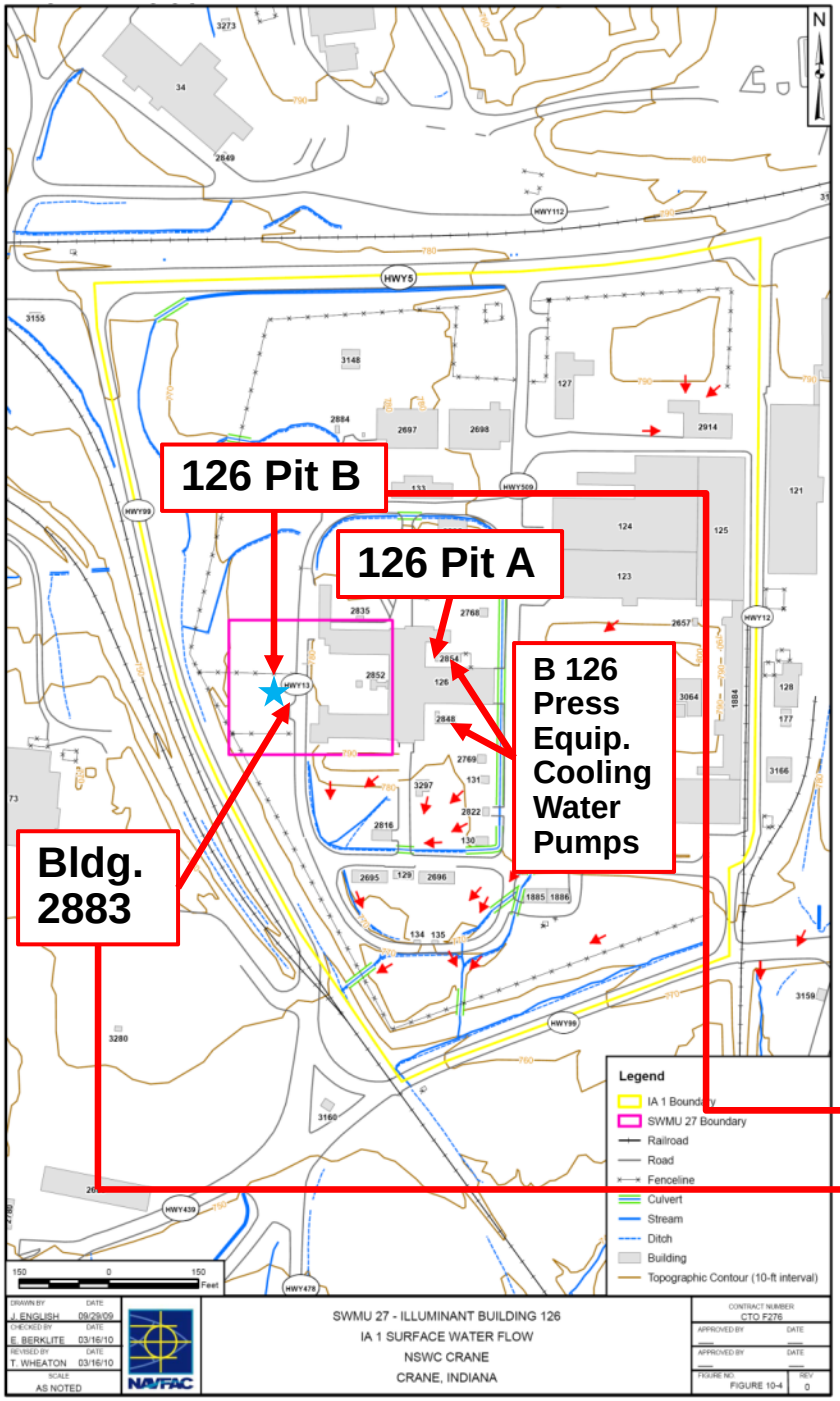
Building 126 (Illuminant Building) - Continued

- Pryotechnic illumination devices have been manufactured in Building 126 since 1943. Industrial presses are used in the manufacturing process.
- All floor drains in Building 126 discharge to a common collector which empties into a large sump outside the building. Process wastewater is also discharged to this same sump (126 Pit B) outside the building next to Building 2883. In 1972 this sump was connected to the sewage treatment plant. The pyrotechnic sludge in the sump is taken to the ordnance burning grounds for disposal.
- Building 2852 is located directly west of Building 126 and is an outdoor storage area for miscellaneous empty containers and chemicals including magnesium powder, aluminum powder, contaminated rags, and a variety of chemicals containing organic constituents.

Building 126 (Illuminant Building) - Continued

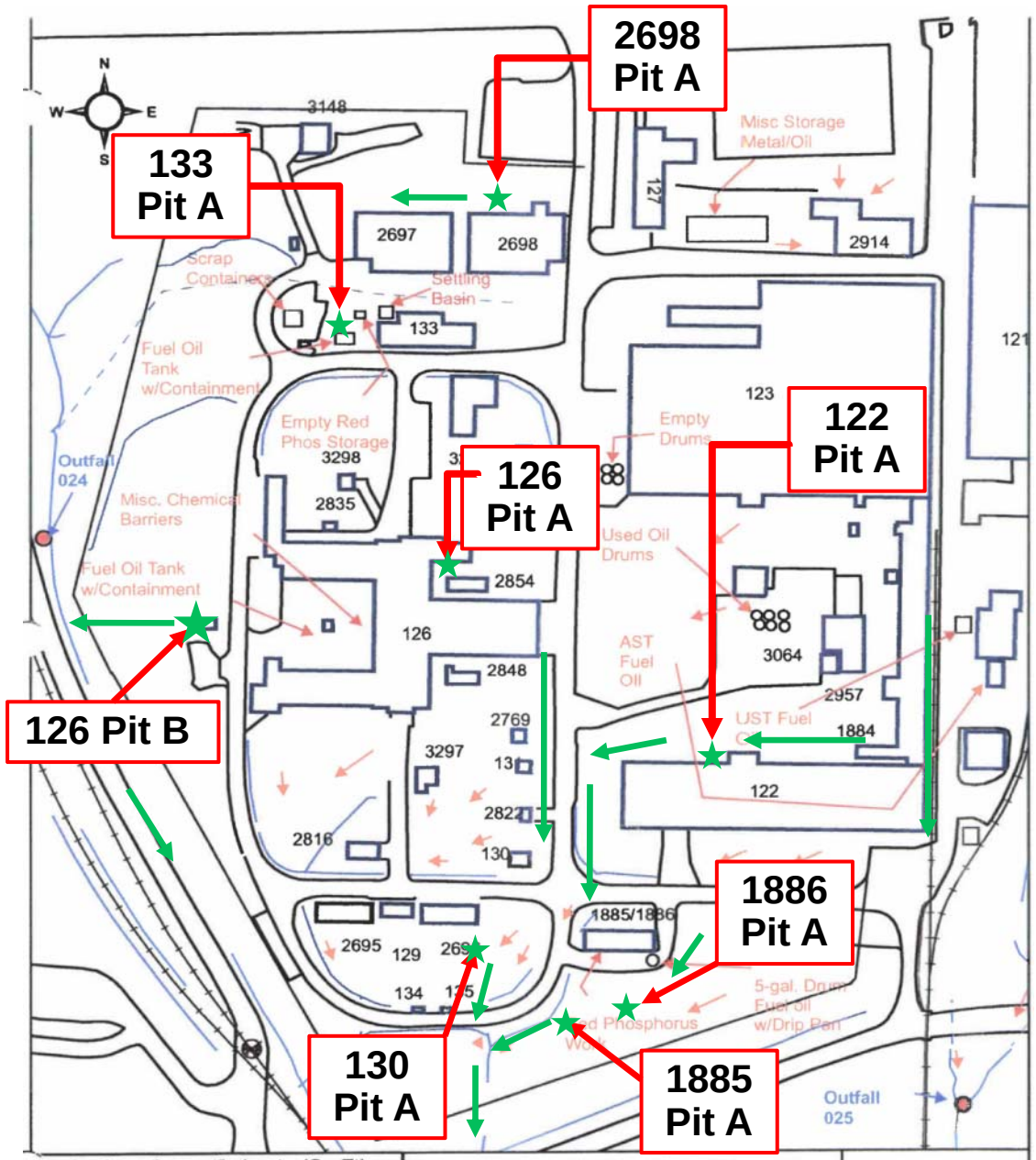
- Presses used in Building 126 require regular cooling, and pumps in Building 2848 and Building 2854 circulate non-contact cooling water through the presses. The cooling water is discharged into floor drains inside each of these buildings.
- Floor drains from the pump buildings likely discharge to 126 Pit A (a three-chambered sump) located outside Building 126 near the pump buildings. The water is transferred to the sanitary sewer and collected solids are taken to ordnance burning grounds.

Building 126 (Illuminant Building) - Continued



- All process wastewater generated is discharged to Settling Basin (126 Pit B) next to Building 2883 prior to discharge to sanitary sewer.
- Building 2883 is a covered caged pad and TYPE II Explosive hazardous waste accumulation site for scrap pyrotechnics in solvent or fuel oil. The scrap material is sent to ABG daily and a fuel oil container is adjacent to location.

Surface Water Flow, Drainages and Source Areas, SWMU 27 (IA 1)



Surface Cover Estimate (Sq Ft)

	Impervious	Pervious
024	146,362	341,512
025	148,105	148,105

Naval Support Activity
Crane, IN

PYRO AREA

0 100 200 400 Feet

FIGURE 2.2.36

- Many buildings in the PPA used sump pits (★) that were pumped out by trucks and the solid residues were taken to the burning grounds. Sump overflows drained to the south and west into the Boggs Creek watershed.
- Surface water flow and runoff direction on surfaces and drainage ditches (⟵) are shown on the adjacent map of the PPA.

Surface Water Flow, Drainages and Source Areas, SWMU 27 (IA 1)

- The sumps, sump pits, and adjacent areas that may have been impacted by sump overflows will be evaluated to determine degree of potential releases. The PPA drains to the south and west into the Boggs Creek watershed.
- There are numerous ditches and sumps in the PPA and Building 126. Examples are presented in the photographs collected from the September 2009 site walk.

Building 133 – Red Phosphorus Building

- Building 133 was used for red phosphorus candle pressing. All process wastewater is discharged to a settling basin prior to the sanitary sewer discharge. Settling basin solids are removed and sent to the ABG.
- The settling basin for Building 133 (133 Pit A) is supplied by a series of drains and covered concrete trenches which lead from Building 133 to a standard three-chamber concrete settling basin.
- The photographs were collected during the September 2009 site walk.

Building 2698 – Applied Science Department

- Building 2698 was used to support the Mk 25 and Mk 58 Marine location marker production efforts and continues to support a number of pyrotechnic production programs.
- Building 2698 was operated by the Applied Science Department and housed a foam machine and other process equipment.
- The building sump (2698 Pit A) is a single-chamber settling basin located on the north exterior wall of the building.
- Oxalic acid, various paints, solvents, VOCs, red phosphorus and the chemiluminescence compound tetrakis diaminoethylene (TMAE) were used in this building at one time.

Building 122 – Flare Assembly Building

- The building sump (122 Pit A) is a three-chamber settling basin on the north exterior wall of the building.
- Building122 supported assembly of 5”/54 caliber parachute flares. Flares were inserted into the projectiles and asbestos parachute lines were cut to length and tied to the parachute rig in Building 122.
- Building 122 was used for the recovery of magnesium and sodium nitrate from Mk 24 and Mk 45 flare candles and also included a paint booth and a polyurethane foam machine for the Mk 58 marine location marker.
- Building 122 was transferred to the Navy from the Army in the 1990s after being renovated to support the 16”/50 caliber projectile rework program. However, that program was discontinued with the Iowa Class Battleships were decommissioned.
- Methanol, methylene chloride, paints, asbestos, polyurethane foam, aluminum and magnesium powder, peroxides, nitrates, chlorates, perchlorates, sulfur, lactose, and other oxidizing agents and oxidizable metals were used in this building at one time.

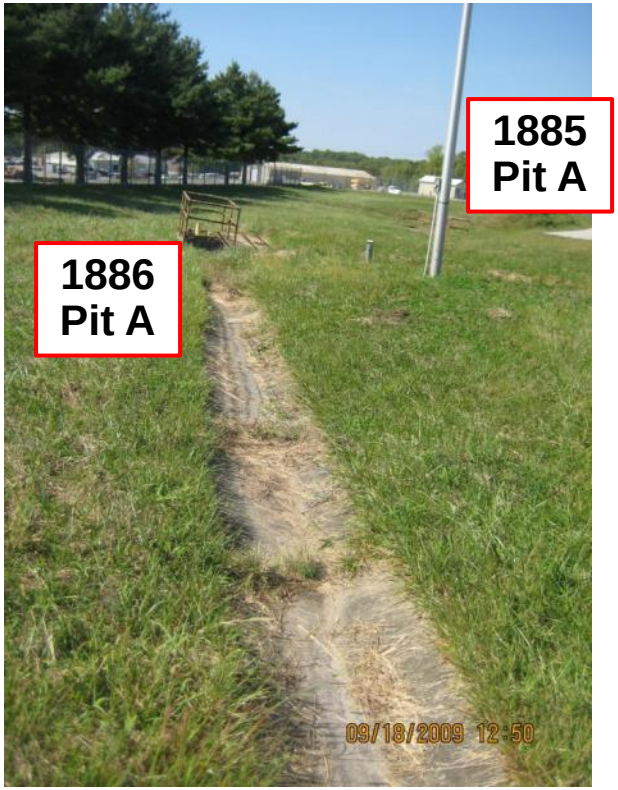
Building 130 – Flare Fuse Assembly Building

- Currently inactive, Building 130 was used for weighing out red phosphorus materials used in pyrotechnic flares and marker candles.
- Building 130 was being used for weighing out red phosphorus and a flash fire occurred when an employee attempted to empty the last remaining red phosphorus from a barrel.
- The employee was uninjured by the slow-evolving fire because he quickly jumped into the trough placed at the door for personnel fire protection purposes.

Building 1885/1886 – Phosphorus Mixing Bldg.

- Buildings 1885 and 1886 were used for mixing the red phosphorus composition used for pyrotechnic flares and marker candles.
- In October 1985 the Crane Safety Director noted that interior structure vents, ducts, door frames, wall cracks in Building 1885/1886 were heavily contaminated with powdered red phosphorus, which was a fire hazard and a violation of explosive safety standards.
- An NSA Crane Ordnance Safety Briefing dated 18 August 2008 noted that Buildings 1885 and 1886 were contaminated with red phosphorus, were no longer in good condition, and needed to be demolished.

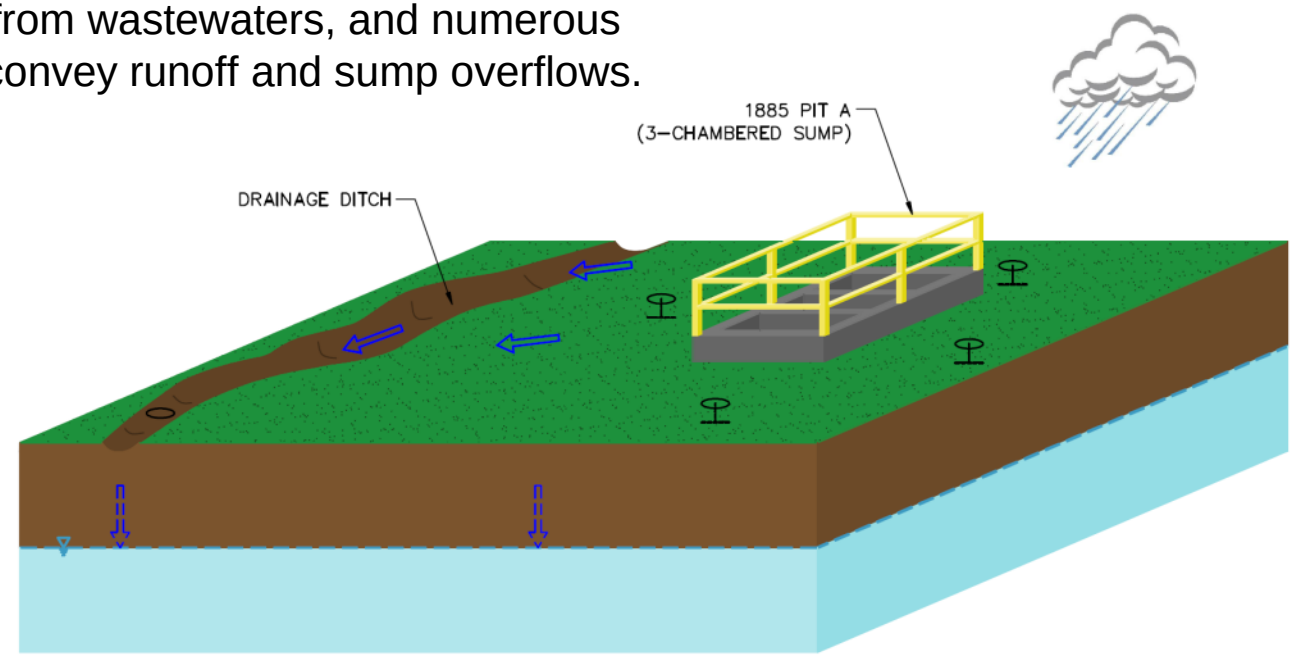
Building 1885/1886 – Phosphorus Mixing Bldg.



- Buildings 1885 and 1886 both feature three-chambered settling basins or pits where solids were separated from wastewaters.
- If these buildings are removed from service and demolished, then the corresponding settling basins (1885 Pit A and 1886 Pit A) will likely require removal or repairs as needed to address surface runoff and local drainages.

PPA Sumps and Surface Drainages

- The PPA features several three-chambered settling basins and pits where solids were separated from wastewaters, and numerous ditches to convey runoff and sump overflows.



LEGEND

- WATER TABLE ELEVATION (ESTIMATED)
- SURFACE RUNOFF FLOW DIRECTION
- INFILTRATION
- 0–2 FT BGS

DRAWN BY CK DATE 4/07/10	NAVFAC	CONCEPTUAL SITE MODEL CATCH BASINS, SETTLING BASINS, AND DRAINAGWAYS NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY DATE			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10–8 REV. 0

Summary of Approach for CTO F276 – RFI for SWMU 27 (PPA) – Metal Plating Rinsewaters

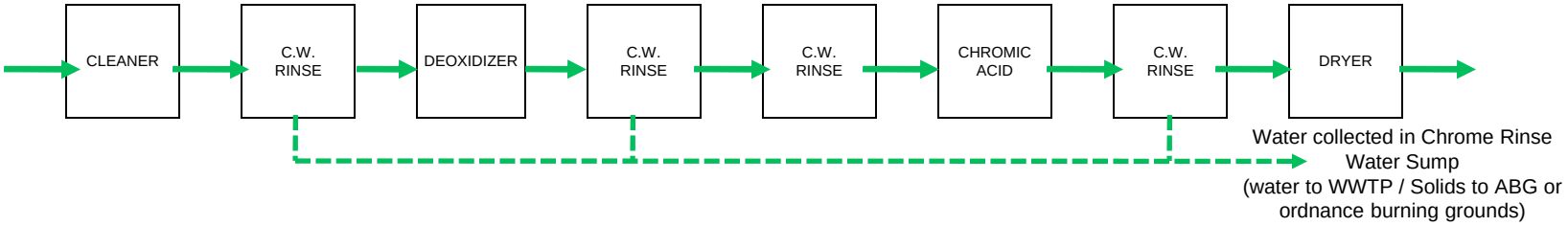
- A metal-plating shop utilizing metals, caustics, acids, and cyanides has been operating on the eastern edge of the PPA (Building 1884).
- Process wastewaters and rinsates were at one time discharged to open ditches and later to building sumps that may have overflowed.
- The ditches, sump areas, and other adjacent areas possibly impacted by discharges or sump overflows will be evaluated to determine degree of potential releases. The PPA drains to the south and west into the Boggs Creek watershed.
- Significant heavy metal contamination (zinc and cadmium) has been experienced from wastewater being discharged into open ditches.
- Treated wastewater is discharged to the sanitary sewer system under an NPDES permit.

Summary of Approach for CTO F276 – RFI for SWMU 27 (PPA) – Metal Plating Rinsewaters

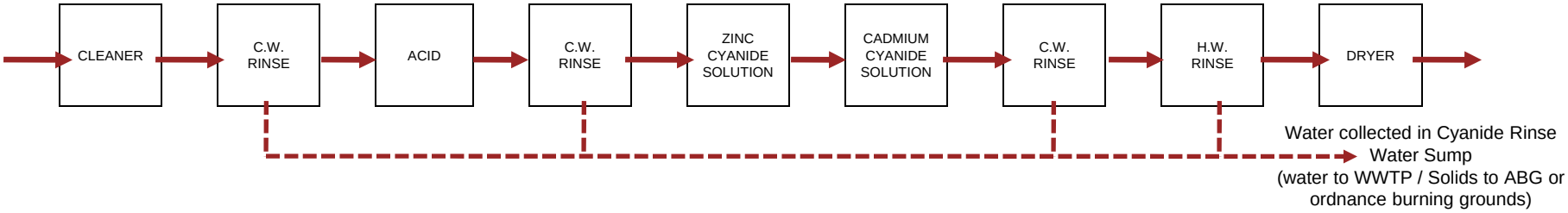
- Few trenches or sumps were identified in the vicinity of the metal plating facility (Building 1884). There was a long concrete paved ditch that drained away from Building 1884, but appeared to be a conveyance for roof drains.
- There sumps and drains near Building 1884 that may have been impacted by overflows of process wastewaters and rinsates discharged to open ditches and later to building sumps.

Summary of Metal Plating Processes at Building 1884
RFI for SWMU 27 (PPA)

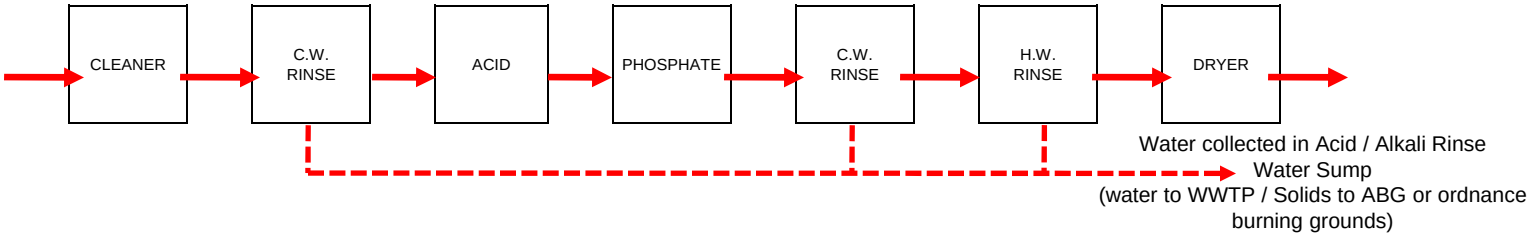
CHROME CONVERSION COATING



CADMIUM AND ZINC CYANIDE PLATING



PHOSPHATE COATING



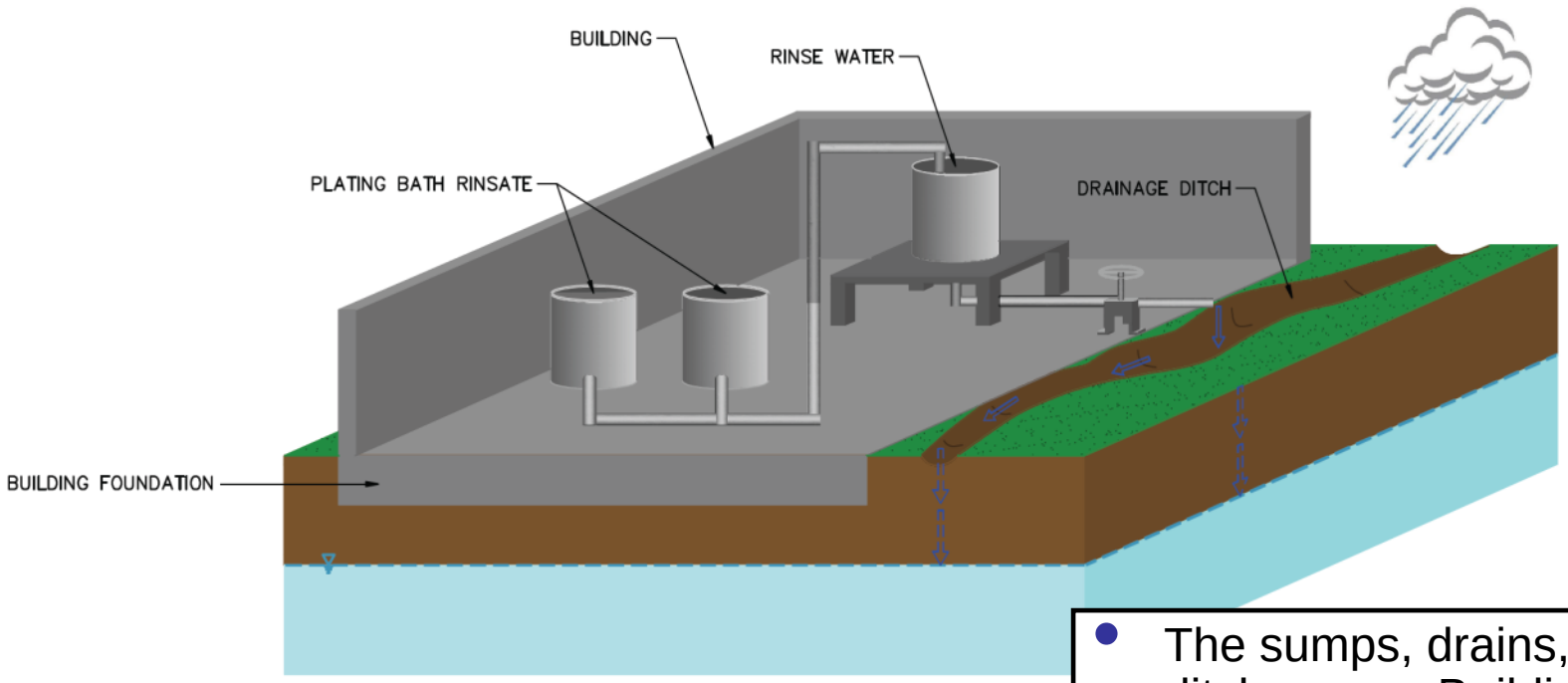
PROCESS FLOW

WASTEWATER FLOW

C.W. - COLD WATER
H.W. - HOT WATER
WWTP - Wastewater Treatment Plant (Building 3064)

Source: Figure E-1 US Army Water Quality Engineering Study No. 32-24-0575-85

Summary of Approach for CTO F276 – RFI for Building 1884 Metal Plating Rinsewaters



LEGEND

SURFACE RUNOFF FLOW DIRECTION

INFILTRATION

WATER TABLE ELEVATION (ESTIMATED)

0–2 FT BGS

DRAWN BY	DATE
CK	4/07/10
CHECKED BY	DATE
REVISED BY	DATE
SCALE NOT TO SCALE	

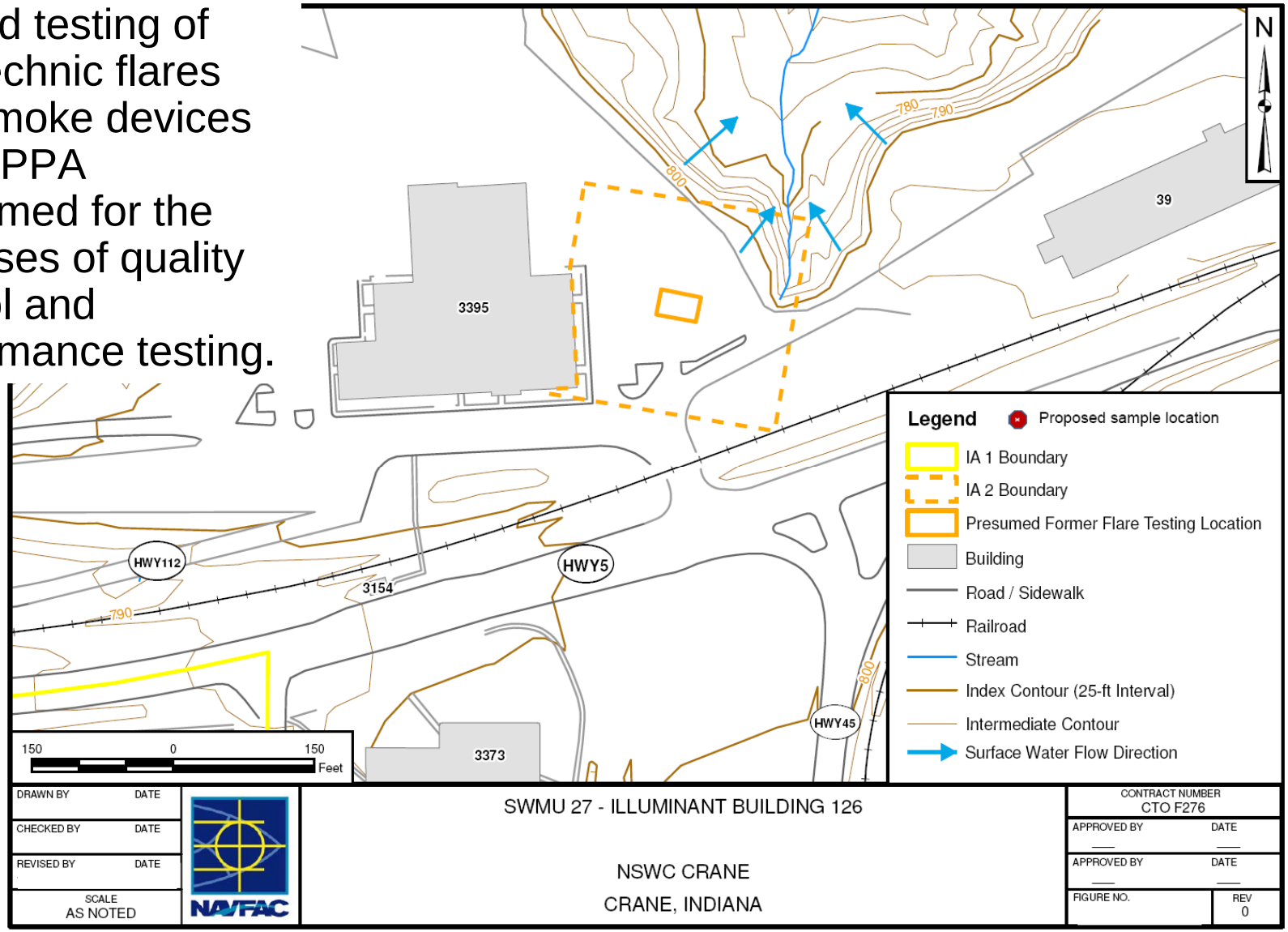
- The sumps, drains, and ditches near Building 1884 may have been impacted by overflows of process wastewaters and rinsates discharged to open ditches and later to building sumps.

Summary of Approach for CTO F276 – RFI for SWMU 27 (PPA) – Flare Testing/Burn Areas

- There was limited testing of pyrotechnic flares and smoke devices in the PPA performed for the purposes of quality control and performance testing.
- The pyrotechnics included red phosphorus, chlorates, dyes, and oxidizers (such as perchlorate).
- There were two identified flare test/burn areas in the PPA, one behind Building 126 (west near Pit 126B) and another location across Highway 5.
- Tests/burns at the sites ceased in 1971.
- Limited sampling will also be performed for discrete PPA areas with known operational fires or explosions.

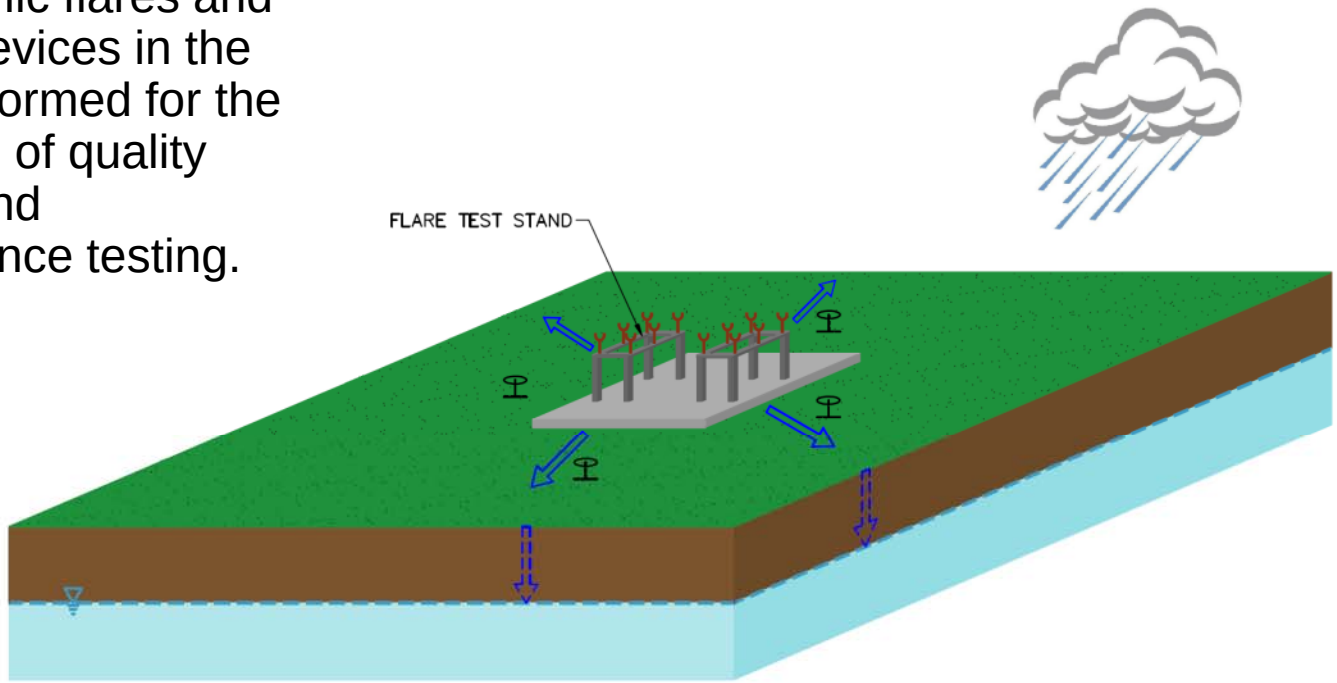
Summary of Approach for CTO F276 – RFI for SWMU 27 (PPA) – Flare Testing/Burn Areas

- Limited testing of pyrotechnic flares and smoke devices in the PPA performed for the purposes of quality control and performance testing.



Summary of Approach for CTO F276 – RFI for SWMU 27 (PPA) – Flare Testing/Burn Areas

- Limited testing of pyrotechnic flares and smoke devices in the PPA performed for the purposes of quality control and performance testing.



LEGEND

SURFACE RUNOFF FLOW DIRECTION

INFILTRATION

WATER TABLE ELEVATION (ESTIMATED)

0–2 FT BGS

DRAWN BY CK DATE 4/07/10	NAVFAC	CONCEPTUAL SITE MODEL FLARE TESTING ACTIVITIES NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY DATE			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10–9 REV. 0

Past Releases/Incidents Recorded in the PPA

- **Building 127** was initially used a supporting laboratory for the work in Building 126. The former north end of Building 127 once housed a burn chamber where experiments were conducted in the 1960's to evaluate the relationship of candle density to illuminating round performance. An explosion occurred when a burn was being conducted with a very low density press loaded candle and the explosion destroyed the burn chamber.
- A **Former Curing Building** located south of Building 126 was destroyed by fire in the late 1980's, and the fire was traced to a malfunctioning heating unit that overheated and cooked off the candles being cured. Building 3297 has been constructed in the same general location as the destroyed curing building.

Past Releases/Incidents Recorded in the PPA

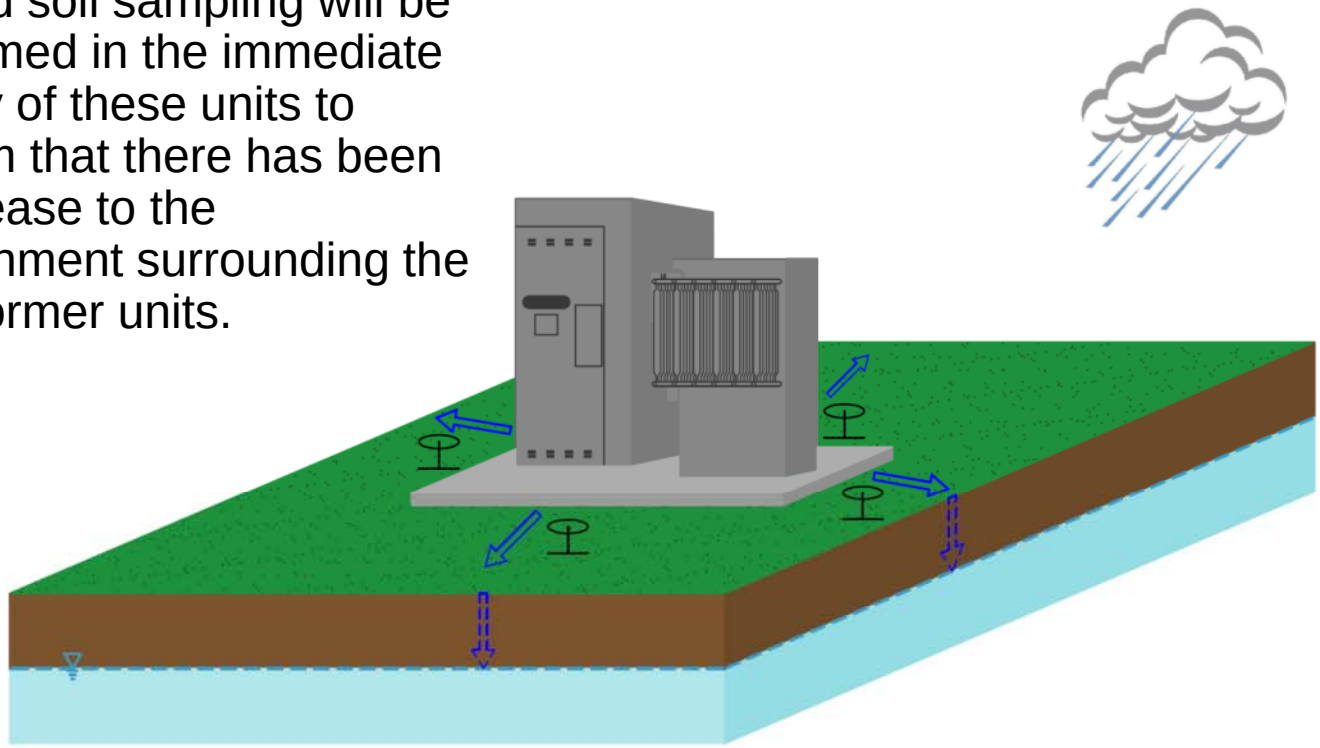
- **Building 130** was being used for weighing out red phosphorus and a flash fire occurred when an employee attempted to empty the last remaining red phosphorus from a barrel. The post-incident investigation revealed the plastic bag lining the barrel was not conductive, but as red phosphorus fines were falling through space inside the bag, a spark ignited the red phosphorus. The employee was uninjured by the slow-evolving fire because he quickly jumped into the trough placed at the door for personnel fire protection purposes.
- **Building 1886** was being used for experiments involved red phosphorus, boron, lead dioxide and a binder material when a flash fire occurred. The ingredients were added into the mixer bowl in a different manner than called for by the test procedure to improve the mixing effort. The lesson learned was that knowing the sensitivity of individual ingredients and the final product may not sufficient to outline the true hazards of the mixing process.

Summary of Approach for CTO F276 – RFI for SWMU 27 (PPA) – PCB Transformers (IA 1)

- There were multiple transformers and former transformer locations noted during the September 2009 site walk. Several units were marked as PCB transformers.
- Limited soil sampling will be performed in the immediate vicinity of these units to confirm that there has been no release to the environment surrounding the transformer units.

Summary of Approach for CTO F276 – RFI for SWMU 27 (PPA) – PCB Transformers (IA 1)

- Limited soil sampling will be performed in the immediate vicinity of these units to confirm that there has been no release to the environment surrounding the transformer units.



LEGEND

SURFACE RUNOFF FLOW DIRECTION

INFILTRATION

WATER TABLE ELEVATION (ESTIMATED)

0–2 FT BGS

DRAWN BY CK DATE 4/07/10	NAVFAC	CONCEPTUAL SITE MODEL POLYCHLORINATED BIPHENYLS (PCBs) TRANSFORMERS NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY DATE			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10–10 REV. 0

Summary of Approach for CTO F276 – RFI for SWMU 27 – A Phased Sampling Approach

- Phase I sampling within the PPA will assess the degree of the potential environmental contamination within SWMU 27:
  - Soil sampling around pyrotechnic process sumps, building drains, and related ditches and trenches that convey water from chemical processing/mixing areas (pyrotechnic compounds like red phosphorus, chlorates, oxidizers, and fuels for flares and smoke munitions). Process wastewaters and rinsates once discharged to open ditches and were later directed to sumps that may have overflowed.
  - Soil sampling within ditches and trenches near the cutting, welding, heat treating, metal-finishing operations in Buildings 123, 124, and 125 (petroleum-based or chlorinated metal-cleaning solvents, metal cutting oils, machine shop device cooling water discharges, etc.)
  - Soil sampling within ditches and trenches near the metal plating operations in Building 1884 (metal-cleaning solvents, heavy metals such as chromium, cadmium, zinc, and also cyanide),
  - Soil sampling around known/suspected PCB transformers (PCB aroclors)
  - Soil sampling around two suspected former test/burn sites (residual metal fuels such as magnesium, manganese, zinc, and also perchlorate)
  - Soil sampling in the general vicinity location of reported process area fires and explosions (residual metal fuels such as magnesium, manganese, zinc, and also perchlorate)

Summary of Approach for CTO F276 – RFI for SWMU 27 – A Phased Sampling Approach

- Phase II sampling will include further sampling of soils, as necessary, to supplement Phase I data to better delineate the lateral and vertical extent of any suspected release areas.

Summary of Approach for CTO F276 – RFI for SWMU 27 – A Phased Sampling Approach

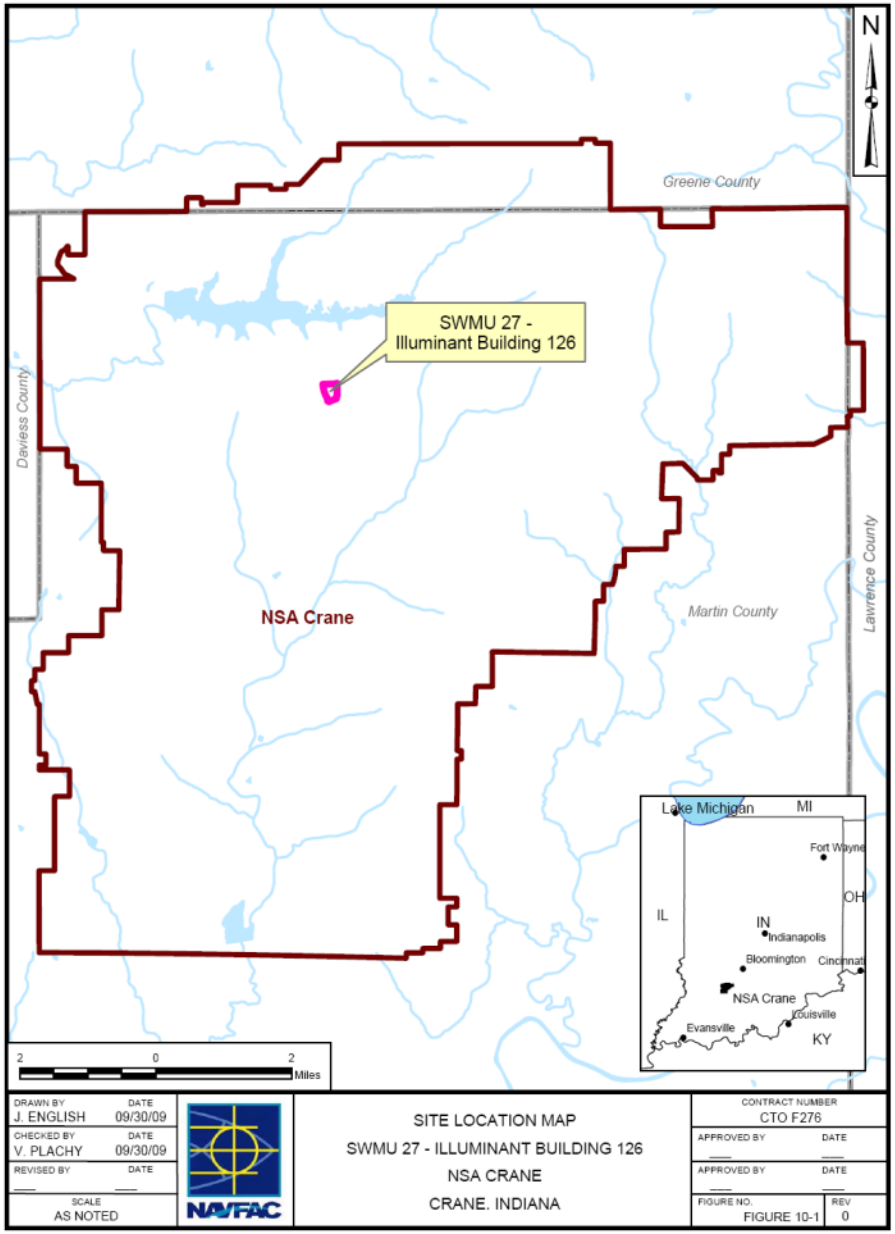
- Phase III sampling will be performed, as necessary, to complete delineation of suspected release areas that were not completed during Phase II.

Solid Waste Management Unit 27 Pyrotechnics Production Area (PPA) RCRA Facility Investigation Project Data Quality Objectives Planning

Naval Support Activity (NSA) Crane, Crane, Indiana

DQO Planning Session
Part 3
June 2010

Naval Support Activity Crane Installation Map



SWMU 27 – Pyrotechnic Production Area (PPA) Building 126 – Illuminant Building

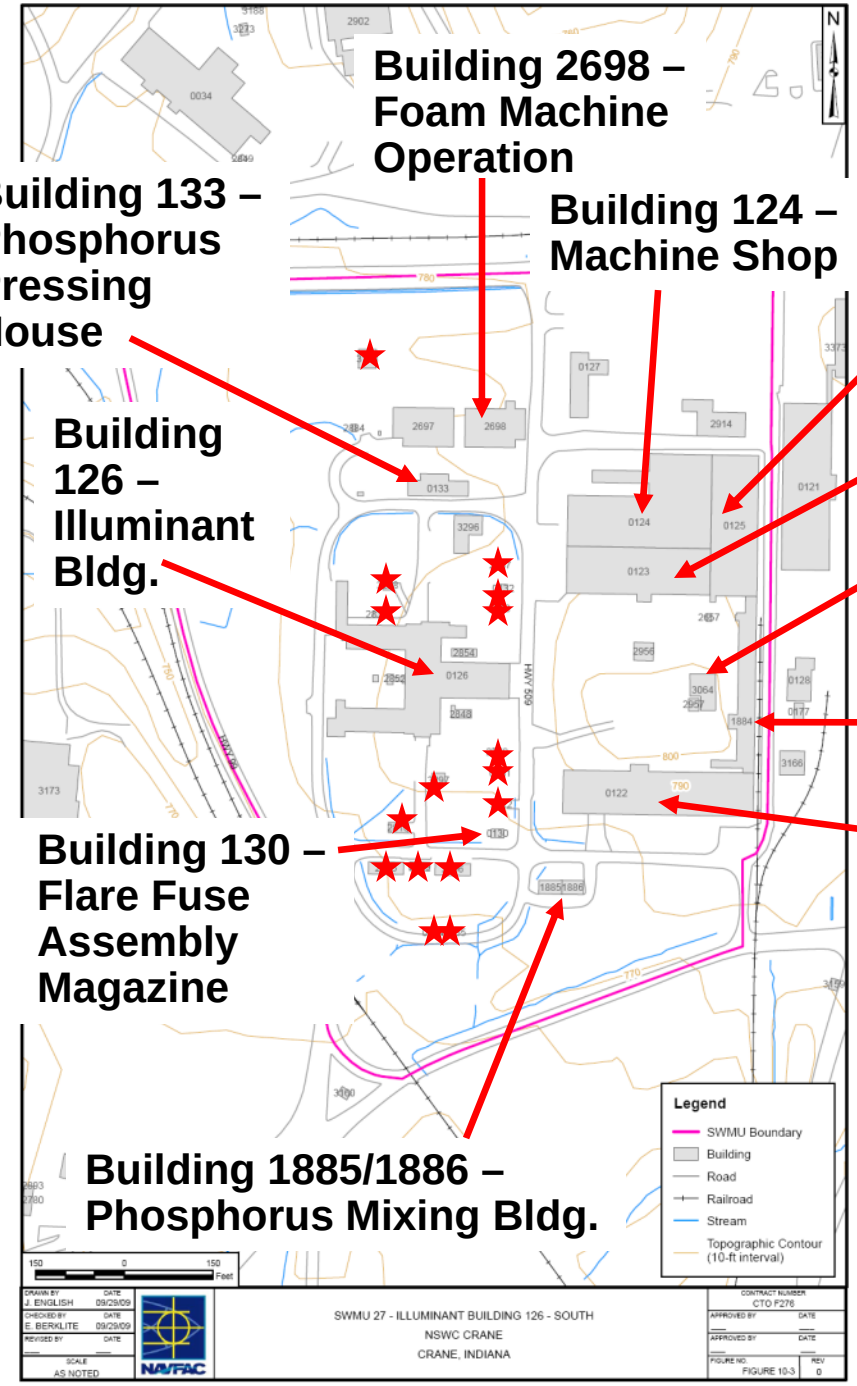
- The Illuminant Building 126 is classified as an IR site by the Navy.
- According to descriptions of this IR site, red phosphorus was discharged to sump pits that overflowed to tributaries of Boggs Creek.
- There has also been heavy metal contamination from zinc and cadmium identified in open ditches that eventually drain into Boggs Creek.
- No remedial investigation studies have been performed for this IR site; therefore, it is difficult to know the extent and location of potential contamination.
- An outdoor burning area was located near Building 126 and another area north of Highway 5. Open burning operations ceased in 1971.

Project Background for CTO F276 – RFI for SWMU 27

Site History from NAVFAC:

- Red Phosphorous is the most significant contaminant with lesser chlorates, dyes, oxidizers and fuels for flares and smoke munitions.
- The building (Building 126) used sump pits that were vacuum pumped out by trucks and taken to the burning grounds.
- All sump overflows drained into the Boggs Creek watershed.
- A metal plating shop that has utilized metals, caustics, acids, and cyanides is also present and significant heavy metal contamination (zinc and cadmium) has been experienced from wastewater being discharged into open ditches.
- In the late 1970's, a pre-treatment facility was constructed for the aforementioned plating shop. It is currently in use and treated wastewater is discharged to the sanitary sewer system under an NPDES permit.
- There are also historic burn areas; one behind Building 126 and one across Highway 5. Burning at those locations ceased in 1971.

SWMU 27 – Pyrotechnic Production Area (PPA) Buildings



Building 125 – Machine Shop

Building 123 – Former Projectile Assembly

Building 3064/2957 – Wastewater Treatment Plant (includes Metal Plating Facility rinse waters).

Building 1884 – Metal Plating Operations

Building 122 – Former Flare Assembly Bldg. and Paint Booth / Urethane Foam Operation

Many smaller buildings in the PPA are locked storage warehouses and bunkers where raw materials used in pyrotechnic production were stored, mixed, or where pyrotechnic items were permitted to cure. These locations are noted on this map with a★.

Problem Definition

- The PPA is currently active and contains multiple warehouses, storage bunkers, manufacturing facilities, and curing buildings. However, certain pyrotechnic process buildings in the PPA are not currently used.
- It is likely that past PPA operations may have resulted in chemical discharges or releases (discharge areas, sump overflows, release areas, flare test areas, transformer equipment areas, pyrotechnic processing and operations areas, burning areas, etc.) to local environmental media (surface soil, sediment, surface water run-off).
- Discharges/releases of pyrotechnic compounds and other contaminants in the PPA may have produced areas of chemical contamination in the local environment above project action levels (PALs) based on Indiana Department of Environmental Management (IDEM) Risk Integrated System of Closure (RISC) Default Closure Levels (DCLs) for residential and industrial receptors (IDEM RISC R-DCLs and IDEM RISC I-DCLs, respectively) and/or EPA Regions 3, 6, and 9 Residential Regional Screening Levels (R-RSLs) (e.g., the human health screening levels (HHRSLs)).

Problem Definition

- The Project Team must determine whether site-related contaminant releases are present in local environmental media and evaluate if those concentrations that are greater than HHRSLs are indicative of potential risk to human health for current and future workers in the PPA. The Project Team will make recommendations for further appropriate action, as necessary, based on the HHRSLs.
- If target analytes are identified in groundwater in excess of human health risk screening, then the Project Team will evaluate the need for permanent monitoring wells.

Information Inputs

- Aerial photos and historic drawings showing locations of former buildings and site features will be used to determine sample locations and to support the CSM.
- Chemical Data:
  - Soil: Select metals, cyanide, perchlorate, VOCs
  - Residual material and surface water in sumps: metals, cyanide, perchlorate, VOCs
  - Groundwater: will depend on detects of analytes in soil during Phase I and II.
- Groundwater Field Parameters: pH, specific conductance, oxidation-reduction potential (ORP), temperature, dissolved oxygen (DO), and turbidity.
- PALs and Background:
  - Background data for the various soil types identified at NSA Crane are described in the Final Base-Wide Background Soil Investigation Report for NSWC Crane (Tetra Tech, 2001).
  - IDEM RISC R-DCLs and RISC Industrial Default Closure Levels (I-DCLs); EPA Regions 3, 6, and 9 Residential Regional Screening Levels (R-RSLs)

Study Area Boundaries

- Horizontal boundaries are defined by the extent of contamination in surface soil (0-2 ft) identified in location of former process area sumps and potential releases including discharges and overflows from process building sumps into adjacent soil and soil in drainages and ditches (0-1 ft), chemical releases from material management areas, releases from material processing areas or material storage areas, release areas, leaks from transformers in operational areas, or releases from pyrotechnic testing/burning areas, etc.).
- Phase I vertical boundaries for sumps is the depth of the sump.
- Phases II and III vertical boundaries are defined by the extent of vertical contamination found in subsurface soil.

Study Area Boundaries

- The vertical boundary for the potential groundwater investigation is defined by the water table.
- Populations of interest include potentially contaminated soil and groundwater in the areas described above.
- Populations of interest include potentially uncontaminated soil and groundwater in the areas described above.

Decision Rules

- If there is no contamination above HHRSLs, then no additional action is required for this site.

- If surface soil contamination is present, then proceed to Phase II of the investigation to determine the extent of surface soil contamination and to determine if contamination is present in subsurface soil.

- If contamination is identified in subsurface soil during Phase II of the investigation, then additional data must be gathered in Phase III of the investigation to delineate the extent of soil contamination and to determine if contamination has migrated to groundwater.

- If no contamination has been identified in groundwater during Phase III, then complete the HHRA with the Phase I and II soil data. If unacceptable risk are present, then proceed to a CMS to evaluate response actions. If no unacceptable risk is present, then no further action is required.

- If contamination is present in groundwater, then complete the HHRA with the Phase I and II soil and groundwater data. If unacceptable risk are present, then proceed to a CMS to evaluate response actions.

Develop the Plan (Sample Design)

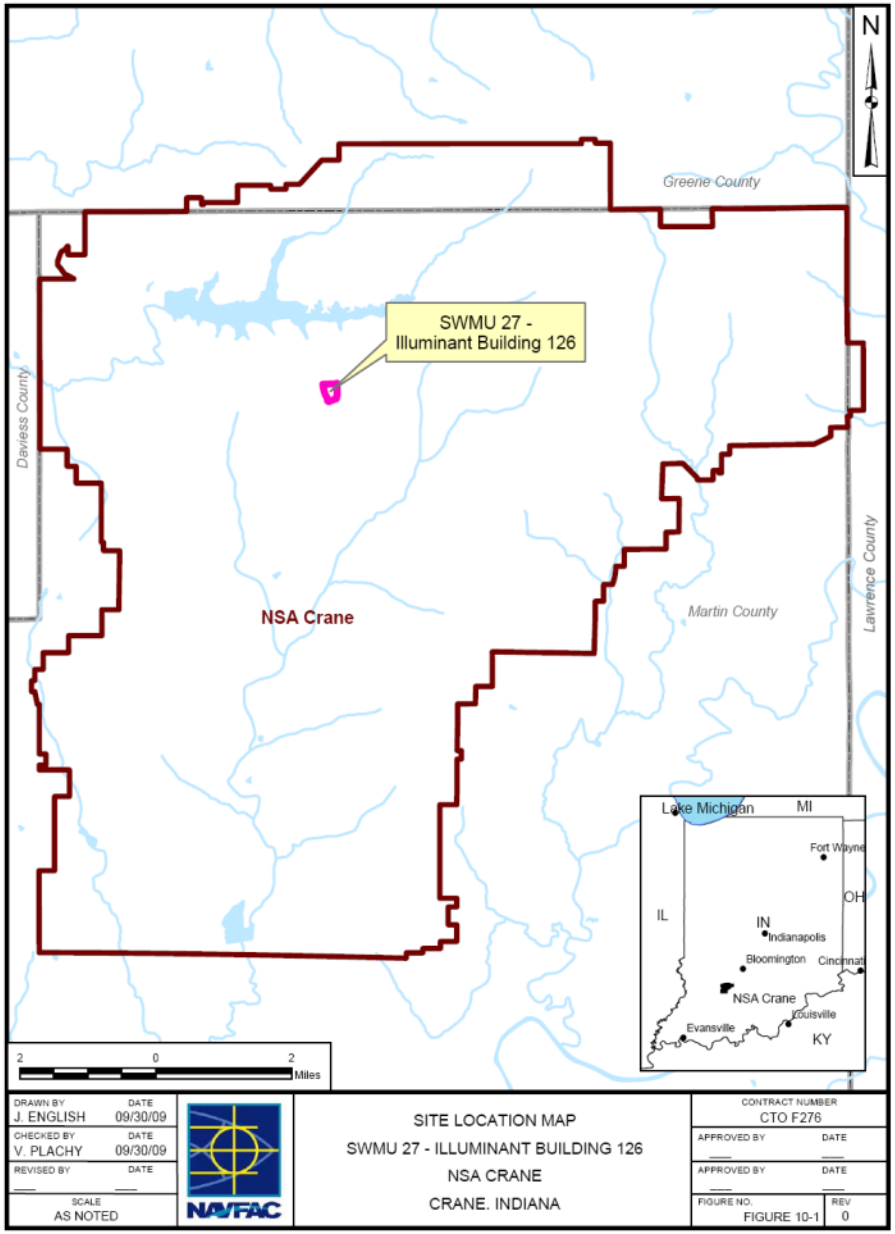
- RFI sampling will be performed to determine local impacts of known or suspected releases or discharge locations such as sump overflow areas, flare burn areas, transformer areas, etc.
- RFI program's focused soil / sediment sampling will improve the characterization of local environmental conditions in PPA areas.
- Initial RFI phase (Phase I) will seek to identify / establish lateral (horizontal) boundaries (to the extent possible) for areas of surface soil (0 to 2 feet deep) and sediment contamination (0 to 6 inches) near known and suspected release sites within SWMU 27.
- Subsequent phases (RFI Phase II and Phase III) will be performed (as necessary) to confirm lateral boundaries and establish vertical components of contamination in subsurface soil (greater than two feet deep) and also to evaluate potential impacts of soil contamination releases to local groundwater at SWMU 27.
- Use newly obtained RFI environmental data to expand/update the CSM using field reconnaissance/survey data
- Evaluate initial <u>human health exposure hazards</u>, summarize risk screening results, and recommend future site actions.

Solid Waste Management Unit 27 Pyrotechnics Production Area (PPA) RCRA Facility Investigation Project Data Quality Objectives Planning

Naval Support Activity (NSA) Crane, Crane, Indiana

RFI Planning Session
Part 4
June 2010

Naval Support Activity Crane Installation Map



SWMU 27 – Pyrotechnic Production Area (PPA) Building 126 – Illuminant Building

- The Illuminant Building 126 is classified as an IR site by the Navy.
- According to descriptions of this IR site, red phosphorus was discharged to sump pits that overflowed to tributaries of Boggs Creek.
- There has also been heavy metal contamination from zinc and cadmium identified in open ditches that eventually drain into Boggs Creek.
- No remedial investigation studies have been performed for this IR site; therefore, it is difficult to know the extent and location of potential contamination.
- An outdoor burning area was located near Building 126 and another area north of Highway 5. Open burning operations ceased in 1971.

Project Background for CTO F276 – RFI for SWMU 27

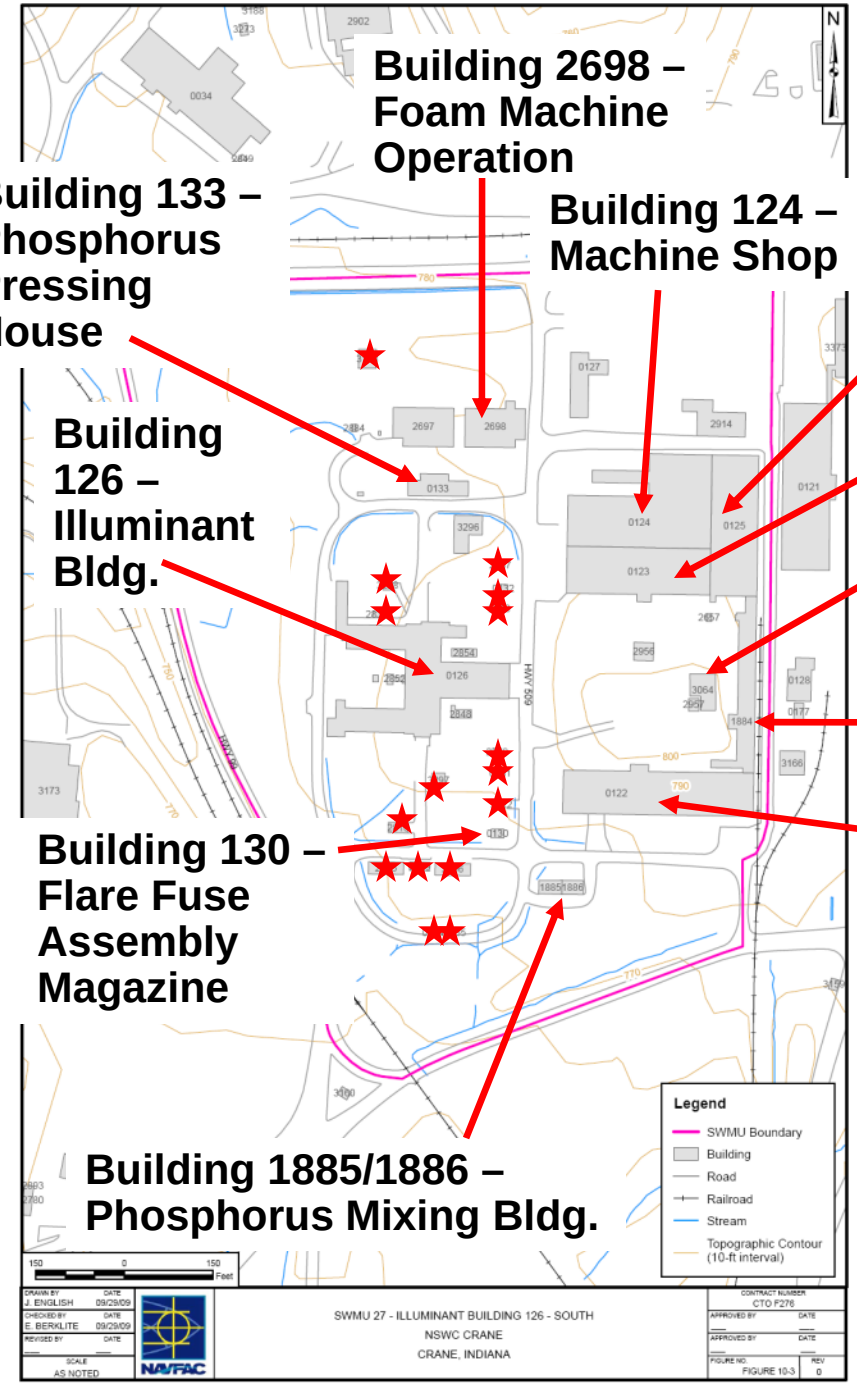
Site History from NAVFAC:

- Red Phosphorous is the most significant contaminant with lesser chlorates, dyes, oxidizers and fuels for flares, and smoke munitions.
- The building (Building 126) used sump pits that were pumped out by vacuum trucks and taken to the burning grounds.
- All sump overflows drained into the Boggs Creek watershed.
- A metal plating shop that has utilized metals, caustics, acids, and cyanides is also present and significant heavy metal contamination (zinc and cadmium) has been experienced from wastewater being discharged into open ditches.
- In the late 1970's, a pre-treatment facility was constructed for the aforementioned plating shop. It is currently in use and the treated wastewater is discharged to the sanitary sewer system under an NPDES permit.
- There are historic burn areas; one behind Building 126 and one across Highway 5. Burning at those locations ceased in 1971.

Data Quality Objectives (DQOs) for RFI at PPA

- **Develop the Plan (Sample Design)**
  - RFI sampling will be performed to determine local impacts of known or suspected releases or discharge locations such as sump overflow areas, flare burn areas, transformer areas, etc.
  - RFI program's focused soil / sediment sampling will improve the characterization of local environmental conditions in PPA areas.
  - Initial RFI phase (Phase I) will seek to identify / establish lateral (horizontal) boundaries (to the extent possible) for areas of surface soil (surface to two feet deep) and sediment contamination near known and suspected release sites within SWMU 27.
  - Subsequent phases (RFI Phase II and Phase III) will be performed (as necessary) to confirm lateral boundaries and establish vertical components of contamination in subsurface soil (greater than two feet deep) and also to evaluate potential impacts of soil contamination releases to local groundwater at SWMU 27.
  - Use newly obtained RFI environmental data to expand / update the Conceptual Site Model (CSM) using field reconnaissance/survey data
  - Evaluate initial <u>human health exposure hazards</u>, summarize risk screening results, and recommend future site actions.

SWMU 27 – Pyrotechnic Production Area (PPA) Buildings



Building 125 – Machine Shop

Building 123 – Former Projectile Assembly

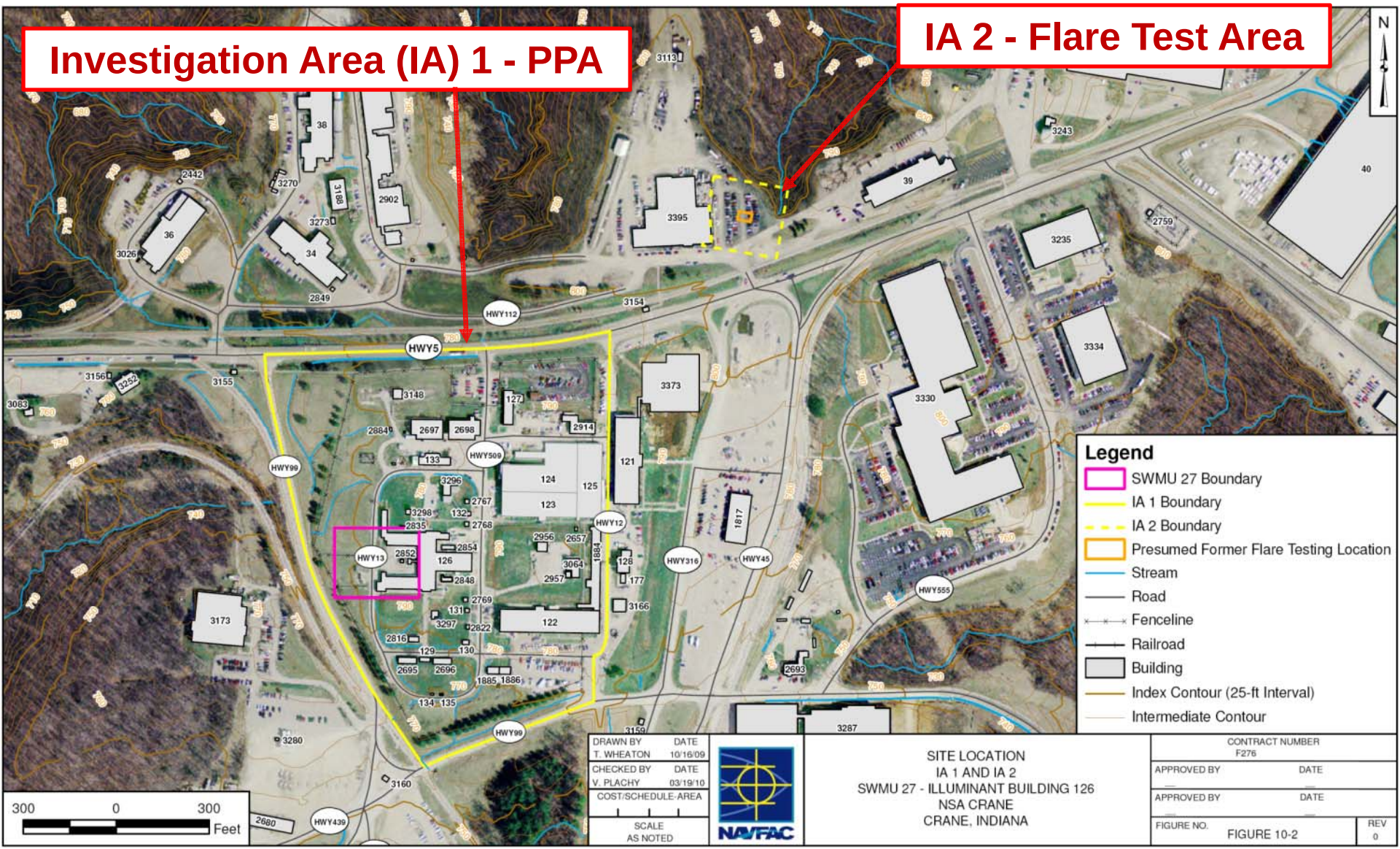
Building 3064/2957 – Wastewater Treatment Plant (includes Metal Plating Facility rinse waters).

Building 1884 – Metal Plating Operations

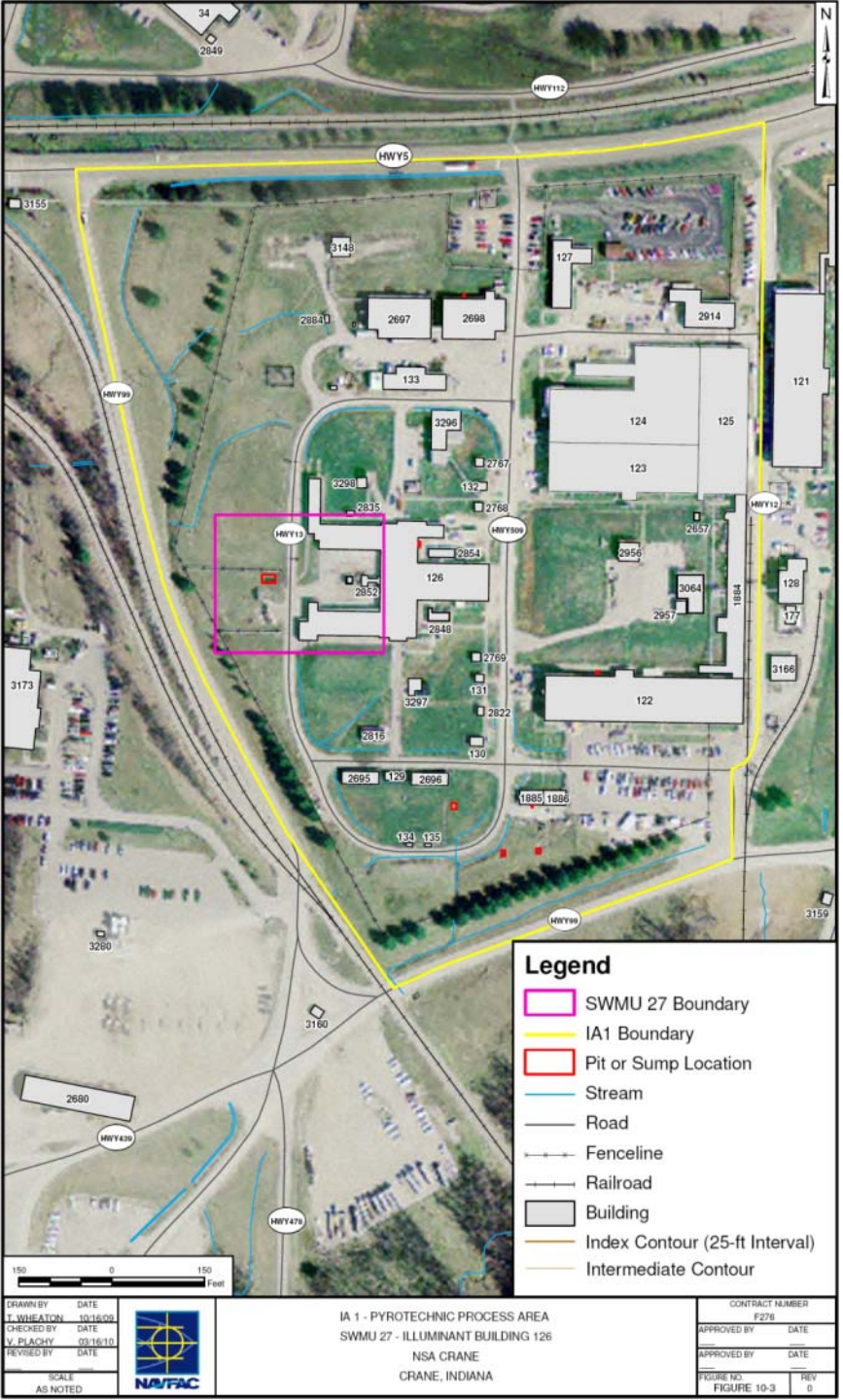
Building 122 – Former Flare Assembly Bldg. and Paint Booth / Urethane Foam Operation

Many smaller buildings in the PPA are locked storage warehouses and bunkers where raw materials used in pyrotechnic production were stored, mixed, or where pyrotechnic items were permitted to cure. These locations are noted on this map with a ★.

PPA Figure for SWMU 27 RFI

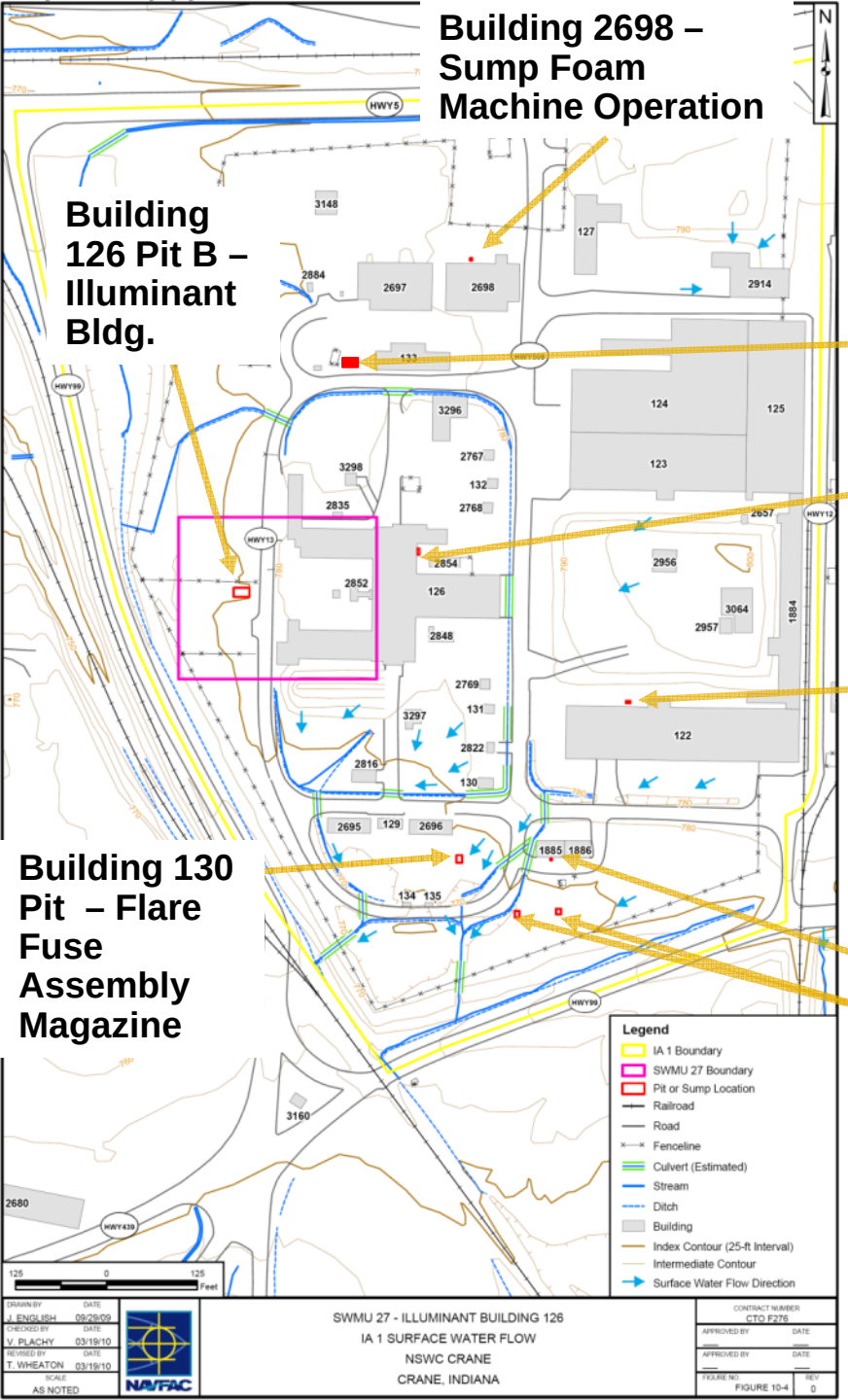


SWMU 27 Investigation Area #1 – Sumps/Pits



- According to descriptions of this IR site, red phosphorus was discharged to sumps and pits overflowed to tributaries of Boggs Creek.
- No remedial investigation studies have been performed for this IR site; therefore, it is difficult to know the extent and location of potential contamination.
- RFI sampling will be performed to determine local impacts of known or suspected releases or discharge locations such as sump overflow areas and drainage ditches
- RFI program's focused soil / sediment sampling will improve the characterization of local environmental conditions in PPA areas.

SWMU 27 Investigation Area #1 – Sumps and Pits



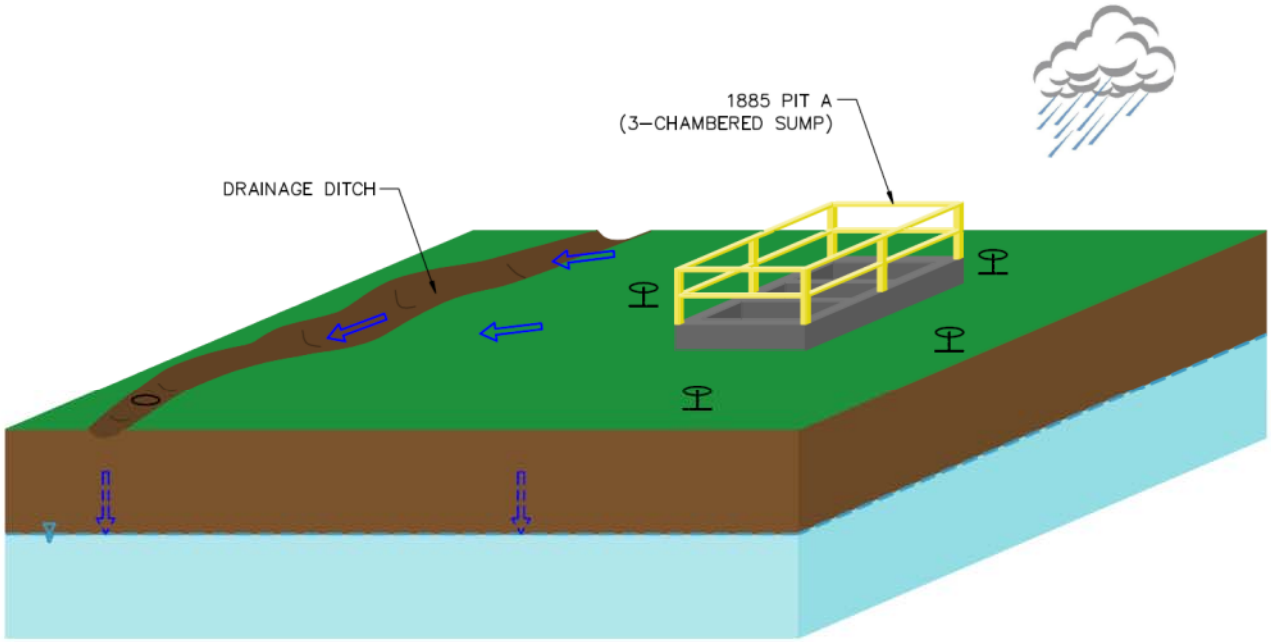
Building 133 Pit A – Phosphorus Pressing House

Building 126 Pit A – Illuminant Bldg.

Building 122 Sump – Former Flare Assembly Bldg. and Paint Booth / Urethane Foam Operation

Building 1885/1886 – Pits and Sumps from Phosphorus Mixing Bldg.

CSM and Sampling Approach
SWMU 27 Sumps



LEGEND

WATER TABLE ELEVATION (ESTIMATED)

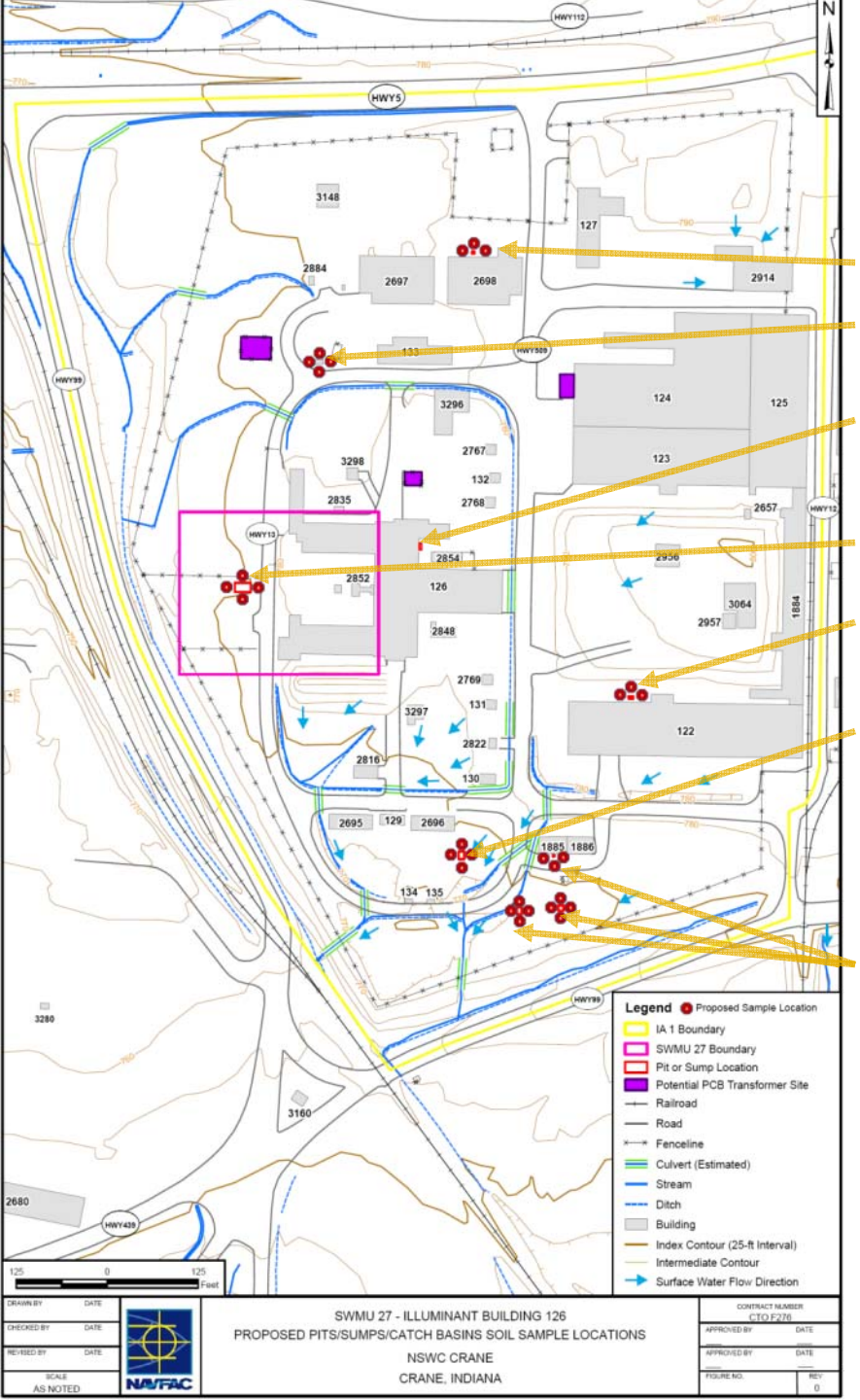
SURFACE RUNOFF FLOW DIRECTION

INFILTRATION

0–2 FT BGS

DRAWN BY CK DATE 4/07/10	NAVFAC	CONCEPTUAL SITE MODEL CATCH BASINS, SETTLING BASINS, AND DRAINAGWAYS NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY DATE			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10–8 / REV. 0

Sampling Approach for SWMU 27 – Sumps/Pits



Building 2698 –Sump Foam Machine Operation

Building 133 Pit A – Phosphorus Pressing House

Building 126 Pit A – Illuminant Bldg. (no samples are proposed for this unit because concrete surrounds the sump.)

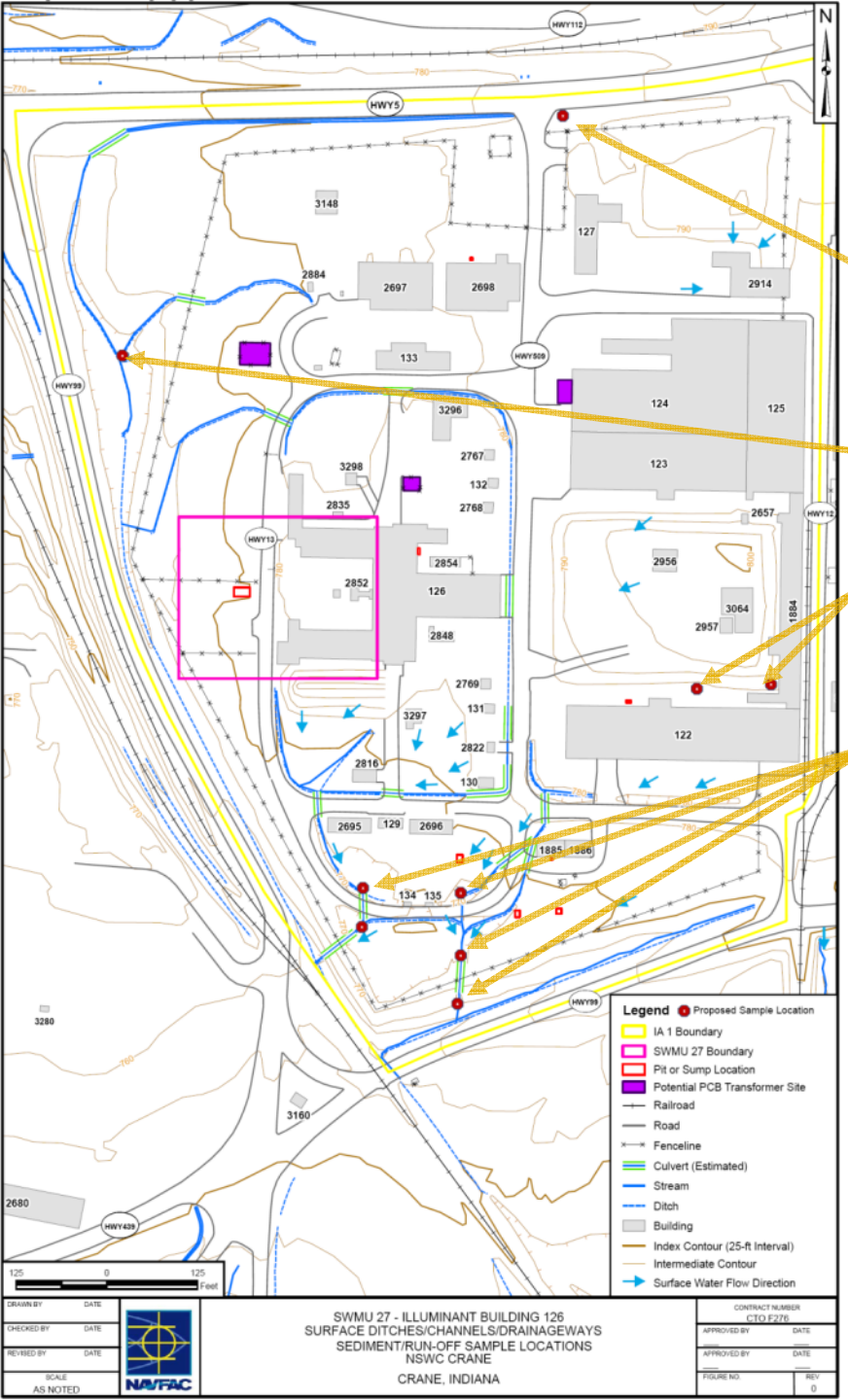
Building 126 Pit B – Illuminant Bldg.

Building 122 Sump – Former Flare Assembly Bldg. and Paint Booth / Urethane Foam Operation

Building 130 Pit – Flare Fuse Assembly Magazine

Building 1885/1886 – Pits and Sumps from Phosphorus Mixing Bldg.

Sampling Approach for SWMU 27 Drainage Ditches



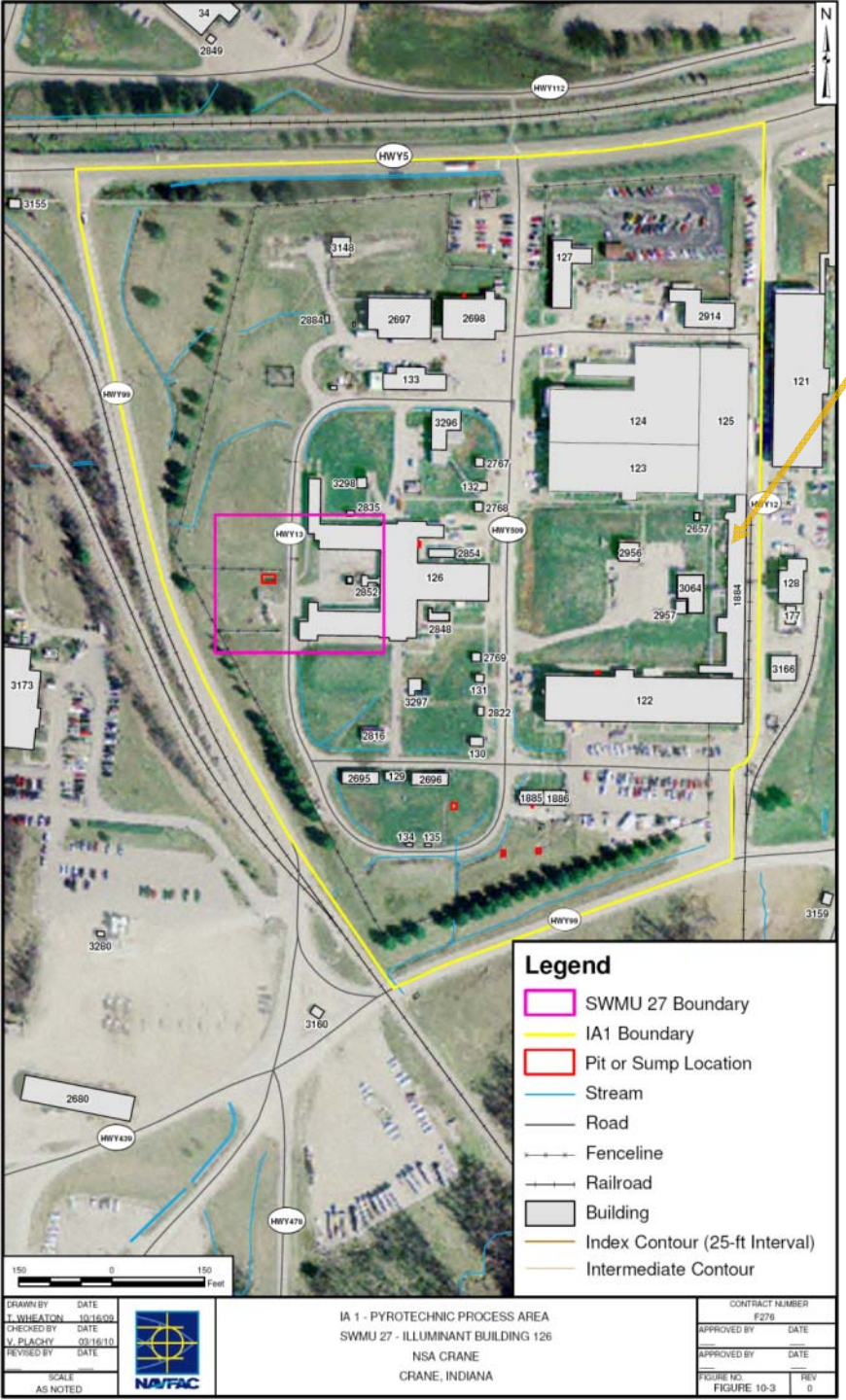
Near discharge from Building 127 – Former testing laboratory. The former north end of Building 127 once housed a burn chamber for flare tests in the 1960's - site of past test chamber explosion .

Drainage ditch that carries surface flow from the northwestern quadrant of the PPA (including Building 133).

Drainage culvert / ditch behind Building 1884 (Metal Plating Operations) where plating rinsewaters may have been discharged.

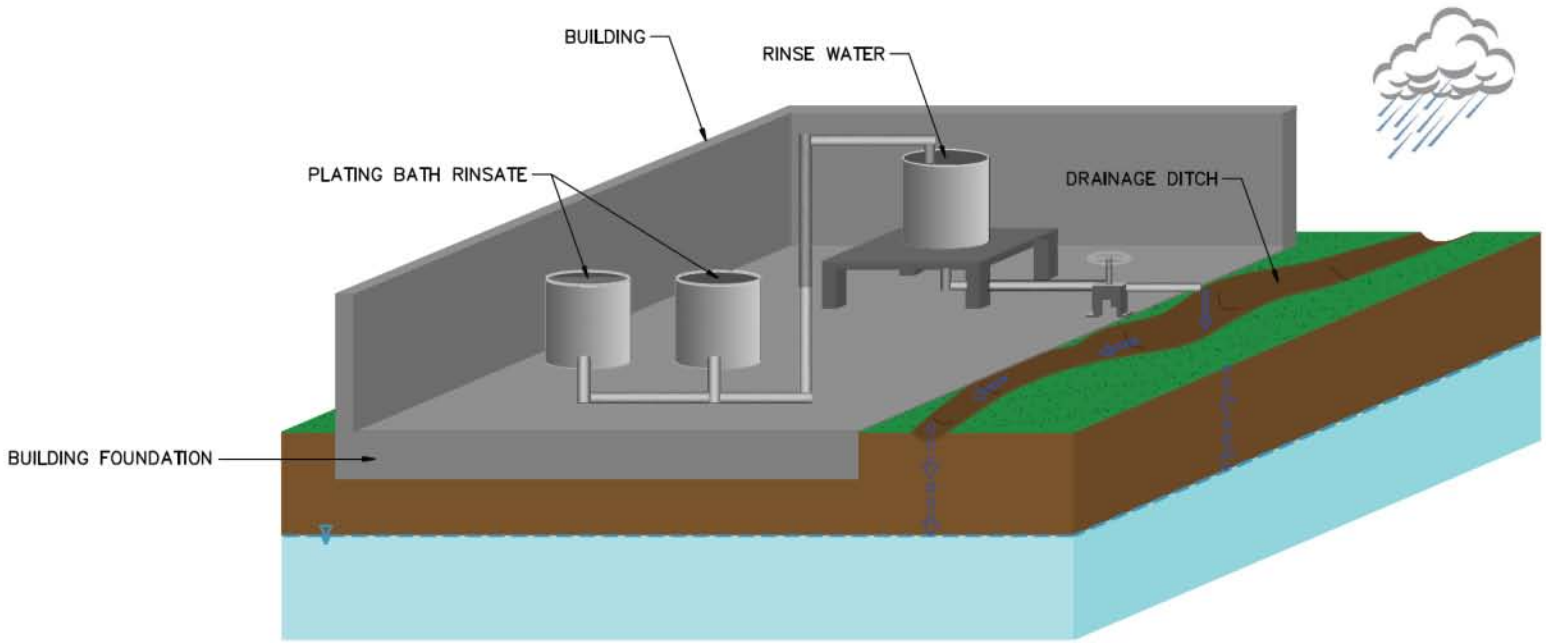
Drainage ditches that are downslope of the Building 130 Pit – Flare Fuse Assembly Magazine, downslope of the Building 1885/1886 – Phosphorus Mixing Building Pits and downslope of other chemical storage buildings and magazines in the southern portion of the PPA.

SWMU 27 Investigation Area #1 – Metals Plating Discharges



- Building 1884, the metal-plating shop has utilized metals, caustics, acids, and cyanides in the PPA and significant heavy metal contamination (zinc and cadmium) has been experienced from wastewater being discharged into open ditches.
- There has been heavy metal (zinc and cadmium) contamination identified in open ditches that drain into Boggs Creek.
- A pre-treatment facility was constructed for the plating shop (late 1970's) and has been used since that time to treat wastewater prior to discharge to the sanitary sewer system under an NPDES permit.
- RFI sampling will be performed to determine local impacts of known or suspected rinsewater discharge locations to drainage ditches.
- The general Building 1884 area has been modified and includes the removal of railroad tracks near the loading dock. Concrete now covers the area.

CSM and Sampling Approach SWMU 27 Plating Rinsewater Discharges to Ditches



LEGEND

SURFACE RUNOFF FLOW DIRECTION

INFILTRATION

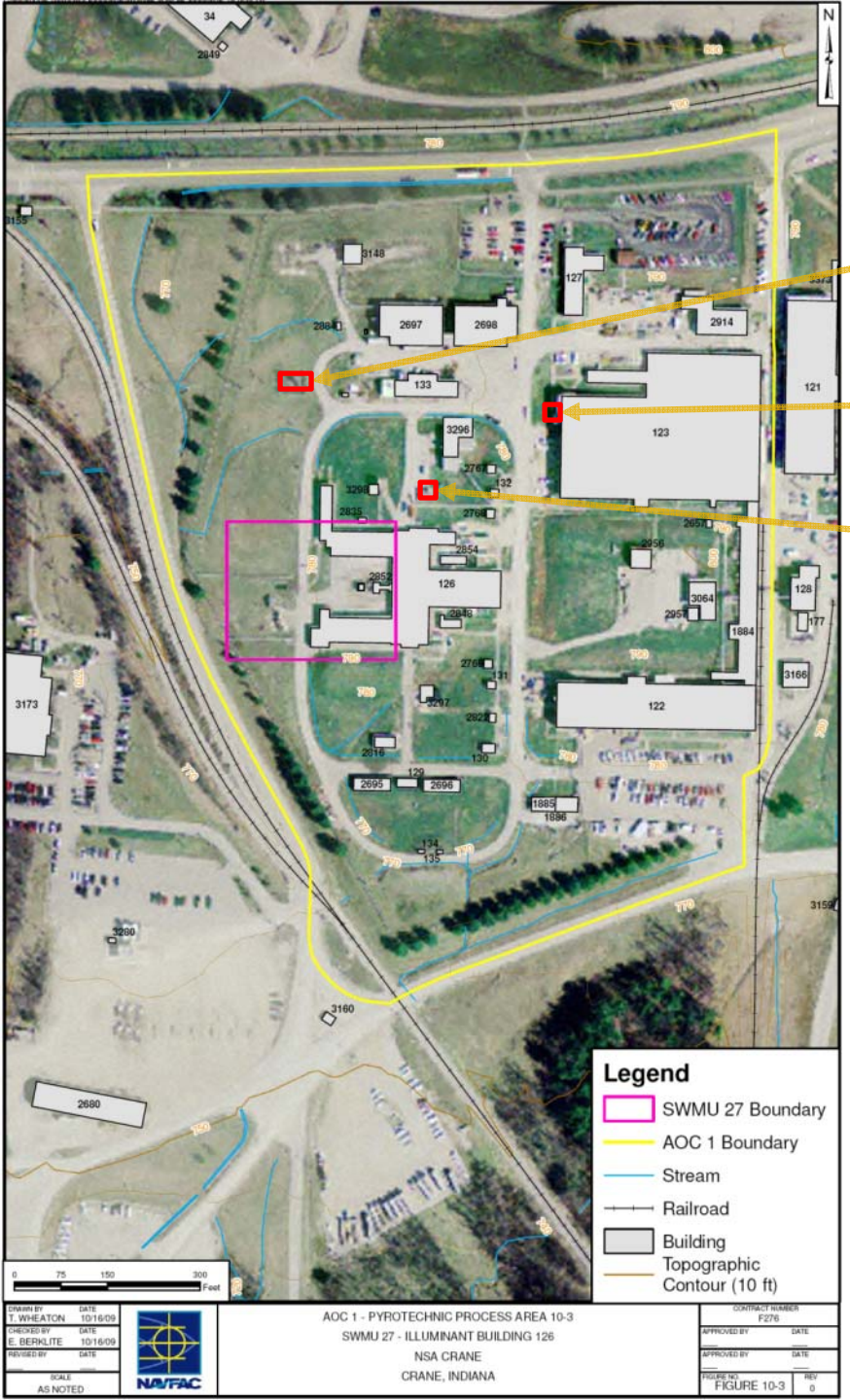
WATER TABLE ELEVATION (ESTIMATED)

0–2 FT BGS

DRAWN BY CK DATE 4/07/10	NAVFAC	CONCEPTUAL SITE MODEL CATCH BASINS, SETTLING BASINS, AND DRAINAGWAYS NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY DATE			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10–11 REV. 0

SWMU 27 Investigation Area #1
Transformer Areas

SWMU 27 RFI Investigation Area #1



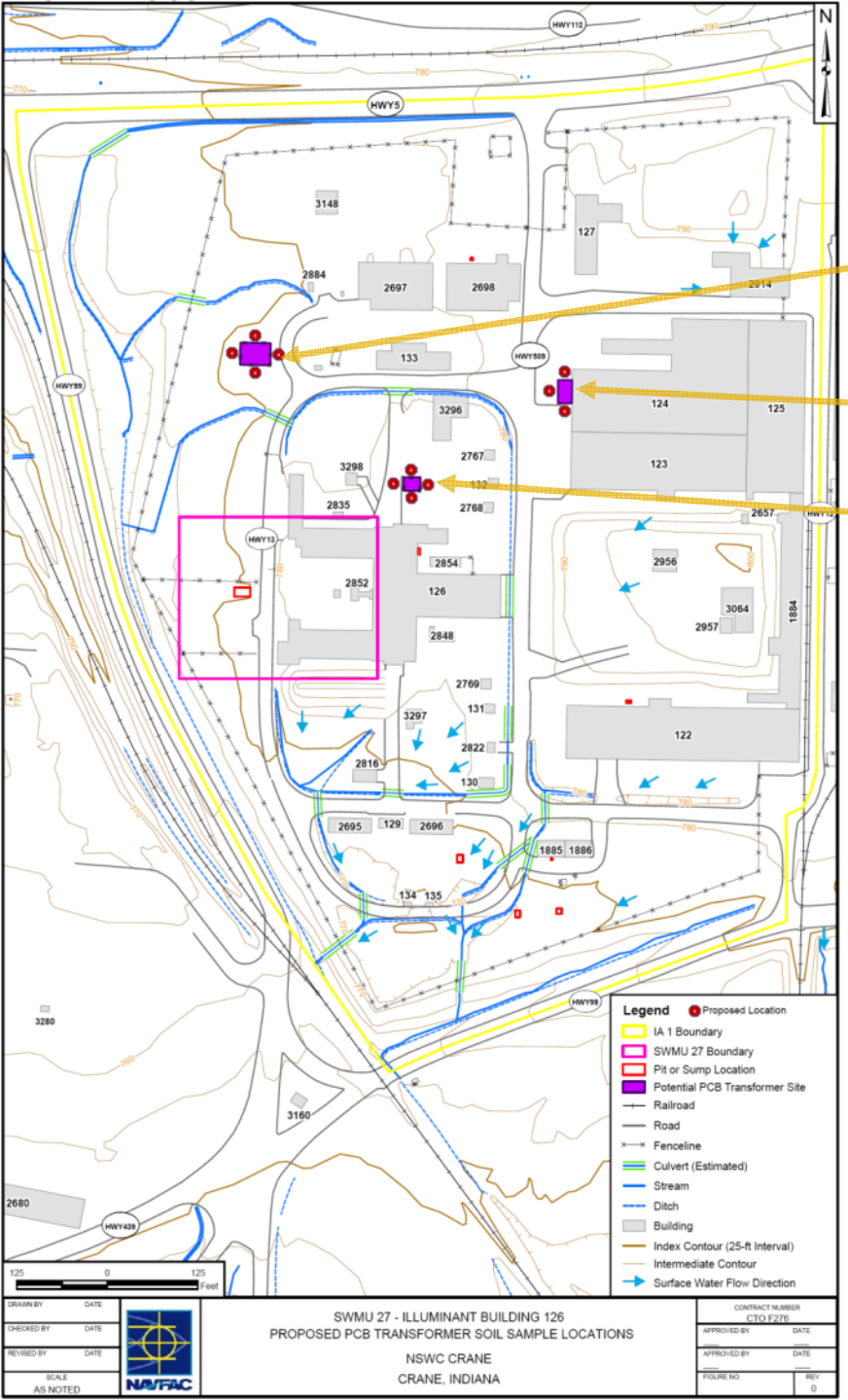
Former Transformer Yard West of Building 133 – Phosphorus Pressing House (all transformers removed from fenced enclosure)

Transformer on West Side of Building 124 – Machine Shop (labeled as containing PCBs).

Central PPA Transformer North of Building 126 – Illuminant Building (labeled as containing PCBs)

- A fenced former transformer yard is present in the northwestern quadrant of the PPA (west of Building 133). The transformers have been removed from this location.
- Active transformers were identified during the site walk and placards on the transformers indicated that they contained PCBs.
- Limited soil sampling will be performed in the immediate vicinity of these units to confirm that there has been no release to the environment surrounding the transformer units.

SWMU 27 RFI Investigation Area #1



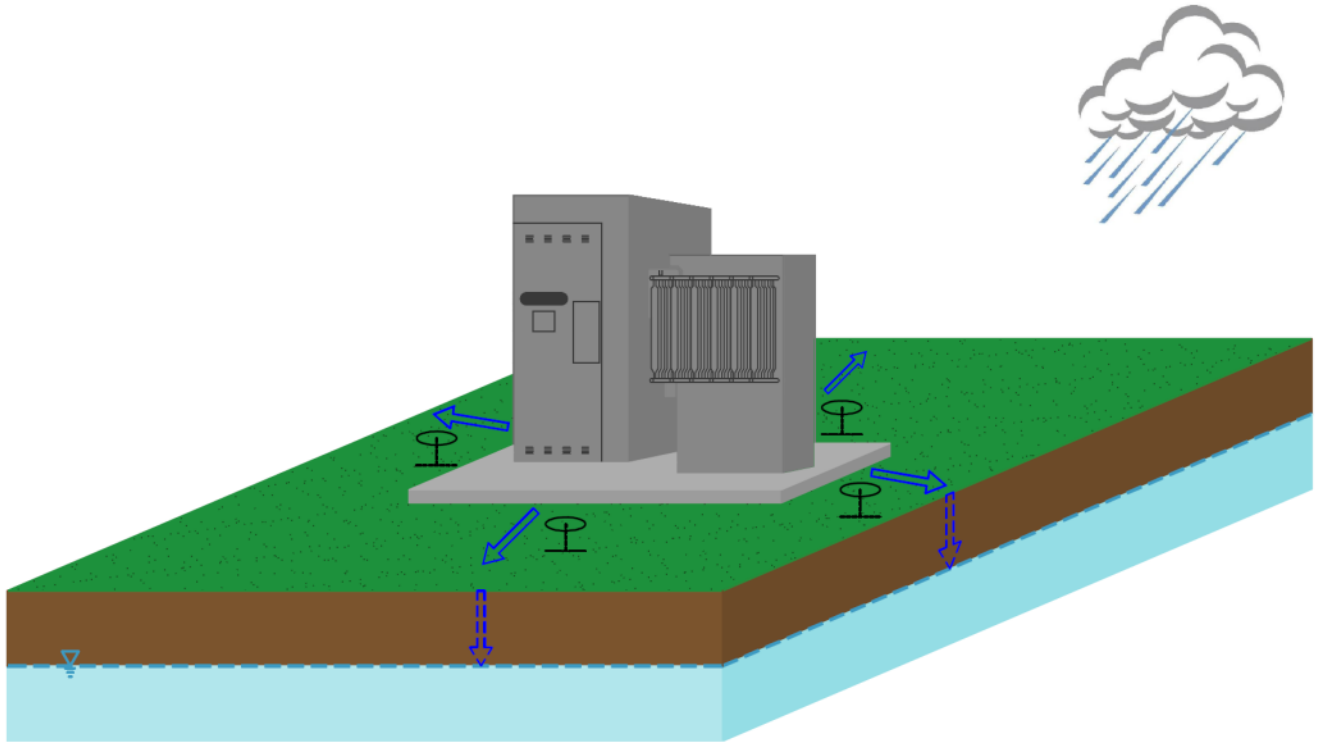
Former Transformer Yard West of Building 133 – Phosphorus Pressing House (all transformers removed from fenced enclosure)

Transformer on West Side of Building 124 – Machine Shop (labeled as containing PCBs).

Central PPA Transformer North of Building 126 – Illuminant Building (labeled as containing PCBs)

- RFI sampling for PCB arochlors will be performed in soil samples collected adjacent to the transformer pads to determine there were leaks of transformer fluids which released PCB arochlors to the local environment (surface soil).
- Phase I will include only adjacent surface soil samples. If necessary to further delineate extent of PCB releases, additional samples may be collected from more distant surface soils or surface drainages (as necessary) during Phases II and III.

CSM and Sampling Approach
SWMU 27 Electrical Transformer Areas



LEGEND

SURFACE RUNOFF FLOW DIRECTION

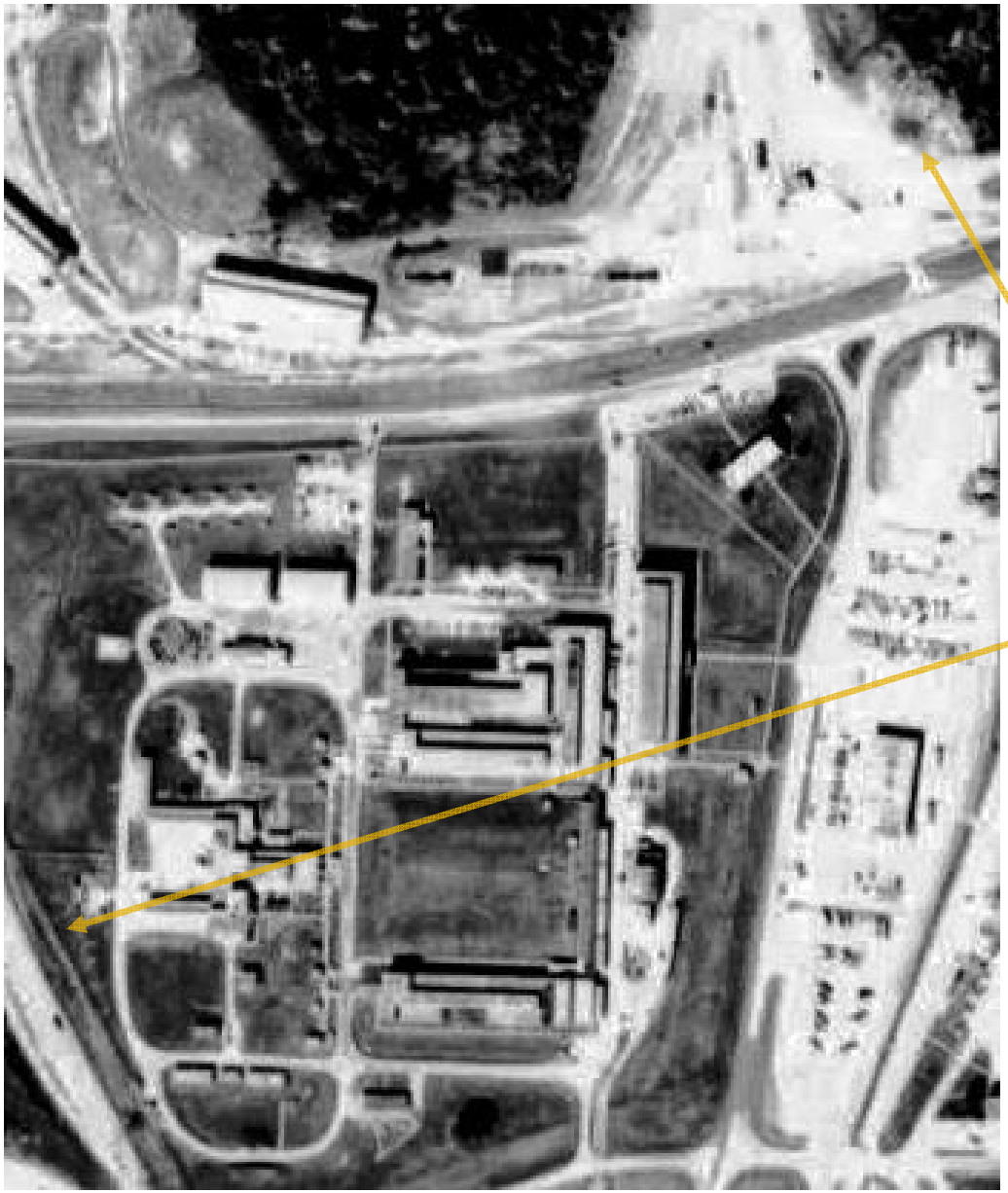
INFILTRATION

WATER TABLE ELEVATION (ESTIMATED)

0–2 FT BGS

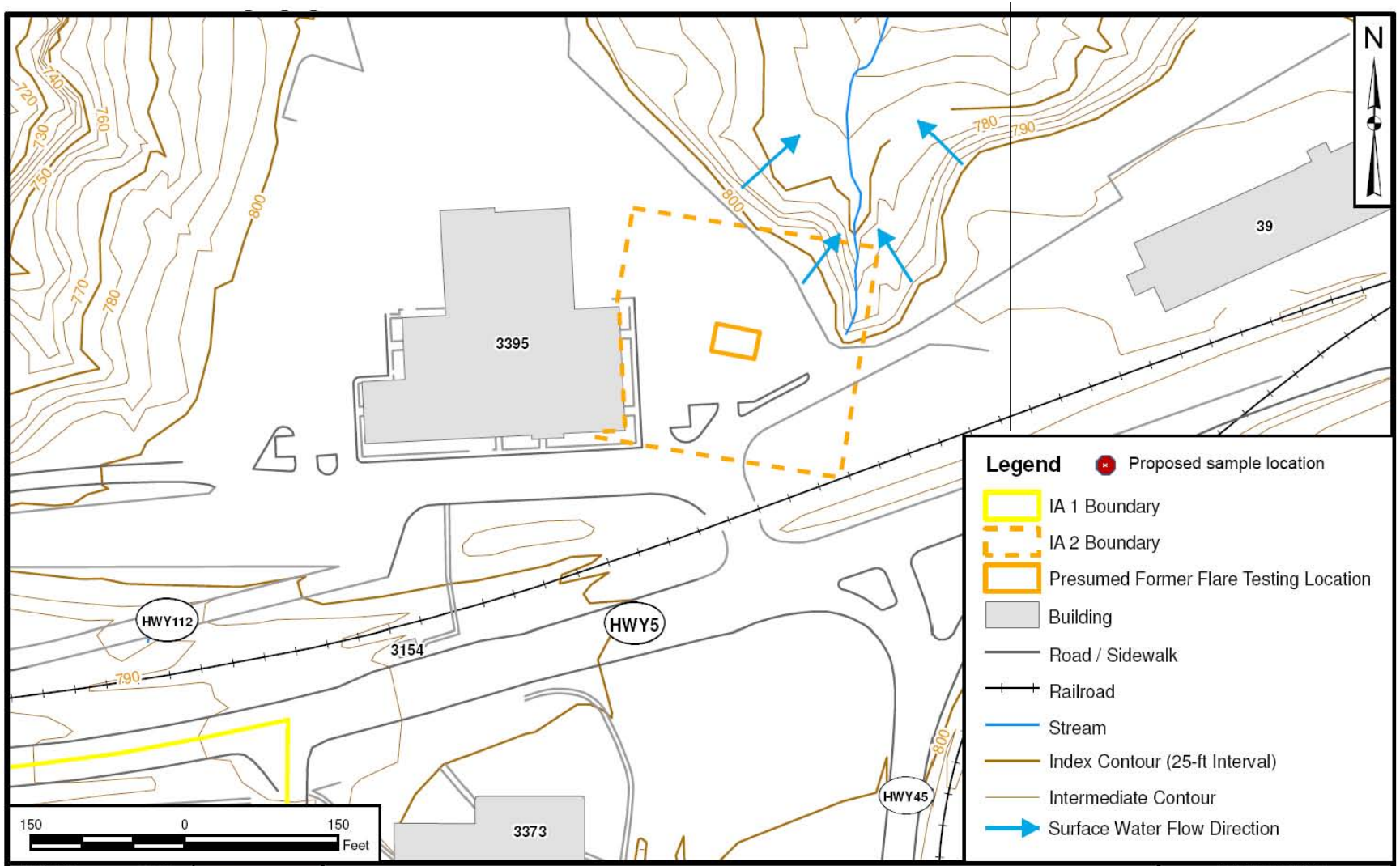
DRAWN BY CK DATE 4/07/10	NAVFAC	CONCEPTUAL SITE MODEL POLYCHLORINATED BIPHENYLS (PCBs) TRANSFORMERS NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY DATE			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10–10 REV. 0

SWMU 27 RFI – Flare Testing / Burn Areas



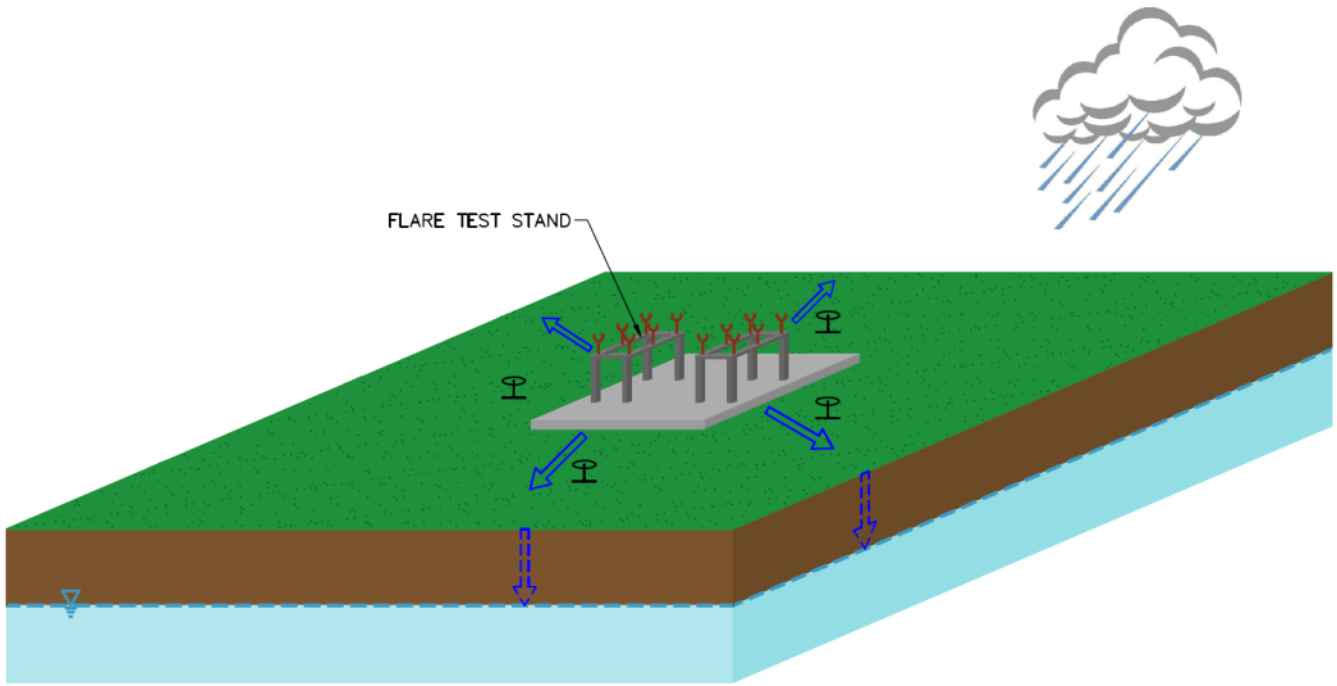
- There were pyrotechnic flare testing and burn areas that were used years ago to support PPA operations.
- Burning at those locations reportedly ceased in 1971.
- This 1966 aerial photograph may reveal the location of one of these test/burn sites that was reportedly across Highway 5. This area has been added to the SWMU 27 RFI as Investigation Area #2
- Another flare testing/burn area was reported to be behind Building 126 and may correspond to a site where two concrete basins were later constructed to support flare testing and burning operations. The basins and the general vicinity were recently sampled during performance of the MRP SI at NSA Crane. No additional sampling is proposed for this location.

PPA Figures for SWMU 27 RFI



DRAWN BY	DATE	SWMU 27 - ILLUMINANT BUILDING 126	CONTRACT NUMBER CTO F276
CHECKED BY	DATE		APPROVED BY ___ DATE ___
REVISED BY	DATE	NSWC CRANE	APPROVED BY ___ DATE ___
SCALE AS NOTED	NAVFAC	CRANE, INDIANA	FIGURE NO. / REV 0

CSM and Sampling Approach
SWMU 27 Flare Test / Burn Areas



LEGEND

SURFACE RUNOFF FLOW DIRECTION

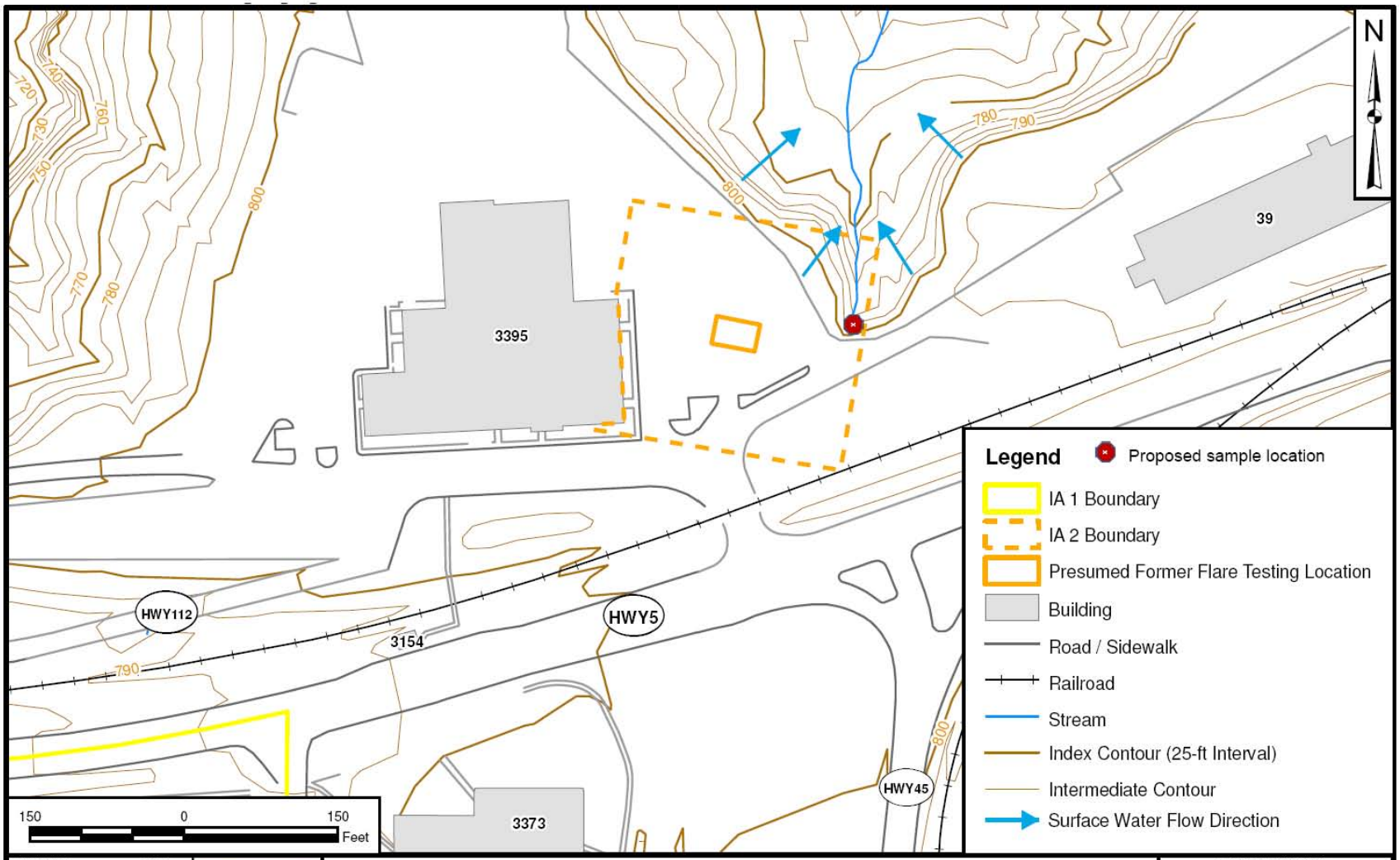
INFILTRATION

WATER TABLE ELEVATION (ESTIMATED)

0–2 FT BGS

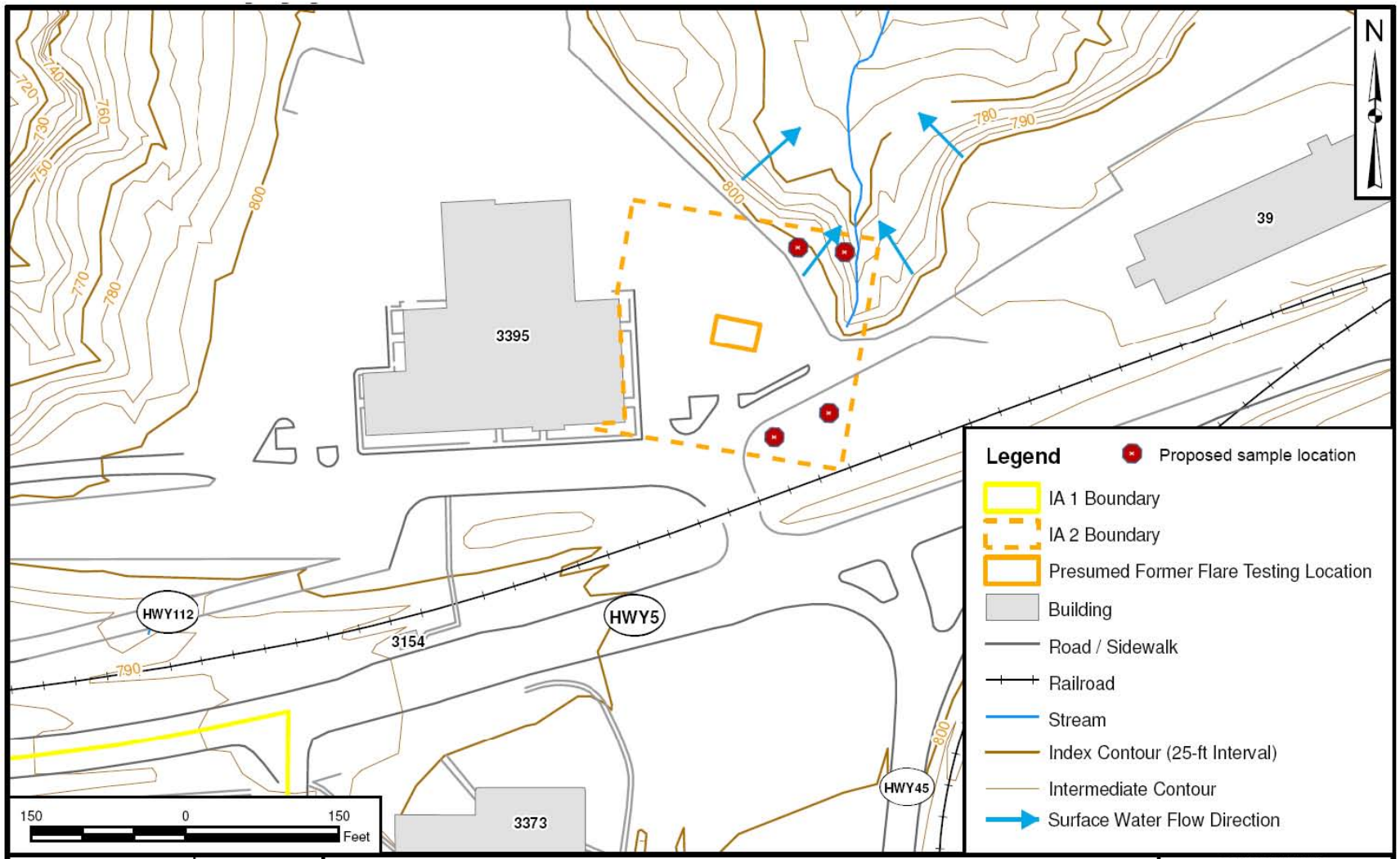
DRAWN BY CK DATE 4/07/10	NAVFAC	CONCEPTUAL SITE MODEL FLARE TESTING ACTIVITIES NSA CRANE CRANE, INDIANA	CONTRACT NO. 2126
CHECKED BY DATE			OWNER NO.
REVISED BY DATE			APPROVED BY DATE
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10–9 REV. 0

Sampling Approach for SWMU 27 Flare Test / Burn Area Ditches/Drainages



DRAWN BY	DATE	SWMU 27 - ILLUMINANT BUILDING 126	CONTRACT NUMBER CTO F276
CHECKED BY	DATE	IA 2 SURFACE DITCHES/DRAINAGEWAYS SEDIMENT/RUN-OFF SAMPLE LOCATIONS	APPROVED BY / DATE
REVISED BY	DATE	NSWC CRANE	APPROVED BY / DATE
SCALE AS NOTED	NAVFAC	CRANE, INDIANA	FIGURE NO. / REV 0

Sampling Approach SWMU 27
Flare Test / Burn Area Soil Samples



DRAWN BY	DATE	SWMU 27 - ILLUMINANT BUILDING 126
CHECKED BY	DATE	IA 1 FLARE TEST AREA PROPOSED SOIL SAMPLE LOCATIONS
REVISED BY	DATE	NSWC CRANE
SCALE AS NOTED		CRANE, INDIANA

NAVFAC

CONTRACT NUMBER CTO F276	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO.	REV 0

June 10, 2010 DQO Meeting

Attendees:

- Doug Griffin (IDEM) (dgriffin@idem.in.gov) ((317) 233-2710)
- Tom Brent (NSA Crane) (thomas.brent@navy.mil) ((812) 854-6160)
- Ralph Basinski (Tetra Tech) (ralph.basinski@tetratech.com) ((412) 921-8308)
- Peggy Churchill (Tetra Tech) (peggy.churchill@tetratech.com) ((321) 636-6470 x1300)
- Rick Barringer (Tetra Tech) (richard.barringer@tetratech.com) ((412) 921-8524)
- Valerie Plachy (Tetra Tech) (valerie.plachy@tetratech.com) ((412) 921-8389)

DISCUSSION NOTES:

1. Overview and discussion of the PPA site including background information, PPA activities, historical / previous investigations (IAS; PR/VSI), historical releases, general terminology.
2. Overview and discussion of the PPA processes and associated chemicals used.

Comments/Decisions:

Initial data collection efforts occurred during a September 16 to18, 2009, site visit to NSA Crane. SWMU 27 historical use and available data were discussed. A Trip Report is provided in Appendix A.

The steps for the phased RFI in accordance with the UFP-SAP format were discussed.

The DQO meeting was conducted on June 10, 2010. The meeting participants developed project quality objectives (PQOs) using USEPA's seven-step DQO process. A site visit/walk was conducted to review proposed sample location. The rationale for the selection of the sample locations and analytical parameters for each location was discussed and consensus was obtained. Tetra Tech was assigned the task of preparing a draft UFP-SAP. Detailed meeting discussion and decision items are presented in Appendix A.

CONSENSUS ITEMS:

1. Analysis for red phosphorus is not required because it will not linger in the environment, and it has been assigned to the unproblematic environmental category (German Environmental Protection Agency) based upon the March 1990 Assessment of Red Phosphorus in the Environment by the US Army Biomedical Research and Development Laboratory at Fort Detrick. Fredrick, Maryland.
2. The metal-plating process (Building 1884) is of concern regarding historical potential use of metals and chlorinated solvents.
3. SWMU 27 (PPA and suspected flare test area north of Highway 5) does not provide ecological habitat because it is located in an industrial area of the facility.
4. SWMU 27 likely does not impact ecological receptors in Boggs Creek and Lake Galimore.
5. Detection of off-site contamination during the RFI could require ecological risk screening. If an ecological risk screening is required, it will be addressed in an addendum.
6. The residue material found in the drainageways and channels within SWMU 27 should be referred to as soil not sediment.
7. Drainageway and channel soil samples will be collected and composited from the 0 feet below ground surface (bgs) to 1 foot bgs.
8. Phase I samples for soil surrounding the catch basins will be four-point composite samples (one grab from each side) except for Pit 126B.
9. Phase I Pit 126B samples will be two four-point composite samples along each side of the concrete.
10. A residual material four-point composite sample will be collected from all non-active catch basins. The active catch basins are Pit 133 and Pit 126B.
11. All Phase I catch basin samples will include one basin exterior composite soil sample at the catch basin low point from 1 foot bgs to the depth of the catch basin.
12. Phase I samples for soil surrounding the PCB transformer pads will be four-point composite samples (one grab from each side) except for the transformer west of Building 124 and the former transformer area.
13. Phase I former transformer area samples will be two four-point composite samples along each side of the concrete.
14. The investigation of each suspected flare test area north of Highway 5 will consist of two four-point composite samples if the area is not covered with asphalt.
15. Asphalted suspected flare test areas north of Highway 5 will not be sampled during Phase I unless a direct-push technology (DPT) rig is required for other sample locations.

16. Volatile organic compounds (VOCs) rather than total petroleum hydrocarbons and benzene, toluene, ethylbenzene, and xylenes (BTEX) will be included in the analytical program.
17. Catch basin, residual sump material, and drainageway/channel soil samples will be analyzed for:
    - VOCs
    - Perchlorate
    - Cyanide (Only in The Plating Shop area and down channel of The Plating Shop)
    - Select metals:
        - Aluminum
        - Barium
        - Cadmium
        - Total Chromium
        - Copper
        - Lead
        - Magnesium
        - Manganese
        - Potassium
        - Strontium
        - Sodium
        - Zinc
18. Samples from PCB transformer areas will be analyzed for:
    - PCBs:
        - Aroclor-1016
        - Aroclor-1221
        - Aroclor-1232
        - Aroclor-1242
        - Aroclor-1248
        - Aroclor-1254
        - Aroclor-1260
        - Aroclor-1262
        - Aroclor-1268
        - Total Aroclor
19. Samples from suspected flare test areas and fire incident areas will be analyzed for:
    - Perchlorate
    - Select metals:
        - Aluminum
        - Barium
        - Cadmium
        - Total Chromium
        - Copper
        - Lead
        - Magnesium
        - Manganese
        - Potassium
        - Strontium

- Sodium
- Zinc

ACTION ITEMS:

1. An internet search will be conducted by Tetra Tech regarding the METX 1726 (P-F) and RA Brite (MIL-ETCH) that were used in Building 1884.
    - See METX 1726 MSDS
    - RA Brite (MIL-ETCH) did not find MSDS for this item.
2. Tetra Tech is to plot the suspected flare test areas north of Highway 5 to determine if they are located within an asphalted area.
    - Plot incorporated in UFP-QAPP/SAP figures for IA 2
3. Tetra Tech will review historical figures and drawings to determine the depth of the exterior catch basin samples.
    - Typical catch basin provided in Appendix A of UFP-QAPP
    - Depth incorporated into incorporated in UFP-QAPP/SAP catch basin figures for IA 1

1. Catch basin, residual sump material, and drainageway/channel soils will be analyzed for:
    - VOCs
    - Perchlorate
    - Cyanide (ONLY IN THE PLATING SHOP AREA AND DOWN CHANNEL OF THE PLATING SHOP)
    - Select metals:
        - Aluminum
        - Barium
        - Cadmium
        - Total Chromium
        - Copper
        - Lead
        - Magnesium
        - Manganese
        - Potassium
        - Strontium
        - Sodium
        - Zinc
2. PCB transformer areas will be analyzed for:
    - PCBs:
        - Aroclor-1016
        - Aroclor-1221
        - Aroclor-1232
        - Aroclor-1242
        - Aroclor-1248
        - Aroclor-1254
        - Aroclor-1260
        - Aroclor-1262
        - Aroclor-1268
        - Total Aroclor
3. Suspected flare test areas and fire incident areas will be analyzed for:
    - Perchlorate
    - Select metals:
        - Aluminum
        - Barium
        - Cadmium
        - Total Chromium
        - Copper
        - Lead
        - Magnesium
        - Manganese
        - Potassium
        - Strontium
        - Sodium
        - Zinc

MACDERMID INCORPORATED -- METEX E-1726 -- 6850-01-151-7067

========== Product Identification ==========

Product ID:METEX E-1726
MSDS Date:11/27/1985
FSC:6850
NIIN:01-151-7067
MSDS Number: BJCSG
=== Responsible Party ===
Company Name:MACDERMID INCORPORATED
Address:526 HUNTINGDON AVENUE
City:WATERBURY
State:CT
ZIP:06720
Info Phone Num:(313) 644-5626
Emergency Phone Num:(203) 575-5700
Preparer's Name:CHERRIE D. GILLIS
CAGE:IFK56
=== Contractor Identification ===
Company Name:MACDERMID INC
Address:50 BROOKSIDE RD
Box:671
City:WATERBURY
State:CT
ZIP:06720
Country:US
Phone:203-575-5700
CAGE:76071
Company Name:MACDERMID INCORPORATED
Address:526 HUNTINGDON AVENUE
City:WATERBURY
State:CT
ZIP:06708
Phone:203-575-5700,313-644-5626
CAGE:IFK56

========== Composition/Information on Ingredients ==========

Ingred Name:SODIUM HYDROXIDE (SARA III)
CAS:1310-73-2
RTECS #:WB4900000
Fraction by Wt: 50%
Other REC Limits:2MG/M3 CEILING NIOSH
OSHA PEL:2 MG/M3
ACGIH TLV:C 2 MG/M3; 9293
EPA Rpt Qty:1000 LBS
DOT Rpt Qty:1000 LBS

========== Hazards Identification ==========

Routes of Entry: Inhalation:YES Skin:NO Ingestion:NO
Reports of Carcinogenicity:NTP:NO IARC:NO OSHA:NO
Health Hazards Acute and Chronic:INHALATION: IRRITATION & BURNS TO EYES, SKIN & MUCOUS MEMBRANES.
Explanation of Carcinogenicity:NONE
Effects of Overexposure:INHALATION: IRRITATION & BURNS TO EYES, SKIN & MUCOUS MEMBRANES.

First Aid Measures

First Aid:EYES: FLUSH W/WATER FOR 15 MINUTES. CONTACT PHYSICIAN. SKIN: FLUSH W/COLD WATER. WASH W/VINEGAR. INGESTION: GIVE WATER. CONTACT PHYSICIAN.

Fire Fighting Measures

Flash Point:NON-FLAMMABLE
Extinguishing Media:AS APPROPRIATE FOR SURROUNDING MATERIALS.
Fire Fighting Procedures:NONE
Unusual Fire/Explosion Hazard:EXOTHERMIC REACTION UPON MIXING W/WATER.

Accidental Release Measures

Spill Release Procedures:SWEEP UP AS MUCH AS POSSIBLE. FLUSH REMAINING W/COLD WATER.
Neutralizing Agent:NEUTRALIZE TO PH OF 6 TO 8 W/DILUTE ACID BEFORE DISCARDING.

Handling and Storage

Handling and Storage Precautions:STORE IN DRY AREA.
Other Precautions:NONE

Exposure Controls/Personal Protection

Respiratory Protection:DUST MASK
Ventilation:MECHANICAL
Protective Gloves:RUBBER
Eye Protection:FACE SHIELD, SAFETY CHEMICAL GOGGLES
Other Protective Equipment:RUBBER BOOTS & APRON
Supplemental Safety and Health

Physical/Chemical Properties

Vapor Pres:0
Solubility in Water:APPRECIABLE
Appearance and Odor:OFF-WHITE GRANULAR MIXTURE
Percent Volatiles by Volume:0%

Stability and Reactivity Data

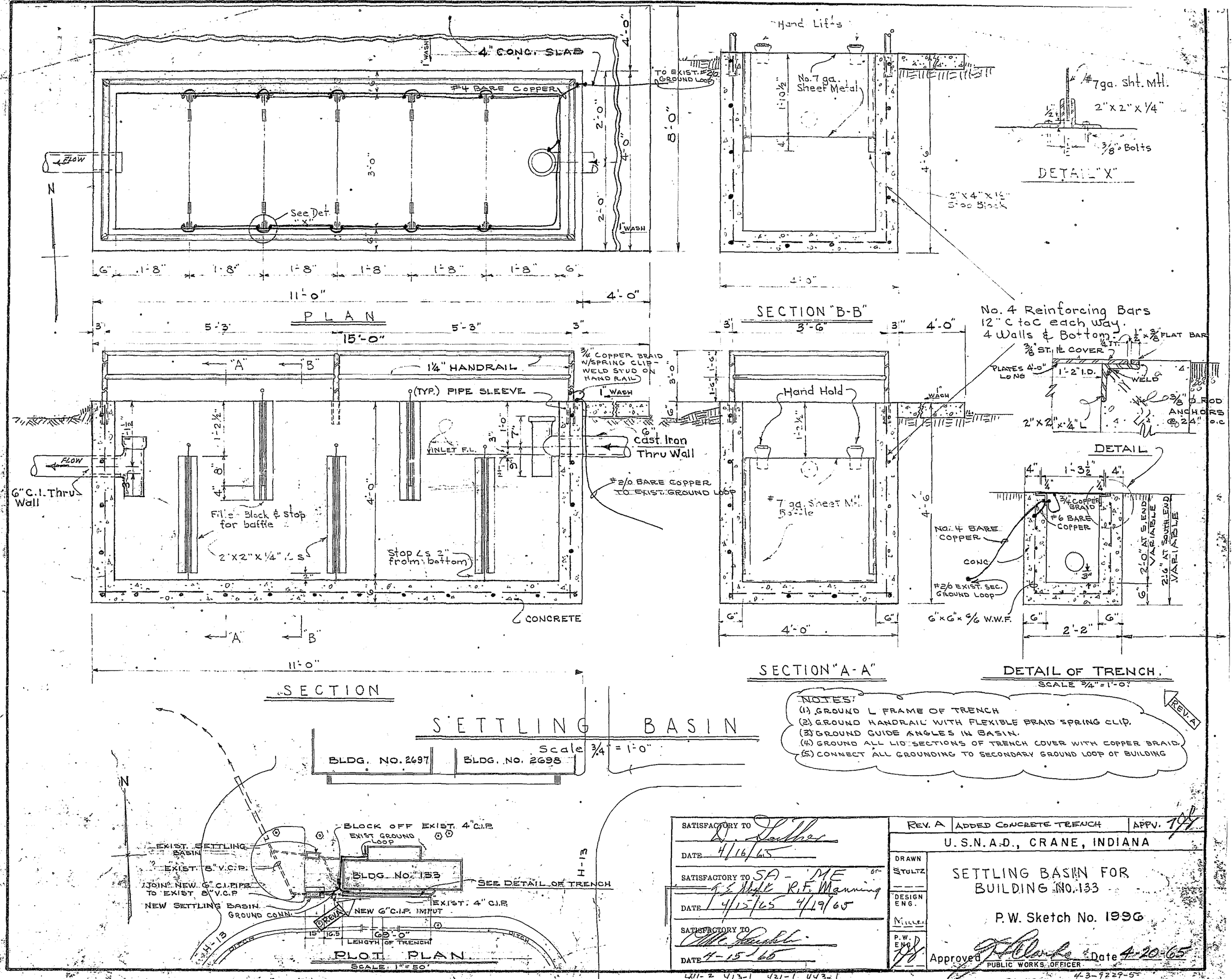
Stability Indicator/Materials to Avoid:YES
ACIDS & ACIDIC MATERIALS
Stability Condition to Avoid:DON'T ADD TO HOT WATER.
Hazardous Decomposition Products:OXIDES OF CARBON

Disposal Considerations

Waste Disposal Methods:CONSULT LOCAL, STATE, OR FEDERAL REGULATIONS PRIOR TO DISPOSAL. CORROSIVE MATERIAL UN1823.

Disclaimer (provided with this information by the compiling agencies): This information is formulated for use by elements of the Department of Defense. The United States of America in no manner whatsoever, expressly or implied, warrants this information to be accurate and disclaims all liability for its use. Any person utilizing this document should seek competent professional advice to verify and assume responsibility for the suitability of this information to their

particular situation.


4" CONC. SLAB
1" WASH
#4 BARE COPPER
TO EXIST. #2/0 GROUND LOOP
FLOW
N
See Det. "X"
3'-0"
2'-0"
4'-0"
8'-0"
6" 1'-8" 1'-8" 1'-8" 1'-8" 1'-8" 1'-8" 6"
11'-0"
4'-0"
PLAN
Hand Lifts
No. 7 ga. Sheet Metal
1'-10½"
4'-6"
2"x4"x¼" S.S. Block
4'-0"
SECTION "B-B"
3'-6"
#7ga. sht. Mtl.
2"x2"x¼"
½"
3/8" Bolts
DETAIL "X"
No. 4 Reinforcing Bars 12" c to c each way. 4 Walls & Bottom.
½"x3/8" FLAT BAR
3/8" ST. ⊥ COVER
¼"
PLATES 4'-0" LONG
1'-2" I.D.
WELD
3/8" ⌀ ROD ANCHORS @ 24" o.c.
2"x2"x¼" L
DETAIL
3 5'-3" 5'-3" 3
15'-0"
"A" "B"
1¼" HANDRAIL
(TYP.) PIPE SLEEVE
¾" COPPER BRAID W/SPRING CLIP - WELD STUD ON HAND RAIL
1" WASH
6" Cast Iron Thru Wall
INLET F.L.
#2/0 BARE COPPER TO EXIST. GROUND LOOP
6" C.I. Thru Wall
FLOW
1'-2½"
8"
4"
Filler Block & stop for baffle
2"x2"x¼" L's
Stop L's 2" from bottom
4'-0"
CONCRETE
11'-0"
SECTION
Hand Hold
#7 ga. Sheet Mtl. Baffle
4'-6"
4'-0"
SECTION "A-A"
4" 1'-3½" 4"
¼"
¾" COPPER BRAID
#6 BARE COPPER
NO. 4 BARE COPPER
CONC.
#2/0 EXIST. SEC. GROUND LOOP
6"x6"x6/6 W.W.F.
2'-0" AT SOUTH END VARIABLE
2'-6" AT SOUTH END VARIABLE
2'-2"
DETAIL OF TRENCH.
SCALE 3/4" = 1'-0"
SETTLING BASIN
Scale 3/4" = 1'-0"
BLDG. NO. 2697
BLDG. NO. 2698
NOTES:
(1) GROUND L FRAME OF TRENCH
(2) GROUND HANDRAIL WITH FLEXIBLE BRAID SPRING CLIP.
(3) GROUND GUIDE ANGLES IN BASIN.
(4) GROUND ALL LID SECTIONS OF TRENCH COVER WITH COPPER BRAID.
(5) CONNECT ALL GROUNDING TO SECONDARY GROUND LOOP OF BUILDING
REV. A
N
BLOCK OFF EXIST. 4" C.I.P.
EXIST. GROUND LOOP
EXIST. SETTLING BASIN
EXIST. 8" V.C.P.
JOIN. NEW 6" C.I. PIPE TO EXIST. 8" V.C.P.
NEW SETTLING BASIN GROUND CONN.
BLDG. No. 133
SEE DETAIL OF TRENCH
EXIST. 4" C.I.P.
NEW 6" C.I.P. INPUT
H-13
15' 16.5 63'-0"
LENGTH OF TRENCH
DITCH
PLOT PLAN
SCALE: 1" = 50'
SATISFACTORY TO D. Gerber
DATE 4/16/65
SATISFACTORY TO SA - ME
J. E. Hyde R.F. Manning
DATE 4/15/65 4/19/65
SATISFACTORY TO
DATE 4-15-65
REV. A | ADDED CONCRETE TRENCH | APPV.
U.S.N.A.D., CRANE, INDIANA
DRAWN STULTZ
DESIGN ENG. MILLER
P.W. ENG.
SETTLING BASIN FOR BUILDING NO. 133
P.W. Sketch No. 1996
Approved Date 4-20-65
PUBLIC WORKS OFFICER
W.R. 31-589-65
4-3-9229-5
411-2 413-1 421-1 443-1
412-1 426-1 441-1

A.4

RED PHOSPHORUS TECHNICAL REPORT

DTIC FILE COPY

AD

AD-A221 704

TECHNICAL REPORT 9005

ASSESSMENT OF RED PHOSPHORUS IN THE ENVIRONMENT

Wayne R. Mitchell
Elizabeth P. Burrows

DTIC
ELECTE
MAY 17 1990
S E D

MARCH 1990

U S ARMY BIOMEDICAL RESEARCH & DEVELOPMENT LABORATORY
Fort Detrick
Frederick, MD 21701-5010

Approved for public release;
distribution unlimited

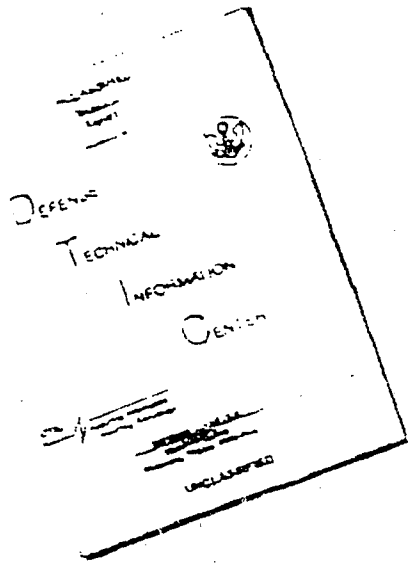
U S ARMY MEDICAL RESEARCH & DEVELOPMENT COMMAND
Fort Detrick
Frederick, MD 21701-5012

90 05 16 174

DISCLAIMER

The findings in this report are not to be construed as an official Department of the Army position unless designated by other authorized documents.

DISCLAIMER NOTICE



THIS DOCUMENT IS BEST QUALITY AVAILABLE. THE COPY FURNISHED TO DTIC CONTAINED A SIGNIFICANT NUMBER OF PAGES WHICH DO NOT REPRODUCE LEGIBLY.

REPRODUCED FROM
BEST AVAILABLE COPY

THIS DOCUMENT CONTAINED
BLANK PAGES THAT HAVE
BEEN DELETED

Unclassified
SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No 0704-0188
Exp Date Jun 30 1986

1a REPORT SECURITY CLASSIFICATION Unclassified	1b RESTRICTIVE MARKINGS
2a SECURITY CLASSIFICATION AUTHORITY	3 DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited
2b DECLASSIFICATION/DOWNGRADING SCHEDULE	
4 PERFORMING ORGANIZATION REPORT NUMBER(S) Technical Report 9005	5. MONITORING ORGANIZATION REPORT NUMBER(S)
6a NAME OF PERFORMING ORGANIZATION U.S. Army Biomedical Research and Development Laboratory 6b OFFICE SYMBOL (If applicable) SGRD-UBG-E	7a NAME OF MONITORING ORGANIZATION
6c. ADDRESS (City, State, and ZIP Code) Fort Detrick Frederick, MD 21701-5010	7b ADDRESS (City, State, and ZIP Code)
8a. NAME OF FUNDING/SPONSORING ORGANIZATION U.S. Army Corps of Engineers 8b OFFICE SYMBOL (If applicable)	9 PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
8c. ADDRESS (City, State, and ZIP Code) 200 Massachusetts Avenue, NW Washington, DC 20003	10. SOURCE OF FUNDING NUMBERS PROGRAM ELEMENT NO / PROJECT NO / TASK NO / WORK UNIT ACCESSION NO

11. TITLE (Include Security Classification)

(U) Assessment of Red Phosphorus in the Environment

12 PERSONAL AUTHOR(S)
Wayne R. Mitchell, Elizabeth P. Burrows

13a. TYPE OF REPORT	13b TIME COVERED	14 DATE OF REPORT (Year, Month, Day)	15 PAGE COUNT
Technical	FROM May 88 TO Feb 90	1990 March 1	17

16. SUPPLEMENTARY NOTATION

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	red phosphorus, phosphate(s), XM819 mortar, smoke(s), phosphine, orthophosphoric acid, obscurant(s), L8A1 grenade(s), environmental fate and effects

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

This report is a brief critical review of the current literature relevant to the environmental and health effects of the use of red phosphorus in smoke and obscurant munitions by the Army. Properties of the element in its red allotropic form and its many combustion products, various oxyacids of phosphorus, are discussed within the context of their release to the environment in testing and military training operations. The possible consequences of increased environmental burdens of acid and phosphate deposition are considered, and toxicological effects are reviewed briefly.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT ☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS	21 ABSTRACT SECURITY CLASSIFICATION Unclassified
22a. NAME OF RESPONSIBLE INDIVIDUAL Wayne R. Mitchell	22b TELEPHONE (Include Area Code) 301-663-2538 / 22c OFFICE SYMBOL SGRD-UBG-E

DD FORM 1473, 84 MAR — 83 APR edition may be used until exhausted. All other editions are obsolete — SECURITY CLASSIFICATION OF THIS PAGE

TABLE OF CONTENTS

INTRODUCTION 3

PHYSICAL AND CHEMICAL PROPERTIES 3

MANUFACTURE AND USE BY THE ARMY 7

ANALYTICAL METHODS 8

ENVIRONMENTAL FATE AND EFFECTS 9

TOXICITY 10

CONCLUSIONS AND RECOMMENDATIONS 11

REFERENCES 12

DISTRIBUTION LIST 16

TABLES

1. Physical and Chemical Properties of Red Phosphorus 5

2. Phosphorus, Selected Phosphorus Compounds, and their Oxidation States 6

FIGURE

1. Three-dimensional structures of two of the allotropic forms of phosphorus: (a) white phosphorus tetrahedron; (b) red phosphorus chain 4

Accession For
NTIS GRA&I ☒
DTIC TAB ☐
Unannounced ☐
Justification
By
Distribution/
Availability Codes
Dist | Avail and/or Special
A-1

DTIC COPY INSPECTED 4

INTRODUCTION

The utilization of smokes and obscurants by the U.S. Army is essential for the achievement of tactical goals such as protection of potential targets and masking personnel and materiel movements. Toward that end and to ensure defense preparedness, large quantities of such munitions are used in military training areas throughout the United States and abroad. Environmental contamination associated with these munitions could occur as a result of their manufacture or their deployment. In view of present day concerns over risks associated with foreign substances in the environment, an understanding of the nature of such chemicals, their entry and fate in the environment, and their impact on living things is necessary to assess the degree of these risks realistically.

Elemental red phosphorus is a principal component in the formulation of several obscurant munitions produced and used by the U.S. Army. In grenades, mortar rounds, and a proposed howitzer shell, the element is deployed explosively over wide areas and burns rapidly to form oxides that can themselves react with water vapor to form aerosol particles of phosphoric acids. It is the purpose of this report to assess current literature regarding red phosphorus obscurant munitions as they relate to health and the environment.

PHYSICAL AND CHEMICAL PROPERTIES

Phosphorus is the 11th most common element in the earth's crust. It occurs commonly in the lithosphere in igneous, sedimentary, and metamorphic rock species and can be found as vast minable reserves in marine deposits [1]. In the biosphere [2] it is an essential nutrient in the formation of structural biomolecules such as membrane phospholipids, functional macromolecules like nucleic acids and adenosine triphosphate, and metabolic intermediates such as the sugar phosphates [3]. Human activities, such as the manufacture and use of detergents, fertilizers, and water softeners, contribute significant amounts of phosphorus to the environment [4]. However, in all of the above cases phosphorus exists in combination with other elements, whereas natural elemental phosphorus is not found free in the environment.

Depending upon the nature of interatomic bonds established during its formation, solid elemental phosphorus can occur in three major allotropic forms: black, white, or red. The black allotrope is the least reactive of the three due to its highly polymerized nature, and has no military application at this time. White phosphorus, the best known and commercially most important phosphorus allotrope, is considerably more reactive than the other solid forms, ignites spontaneously in air, and can be dissolved in various organic solvents. Its atoms exist as P4 tetrahedra (figure 1a), accounting for its solubility and reactivity [5]. Because of its impact on health and the environment, white phosphorus has been reviewed recently in a context of its military applications [6].

The red allotropic form of elemental phosphorus is intermediate to the black and white varieties in reactivity, has military application as noted above, but has received considerably less attention in the literature than the white allotrope. Red phosphorus atoms are thought to form high polymers in which single bonds of phosphorus tetrahedra are broken and linked together forming chains and rings [7] (figure 1b). In this configuration, phosphorus is less reactive than

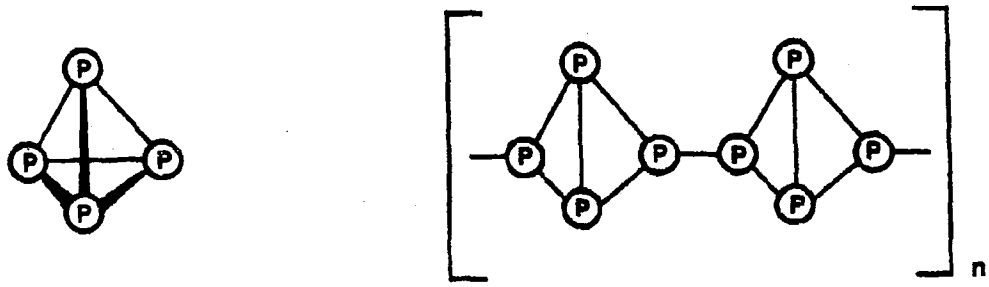


Figure 1. Three-dimensional structures of two of the allotropic forms of phosphorus: (a) white phosphorus tetrahedron; (b) red phosphorus chain

in the white allotropic form and is insoluble in water as well as in organic solvents. Its greater stability (and insolubility) is explained by the necessity of breaking polymeric phosphorus bonds prior to the formation of new ones [8]. Nevertheless, red phosphorus can react slowly with oxygen and water vapor to evolve phosphine [9]. Moreover, since its oxidation is exothermic, spontaneous combustion has been noted in storage piles of this elemental form. Metallic impurities in the stored solid can catalyze the oxidation [5]. In addition to heating and exposure to oxidizing substances, ignition of red phosphorus can be accomplished by percussion, contact, and friction [5,10]. The chemical and physical properties of elemental red phosphorus are summarized in Table 1.

Table 1. Physical and Chemical Properties of Red Phosphorus

Property	Value	Reference
CAS NO.	7723-14-0	5
Formula	polymeric $(P4)_n$	7
Molecular Weight	$(123.9)_n$	11
Appearance	red-brown	11
Density	2.34 g/cm^3	11
Melting Point	sublimes at 416°C	11
Heat of Sublimation	19.7 kcal/mol	11
Ignition Temperature	280°C in air	11
Critical Temperature	589°C	11
Critical Pressure	43.1 atm	11
Heat Capacity	5.1 cal/deg/mol (P)	11
Solubility	insoluble in organic solvents, negligible in water	10
Uses	pyrotechnics, safety matches, fertilizers, pesticides, incendiaries, smoke bombs	10

As a Group VA element in the periodic table, with 5 electrons in its valence shell (3p), phosphorus commonly bonds three or four adjacent atoms forming molecules with three or five shared electrons. However, the element can also form stable compounds in which its oxidation number ranges from -3 to +5 owing to its capacity to fill s, p, and d orbitals [8,12]. Examples of elemental phosphorus and some of its more important compounds and their various oxidation states are given in Table 2.

Since the military application of red phosphorus as an obscurant involves combustion, its oxides (Table 2) are of practical importance in that they will be released directly to the environment. When combusted in a free supply of air, red phosphorus burns to form phosphorus pentoxide as a predominant combustion product. Alternatively, under conditions of oxygen deficiency, the formation of phosphorus trioxide is favored [8], and phosphorus tetroxide may also be present [13]. The phosphorus oxides themselves hydrolyze readily in water to form corresponding oxyacids, and in the case of phosphorus pentoxide, further condensation reactions result in the formation of polymeric phosphate products (Table 2). Thus, depending upon the availability of oxygen for combustion, water (or its vapor) for product dissolution, and heat to drive reactions, the burning of elemental phosphorus can result in complex mixtures of phosphorus compounds. Monomeric oxyacids such as phosphoric, phosphorous, and hypophosphorous acids and condensation products such as the linear pyro- and polyphosphates and cyclic metaphosphates can occur simultaneously in solution following combustion of the element [13-16].

Table 2. Phosphorus, Selected Phosphorus Compounds, and their Oxidation States

Substance				Oxidation State
Phosphines				
Phosphine	PH_3			-1
Diphosphine	P_2H_4			-2
Elemental Phosphorus				
White phosphorus	P_4			0
Red phosphorus	$(P_4)_n$			0
Oxides and their Oxyacids				
-		Hypophosphorous Acid	H_3PO_2	+1
Phosphorus trioxide	P_4O_6	Phosphorous Acid	H_3PO_3	+3
Phosphorus tetroxide	P_2O_4	Hypophosphoric Acid	$H_4P_2O_6$	+4
Phosphorus Pentoxide	P_4O_{10}	Orthophosphate (Phosphoric Acid)	H_3PO_4	+5
		Pyrophosphate (Diphosphoric Acid)	$H_4P_2O_7$	+5
		Polyphosphates (linear) (Triphosphoric Acid)	$H_5P_3O_{10}$	+5
		Tetraphosphoric Acid	$H_6P_4O_{13}$	
		Metaphosphates (cyclic) (Trimetaphosphoric Acid)	$H_3P_3O_9$	+5
		Tetrametaphophoric Acid	$H_4P_4O_{12}$	

Phosphine, a highly toxic gas, can be formed along with the above-described mixture of oxyacids from red phosphorus in oxidations taking place in the presence of water vapor and less than stoichiometric quantities of oxygen [13]. The reaction is extremely slow at normal temperatures and humidities [17] and it would not be a factor in the deployment of phosphorus munitions in military field exercises.

MANUFACTURE AND USE BY THE ARMY

The manufacture of white phosphorus from natural raw materials, its conversion to red phosphorus, and the many pollution problems associated with industrial phosphorus production and military white phosphorus processing have been described previously [5,6,18]. Since red phosphorus is not manufactured by the Army but is obtained commercially from sources abroad, production wastes are not a problem in this country [13]. However, blending and filling operations for red phosphorus munitions, as well as their testing, take place at Pine Bluff Arsenal (PBA), Arkansas. The principal munitions to be manufactured in the future will be smoke-forming grenades and mortar rounds.

There are currently 226,000 L8A1 grenades in the inventory [19]. This munition, which ignites while airborne, is designed to obscure vehicles from which grenade clusters are launched. The obscurant portion of the L8A1 grenade consists of 360 grams of a 95 to 5 mixture of red phosphorus and butyl rubber formulated in granules [11,20]. These grenades were originally obtained by the U.S. Army from Great Britain and were then produced at PBA until 1985, after which time their production was discontinued. In 1987, production began on the L8A3 grenade, a modification of the L8A1 that will eventually replace it [19]. The newer model is of similar design to the L8A1 but employs silica instead of talc as the granule coating material, to reduce phosphorus clumping and thereby improve its burning qualities [21]. During grenade production, red phosphorus is plasticized with butyl rubber in an organic solvent and extruded. The resulting granules are dried, pressed into pellets, and then inserted into the rubber sleeve of the grenade [11]. It has been estimated that at full production as many as 10,000 L8A3 grenades per month could be produced at PBA [22].

Red phosphorus is also the major smoke-generating ingredient in the XM819 mortar round. In this munition, it is compounded with sodium nitrate and an epoxy binder in an approximate ratio of 80:14:6 parts by weight, respectively [23]. Fill for the mortar rounds will be prepared by blending powdered red phosphorus, sodium nitrate, and epoxy binder with appropriate curing agents and solvents, granulating the resulting slurry, and pressing it into wedges for loading. During loading, mortar rounds will receive 28 wedges weighing approximately 43 grams each. The munition is designed so that following its detonation, ignited wedges will disperse both in the air and on the ground [23]. Consideration is also currently being given to the design and production of a third red phosphorus munition, the XM803 howitzer round, for even larger scale smoke generation [24].

ANALYTICAL METHODS

While direct chemical analysis of elemental red phosphorus is not feasible due to its polymeric nature and insolubility, it is readily oxidized to the +5 state and then analyzed by standard gravimetric and volumetric [25], potentiometric [26], or colorimetric [27] methods for orthophosphate. For example, red phosphorus was first solubilized with aqua regia, oxidized with ammonium persulfate and sulfuric acid to orthophosphate, and then analyzed spectrophotometrically as a phosphomolybdate complex [28]. Alternatively, Spanggord [26] recently assayed red phosphorus by combusting it to completion, boiling the collected products in water to ensure conversion to orthophosphate, and measuring total phosphorus by titrating the one strongly ionized hydrogen present per phosphorus atom. Application of such methods to environmental samples containing elemental phosphorus would encounter interferences from naturally occurring phosphates and would not differentiate between the red and white allotropes. Conceivably, interfering phosphates could be removed from such samples by aqueous extraction (converted to a water soluble form if necessary) and white phosphorus could similarly be removed by extraction with an organic solvent prior to oxidation or combustion as described above.

In addition, phosphates can be assayed more rigorously and specifically by a number of fluorometric, radiological, atomic adsorption, and nuclear magnetic resonance techniques [13]. However, since the burning of phosphorus and dissolution of its oxides in water results in the formation of a variety of compounds, more general methods appropriate to the assay of phosphate mixtures have been developed. Paper, thin layer, and open-column chromatographic systems have been applied to the mixtures with varying degrees of success. One thin layer system, in which elution was conducted with acidic and then basic solvents, separated several of the monomeric phosphorus oxyacids, pyrophosphate, and a few meta- and polyphosphates. Similarly, open column ion exchange and gel chromatographic systems have resulted in partial separations of monomeric and condensed phosphate species in the mixtures [13]. More recently, high performance liquid chromatographic (HPLC) procedures coupling ion exchange columns to potentiometric or spectrophotometric detection systems have expanded phosphate separations (26,29-31]. Typically, Spanggord et al. [26] resolved ortho-, pyro-, tri-, and tetraphosphoric acids, as well as homologous linear condensed polyphosphates (P_6 to P_{16}) and several cyclic metaphosphate species by means of such columns eluted with sodium chloride gradients and quantified them as colored molybdate complexes. Likewise, modification of ion chromatographic systems has facilitated the differential analysis of lower oxyacids of phosphorus including separations of orthophosphate, phosphinate and phosphonate species [31,32].

Both paper strip and silica gel collector systems have been used for the colorimetric detection of phosphine. In the former case, mercuric chloride in methyl yellow was used as an indicator for HCl produced during the formation of $P(HgCl)_3$, whereas in the latter, color changes resulted from reaction of phosphine with impregnating $AuCl_3$ [13]. Gas chromatography has provided more sophisticated quantitative analysis of phosphine in combustion product vapors. Levels of 0.04 ppm and perhaps less have been demonstrated by means of isothermal analysis (75°, 80/100 mesh Porapak P columns) and nitrogen-phosphorus detection [26].

ENVIRONMENTAL EFFECTS AND FATE

Because of its unreactive polymeric nature, low vapor pressure, and insolubility, undisturbed red phosphorus is relatively inert in the environment. Its presence unchanged in soils as a consequence of Army operations is innocuous, with one important exception to be described shortly. Indeed, red phosphorus has been intentionally applied to soils experimentally as a possible fertilizer [33,34]. Surface films of oxides formed slowly (these have also been observed in the laboratory), but no further reaction or dissolution was found. However, as a consequence of both testing of munitions at PBA and deployment of grenades in military training exercises elsewhere, incompletely combusted clumps of oxide-coated red phosphorus have been observed and are believed to account for fires occurring at the training sites [19]. Thus the possible ignition of these residual clumps either spontaneously or by friction or other disturbance presents a potential threat for environmental damage from forest and grass fires.

The environmental impact of the combustion products of red phosphorus was an important consideration. Laboratory analyses have shown that essentially the same products result from burning of either the red or white allotrope: oxides of phosphorus which in the presence of atmospheric moisture form complex mixtures of polymeric phosphoric acids and other oxyacids of phosphorus. In the environment the polymeric oxyacids are known to break down both chemically and microbiologically with increasing time to the ultimate product, orthophosphoric acid [16,26,30,31]. Two possible adverse effects then needed to be evaluated: an increase in acid deposition and an increase in phosphate levels in the local waters. It can be generally concluded that pH effects in water will be of little consequence unless the buffering capacity of the system is exceeded. For experimental aquatic systems, a relationship between buffering capacity, pH, and total phosphorus has been developed which indicates that excessive smoke deposition is necessary to bring about pH changes [31]. Specifically, a pond ($3000\ m^2 \times 1.5\ m$) containing 4.5×10^6 L of water at an alkalinity of 100 mg/L $CaCO_3$ would require the deposition of approximately 230 kg of total combusted phosphorus to lower the pH to 6. As a matter of perspective, such a change would require the complete combustion and total obscurant deposition in the system of somewhat more than 700 L8-series grenades.

On the other hand, it has been shown that combustion products of phosphorus can stimulate algal growth in experimental systems at levels which do not significantly acidify poorly buffered growth media. Optimal phosphorus-phosphate for growth of green algae occurs in the range of 0.02 to 0.6 mg/L whereas that for blue-green species occurs at higher levels of up to 3.0 mg/L [31]. Because of these growth responses at low phosphate levels, relatively small amounts of red phosphorus combustion products could affect algal levels without producing detectable pH changes. As part of the lake eutrophication process, the excessive input of phosphorus to water systems leading to nuisance algal blooms is well known [35]. Thus the repeated high level deposition of red phosphorus obscurant combustion products to aqueous systems, either directly or as a result of runoff, has the potential to decrement surface water quality. Even then, however, the degree to which such an effect would be manifested would be a function of the nature of the receiving body, its trophic state, and the time of the year. For example, an algal bloom would more likely be encountered in a small lake with a low flushing rate during spring when phosphorus is limiting than in a rapidly

moving stream in summer and fall when nitrogen may be deficient [36].

Analysis of soils following the experimental burning of red (and white) phosphorus in two different microcosm systems has defined no more than minor ecological effects [24,37]. In neither study were significant pH effects noted even at high aerosol concentrations. However, in the first study, nutrient losses (particularly calcium) in leachates were detected after the application of phosphorus smokes at concentrations far exceeding those anticipated during field use (1500 mg/m^3), but were attributed to biological and not physicochemical processes. Similarly, in the second study, transient mobility of trace elements was noted following repeated applications of phosphorus smokes and was attributed to their interactions with various phosphates formed after deposition. Such mobility was shown to decrease with the soil volume in the microcosm studies, and for that reason has been suggested to be minimal on a field scale.

In terrestrial microcosm studies little effect has been noted on total respiration of environmental microorganisms in soil even after exposure to extremely high concentrations of smokes [37]. On the other hand, exposure of some soils to elevated smoke concentrations can reduce nitrification and microbial dehydrogenase activity, ostensibly due to acidification, and depress levels of microbial phosphatase activity, possibly as a result of orthophosphate end product inhibition [39]. Even then, however, it has been concluded that the effects occur predominantly at soil surfaces, and in the case of nitrification are transient [24]. For plant species including sweet clover, rye-grass, and wheat exposed to concentrations several times higher than typical field levels (50-400 mg/m^3), no more than minor adverse effects were detected; and it has been concluded based on these and similar results that repeated deployment of the smokes at or below 600 mg/m^3 for a period of eight weeks should be possible without adverse ecological effects [37]. Moreover, toxic effects noted at high mass loadings on other plant species including pine seedlings, bushbeans, blando bromegrass, and sagebrush were, with the exception of the bromegrass, reported to be transient and could be ameliorated by rainfall [24]. For aquatic organisms including several fish, invertebrates, and an algal species, combustion products of phosphorus were not toxic except at high concentrations, in which case the effects were related to acidification of water after its buffering capacity was exhausted rather than to the phosphates themselves [31].

TOXICITY

Little if any significant toxicity appears to be associated with elemental red phosphorus unless it is contaminated with the white allotropic form [39]. When applied under patches to the intact or abraded skin of rabbits at doses of 0.5 g per site, no dermal irritation was noted following 24 hours of exposure to red phosphorus. Similarly, dermal application of the element to guinea pigs resulted in no skin irritation or sensitization responses, and interdermal injection resulted in only slight irritation with no skin sensitization. Doses of 100 mg did not result in rabbit eye irritation. Even at oral doses of 10,000 mg/Kg, lethality in Fischer 344 male and female rats was less than 20% of the experimental subjects after 14 days, and the oral LD50 was reported as being greater than 10g/kg [28].

For phosphorus combustion products, studies have been conducted both to

establish toxicity levels for laboratory animals and humans and to estimate toxic effects on environmental fauna. Phosphorus smoke toxicity levels for mammalian species have been listed recently [38] and extensively discussed in a context of the manufacture and military use of red and white phosphorus obscurant munitions [11]. Recent results of rat aerosol exposure studies indicate that the concentration of phosphorus smokes is a much more important factor in determining mortality than is the duration of exposure [40]. With the exception of guinea pigs, which are highly susceptible to the smokes, data for laboratory animals (mice, rats, dogs, and goats) for one hour exposures indicate that the critical concentration for LC_{50} for most species is in the vicinity of 2000 mg/m^3 [11]. Lethal effects were both acute and chronic in nature and were accompanied by symptoms of respiratory distress, most commonly including pulmonary congestion, edema, and atelectasis [16,41]. In contrast, repeated short term exposures (8 minutes per day during 5 day periods for 12 weeks) of mice, rats, and rabbits to phosphorus smokes at anticipated field concentrations resulted in no irreversible toxic effects and no latent toxicity, tumor formation or reproductive defects [20]. Toxicity levels for man are unavailable; however, exposure of volunteers to concentrations of between 100 and 700 mg/m^3 for between 2 and 15 minutes resulted in significant but reversible respiratory distress symptoms, as well as mucus membrane and eye irritation [38,41]. It is estimated that human exposure to concentrations in the neighborhood of 2000 mg/m^3 for longer than 15 minutes would result in increasing deaths; concentrations of 1000 mg/m^3 are considered intolerable and 700 mg/m^3 is the minimum harassing concentration, or that concentration at which masking is mandatory [11,41].

An apparent exception to the mammalian scenario summarized above has just been published [42]. A laboratory study on two species of wildlife, rock doves and black-tailed prairie dogs, reported that these remarkable creatures survived several multiple 80 minute exposures to extremely high concentrations of up to 4000 mg/m^3 phosphorus smoke. Transient adverse behavioral and physiological responses were noted, and the birds were the more vulnerable of the two species.

CONCLUSIONS AND RECOMMENDATIONS

Except for its possible association with grass and forest fires and its potential to produce phosphine in confined spaces, elemental red phosphorus has not been demonstrated to pose a threat to the environment or health. In the former case, new red phosphorus munitions, particularly mortar rounds and howitzer shells designed to ignite close to the ground, should be thoroughly evaluated in the light of their fire hazard potential. In the latter case, the U.S. Army should determine if phosphine production and worker exposure exist at red phosphorus munitions facilities and, if so, define the steps necessary to obviate the problem. For the military burning of phosphorus during obscurant generation, it should be emphasized that levels of phosphate entering the environment are minute in comparison to those from other sources such as agricultural runoffs and natural leaching. However, the potential exists at local levels for problems resulting from the heavy and repeated use of phosphorus munitions. In that context and in the light of the extensive environmental effects data for phosphates and phosphorus smokes cited in this report, a future U.S. Army effort could be the establishment of practical site-specific guidelines for phosphorus munitions usage to preclude local environmental damage.

REFERENCES

1. McKelvey, V.E. 1973. Abundance and Distribution of Phosphorus in the Lithosphere. In Environmental Phosphorus Handbook. Griffith, E.J., A. Beeton, J.M. Spencer and D.T. Mitchell, Eds. John Wiley and Sons, Inc. New York, NY, pp. 13-32.

2. Hooper, F.F. 1973. Origin and Fate of Organic Phosphorus Compounds in Aquatic Systems. In Environmental Phosphorus Handbook. Griffith, E.J., A. Beeton, J.M. Spencer, and D.T. Mitchell, Eds. John Wiley and Sons, Inc. New York, NY, pp. 179-202.

3. Stryer, L. 1988. Biochemistry. 3rd Ed. W.H. Freeman and Co. New York, NY.

4. Jones, E.D. 1973. Phosphorus as a Factor in the United States Economy. In Environmental Phoshorus Handbook. Griffith, E.J., A. Beeton, J.M. Spencer, and D.T. Mitchell, Eds. John Wiley and Sons, Inc. New York, NY, pp. 669-695.

5. Van Wazer, J.R. 1982. Phosphorus and the Phosphides. In Kirk-Othmer Encyclopedia of Chemical Technology, Vol. 17. 3rd Ed. Grayson, M. and D. Eckroth, Eds. John Wiley and Sons, Inc. New York, NY, pp. 473-490.

6. Davidson, K.A., P.S. Hovatter, and C.F. Sigmon. 1987. Water Quality Criteria for White Phosphorus. Final Report. AD-A186613. Project Order 84PP4845. Oak Ridge National Laboratory, Oak Ridge, TN.

7. Corbridge, D.E.C., M.S. Pearson, and C. Walling. 1966. The Structural Chemistry of Phosphorus Compounds. In Topics in Phosphorus Chemistry, Vol. 3. John Wiley and Sons, Inc. New York, NY, pp. 57-363.

8. Pauling, L. 1970. General Chemistry. 3rd Ed. W.H. Freeman and Co. San Francisco, CA.

9. Hawley, G.G. 1981. The Condensed Chemical Dictionary. 10th Ed. Van Nostrand Reinhold Co. New York, NY, pp. 810.

10. Windholz, M., S. Budavari, R.F. Blumetti, and E.S. Otterbein. 1983. The Merck Index. An Encyclopedia of Chemicals, Drugs, and Biologicals. 10th Ed. Merck and Co., Inc, Rahway, NJ, pp. 1059-1060.

11. Berkowitz, J.B., G.S. Young, R.C. Anderson, A.J. Colella, W.J. Lyman, W.D. Steber, R.G. Thomas, R.G. Vranka, and A.L. Preston. 1981. Research and Development for Health and Environmental Hazard Assessment-Task Order 5. Occupational and Environmental Hazards Associated with the Formulation and Use of White Phosphorus-Felt and Red Phosphorus-Butyl Rubber Screening Smokes. Final Report. AD-A116956. Contract DAMD17-79-C-9139. Arthur D. Little, Inc., Cambridge, MA.

12. Van Wazer, J.R. 1973. The Compounds of Phosphorus. In Environmental Phosphorus Handbook. Griffith, E.J., A. Beeton, J.M. Spencer, and D.T. Mitchell, Eds. John Wiley and Sons, Inc. New York, NY, pp. 169-178.

13. Spanggord, R.J., R.T. Podoll, R.T. Rewick, T-W. Chou, R.B. Wilson, J. Backovsky, and D.L. Roberts. 1983. Environmental Fate of White Phosphorus/Felt and Red Phosphorus/Butyl Rubber Military Screening Smokes: Phase I-Literature Review. Final Report. AD-A150754. Contract DAMD17-82-C-2320. SRI International, Menlo Park, CA.

14. Hudson, R.B. and M.J. Dolan. 1982. Phosphoric Acids and Phosphates. In Kirk-Othmer Encyclopedia of Chemical Technology, Vol. 17. 3rd Ed. Grayson, M. and D. Eckroth, Eds., pp. 426-473.

15. Boenig, I.A., M.M. Crutchfield, and C.W. Heitsch. 1982. Phosphorus Compounds. In Kirk-Othmer Encyclopedia of Chemical Technology, Vol. 17. 3rd Ed. M. Grayson and D. Eckroth, Eds., pp. 490-539.

16. Ballou, J.E. 1981. Chemical Characterization and Toxicologic Evaluation of Airborne Mixtures. Final Report. AD-A102628. Contract DAMD17-79-C-9160. Battelle Pacific Northwest Laboratories, Richland, WA.

17. Van Wazer, R.J. 1958. Phosphorus and Its Compounds. Vol. I. Interscience Publishers. New York, NY.

18. Jangaard, P.M. 1970. The Role Played by the Fisheries Research Board of Canada in the "Red" Herring Phosphorus Pollution Crisis in Placentia Bay, Newfoundland. Circular No. 1. Fisheries Research Board of Canada. Office of the Atlantic Regional Director, Research, Halifax, N.S.

19. Allen, J.T. 1988. Investigative Data on the L8A1 Smoke Grenade. Memorandum Report 15-88. U.S. Army Biomedical Research and Development Laboratory, Fort Detrick, Frederick, MD.

20. Weimer, J.T., G.E. Affleck, R.L. Farrand, F.K. Lee, and R.J. Pellerin. 1980. The Acute and Chronic Effects of Repeated Exposures to United Kingdom Red Phosphorus Screening Smokes in Rats, Mice, Guinea Pigs, and Rabbits. Technical Report ARCSL-TR-79053. AD-B051834L. Chemical Systems Laboratory, Aberdeen Proving Ground, MD.

21. Fiala, J.P. 1988. Quantitative Evaluation of L8 Screening Smoke Grenades. Memorandum for Record, SMCCR-MUP-S, 28 July 1988.

22. Garcia, D.J., J.D. Marshall, M.D. Frauenthal, L. Dickson, W.L. Fortner, and T.E. Shook. 1985. Red Phosphorus Pollution Abatement Evaluations. Technical Report SMCPB-TR-28. AD not available. Pine Bluff Arsenal, Pine Bluff, AR.

23. Ramsey, R.S., J.H. Moneyhun, and R.W. Holmberg. 1985. The Chemical and Physical Characterization of XM819 Red Phosphorus formulation and the Aerosol Produced by Its Combustion. Final Report. AD-A164442. Project Order 82PP2802. Oak Ridge National Laboratory, Oak Ridge, TN.

24. Van Voris, P., D.A. Cataldo, M.W. Ligotke, T.R. Garland, K.M. McFadden, J.K. Fredrickson, S.W. Li, R.M. Bean, B.L. Thomas, and D.W. Carlile. 1987. Evaluate and Characterize Mechanisms Controlling Transport, Fate, and Effects of Army Smokes in the Aerosol Wind Tunnel. Transport, Transformations, Fate, and

Terrestrial Ecological Effects of Red Phosphorus-Butyl Rubber and White Phosphorus Obscurant Smokes. Final Report. AD-A191109. Project Order 84PP4819. Battelle Pacific Northwest Laboratory, Richland, WA.

25. Gorzny, K. 1972. Analysis of Elemental Phosphorus. In Analytical Chemistry of Phosphorus Compounds. M. Halmann, Ed. John Wiley and Sons, Inc. New York, NY.

26. Spanggord, R.J., R. Rewick, T-W. Chou, R. Wilson, R.T. Podoll, T. Mill, R. Parnas, R. Platz, and D. Roberts. 1985. Environmental Fate of White Phosphorus/Felt and Red Phosphorus/Butyl Rubber Military Screening Smokes. Final Report. AD-A176922. Contract DAMD17-82-C-2320. SRI International, Menlo Park, CA.

27. USEPA. 1974. Manual of Methods for Chemical Analysis of Water and Wastes. EPA-625-/6-74-003. U.S. Environmental Protection Agency. National Environmental Research Center, Cincinnati, OH. pp. 249-263.

28. Henry, M.C., J.J. Barkley, and C.D. Rowlett. 1981. Mammalian Toxicological Evaluation of Hexachloroethane Smoke Mixture and Red Phosphorus. Final Report. AD-A109593. Conducted as Contract DAMD17-C-78-C-8086 by Litton Bionetics, Inc., Kensington, MD, for the U.S. Army Medical Bioengineering Research and Development Laboratory, Fort Detrick, Frederick, MD.

29. Yamaguchi, H., T. Nakamara, Y. Hirai, and S. Ohashi. 1979. High Performance Liquid Chromatographic Separation of Linear and Cyclic Condensed Phosphates. J. Chromatogr. 172:131-140.

30. Brazell, R.S., R.W. Holmberg, and J.H. Moneyhun. 1984. Application of High-Performance Liquid Chromatography-Flow Injection Analysis for the Determination of Polyphosphoric Acids in Phosphorus Smokes. J. Chromatogr. 290:163-172.

31. Poston, T.M., B.L. Thomas, K.M. McFadden, B.W. Killand, R.M. Bean, L.A. Prohammer, M.L. Clark, and D.R. Kalkwarf. 1986. Acute Toxicity of Smoke Screen Materials to Aquatic Organisms, White Phosphorus-Felt, Red Phosphorus-Butyl Rubber and SGF No. 2 Fog Oil. Final Report. AD-A167900. Project Order 84PP4800. Battelle Pacific Northwest Laboratory, Richland, WA.

32. Hirai, Y., N. Yoza, and S. Ohashi. 1981. Flow Injection System as a Post-Column Reaction Detector for High Performance Liquid Chromatography of Phosphonate and Orthophosphate. J. Chromatogr. 206:501-509.

33. Jackman, R.H., J.P. Lambert, and H.P. Rothbaum. 1970. Red Phosphorus as a Fertilizer for Grass-Clover Pasture. N. Z. J. Agr. Res. 13:242-251.

34. Sokolov, A.V., N.D. Talanov, K.F. Gladkova, G.V. Speranskaya, V.G. Bulaeva, and L.V. Vasil'eva. 1976. Use of Red Phosphorus as Fertilizer. Khim. Sel'sk. Khoz. 14:22-24.

35. Theis, T.L. and P.J. McCabe. 1978. Retardation of Sediment Phosphorus Release by Fly Ash Application. J. Water Pollut. Control Fed. 50:2666-2676.

36. Collins, A.G. and T.C. Young. 1988. J. Environ. Engr. 114:337-351.

37. Tolle, D.A., M.F. Arthur, J. Chesson, K.M. Duke, D.R. Jackson, V. Kogan, R.R. Kuhlman, and D.P. Margeson. 1988. Terrestrial Microcosm Evaluation of Two Army Smoke-Producing Compounds. Final Report. AD-A190797. Contract DAMD17-84-C4001. Battelle, Columbus Division, Columbus, OH.

38. Uhrmacher, J.C., P.P. Werschulz, D.O. Schultz, and D.O. Weber. 1985. A Health and Environmental Effects Data Base Assessment of U.S. Army Waste Material, Phase II. Final Report. AD-A175274. Contract No. DAMD17-84-C-4133. Carltech Associates, Columbia, MD.

39. Aranyi, C. 1983. Research and Development on Inhalation Toxicologic Evaluation of Red Phosphorus/Butyl Rubber Combustion Products. Phase II. Final Report. AD-A158323. Contract No. DAMD17-82-C-2121. IIT Research Institute, Chicago, IL.

40. Watsi, K., K.J.R. Abaidoo, J.E. Villaume, and P.N. Craig. 1978. Occupational Health and Safety Aspects of Phosphorus Smoke Compounds, Vol II. Final Report. AD-A056018. Contract No. DAMD17-77-C-7020. The Franklin Institute Research Laboratories, Philadelphia, PA.

41. Li, S.W., J.K. Fredrickson, M.W. Ligotke, P. Van Voris, and J.E. Rogers. 1988. Influence of Smoke Exposure on Soil Enzyme Activities and Nitrification. Biol. Fertil. Soils 6:341-346.

42. Sterner, R.T., S.A. Shumake, R.D. Thompson, and B.E. Johns. 1990. Behavioral-Physiological Effects of Red Phosphorus. Final Report. AD not yet assigned. Project Order No. 85PP5847. U.S. Department of Agriculture, Denver Wildlife Research Center, Denver, CO.

Accession Number : ADA221704

Title : Assessment of Red Phosphorus in the Environment

Descriptive Note : Technical rept. May 1988-Feb 1990

Defense Technical Information Center

Corporate Author : ARMY BIOMEDICAL RESEARCH AND DEVELOPMENT LAB FORT DETRICK MD

Personal Author(s) : Mitchell, Wayne R. ; Burrows, Elizabeth P.

Handle / proxy Url : http://handle.dtic.mil/100.2/ADA221704 Check NTIS Availability...

Report Date : 01 MAR 1990

Pagination or Media Count : 18

Abstract : This report is a brief critical review of the current literature relevant to the environmental and health effects of the use of red phosphorus in smoke and obscurant munitions by the Army. Properties of the element in its red allotropic form and its many combustion products, various oxyacids of phosphorus, are discussed within the context of their release to the environment in testing and military training operations. The possible consequences of increased environmental burdens of acid and phosphate deposition are considered, and toxicological effects are reviewed briefly. Keywords: Red phosphorus, Phosphate(s), XM819 mortar, Smoke(s), Phosphine, Orthophosphoric acid, Obscurant(s), L8A1 grenade(s), Environmental fate and effects.

Descriptors : *SMOKE MUNITIONS, *ENVIRONMENTAL IMPACT, *OBSCURATION, *RED PHOSPHORUS, MILITARY OPERATIONS, ENVIRONMENTS, HEALTH, DEPOSITION, SMOKE, RELEASE, MILITARY TRAINING, PHOSPHINE, TOXICOLOGY, PHOSPHATES, PHOSPHORIC ACIDS, COMBUSTION PRODUCTS.

Subject Categories : INORGANIC CHEMISTRY
PYROTECHNICS
AIR POLLUTION AND CONTROL

Distribution Statement : APPROVED FOR PUBLIC RELEASE

Search DTIC's Public STINET for similiar documents.

Members of the public may purchase hardcopy documents from the National Technical Information Service.

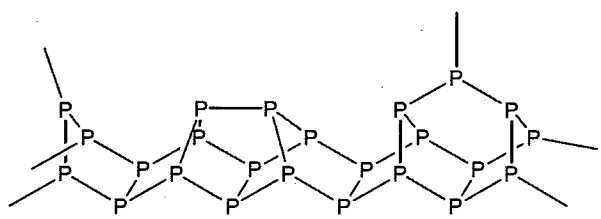
European Flame Retardants Association

FLAME RETARDANTS FACT SHEET

Red Phosphorus (RP)

SUMMARY PROFILE

Red Phosphorus is an amorphous polymeric modification of the chemical element phosphorus. Elemental phosphorus is readily oxidized and this high reactivity results in hazardous properties of the material. However, red phosphorus is much less reactive and even more less toxic than yellow (also called white) phosphorus[1] (see appendix). Since red phosphorus is a polymer, an exact molecular formula does not exist. The following structure serves as an example to illustrate how each phosphorus atom is bonded to three others:



APPLICATIONS

- Red Phosphorus is active as sole additive in polymers like polyamides, polyesters, polyurethanes, polyisocyanates and epoxy resins while in polymers like polyolefins, styrenics and rubbers it has to be applied in combination with spumific and carbonific coagents;
- typical required concentrations are 2 ... 10 %

BENEFITS

- Red Phosphorus is the more concentrate source of phosphorus therefore it is an effective flame retardant at low concentration.

1 Although red and white phosphorus are totally different chemicals they do share the same EINECS-number (231-768-7, European inventory of existing chemical substances) so that care has to be taken when data are extracted from sources based on the EINECS-number.

- Red Phosphorus does not affect the mechanical properties of polymer articles and usually retains or even improves good electrical properties of the polymers (e.g. high current tracking index, CTI).
- Red Phosphorus is endued with high thermal stability therefore can overcome drastic processing conditions up to 300°C.
- Red Phosphorus avoids the formation of greater amounts of toxic smoke and heat.
- The flame retarding effect of Red Phosphorus prevents the outbreak of large fires from small ignition sources, so that substantial harm to human beings, buildings, etc. can be prevented.

Mode of action:

Phosphorus containing flame retardants mainly influence the reactions taking place in the solid phase. They are particularly effective in materials with high oxygen content, such as cellulose and oxygen containing plastics. The flame retardant is converted by thermal decomposition to phosphoric acid, which in the condensed phase extracts water from the pyrolysing substrate, causing it to form a protective char layer on the surface of the burning item.

ENVIRONMENTAL / HEALTH ASPECTS

Health:

Red Phosphorus has a very low acute toxicity to mammalians, e.g. the LC_{50} for rats by oral application is greater than 15 000 mg per kg bodyweight. Moreover, when applied as a flame retardant, red phosphorus is usually completely embedded into a polymer matrix so that it cannot react with air or water. Due to its own polymeric nature it will not migrate or evaporate from the final product.

Environment:

Recent toxicological studies revealed an aquatic toxicity (LC_{50} or EC_{50}) in the range of 10 to 100 mg/L for fish, Daphnia and algae. In the environment red phosphorus will slowly degrade by disproportionating and hydrolysis to phosphine (PH3) and phosphorus acids (oxidation levels of I, III, V). Although the intermediate phosphine is toxic, it is also quite reactive and will be oxidized. The final products will be phosphates which are ubiquitous and harmless. From wastewater, red phosphorus is eliminated mainly by adsorption to sewage sludge. In an accidental fire or waste incineration the phosphorus will be oxidized to phosphorus oxides which are properly removed in flue gas treatment systems of state of the art incinerators or which will remain as polymeric phosphoric acid or phosphates in the bottom ash.

ISSUES

In a study on environmental properties of flame retardants, the German Environmental Protection Agency (Umweltbundesamt, 2001) has assigned red phosphorus to the category »use is unproblematic«.

SAFETY

Hazardous substance data and risk symbols (European classification and labeling):

- sign »F« highly flammable
- R 11 Highly flammable
- R 16 Explosive when mixed with oxidizing substances
- R 52 / R 53 harmful to aquatic organisms / may cause long term adverse effects in the aquatic environment
- S 7 Keep container tightly closed
- S 43.1 In case of fire, use water

Red phosphorus is very sensitive to mechanical forces like friction or impact when dry and concentrated. The safety of handling Red Phosphorus in the workplace can be increased by adding stabilisers, dust suppressants, and coating the surface by micro encapsulation.

The safest and most convenient way of processing Red Phosphorus is the use of concentrates available in a wide variety of polymer carriers covering almost all the applications.

Red Phosphorus in this form in NOT flammable, NOT classified as an hazardous product for the transportation, NOT hazardous for the handling and the industrial safety (unless carrier material is).

MANUFACTURERS / REFERENCES

EFRA
European Flame Retardants Association
Dr. Brigitte Dero
Sector Group Manager
European Chemical Industry Council (Cefic)
Av. E. Van Nieuwenhuyse 4
B - 1160 Brussels
Tel : + 32 2 676 7259
Fax: + 32 2 676 7392
Website: http://www.cefic-efra.com

MANUFACTURERS:

Clariant
Pigments and Additives Division
Industriestrasse 1
D-50354 Huerth
Telephone: +49 2233 48-6114
Fax: +49 2233 41236
Website: www.exolit.com

Italmatch Chemicals Spa
v. P. Chiesa 7/13
Torri Piane, San Benigno
I-16149 Genova
Telephone: +39 010 642081
Fax: +39 010 4695296
Website: www.italmatch.it

REFERENCES:

Umweltbundesamt 2001: Research project on "Substituting Environmentally Relevant Flame Retardants: Assessment Fundamentals". Berlin, Umweltbundesamt, UBA-FB 000171/1-3

APPENDIX: Comparison of white and red phosphorus		
	white phosphorus	**red phosphorus**
CAS-number	12185-10-3	7723-14-0
EINECS-number	231-768-7 !	
structure	discrete P_4 molecules	highly polymeric P_n
appearance	crystalline, waxy, translucent	amorphous or crystalline, opaque, dark red or brownish
melting point	44 °C	585 – 610 °C
vapour pressure	high	very low
density	1.83 g/cc	2.0 ... 2.4 g/cc
solubility: in organic solvents: in water:	 yes only ~3 mg / L	 no no (very slow hydrolysis)
toxicity	highly toxic	very low toxicity
toxicity LC_{50} 48 h, fish LD_{50}, oral, rat	highly toxic low µg/L levels 3 mg/kg body weight	very low toxicity > 100 mg/L (nominal loading) > 15 000 mg/kg body weight
heat of sublimation	13.4 k cal/mol	30.0 k cal/mol
chemiluminescence	yes	no
ignitability	- spontaneous ignition in air at room temperature - spontaneously ignites in chlorine	- ignites only above 260 °C - heat necessary for ignition in chlorine
smell	characteristic	no smell
reaction with aqueous alkali	produces phosphine	none

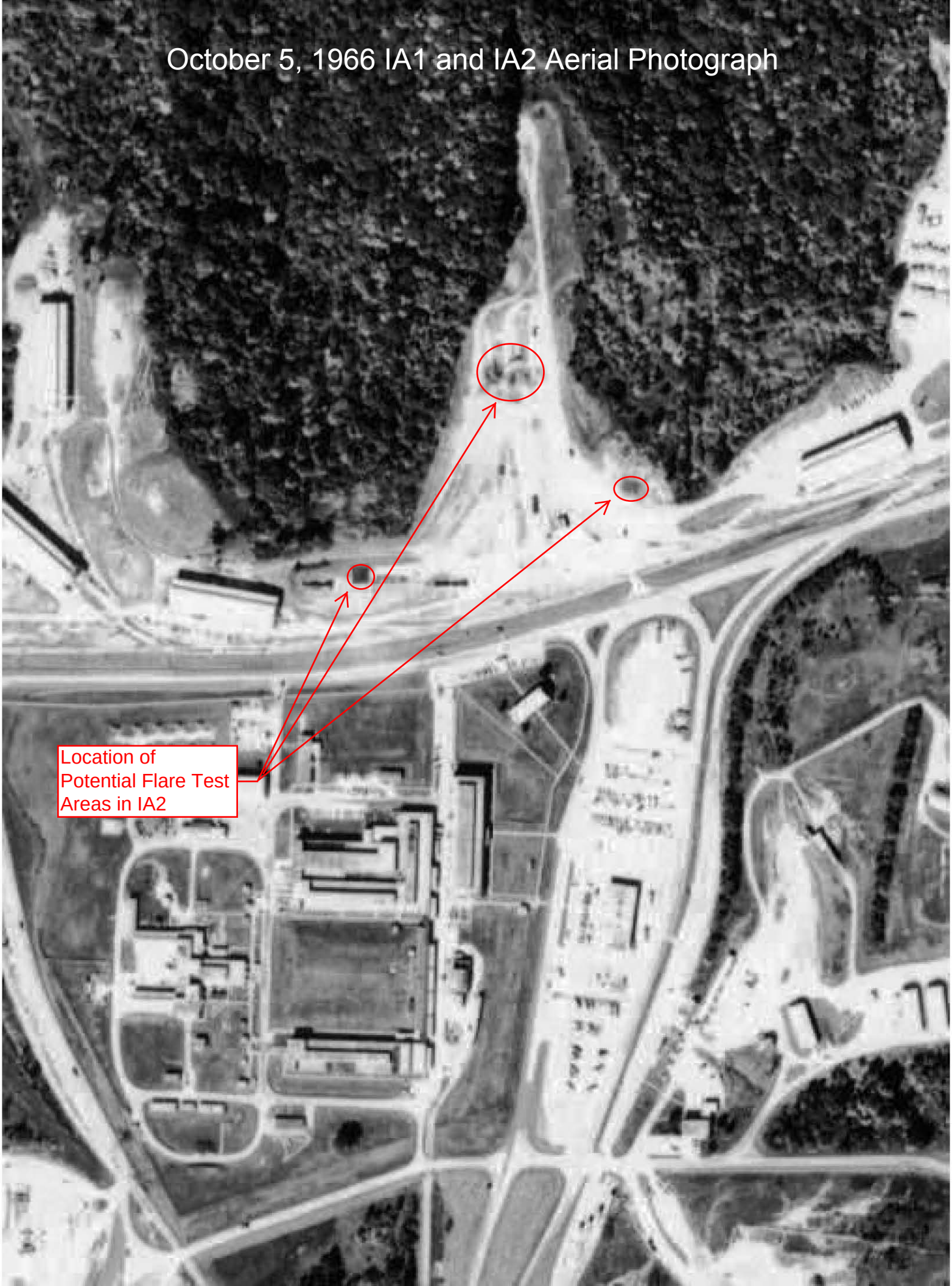
DISCLAIMER

This information was compiled with great care and scrutiny – it reflects the current knowledge about this product at the time of completion of this record. This fact sheet is meant to provide users of the product and all interested parties information on health, environmental and regulatory issues. However, this is no replacement for a safety data sheet or any other legally required document. Furthermore, these data do not represent a specification of any commercial product.

A.5

HISTORICAL AERIAL PHOTOGRAPHS

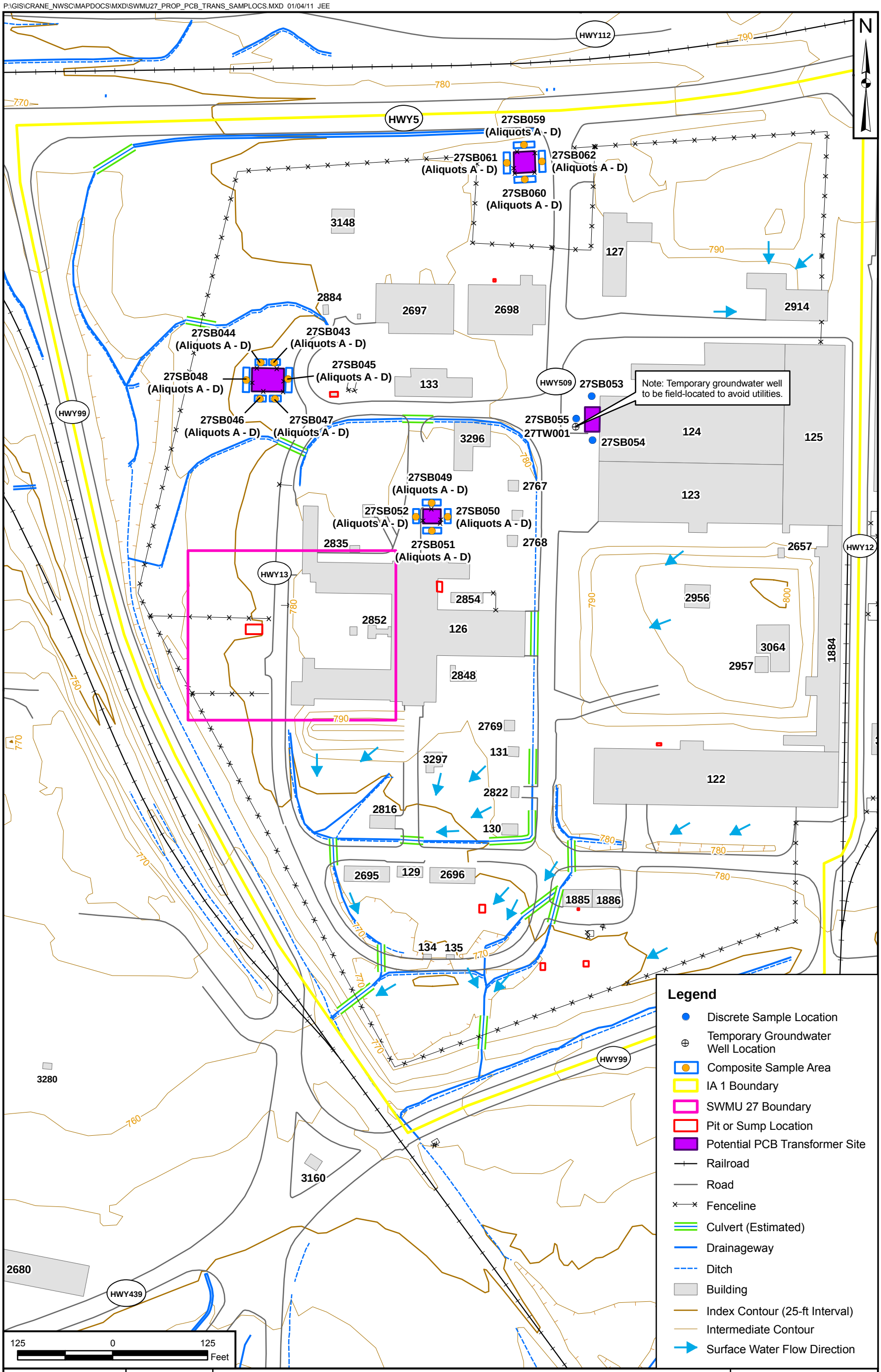
October 5, 1966 IA1 and IA2 Aerial Photograph



September 23, 1974 Aerial Photograph

March 24, 1998 SWMU 27 Aerial Photograph
Mar 23, 1998
317
315
SWMU 27
Naval Weapons Support Center
Image U.S. Geological Survey
©2009 Google
© 2009 Tele Atlas
Imagery Date: Mar 24, 1998
38°52'07.65" N 86°49'19.26" W elev 244 m
Eye alt 1.88 km

P:\GIS\CRANE_NWSC\MAPDOCS\MXD\SWMU27_PROP_PCB_TRANS_SAMPLOCS.MXD 01/04/11 JEE



DRAWN BY	DATE
J. ENGLISH	01/03/11
CHECKED BY	DATE
V. PLACHY	01/04/11
REVISED BY	DATE
—	—
SCALE	
AS NOTED	

NAVFAC

PCB TRANSFORMER SURFACE SOIL
AND GROUNDWATER SAMPLE LOCATIONS
SWMU 27 - ILLUMINANT BUILDING 126
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER	
CTO F276	
APPROVED BY	DATE
—	—
APPROVED BY	DATE
—	—
FIGURE NO.	REV
FIGURE 17-6	0

APPENDIX B

FIELD STANDARD OPERATING PROCEDURES

SOP-01	Global Positioning System
SOP-02	Sample Labeling
SOP-03	Sample Identification Nomenclature
SOP-04	Sample Custody And Documentation Of Field Activities
SOP-05	Sample Preservation, Packaging, And Shipping
SOP-06	Decontamination Of Field Sampling Equipment
SOP-07	Soil Coring And Sampling Using Hand Auger Techniques
SOP-08	Soil Sample Logging
SOP-09	Management Of Investigation-Derived Waste
SOP-10	Calibration And Use Of Photoionization Detector
SOP-11	Subsurface Soil And Groundwater Sampling Using Direct-Push Technology
SOP-12	Monitoring Well Installation
SOP-13	Monitoring Well Development
SOP-14	Measurement Of Water Levels
SOP-15	Low-Flow Well Purging And Stabilization
SOP-16	Monitoring Well Sampling
SOP-17	Calibration And Care Of Water Quality Meters

STANDARD OPERATING PROCEDURE

SOP-01

GLOBAL POSITIONING SYSTEM DATA COLLECTION AND TRANSFER

1.0 PURPOSE

The primary purpose of this Standard Operating Procedure (SOP) is to provide the Field Technicians with basic instructions for operating a handheld Global Positioning System (GPS) unit allowing them to set GPS parameters in the receiver, record GPS positions on the field device, and transfer the data for integration into existing Geographic Information System (GIS) figures.

This SOP is specific to GIS quality data collection for Trimble-specific hardware and software.

If possible, the Trimble GeoXT or XH Operators Manual should be downloaded onto the operator's personal computer for reference before or while in the field. The manual can be downloaded at the following website:
http://trl.trimble.com/docushare/dsweb/Get/Document-311749/TerraSyncReferenceManual.pdf

Unless the operator is proficient in the setup and operation of the GPS unit, the Project Manager (or designee) should have the GPS unit shipped to the project-specific contact listed below in the Pittsburgh, Pennsylvania office at least five working days prior to field mobilization so project-specific data files (i.e. shape files), background images, data dictionaries, and correct coordinate systems can be uploaded into the unit.

Tetra Tech NUS
Attn: Rick Barringer
661 Anderson Drive, Bldg #7
Pittsburgh, PA 15220

The SOP also describes how field collected data is to be transferred through the use of the MRP Website. (http://www.ttnus.com/MRPRepository/). This website serves as a centralized portal to facilitate data exchange for field personnel, GIS staff, and project managers. The website contains a "Reference" page that will contain the latest version of this SOP and other valuable documentation.

For technical questions regarding operation of the GPS units and data collection, please contact John Wright (john.wright@tetratech.com). For general questions about this SOP and use of the MRP website, please contact Mark Maguire (mark.maguire@tetratech.com).

2.0 REQUIRED EQUIPMENT

The following hardware and software should be utilized for locating and establishing GPS points in the field:

2.1 GPS Hardware and Equipment

- Hand-held GPS Unit capable of sub-meter accuracy. This includes the docking cradle, a/c adapter, stylus, and USB cable for data transfer. Two models, the GeoXH and GeoXT, are acceptable for use. The XH yields higher accuracy (in both real-time and post-processed) and **should always be requested** when highly precise data is required.

- An external antenna will yield better satellite reception, especially in heavy tree canopy. Associated accessories include a range pole and hardware clamp, for mounting the GPS unit to the pole.

- Indelible marker.

- Non-metallic pin flags for temporary marking of positions.

2.2 GPS Software

The following software is required to transfer data from the handheld GPS unit to a personal computer:

- Trimble TerraSync version 2.6 or later (pre-loaded onto GPS unit from vendor)

- Microsoft ActiveSync version 4.5 or later. Download to personal computer from:
  http://www.microsoft.com/windowsmobile/en-us/downloads/microsoft/activesync-download.mspx

  Note: Windows Vista and Windows 7 users should download Windows Mobile Device Center version 6.1 or later from the following site, if it is not already loaded on the machine:
  http://www.microsoft.com/windowsmobile/en-us/downloads/microsoft/device-center-download.mspx

- Trimble Data Transfer Utility (freeware version 2.1 or later). Download to personal computer from:

http://www.trimble.com/datatransfer.shtml

3.0 START-UP PROCEDURES

Prior to utilizing the GPS in the field, ensure the unit is fully charged. The unit may come charged from the vendor, but an overnight charge is recommended prior to fieldwork.

The Geo-series GPS units require a docking cradle for both charging and data transfer. The Geo-series GPS unit is docked in the cradle by first inserting the far domed end in the top of the cradled, then gently seating the contact end into the latch. The power charger is then connected to the cradle at the back end using the twist-lock connector. Attach a USB cable as needed between the cradle (B end) and the laptop/PC (A end).

It is recommended that the user also be familiar and check various Windows Mobile settings. One critical setting is the Power Options. The backlight should be set as needed to conserve power when not in use.

3.1 Initial Start Up

1) Power on the GPS unit by pushing the small green button located on the lower right front of the unit.

2) Utilizing the stylus that came with the GPS unit, launch **TerraSync** from the Windows Operating System by tapping on the start icon located in the upper left hand corner of the screen and then tap on **TerraSync** from the drop-down list.

3) If the unit does not default to the Setup screen, tap the Main Menu (uppermost left tab, just below the Windows icon) and select Setup.

4) If the unit was previously shipped to the Pittsburgh office for setup, you can skip directly to Section 4.0. However, to confirm or change settings, continue on to Section 3.1.

3.2 Confirm Setup Settings

Use the Setup section to confirm the TerraSync software settings. To open the Setup section, tap the Main Menu and select Setup. (Note that if the unit was shipped from the Pittsburgh office, these settings should have been set for your specific project. Feel free to contact Pittsburgh staff with any questions.)

1) Tap on the Coordinate System.
2) Verify the project specs are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu. Note: It is always best to utilize the Cancel tab rather than the OK tab if no changes are made since configurations are easily changed by mistake.
3) Tap on the Units.
4) Verify the user preferences are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
5) Tap Real-time Settings.
6) Verify the Real-time Settings are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
7) The GPS unit is now configured correctly for your specific project.

3.3 Antenna Connection

1) If a connection has been properly made with the internal antenna, a satellite icon along with the number of usable satellites will appear at the top of the screen next to the battery icon. If no connection is made (e.g.: no satellite icon), tap on the GPS tab to connect antenna.
2) At this point, the GPS unit is ready to begin collecting data.

3.4 Loading a Backup File

This section provides instructions on pulling in a pre-loaded background file. These files are helpful in visualizing your current location.

From the Main Menu select Map, then tap on Layers, select the background file from drop down list.

Select the project-specific background file from the list of available files.

Once the selected background file appears, the operator can manipulate the screen utilizing the +/- and <-/-> functions at the bottom of the screen.

In operating mode, the operator's location will show up on the background file as a floating "x."

4.0 FILED DATA COLLECTION

For MRP data collection activities, a new GPS file should be created **every day** and transferred **nightly** using the MRP website (see Section 9.0). This is to insure the timely transfer of data, file organization in the database, and allow for next-day GIS mapping. Also, individual GPS data files should be **unique to a**

particular site or unit (typically a UXO number). If multiple sites are visited in a single data, multiple files should be created.

4.1 Creating a Data File

1) From the Main Menu select Data.
2) From the Sub Menu (located below the Data tab) select New which will bring up the New Data File menu.
3) An auto-generated filename appears and should be edited for your specific project. The following naming convention should be followed as closely as possible: **IH-UXO4-01012010-TeamA**, where "IH" is the installation abbreviation (Indian Head), "UXO04" is the site, and "01012010" is the data in MMDDYYYY format. If multiple teams are being deployed across an individual site on the same day, it is important to specify the field team name at the end of the file name ("TeamA"). If the integral keyboard does not appear, tap the small keyboard icon at the bottom of the screen.
4) Select the data dictionary that will be used to collect features. The data dictionary provides predefined fields and drop-down menus to facilitate data collection as it relates to specific MRP data types. The MRP data dictionary is entitled "**MRP Data Collection**" and should appear in the data dictionary drop-down list. This should have been pre-loaded into the GPS prior to use. The data dictionary file is available on the MRP website under the "Reference" section.
5) After entering the file name and selecting the data dictionary, tap Create to create the new file.
6) Confirm antenna height if screen appears. Antenna height is the height that the GPS unit will be held from the ground surface (Typically 3 to 4 feet)
7) The Choose Feature screen appears.

4.2 Collecting Features

1) If not already open, the Collect Feature screen can be opened by tapping the Main Menu and selecting Data. The Sub Menu should default to Collect.
2) **Do not begin the data logging process until you are at the specific location for which you intend to log the data.**
3) A known reference or two should be shot at the beginning and at the end of each day in which the GPS unit is being used. This allows for greater accuracy during post-processing of the data.
4) Upon arriving at the specific location, select the proper feature type from the data dictionary list (MEP Object, Transect End Point, GPS QC Point, or General Point).
5) Tap Create to begin data logging.

6) As the GPS is collecting positions, enter the feature attributes, starting with the Item ID. This field is required and will not allow the user to continue or save the position without entering a value. Enter any additional notes or feature descriptions in the appropriate fields.
7) Data logging can be confirmed by viewing the writing pencil icon in the upper part of the screen. Also, the logging counter will begin. As a Rule of Thumb, accumulate a minimum of 20 readings on the counter, per point, as indicated by the logging counter before saving the GPS data.
8) Once the counter has reached a minimum number of counts (i.e. 20), tap on OK to save the data point to the GPS unit. Confirm the feature. All data points are automatically saved within the GPS unit.
9) Repeat steps 2 through 8, giving each data point a unique name or number.

Note: If the small satellite icon or the pencil icon is blinking, this is an indication the GPS unit is not collecting data. A possible problem may be too few satellites. While still in data collection mode, tap on Main Menu in upper left hand corner of the screen and select Status. Skyplot will display as the default showing the number of available satellites. To increase productivity (number of usable satellites) use the stylus to move the pointer on the productivity and precision line to the left. This will decrease precision, but increase productivity. The precision and productivity of the GPS unit can be adjusted as the number of usable satellites changes throughout the day. To determine if GPS is correctly recording data, see Section 5.2. If the precision toggle is decreased, the user should frequently check the Skyplot display to restore the default values as soon as possible.

4.3 Navigation

This section provides instructions on navigating to saved data points in an existing file within the GPS unit.

1) From the Main Menu select Map.
2) Using the Select tool, pick the point on the map to where you want to navigate.
3) The location you select will have a box placed around the point.
4) From the Options menu, choose the Set Nav Target (aka set navigation target).
5) The location will now have double blue flags indicating this point is you navigation target.
6) From the Main Menu select Navigation.
7) The dial and data on this page will indicate what distance and direction you need to travel to reach the desired target.
8) Follow the navigation guide until you reach the point you select.

9) Repeat as needed for any map point by going back to Step 1.

4.4 Data Quality Control

Quality control checks should be performed each day of data collection and/or data navigation. QC checks are important both to understand real-time accuracy while in the field, and also to provide control data needed during post-processing.

1) Known survey benchmarks, surveyed monitoring wells, or other established and documented control points should be identified
2) GPS equipment should be placed on known control points and positions recorded
3) For data collection tasks - QC check data should be collected at least at the start and completion of the fieldwork for the day of data collection. Additional occupation and collection of control point data should occur as possible during the work day, and should increase in frequency as the number of data points increase and the need for accurate data collection increases
4) For navigation tasks such as stake placement for planned sample locations, QC data checks should be done at least at the start and completion of the fieldwork for each day. Known visible targets should be occupied and observed by the user, while the GPS satellite status and other user interface data is reviewed. The user should assess whether the real-time accuracy settings on the GPS are within the tolerance of the observed visual reference points.

4.5 Viewing Data or Entering Additional Data Points to the Current File

1) To view the stored data points in the current file, tap on the Main Menu and select Map. Stored data points for that particular file will appear. Use the +/- and <-/-> icons in lower left hand corner of screen to zoom in/out and to manipulate current view.
2) To return to data collection, tap on the Main Menu and select Data. You are now ready to continue to collect additional data points.

4.6 Viewing Data or Entering Data Points from an Existing File

1) To view data points from a previous file, tap on Main Menu and select Data, then select File Manager from the Sub Menu.
2) Highlight the file you want to view and select Map from the Main Menu.
3) To add data points to this file, tap on Main Menu and select Data. Continue to collect additional data points.

4.7 Shutting Down

This section provides instruction for properly shutting down the GPS unit.

1) When shutting down the GPS unit for the day, first click on the “X” in the upper right hand corner.
2) You will be prompted to ensure you want to exit TerraSync. Select Yes.
3) Power off the GPS unit by pushing the small green button located on the bottom face of the unit.
4) Place the GPS unit in its cradle to recharge the battery overnight. Ensure the green charge light is visible on the charging cradle.

5.0 DATA TRANSFER

This section describes how data should be downloaded from the GPS units and uploaded to a central website for post-processing and integration into GIS datasets. GPS data collected on a given day should be transferred **that night** for post-processing by GIS staff the next morning. Once post-processed, the GPS data will be plotted on a map and be immediately provided to the project team for review. Data upload, download, and review will be facilitated through a secure MRP website: http://www.ttnus.com/MRPRepository/

5.1 Load Data from the GPS Unit to Your Computer

1) Install the Data Transfer and ActiveSync software installed on your PC (see section 2.2)
2) Connect the GeoXH/XT to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
3) ActiveSync should auto-detect the connection and recognize the data collector
4) Make sure the data file desired is CLOSED in TerraSync prior to transfer
5) Connect via ActiveSync as a guest (not a partnership)
6) Run the Trimble Data Transfer Utility program on your PC
7) Select "GIS Datalogger on Windows CE" or similar selection
8) Hit the green connect icon to the right - the far right area should say "Connected to ...." if successful
9) Select the "Receive" data tab (under device)
10) Select "Data" from file types on the right
11) Find the file(s) needed for data transfer. You can sort the data files by clicking on the date/time header

12) Select or browse to a C-drive folder you can put this file for upload

13) When the file appears on the list, hit the "Transfer All". Once complete, a packet of multiple data files will appear on your computer in the specified folder.

5.2 Gain Access to MRP Website

1) Confirm that your computer has internet access
2) Click on the following link: http://www.ttnus.com/MRPRepository/
3) To register for the website, click on the "Register here" link. Enter your information and click "Submit." NOTE: Requests for registration are sent to Ralph Basinski, Program Manager, for approval. Please contact mark.maguire@tetratech.com if you experience any access issues.
4) Enter your username (Tetra Tech email address) and password to log in.

5.3 Upload GPS Data from Your Computer to the MRP Website

1) From the main page, select "Upload" from the menu at left.
2) Select the type of data you are uploading, typically "GPS Field Data"
3) Select the appropriate Installation and Site. Remember that GPS files should be unique for each site, even if multiple sites are visited in one day. If collected data is not associated with a site, select "Other."
4) Select "browse" to navigate to the appropriate *.SSF file on your computer. When you use the Trimble download utility to grab data from the GPS unit, multiple files will appear on your computer. You only need to the upload the *.SSF file.
5) Populate the "Comments" field to describe the dataset and any other pertinent information. This information will be provided to the GIS analyst who will be integrating the dataset, so be sure to be as descriptive as possible especially if there are any issues with the data. (For example, if you were to sample 16 points and for some reason you believe only 15 were logged, it is helpful to share this information.)
6) Select "Upload." Users will be notified if the files were uploaded successfully.

5.4 Download Data from the MRP Website to Your Computer

The download utility on the MRP website will serve different user types. **Field staff** will use the utility to download GIS figures (in PDF format) and view the previous day(s) field data on aerial photographs,

checking for any discrepancies or missing data elements. **Project Managers** will also have the ability to download and view these figures, to visualize the data and track project progress. This utility will also allow **GIS Analysts** to download the *.SSF files posted by field staff for post-processing and map plotting.

To download GIS Figures:

1) From the main page, select “Download” from the menu at left.
2) Select an Installation and Site
3) Users can view Figures for a particular date or by a range of dates, by selecting the appropriate options. <u>To search all dates, leave all of these fields as the default.</u>
4) Select “Search”
5) A table will appear showing the files available for download. Simply click on the link to the file and you will be prompted to save it to your computer.

STANDARD OPERATING PROCEDURE

SOP-02

SAMPLE LABELING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used for labeling sample containers. Sample labels are used to document the sample ID, date, time, analysis to be performed, preservative, matrix, sampler, and the analytical laboratory. A sample label will be attached to each sample container.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)
Disposable medical-grade gloves (e.g. latex, nitrile)
Sample log sheets
Required sample containers: All sample containers for analysis by fix-based laboratories will be supplied and deemed certified clean by the laboratory.
Sample labels
Chain-of-custody records
Sealable polyethylene bags
Heavy-duty cooler
Ice

3.0 PROCEDURES

3.1 The following information will be electronically printed on each sample label prior to mobilizing for field activities. Additional “generic” labels will also be printed prior to mobilization to be used for field QC and backups.

- Project number (CTO F276)
- Sample location ID
- Contract Task Order number
- Sample ID
- Matrix

- Preservative
- Analysis to be performed
- Laboratory name

3.2 Select the container(s) that are appropriate for a given sample. Select the sample-specific ID label(s), complete date, time, and sampler name, and affix to the sample container(s).

3.3 Fill the appropriate containers with sample material. Securely close the container lids without overtightening.

3.4 Place the sample container in a sealable polyethylene bag and place in a cooler containing ice.

Example of a sample label is attached at the end of this SOP.

4.0 ATTACHMENTS

1. Sample Label

ATTACHMENT 1

SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** **Location:** **CTO:**	
Sample No:		**Matrix:**
Date:	**Time:**	**Preserve:**
Analysis:		
Sampled by:	**Laboratory**	

STANDARD OPERATING PROCEDURE

SOP-03

SAMPLE IDENTIFICATION NOMENCLATURE

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a consistent sample nomenclature system that will facilitate subsequent data management at the Naval Support Activity (NSA) Crane. The sample nomenclature system has been devised such that the following objectives can be attained.

- Sorting of data by site, location, or matrix
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints
- Ease of sample identification

The NSA Crane Environmental Protection Department must approve any deviations from this procedure.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Pen with indelible ink
Sample tags
Sample container labels

3.0 SAMPLE IDENTIFICATION NOMENCLATURE

3.1 <u>Confirmation Samples</u>

All confirmation samples will be properly labeled with a sample label affixed to the sample container. Each sample will be assigned a unique sample tracking number.

3.1.1 Confirmation Sample numbering Scheme

The sample tracking number will consist of a four- or five-segment alpha-numeric code that identifies the sample's associated Solid Waste Management Unit (SWMU), sample type, location, and sample depth. For soil samples, the final four tracking numbers will identify the depth in units of feet below ground surface (bgs) at which the sample was collected (rounded to the nearest foot). For sediment samples, the final four tracking numbers will identify the depth in units of inches bgs at which the sample was collected.

The alphanumeric coding to be used is explained in the following diagram and subsequent definitions:

AN	**AA**	**NNNA**	**NNNN (Soils and Sediment only)**
SWMU Number	Matrix	Sample Location Number and Sample Type	Sequential depth interval from freshly exposed surface

Character Type:

A = Alpha

N = Numeric

SWMU Number (AN):

= 27

Matrix Code (AA):

SS = Surface Soil Sample

SB = Subsurface Soil Sample

GW = Groundwater Sample

Location Number (NNNA):

Sequential number beginning with "001" for each matrix, followed by a letter indicating the sample type [grab (G) or composite (C) sample, if appropriate].

Depth Interval (NNNN):

This code section will be used for soil samples only. For soil samples, the final four tracking numbers will identify the depth in units of feet. Surface soil samples will be collected from 0- to 2-feet bgs. Subsurface soil samples will be collected at depths greater than 2-feet bgs.

The depth code is used to note the depth bgs at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval of the sample depth. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc. (If composite samples are collected: "location" refers to a particular sampling grid represented by a composite sample.

3.1.2 Examples of Confirmation Sample Nomenclature

A surface soil grab sample collected from SWMU 27, sampling location 001, at a depth of 0- to 2-foot bgs would be labeled as "27SS001G0002."

A surface soil composite sample collected from SWMU 27, sampling location 002, would be labeled as "27SS002C0002."

3.3 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

Field QA/QC samples are described in the UFP-SAP. They will be designated using a different coding system than the one used for regular field samples.

3.3.1 QC Sample Numbering

The QC code will consist of a four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AN	AA	NNNNNN	NN
SWMU Number	QC Type	Date	Sequence Number (per day)

The QC types are identified as:

RB = Rinsate Blank
FD = Field Duplicate

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be "0000" so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

3.3.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day at SWMU 27 for a surface soil sample collected on March 25, 2011 would be designated as “27FD03251101.”

The third duplicate of the day taken at SWMU 27 of a surface soil sample collected on March 3, 2011 would be designated as “27FD03031103.”

The first rinsate blank associated with samples collected on March 3, 2011 would be designated as “27RB03031101.”

STANDARD OPERATING PROCEDURE
SOP-04

SAMPLE CUSTODY AND DOCUMENTATION OF FIELD ACTIVITIES

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for sample custody and documentation of field sampling and field analyses activities.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following logbooks, forms, labels, and equipment are required.

Writing utensil (preferably black pen with indelible ink)
Site logbook
Field logbook
Sample label
Chain-of-Custody Form
Custody seals
Equipment calibration log
Soil and Sediment Sample Log Sheet

3.0 PROCEDURES

This section describes custody and documentation procedures. All entries made into the logbooks, custody documents, logs, and log sheets described in this SOP must be made in indelible ink (black is preferred). No erasures are permitted. If an incorrect entry is made, the entry will be crossed out with a single strike mark, initialed, and dated.

3.1 <u>Site Logbook</u>

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, the following activities and events will be recorded (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Start or completion of sampling activities
- Daily on-site activities performed each day
- Sample pickup information
- Health and safety issues
- Weather conditions

The site logbook is initiated at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day that on-site activities take place.

The following information must be recorded on the cover of each site logbook:

- Project name
- Project number
- Book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). At the completion of each day's entries, the site logbook must be signed and dated by the field operations leader (FOL).

3.2 Field Logbooks

The field logbook is a separate dedicated notebook used by field personnel to document his or her activities in the field. This notebook is hardbound and paginated.

3.3 Sample Labels

Adhesive sample container labels must be completed and applied to every sample container. Information on the label includes the project name, location, sample number, date, time,

preservative, analysis, matrix, sampler's initials, and the name of the laboratory performing the analysis.

3.4 Chain-of-Custody Form

The Chain-of-Custody Form (COC) is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as it is transferred from person to person. Each COC is numbered. This form must accompany any samples collected for laboratory chemical analysis. A copy of a blank COC form is attached at the end of this SOP.

The FOL must include the name of the laboratory in the upper right hand corner section to ensure that the samples are forwarded to the correct location. If more than one COC is necessary for any cooler, the FOL will indicate "Page __ of __" on each COC. The original (top) signed copy of the COC will be placed inside a sealable polyethylene bag and taped inside the lid of the shipping cooler. Once the samples are received at the laboratory, the sample custodian checks the contents of the cooler(s) against the enclosed COC(s). Any problems are noted on the enclosed COC Form (bottle breakage, discrepancies between the sample labels, COC form, etc.) and will be resolved through communication between the laboratory point-of-contact and the Project Manager (PM). The COC form is signed and retained by the laboratory and becomes part of the sample's corresponding analytical data package.

3.5 Custody Seal

The custody seal is an adhesive-backed label, and it is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transit to the laboratory. The custody seals are signed and dated by the samplers and affixed across the opening edges of each cooler (two seals per cooler) containing environmental samples. The laboratory sample custodian will examine the custody seal for evidence of tampering and will notify the TtNUS PM if evidence of tampering is observed.

3.6 Equipment Calibration Log

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device requiring calibration. Entries must be made for each day the equipment is used.

3.7 Sample Log Sheets

The Soil and Sediment Sample Log Sheets are used to document the sampling of soil and sediment (see SOPs-09, -10, -11, and -12).

4.0 ATTACHMENTS

1. Chain-of-Custody Record
2. Equipment Calibration Log
3. Soil and Sediment Sample Log

ATTACHMENT 1

CHAIN-OF-CUSTODY RECORD

CHAIN OF CUSTODY | NUMBER | PAGE ____ OF _____

PROJECT NO:	SITE NAME:	PROJECT MANAGER AND PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER AND PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER	CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

DATE YEAR ____	TIME	SAMPLE ID	MATRIX	GRAB (G) COMP (C)	No. OF CONTAINERS	CONTAINER TYPE PLASTIC (P) or GLASS (G) / PRESERVATIVE USED / TYPE OF ANALYSIS								COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

3/99
FORM NO. TtNUS-001

ATTACHMENT 2

EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

ATTACHMENT 3

SOIL AND SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE

SOP-05

SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for sample preservation, packaging, and shipping to be used in handling soil and aqueous samples.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Shipping labels
Custody seals
Chain-of-custody (COC) form(s)
Sample containers with preservatives: All sample containers for analysis by fixed-base laboratories will be supplied, with preservatives added (if required) and deemed certified clean by the laboratory.
Sample shipping containers (coolers): All sample shipping containers are supplied by the laboratory.
Packaging material: Bubble wrap, sealable polyethylene bags, strapping tape, etc.

3.0 PROCEDURES FOR SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

3.1 The laboratory provides sample containers with preservative already included (as required) for the analytical parameter for which the sample is to be analyzed. All samples will be held, stored, and shipped at 4°C. This will be accomplished through refrigeration (used to hold samples prior to shipment) and/or ice.

3.2 The sampler shall maintain custody of the samples until the samples are relinquished to another custodian or to the common carrier.

3.3 Check that each sample container is properly labeled, the container lid is securely fastened, and the container is sealed in a polyethylene bag.

3.4 If the container is glass, place the sample container into a bubble-out shipping bag and seal the bag using the self-sealing, pressure sensitive tape supplied with the bag.

3.5 Inspect the insulated shipping cooler. Check for any cracks, holes, broken handles, etc. If the cooler has a drain plug, make certain it is sealed shut, both inside and outside of the cooler. If the cooler is questionable for shipping, the cooler must be discarded.

3.6 Line the cooler with large plastic bag, and line the bottom of the cooler with a layer of bubble wrap. Place the sample containers into the shipping cooler in an upright position (containers will be upright, with the exception of any 40-ml vials). Continue filling the cooler with ice until the cooler is nearly full and the movement of the sample containers is limited.

3.7 Wrap the large plastic bag closed and secure with tape.

3.8 Place the original (top) signed copy of the COC form inside a sealable polyethylene bag. Tape the bag to the inside of the lid of the shipping cooler.

3.9 Close the cooler and seal the cooler with approximately four wraps of strapping tape at each end of the cooler. Prior to wrapping the last wrap of strapping tape, apply a signed and dated custody seal to each side of the cooler (one per side). Cover the custody seal with the last wrap of tape. This will provide a tamper evident custody seal system for the sample shipment.

3.10 Affix shipping labels to each of the coolers, ensuring all of the shipping information is filled in properly. Overnight (e.g., FedEx Priority Overnight) courier services will be used for all sample shipments.

3.11 All samples will be shipped to the laboratory no more than 72 hours after collection. Under no circumstances should sample hold times be exceeded.

STANDARD OPERATING PROCEDURE

SOP-06

DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures to be followed when decontaminating non-dedicated field sampling equipment during the field investigations.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)
Non-latex rubber or plastic gloves
Cotton gloves
Field logbook
Potable water
Deionized water
Isoproponal (optional)
LiquiNox detergent
Brushes, spray bottles, paper towels, etc.
Container to collect and transport decontamination fluids

3.0 DECONTAMINATION PROCEDURES

3.1 Don non-latex and/or cotton gloves and decontaminate sampling equipment (in accordance with the following steps) prior to field sampling and between samples.

3.2 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.3 Wash the equipment with a solution of LiquiNox detergent. Prepare the LiquiNox wash solution in accordance with the instructions on the LiquiNox container. Collect the LiquiNox wash solution into a container. Use brushes or sprays as appropriate for the equipment. If oily residue has accumulated on the sampling equipment, remove the residue with an isopropanol wash and repeat the LiquiNox wash.

3.4 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.5 Rinse the equipment with deionized water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the deionized water rinsate into a container.

3.6 Remove excess water by air drying, shaking, or by wiping with paper towels as necessary.

3.7 Document decontamination by recording it in the field logbook.

3.8 Containerized decontamination solutions will be managed in accordance with the procedures described in SOP-13 and this UFP SAP.

STANDARD OPERATING PROCEDURE

SOP-07

SOIL CORING AND SAMPLING USING HAND AUGER TECHNIQUES

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for collecting surface and subsurface soil cores from unconsolidated overburden materials using hand augering techniques.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Disposable medical-grade gloves (e.g., latex, nitrile)
Writing utensil (preferably black pen with indelible ink)
Indelible marker
Stainless Steel Auger Buckets
Stainless Steel Extension Rods
Cross Handle
Required decontamination materials
Bentonite pellets
Sealable polyethylene bags
Sample labels
Shipping containers (containing ice)
Disposable plastic trowels or stainless steel trowels
Stainless steel mixing bowls
Sample containers: Sample containers are certified clean by the laboratory supplying the containers.
Soil Sample Log Forms
Daily Activity Logs
Chain-of-Custody Form

3.0 SOIL SAMPLING USING A HAND AUGER

Hand Augers may be employed to collect the soil cores. A hand augering system generally consists of a variety of all stainless steel bucket bits (i.e. cylinders 6-1/2” long and 2-3/4”, 3-1/4”, and 4” in diameter), a series of extension rods (available in 2’, 3’, 4’ and 5’ lengths), a cross handle.

3.1 The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil, both from the surface, or to depths in excess of 12 feet. However, the presence or rock layers and the collapse of the borehole normally contribute to its limiting factors.

Attach a properly decontaminated bucket bit into a clean extension rod and further attach the cross handle to the extension rod.

3.2 Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, letter, etc.)

3.3. Turn the hand auger sampler into the ground to a depth of 6-inches. The 0- to 6-inch depth soil interval is considered to be the surface soil.

3.4 After reaching the desired depth, slowly and carefully withdraw the apparatus from the borehole.

3.4 Utilizing a properly decontaminated stainless steel trowel or disposable trowel, remove the sample material from the bucket bit and place into a sealable polyethylene bag. Note in a field notebook or on a standardized data sheet any changes in the color, texture or odor of the soil.

3.5 Thoroughly homogenize the sample material and write sample ID, date, and time on the bag with an indelible marker.

3.6 Complete required information on the Soil Sample Log Sheet (copy attached at the end of this SOP). Update the Chain-of-Custody (COC) Form.

3.7 Excess soil core materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole, and hydrated with potable water.

3.8 Decontaminate all soil sampling equipment in accordance with SOP-08 before collecting the next sample.

3.9 Soil samples shipped to a fixed-base laboratory for analysis will be in sample containers supplied by the laboratory. The sample labels will be completed and affixed to the sample container. The samples will then be packaged and shipped to the fixed-base laboratory in accordance with SOP-07.

4.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

ATTACHMENT 1

SOIL AND SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE

SOP-08

SOIL SAMPLE LOGGING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the standard procedures and technical guidance on the logging of soil samples.

2.0 FIELD FORMS AND EQUIPMENT

Knife
Ruler (marked in tenths and hundredths of feet)
Boring Log: An example of this form is attached.
Writing utensil (preferably black pen with indelible ink)

3.0 RESPONSIBILITIES

A field geologist or engineer is responsible for supervising all activities and assuring that each soil sample is properly and completely logged.

4.0 PROCEDURES FOR SAMPLE LOGGING

To maintain a consistent classification of soil, it is imperative that the field geologist understands and accurately uses the field classification system described in this SOP. This identification is based on visual examination and manual tests.

4.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (attached to this SOP).

This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no distinguishable size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils will be divided into categories: rock fragments, sand, or gravel. The terms "sand" and "gravel" not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term "rock fragments" will be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges that are typically observed indicate little or no transport from their source area; and therefore, the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used, it will be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

4.2 Color

Soil colors will be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Because color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples will be broken or split vertically to describe colors. Samplers tend to smear the sample surface, creating color variations between the sample interior and exterior.

The term "mottled" will be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

4.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are non-cohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in the following table.

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist.
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb.
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort.
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort.
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail.
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined by hand by determining the resistance to penetration by the thumb. The thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample. The sample will be broken in half and the thumb pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. One of the other methods will be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in the above-listed table.

4.4 Weight Percentages

In nature, soils are consist of particles of varying size and shape and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., sandy)	31 - 50 percent

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

4.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the gloved hand or on a porous surface liberates water (i.e., dirties or muddies the surface). Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire field activity.

4.6 Classification of Soil Grain Size for Chemical Analysis

To determine the gross grain size classification (e.g., clay, silt, and sand) from the USCS classification described above, the following table will be used.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
Clay	CL	inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	CH	inorganic clays of high plasticity, fat clays.
	OH	organic clays of medium to high plasticity, organic silts.
Silt	ML	inorganic silts and very fine sands, rock four, silty or clayey fine sands with slight plasticity.
	OL	organic silts and organic silty clays of low plasticity.
	MH	inorganic silts, micaceous or diatomaceous fine sand or silty soils.
Sand	SW	well graded sands, gravelly sands, little or no fines.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
	SP	poorly graded sands, gravelly sands, little or no fines.
	SM	silty sands, sand-silt mixtures.
	SC	clayey sands, sand-clay mixtures.

4.7 Summary of Soil Classification

In summary, soils will be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (optional)
- Soil types
- Moisture content
- Other distinguishing features
- Grain size
- Depositional environment

5.0 ATTACHMENTS

1. Figure 1 - Unified Soil Classification System
2. Boring Log

ATTACHMENT 1

FIGURE 1 - UNIFIED SOIL CLASSIFICATION SYSTEM

Unified Soil Classification System

Coarse Grained Soils (more than half of soil > No. 200 sieve)	Gravels (More than half of coarse fraction > no. 4 sieve size)	GW	Well graded gravels or gravel-sand mixtures, little or no fines
		GP	Poorly graded gravels or gravel-sand mixtures, little or no fines
		GM	Sandy gravels, gravel-sand-silt mixtures
		GC	Clayey gravels, gravel-sand-silt mixtures
	Sands (More than half of coarse fraction < no. 4 sieve size)	SW	Well graded sands or gravelly sands, little or no fines
		SP	Poorly graded sands or gravelly sands, little or no fines
		SM	Silty sands, sand-silt mixtures
		SC	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands or clayey silts with slight plasticity
Fine Grained Soils (more than half of soil < No. 200 sieve)	Silts and Clays LL = < 50	ML	Inorganic silts and very fine sands, rock flour, silty fine sands or clayey silts with slight plasticity
		CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, lean clays
		OL	Organic silts and organic silty clays of low plasticity
	Silts and Clays LL = > 50	MH	Inorganic silts, micaceous or diatomaceous fine sand or silty soils, elastic silts
		CH	Inorganic silts of high plasticity, fat clays
		OH	Organic clays of high plasticity, organic silty clays, organic silts
Highly Organic Soils		Pt	Peat and other highly organic soils

Grain Size Chart

Classification	Range of Grain Sizes	
	U.S. Standard Sieve Size	Grain Size In Millimeters
Boulders	Above 12"	Above 305
Cobbles	12" to 3"	305 to 76.2
Gravel	3" to No. 4	76.2 to 7.76
coarse	3" to 3/4"	76.2 to 4.76
fine	3/4" to No. 4	19.1 to 4.76
Sand	No. 4 to No. 200	4.76 to 0.074
coarse	No. 4 to No. 10	4.76 to 2.00
medium	No. 10 to No. 40	2.00 to 0.420
fine	No. 40 to No. 200	0.420 to 0.074
Silt and Clay	Below No. 200	Below 0.074

Relative Density (SPT)

SANDS AND GRAVELS	BLOWS/FOOT
VERY LOOSE	0 – 4
LOOSE	4 – 10
MEDIUM DENSE	10 – 30
DENSE	32 – 50
VERY DENSE	OVER 50

Consistency (SPT)

SILTS AND CLAYS	BLOWS/FOOT
VERY SOFT	0 – 2
SOFT	2 – 4
MEDIUM STIFF	4 – 8
STIFF	8 – 16
VERY STIFF	16 – 22
HARD	OVER 32

ATTACHMENT 2

BORING LOG

Tetra Tech NUS, Inc.

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS +	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole*	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): ____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

STANDARD OPERATING PROCEDURE

SOP-09

MANAGEMENT OF INVESTIGATION-DERIVED WASTE

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes how investigation-derived waste (IDW) will be collected, segregated, classified, and managed during the field investigations at NSA Crane. The following types of IDW will be generated during this investigation:

- Soil sampling residues
- Monitoring well development and well purge waters
- Decontamination solutions
- Personal protective equipment and clothing (PPE)
- Miscellaneous trash and incidental items

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Health and safety equipment (with PPE)
Hand augers, plastic or stainless steel trowels
Bucket (with collected development/purge water)
Decontamination equipment
Field logbook
Writing utensil (preferably black pen with indelible ink)
Plastic sheeting and/or tarps
55-gallon drums with sealable lids
IDW labels for drums
Plastic garbage bags

3.0 PROCEDURES

Management of IDW includes the collection, segregation, temporary storage, classification, final disposal, and documentation of the waste-handling activities if necessary.

3.1 Liquid Wastes

Liquid wastes that will be generated during the site activities include decontamination solutions from sampling equipment. These wastes will be collected and containerized in a central location at NSA Crane for proper disposal.

3.2 Solid Wastes

Solid wastes that may be generated during site activities include soil and sediment sampling residues. Excess soil core/sampling materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole, and hydrated with potable water. Excess sediment sampling materials will be returned to the point of collection. The disposition of this materials will be carried out in a manner such as not to contribute further environmental degradation or pose a threat to public health or safety.

3.3 PPE and Incidental Trash

All PPE wastes and incidental trash materials (e.g., wrapping or packing materials from supply cartons, waste paper) will be decontaminated (if contaminated), double bagged, securely tied shut, and placed in a designated waste receptacle at NSA Crane.

STANDARD OPERATING PROCEDURE

SOP-10

CALIBRATION AND USE OF PHOTOIONIZATION DETECTOR

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes procedures for the maintenance, calibration, and use of a photoionization detector (PID). The Photovac 2020 Photoionization Air Monitor will be used at NSWC Crane. The procedures for its use are discussed in detail in the following sections.

2.0 GLOSSARY

Electron-volt (eV) - A unit of energy equal to the energy acquired by an electron when it passes through a potential difference of 1 volt in a vacuum.

Intrinsically Safe (I.S.) - Based on wiring, configuration, design, operation, gasketing, construction, this instrument may be employed within locations where flammable gases and/or vapors may exist.

Ionization Potential (I.P.) - The energy required to remove an electron from a molecule yielding a positively charged ion and a negatively charged free electron. The instrument measures this energy level.

Photoionization Detector (PID) - Photoionization detector is employed as the general reference to air monitors of this type. PID's detection method employs ultraviolet (UV) radiation as an energy source. As air and contaminants are drawn through the ionization chamber, the UV light source causes the contaminant with ionization potentials equal to or less than the UV source to break into positive and negatively charge ions. The created ions are subjected to an electrostatic field. The voltage difference is measured in proportion to the calibration reference and the concentration of the contaminant.

Ultraviolet Radiation (UV) Lamp - Ultraviolet radiation is the energy source employed by the instrument to ionize collected sample gas streams. The UV lamp source is required to be equal to or greater than the ionization potential of the substance drawn through the instrument in order to create separate ionized species.

3.0 REQUIRED EQUIPMENT

Pen

Equipment Calibration Form

Photoionization detector

Isobutylene calibration gas (i.e., span gas)

Regulator

4.0 PROCEDURES

4.1 Principle of Operation

The Photovac portable photoionizer detects many organic (and a few inorganic) species. The basis for detection of this instrument is the ionization of components in gaseous streams. The incoming gas molecules are subjected to ultraviolet (UV) radiation, which is energetic enough to ionize many compounds associated with industrial activities. Molecules are transformed into charged-ion pairs, creating a current between two electrodes. Each molecule has a characteristic ionization potential, which is the energy required to remove an electron from the molecule, yielding a positively charged ion and a free electron. The instrument measures this energy level.

This instrument measures the concentration of airborne photoionizable gases and vapors and automatically displays and records these concentrations. It does not distinguish between individual substances. Readings displayed represent the total concentration of all photoionizable chemicals present in the sample. This instrument is factory-set to display concentration in units of ppm or mg/m^3. The meter display updates itself once per second.

The 2020 also performs short-term exposure limit (STEL), time-weighted average (TWA), and PEAK calculations. Any of these results can be viewed, but only one mode may be viewed at a time.

The 2020 has six keys for alphanumeric entry and for accessing multiple functions. The keys are used to set up and calibrate 2020. They allow the user to manipulate the concentration data in various ways.

All information entered with the keys and stored in the 2020's memory is retained when the instrument is switched off. The clock and calendar continue to operate and do not need to be set each time the 2020 is turned on.

4.1.1 Displays

The 2020 has a meter display for reporting detected concentration and a display used for status/ information to guide you through configuration options. All functions of the 2020 will be controlled or reported using one of these displays.

4.1.1.1 Meter Display

The meter display is four digits. It will always be used for reporting detected concentration. When the detector and pump are off, the meter display will be blank.

In order to accommodate the range of concentrations the 2020 can detect, the meter reading will be reported using one of two resolutions. A resolution of 0.1 will be used for concentrations below 100 ppm, and a resolution of 1 will be used for concentrations above 100 ppm.

4.1.1.2 Status Display

The status display is a two- line by 16-character display. The top line is used to display status information and prompts the user for inputs. The bottom line is used for soft key names. Up to three names can be displayed for the three soft keys. If a name does not appear for a soft key, then the soft key has no associated function.

4.1.2 Keys

4.1.2.1 Fixed Keys

The three round keys below the soft keys each have a fixed function. The first key is the ON/OFF key, the middle key is the EXIT key, and the last key is the ENTER key.

The ON/OFF key is used to both turn power on and off. To turn on the 2020, press the ON/OFF key. To turn the power off, press the ON/OFF key and hold it down for 2 seconds, and then release it. This is done to prevent accidental power off.

The EXIT key provides a way of returning to the default display. In the functional map, the soft keys allow the user to advance and the EXIT key provides a way to go back. If the user is at the initial entry of the menu, EXIT will return you to the default display.

The ENTER key has a context sensitive function. When the user is operating or navigating through the function map, the ENTER key is used to exit the functions and return to the default display. When entering data such as a name, number, date, or time, ENTER is used to confirm the entry.

4.1.2.2 Soft Keys

The three soft keys on the 2020 are located directly below the status display. Each key has varying functions for configuring the 2020, editing the data logger, and controlling the display. Because only three soft keys are available, each function is broken down into a path.

4.1.2.3 Entering Text with the Soft Keys

For all information that is entered, the left, center, and right soft keys correspond to the up, down, and right arrow.

The up and down arrows are used to change the character highlighted by the cursor. The right arrow is used to advance the cursor to the next character on the right. When the cursor is advanced past the right most character, it wraps around to the first character again. To accept the changes, press the ENTER key. To ignore the change, press EXIT.

Formatting characters, such as the colon (:) in the time, the decimal (.) in a concentration, and the slash (/) in the date, are skipped when advancing the cursor.

All inputs are an eight-character input, which is displayed on the right side of the top line of the status display. The prompt, describing the input, occupies the left half of the top line. The soft keys are defined on the bottom line of the status display.

4.2 Default Display

The meter display shows the detected concentration. The resolution of the display changes with the magnitude of the reading. A reading of 0 to 99.9 will be displayed with a resolution of 0.1 ppm. A reading greater than 99.9 will be shown with a resolution of 1 ppm or 1 mg/m^3. The meter will display concentrations up to 2000 ppm or 2(99) mg/m^3.

The status display is used to display the instrument status, date, time, units, and active soft keys.

The default display provides the following information: instrument status, current detected concentration, time, date, and measurement units. The status display toggles between showing time and units and then the date.

When the display mode is MAX, the date and time correspond to the date and time the MAX concentration was recorded. In TWA mode, the time represents the number of hours and minutes during which the TWA has been accumulating. For PEAK and STEL monitoring, the date and time correspond to the current date and time.

4.3 Monitoring

4.3.1 Instrument Status

The instrument status is shown on the left of the first line of the status display and on the Table and Graph outputs. Each status has a priority assigned to it. If more than one status is in effect, then the status with the highest priority is displayed until the condition is corrected or until the option is turned off.

4.3.2 Alarms

While operating the instrument, any one of three alarm conditions can occur. To accurately identify the source of the alarm, each type of alarm has been given a unique status.

In addition to the status, the 2020 also has an audible alarm and a visual alarm LED. To conserve power, the 2020 alternates between these two alarm indicators, rather than operating both concurrently. Different alarms are identified by the frequency at which the 2020 alternates as follows: PEAK alarm, 5 times per second; STEL alarm, 2.5 times per second; and TWA alarm, 1.25 times per second.

The left soft key is used for acknowledging alarms, and is labeled "Ack." If no alarm exists, then the "Ack" key is not shown. To clear the alarm, press the "Ack" key. Once acknowledged, the alarm indicators are cleared. The alarm status will remain until the alarm condition clears.

The 2020 updates the peak concentration once every second. Following every update, the peak concentration is compared to the peak alarm level, and, if exceeded, an alarm is triggered.

If a 15-minute average concentration exceeds the selected STEL, a STEL alarm is generated.

The TWA alarm is generated when the current average concentration over an 8-hour period, since the TWA was last cleared, has exceeded the TWA exposure limit.

During calibration, all alarms are disabled. Once the calibration is complete, the alarms are re-enabled.

4.4 STEL, TWA, MAX, and PEAK Operation

The 2020's meter display can be configured to show one of four values: STEL, TWA, PEAK, and MAX.

4.4.1 Short-term Exposure Limit Mode

The STEL mode displays the concentration as a 15-minute moving average. The 2020 maintains 15 samples, each representing a 1-minute averaging interval.

Once every minute, the oldest of the 15 samples is replaced with a new 1-minute average. This moving average provides a 15-minute average of the last 15 minutes with a 1-minute update rate. Because the average is calculated using 15 one-minute averages, the meter display will only update once every minute.

The STEL is set to zero each time the instrument is turned on. Because STEL is a 15-minute moving average, there is no need to clear or reset the STEL.

STEL calculations are always being performed by the 2020. The user can display the results of the calculations by selecting “STEL” as the Display mode.

4.4.2 Time-weighted Average (TWA) Mode

The TWA accumulator sums concentrations every second until 8 hours of data have been combined. If this value exceeds the TWA alarm setting, a TWA alarm is generated. The TWA is not calculated using a moving average. Once 8 hours of data have been summed, the accumulation stops. In order to reset the TWA accumulator, press the "Clr" key.

This sum will only be complete after 8 hours, so the meter displays the current sum divided by 8 hours. While in TWA mode, the time on the status display will show the number of minutes and hours of data that TWA has accumulated. When this reaches 8 hours, the 2020 stops accumulating data and the TWA is complete.

TWA calculations are always being performed by the 2020. The user can display the results of the calculations by selecting TWA as the Display mode.

4.4.3 MAX Mode

The MAX mode displays the maximum signal, with the date and time that it was recorded. The 2020 continues to log data according to the selected averaging interval, but only the maximum detected concentration is displayed on the meter display.

The right soft key is used to clear the meter when displaying MAX. The "Clr" key only affects the reading that the meter is displaying. For example, if the MAX reading is displayed, and "Clr" is pressed, only the MAX value is cleared. The TWA is still accumulating in the background.

4.4.4 PEAK Mode

The PEAK mode displays the current detected concentration. The reading is updated once a second. In the background, the 2020 data logger is sampling the concentration and measuring minimum, maximum, and average concentrations for the selected averaging interval. At the end of every interval, one entry is placed in the data logger until the data logger is full. Operation within the other specialized modes is the responsibility of the SSO.

4.5 <u>Set Functions</u>

Set functions are used to set up the 2020. Three functions can be set on the 2020: Pump, Clock, and Calibration.

4.5.1 Pump

The Pump function is used to control the pump. After selecting “Set Pump,” the 2020 responds by displaying the new pump status.

The detector is also turned off when the pump is turned off. This prevents the detector from being damaged when there is no sample flowing through the detector.

When the pump and the detector are off, the meter display will be blank. Turn the pump and detector off when concentration measurements are not necessary, and the 2020 will only be used for reviewing data

or generating reports. By operating the instrument with the pump and detector off when they are not needed, the lives of the battery and ultraviolet (UV) lamp will be conserved.

To set the pump:

1. Press the ENTER key. The top line of the status display changes to "Select?" The bottom line displays three soft key names: "Set," "Log," and "Disp."

2. Press the soft key below "Set."

3. The names of the soft keys change to reflect the Set options. The display now shows three devices that can be set: "Clock," "Pump," and "Cal." Press the "Pump" key.

4. The 2020 turns the pump off. If the pump was off, pressing "Pump" will turn the pump on.

5. A message will be displayed to show the status of the pump. The 2020 reverts back to the previous menu after a few seconds.

6. To return to the default display, press the ENTER key.

4.5.2 Clock

The Clock function is used to set both the current date and time.

To set the clock:

1. Press the ENTER key.

2. Press the "Set" key.

3. When the names of the soft keys change, press the "Clock" key.

   The up and down arrows are used to change the character underlined by the cursor. The right arrow is used to advance the cursor to the next character on the right. When the cursor is advanced past the right-most character, it wraps around to the first character again.

Formatting characters, such as the colon (:) in the time and the slash (/) in the date are skipped when advancing the cursor.

4. Use the "arrow keys" to enter the correct time. The time is formatted as Hour:Minute:Second.

5. Press the ENTER key to confirm the time and move to the date option.

6. When setting the date, the 2020 prompts for the current date formatted as Year/Month/Day. Use the "arrow keys" to enter the correct date.

7. Press the ENTER key to confirm the date and return to the Set options. Wait for the display to timeout or press ENTER to return to the default display.

4.5.3 Calibration (Cal)

“Cal” allows the user to set up and calibrate the 2020. There are three options under the Cal function: "Zero," “Span," and "Mem."

A calibration memory consists of a name, a response factor, and PEAK, TWA, and STEL alarm levels.

The "Zero" and "Span" keys are covered in detail in the manufacturer's operations manual for the instrument.

To edit the calibration memory, select "Mem" and then "Chng." The 2020 prompts the user with two new soft keys: "User" and "Lib."

4.5.4 Library (Lib)

Library selections simplify Cal Memory programming and provide standard response factors for approximately 70 applications. "Lib" allows the user to select an entry from a pre-programmed library. The name, response factor, and three alarm levels are all set from the library. To select a library entry to program the selected Cal Memory:

1. Select "Set," "Cal," "Mem," "Chng," and "Lib."
2. Use the "Next" and "Prev" keys to scroll through the list. See the manufacturer's manual Appendix 8.7 for a list of the library entries.

4.6 Preparing for Field Operation of the Photovac 2020

Turning The 2020 On

1. Turn the 2020 on by pressing the ON/OFF key.

2. The 2020 will display the software version number. Wait for the 2020 to proceed to the default display.

3. Allow 10 minutes for the instrument to warm up and stabilize.

4. Press the Enter Key. The default display will provide three soft key selections, "Set," "Log," and "Display."

5. Press "Set." From this option, three other soft key selections will be offered: "Pump," "Clock," and "Cal."

6. Press "Cal." This will begin the calibration sequence. The first selection is to zero the instrument.

7. Press Enter; zeroing will begin. (Note: When employing zero gas, attach and activate zero gas supply at this time.)

8. The next selection offered will be Span. Press Enter, at which time the concentration will be requested. The isobutylene calibration gas employed under general service will be marked on the side of the container. Use the soft keys to toggle into position and to log the concentration. Once the concentration is logged, press "Enter." The direction on the status display will indicate spanning. At this time, hook up the span gas with a regulator to the Photovac 2020 and open it to supply enough flow to elevate the flow rate indicator to the green indicator line (1/8 inch from the rest position).

9. Once spanning is complete, the alarms, which have been disabled during calibration, will activate, indicating that calibration is complete.

10. Document this calibration procedure using a Document of Calibration form (included in Appendix A).

This instrument is ready for general purpose application.

Calibration is to be performed daily or prior to each use, in accordance with this section.

4.7 Maintenance and Calibration Schedule

Function	Frequency
Routine Calibration	Prior to each use.
Factory Inspection and Calibration	Once a year, or when malfunctioning.
Wipe Down the Outer Casing of the Unit	After each use.
Clean UV Light Source	Every 24 hours of operation.
Sample Inlet Filter	Change on a weekly basis or as required by level of use.
Battery charging	After each use.
Clean ionization chamber	Monthly.

4.7.1 Cleaning the UV Light Source Window

1. Turn the FUNCTION switch to the OFF position. Use the 2020 multi-tool and remove the lamp housing cover. CAUTION: The UV lamp is delicate and expensive; handle carefully.

2. Tilt the lamp housing with one hand over the opening; slide the lamp out of the housing.

3. The lamp window may now be cleaned with any of the following compounds using lens paper:
   a. 11.7 eV Lamp - Dry Aluminum Oxide Powder (3.0 micron powder)
   b. All other lamps-HPLC Grade Methanol

4. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Replace the o-ring as necessary, reinstall the lamp housing cover, and tighten it using the 2020 multi-tool. (Do not over tighten.)

5. Recalibrate the instrument as per Section 4.6.

4.7.2 Cleaning the Ionization Chamber

1. Turn the FUNCTION switch to the OFF position and remove the lamp housing cover and lamp as per Section 4.7.1.

2. Using a gentle jet of compressed air, gently blow out any dust or dirt.

3. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Replace the o-ring as necessary, reinstall the lamp housing cover, and tighten it using the 2020 multi-tool. (Do not over tighten.)

4. Recalibrate the instrument as per Section 4.6.

4.8 Instrument Advantages

The Photovac 2020 is easy to use in comparison to many other types of monitoring instrumentation. Its detection limit range is in the low parts-per-million range. Response rapidly reaches 90 percent scale of the indicated concentration (less than 3 seconds for benzene). This instrument's automated performance covers multiple monitoring functions simultaneously, incorporating data logging capabilities.

4.9 Limitations of the Photovac 2020 Photoionization Monitor

- Because the 2020 is a nonspecific total gas/vapor detector, it cannot be used to identify unknown chemicals; it can only quantitate them in relationship to a calibration standard (relative response ratio).

- For appropriate application of the 2020, ionization potentials of suspected contaminants must be known.

- Because the types of compounds that the 2020 can potentially detect are only a fraction of the chemicals possibly present at a hazardous waste site or incident, a background or zero reading on this instrument does not necessarily signify the absence of air contaminants.

- The 2020 instrument can only monitor certain vapors and gases in air. Many nonvolatile liquids, toxic solids, particulates, and other toxic gases and vapors cannot be detected.

- PIDs are generally not specific. Their response to different compounds is relative to the calibration gas used. This is referred to as relative response ratio. Instrument readings may be higher or lower than the true concentration. This can be an especially serious problem when monitoring for total contaminant concentrations if several different compounds are being detected at once.

- The 2020 is a small, portable instrument that cannot be expected to yield results as accurately as laboratory instruments.

4.9.1 Variables Affecting Monitoring Data

Monitoring a hazardous waste site environment can pose a significant challenge in assessing airborne concentrations and the potential threats to site personnel. Several variables may influence both dispersion and the instrument's ability to detect actual concentrations. Some of the variables, which may impact these conditions, are as follows:

- Temperature - Changes in temperature or pressure will influence volatization and affect airborne concentrations. Additionally, an increase or decrease in temperature ranges may have an adverse effect on the instrument's ability to detect airborne concentrations.

- Humidity - Excessive levels of humidity may interfere with the accuracy of monitoring results.

- Rainfall - Through increased barometric pressure and water, rainfall may influence dispersion pathways affecting airborne emissions.

- Electromagnetic interference - High voltage sources, generators, other electrical equipment may interfere with the operation and accuracy of direct-reading monitoring instruments.

5.0 TROUBLESHOOTING

5.1 <u>Fault Messages</u>

When the "Fault" status is displayed, the 2020's operation is compromised.

Fault 1: Signal from zero gas is too high.

Cause: If another fault occurred while the 2020 was setting its zero point, then this fault is displayed.
Action: Ensure no faults are occurring and calibrate the 2020 again.
Cause: Contamination of sample line, sample probe, or fittings before the detector.
Action: Clean or replace the sample line, sample probe, or the inlet filter.
Cause: Span gas and zero air are switched.
Action: Ensure clean air is used to zero the 2020. If you are using gas bags, mark the calibration and zero gas bags clearly.

Cause: Ambient air is contaminated.

Action: If you are unsure about the quality of ambient air, use a supply of commercial zero grade air to zero the 2020.

Fault 2: Signal from span gas is too small.

Cause: Operator may have switched the span gas and zero air.

Action: Ensure clean air is used to zero the 2020. If you are using gas bags, mark the calibration and zero gas bags clearly.

Action: Ensure the span gas is of a reliable concentration.

Cause: UV lamp window is dirty.

Note: Do not remove the detector lamp in a hazardous location.

Action: Clean the UV lamp window.

Cause: UV lamp is failing.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp.

Cause: Incompatible application.

Action: The concentration and sample gas are incompatible for use with the 2020.

Fault 3: UV lamp fault. UV lamp has not started.

Cause: The UV lamp has not started immediately.

Action: This fault may be seen momentarily when 2020 is first turned on. Allow 30 to 60 seconds for the UV lamp to start and the fault to clear.

Cause: The UV lamp serial number label is blocking the photocell.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the label is blocking the photocell. Rotate the lamp approximately 90 degrees and then try to start the 2020 again. If the fault persists, replace the lamp.

Cause: the UV lamp is not installed.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a UV lamp.

Cause: The UV lamp has failed.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp.

Cause: Electronic problem.

Action: If a new UV lamp still generates this fault, then contact the Photovac Service Department.

Fault 4: Pump current too low or too high.

Cause: If the pump sounds labored, then the pump is operating beyond normal operating parameters.

Action: Check for an obstruction in the sample line. Make sure sample line, sample probe, or inlet filter are not plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace the inlet filter.

Action: Ensure the sample outlet, located on the underside of the 2020, is not obstructed.

Cause: The UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the TtNUS equipment manager.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the TtNUS equipment manager.

Cause: The pump has failed.

Action: Contact the TtNUS equipment manager.

5.2 Specific Problems

Problem: Very low or no instrument response detected, yet compounds are known to be present.

Cause: The 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in Section 4.6 of the User's Manual. After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the TtNUS equipment manager.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Disconnect the battery charger before calibrating the 2020.

Cause: Calibration memories have not been programmed correctly.

Action: Program all the calibration memories you require for your application. You must use the correct calibration gas and concentration for each Cal Memory.

Cause: The response factor has been set to zero.

Action: Enter the correct response factor. Refer to Appendix 8.6 for a list of response factors. If the compound is not listed in Appendix 8.6 or you are measuring gas mixtures, then enter a value of 1.0. See User's Manual provided by the manufacturer.

Cause: You are not using the correct Cal Memory.

Action: Select the correct Cal Memory for your application.

Note: It does not matter which Cal Memory is selected or which response factor is entered. The 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: Detector is leaking. A decrease in sensitivity may be due to a leak in the detector.

Note: Do not remove or replace the detection lamp in a hazardous location.

Action: Ensure the UV lamp has been installed correctly.

Action: Ensure the lamp cover has been tightened down. Do not overtighten the cover.

Action: Ensure the o-ring seal on the lamp cover is positioned correctly.

Cause: The UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the TtNUS equipment manager.

Cause: UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the TtNUS equipment manager.

Cause: The sampling environment is extremely humid.

Action: Water vapor is not ionized by the PID, but it does scatter and absorb the light and results in a lower reading. The 2020 detector has been designed to operate under high humidity conditions. Under extreme conditions, you may notice decreased response due to humidity.

Cause: The UV lamp is failing.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: High concentration of non-ionizable compounds. Chemical compounds, such as methane, with IPs greater than the 10.6 eV scatter and absorb the UV light. Sensitivity may be decreased significantly. Application with high backgrounds of such materials may be incompatible with the 2020. Contact the Photovac Applications Group for more information.

Problem: Erroneously high readings.

Cause: Sampling environment is extremely humid.

Action: Water vapor may contain mineral salts, which carry a charge. The water vapor becomes an electrolytic solution, which becomes ionized when it enters the detector. Atmospheric water in areas around the sea or stagnant water may produce a response in the absence of contaminants.

The same effect may be seen when conducting ground water investigations in areas where the water is hard because it contains a significant concentration of minerals.

Cause: The 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in Section 4.6. After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the TtNUS equipment manager.

Cause: Cal Memories have not been programmed correctly.

Action: Program all the Cal Memories you require for your application. You must use the correct calibration gas and concentration for each Cal Memory. See Section 3.4 of the User's Manual.

Cause: You are not using the correct Cal Memory.

Action: Select the correct Cal Memory for your application. See Section 3.2.2 or 3.3.2 of the User's Manual.

Note: It does not matter which Cal Memory is selected or which response factor is entered. The 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: The detector has been short circuited by foreign matter in the detector cell.

Note: Do not service the 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the TtNUS equipment manager.

Problem: Date and time settings are not retained.

Cause: The battery pack was removed before the 2020 was turned off.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Replace the battery pack and reset the time and date. Ensure that the 2020 has been turned off before removing the battery pack.

Cause: The 2020 has not been used for 3 months or more and the internal battery (not the external battery pack) has discharged.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Connect the 2020 to the AC adapter and turn the 2020 on. Turn the pump off. While the 2020 is running, the internal battery is charging. Leave the instrument running for approximately 24 hours.

Problem: Instrument status shows "Over."

Cause: High concentrations of gases and vapors will cause a rapid change in signal level. The detector and associated electronics may become temporarily saturated.

Action: Wait a few seconds for the status to return to normal. PIDs are designed to detect relatively low concentrations of gases and vapors. Exposure to very high concentrations may result in a very high or maximum response.

Cause: The detector has become saturated.

Action: Move the 2020 to a location where it can sample clean air. Sample clean air until the reading stabilizes around 0.

Cause: Detector has been short circuited by foreign matter in the detector cell.

Note: Do not service the 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust or dirt in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the TtNUS equipment manager.

Problem: Display is blank.

Cause: Battery pack is critically low.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Replace the battery pack or connect the 2020 to the AC adapter.

Cause: The battery pack is not connected to the instrument correctly.

Action: Ensure the battery pack connector is securely attached to the connector on the 2020.

Cause: There is an undetermined problem.

Action: Reset the 2020. You must leave the instrument on while you disconnect the battery pack. This will reset the instrument. Reconnect the battery pack and close the battery hatch. Turn on the 2020, set the time and date, and program all the calibration memories that you are using.

Action: Contact the TtNUS equipment manager.

Problem: Sample flow rate is less than 300 ml/min.

Cause: The inlet filter is plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace the inlet filter.

Cause: The inlet filter has not been installed properly.

Action: Ensure that the inlet filter has been installed correctly.

Cause: The UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the TtNUS equipment manager.

Cause: The UV lamp is too wide, causing flow to be restricted.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the TtNUS Equipment Manager.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the TtNUS equipment manager.

Cause: Sample outlet is obstructed.

Action: Ensure the sample outlet is not obstructed in any way.

Cause: Pump has been damaged.

Action: Contact the TtNUS equipment manager.

Problem: Liquid has been aspirated.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter.

Action: Contact the TtNUS equipment manager.

Problem: Corrosive gases and vapors have been sampled.

Cause: The 2020 has been exposed to corrosive gases and vapors.

Action: Corrosive gases and vapors can affect the electrodes within the detector as well as the lamp window. Prolonged exposure to corrosive materials may result in permanent fogging or etching of the window. If the 2020 is exposed to corrosive material, contact the TtNUS equipment manager.

6.0 SHIPPING

The Photovac may be shipped as cargo or carried on as luggage if there is no calibration gas cylinder accompanying the kit. When shipping or transporting the calibration gas, a Hazardous Airbill must be completed.

7.0 REFERENCES

Photovac 2020 Photoionization Monitor User's Manual, 1995.

8.0 ATTACHMENTS

1. Equipment Calibration Log

ATTACHMENT 1

EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : ____________________ INSTRUMENT NAME/MODEL: ____________________

SITE NAME: ____________________ MANUFACTURER: ____________________

PROJECT No.: ____________________ SERIAL NUMBER: ____________________

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

STANDARD OPERATING PROCEDURE
SOP-11

SUBSURFACE SOIL AND GROUNDWATER SAMPLING USING DIRECT-PUSH TECHNOLOGY

1.0 PURPOSE

This procedure provides general guidance and reference information on direct-push technology (DPT). DPT is designed to collect soil and groundwater samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, ability to sample in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells.

The methods described herein are specific for soil, groundwater, and soil gas samples at Naval Support Activity (NSA) Crane. Guidelines by Southern Division, Naval Facilities Engineering Command (South Div NAVFAC, 1997) and the State of Indiana regulatory requirements in Article 16 (Water Well Drillers) of Chapter 310 of the Indiana Administrative Code (310 IAC 16) should be consulted.

2.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction.

Field Geologist - The Tetra Tech Field Geologist supervises and documents DPT activity performed by the driller, and insures that the soil and groundwater samples collected accurately representative the desired media and sample interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

All Field Personal – All field personal including, the drilling contractor personnel and other field staff, must have all of the health and safety training required to perform the work, as specified in the Health and

Safety Plan (HASP). All field personnel shall be aware of the potential presence of underground utilities. Proper utility clearance must be obtained by the Tetra Tech Project Manager (PM) prior to any DPT activity.

3.0 REQUIRED EQUIPMENT/ITEMS

The list of equipment and items required for DPT sampling includes, but is not limited to:

Health and safety equipment as required by the HASP and the Site Safety Officer.

DPT Rig is supplied by the drilling subcontractor and may include the following:

- 4-foot x 2-inch diameter macrocore sampler
- Probe sampling adapters
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Stainless steel screen point groundwater sampler (Geoprobe®, HydroPunch™, or equivalent)

55-gallon drums to containerize IDW (supplied by the drilling subcontractor).

Required decontamination materials including distilled water, deionized water, paper towels, and stainless steel clamps.

Writing utensil (preferably black ink), non-latex gloves, bound field logbook, chain-of-custody forms, sample labels, boring log, sample logsheets, engineer's tape (or equivalent), and stainless-steel spoon or trowel.

Required sample containers with appropriate preservative: All sample containers for analysis by fixed-base laboratories will be supplied and deemed certified clean by the laboratory. Additional sampling equipment as needed, such as photo-ionization detector (PID), flame-ionization detector (FID), Ziplock bags, calculator, wristwatch, and timer, and cooler (containing ice), peristaltic pump, inertial lift pump, silicon tubing, polyethylene (PE) tubing, water quality meter with a flow through cell, LaMotte 2020 Turbidity Meter (or equivalent), water level indicator, 0.45 micron filter cartridge, trip blanks, and bucket to collect development/purge water.

4.0 GLOSSARY

Direct Push Technologies (DPT) -DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools.

Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photoionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

5.0 DPT SOIL SAMPLING PROCEDURES

General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

DPT Sampling Methodologies

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Tetra Tech PM in accordance with the project-specific plan.

Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.

The sampler is advanced continuously in 4-foot intervals, or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.

The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.

- Attach the metal trough from the Geoprobe® Sampling Kit (or equivalent) firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.
- Place the acetate liner containing the soils in the trough.
- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife. Then remove the strip of acetate from the trough to gain access to the collected soils. **Do not** attempt to cut the acetate liner while holding it in your hand.
- Field screen the sample with an FID or PID (according to manufacturer's Standard Operating Procedure [SOP]) and observe/examine the sample. If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organic compounds (VOCs) analysis.
- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.
- In the event the direct push van, truck, or track mounted rig cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).
- Sampling equipment is decontaminated prior to collecting the next sample.

6.0 GROUNDWATER SAMPLING PROCEDURES

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling. Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

6.1 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited to the following:

- 2-foot x 1 -inch diameter mill-slotted (0.005 to 0.02-inch) well point Connecting rods
- Roto-hammer with 1.5-inch bit Mechanical jack
- 1/4-inch outside diameter (OD) PE tubing
- 3/8-inch OD PE tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

6.2 DPT Sampling Methodologies

Once the water table has been encountered, a stainless steel screen point groundwater sampler (Geoprobe®, HydroPunch™, or equivalent) will be driven by DPT to collect a groundwater sample at the water table.

- Field screening of VOC vapors in the borehole shall be done using a FID or PID.
- The screen point will be allowed to equilibrate for at least 15 minutes.
- Once equilibration occurs, measurement of the static water level will be taken. This initial water level measurement will be used to assess the amount of water present in the screen point and to determine the amount of silt and/or sand infiltration.
- Development of the screen point will be accomplished using a peristaltic pump.
- Insert the intake end of a length of dedicated PE tubing to the bottom of the screen point and attach a length of silicon tubing (approximately 1 foot) to the discharge end of the PE tubing. The silicon tubing will be threaded around the rotor of the pump and out of the pump.

- The PE tubing will be lifted and lowered slightly while the pump is operating. The maximum pump rate will be approximately 2 liters per minute during development; however the yield of the formation will dictate the pumping rate.
- Measurement of pH, specific conductance, turbidity, dissolved oxygen, eH, salinity, and temperature shall be recorded every 5 to 10 minutes during the development process using a water quality meter and flow-through cell, with the exception of turbidity. Turbidity measurements will be taken with a Lamotte Turbidity Meter from water collected from a T-connector with a valve inserted in the pump discharge tubing prior to entering the flow-through cell.
- After removal of sediment from the bottom of the screen point, the screen point will be pumped until discharge water is visibly clear and no further sediments are being generated.
- Stabilization is achieved after two consecutive readings taken at 5 to 10 minutes intervals of the following field parameters has occurred:

    - pH +/- 0.1 standard units
    - Turbidity +/- 10% for values greater than 1 NTU
    - Specific conductance +/- 3%
    - Temperature +/- 3%
    - eH +/- 10 millivolts
    - Dissolved oxygen +/- 10%

- Samples will be collected using the peristaltic pump set at 0.2 liters per minute or lower, depending on the yield of the formation. Samples (with the exception of samples to be analyzed for VOCs) will be collected directly from the pump discharge. The pump shall continuously operate between development, purging, and sampling.

- If the above condition(s) have not been met after three well volumes have been removed, this will be recorded on the field sample form and the groundwater sample will be collected.

- Record the sample date and time (using military time) on a Tetra Tech Groundwater Sample Log Sheet and on a chain-of-custody form.

- Record the sample date and time (using military time) on an adhesive-backed sample label and affix the sample label securely to the sample container.

- With the pump continuing to run, allow the pump discharge to flow gently down the inside of the sample container with minimal turbulence when filling sample containers. Avoid immersing the discharge tube into the sample as the sample container is being filled.

- Cap each container immediately after filling.

- Place the sample container into a ziplock® bag and then into a cooler containing ice.

- Repeat the last four steps for each sample container collected.

- The pump rate should not be adjusted after sampling has commenced. If it becomes necessary to adjust the pump rate, document the change on the Tetra Tech Groundwater Sample Log Sheet.

- All samples will be collected into pre-preserved bottles (if required) supplied by an approved laboratory. The hierarchy of filling sample containers is as follows:

  - VOCs
  - Polychlorinated Biphenyls (PCBs)
  - Total metals
  - Dissolved metals
  - Perchlorates

- This hierarchy takes into consideration the volatilization sensitivity of groundwater samples. The only deviation from this order will be the collection of samples for VOC analysis. The collection of VOCs will be the final parameter collected due to the fact that VOCs will not be collected using the peristaltic pump.

- A single-use, disposable, in-line 0.45-micron filter cartridge shall be used to collect dissolved metals samples. Attach the filter cartridge to the discharge end of the pump tubing. Prior to filling containers with filtered sample, rinse the filter cartridge with approximately 100 milliliters (mL) of water from the boring to be sampled. Direct the discharge from the filter cartridge into the sample bottle and collect the filtered sample. The laboratory will supply all sample containers, and the laboratory will pre-preserve sample containers where appropriate.

- Once all of the sample containers have been filled (with the exception of those sample containers for VOC analysis), the pump shall be shut off. Record the sample date and time (in military time) on an adhesive-backed sample label and affix the sample label securely to the sample container. Sample containers for VOCs will be filled by crimping the discharge end of the PE tubing (immediately after shutting off the pump). Remove the inlet end of the PE tubing from the well, suspend the inlet tubing above the VOC sample container (pre-preserved 40 mL vial), and slowly allow water to fill each VOC sample container by gravity flow. The discharge of sample from the PE tubing shall be accomplished in a manner that allows the water to gently flow down the inside of the sample container. Sample containers for VOCs must be completely filled so that no headspace exists in the container. Record the end time for sampling on a Tetra Tech Groundwater Sample Log Sheet.

- Once collection of samples is complete, the driller shall remove the screen point and the screen point will be decontaminated in accordance with the procedures outlined in the decontamination SOP.

- If needed, continuous soil and groundwater sampling using DPT below the water table shall be done in accordance with those procedures outlined above.

- After the groundwater samples have been collected, the driller shall retract the screen point sampler from the borehole and proceed to abandon the borehole with a grout pump using a cement bentonite grout mix from the bottom up to the ground surface.

- When advancing a boring using DPT and refusal is encountered, the boring shall be deemed complete, drilling shall cease, and the borehole shall be abandoned with a grout pump using a cement bentonite grout mixture.

STANDARD OPERATING PROCEDURE
SOP-12

MONITORING WELL INSTALLATION

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper design and installation of ground water monitoring wells. The methods described herein are specific for monitoring well construction at Naval Support Activity (NSA) Crane. Guidelines by Southern Division, Naval Facilities Engineering Command (South Div NAVFAC, 1997) and the State of Indiana regulatory requirements in Article 16 (Water Well Drillers) of Chapter 310 of the Indiana Administrative Code (310 IAC 16) should be consulted.

2.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The drilling contractor personnel must have all of the health and safety training required to perform the work, as specified in the Health and Safety Plan (HASP).

Field Geologist - The Tetra Tech Field Geologist supervises and documents well installation and construction performed by the driller, and insures that the screen interval for each monitoring well is properly placed to provide representative groundwater data from the monitored interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

3.0 REQUIRED EQUIPMENT/ITEMS

The following list includes equipment and items required for monitoring well installation:

Health and safety equipment as required by the HASP and the Site Safety Officer.

Well drilling and installation equipment with associated materials (typically supplied by the driller).

Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineer’s rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink

marker for marking monitoring wells, sample jars, well installation forms, boring logs, soil sample log forms, chain-of-custody records, sample coolers with ice, and a field notebook).

4.0 WELL DESIGN AND CONSTRUCTION

New wells will be installed only with Navy concurrence. Based on observations and information gathered during the drilling of each hole, the total depth of the hole and the placement of the well screen will be determined at the discretion of the Tetra Tech Field Geologist or the Tetra Tech Field Operations Leader (FOL). The decision concerning the monitored interval and well depth will be based on the following (and possibly other) information collected while the well bore is being drilled and logged:

- The specific depths where poorly-cemented sandstone units, fractured rock, or other permeable rock zones are encountered,
- The specific depths where above-average rates of ground water were brought to the surface during drilling,
- The specific depth interval where contaminants (i.e., VOCs), if any, are encountered during drilling.

All of this information will be recorded on the borehole log as the hole is drilled.

Overburden drilling followed by diamond coring (if necessary) will be performed at borehole locations. For each well, the coring will proceed to the final depth of the borehole. Once the coring has been completed and the core has been logged, then the hole will be reamed out with a 6 to 8-inch diameter air rotary bit. The air rotary equipment must have a filter on the compressed air line going to the borehole to prevent oil and other organics from being introduced. Once the hole has been completed to depth, the boring shall be cleaned out using the compressed air of the rig. Note: all drilling equipment must be decontaminated before it is placed in a borehole.

A 6-inch diameter steel isolation casing will be installed and pressure grouted in the deep wells to seal the upper groundwater from deep groundwater. The grout will be allowed to cure for a minimum of 24 hours before resuming coring and reaming to the total depth of the borehole.

All monitoring wells will be constructed of schedule-40, flush-joint threaded, 2-inch inside diameter (ID) polyvinyl chloride (PVC) riser pipe and flush joint threaded, factory slotted well screen with a threaded end cap. The well screens will be factory slotted to 0.020-inch size. Each section of well casing and screen shall be National Sanitation Foundation (NSF) approved. Well screens will be 10-feet long, but

may be longer or shorter based on the subsurface conditions encountered. A PVC cap will be placed on the bottom and will also be flush-threaded. Thermoplastic pipe shall comply with ASTM F-480 (1981). Other means of joining casings using glue, gaskets, pop rivets or screws are not allowed. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material.

Monitoring wells will be installed immediately upon completion of drilling. A well screen section with bottom cap and the proper amount of riser pipe will be assembled and lowered down the borehole. Spacers may be used to ensure that the casing and screen are centered and are aligned straight. Clean silica sand pack will be installed through the borehole. The sand pack will be extended from 0.5 feet below the well screen to 2.0 feet above the top of the well screen. Clean silica sand of U.S. Standard Sieve Size No. 20 to 40 will be used.

A minimum 2-foot thick bentonite pellet seal will be installed above the filter pack and allowed to hydrate as determined by field geologist before grout is added above the seal. Only 100-percent, certified pure, sodium bentonite will be used for well construction. The depths of backfill materials will be constantly monitored during well installation using a weighted stainless steel or fiberglass tape measure.

The remaining annulus above the hydrated bentonite seal will be backfilled to the surface using a tremie pipe, with a 20:1 cement/bentonite grout. A maximum of 10 gallons of water per 94-pound bag of Type-1 cement will be used. The grout mixture should be blended in an above-ground rigid container or mixer to produce a thick lump-free mixture.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets extending to the surface. The grout effectively seals the well and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom of the hole upward, to prevent bridging, and to provide a better seal. However, in shallow boreholes that do not collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

When the well is completed and grouted to the surface, a protective steel surface casing is placed over the top of the well. The finished well casing shall extend at least 2 ft above the ground level. This casing will have a cap that will be locked to prevent vandalism. A vent hole shall be provided in the cap to allow venting of gases and maintain atmospheric pressure as water levels rise or fall in the well. The protective casing has a larger diameter than the riser pipe and is set into the wet cement grout over the riser upon completion. In addition, one hole is drilled just above the cement collar through the protective casing

which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

Four barrier posts shall be placed at the corner of a 3-foot by 3-foot by 6-inch thick concrete pad located at the ground surface.

5.0 DOCUMENTATION OF FIELD ACTIVITIES

A critical part of monitoring well installation is recording of significant details and events in the site logbook, on field forms, and a field logbook.

All installed wells must be registered with the NSA Crane Environmental Protection Department. The following information must be supplied to NSA Crane for each well as soon as this information is known:

- Tag number
- Installation Name (i.e., NSA Crane)
- Contract Task Order number (CTO F276)
- Tetra Tech project number
- Well identification number
- Date installed
- Installer (i.e., Tetra Tech)
- Total well depth
- Screened interval
- Elevation (Top of casing)
- Northing coordinate (ft)
- Easting coordinate (ft)
- Survey coordinate reference system
- Information point of contact

6.0 ATTACHMENTS

1. Bedrock Monitoring Well Sheet
2. Overburden Monitoring Well Sheet

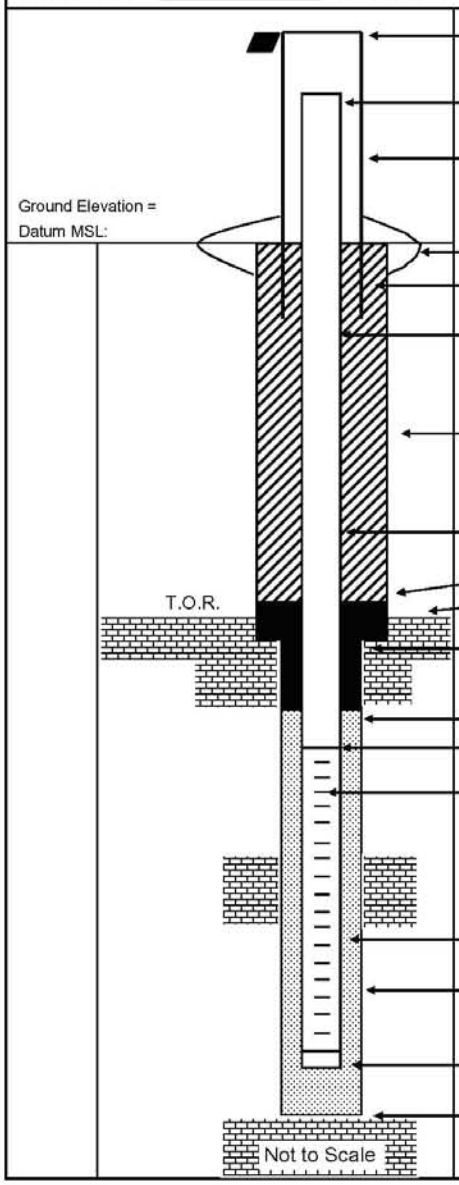
ATTACHMENT 1

BEDROCK MONITORING WELL SHEET

BEDROCK
MONITORING WELL SHEET

WELL No.: _____
PERMIT No:

PROJECT:		DRILLING Co.:		BORING No.:	
PROJECT No.:		DRILLER:		DATE COMPLETED:	
SITE:		DRILLING METHOD:		NORTHING:	
GEOLOGIST:		DEV. METHOD:		EASTING:	



Elevation of Top of Casing: _____

Stick Up of Casing Above Ground Surface: _____
Elevation of Top of Riser: _____

I.D. of Surface Casing: _____

Type of Surface Casing: _____

Type of Surface Seal: _____

I.D. of Permanent Casing: _____

I.D. of Riser: _____

Type of Riser: _____

Borehole Diameter: _____

Type of Backfill: _____

Elevation / Depth Top of Seal: _____ / _____
Elevation / Depth Top of Bedrock: _____ / _____

Type of Seal: _____

Elevation / Depth of Top of Fine Sand: _____ / _____
Elevation / Depth of Top of Filter Pack: _____ / _____
Elevation / Depth of Top of Screen: _____ / _____

Type of Screen: _____

Slot Size x Length: _____

I.D. of Screen: _____

Type of Filter Pack: _____

Diameter of Hole in Bedrock:
Core / Ream: _____

Elevation / Depth of Bottom of Screen: _____ / _____

Elevation / Total Depth of Borehole: _____ / _____

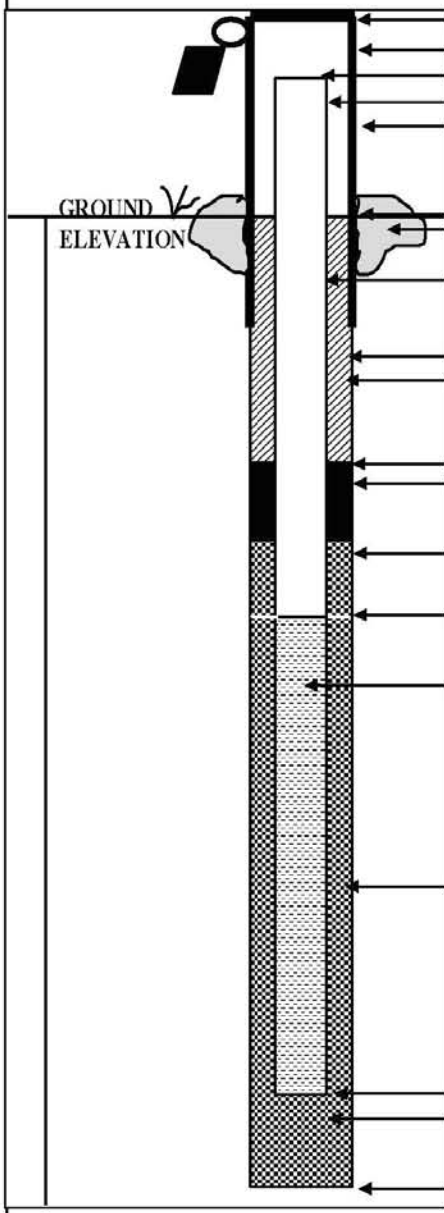
ATTACHMENT 2

OVERBURDEN MONITORING WELL SHEET

BORING NO.: _____

OVERBURDEN MONITORING WELL SHEET

PROJECT:		DRILLING Co.:		BORING No.:	
PROJECT No.:		DRILLER:		DATE COMPLETED:	
SITE:		DRILLING METHOD:		NORTHING:	
GEOLOGIST:		DEV. METHOD:		EASTING:	



ELEVATION OF TOP OF SURFACE CASING: _____
STICK -UP TOP OF SURFACE CASING: _____
ELEVATION OF TOP OF RISER PIPE: _____
RISER STICK-UP ABOVE GROUND SURFACE: _____
I.D. OF SURFACE CASING: _____
TYPE OF SURFACE CASING: _____

GROUND ELEVATION: _____
TYPE OF SURFACE SEAL: _____

RISER PIPE I.D.: _____
TYPE OF RISER PIPE: _____

BOREHOLE DIAMETER: _____
TYPE OF SEAL: _____

ELEVATION / DEPTH OF SEAL: _____ / _____
TYPE OF SEAL: _____

ELEVATION / DEPTH TOP OF FILTER PACK: _____ / _____

ELEVATION / DEPTH TOP OF SCREEN: _____ / _____

TYPE OF SCREEN: _____

SLOT SIZE X LENGTH: _____

I.D. OF SCREEN: _____

TYPE OF FILTER PACK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: _____ / _____
ELEVATION / DEPTH BOTTOM OF FILTER PACK: _____ / _____
TYPE OF BACKFILL BELOW WELL: _____
ELEVATION / DEPTH OF BOREHOLE: _____ / _____

STANDARD OPERATING PROCEDURE
SOP-13

MONITORING WELL DEVELOPMENT

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper development of new and existing monitoring wells. The methods described herein are specific for monitoring wells located at Naval Support Activity (NSA) Crane. Guidelines by Southern Division, Naval Facilities Engineering Command (South Div NAVFAC, 1997) and the State of Indiana regulatory requirements in Article 16 (Water Well Drillers) of Chapter 310 of the Indiana Administrative Code (310 IAC 16) should be consulted.

2.0 RESPONSIBILITIES

The drilling contractor will provide adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing the development of monitoring wells. The drilling contractor personnel must have all of the health and safety training required to perform the work, as specified in the Health and Safety Plan (HASP).

3.0 REQUIRED EQUIPMENT/ITEMS

The following list includes equipment and items required for monitoring well installation:

Health and safety equipment as required by the HASP and the Site Safety Officer.

Well development equipment with associated materials (typically supplied by the driller).

Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).

4.0 WELL DEVELOPMENT METHODS

The development of new wells shall not occur until at least 24 hours after the well has been installed and grouted. This time is required so that the grout in the annulus can set and harden. The purpose of well development is to stabilize and increase the permeability of the sand pack and the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water, if any, is removed from the well.

Sequential measurements of pH, specific conductance, turbidity, and temperature will be taken during development. Development should proceed until criteria are met as stated in Navy Guidelines.

Vigorous on-and-off pumping or a surge block will be used for development.

A surge block that is approximately the same diameter as the well riser will be used to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers; solid and valved surge plungers. Site-specific conditions will dictate which type will be used. In formations with low yields, a valved surge plunger may be preferred, as solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

Development should proceed until three consecutive pH, specific conductance, and temperature readings are within 10 percent of each other and three consecutive turbidity readings are within 5 Nephelometric Turbidity Units (NTUs) of each other. If these criteria cannot be met after five volumes of water have been removed, then one additional well volume will be removed and well development will be considered complete.

If for any reason the above criteria cannot be met, the site geologist should document the event in writing and consult with the Tetra Tech Project Manager (PM) regarding an alternate plan of action.

Well development must be completed at least 24 hours before well sampling. The intent of this hiatus is to provide time for the newly installed well and backfill materials to sufficiently equilibrate to their new environment and for that new environment to re-stabilize after the disturbance of drilling.

5.0 ATTACHMENTS

1. Monitoring Well Development Record

ATTACHMENT 1

MONITORING WELL DEVELOPMENT RECORD

Tetra Tech NUS, Inc.

MONITORING WELL DEVELOPMENT RECORD

Page ___ of _____

Well: _____ Depth to Bottom (ft.): _____ Responsible Personnel: _____
Site: _____ Static Water Level Before (ft.): _____ Drilling Co.: _____
Date Installed: _____ Static Water Level After (ft.): _____ Project Name: _____
Date Developed: _____ Screen Length (ft.): _____ Project Number: _____
Dev. Method: _____ Specific Capacity: _____
Pump Type: _____ Casing ID (in.): _____

Time	Estimated Sediment Thickness (Ft.)	Cumulative Water Volume (Gal.)	Water Level Readings (Ft. below TOC)	Temperature (Degrees C)	pH	Specific Conductance (Units _____)	Turbidity (NTU)	Remarks (odor, color, etc.)

STANDARD OPERATING PROCEDURE

SOP-14

MEASUREMENT OF WATER LEVELS

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes procedures for determining water levels in monitoring wells.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following equipment and field forms are required for determining water levels in monitoring wells.

Groundwater Level Measurement Form: A copy of the Water Level Measurement Sheet is attached.
Bound Field Log Book
Photoionization Detector (PID)
Well Key
Electronic Water-Level Indicator: The water level indicator must have a cable of sufficient length to reach the water surface and be capable of measurements of 0.01 feet.
Decontamination Supplies

3.0 WATER-LEVEL MEASUREMENT PROCEDURES

3.1 Check the operation of the electronic water level indicator or interface meter.

3.2 Record the well identification (ID), date, and time (using military time) on the Water Level Measurement Sheet.

3.3 Unlock the well and remove the well cap.

3.4 Place the well cap on a clean piece of plastic.

3.5 Check the well for the presence of organic vapors in the 2-inch PVC riser pipe as follows:

1. Calibration of the PID shall be done in accordance with appropriate calibration procedures.

2. Insert the PID sample inlet straw approximately three inches into the riser pipe.

3. Record the PID reading on the Water Level Measurement Sheet. If the reading is less than concentrations specified in the site-specific Health and Safety Plan (HASP), proceed to step 3.6. If the reading is greater than the concentration specified in the HASP, measure the concentration in the breathing zone. If the concentration in the breathing zone is less than the concentration specified in the HASP, proceed to Step 3.6. If the reading is greater than the specified concentration, allow the riser pipe to ventilate for ten minutes and repeat the measurement of breathing zone concentrations until the concentrations fall below the level specified in the HASP before proceeding to step 3.6.

3.6 Ensure that the water level indicator probe has been decontaminated before use.

3.7 Slowly lower the probe into the well riser pipe (or into the surface water for staff gages) until an audible and/or visible signal is produced, indicating contact with the water surface.

3.8 Read the water level measurement from the top of the inner casing (or from the staff gage reference point) at the surveyed reference point to the nearest 0.01-foot.

3.9 Record the water level measurement on the Water Level Measurement Sheet.

3.10 Wind the meter cable measuring tape back onto the spool.

3.11 Replace the well cap and lock.

3.12 Decontaminate the meter's probe and cable.

4.0 ATTACHMENTS

1. Water Level Measurement Sheet

ATTACHMENT 1

WATER LEVEL MEASUREMENT SHEET

WATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____
Location: _____ **Personnel:** _____
Weather Conditions: _____ **Measuring Device:** _____
Tidally Influenced: Yes ___ No ___ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Total Well Depth (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Groundwater Elevation (feet)*	Comments

* All measurements to the nearest 0.01 foot

Page ____ of ____

STANDARD OPERATING PROCEDURE

SOP-15

LOW-FLOW WELL PURGING AND STABILIZATION

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for well purging and stabilization utilizing low-flow techniques.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for low-flow purging.

Low-Flow Purge Data Sheet: A copy of this form is attached at the end of this SOP.
Groundwater Sample Log Sheet: A copy of this form and instructions for its completion are included in SOP-16.
Bound Field Log Book
Well key
Electronic water level indicator: The water level indicator must have a cable of sufficient length to reach the water surface and be capable of measurements of 0.01-feet.
Electronic Programmable Controller, model 400 or comparable: This controller regulates air flow in a bladder pump.
Cylinder of compressed nitrogen with regulator: Compressed gas serves as the power source for the bladder pump.
Multiple parameter water quality meter: This unit measures and displays field parameters measured in the field including pH, dissolved oxygen (DO), oxidation-reduction potential (ORP), temperature, and specific conductance.
Flow-through cell adapter for water quality meter
LaMotte Turbidity Meter or comparable: Used to measure turbidity.
Purge water containers
Graduated cylinder and stopwatch: Used to calculate flow rate.

3.0 PROCEDURES FOR WELL PURGING

3.1 Prior to mobilizing to the site, clean, check for proper operation, and calibrate as per manufacturer requirements above equipment as necessary.

3.2 Obtain a static water level measurement of the well to be purged. Record the information on the Groundwater Sample Log Sheet and the Low-Flow Purge Data Sheet. Leave the water level meter suspended in the well casing.

3.3 Calculate one well casing volume as follows:

1. Obtain the total depth of the well.

2. Using the static water level determined in Step 3.2 of this SOP and the total depth of the well, calculate the well casing volume using the following formula:

$$\mathbf{V = (0.163)(T)(r^2)}$$

where:

V	=	Static casing volume of well (in gallons).
T	=	Vertical height of water column (linear feet of water).
0.163	=	A constant conversion factor which compensates for the conversion of the casing radius from inches to feet, the conversion of cubic feet to gallons, and pi.
r	=	Inside radius of the well casing (in inches).

Note: For wells of 1-inch radius (2-inch diameter) V = 0.163 gallons per foot of water column.

3.4 Connect the pump controller to the well pump air supply (at the well cap) by following the instructions in the pump control manual. The pump controller must be turned off when being connected.

3.5 Connect the nitrogen cylinder to the pump controller. The nitrogen cylinder valve must be closed and the regulator line pressure set at zero pounds per square inch (PSI) when being connected.

3.6 Following the instructions found in the water quality meter manual, connect the flow-through cell to the pump discharge line (at the well cap).

3.7 Place the discharge tubing from the flow-through cell to direct the purge water discharge into the graduated cylinder or purge-water container.

3.8 Following the instructions in the pump controller manual, start pumping water from the well.

3.9 Start with the initial pump rate set at approximately 0.1 liters/minute. Use the graduated cylinder and stopwatch to measure the pumping rate. Adjust pumping rates as necessary to prevent drawdown from exceeding 0.3 feet during purging. If no drawdown is noted, the pump rate may be increased (to a max of 0.4 liters/minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 Nephelometric Turbidity Units (NTUs) after all other field parameters have stabilized. If ground water is drawn down below the top of the well screen, purging will cease and the well will be allowed to recover before purging continues. Slow recovering wells will be identified and purged at the beginning of the workday. If possible, samples will be collected from these wells within the same 8-hour workday and no later than 24 hours after the start of purging.

The time to sample any given well will vary greatly due to the many variables associated with low flow purging and sampling, such as:

- Stabilization of parameters
- Possible draw down
- Analytical changes from quarter to quarter
- Varying quality assurance (QA) sample requirements from quarter to quarter
- Variable pump rates

Normally, the time from the start of purging to the end of sampling will be between 1 to 4 hours.

3.10 Measure the well water level using the water level meter every five to ten minutes. Record the well water level on the Low-Flow Purge Data Sheet (attached at the end of this SOP).

3.11 Record on the Low-Flow Purge Data Sheet every five to ten minutes the water quality parameters (pH, specific conductance, temperature, turbidity, ORP, and DO) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell

with distilled water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Sheet.

3.12 Measure the flow rate using a graduated cylinder. Remeasure the flow rate any time the pump rate is adjusted.

3.13 During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections.

3.14 Stabilization is achieved and sampling can begin when a minimum of one casing volume has been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits:

pH ± 0.1 standard units (S.U.)
Specific conductance ± 3%
Temperature ± 1.0 ºC
Turbidity less than 10 NTUs

If the above conditions have still not been met after the well has been purged for four hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data Sheet onto the Ground Water Sample Log Sheet.

If there is a need to leave a well during purging, there are two options:

- One, if the sampler must move for 30 minutes or less but still has a clear line of sight to the well, the sampler may leave the pump running and watch the well until the sampler is able to return to the well.

- Two, if for whatever reason, the sampler must stop purging for an extended period of time or a clear line of sight cannot be maintained, the pump and cell will be shut down. All equipment and supplies will be loaded into the sample vehicle, and the well will be secured before departing.

In both cases, the time purging was stopped and restarted will be noted on the Low-Flow Purge Data Sheet.

3.15 Once sampling activities have been completed, turn the pump off. Remove pump, hoses, cables, and other equipment from the well.

3.16 Decontaminate pumps, hoses, cables, flow-through cell, and other equipment.

4.0 ATTACHMENTS

1. Low-Flow Purge Data Sheet

ATTACHMENT 1

LOW-FLOW PURGE DATA SHEET

LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: _____ WELL ID.: _____
PROJECT NUMBER: _____ DATE: _____

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	S. Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Salinity % or ppt	Comments

SIGNATURE(S): _____ CTO G276 PAGE___OF___

STANDARD OPERATING PROCEDURE
SOP-16

MONITORING WELL SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for monitoring well sampling. Low-flow sampling techniques will be used for ground water sampling at Naval Support Activity (NSA) Crane.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for low-flow sampling of monitoring wells:

Ground Water Sample Log Sheet: A copy of this form is attached at the end of this SOP.

Bound field log book

Chain-of-Custody Form

Bladder Pump

Surgical Gloves

Labeled sample containers: Sample containers are certified clean by the laboratory supplying the sample containers.

Tag for each sample container

Plastic storage bags

Shipping containers with ice

3.0 MONITORING WELL SAMPLING PROCEDURES

3.1 Ground water sampling may be initiated when the monitoring well has been purged and stabilized.

3.2 Record the sample start time (using military time) on the Groundwater Sample Log Sheet. Record the field measurements for pH, oxidation-reduction potential (ORP), specific conductance, temperature, dissolved oxygen (DO), and turbidity.

3.3 With the pump continuing to run, disconnect the flow-through cell from the pump discharge tube and immediately start filling sample bottles directly from the pump discharge. All sample

containers will be supplied by the laboratory, and the laboratory will pre-preserve all sample containers, where appropriate.

3.4 Allow the pump discharge to flow gently down the inside of the container with minimal turbulence when filling sample containers. Avoid immersing the discharge tube into the sample as the sample container is being filled. Sample containers for volatile organic compounds (VOCs) must be completely filled so that no headspace exists in the container. The VOC vials shall be filled to the top so that a convex meniscus is formed. Gently secure the cap, turn the vial upside down, and check to see if any air has been trapped inside the vial. If so, open the cap, reform the meniscus, and attempt again to secure the lid without trapping air in the sample. All other sample containers can have air space included when the container lid is secured.

3.5 Cap each container immediately after filling.

3.6 Record the sample time on the Groundwater Sample Log Sheet, the sample tag, and on the sample label.

3.7 Secure the associated tag to each sample container.

3.8 Place the tagged sample container into a plastic storage bag and then into a cooler containing ice.

3.9 Enter the proper information on the Chain-of-Custody form for each sample container.

3.10 Repeat steps 3.3 through 3.9 for each sample container collected.

3.11 The pump rate should not be adjusted after sampling has commenced. If it becomes necessary to adjust the pump rate, document the change on the Groundwater Sample Log Sheet.

3.12 All samples will be collected into pre-preserved bottles (if required) supplied by an approved laboratory. All samples will be collected in the following sequence (where applicable):

VOCs
Polychlorinated Biphenyls (PCBs)
Total Metals
Dissolved Metals

Total Organic Carbon (TOC)

3.13 If the last turbidity measurement prior to the commencement of sampling showed turbidity to be greater than 5 Nephelometric Turbidity Units (NTUs), then filtered aliquots of ground water will be collected and analyzed for dissolved metals and dissolved thorium isotopes. Without turning off the pump, attach a disposable, inline, 0.45-um filter cartridge at the end of the discharge tube. Fill sample containers marked for "dissolved metals" so that the laboratory knows that these aliquots are distinct sample fractions and that the results should be reported as dissolved analytes. Samples scheduled for VOC analysis shall not be filtered.

3.14 Repeat steps 3.5 through 3.9 for the filtered sample containers.

3.15 After completion of sample collection, remove the bladder pump from well and decontaminate.

3.16 Replace the outer protective well cap and lock the well.

3.17 All equipment should be cleaned and packed into the sample vehicle, along with the sample cooler for transport. Disposable gloves and other equipment should be placed in a plastic trash bag and handled as investigation derived waste.

4.0 ATTACHMENTS

1. Groundwater Sample Log Sheet

ATTACHMENT 1

GROUNDWATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Domestic Well Data
[] Monitoring Well Data
[] Other Well Type: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

Date: / Time:	Color (Visual)	pH (S.U.)	S.C. (mS/cm)	Temp. (°C)	Turbidity (NTU)	DO (mg/l)	Salinity (%)	Other
Method:								

PURGE DATA:

Date:	Volume	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Method:								
Monitor Reading (ppm):								
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:

MS/MSD	Duplicate ID No.:	Signature(s):

STANDARD OPERATING PROCEDURE

SOP-17

CALIBRATION AND CARE OF WATER QUALITY METERS

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for the calibration and maintenance of field instruments used to measure water quality and for the proper documentation of calibration and maintenance. The YSI 600-Series Environmental Monitoring System or the Horiba U20–Series multi-parameter water quality monitoring system will be used to measure pH, temperature, oxidation-reduction potential (ORP), specific conductance (SC), and dissolved oxygen (DO) in water. A LaMotte turbidity meter will be used in conjunction with the water quality meter to measure turbidity. The water quality meter will have a multiprobe sensor that can be used in conjunction with a flow-through cell attached to a pump discharge tube to measure water-quality parameters in a groundwater discharge or can be immersed in a surface water body such as a stream, pond, or drainage ditch. The LaMotte is a hand held meter that uses a multi-detector optical configuration to assure long term stability and minimize stray light and color interferences. All comparable equipment used in place of the equipment items identified in Section 2.0 below must be comparable in terms of sensitivity, accuracy, and precision.

2.0 FIELD FORMS AND EQUIPMENT LIST

The following logbooks, forms, equipment, and supplies are required:

Site logbook
Equipment calibration log sheet
YSI Model 600 Series and Sonde or Horiba U20 Series, or comparable: multi-parameter water-quality meter with flow through cell.
LaMotte Turbidity Meter, or comparable
Equipment manual
Calibration kit
Deionized water, paper towels, spray bottle, etc.
Disposable medical-grade gloves (e.g., latex, nitrile)

3.0 PROCEDURES

This section describes the calibration procedures for the YSI Model 600 series, the Horiba U20 series, and the LaMotte. Each meter is supplied with an instruction manual and will be on-site and will be used as the calibration guidance documents. These procedures will list requirements for frequency of calibration and checks to be performed on the meter.

3.1 YSI Model 600 Series and Horiba U20 Series

The YSI Model 600 series and Sonde and the Horiba U20 series are multi-parameter, water-quality meters that may be used to measure open water bodies (streams, ponds, springs, etc.) with the probe guard installed. With the flow through cell attached, the meters have the ability to measure water-quality parameters in groundwater via a pump discharge line. By performing the measurements in the discharge line coming directly from the well, the parameters are measured before the groundwater comes in contact with the atmosphere. The parameters measured by the YSI or the Horiba for this field effort are as follows:

- DO
- SC
- Temperature
- pH
- ORP
- Turbidity

3.1.1 Documentation

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including the frequency of calibration, type of standards used, and checks performed on calibration during the course of using the equipment. An Equipment Calibration Log must be maintained for each measuring device that requires calibration. Entries must be made for each day the equipment is used. A blank Equipment Calibration Log form is attached at the end of this SOP.

3.1.2 Calibration

All the parameters listed in Section 3.0 must be calibrated prior to the start of each field effort. After this initial calibration, the meter will be checked each day that it is used. If the check shows any out-of-

specification readings, the specific probe will be recalibrated. Meter specifications can be found in the equipment manual, starting on page 248 (YSI) or page 93 (Horiba). Calibration and calibration checks will be documented in the field logbook and on the Equipment Calibration Log. The name, lot number, and expiration date for all calibration buffers and standards used will be recorded on the Equipment Calibration Log. The meter's model, serial number, and name of the rental company will also be recorded on the equipment calibration form.

3.1.3 Tips for Good Calibration

- The DO calibration is a water-saturated air calibration. Make certain to loosen the calibration cup seal to allow pressure to equilibrate before calibrating.

- Make certain that sensors are completely submersed in solution and readings are stable when calibration values are entered.

- Use a small amount of calibration solution (previously used solution may be used, then discarded for this purpose) to pre-rinse the sonde.

- Fill a bucket with ambient temperature water to rinse the sonde between calibration solutions.

- Make sure to rinse and dry the probe between calibration solutions. This will reduce carry-over contamination and increase the accuracy of the calibration.

3.2 Lamotte Turbidity Meter

The Lamotte turbidity meter is a hand held meter that measures the amount of suspended matter in water using the Nephelometric method.

3.2.1 Documentation

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including the frequency of calibration, type of standards used, and checks performed on calibration during the course of using the equipment. An Equipment Calibration Log must be maintained for each measuring device that requires calibration. Entries must be made for each day the equipment is used. A blank Equipment Calibration Log form is attached at the end of this SOP.

3.2.2 Calibration

Turbidity must be calibrated prior to the start of each field effort. After this initial calibration, the LaMotte will be calibrated each day that it is used. If the check shows any out-of-specification readings, the meter will be recalibrated. Meter specifications can be found in the equipment manual. Calibration and calibration checks will be documented in the field logbook and on the Equipment Calibration Log. The name, lot number, and expiration date for all calibration standards used will be recorded on the Equipment Calibration Log. The meter's model, serial number, and name of the rental company will also be recorded on the equipment calibration form.

3.2.3 Tips for Good Calibration

- Thoroughly clean the standard vial with a chem wipe to remove finger prints.
- Make sure that the vial is properly aligned according the manual recommendations.

4.0 MAINTENANCE

The YSI and/or Horiba Meter and LaMotte will be rented for the duration of each brief field effort. Therefore, little field maintenance will be required. For any maintenance other than the routine cleaning, calibrating, or battery charging, the instrument should be returned to the vendor and a replacement sent immediately to the job site.

4.1 Meter Storage for the YSI and Horiba

For this field effort, the meter storage will be short term, [i.e. over night or between work shifts (4-day break)]. During these breaks, the meter will be charged. One-half inch of tap or distilled water will be placed in the meter calibration cup and the cup threaded onto the sonde. The key for short-term storage of probes is to use a minimal amount of water so the calibration cup will remain at 100 percent humidity. The water level must be low enough so that none of the probes are actually immersed. Proper storage of the sonde between usage will extend its life and will also ensure that the unit is ready for use as quickly as possible for the next application.

Multi-parameter short term storage key points:

- Use enough water to provide humidity but not enough to cover the probe surfaces.
- Make sure the storage vessel is sealed to minimize evaporation.
- Check periodically to make certain that water is still present.

4.2 Probe Cleaning

- Rinse the probe thoroughly with potable water.
- Wash the probe in a mild solution of Liquinox and water and wipe with paper towels and/or cotton swabs.
- Rinse and soak the probe in deionized water.
- If stronger cleaning is required, consult Section 2.10 on page 89 (YSI) or Section 7.1 on page 86 (Horiba) of the equipment manual.

Note: Reagents that are used to calibrate and check the water quality meter may be hazardous. Review the health and safety plan and Material Safety Data Sheets (MSDSs), all of which are on file in the field trailer.

4.3 Meter Storage for the LaMotte

For this field effort, the meter storage will be short term, [i.e. over night or between work shifts (4-day break)]. Proper storage of the meter between usage will extend its life and will also ensure that the unit is ready for use as quickly as possible for the next application.

Short term storage key points:

- Make sure the storage vessel is moisture free and sealed.

4.4 Sample Vial Cleaning

- Rinse the vial thoroughly with potable water to remove sediments.
- Wipe with chem-wipes or cotton swabs.

5.0 ATTACHMENTS

1. Equipment Calibration Log

ATTACHMENT 1

EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

APPENDIX C

LABORATORY STANDARD OPERATING PROCEDURES AND CERTIFICATIONS

SOP NUMBER	REVISION	TITLE
100	R20 20100427	Metals Digestion/Preparation
105	R16 20100411	Metals By Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) Technique
164	R15 20100325	Empirical Laboratories, LLC Standard Operating Procedure Distillation of Aqueous/Solid Samples for cyanide, total and amenable
175	R10 20091215	Post-Distillation Analysis For Cyanide Using Lachat Flow Injection Analyzer
202	R22 20090930	GC/MS Volatiles By EPA Method E624 & SW846 Method 8260B Including Appendix IX Compounds
211	R22 20100707	Gas Chromatography/Electron Capture Detector (GC/ECD) Organochlorine Pesticides/Polychlorinated Biphenyls (PCBs)
225	R08 20080924	GC/MS Volatile Non-Aqueous Matrix Extraction Using Sw-846 Method 5035 For 8260B Analysis
302	R17 20100426	Pesticide/PCBs Aqueous Matrix Extraction For EPA Method 608/608.2 And SW846 Method 8081/8082 Using SW846 Method 3510C
404	R13 20090629	Laboratory Sample Receiving Log In and Storage Standard Operating Procedures
405	R05 20090623	Analytical Laboratory Waste Disposal
410	R07 20090623	Analytical Laboratory Waste Disposal
239	R2 20090721	Standard Operating Procedure Perchlorate in water, soil, and solid waste using high performance liquid chromatography/electrospray ionization/mass spectrometry
343	080109 REV0	BNA and Pesticide/PCBs and TPH Non aqueous Matrix (Microwave Extraction) using SW-846 Method 3546

LABORATORY ACCREDITATION BUREAU

Certificate of Accreditation

ISO/IEC 17025:2005 ***Certificate Number L2226***

Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: November 30, 2012

R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 30th of November 2009

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements
Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2226

Scope of Accreditation For Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228
Marcia K. McGinnity
1-877-345-1113

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Empirical Laboratories, LLC to perform the following tests:

Accreditation granted through: **November 30, 2012**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	8260B	1,1,2-Trichloroethane
GC/MS	8260B	1,1,2,2-Tetrachloroethane
GC/MS	8260B	1,1,1,2-Tetrachloroethane
GC/MS	8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	8260B	1,2,3-Trichlorobenzene
GC/MS	8260B	1,2,4-Trichlorobenzene
GC/MS	8260B	1,2,3-Trichloropropane
GC/MS	8260B	1,2,4-Trimethylbenzene
GC/MS	8260B	1,3,5-Trimethylbenzene
GC/MS	8260B	1,2-Dibromoethane (EDB)
GC/MS	8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	8260B	1,2-Dichlorobenzene
GC/MS	8260B	1,2-Dichloroethane (EDC)
GC/MS	8260B	1,2-Dichloropropane
GC/MS	8260B	1,3-Dichlorobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B	1,4-Dichlorobenzene
GC/MS	8260B	1,1-Dichloropropene
GC/MS	8260B	1,3-Dichloropropane
GC/MS	8260B	2,2-Dichloropropane
GC/MS	8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	8260B	Acetone
GC/MS	8260B	Benzene
GC/MS	8260B	Bromochloromethane
GC/MS	8260B	Bromodichloromethane
GC/MS	8260B	Bromobenzene
GC/MS	8260B	Bromoform
GC/MS	8260B	Bromomethane
GC/MS	8260B	n-Butylbenzene
GC/MS	8260B	sec-Butylbenzene
GC/MS	8260B	tert-Butylbenzene
GC/MS	8260B	Carbon Disulfide
GC/MS	8260B	Carbon Tetrachloride
GC/MS	8260B	Chlorobenzene
GC/MS	8260B	Chloroethane
GC/MS	8260B	Chloroform
GC/MS	8260B	Chloromethane
GC/MS	8260B	2-Chlorotoluene
GC/MS	8260B	4-Chlorotoluene
GC/MS	8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	8260B	cis-1,3-Dichloropropene
GC/MS	8260B	Cyclohexane
GC/MS	8260B	Dibromochloromethane
GC/MS	8260B	Dibromomethane
GC/MS	8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	8260B	Ethylbenzene
GC/MS	8260B	Hexachlorobutadiene
GC/MS	8260B	Isopropylbenzene (Cumene)
GC/MS	8260B	p-Isopropyltoluene
GC/MS	8260B	Methyl Acetate
GC/MS	8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	8260B	Methylcyclohexane
GC/MS	8260B	Methylene Chloride, or Dichloromethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B	Naphthalene
GC/MS	8260B	n-Propylbenzene
GC/MS	8260B	Styrene
GC/MS	8260B	Tetrachloroethene (PCE; PERC)
GC/MS	8260B	Toluene
GC/MS	8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	8260B	trans-1,3-Dichloropropene
GC/MS	8260B	Trichloroethene (TCE)
GC/MS	8260B	Trichlorofluoromethane (CFC-11)
GC/MS	8260B	Vinyl Chloride (VC)
GC/MS	8260B	Xylenes (Total)
GC/MS	8260B	Acrolein
GC/MS	8260B	Acrylonitrile
GC/MS	8260B	Di-isopropyl ether
GC/MS	8260B	ETBE
GC/MS	8260B	Ethyl methacrylate
GC/MS	8260B	Iodomethane
GC/MS	8260B	Methyl methacrylate
GC/MS	8260B	t-Butyl alcohol
GC/MS	8260B	tert-Amyl methyl ether
GC/MS	8260B	2-Chloroethyl vinyl ether
GC/MS	8260B	1-Chlorohexane
GC/MS	8260B	Tetrahydrofuran
GC/MS	8260B	Vinyl acetate
GC/MS	8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	8270C/D	1,2-Dichlorobenzene
GC/MS	8270C/D	1,3-Dichlorobenzene
GC/MS	8270C/D	1,4-Dichlorobenzene
GC/MS	8270C/D	2,4,5-Trichlorophenol
GC/MS	8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	8270C/D	2,4-Dimethylphenol
GC/MS	8270C/D	2,4-Dinitrophenol
GC/MS	8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	8270C/D	2,6-Dichlorophenol
GC/MS	8270C/D	2,6-Dinitrotoluene
GC/MS	8270C/D	1,2-Diphenylhydrazine
GC/MS	8270C/D	2-Chloronaphthalene
GC/MS	8270C/D	2-Chlorophenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C/D	2-Methylnaphthalene
GC/MS	8270C/D	2-Methylphenol (o-Cresol)
GC/MS	8270C/D	2-Nitroaniline
GC/MS	8270C/D	2-Nitrophenol (ONP)
GC/MS	8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	8270C/D	3-Methylphenol
GC/MS	8270C/D	3-Nitroaniline
GC/MS	8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	8270C/D	4-Bromophenyl phenyl ether
GC/MS	8270C/D	4-Chloro-3-methylphenol
GC/MS	8270C/D	4-Chloroaniline
GC/MS	8270C/D	4-Chlorophenyl phenyl ether
GC/MS	8270C/D	4-Methylphenol (p-Cresol)
GC/MS	8270C/D	4-Nitroaniline (PNA)
GC/MS	8270C/D	4-Nitrophenol (PNP)
GC/MS	8270C/D	Acenaphthene
GC/MS	8270C/D	Acenaphthylene
GC/MS	8270C/D	Acetaphenone
GC/MS	8270C/D	Anthracene
GC/MS	8270C/D	Benzo(a)anthracene
GC/MS	8270C/D	Benzo(a)pyrene
GC/MS	8270C/D	Benzo(b)fluoranthene
GC/MS	8270C/D	Benzo(g,h,i)perylene
GC/MS	8270C/D	Benzo(k)fluoranthene
GC/MS	8270C/D	Benzyl alcohol
GC/MS	8270C/D	Benzoic Acid
GC/MS	8270C/D	bis(2-Chloroethoxy)methane
GC/MS	8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	8270C/D	Carbazole
GC/MS	8270C/D	Chrysene
GC/MS	8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	8270C/D	Dibenz(a,h)anthracene
GC/MS	8270C/D	Dibenzofuran (DBF)
GC/MS	8270C/D	Diethyl phthalate (DEP)
GC/MS	8270C/D	Dimethyl phthalate (DMP)
GC/MS	8270C/D	Fluoranthene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C/D	Fluorene
GC/MS	8270C/D	Hexachlorobenzene (HCB)
GC/MS	8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	8270C/D	Hexachloroethane (HCE)
GC/MS	8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	8270C/D	Isophorone
GC/MS	8270C/D	N-Nitrosodimethylamine
GC/MS	8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	8270C/D	Naphthalene
GC/MS	8270C/D	Nitrobenzene
GC/MS	8270C/D	Pentachlorophenol
GC/MS	8270C/D	Phenanthrene
GC/MS	8270C/D	Phenol
GC/MS	8270C/D	Pyrene
GC/MS	8270C/D	Pyridine
GC/MS	8270C/D	1,2,4-Trichlorobenzene
GC/MS	8270C/D	1,1'-Biphenyl
GC/MS	8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	8270C/D	1,4-Dioxane
GC/MS	8270C/D	1-Methylnaphthalene
GC/MS	8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	8270C/D	Aniline
GC/MS	8270C/D	Atrazine
GC/MS	8270C/D	Benzaldehyde
GC/MS	8270C/D	Benzidine
GC/MS	8270C/D	7,12-Dimethylbenz(a)anthracene
GC/MS	8270C/D	Caprolactam
GC/ECD	8081A/B	4,4'-DDD
GC/ECD	8081A/B	4,4'-DDE
GC/ECD	8081A/B	4,4'-DDT
GC/ECD	8081A/B	Aldrin
GC/ECD	8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	8081A/B	alpha-Chlordane
GC/ECD	8081A/B	beta-BHC (beta-HCH)
GC/ECD	8081A/B	delta-BHC (delta-HCH)
GC/ECD	8081A/B	Dieldrin
GC/ECD	8081A/B	Endosulfan I

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8081A/B	Endosulfan II
GC/ECD	8081A/B	Endosulfan sulfate
GC/ECD	8081A/B	Endrin
GC/ECD	8081A/B	Endrin aldehyde
GC/ECD	8081A/B	Endrin ketone
GC/ECD	8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	8081A/B	gamma-Chlordane
GC/ECD	8081A/B	Heptachlor
GC/ECD	8081A/B	Heptachlor epoxide
GC/ECD	8081A/B	Methoxychlor
GC/ECD	8081A/B	Chlordane
GC/ECD	8081A/B	Toxaphene
GC/ECD	8082 /A	Aroclor-1016
GC/ECD	8082 /A	Aroclor-1221
GC/ECD	8082 /A	Aroclor-1232
GC/ECD	8082 /A	Aroclor-1242
GC/ECD	8082 /A	Aroclor-1248
GC/ECD	8082 /A	Aroclor-1254
GC/ECD	8082 /A	Aroclor-1260
GC/ECD	8151A	2,4,5-T
GC/ECD	8151A	2,4,5-TP (Silvex)
GC/ECD	8151A	2,4-D
GC/ECD	8151A	2,4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichlorprop
GC/ECD	8151A	Dinoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP (Mecoprop)
HPLC/UV	8330A	1,3,5-Trinitrobenzene
HPLC/UV	8330A	1,3-Dinitrobenzene
HPLC/UV	8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	8330A	2,6-Dinitrotoluene
HPLC/UV	8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	8330A	2-Nitrotoluene (ONT)
HPLC/UV	8330A	3-Nitrotoluene
HPLC/UV	8330A	4-Amino-2,6-dinitrotoluene

Non-Potable Water		
Technology	**Method**	**Analyte**
HPLC/UV	8330A	4-Nitrotoluene (PNT)
HPLC/UV	8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	8330A	Nitroglycerin
HPLC/UV	8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	8330A	3,5-Dinitroaniline
HPLC/UV	8330A	Nitrobenzene
HPLC/UV	8330A	PETN
HPLC/UV	8330A	Nitroguanidine
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	8015B	TPH DRO
GC/FID	8015B	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	8011	1,2-Dibromoethane (EDB)
GC/ECD	8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	6850	Perchlorate
ICP	6010B/C	Aluminum
ICP	6010B/C	Antimony
ICP	6010B/C	Arsenic
ICP	6010B/C	Barium
ICP	6010B/C	Beryllium
ICP	6010B/C	Cadmium
ICP	6010B/C	Calcium
ICP	6010B/C	Chromium, total
ICP	6010B/C	Cobalt
ICP	6010B/C	Copper
ICP	6010B/C	Iron
ICP	6010B/C	Lead
ICP	6010B/C	Magnesium
ICP	6010B/C	Manganese
CVAA	7470A	Mercury
ICP	6010B/C	Nickel
ICP	6010B/C	Potassium
ICP	6010B/C	Selenium
ICP	6010B/C	Silver
ICP	6010B/C	Sodium
ICP	6010B/C	Thallium
ICP	6010B/C	Vanadium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP	6010B/C	Zinc
ICP	6010B/C	Molybdenum
ICP	6010B/C	Tin
ICP	6010B/C	Strontium
ICP	6010B/C	Titanium
IC	300.0	Chloride
IC	300.0	Fluoride
IC	300.0	Nitrate
IC	300.0	Nitrite
IC	300.0	Sulfate
IC	9056A	Chloride
IC	9056A	Fluoride
IC	9056A	Nitrate
IC	9056A	Nitrite
IC	9056A	Sulfate
Titration	SM 2320B 20th ed.	Alkalinity
ISE	SM 4500 B, D, 20th ed.	Ammonia
UV/Vis	7196A	Hexavalent Chromium
Colorimetric	353.2	Nitrocellulose
Colorimetric	353.2	Nitrate/Nitrite
Titration	Chap.7, Sect. 7.3 Mod.	Reactive Sulfide
Titration	SM 4500 S-2CF, 20th edition	Sulfide
UV/Vis	SM 4500 P B5, E, 20th edition	Total Phosphorus (as P)
UV/Vis	SM 4500 PE, 20th edition	Ortho-Phosphate (as P)
TOC	9060A/SM5310C, 20th edition	Total Organic Carbon
Gravimetric	SM 2540C, 20th edition	TDS
Colorimetric	9012A/B	Cyanide
Physical	1010A	Ignitability
Physical	9095B	Paint Filter
Probe	9040B/C	pH
Preparation	**Method**	**Type**
Preparation	1311	TCLP
Preparation	3005A	Metals digestion
Preparation	3010A	Metals digestion
Preparation	3510C	Organics Liquid Extraction
Preparation	5030A/B	Purge and Trap Water

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	8260B	1,1,2-Trichloroethane
GC/MS	8260B	1,1,2,2-Tetrachloroethane
GC/MS	8260B	1,1,1,2-Tetrachloroethane
GC/MS	8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	8260B	1,2,3-Trichlorobenzene
GC/MS	8260B	1,2,4-Trichlorobenzene
GC/MS	8260B	1,2,3-Trichloropropane
GC/MS	8260B	1,2,4-Trimethylbenzene
GC/MS	8260B	1,3,5-Trimethylbenzene
GC/MS	8260B	1,2-Dibromoethane (EDB)
GC/MS	8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	8260B	1,2-Dichlorobenzene
GC/MS	8260B	1,2-Dichloroethane (EDC)
GC/MS	8260B	1,2-Dichloropropane
GC/MS	8260B	1,3-Dichlorobenzene
GC/MS	8260B	1,4-Dichlorobenzene
GC/MS	8260B	1,1-Dichloropropene
GC/MS	8260B	1,3-Dichloropropane
GC/MS	8260B	2,2-Dichloropropane
GC/MS	8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	8260B	Acetone
GC/MS	8260B	Benzene
GC/MS	8260B	Bromochloromethane
GC/MS	8260B	Bromodichloromethane
GC/MS	8260B	Bromobenzene
GC/MS	8260B	Bromoform
GC/MS	8260B	Bromomethane
GC/MS	8260B	n-Butylbenzene
GC/MS	8260B	sec-Butylbenzene
GC/MS	8260B	tert-Butylbenzene
GC/MS	8260B	Carbon Disulfide
GC/MS	8260B	Carbon Tetrachloride
GC/MS	8260B	Chlorobenzene
GC/MS	8260B	Chloroethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	8260B	Chloroform
GC/MS	8260B	Chloromethane
GC/MS	8260B	2-Chlorotoluene
GC/MS	8260B	4-Chlorotoluene
GC/MS	8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	8260B	cis-1,3-Dichloropropene
GC/MS	8260B	Cyclohexane
GC/MS	8260B	Dibromochloromethane
GC/MS	8260B	Dibromomethane
GC/MS	8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	8260B	Ethylbenzene
GC/MS	8260B	Hexachlorobutadiene
GC/MS	8260B	Isopropylbenzene (Cumene)
GC/MS	8260B	p-Isopropyltoluene
GC/MS	8260B	Methyl Acetate
GC/MS	8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	8260B	Methylcyclohexane
GC/MS	8260B	Methylene Chloride, or Dichloromethane
GC/MS	8260B	Naphthalene
GC/MS	8260B	n-Propylbenzene
GC/MS	8260B	Styrene
GC/MS	8260B	Tetrachloroethene (PCE; PERC)
GC/MS	8260B	Toluene
GC/MS	8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	8260B	trans-1,3-Dichloropropene
GC/MS	8260B	Trichloroethene (TCE)
GC/MS	8260B	Trichlorofluoromethane (CFC-11)
GC/MS	8260B	Vinyl Chloride (VC)
GC/MS	8260B	Xylenes (Total)
GC/MS	8260B	Acrolein
GC/MS	8260B	Acrylonitrile
GC/MS	8260B	Ethyl methacrylate
GC/MS	8260B	Iodomethane
GC/MS	8260B	Methyl methacrylate
GC/MS	8260B	Vinyl acetate
GC/MS	8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	8270C/D	1,2-Dichlorobenzene
GC/MS	8270C/D	1,3-Dichlorobenzene
GC/MS	8270C/D	1,4-Dichlorobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	8270C/D	2,4,5-Trichlorophenol
GC/MS	8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	8270C/D	2,4-Dimethylphenol
GC/MS	8270C/D	2,4-Dinitrophenol
GC/MS	8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	8270C/D	2,6-Dichlorophenol
GC/MS	8270C/D	2,6-Dinitrotoluene
GC/MS	8270C/D	1,2-Diphenylhydrazine
GC/MS	8270C/D	2-Chloronaphthalene
GC/MS	8270C/D	2-Chlorophenol
GC/MS	8270C/D	2-Methylnaphthalene
GC/MS	8270C/D	2-Methylphenol (o-Cresol)
GC/MS	8270C/D	2-Nitroaniline
GC/MS	8270C/D	2-Nitrophenol (ONP)
GC/MS	8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	8270C/D	3-Methylphenol
GC/MS	8270C/D	3-Nitroaniline
GC/MS	8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	8270C/D	4-Bromophenyl phenyl ether
GC/MS	8270C/D	4-Chloro-3-methylphenol
GC/MS	8270C/D	4-Chloroaniline
GC/MS	8270C/D	4-Chlorophenyl phenyl ether
GC/MS	8270C/D	4-Methylphenol (p-Cresol)
GC/MS	8270C/D	4-Nitroaniline (PNA)
GC/MS	8270C/D	4-Nitrophenol (PNP)
GC/MS	8270C/D	Acenaphthene
GC/MS	8270C/D	Acenaphthylene
GC/MS	8270C/D	Acetaphenone
GC/MS	8270C/D	Anthracene
GC/MS	8270C/D	Benzo(a)anthracene
GC/MS	8270C/D	Benzo(a)pyrene
GC/MS	8270C/D	Benzo(b)fluoranthene
GC/MS	8270C/D	Benzo(g,h,i)perylene
GC/MS	8270C/D	Benzo(k)fluoranthene
GC/MS	8270C/D	Benzyl alcohol
GC/MS	8270C/D	Benzoic Acid
GC/MS	8270C/D	bis(2-Chloroethoxy)methane
GC/MS	8270C/D	bis(2-Chloroethyl)ether (BCEE)

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	8270C/D	Carbazole
GC/MS	8270C/D	Chrysene
GC/MS	8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	8270C/D	Dibenz(a,h)anthracene
GC/MS	8270C/D	Dibenzofuran (DBF)
GC/MS	8270C/D	Diethyl phthalate (DEP)
GC/MS	8270C/D	Dimethyl phthalate (DMP)
GC/MS	8270C/D	Fluoranthene
GC/MS	8270C/D	Fluorene
GC/MS	8270C/D	Hexachlorobenzene (HCB)
GC/MS	8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	8270C/D	Hexachloroethane (HCE)
GC/MS	8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	8270C/D	Isophorone
GC/MS	8270C/D	N-Nitrosodimethylamine
GC/MS	8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	8270C/D	Naphthalene
GC/MS	8270C/D	Nitrobenzene
GC/MS	8270C/D	Pentachlorophenol
GC/MS	8270C/D	Phenanthrene
GC/MS	8270C/D	Phenol
GC/MS	8270C/D	Pyrene
GC/MS	8270C/D	Pyridine
GC/MS	8270C/D	1,2,4-Trichlorobenzene
GC/MS	8270C/D	1,1'-Biphenyl
GC/MS	8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	8270C/D	1,4-Dioxane
GC/MS	8270C/D	1-Methylnaphthalene
GC/MS	8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	8270C/D	Aniline
GC/MS	8270C/D	Atrazine
GC/MS	8270C/D	Benzaldehyde
GC/MS	8270C/D	Benzidine
GC/MS	8270C/D	Benzo(a)anthracene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	8270C/D	Caprolactam
GC/ECD	8081A/B	4,4'-DDD
GC/ECD	8081A/B	4,4'-DDE
GC/ECD	8081A/B	4,4'-DDT
GC/ECD	8081A/B	Aldrin
GC/ECD	8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	8081A/B	alpha-Chlordane
GC/ECD	8081A/B	beta-BHC (beta-HCH)
GC/ECD	8081A/B	delta-BHC (delta-HCH)
GC/ECD	8081A/B	Dieldrin
GC/ECD	8081A/B	Endosulfan I
GC/ECD	8081A/B	Endosulfan II
GC/ECD	8081A/B	Endosulfan sulfate
GC/ECD	8081A/B	Endrin
GC/ECD	8081A/B	Endrin aldehyde
GC/ECD	8081A/B	Endrin ketone
GC/ECD	8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	8081A/B	gamma-Chlordane
GC/ECD	8081A/B	Heptachlor
GC/ECD	8081A/B	Heptachlor epoxide
GC/ECD	8081A/B	Methoxychlor
GC/ECD	8081A/B	Chlordane
GC/ECD	8081A/B	Toxaphene
GC/ECD	8082 /A	Aroclor-1016
GC/ECD	8082 /A	Aroclor-1221
GC/ECD	8082 /A	Aroclor-1232
GC/ECD	8082 /A	Aroclor-1242
GC/ECD	8082 /A	Aroclor-1248
GC/ECD	8082 /A	Aroclor-1254
GC/ECD	8082 /A	Aroclor-1260
GC/ECD	8151A	2,4,5-T
GC/ECD	8151A	2,4,5-TP (Silvex)
GC/ECD	8151A	2,4-D
GC/ECD	8151A	2,4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichlorprop
GC/ECD	8151A	Dinoseb
GC/ECD	8151A	MCPA

Solid and Chemical Materials

Technology	Method	Analyte
GC/ECD	8151A	MCPP (Mecoprop)
HPLC/UV	8330A	1,3,5-Trinitrobenzene
HPLC/UV	8330A	1,3-Dinitrobenzene
HPLC/UV	8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	8330A	2,6-Dinitrotoluene
HPLC/UV	8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	8330A	2-Nitrotoluene (ONT)
HPLC/UV	8330A	3-Nitrotoluene
HPLC/UV	8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	8330A	4-Nitrotoluene (PNT)
HPLC/UV	8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	8330A	Nitroglycerin
HPLC/UV	8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	8330A	Nitrobenzene
HPLC/UV	8330A	PETN
HPLC/UV	8330A	Nitroguanidine
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	8015B	TPH DRO
GC/FID	8015B	TPH GRO
HPLC/MS	6850	Perchlorate
ICP	6010B/C	Aluminum
ICP	6010B/C	Antimony
ICP	6010B/C	Arsenic
ICP	6010B/C	Barium
ICP	6010B/C	Beryllium
ICP	6010B/C	Cadmium
ICP	6010B/C	Calcium
ICP	6010B/C	Chromium, total
ICP	6010B/C	Cobalt
ICP	6010B/C	Copper
ICP	6010B/C	Iron
ICP	6010B/C	Lead
ICP	6010B/C	Magnesium
ICP	6010B/C	Manganese
CVAA	7471A/B	Mercury
ICP	6010B/C	Nickel
ICP	6010B/C	Potassium

LABORATORY ACCREDITATION BUREAU

Certificate # L2226

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	6010B/C	Selenium
ICP	6010B/C	Silver
ICP	6010B/C	Sodium
ICP	6010B/C	Thallium
ICP	6010B/C	Vanadium
ICP	6010B/C	Zinc
ICP	6010B/C	Molybdenum
ICP	6010B/C	Tin
ICP	6010B/C	Strontium
ICP	6010B/C	Titanium
UV/Vis	7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	353.2	Nitrocellulose
Colorimetric	9012A/B	Cyanide
Titration	Chap.7, Sect. 7.3 Mod.	Reactive Sulfide
Titration	9034	Sulfide
Probe	9045D	pH
Preparation	**Method**	**Type**
Preparation	1311	TCLP
Preparation	1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	3050B	Metals Digestion
Preparation	3546	Organics Microwave Extraction
Preparation	3541	Organics Soxhlet Extraction
Preparation	3550B	Organics Sonication
Preparation	SM 2540B 20th edition	Percent Solids (Percent Moisture)
Preparation	5035 /A	Purge and Trap Solid

Notes:

1) This laboratory offers commercial testing service.

Approved By: _____ Date: March 31, 2010

R. Douglas Leonard
Chief Technical Officer

Issued: 11/30/09 Revised: 2/9/10 Revised: 3/31/10

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP100 REVISION #: 20 EFFECTIVE DATE: 042710

METALS DIGESTION/PREPARATION

References:
Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)

APPROVALS:

Lab Director: _____ Date: 4/28/10

Data Quality Manager: _____ Date: 4/28/10

Section Supervisor: _____ Date: 4/28/10

Changes Summary

Revision 20, 4/27/10

- The SOP is an update from Revision 19 dated 04/20/09.
- References to oil sample preparation have been removed.
- Extraction volumes for TCLP have been updated.

METALS DIGESTION/PREPARATION

References:
Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)

I. SCOPE AND APPLICATION

A. AQUEOUS

1. Method 3005A and USEPA CLP ILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
   a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry"
   a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
   a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.
4. Method 3030C (Standard methods), "Preliminary Treatment for Acid-Extractable Metals".
   a. This method is used to prepare ground water samples from North Carolina for analysis by ICP.

B. SOLIDS

1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".
   a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.
   b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.
2. USEPA CLP ILM0 4.1, “Acid Digestion of Soil/Sediment”
   a. This method is used to prepare sediments and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

D. **NOTES:**

1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.
2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion.

II. SUMMARY OF METHODS

A. A representative sample of water or soil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower than is customary, a four times concentration or alternate soil digestion ratio must be used in order to reach that lower level. Care must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike (not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

III. SAMPLE HANDLING AND PRESERVATION

A. AQUEOUS
   1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated HNO_3 to a pH <2 immediately once sampled. If dissolved metals are to be analyzed the sample should be filtered before the HNO_3 is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

B. SOLIDS
   1. Samples are taken in high density polyethylene(CLP only) or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

IV. INTERFERENCES

A. AQUEOUS
   1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

B. SOLIDS
   1. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

V. SAFETY

A. Normal accepted laboratory safety practices should be followed while performing this analysis.
B. Be certain the exhaust hood is functioning before you begin the digestion procedure.
C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

VI. EQUIPMENT/APPARATUS

A. Fume hood, Labconco or equivalent.
B. Hot plate, Thermolyne cimarec-3 or equivalent source for use at 95°C. The temperature of the hot plate must be monitored via the use of a temperature blank.
C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.

D. Vacuum pump for filtering dissolved metals- Gast or equivalent.
E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.
F. Beckman CS-6R centrifuge.
G. Various class A volumetric glassware and ribbed watchglasses, Pyrex or equivalent.
H. Whatman No. 41 filter paper or equivalent.
I. Whatman No. 42 filter paper or equivalent.
J. Whatman 0.45 micron filter paper or equivalent.
K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes, watch glasses and caps.
L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.
M. Manual Sample Mill
N. Wiley Sample Mill
O. Clippers for cutting vegetation

NOTE: All glassware should be acid washed.

VII. REAGENTS AND STANDARD PREPARATION

A. REAGENTS

1. Metals grade Nitric acid (HNO_3). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
4. Metals grade Sulfuric acid (H_2SO_4). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
5. Reagent water (Deionized water).
6. Potassium Permanganate - Ultra pure grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
7. Ammonium hydroxide, concentrated, reagent grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
8. Ammonium phosphate, reagent grade- Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

B. STANDARDS

1. Traceability

a. A LIMS record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label.

b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the information is recorded in LIMS. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in LIMS. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out and initial/date the unused area of each logbook page.

2. PREPARATION

A. Laboratory control sample

1. Aqueous

a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL (100 mL for strict CLPIlM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample digestion logbook/LIMS.

b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO_3 and 5% HCl. Use 0.125 mLs HNO_3 and 0.3125 mLs HCl to each 50 mL vessel.

2. Solids:

a. 1.0 ±0.02 (or 2.0 ±0.02) gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. LCSS(date)A and then B etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and the weight is recorded in a bound logbood and transfer to LIMS.

B. Spiking solution

1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.

2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.

3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less then 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

VIII. CALIBRATION

A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. Record on LIMS batch sheet for later transfer into LIMS.

IX. PROCEDURE

A. Glassware preparation for digestion or when the hot-block can not be used:
   1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)
   2. Rinse glassware with reagent water that contains 5% HNO_3 and 5% HCl followed by a rinse with reagent water.
   3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.

B. Aqueous sample filtration (for dissolved metals):
   1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO_3 followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
   2. Rinse a 0.45 micron filter with 1:5 HNO_3 thoroughly, followed by D.I. water.
   3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
   4. Discard the first 50 to 100 mL.
   5. A preparation blank must be taken through the filtration step and analyzed with the sample.
   6. Preserve the sample with HNO_3 to pH<2.
   7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO_3. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**

C. Aqueous sample preparation
   1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP ".**
      a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
      b. Add 0.50 mL (1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO_3 to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO_3 to the sample.
      c. Add 2.5 mL (5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.
      d. Cover the sample with a ribbed watch glass or equivalent source.
      e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the digestion log book. Take the volume down to between 5 to 10 mL, (

12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool

f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
g. Bring sample to its predigestion volume (or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the digestion log book.
h. The sample is now ready for analysis.
i. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.

2 Method 200.7, "**Acid digestion procedure for total recoverable metals**".

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.
b. Add 1.0 mL concentrated HNO_3 to the sample.
c. Add 2.50 mL concentrated HCl to the sample.
d. Cover the sample with a ribbed watch glass or equivalent source.
e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes.**
f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.
g. Bring sample to its predigestion volume with DI water in the digestion vessel.
h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
i. The sample is now ready for analysis.
j. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".

a. Shake sample thoroughly and pour 50 mL (5ml diluted to 50mL for TCLP, full 50ml volume for SPLP) of the well-mixed sample into the digestion vessel.
b. Add 1.5 mL concentrated HNO_3 to the sample.
c. Cover the sample with a ribbed watch glass.
d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature being recorded in the log book. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the**

bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries. Remove the sample from the hot plate and cool.

e. Add another 1.5 mL portion of concentrated HNO_3 to the sample.
f. Cover the sample with a ribbed watch glass.
g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).
h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.
i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).
j. Cover the digestion vessel and reflux for an additional 15 minutes.
k. Bring sample to its predigestion volume in digestion vessel.
l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

   Note: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

m. The sample is now ready for analysis.
n. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals"**

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.
b. Add 2.5 mL 1:1 HCl to the sample.
c. Heat 15 minutes in a hot bath.
d. Filter through a membrane filter.
e. Adjust filtrate volume to 50 mL with DI water.
f. Transfer to ICP analyst.

D. Solid sample preparation

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- *The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

Grinding of Vegetation Samples

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, **"Acid digestion of Soil/Sediment"**
   a. Mix the sample thoroughly to achieve homogenity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.
   b. Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated HNO_3, replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.
   c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.
   d. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2.)
   e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivelent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated HNO_3, mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v) HNO_3. The sample is now ready for analysis.
   f. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.
2. Method 3050B, **"Acid digestion of Sediments, Sludges and Soils"**

a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.
b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the digestion log.

   NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.
c. Add 5 mL D.I. water and 5 mL concentrated HNO_3(1:1), mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at 95°C for 10 to 15 minutes being certain that the sample does not boil. Record temperature in digestion log book
d. Allow the sample to cool. Add 5 mL concentrated HNO_3, replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) over and over until <u>no</u> brown fumes are given off by the sample indicating the complete reaction with HNO_3. Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at 95°C $\pm$ 5°C for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.
e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of H_2O_2 if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H_2O_2 to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2.)
f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at 95°C $\pm$ 5°C without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.
g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling
h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.

NOTE1: When preparing DoD project samples, if any sample in a digestion batch requires filtration , all samples (including QC samples) must be treated in the same manner.

NOTE2: To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.

j. The sample is now ready for analysis.

k. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

X. CALCULATIONS

A. The analyst must be supplied with both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the digestion log.

XI. QUALITY CONTROL

A. Digestion

1. Temperature blank
   a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.
   b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.
2. Blanks
   a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples.
   b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.
   c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
   d. Sample is given a unique identifier in the digestion log.
3. Laboratory Control Samples
   a. For water samples, one LCS is digested with every batch of samples digested (20 sample maximum).
   b. For water samples, a LCS is digested every day for each type of digestion, every 20 samples.
   c. For soil/sediment samples, a soil matrix standard reference material (SRM) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.
   d. Sample is given a unique identifier in the digestion log.
4. Duplicates
   a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.

      NOTE: Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.
5. Blank Spike
   a. This is required for certain projects.

B. Sample Matrix

NOTE: Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

1. Matrix spike
   a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

      NOTE: For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.

   b. The following metals do not get digested spikes when using CLP spike.
      Calcium
      Magnesium
      Sodium
      Potassium
   c. For TCLP samples, a spike must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquot must be added to the extract after filtration but before preservation.)
   d. The CLH project requires that a high and a low spike be prepared and analyzed. Spikes shoud be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.

XII. CORRECTIVE ACTIONS

A. Sample boils during digestion.
   1. Redigest another sample aliquot.

B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.
   1. Redigest another sample aliquot.
   2. Glass beaker dry for an extended period of time? Discard beaker.

XIII. SPECIAL NOTES

A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.

B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.

C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.

D. There are several precautions that must be taken to minimize the possibility of contamination.
   1. All metals glassware must be kept separate from all other laboratory glassware.

2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.

E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.

F. Please consult Waste Disposal SOP-QS14, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

Addendum for USEPA CLPILM 05.2 AQUEOUS & SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. Soluble samples are required to be digested unless the chain of custody specifically states that digestion is not required. An MDL study must be done on the unprepared MDL solution in order to provide MDL levels for samples that are not digested. When digestion is not required an LCSW and post digestion spike are not required.
2. Digestates must be stored until 365 days after delivery of a complete, reconciled data package.
3. Preparation codes are used on form 13's. They are found in the 5.2 statement of work page B-39 3.4.12.2.4.

DEFINITIONS – Refer to SOP-QS08 for common environmental laboratory definitions.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 105 REVISION #: 16 EFFECTIVE DATE: 041110

METALS
BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY (ICP-AES) TECHNIQUE

References: SW-846, Method 6010B, December 1996; SW-846, Method 6010C, Revision 3 February 2007; USEPA, Method 200.7, June 1991; Standard Methods 19th Edition 2340B; 1995 USEPA CLP, ILM 04.1. See Addendum for USEPA CLPILM 05.2

APPROVALS:

Lab Director: _____ Date: 4/12/10

Data Quality Manager: _____ Date: 4/11/10

Section Supervisor: Betty DeVille Date: 4/13/10

Changes Summary

Revision 16, 04/11/10

- The SOP is an update from Revision 15 dated 05/08/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

1. Identification of the Test Method

This SOP is compliant with methods – SW846 6010B, SW846 6010C, EPA 200.7, (SM 19th Edition 2340B) Hardness Calculation, (USEPA CLP) ILMO 4.1 (NJDEP does not accept CLPILM 04.1 after June, 2003) and Addendum for USEPA CLPILM 05.2.

2. Applicable Matrix or Matrices

This SOP is applicable to all matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludge samples, sediments, and other solid wastes, require digestion prior to analysis.

3. Detection Limit: Detection limits, sensitivity, and optimum ranges of the metals may be found in the ICP method file.

4. Scope of Application, Including components to be Analyzed

Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Method Detection Limit and the Reporting Limit (also defined as the Limit of Quantitation).

5. Summary of the Test Method

5.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g., Methods 3005-3050 and SOW ILM 04.1/05.2). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis.

NOTE: When selenium is required soluble samples must always be digested.

5.2 This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving a measurement of the concentration of each element type in the original sample. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analytic wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Control of the spectrometer is provided by PC based *iTEVA* software.

5.3 Inductively Coupled Argon Plasma (ICAP) primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments

utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.

5.4 It is standard procedure to use an internal standard (scandium) with samples to increase the stability of the instrument as recommended by the manufacturer (Thermo Fisher). (When samples are suspected of containing scandium, internal standard cannot be used.)

6. Definitions

Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

Additional definitions specific to this SOP are listed below:

6.1 **ICP or ICAP**- Inductively Coupled Plasma or Inductively Coupled Argon Plasma.
6.2 **Inter-element correction (IEC)**- Defined as a correction factor applied by the instrument when there is an overlap of the spectrum from the plasma gases or from another metal into the spectrum of another metal causing that metals concentration to either be inflated or deflated.

7. Interferences

7.1 Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high- concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

7.1.1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods

using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.

7.1.2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a 200 mg/L or 500 mg/L concentration near the upper analytical range limit.

7.1.3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

7.1.4. When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that Arsenic is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Aluminum. According to Table 2 from the method, 100 mg/L of Aluminum would yield a false signal for Arsenic equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Aluminum would result in a false signal for Arsenic equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The

interference effects must be evaluated for each individual instrument since the intensities will vary.

7.1.5. Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.

7.1.6. The interference effects must be evaluated for each individual instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The instrument utilizes a computer routine for automatic correction on all analyses.

7.1.7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

7.1.8 When inter-element corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions (IFA/IFB). If the correction factors or multivariate correction matrices tested on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at ± one reporting limit from zero, daily verification is not required. All inter-element spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation-change, such as in the torch, nebulizer, injector, or plasma conditions occurs.

Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

7.2. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.

7.3. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized

7.4 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. When the instrument displays negative values, dilution of the samples may be necessary.

8. Safety

Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab-wide.

8.1 Normal accepted laboratory safety practices should be followed while performing this analysis.

8.1.1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of appropriate safety gloves and lab coats is highly recommended.

8.1.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.1.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Quality Assurance Officers office.

9. Equipment & Supplies

9.1. Inductively coupled argon plasma emission spectrometer: Thermo Scientific 6500 DUO.

9.2. Computer-controlled emission spectrometer with background correction: Thermo Scientific 6500 DUO or equivalent.

9.3. Radio frequency generator compliant with FCC regulations: Thermo Fisher or equivalent.

9.4. Auto-sampler: Thermo Fisher or equivalent.

9.5. Printer capable of printing results every 4 minutes.

9.6. Cooling Water recycler.

9.7. *Iteva* software.

9.8. Argon gas supply – Liquid Argon

9.9. Class A volumetric flasks

9.10. Analytical balance - capable of accurate measurement to a minimum of three significant figures (0.001gm).

9.11. Variable Eppendorf Pipettes 1000µL; 5000µL

9.12. Disposable beakers 10, 20 and 50 mL size.

9.13. Hood system capable of venting the heat from the system off of the instrument during analysis.

10. Reagents and Standards

The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

10.1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.

10.2. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

10.3. Hydrochloric acid (concentrated), HCl. A method blank is digested and analyzed before a new lot number of HCl is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.

10.4. Nitric acid (concentrated), HNO_3. A method blank is digested and analyzed before a new lot number of HNO3 is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.

10.5. Calibration standards

10.5.1. All standards have an acid matrix of 2% HNO_3 and 5% HCl and should be prepared using class A volumetric flasks and calibrated Eppendorfs).

10.5.2. CAL1 is the calibration blank: Reagent grade water **matrix matched as in 10.5.1. Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. Prepare this solution fresh daily.**

10.5.3. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.

10.5.4. Stock QC7 solution: Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals- (50 ug/mL)- silver; (100 ug/mL)- aluminum, boron, barium and sodium; (1000 ug/mL)- potassium; (500 ug/mL or 100 ug/mL note we use two sources of this standard and each have different concentrations for Si) –Silica.

10.5.5. Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.6. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.7. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.8. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.9. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier. Note: Two sources are needed.

10.5.10. Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.11. Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.12. Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.13. Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.14. Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.15. Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.16. Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.17. Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.18. Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.19. Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.20. Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.21. Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.22. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.23. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.24. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.25. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.26. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.27. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.28. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.29. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.30. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.31. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.32. Stock Scandium solution (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.6. Calibration and Calibration Verification standards

10.6.1. The calibration standards and calibration verification standards preparations are recorded in Element. Please find method of preparation in Appendix I.

10.6.2. The CRL solution is analyzed to check the accuracy of the instrument at the reporting limit. The stock standard solutions A and B are prepared from single element standards listed in 10.5 above. Please find method of preparation in Appendix I. This solution is stable for 6 months. The working solutions are made up as needed or every 3 months as follows: Prepared by adding 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B to de-ionized water with 2% HNO3 and 5% HCL matrix and diluting to 100 mLs , mix well. This solution is stable for 3 months.

10.6.3. The interference check standard solutions (IFA and IFB) are prepared to provide an adequate test of the IECs. A purchased solution containing 500

ug/mL Al, Ca, Mg and 200 ug/mL Fe is diluted 10x to prepare the IFA. The IFB is prepared by diluting 100x a purchased solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x. These solutions are prepared as needed or monthly and assigned an Element # for traceability.

10.7 Digestion standards

10.7.1 The Blank Spike (BS) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B; and 0.50 mLs of the 1000 ug/mL single element standards for Molybdenum, Boron, Titanium and Strontium is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3 and 0.050 mLs of 10000 ug/mL Tin. 25 mL of HCl and 10 mL of HNO3 are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the BS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier within Element. The BS is prepared from a source independent from that used in the calibration standards. This solution is prepared daily or as needed. 50 mLs of this solution is used for digestion for normal level water samples and the sample is brought back to 50 mLs after digestion. Low level water samples start with two 50 mLs vials with only 1.0 mL of the stock blank spike solution in each taken to 50 mLs. The samples are cooked down to below 25 mLs and combined and then cooked down to below 25 mLs again and then brought back to 25 mLs. This low level BS is given a unique identifier in Element.

10.7.2. The solid BS used with soil samples is prepared by weighing up 1.0 gram of Teflon chips for regular level and 2.0 grams of Teflon chips for low level and spiking using the same spiking solutions used to spike the sample matrix. This standard is given a unique identifier i.e. Batch #-BS1. Note: Amount of spiking solution used varies according to whether the samples are being digested for normal level or low level soils. See spiking solutions in 10.7.3.1 for how to prepare the BS for a solid sample, it is prepared the same way that a soil spike is prepared only the known amounts of metals are added to laboratory water.

10.7.3. The spiking solutions are prepared as follows:

10.7.3.1.Stock Multi-element Spiking Solutions: High Purity <u>CLP-CAL-1 solution A</u>: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; <u>solution B</u>: 250 ug/mL Ag; <u>CLP-CAL-2</u>: 1000 ug/L Sb; <u>CLP-CAL-3</u>: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier within Element. Add 0.050 mL for water samples and 0.20 mL for normal level soil samples and 0.10 for low

level soil samples of CLP-CAL-1 solutions A and B, and 0.0125 mL for water samples and 0.05 mLs for normal level soil samples and 0.025 mLs for low level soil samples of CLP-CAL-2 and 3 to 50 mL of sample for water samples and 1gram of sample for normal level soils and 2 grams of sample for low level soils for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.

10.7.3.2. TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion:
2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL . Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed.

10.7.3.3. TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion:
0.40 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store this solution in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 200 ug/L. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. Also this solution is not very stable and may require fresh preparation at least weekly.

11. Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.1. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been pre-filtered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).

11.2. Sample digestates are stored at room temperature for at least 2 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. All metal samples are neutralized before disposal in the receiving section of the laboratory.

11.3. **The appropriate SOPs should be consulted regarding sample preparation.** The following is a brief summary of the methods we use for metals preparation.

11.3.1. Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and HNO_3 prior to metal determination.

11.3.2. Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.

11.3.3. Standard Methods 19th Edition Method 3030C prepares ground-waters and surface water samples for acid extractable metals: (lead and chromium.) This preparation has a holding time of 72 hours. The samples are preserved at collection with 5mL/L of HNO3, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. The sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.

11.3.4. Method 3050B prepares wastes samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

12. Quality Control

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.1. **Daily run and batch QC**

12.1.1. Calibration is required daily. Either a blank and a high standard or a client specific three standard concentration points and a blank calibration is required daily.

12.1.2. IEC correction standards for aluminum and iron are required daily.

12.1.3. ICV within ±5% for 200.7 and within ±10% for all other methods.

12.1.4. ICB/CCB less than two times ± MDL or less than ± LOD for DOD. The ICB/CCB must immediately follow the ICV/CCV.

12.1.5. RL standard run against the curve within ±20% initially and client specific requirement of ±30% at the end of the analysis.

12.1.6. IFA/IFB analyzed daily. IFA must be less than two times ±MDL or less than ±LOD unless verified standard contamination for DOD. The IFB must recover within ±20% for all analytes in the IFB standard solution. If the IFA/IFB solution is not within the required limits- if possible reanalyze all associated samples, if not possible to reanalyze all associated samples must be flagged with an "Q" on the final report for DOD.

12.1.7. CCV must be analyzed every ten samples or at the end of the analysis within ±10% or the samples are reanalyzed if possible. If samples cannot be reanalyzed, all samples are flagged with a "Q" for DOD.

12.1.8. CCB must be analyzed every ten samples immediately following the CCV or at the end of the analysis less than two times ±MDL or <±LOD for DOD. If the CCB is out of the allowable range the samples are flagged with "B".

12.1.9. *The following should be analyzed with each preparation batch containing a matrix spike.*

- Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (volumetric glassware must be used) should agree within ±10% of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

- Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value for SW6010B and 80 to 120% for SW6010C and is required especially if the pre-digestion matrix spike is outside of control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. Run all associated samples in the preparatory batch by method of standard additions (MSA) or apply "J" flag. The analyst and or section manager must note this situation on the final analytical report. Apply "J" flag if the post spike is outside the range of 75 to 125% for 6010B or 80 to 120% for 6010C.

12.2 Quarterly and/or every six months

12.2.1. Linear range standards must be analyzed at a frequency no less than once every six months. The linear range standard is required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. The linear range standard must be within +/-10% of true value. This standard can be analyzed as the linear dynamic range.

12.2.2. The inter-element correction factors (IEC) should be verified at the time the linear range standards are analyzed or whenever there is any question about whether an IEC is correcting correctly.

12.2.3. IDL's, linear range and IEC checks must be performed quarterly if straight CLP work is required.

12.3. Digested Batch QC

12.3.1. All quality control data should be maintained and available for easy reference or inspection.

12.3.2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank (BLK), sometimes referred to as the preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. The result for the method blank should not indicate contamination greater than ± ½ RL for DOD or ±RL/CRDL for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The extracted blank associated with TCLP batches must be less than 100 X the regulatory limit for barium.

12.3.3. Employ a minimum of one blank spike (BS) for aqueous samples or one Teflon chip spiked sample per sample batch to verify the digestion procedure. These blank spikes are taken through the same digestion/preparation steps as the sample being tested. The control limits are ±15% method 200.7 - aqueous and soil samples or ±20% for all other methods aqueous and soil samples. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be re-digested. Consult your supervisor for further action. Qualifying the associated data may not be permissible for some clients.

12.4. Sample

12.4.1. Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. The control limits are less than or equal to 20% RPD (if both are >5x RL) or ± the RL (if either are <5X RL). Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. Apply "J" flag for DOD if acceptance criteria are not met. Apply "*" flag for CLP and other work if acceptance criteria are not met.

12.4.2. Analyze a minimum of one spiked sample and/or spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. Project

specific requirements will take precedence in determining whether a matrix spike duplicate is employed in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within ±20% of the true value. If not, and sufficient sample volume exist, a post digestion spike should be analyzed. Apply "J" flag for DOD if acceptance criteria are not met. Apply "N" flag or CLP and other work if acceptance criteria are not met.

13. Calibration and Standardization

Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

13.2. Operating conditions - **The instrument settings can be found in method file within the iTEVA software.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

13.3. Auto-peak when some change has been made to the introductory system and calibrate the instrument according to the instrument manufacturers recommended procedures, using the specified calibration standard solutions. Flush the system with 2% HNO_3 / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ($r \geq 0.998$). If a three point calibration curve is not required for the client samples being analyzed by Empirical Laboratories may use a blank and one standard as referenced in USEPA - CLP protocols.

13.4. Before beginning the sample run, analyze single element Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs using the iTEVA software. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV (± 10%) for 200.7 (± 5%) and ICB (< ±2xMDL, <±LOD-DOD or ±RL/CRDL for others or CLP, first, then analyze a reporting limit standard (a standard at the concentration of the reporting limit). This standard should be within ±20% for DOD projects and ±30% for samples analyzed for 6010C. Then reanalyze the

highest mixed calibration standard(s) as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5%. If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.5. For **CLP projects**, verify the validity of the curve in the region of 2x the contract required detection limit (CRDL) before and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB (CCB criteria: < ±MDL or ±RL/CRDL for others or CLP, or twice during every 8-hour work shift, whichever is more frequent. Results should be within ±20%. Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. (For Internal QC)

13.6. Verify the inter-element and background correction factors at the beginning of the sequence in the specific order of IFA, IFB, CCV and CCB (IFA criteria: non-spiked analytes < ±2xMDL or <±LOD for DOD beginning of sequence**.** Do this by analyzing the interference check solution IFA and IFB. Absolute value of concentration for all non-spiked analytes in the IFA must be <LOD (unless they are verified trace impurity from one of the spiked analytes) for DOD. Results must be within ±20% of the true value for IFB. If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS. (CRI, ICSA and ICSAB required at the end for CLP projects only).

Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.7. The instrument must be calibrated once every 24 hours.

13.8. Instrument Autosampler Report example:

Calibration Rack (used by instrument software to insert QC)

1) Cal Std 1 (blank)
2) Cal Std 2 (Low Cal)
3) Cal Std 3 (Mid Cal)
4) Cal Std 4 (Ba @ 5000 ppb)
5) Cal Std 5 (QC5)
6) Cal Std 6 (QC 21)
7) Cal Std 7 (NAK 100)
8) Cal Std 8 (QC3)
9) Cal Std 9 (Ag)
10) Al IEC-(correction using ITEVA software)
11) Fe IEC-(correction using ITEVA software)

Sample Sequence RACK 1

1) SEQ-ICV
2) SEQ-ICB
3) SEQ-CRL1-reporting limit standard 1
4) SEQ-CRL2-reporting limit standard 2
5) Ba@ 5000 ppb (readback)
6) QC5
7) NAK High-(readback)
8) QC 21 High-(readback)
9) Salt Cal at 500 ppm (readback)
10) Rinse
11) SEQ-IFA1
12) SEQ-IFB1
13) Rinse
14) SEQ-CCV
15) SEQ-CCB
16) Method Blank (*Batch #* -BLK1)
17) Blank Spike (*Batch #* -BS1)
18) Sample 1
19) Sample 2
20) Sample 3
21) Sample 4
22) Sample 5
23) Sample 6
24) Sample 7
25) Sample 8
26) Sample 9
27) Sample 10
28) SEQ-CCV
29) SEQ-CCB
30) Sample 11
31) Sample 12
32) Sample 13
33) Sample 14
34) Sample 15
35) Sample 16
36) Sample 17
37) Sample 18
38) Sample 19
39) Sample 20
40) Sample matrix spike (*batch#*- MS1)
41) Sample matrix spike duplicate (*batch#* -MSD1)
42) Sample post digestion spike (*batch#* -PS1)
43) Sample serial dilution (*batch#* -DUP1)
44) SEQ-CCV

45) SEQ-CCB
46) Preparation Blank (*batch#* -BLK1)
47) Blank Spike (*batch#* -BS1)
48) Sample 1
49) Sample 2
50) Sample 3
51) Sample 4
52) Sample 5
53) Sample 6
54) Sample 7
55) Sample 8
56) Sample 9
57) Sample10
58) SEQ-CCV
59) SEQ-CCB
60) Sample 11

RACK 2

1) Sample 12
2) Sample 13

Etcetera…

Each rack holds 60 samples and there are 4 racks that are used for samples, CCVs and CCBs and run QC.

14. Procedure

14.1. Once the instrument has been calibrated, begin the analysis of samples.

14.2. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. **In the event USACE samples are filtered, all USACE samples and the QC samples in that QC batch must be filtered. All USACE solid samples and their associated batch QC samples must be filtered prior to analysis.**

14.3. Flush the system with 2% HNO_3 / 5% HCl for at least 1 minute before the analysis of each sample.

14.4. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7, $\pm$ 10% of the linear range standard. **In the case of USACE samples, the criterion changes and requires dilution and reanalysis of all samples which produce a concentration that exceeds the highest calibration standard. Sample results detected between the MDL and LOQ are flagged as estimated with a "J" flag.**

14.5. Verify calibration every 10 samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using a continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

14.5.1. The results of the CCV are to agree within ±10% for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) < 5% from 3 replicates (minimum of three integrations). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run, and after conferring with the section manager it may be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours.

14.5.2. The results of the calibration blank (this is not the method/preparation blank) are to be < 2x ±MDL, for CLP <RL, for **DOD no analytes detected >±LOD**. If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank < 1/10 the concentration of the action level of interest and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).

14.6. Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

15. Data Analysis and Calculations

Quality Systems SOP QS09 "General and commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.1. Total hardness is reported from HNO_3 preserved sample. The final concentration is calculated from the calcium and magnesium results as follows: Ca mg/L x 2.5 + Mg mg/L x 4.1 = total Hardness in mg/L as $CaCO_3$.

15.2. The instrument will generate data results in mg/L or µg/L (labeled appropriately). Each result represents an average of three individual readings per metal channel.

15.3. For aqueous samples, if a post/pre-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.

15.4. For solid samples, if a post-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

SR (ug/g or mg/kg) = IR*DF*FED/SM

SR = Sample result
IR = Instrument result (µg/L)
DF = Dilution factor (post digestion)
FED = Final volume of digestate (L)
SM = Sample mass digested (g)

16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality Manuel is completed by each analyst and then provided to the supervisor for further processing and approval.
DOC LCS Preparation: See BS preparation under 10.7.1 through10.7.3 above.
DOC Accuracy and Precision Criteria: The LOD is analyzed at 2 times the MDL and must result in an concentration 3 times the noise. The LOQ is analyzed at the RL or 2 times the RL and must be recovered within ±50%.

17. Pollution Prevention:
Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. Table 2 of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data
Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

CORRECTIVE ACTIONS

19.1. INSTRUMENT RELATED

19.1.1. ICV not within ± 10% or ± 5% for 200.7
a. Is the problem with the solution?
i. Re-prepare or obtain new stock.

b. Is the problem with the calibration?
   i. Recalibrate through analysis of appropriate standards and recheck ICV.

19.1.2. ICB not ±MDL or within ± 3X IDL or CRDL for CLP, **DOD no analytes detected >LOD**

a. Is the problem with the solution?
   i. Re-prepare
b. Is the problem with the calibration?
   i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.

19.1.3. Check standards not within ± 5%
a. Is the problem with the solution?
   i. Re-pour, re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.4. CLP only-CRI not within ± 20% (Internal QC, only required for CLP work).
a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.5. IFA metals not present are not less than the detection limit for that metal, **for IFA DOD, absolute value of concentration for all non-spiked analytes <±LOD.**
a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.6. IFB not within ± 20%
a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.7. CCV not within ± 10%
a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified.

19.1.8.. CCB not ±2xMDL or CRDL for CLP, DOD no analytes detected >±LOD.

a. Is the problem with the solution?
   i. Re-prepare
b. Is the problem with the calibration?
   i. Re-calibrate and reanalyze.

19.2. DIGESTION RELATED

19.2.1. Preparation blank (BLK) not within ± ½ RL and ± RL for common contaminants DOD or RL/CRDL for other or CLP

a. Is the problem with the instrument?
   i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
b. Is the problem with the digestion?
   i. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be re-digested or the data must be qualified on the final report.

19.2.2. BS not within control limits

a. Is the problem with the instrument?
   i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
b. Is the problem with the digestion?
   i. If biased low, associated samples must be re-digested.
   ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

19.3. SAMPLE MATRIX RELATED

19.3.1. Replicate analysis RPD not within ±20% (if both are >5X CRDL) or ± the CRDL (if either are <5X CRDL).

a. The associated sample data must be qualified on the final report.

19.3.2. Spike analysis recovery not within ±20%.

a. Is the analyte level in the sample greater than 4X the spiking level?
   i. If yes, the spike recovery is not evaluated.
   ii. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.

19.3.3. When required, post digestion spike analysis recovery not within ±25% for SW6010B, DOD or ± 20% SW6010C.

a. The associated sample data must be qualified on the final report.
b. For USACE analysis by MSA is required.

19.3.4. Serial dilution analysis percent difference not within ±10%

a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?

i. If no, the serial dilution data can not be evaluated.

iii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

20. **Waste Management**

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. **References**

21.1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 6010B and Method 6010C.

21.2. USEPA *Code of Federal Requlations, 40, CH 1,PT 136;* Method 200.7; APX-B.

21.3. USEPA *Contract Laboratory Program (CLP) for Inorganics ILM04.1; ILM05.2*

21.4. DOD Quality Systems Manual for Environmental Laboratories Version 4.1. (Based on NELAC Voted Revision June 5, 2003. 4/22/09

22. **Tables, Diagrams, Flowcharts and Validation Data**

Table 1 contains all applicable parameters with the applicable RL/LOQ, LOD and Detection Limit.

Table 1A, contains a list of the wavelengths used for each analyte.

Table 2, for all technical methods, contains the QA/QC summary table.

Table 3, Technical Completeness / Accuracy Checklist

Table 4, Data Reviewers Checklist

Table 1 Water				
Analyte	MDL	LOD	MRL	Units
Aluminum	50.0	100	200	ug/L
Antimony	5.00	8.00	15.0	ug/L
Arsenic	3.00	6.00	10.0	ug/L
Barium	5.00	10.0	40.0	ug/L
Beryllium	1.00	2.00	5.00	ug/L
Boron	10.0	20.0	30.0	ug/L
Cadmium	1.00	2.00	5.00	ug/L
Calcium	1000	2000	5000	ug/L
Chromium	2.00	4.00	10.0	ug/L
Cobalt	5.00	10.0	12.5	ug/L
Copper	4.00	8.00	10.0	ug/L
Iron	30.0	60.0	100	ug/L
Lead	1.50	3.00	3.00	ug/L
Magnesium	1000	3000	5000	ug/L
Manganese	3.00	6.00	15.0	ug/L
Molybdenum	5.00	10.0	15.0	ug/L
Nickel	3.00	6.00	10.0	ug/L
Potassium	1000	3000	5000	ug/L
Selenium	3.00	5.00	6.00	ug/L
Silver	1.00	2.00	10.0	ug/L
Sodium	1000	3000	5000	ug/L
Thallium	3.00	4.00	8.00	ug/L
Tin	10.0	20.0	30.0	ug/L
Titanium	5.00	10.0	15.0	ug/L
Vanadium	5.00	10.0	12.5	ug/L
Zinc	5.00	10.0	20.0	ug/L

Table 1 TCLP				
Analyte	MDL	LOD	MRL	Units
Antimony	0.00500	0.00800	0.0150	mg/L
Arsenic	0.00300	0.00600	0.0100	mg/L
Barium	0.00500	0.0100	0.0400	mg/L
Cadmium	0.00100	0.00200	0.00500	mg/L
Chromium	0.00200	0.00400	0.0100	mg/L
Copper	0.00400	0.00800	0.0100	mg/L
Lead	0.00150	0.00300	0.00300	mg/L
Selenium	0.00300	0.00500	0.00600	mg/L
Silver	0.00100	0.00200	0.0100	mg/L

Table 1 Soil				
Analyte	MDL	LOD	MRL	Units
Aluminum	10.0	20.0	40.0	mg/Kg
Antimony	1.00	1.60	3.00	mg/Kg
Arsenic	0.600	1.20	2.00	mg/Kg
Barium	1.00	2.00	8.00	mg/Kg
Beryllium	0.200	0.400	1.00	mg/Kg
Boron	2.00	4.00	6.00	mg/Kg
Cadmium	0.200	0.400	1.00	mg/Kg
Calcium	200	400	1000	mg/Kg
Chromium	0.400	0.800	2.00	mg/Kg
Cobalt	1.00	2.00	2.50	mg/Kg
Copper	0.800	1.60	2.00	mg/Kg
Iron	6.00	12.0	20.0	mg/Kg
Lead	0.300	0.600	0.600	mg/Kg
Magnesium	200	600	1000	mg/Kg
Manganese	0.600	1.20	3.00	mg/Kg
Molybdenum	1.00	2.00	3.00	mg/Kg
Nickel	0.600	1.20	2.00	mg/Kg
Potassium	200	600	1000	mg/Kg
Selenium	0.600	1.00	1.20	mg/Kg
Silver	0.200	0.400	2.00	mg/Kg
Sodium	200	600	1000	mg/Kg
Thallium	0.600	0.800	1.60	mg/Kg
Tin	2.00	4.00	6.00	mg/Kg
Titanium	1.00	2.00	3.00	mg/Kg
Vanadium	1.00	2.00	2.50	mg/Kg
Zinc	1.00	2.00	4.00	mg/Kg

TABLE 1A

METAL	WAVELENGTH
Aluminum	396.1
Antimony	206.8
Arsenic	189.0
Barium	233.5
Beryllium	313.0
Boron	249.7
Cadmium	228.8
Calcium	317.9
Chromium	267.7
Cobalt	228.6
Copper	324.7
Iron	261.1
Lead	220.3
Magnesium	279.0
Manganese	257.6
Molybdenum	202.0
Nickel	231.6
Potassium	766.4
Selenium	196.0
Silver	328.0
Sodium	589.5
Strontium	421.5
Thallium	190.8
Tin	189.9
Titanium	334.9
Vanadium	292.4
Zinc	206.2

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
Interference Check	• once per calibration	• IFA less than LOD if not verified contamination of standard. IFB must be within ±20%.	• Check IEC corrections for metals in the IFA.
Calibration Curve	• Prior to analyzing any samples • A minimum of a blank and 3-points for linear fits client specific requirement or a blank and high standard. • Low standard at the RL level run against the curve within 20% initially and within 30% for subsequent analysis (6010C).	• Linear calibration Corr. of 0.998 • Must follow curve processing requirements from SOP QS08	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICB	At the beginning of every sequence	Must meet the <±LOD for DOD or < 2xMDL	Re-run
ICV	Alternate source standard to be analyzed after every calibration curve	• Must be in the range 90 to 110% for 6010B&C, or 95 to 115% for 200.7.	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	• At the beginning of every sequence • For every 10-client samples	• Must be in the range 90 to 110%	• Samples must be reanalyzed if possible, if not samples are flagged with a "Q".
Closing CCV	• At the end of every sequence	• Must be in the range 90 to 110%	• Samples must be reanalyzed if possible, if not samples are flagged with a "Q".
BLK	One per prep batch	• Must be less than ½ ±RL.	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
BS	One per prep batch	Must be in the range of 80 to 120% for 6010B, DOD; or 85 to 115% for 200.7.	• Rerun to confirm problem. • All samples associated with the LCS must be re-digested, reanalyzed if possible. • NCR will be required for data reported • If samples cannot be re-digested or re-analyzed Final Report data flagging will be required
MS	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
MSD	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
Sample Duplicate	One per prep batch	20%	Flag samples
Post Digestion Spike	One per batch	±25% for DOD/6010B, ±20% 6010C	If possible MSA required, Flag samples
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Must meet the criteria of the BS for average accuracy	• Re-prep and / or • Re-analysis
MDL Study	Once per year		
LOD Verification	Every quarter		
LOQ Verification	Every quarter		
Linear Dynamic Range Study (LDR)	Every six months		

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
7. Were proper data qualifiers applied to the data in LIMS
8. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST Sample Number(s):
Batch Number(s):
Method: 6010B or 6010C (ICP)

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did BS or blank spike meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank (BLK) below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor?				
10. Sample preparation information is correct and complete.				
11. Analytical results are correct and complete.				
12. The appropriate SOP's have been used and followed.				
14. "Raw data" including all manual integration's have been correctly interpreted.				
15. "Special" sample preparation and analytical requirements have been met.				
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.				

Comments on any "No" response:

_____

_____

_____

_____

_____

_____

Analyst: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 164 REVISION #: 15 EFFECTIVE DATE: 20100325

DISTILLATION OF AQUEOUS/SOLID SAMPLES FOR CYANIDE, TOTAL AND AMENABLE

REFERENCE:
SW846 METHOD 9012A, USEPA Methods 335.1, 335.4, Standard Methods SM 4500-CN C,G, 18th, 19th ED. / CLP ILMO 4.1
See Addendum for USEPA CLPILM 05.2 (Aqueous, Soil/Sediment)

APPROVALS:

Lab Director: _____ Date: 3/30/10

Data Quality Manager: _____ Date: 3/30/10

Section Supervisor: _____ Date: 3/30/10

Changes Summary:

Revision Date: 03/25/2010

- The SOP has been reviewed for accuracy and completeness. No changes have been made.

DISTILLATION OF CYANIDE, TOTAL AND AMENABLE

REFERENCE:
SW846 METHOD 9012A, USEPA Methods 335.1, 335.4, Standard Methods SM 4500-CN C,G, 18th, 19th ED. / CLP ILMO 4.1
See Addendum for USEPA CLPILM 05.2 (Aqueous, Soil/Sediment)

I. SCOPE AND APPLICATION

A. This method is applicable to the distillation of cyanide from drinking, surface, and saline waters, domestic and industrial wastes, and soil/sediments.

B. The limit of detection for waters is 0.0050 mg/L and the limit of quantitation is 0.010 mg/L. The limit of detection for soils is 0.13 mg/kg and the limit of quantitation is 0.25 mg/kg.

II. SUMMARY OF METHOD

A. The cyanide as hydrocyanic acid is released from cyanide complexes by means of a reflux distillation operation, and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined colorimetrically.

B. Cyanide is defined as cyanide ion (CN^-) and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

III. SAMPLE HANDLING AND PRESERVATION

A. The sample should be collected in plastic or glass bottles of 100 mLs. All bottles must be thoroughly cleansed and thoroughly rinsed to remove soluble material from containers.

B. Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.06 g of ascorbic acid for each liter of sample volume.

C. Samples (not including soils/sediments) must be preserved with 2 mL of ten normal sodium hydroxide per liter of sample (pH $\geq$12) at time of collection.

D. Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator to maintain temperature at 4°C.

E. The holding time for cyanide is 14 days counting the day of sampling as the first day. This applies for soils as well as waters. Samples must be distilled and analyzed within 14 days of sampling.

IV. INTERFERENCES

A. Interferences are eliminated or reduced by using the distillation procedure.

B. Sulfides adversely affect the colorimetric procedures. Samples that contain hydrogen sulfide, metal sulfides, or other compounds that may produce hydrogen sulfide during the distillation, must be treated by adding lead acetate, or if the sulfide concentration is too high, add powdered lead carbonate [$Pb(CO_3)_2$] to avoid significantly reducing pH. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper. Filter sample before raising pH for stabilization.

C. High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under test conditions to generate HCN. Nitrate and nitrite interference is eliminated by adding 0.5 ml of sulfamic acid solution (VII.A.7) before distillation.

D. The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoam agent will prevent the foam from collecting in the condenser.

V. SAFETY

A. **CAUTION: KCN is highly toxic. Avoid contact with standard solutions.**

B. If an alkaline solution containing simple cyanide is acidified, the simple cyanide will be released.

C. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential heath hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

D. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be warn when handling samples and chemicals.

VI. EQUIPMENT/APPARATUS

A. Reflux distillation apparatus is a midi-distillation apparatus which uses Hammett Scientific Glassware Note: This glassware is made to fit our specific block digester for cyanide. Glassware orders must be made in advance since they are not made until ordered.

VII. REAGENTS AND STANDARDS PREPARATION

A. Reagents

1. **Sodium Hydroxide, 0.25N**: Dissolve 10 g grams of NaOH in DI water and dilute to 1 liter with DI water. Store in a labeled plastic container at room temperature.

2. **Sulfuric Acid 1:1:** Slowly add 500 mL of concentrated H_2SO_4 to 500 mL DI water. Use caution because solution will become extremely hot!! Allow to cool while continuing to stir. Store in a labeled container at room temperature.

4. **Magnesium Chloride Solution**: Weigh 510 g of $MgCl_2 \cdot 6H_2O$ into a 1 liter flask; dissolve and dilute to 1 liter with DI water. Store in a labeled plastic container at room temperature.

5. **Chlorine Bleach Made with Sodium Hypochlorite**, such as Chlorox.

6. **Rhodanine Indicator**:
   Dissolve 20 mg of p-dimethyl-amino-bensalrhodanine in 100 mL acetone.

7. **Sulfamic Acid:** Dissolve 40 g of sulfamic acid in DI water, dilute to 100 mL (saturated solution). **NOTE:** When making this reagent for use with the maxi-distillation system the 40 g of sulfamic are diluted to one liter with DI water

8. **Phenolphthalein solution:** Commercially prepared.

9. **Lead Acetate Test Paper**: Commercially prepared.

10. **Acetate Buffer Solution, pH 4.0:** Dissolve 146g anhydrous $NaC_2H_3O_2$, or 243 g $NaC_2H_3O_2.3H_2O$, in 400 mL distilled water, add 480 g conc. acetic acid, and dilute to 1 L with chlorine-demand-free water.

11. **Lead Acetate**

12. **Powdered Lead Carbonate [$Pb(CO_3)_2$]**

B. Standards

1. Traceability

a. All reference materials must be recorded in Element and given an element identification number. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in Element as well as on the container's label.

b. All working standards made from reference materials shall be labeled with a unique ID number from Element with complete information on preparation date, concentration of

each compound, solvent, preparer's name, expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded on the bench sheet. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

2. **Preparation**

   a. Stock Cyanide Solution at 1000 mg/L: Commercially prepared.

   b. Working Standard Cyanide Solution: Prepare fresh daily by diluting 1 mL of stock cyanide solution to 100 mL in a volumetric flask with using 0.25N NaOH as diluent. 1 mL - 10 µg CN. Record the preparation in Element.

   c. Performance Evaluation (PE) samples which we have analyzed and received true values for are used for the Laboratory Control Samples. These PE samples are given a unique identifier and all information such as expiration date (typically one year from the date made), source, concentration and preservation are recorded in Element and the Element # assigned is used to label this PE sample and is used on all data to trace it back to its source and true concentration.

VIII. CALIBRATION

A. Not applicable. See SOP-175 "**POST DISTILLATION ANALYSIS OF CYANIDE BY THE LACHAT"** for analysis.

IX. PROCEDURE

A. Pretreatment for cyanides amenable to chlorination

1. Two sample aliquots are required to determine cyanides amenable to chlorination. Place one 25 mL aliquot or a volume diluted to 25 mL, in a 100 mL beaker, under the hood, add a stirring bar, and place on a magnetic stirrer, and start stirring bar turning.

2. Check pH and adjust to between 11 and 12 units, with 1.25N NaOH. Use NaOH pellets if solution is highly buffered.

3. Add sodium hypochlorite chlorine bleach (Chlorox) solution dropwise until a drop of well-mixed sample turns KI starch paper a distinct blue color. Add about 10 drops excess bleach. (Caution: the initial reaction product of alkaline chlorination is the very toxic gas cyanogen chloride.) Maintain this excess for one hour, continuing agitation. Check periodically during the hour (15 minute intervals) to make sure an excess is maintained. If necessary, add additional hypochlorite bleach solution. Also check the pH and adjust back up to 11 to 12 units if necessary.

4. After one hour, add 0.025 g portions of ascorbic acid until KI starch paper shows no residual chlorine. Add an additional 0.025 g of ascorbic acid to ensure the presence of excess reducing agent.

5. Turn off stirring plate and remove stirring bar. Proceed with distillation as in the procedure.

6. Test for total cyanide in both the chlorinated and unchlorinated aliquots. (The difference of total cyanide in the chlorinated and unchlorinated aliquots is the cyanide amenable to chlorination.)

B. Spikes and high and low check standards

1. **Spikes**: Matrix spikes are prepared by diluting 2.5 mL of the High DCV solution in #3 below, to 25 mL with sample (adding 22.5 mL of sample and 2.5 mL of DCV high to the distillation tube). The concentration of this spike is 0.10 mg/L.

2. **Low Distilled Check standard (low DCV).** Dilute 2.0 mL of the 10 mg/L working standard to 100 mLs in a volumetric flask. Then dilute 6.0 mLs of this solution to 25 mLs with 0.25N NaOH, to make a 0.048 mg/L standard.

3. **High Distilled Check standard (high DCV).** Dilute 10 mLs of the 10 mg/L working standard to 100 mLs in a volumetric flask with DI water. Then dilute 5 mLs of this solution to 25 mLs with 0.25N NaOH, to make a 0.20 mg/L standard.

C. Distillation

1. For Waters: Place 25 mL of sample, or an aliquot diluted to 25 mL, in a sample tube. 0.25 N NaOH must be used for all sample dilutions following the distillation.

2. For Soils or Sludges:

   a.) It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

   - The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.
   - Two quarters should then be mixed to form halves.
   - The two halves should be mixed to form a homogenous matrix.

   This procedure should be repeated several times until the sample is adequately mixed.

   NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a

representative sub-sample aliquot from the center or middle section of the sample container.

b.) Accurately weigh a representative 1-g minimum (record actual weight) portion of wet-weight sample (unless there are project-specific requirements where a lower detection limit is required) and transfer it to a sample tube. Add 25 mL of 0.25 N NaOH. Shake or stir the sample so that it is dispersed.

3. Add one drop of Phenolphthalein solution to verify preservation. A positive test is indicated by a bright pink color. If not add a small amount of NaOH.

4. To test for sulfide, place a drop of sample on lead acetate paper, previously moistened with acetic acid buffer solution (pH 4). A positive test is indicated by a black color on the paper.

   a.) If positive the sample will need to be filtered through 934AH filter paper. (4.5cm size) The filtrate will be treated and the filter added back to the treated filtrate before distillation.

   b.) Treat filtrate with powdered lead carbonate [$Pb(CO_3)_2$]. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper.

   c.) Filter the resulting precipitate (should form a lead sulfide which will be black in appearance).

   d.) Once the filtrate is treated and filtered, the filter with the original solids from the pretreated sample will be added back to the treated filtrate and then distilled.

5. Add 25 mL of sodium hydroxide solution 0.25N to the absorber and assemble the apparatus as shown in Fig. 1.

6. Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum such that air bubbles from the thistle tube in the flask at a rate of 3 to 5 per second.

   Note: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It may be necessary to readjust the air rate occasionally to prevent the solution in the sample tube from boiling and spewing out through the thistle tube.

7. Turn on condenser water.

8. Add 0.5 mLs of sulfamic acid through the thistle tube.

9. Slowly add 2 mL 1:1 H_2SO_4 through the thistle tube.

10. Add 2 mL 2.5 <u>M</u> $MgCl_2$ through the thistle and rinse it down with deionized water.

11. Reflux at 100°C for 90 minutes, turn off heat, allow to cool with pump on for 15 minutes.

12. Disconnect absorber and transfer the solution from the absorber into a storage container.

14. Record distillation information in the distillation log. Include date, analyst, sample number, client, matrix, position, sample mass / volume, distillation time, cool down time, when transferred to analyst, when analyzed.

X. CALCULATIONS

A. Sample volumes / masses must be recorded accurately in the distillation log to allow the analyst to correctly calculate the final concentration of cyanide correctly.

XI. DISTILLATION GLASSWARE CLEANING & LABELING

A. The distillation glassware is cleaned thoroughly with hot soapy water between distillations. 1:1 HCl is aerated through the frits on the block to ensure that the frits are clean. Deionized water is forced up through the glassware to rinse it. The cold fingers are dipped in mild acid, rinsed and wiped off.

B. All glassware must be labeled and each position's label must be recorded in the distillation log with the sample that was distilled in that particular vessel.

C. When sample concentrations are at or exceed 1000 mg/L a deionized water blank must be distilled in the exact glassware used for that sample before processing any other client's sample to ensure that the glassware is cleaned and has no residual cyanide to carryover.

XII. QUALITY CONTROL

A. With each distillation batch of samples (maximum of 20 samples) a preparation blank (BLK) must be distilled.

B. Each distillation batch of aqueous samples **requires a laboratory control sample (BS) each day or per 20 samples whichever is more frequent.** Under CLP the BS serves as the distilled ICV.

C. With each distillation batch of soil samples a soil BS (boiling chips are used) **each day or per 20 samples whichever is more frequent.**

D. A high and low check standard (DCV/ICV) must be distilled each day or per 20 samples whichever is more frequent. A non-distilled check standard must be prepared daily. This will be used to verify the preparation of the DCV if the DCV results are outside the specification limits. Under CLP the aqueous BS serves as the ICV.

E. A sample spike (MS) and sample spike duplicate (MSD) are performed with each set of twenty samples. CLP requires 1 duplicate and 1 spike per batch. All QC samples should be taken through the whole procedure including distillation. Spikes are added to the boiling flask in the same manner as standards adding 25 mL of samples or a dilution as used for the unspiked

sample. Spike concentration is listed in the method. The addition of the spiking solution must be witnessed and such must be noted in the digestion log.

XIII. CORRECTIVE ACTION

A. If any problems, such as low or high recovery for check standards or BSs occur, contact the Group Leader. Problems most frequently encountered (for the distillation procedure) are listed below.

B. Recoveries are affected in various ways.

1. If the spiking solution is not prepared properly (e.g., making the proper dilutions), the check standard will be out of control. Caution should be used in calculating the dilution and performing the pipetting.

2. Also, the air flow from the vacuum pumps can cause low recovery. Pay attention to the air flow (it should be in the area of 1 to 2 bubbles per second). If it is too great the HCN does not have an adequate amount of time to be absorbed by the NaOH solution.

3. If the heating block is not heating properly, this will also lead to low recovery. During the distillation, periodically check the sample tubes for the presence of heat (tubes should be very hot to the touch).

4. The addition of all reagents, including the spiking solution, is essential. Without the $MgCl_2$, 1:1 H_2SO_4, or spiking solution, the percent recovery will suffer greatly for complex cyanide. The recovery of simple cyanides will usually be unaffected.

5. If the volume of 0.25 N NaOH and reagent water is not accurate in the NaOH absorber tubes, low recoveries will usually occur.

B. Spike recoveries are affected in similar ways. Refer to the above suggestions. Also, if possible, do not spike a sample that is known to be high in cyanide. This may cause recovery and calculation problems.

XIV. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. The ICV shall be distilled.

2. Boiling chips are to be added to each sample. Midi-Distillation is reflux 1.5 hours then the heat and vacuum is turned off and the samples cool an additional 15 minutes.

3. The QC criteria for both the Distillation Check QC and the distilled ICV is ±15%.

4. A CRI is required at the beginning and end of each run and for every 20 samples. The QC criteria for the CRI is ±30%.

5. Rounding rule for the appropriate level of precision is that the figure following those to be retained is $\geq$ 5, round up: otherwise round down. (examples: 1.5 and 2.5 would be 2 and 3 respectively rounded up; 1.4 and 2.4 would be 1 and 2 respectively rounded down) Please see Exhibit B, Section 3, (3.3.9.1) of SOW ILM0 5.2 for more guidance on rounding significant figures.

XV. DEFINITIONS

See SOP-431 for a list of definitions

POST-DISTILLATION ANALYSIS FOR CYANIDE USING LACHAT FLOW INJECTION ANALYZER

METHODS 335.4;(SW846) 9012A, Standard Methods 21st Edition

USEPA-CLP 4.1

(NJDEP does not accept CLPILM 04.1 after June, 2003)

Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT

SOP NUMBER: **SOP-175**

REVISION NUMBER: **10**

APPROVED BY:

SECTION MANAGER

DATA QUALITY MANAGER

EFFECTIVE DATE: **12/15/09**

DATE OF LAST REVISION: **12/15/09**

POST DISTILLATION ANALYSIS OF CYANIDE BY THE LACHAT
0.005-0.500 mg CN^-/L

References:
USEPA METHOD 335.4/(SW846) 9012A/ Standard Methods 21st Edtion 4500CNE/USEPA CLP ILMO 4.1
See Addendum for USEPA CLPILM 05.2 (Soil/Sediment)

I. PRINCIPLE

Cyanide from alkaline distillates is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH lower than 8. The CNCl then forms a red-blue dye by reacting with pyridine-barbituric acid reagent. The color is read at 570 nm.

II. INTERFERENCES

1. For total cyanide, most interferences are eliminated or minimized by the distillation procedure. Sulfides are removed by treatment with lead acetate or powdered lead carbonate. See Empirical Laboratories Method SOP-164 for distillation procedure.

   Note: method 9012A says that sulfides "should" be treated with bismuth nitrate. Empirical Laboratories attempted to use that procedure and found that bismuth nitrate also removes cyanide from standard solutions as well. This sulfide treatment is not used. See Empirical Laboratories Method SOP-164 for distillation procedure.

III. SPECIAL APPARATUS

1. Lachat Quick Chem AE Automated Ion Analyzer

2. Cyanide manifold (Lachat)

IV SAMPLE HANDLING AND PRESERVATION:

The color reaction is pH sensitive. Therefore, distillates and standards should be carefully matched with respect to NaOH concentration. The distillates are 0.25 N NaOH.

V. SAMPLE PREPARATION

The samples for analysis come directly from the midi-scale distillation procedure.

VI. PREPARATION OF REAGENTS

Use deionized water (<10 megaohm) for all solutions.

Reagent 1. Carrier, 0.25 M Sodium Hydroxide

In a 1L vol. flask add 200 mL of 1.25N NaOH (which is used in the SOP-164 distillation method) to approximately 600 mL of D.I. water. Dilute to l L, invert to mix. This will ensure that the concentration of the NaOH used for the distillates will be the same concentration used for the carrier solution.

Reagent 2. Phosphate Buffer, 0.71 M

In a 1 L volumetric flask, dissolve 97 g anhydrous potassium dihydrogen phosphate (potassium phosphate, monobasic, anhydrous, KH_2PO_4) in approximately 800 mL water. Dilute to the mark and invert to mix. Prepare fresh monthly.

Reagent 3. Chloramine-T Hydrate

To a 500 mL volumetric flask add about 250 mL water, then add 2.0 g chloramine-T [$CH_3C_6H_4SO_2N(Cl)Na \times H_2O$]. Dilute to the mark and invert to mix. Prepare fresh daily. The shelf life of the chloramine-T can be a critical factor in the Lachat run. New Chloramine-T should be ordered every 6 months. (Analyst discretion should be used).

Reagent 4. Pyridine-Barbituric Acid Reagent

In the fume hood, place 15.0 g barbituric acid in a 1 L beaker and add 100.0 mL water, rinsing down the sides of the beaker to wet the barbituric acid. Add 75 mL pyridine (C_5H_5N) with stirring and mix until the barbituric acid dissolves. Add 15 mL concentrated hydrochloric acid (12 M HCl) and mix. Transfer to a 1 L volumetric flask, dilute to the mark, and invert to mix. This reagent is stable for approximately six months if stored in a cool, dark place.

VII. PREPARATION OF STANDARDS

Traceability

All reference materials must be assigned an Element # and labeled accordingly. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label.

All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded in the batch sheet in Element. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

Standard 1. 1000 mg CN/L [~1,000 mg CN^-/L (Stock)]

The Stock Standard is purchased from a vendor. **CAUTION: KCN is highly toxic. Avoid contact with standard solutions. Use gloves and protective equipment when handling standards and samples.** This solution is given a unique identifier.

Record preparation in Element and label standards with appropriate Element #'s.

Standard 2. Working Standard (10.0 mg CN/L)

Pipette 2.00 mL Standard 1 into a 200 mL volumetric flask. Dilute to the mark with Reagent 1, 0.25 M Sodium Hydroxide. Invert to mix.

Set of six calibration Standards:

10 mL of 10 mg/L diluted to 100 mL = 1.0 mg/L std
5.0 mL of 10 mg/L diluted to 100 mL = 0.500 mg/L std
2.0 mL of 10 mg/L std diluted to 100 mL = 0.200 mg/L std
1.0 mL of 10 mg/L std diluted to 100 mL = 0.100 mg/L std
4 mL of 0.500 mg/L std diluted to 100 mL = 0.020 mg/L std
2 mL of 0.500 mg/L std diluted to 100 mL = 0.010 mg/L std
Blank consists of 0.25 M NaOH
10 mL of 10.0 mg/L std diluted to 200 mL = 0.500 mg/L chk std

The diluent for all standards is 0.25 M NaOH.

VIII. INSTRUMENT INFORMATION (see page 12 for manifold diagram)

Analyst should confirm the following:

A. Timing:

Sample throughput: 80 samples/h; 40 s/sample
Pump speed: 35 RPM

Cycle Period: 45 s
Inject to start of peak period: 28 s
Inject to end of peak period: 61 s

B. QuikChem AE Settings:

1) Parameter, Data Window:

Top Scale Response: 0.50 abs
Bottom Scale Response: 0.00 abs

2) Segment/Boundaries

A - 0.50 mg CN/L
F - 0 mg CN/L

3) Results/Approval, Reports

In the default Report definition file (RDF), change:
-Set Default Chord 0 to
-Set Default Chord 3
(Peak should be centered in chord 3)
This change must be made to both the sample and the calibration RDF's.

C. System Notes:

1. Allow enough time for heating unit to warm-up to 60°C with D.I. water pumping through all reagent lines at full speed. (approximately 30 min.)

IX. CALCULATIONS

A. Aqueous Samples:

The Lachat reports the concentration of the final distillate in mg/L. The method detection limit (MDL) is 0.005 mg/L and the reporting limit (RL) is 0.010 mg/L.

B. Solid Samples:

The Lachat reports the concentration of the final distillate in mg/L. Results for solid samples should be in mg/kg and can be obtained by entering the appropriate dilution factor when defining the tray. Conversion from mg/L to mg/kg can be accounted for by the following formula:

$$\left[\frac{\text{mg CN}^-}{\text{Kg sample}}\right]=\left(\frac{\text{N mg}}{\text{1 L soln}}\right)\left(\frac{\text{0.50 L soln}}{\text{g sample}}\right)\left(\frac{\text{1,000 g sample}}{\text{1 Kg sample}}\right)(\text{Wet Weight})$$

From this formula a dilution factor can be obtained. For example if 2 grams of soil were used then this would be a x 250 dilution. When the x 0.50 correction factor is included a final manual dilution factor of x 125 is obtained.

N would be the value obtained from the Lachat without a correction factor. The minimum value for N is 0.01 mg/L soln.

C. Data reporting

1. Reduce data to the result which will be reported.

2. Complete the data review checklist (attached). This must be completed and attached to each set of USACE data.

X. QUALITY CONTROL (Reference SW-846, Update III and USEPA CLP ILMO 4.1 for further clarifications)

A. QA/QC, distilled:

1. PB (preparation blank)

   25 mL of DI water is distilled. When brought up to 25 mL volume following the distillation process a 0.25 M NaOH solution is obtained. A blank is distilled every batch of samples. If the absolute value of the PBW is not below the CRDL or the RL **or ½ the CRDL for Navy Projects** the analyst's supervisor should be consulted before proceeding.

2. DCV (distilled calibration verification)/ICV (distilled/CLP)

   a. The DCV high and low are check standards generally of a concentration of about 0.200 mg/L for the high and 0.050 mg/L for the low. A DCV high and low are distilled with every batch of distilled samples. There is a maximum of 20 samples in a batch. If the DCV is not within ±15 % of the undistilled value the analyst's supervisor should be consulted before proceeding. **A corrective action report (CAR) will be required if the limit is exceeded.**

   b. CLP requires the ICV to be distilled and from a second source. Must be analyzed immediately after calibration. Since the matrix for the soil LCS is Teflon chips the LCS serves as a distilled ICV. The control limit is ± 15%.

3. LCSW (laboratory control sample-water)

   The laboratory control sample is a second source check on the calibration and must be run once every 20 aqueous samples. The control limits are in-house-generated. The CLP control limits is 80 to 120%. If the LCSW is not within control limits the analyst's supervisor should be consulted before proceeding. This sample is given a unique identifier in the distillation log.

4. LCSS (laboratory control sample-soil)

   The LCSS is a second source check with a soil matrix (Teflon chips are used for this purpose) and must be run once every 20 solid phase samples. If the LCSS is not within control limits the analyst's supervisor should be consulted before proceeding. This sample is given a unique identifier in Element.

5. MS/MSD (matrix spike/matrix spike duplicate)

   A spike and spike duplicate (CLP requires 1 duplicate and 1 matrix spike) are done with each batch or 20 samples. The spike concentration is 0.10 mg/L. If the percent recovery for the MS or MSD is not within ±25 percent of the spike concentration, the analyst's supervisor should be consulted before proceeding.

6. Documentation of Capability (DOC) - Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

B. QA/QC, undistilled:

1. ICV (initial calibration verification)

   The ICV is an undistilled check standard (except when analyzing by CLP methods and then the ICV is distilled) made from the working standard solution. The concentration is usually around the midpoint of the calibration curve (about 0.500 mg/L). The ICV is analyzed at the beginning of each Lachat run. If the ICV is not within ±10 percent of its true value, the analyst's supervisor should be consulted before proceeding. The calibration verification standard must be prepared fresh daily.

2. ICB (initial calibration blank)

   The ICB is 0.25 M NaOH solution and follows the ICV at the beginning of each Lachat run. If the absolute value of the ICB is not <± 2x MDL or < ± CRDL **or ½ the RL for Navy Projects**, the analyst's supervisor should be consulted before proceeding.

3. CCV (continuing calibration verification) **From primary std. that curve is made from.**

   The CCV will be analyzed after every 10 samples and at the end of the tray. The sampling of the CCV's will be done automatically by the Lachat if it is included in the tray definition. The CCV scheduling can be done under the Data Quality Management option in the Tray Definition screen. If the CCV is not within ±10 percent of its true value a CHK STD failure signal will be given by the Lachat and the analyst's supervisor should be consulted before proceeding.

4. ICB/CCB (initial and continuing calibration blank)

   This sample verifies the instrumental baseline and must be analyzed immediately after every ICV/CCV. If the absolute value of the ICB/CCB is not below the minimum detection limit the analyst's supervisor should be consulted before proceeding.

C. Calibration Approval:

For a cyanide calibration to be approved, segment (A-F) of the curve must have a correlation of at least 0.995 in chord 3.

D. Method Detection Limit (MDL), Empirical Laboratories Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

(MDLS are performed annually or whenever a change in the method is made.)

TABLE I

Aqueous and Soil Method Detection Limits(MDL), Empirical Laboratories Reporting Limits(ERL), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)

Analytes by EPA 335.1, 335.3, 9012A , SOW 4.1 & 5.2	AQUEOUS MDL(ug/L)	AQUEOUS ERL(ug/L)	AQUEOUS CRQL ILMO 4.1 (ug/L)	AQUEOUS CRQL ILMO 5.2 (ug/L)	SOLID/SOIL MDL (mg/Kg)	SOLID/SOIL ERL (mg/Kg)	SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)	SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)
Cyanide	0.0050	0.005	10	10	0.125	0.13	0.25	0.25

TABLE II

ANALYTE WAVELENGTH	
Cyanide	**570.0**

XI. CORRECTIVE ACTIONS

A. INSTRUMENT RELATED

1. ICV not within ± 10%
   a. If the problem is with the solution.
      i. Reprepare, obtain new stock if necessary.
   b. If the problem is with the calibration.
      i. Recalibrate thru analysis of appropriate standards and recheck ICV.
2. CCV not within ± 10%
   a. If the problem is with the solution.
      i. Reprepare, obtain new stock if necessary.
   b. If the problem is with the calibration.

i. Recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.

a. If the CCV was biased high, any of the previous ten samples which were BMDL do not require reanalysis.

b. If the CCV was biased low, the previous ten samples must be reanalysed.

3. Ending CCB not ± RL or CRDL **or ½ the CRDL for Navy Projects**
   a. If the CCB is biased high.

      i. Any samples BDL or greater than 10X the CCB bias need not be reanalyzed.
      ii. Any samples above the detection limit but less than 10X the CCB level must be reanalyzed after the problem is corrected.

   b. If the CCB is biased low.

      i. Any samples greater than 10X the absolute CCB bias need not be reanalyzed.
      ii. All other samples must be reanalyzed after the problem is corrected.

B. DISTILLATION RELATED

1. The preparation blank is not less than the ± RL or CRDL **or ½ the CRDL for Navy Projects**.

   a. If the problem with the instrument.

      i. Analyze a CCB to determine this.
      ii. If the problem was with the instrument correct the situation and reanalyze the preparation blank.

   b. If the problem is with the distillation.

      i. All associated samples which are <MDL or have a level of cyanide greater than 10X the level found in the preparation blank can be reported. If the level of cyanide in an associated sample is not <MDL nor greater than 10X the level found in the preparation blank, the sample must be redistilled/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.

2. LCS not within control limits (±20%).

   a. If the problem is with the instrument.

      i. Reanalyze when instrument is in control.

b. If the problem is with the distillation.

i. If biased low, associated samples must be redistilled.
ii. If biased high, the impact upon the data user must be evaluated. The samples will be redistilled or the data will be qualified on the final report.

3. Distilled check standard not within control limits of ±15%.

a. If the problem is with the instrument.

i. Reanalyze when instrument is in control.

b. If the problem is with the distillation.

i. If biased low, associated samples must be redistilled.
ii. If biased high, the impact upon the data user must be evaluated. The samples will be redistilled or the data will be qualified on the final report.

C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20%

i. The associated sample data must be qualified on the final report.

2. Spike analysis recovery not within ±25%

i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.

ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report.

XII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

XIII. SOURCES/REFERENCES:

1. Lachat manual; Quick Chem method 10-204-00-1-A.

2. U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983, Method 335.4.

3. Standard Methods for the Examination of Water and Wastewater, 21st Edition, APHA-AWWA-WCPC, Park 413D, pp. 370-372.

4. SW-846, 9012A, Revision September 1986.

5. U.S. Environmental Protection Agency, C.L.P. S.O.W. ILMO 4.1.

6. QuickChem AE, Automated Ion Analyzer Training Manual, Lachat Instruments.

7. QuickChem AE, Automated Ion Analyzer Software Reference Manual, Lachat Instruments.

Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. The ICV shall be distilled.

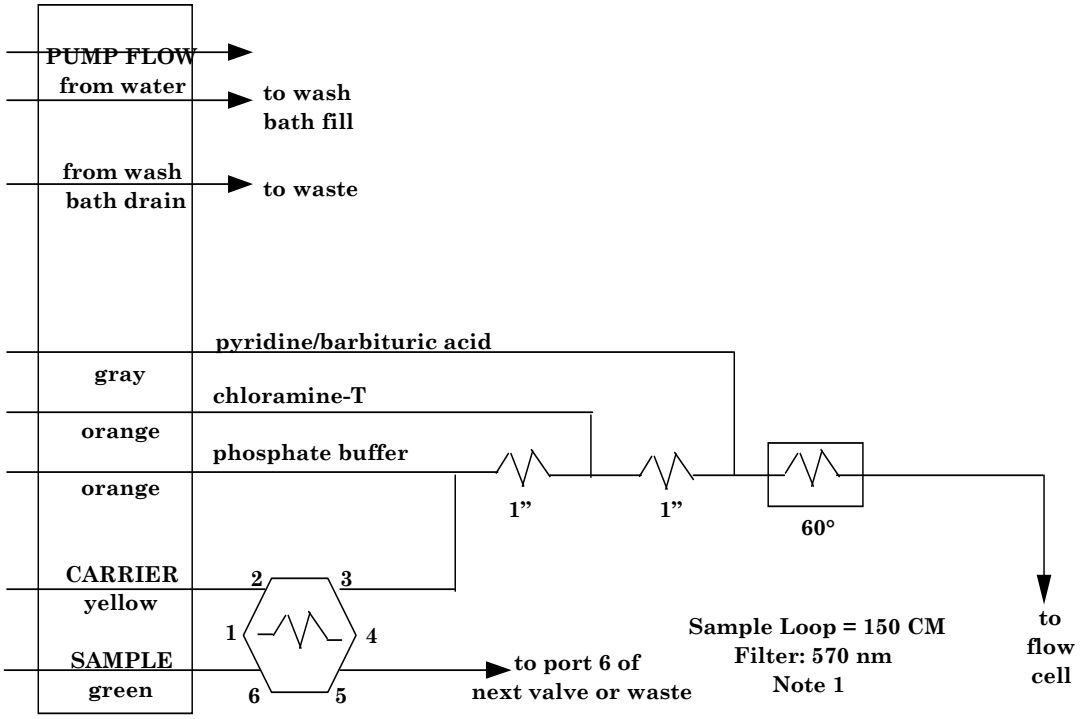
2. Boiling chips are to be added to each sample. Midi-Distillation is reflux 1.5 hours then the heat and vacuum is turned off and the samples cool an additional 15 minutes.

3. The QC criteria for both the Distillation Check QC and the distilled ICV is ±15%.

4. A CRI is required at the beginning and end of each run and for every 20 samples. The QC criteria for the CRI is ±30%.

5. Rounding rule for the appropriate level of precision is that the figure following those to be retained is $\geq$ 5, round up: otherwise round down. (examples: 1.5 and 2.5 would be 2 and 3 respectively rounded up; 1.4 and 2.4 would be 1 and 2 respectively rounded down) Please see Exhibit B, Section 3, (3.3.9.1) of SOW ILM0 5.2 for more guidance on rounding significant figures.

MANIFOLD DIAGRAM



CARRIER is 0.25 M Sodium Hydroxide

1" is 70.0 cm of tubing on a 1 in coil support

APPARATUS: Standard valve, flow cell, and detector head modules are used. The box shows 650 cm of heated tubing.

All manifold tubing is 0.8 mm (0.032 in) i.d., This is 5.2 µL/cm.

Note1: A 3.0 m x 0.022" id back pressure loop is placed at the exit of the flow cell.

Manifold Diagram Revision Date: 13 May

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	**9012A (Cyanide)**

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did LCS or blank spike meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was distillation temperature monitored/documented and did you apply the thermometer correction factor?				
10. Sample preparation information is correct and complete.				
11. Analytical results are correct and complete.				
12. The appropriate SOP's have been used and followed.				
14. "Raw data" including all manual integration's have been correctly interpreted.				
15. "Special" sample preparation and analytical requirements have been met.				
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.				

ANALYST DATA REVIEW CHECKLIST
9012A (Cyanide)

Comments on any "No" response:

_____
_____
_____
_____
_____
_____

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 202 **REVISION #: 22** **EFFECTIVE DATE: 093009**

GC/MS VOLATILES BY EPA METHOD E624 & SW846 METHOD 8260B INCLUDING APPENDIX IX COMPOUNDS

APPROVALS:

Lab Director: _____ Date: 10/5/09

Data Quality Manager: _____ Date: 10/5/09

Section Supervisor: _____ Date: 10/7/09

Changes Summary

Revision 22, 9/30/09

- The SOP is an update from Revision 21 dated 09/11/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

1. Identification of the Test Method

1.1 This SOP is compliant with methods – EPA Method 624 and SW-846 Method 8260B

2. Applicable Matrix or Matrices

2.1 This SOP is applicable to – The analysis of volatile organic compounds in a variety of matrices including but not limited to soils, sediments, ground and surface waters, aqueous sludge, oily wastes, etc.

3. Detection Limit: See Table 1 of this SOP.

4. Scope of Application, Including components to be Analyzed

4.1 This SOP is based primarily on SW-846 Method 8260B. Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

5. Summary of the Test Method

5.1 After sample preparation, the sample is introduced into the GC/MS generally using purge and trap but sometimes using direct injection (see SW-846 Methods 5030B, 5035 and 3585 for preparation). In purge and trap, the analytes are stripped from the sample using helium and trapped on an adsorbent tube. The tube is heated while being backflushed with helium to carry the analytes to the GC/MS system. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra of the sample. Analytes are quantitated relative to known standards using the internal standard method.

6. Definitions

6.1 Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2 Additional definitions specific to this SOP are listed below:

amu	atomic mass unit
BFB	Bromofluorobenzene

°C	degrees Centigrade
CLP	Contract Laboratory Program
DOD	Department of Defense
EICP	extracted ion current profile
G	gram or grams
GC/MS	Gas Chromatograph/Mass Spectrometer
I.D.	inner diameter
ISTD	internal standard
µm	micometer
µL	microliter
mL	milliliter
mm	millimeter
ng	nanogram
P&T	purge and trap
SURR	surrogate
SPCC	System Performance Check Compound
TCLP	Toxicity Characteristic Leaching Procedure
USACE	United States Army Corps Of Engineers
VOA	volatile organic analysis

7. Interferences

7.1 Section 3.0 of SW-846 Method 8260B details interferences and potential problems which may be encountered when dealing with volatile analyses.

8. Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

9. Equipment & Supplies

9.1 GC : HP 5890 or 6890, temperature programmable, suitable for split or splitless injection.

9.2 Column: DB-VRX 60 meter x 0.25 mm I.D. 1.4 µm film thickness or 20 meter x 0.18 mm ID 1.0 µm film thickness silicon coated fused silica capillary column or equivalent.

9.3 M.S. : HP 5971, 5972 or 5973 capable of scanning 35 to 500 amu every one second or less, using 70 volts electron energy in electron impact ionization mode. The MS is capable of producing a mass spectrum for p-Bromofluorobenzene, BFB, which meets all tuning criteria for EPA methods [when 1 µL (50 ng) of the GC/MS tuning standard is introduced to the GC.]

9.4 Purge and Trap Unit

9.4.1 Concentrators: Tekmar LSC 2000 or Tekmar/Dohrmann 3000/3100 Sample Concentrator equipped with Supelco trap number 2-1066-U or 2-4920-U VOCARB 3000 providing good delivery for all target compounds.

9.4.2 Autosamplers: Varian Archon 51 position programmable autosampler with 5ml to 25ml water and heated soil capability.

9.5 Acquisition Software: HP chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

9.6 Data Processing Software: TargetDB on Windows NT data system interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances of any EICP between specified time or scan-number limits. NBS75K mass spectral library is installed.

9.7 Microsyringes – 1.0, 5.0,10, 25, 100, 250, 500 and 1000 µL.

9.8 Syringes – 5, 25 and 50 mL, gas-tight with Luer end.

9.9 Balance - analytical, 0.0001 g; top-loading, 0.01 g.

9.10 Disposable pasteur pipets.

9.11 Volumetric flasks, Class A - 2 mL, 5 mL, 10 mL, 50 mL, 100 mL and 250 mL with ground-glass stoppers.

9.12 Spatula - stainless steel.

9.13 Glass scintillation vials - 20mL with screw caps.

9.14 Nitrile Gloves

9.15 pH paper (measures pH from 0-14).

10. Reagents and Standards

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

10.2 Organic-free reagent water - obtained from a modulab system.

10.3 Methanol - Purge and trap grade (EM-Omnisolv EM-0482-6 or equivalent)

10.4 Methanol - suitable for use in gas chromatography (B&J Omnisolv MX0484-1, or equivalent)

10.5 Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).

10.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the LIMS system along with their lot number and vendor and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date**. All stocks and standards are stored in the freezer at a temperature of -15°C $\pm$ 5°C or less from the date they are received/prepared. The freezer

temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the VOA refrigerator/freezer logbook. Makeup of common standards is detailed below. See standard ID in LIMS system for makeup of other standards.

10.6.1 The Bromofluorobenzene (BFB) tuning standard is prepared as follows: Using a 50µL syringe, 40µL of standard (BFB @ 2500ng/µL) is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same making a 50ng/µL standard. After capping and inverting 3 times, the solution is transferred to a labeled 2ml, teflon-lined, screw-capped vial and stored in the freezer at -15°C ± 5°C or less for up to 6 months **(1 week for South Carolina samples)**. A direct injection of 1µL (or equivalent purge) is used to tune the instrument.

10.6.2 The internal and surrogate standards are prepared as follows: Using the indicated syringe, the indicated amount of standard is injected into a 50 mL volumetric flask containing P&T methanol (Vendor,Lot) and diluted to volume with same making a 150ng/µL standard. After capping and inverting 3 times, the solution is transferred to the Archon standard vial and stored under helium for 1 month or less. Each 8260/624 sample is automatically injected with 1µL of this standard. The internal standard/surrogate solution will be replaced if the –50%-200% criteria fails in the CCV when calculated against the previous CCV.

Standard	Conc. (ng/µL)	Syringe (µL)	Amount (µL)
8260 ISTD Mix	2500	1000	3000
Surr. Mix	2500	1000	3000

10.6.3 Calibration standards are prepared from the vendor stock standards at appropriate concentrations as follows. Occasionally unusual compounds are added to the mix so it is best to check the VOA standards log book for exact standard makeup. Note: for laboratory control spikes (LCS), alternate sources or lot numbers from the main calibration standard are used to make the LCS standard. See the appendix for analytes in the main mixes.

10.6.3.1 Primary Standard: Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50µg/L (5mL purge) standard is made using 25µL of this standard to 50mLof reagent water.

Stock Standard(CCV)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
2-CEVE **(Cat#30265)**	20000	25	20	200
Vinyl Acetate **(#3766)**	5000	100	80	200
Ketones **(cat#30006)**	5000	100	80	200
Liquid mix **(C-349H-07)**	2000	100	100	100
Custom mix **(CCS-1037)**	5000	50	40	100
Gases **(cat#30042)**	2000	100	100	100
Acrolein/Acrylonitrile **(CC2098.10)**	20,000	50	50	500

Additional compounds may be added such as Appendix IX. Refer to standard ID in LIMS system.

10.6.4 ICV/LCS/Matrix Spike Mix: A second source standard is used to check the validity of the gas and primary calibration standards used in analyzing the calibration curve. Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50µg/L ICV/LCS/Matrix Spike is made using 25µL of this standard to 50mL of reagent water/Sample Matrix.

Stock Standard(ICV/LCS)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
2-CEVE	20,000	25	20	200
Vinyl Acetate	5000	100	80	200
Ketones	5000	100	80	200
Liquid mix	2000	100	100	100
Custom Mix	5000	50	40	100
Gases	2000	100	100	100
Acrolein/Acrylonitrile	50,000	50	50	500

11. Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage. All water samples are stored in the BlueIce refrigerator in the VOA lab at a temperature of 4°C. All unpreserved soil samples in TerraCore or encores are stored in the freezer in the VOA lab. All soil samples in bulk jars or chemically preserved TerraCore are stored in the soil walk-in refrigerator at a temperature of 4°C. Non-preserved water volatile samples have a holding time of 7 days from date of sampling. Preserved water samples and soil volatile samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project).

12. Quality Control

12.1 Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.1 Internal Standards - All samples and QC are spiked with internals. See **Table 2** for criteria and corrective action.

12.2 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are used to generate control limits. See section 14.5.2 of this SOP for criteria and corrective action. **When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**

12.3 LCS Sample - An LCS is analyzed every 12 hour tune. To prepare the LCS, a blank is spiked with standards prepared from an alternate vendor or lot number from the calibration standards. Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). The recoveries are used to generate control limits. The limits are in-house generated matrix spike limits or client specified limits for matrix spike analytes and 70-130% (or client specified limits) recovery for waters or soils for all other analytes if limits have not been generated. Limits for 624 LCSs are taken from table 5 of method 624. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reanalyzed for the failed analytes only. **When analyzing samples for DOD QSM Version 4.1, DOD limits will be used. South Carolina limits are 70-130% except for poor purgers which are 60-140%.**

12.4 Method Blanks - The concentration for method target analytes must be < ½ the Reporting Limit (also defined as the Limit of Quantitation). The first step of corrective action is to assess the affect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, would likely be reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank. A method blank is analyzed every 12 hour tune.

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for an MS/MSD with the LCS standard. Criteria for the MS/MSD recoveries are the same as the LCS limits. Limits for the RPDs are 30% RPD for water and soil.. Samples that do not meet these criteria due to matrix must be flagged on the final report for QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the

failed analytes only. MS data evaluation must include the consideration of the following factors. **When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**

12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. A water sample which was taken from the same VOA vial for the original sample and the MS/MSD should have very good RPDs unless there has been a method problem. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

12.5.3 MS vs. MSD - If a spiked compound has a problem in both the MS and MSD, review the LCS and if acceptable no further action may be necessary since it is attributable to matrix effect.

12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

13. Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Chromatographic conditions – Refer to corresponding instrument maintenance log for current gas chromatograph, mass spectrometer, and concentrator conditions.

13.3 System Bakeout - Prior to analysis an instrument blank is analyzed.

NOTE: Further cleaning may be accomplished by backflushing the lines with methanol and then analyzing blanks overnight.

13.4 Tuning - Prior to any calibration or analysis, BFB tuning criteria must be met for a 1.0µL injection of the tuning standard [see below]. Tune must be met every 12 hours sample analysis is to be performed **(every 24 hours for *Federal Register* Method 624 except for South Carolina which only allows 12 hours).** The mass spectrum of BFB is acquired as follows: by using the BFB method in Target (which uses three scans with backround subtraction) to process the BFB data file. If the BFB tune does not pass criteria corrective action should be taken

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

13.5 **Calibration:** Calibration standards are made up in water using the appropriate amount of the methanol standard. **Calibration for soils for South Carolina requires that 5mL of sodium bisulfate solution is added to each calibration standard made if the samples will be preserved with sodium bisulfate.** All calibration standard manual integrations must be approved by for acceptability.

13.5.1 Initial Calibration - An initial calibration curve at no less than five (six if using a quadratic curve fit) concentration levels must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. **For Arizona samples the surrogates must also be calibrated at a minimum of five concentrations.** Method 624 requires that the %RSD be less than 35% to use the average response factor for quantitation, the curve is to be used otherwise and should have a correlation coefficient (***r***) of $\geq$0.995. Method 8260B requires that the %RSD be less than 15% to use the average response factor for quantitation, the curve is to be used otherwise as long as ***r*** is $\geq$0.995 linear or $\geq$ 0.99 quadratic. In addition, there are calibration check compounds (CCCs) listed below which must have a %RSD less than 30% and five system performance check compounds (SPCCs) which must meet the average response factor criteria listed below. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All calibration manual integrations must be approved by management. Any response factors less than 0.050 must be supported by the mass spectrum of the lowest standard. **No quadratic curves for South Carolina.**

CCCs:	1,1-Dichloroethene	Toluene
	Chloroform	Ethylbenzene
	1,2-Dichloropropane	Vinyl chloride

SPCCs:	Chloromethane	0.10
	1,1-Dichloroethane	0.10
	Bromoform	0.10
	Chlorobenzene	0.30

1,1,2,2-Tetrachloroethane 0.30

13.5.2 Initial Calibration Verification - A second source standard at the 50 µg/L (5mL purge) level is used to check the validity of the curve. The standard recovery for all analytes must be between 75 and 125%. **When analyzing samples for DOD QSM Version 4.1, DOD limits (80-120%) will be used.** If the second source recovery is above 125%, the main standard has probably deteriorated for that compound. That standard must be replaced and a new curve generated. If the second source recovery is below 75%, the second source standard has probably deteriorated for that compound and must be replaced. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All calibration manual integrations must be approved by management

13.5.3 Continuing Calibration Verification (every 12 hours) - A midpoint calibration standard (generally 50 µg/L - 5mL purge) must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. Acceptance criteria for method 8260B consists of the same SPCC criteria as above and ≤20% drift or difference (calculations given in section 7.10) for the CCCs as listed above. The internal standards must also be evaluated as listed below. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All calibration manual integrations must be approved by management. Samples are then quantitated against the initial calibration curve. Note: If any compound in the continuing calibration not subject to the criteria above exceeds 30% D, it must be evaluated following the guidelines outlined in SOP QS05. If deemed acceptable, the analyst may continue analysis. **When analyzing samples for DOD QSM Version 4.1, DOD acceptance criteria of ≤ 20% drift or difference for all analytes will be used.**
NOTE: Acceptance criteria for method 624 consists of meeting recovery limits found in table 5 of the method for a QC check sample. This QC check sample is made from a separate source or lot number than the calibration standard at a concentration of 20 µg/L.

Internal standard checks

13.5.3.1 Retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

13.5.3.2 Response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +

100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

14. **Procedure**

14.1 LCS - An LCS is analyzed every 12 hour tune.Using standards prepared from an alternate vendor or lot number, blank water is spiked at the 50 µg/L (5mL/soil) or 10 µg/L (25mL) level. See section 12 above for criteria and corrective action. **Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**

14.2 Method Blank - Prior to sample analysis, the system must be shown to be free of contamination through analysis of a method blank. See **Table 2** for criteria and corrective action.

14.3 Sample Analysis - Prior to analysis, the samples are prepared for chromatography using the appropriate sample preparation method (5mL water, 25mL water, low soil, high soil, etc.) See SOP 225 for preparation of a 5035 soil sample. For a 5mL/25mL water sample, use the following procedure:

14.3.1 Load the vial into the Archon autosampler in the expected position.

14.3.2 Program the Archon for the loaded vial range and necessary dilutions, making sure the programmed method is set for the same volume as the purge vessel on the front of the LSC 2000 or 3000/3100 and that the Chemstation sequence matches the Archon sequence. Note: TCLP samples are analyzed at a 10x dilution. One TCLP sample is spiked per batch at receipt of leachates.

14.3.3 After analysis of the sample has been completed, check the pH of the sample using pH paper and verify it to be less than a pH of 2 (recorded on the sequence log). If it is not, record the pH on the sequence log and generate a corrective action report. The sample report will have to be qualified for preservation if the analysis is being performed more than 7 days after sampling. [Note: TCLP samples do not require a pH check.]

14.4 **Instrument sequence**

An example of a typical instrument sequence log follows:

1-BFB Tune (12:00 am)
2-CCV
3-LCS
4-Method Blank
5-Sample
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample
15-Sample
16-Sample
17-Sample MS
18-Sample MSD
19-BFB (12:00pm - 12 hours since last BFB/CCV)
20-CCV
21-LCS
22-Method Blank
23-Sample
24-Sample

14.5 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through the TargetDB on Windows NT data system. Quantitative measurements are performed using the calculations found in section 15.2 of this SOP. The following must be checked to determine if the sample will need any reanalysis or dilution. Formal data evaluation is detailed in SOP QS05. **See SOP QS07 for guidance on manual integrations.**

14.5.1 Internal Standards - Areas should be within 50 to 200 percent of the area of the continuing calibration. Retention time should be within 30 seconds of the retention time of the continuing calibration. Note: criteria applies to the continuing calibration, not samples, but is used as an indication of the sample analysis validity. If not, the sample and historical data should be evaluated to determine the cause of the problem. Reanalysis is expected if it appears to be from a leak. If matrix effect is confirmed by reanalysis or historical data, complete a corrective action report and flag the affected compounds on the final report for matrix effect.

14.5.2 Surrogates – Control limits are determined by charting LCSs and method blanks. All of the surrogates must be within these limits in order for the analysis to be in control. If not, the reason for the malfunction must be determined and reanalysis may be necessary. If historical data indicates matrix, the sample would be flagged appropriately. When the surrogates exceed either the control limits, a corrective action report must be completed.
Federal Register **Method 624 contains no criteria for surrogate recovery. When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**

Surrogate	**WATER**	**SOIL/SEDIMENT**
Dibromofluoromethane	85-120	80-125
1,2-Dichloroethane-d4	85-135	75-140
Toluene-d8	85-115	80-120
Bromofluorobenzene	80-120	80-125

14.5.3 Analyte concentration must be within the range of the calibration curve after rounding to 2 significant figures. If an analyte exceeds the curve, a dilution must be performed, the next sample must be checked for carryover and the sparge position must be checked for contamination through the analysis of a system blank. Any dilution should keep the concentration of the analyte in question within the mid-range of the curve.

14.5.4 Qualitative identification is made as indicated below.

14.5.4.1 The mass spectra are compared to reference spectra in a user-created data base especially compiled to contain relatively uncontaminated mass spectra of each target compound. Note: Such a file cannot be obtained from the daily calibrations during each 12 hour analytical period due to overlapping peaks in the mixes.

14.5.4.2 The GC/MS analyst uses intelligence guided by experience to make the identifications.In uncontaminated spectra where ions are missing due to low concentration, if the major ions are present in the correct ratios at the correct retention time, the identification will be considered positive. In contaminated spectra, special emphasis will be placed upon higher mass ions, and the major ions will usually need to be present as major components of the spectrum (either unsubtracted or subtracted) for the identification to be positive. All assessments of relative intensities of ions will be made by visual estimates from the spectra.

15. Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Calculations:

15.2.1 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

15.2.2 Calibration verification involves the calculation of the percent drift (linear) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±20% for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

15.2.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume purged in ug/L.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = For purge-and-trap analysis, V_i is not applicable and is set at 1.

$\overline{RF}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample purged (mL). If units of liters are used for this term, multiply the results by 1000.

15.2.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s, A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples.

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

16. Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

17. Pollution Prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. **Data Assessment and Acceptance Criteria for Quality Control Measures**

18.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. **Contingencies for Handling out-of-control or unacceptable data**

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20. **Waste Management.**

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. **References**

21.1 40 CFR, Part 136; Appendix A
21.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition and updates
21.3 National Environmental Laboratory Accreditation Conference; CH. 5, 2001
21.4 USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
21.5 DOD Quality Systems Manual for Environmental Laboratories version 3, 3/2005
21.6 DOD Quality Systems Manual for Environmental Laboratories version 4.1, 4/2009

22. Tables, Diagrams, Flowcharts and Validation Data

TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard				
Parameter	**RL Water ug/L**	**LowCal Water ug/L**	**RL Soil ug/KG**	**LowCal Soil ug/KG**
1,1,1 Trichloroethane	1.0	1.0	5.0	2.0
1,1,1,2-Tetrachlorethane	1.0	1.0	5.0	2.0
1,1,2,2-Tetrachloroethane	1.0	1.0	5.0	2.0
1,1,2-Trichloroethane	1.0	1.0	5.0	2.0
1,1-Dichloroethane	1.0	1.0	5.0	2.0
1,1-Dichloroethene	1.0	1.0	5.0	2.0
1,2,4 Trichlorobenzene	1.0	1.0	5.0	2.0
1,2-Dibromo-3-chloropropane	1.0	1.0	5.0	2.0
1,2-Dibromoethane	1.0	1.0	5.0	2.0
1,2-Dichlorobenzene	1.0	1.0	5.0	2.0
1,2-Dichloroethane	1.0	1.0	5.0	2.0
1,2-Dichloropropane	1.0	1.0	5.0	2.0
1,3-Dichlorobenzene	1.0	1.0	5.0	2.0
1,4-Dichlorobenzene	1.0	1.0	5.0	2.0
2-Butanone	10	2.0	50	4.0
2-Hexanone	5.0	2.0	10	4.0
4-Methyl-2-pentanone	5.0	2.0	10	4.0
Acetone	10	2.0	50	4.0
Benzene	1.0	1.0	5.0	2.0
Bromochloromethane	1.0	1.0	5.0	2.0
Bromodichloromethane	1.0	1.0	5.0	2.0
Bromoform	1.0	1.0	5.0	2.0
Bromomethane	2.0	1.0	10	2.0
Carbon disulfide	1.0	1.0	5.0	2.0
Carbon tetrachloride	1.0	1.0	5.0	2.0
Chlorobenzene	1.0	1.0	5.0	2.0
Chloroethane	2.0	1.0	10	2.0
Chloroform	1.0	1.0	5.0	2.0
Chloromethane	2.0	1.0	10	2.0
Cis-1,2-Dichloroethene	1.0	1.0	5.0	2.0
Cis-1,3-Dichloropropene	1.0	1.0	5.0	2.0
Dibromochloromethane	1.0	1.0	5.0	2.0
Dibromomethane	1.0	1.0	5.0	2.0
Dichlorodifluoromethane	2.0	1.0	10	2.0
Ethylbenzene	1.0	1.0	5.0	2.0
Methylene chloride	2.0	1.0	10	2.0
M,p-Xylene	1.0	2.0	5.0	4.0
o-Xylene	1.0	1.0	5.0	2.0
Styrene	1.0	1.0	5.0	2.0

TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard				
Parameter	**RL Water ug/L**	**LowCal Water ug/L**	**RL Soil ug/KG**	**LowCal Soil ug/KG**
Tetrachloroethene	1.0	1.0	5.0	2.0
Toluene	1.0	1.0	5.0	2.0
Trans-1-2 Dichlorethene	1.0	1.0	5.0	2.0
Trans-1-3-Dichloropropene	1.0	1.0	5.0	2.0
Trichloroethene	1.0	1.0	5.0	2.0
Trichloroflurorometh ane	2.0	1.0	10	4.0
Vinyl chloride	2.0	1.0	10	4.0
MTBE	1.0	1.0	5.0	2.0
Naphthalene	1.0	1.0	5.0	2.0

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
Tune	At the beginning of sequence and every 12 hours	See section 13.4 for criteria.	• Follow guidelines from SOP QS05
Calibration Curve	• Prior to analyzing any samples • A minimum of 5-points for linear fits • A minimum of 6-points for quadratic fits • Low standard at or below the RL/LOQ level	• For Linear or Quadratic calibration fits a RF of 0.995 • Average RSD for CCCs $\leq$ 30%, to use avg. RF $\leq$ 15%, Min. RF for SPCCs per method • Manual integrations on curve standards must have supervisory approval • Must follow curve processing requirements from SOP QS08	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICV	Alternate source standard to be analyzed after every calibration curve	75-125% for 8260B, 80-120% for DOD QSM 4.1	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	• At the beginning of every sequence • Every 12 hours	See section 13.5.3 for criteria.	• Follow guidelines from SOP QS05
MB	One per prep batch	• Must be < ½ the RL/LOQ	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
LCS	One per prep batch	Most stringent criteria listed within the LIMS.	• Follow guidelines from SOP QS05
LCSD	One per prep batch, when MS/MSD not included.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
Internal Standard	• A mix is used per sample post - prep	• 50 – 150 % of the IS from CCV	• If holding time is expired, fill out a NCR and follow directions from PM • Evaluate sample matrix and other applicable results to determine if re-analysis is required at a dilution • Re-injection or analysis • Re-prep followed by re-analysis • Follow guidelines from SOP QS05
Surrogates	• A mix is used per sample prior to sample prep	Criteria listed within LIMS or specified by client.	• If holding time is expired, fill out a NCR and follow directions from PM • Evaluate sample matrix and other applicable results to determine if re-analysis is required at a dilution • Re-injection or analysis • Re-prep followed by re-analysis • Follow guidelines from SOP QS05
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Must meet the criteria of the LCS for average accuracy	• Re-prep and /or re-analysis
MDL Study	Once per year	○ Calculated value must be greater than 10% of the Spike Level ○ Calculated value must be less than the Spike level	• Re-prep and /or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	○ Parameter must be detected ○ Check for Ion Abundance on MS methods ○ the response must be 3-times the noise level	• Re-prep and /or re-analysis • Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
LOQ Verification	Every quarter	○ Bias Requirement: Inorganics 50-150% Organics 10-150% ○ The LOQ value must be greater than the LOD value	• Re-prep and /or re-analysis • Follow guidelines from SOP QS05

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory approval for manual integrations on standards and QC samples
7. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 211 **REVISION #: 22** **EFFECTIVE DATE: 070710**

GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD) ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS (PCB) BY EPA METHOD 608/608.2 or SW846 METHOD 8081A/8082 or 8081B/8082A

APPROVALS:

Lab Director: _____ Date: 7/8/10

Data Quality Manager: _____ Date: 7/7/10

Section Supervisor: _____ Date: 7/7/10

Changes Summary

Revision 22, 07/07/10

- The SOP is an update from Revision 21 dated 04/11/10.
- The SOP has been updated to move specific requirements to tables at the back of the SOP and add Mirex, PCB-1262, PCB-1268 as analytes.

Revision 21, 04/11/10

- The SOP is an update from Revision 20 dated 04/27/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flow charts and Validation Data

1.0 Identification of the Test Method

This SOP is compliant with SW-846 Methods 8000B/8081A/8082 and 8000C/8081B/8082A. *Federal Register* Method 608/608.2 and CLP Method for Pesticides have also been used in the development of this SOP.

2.0 Applicable Matrix or Matrices

This Standard Operating Procedure, SOP, is used for the analysis of Pesticide/PCB organic compounds in a variety of matrices (soils, sediments, waters, etc.).

3.0 Detection Limits

See Table1.

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Detection Limit/Method Detection Limit, Limit of Detection and Reporting Limit/Limit of Quantitation for each analyte.

4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the ECD. Pesticide analytes are identified and confirmed based on the retention time of known standards. PCB and multi-component pesticide analytes are identified based on pattern recognition. Analytes are quantitated relative to known standards using the external standard method.

6.0 Definitions

Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

Section 3.0 of SW-846 Methods 8081A/8082 and Section 4.0 of Methods 8081B/8082A details interferences and potential problems which may be encountered when dealing with pesticide/PCB analyses. Please see sample clean-up SOPs (307, 308, 309 and 330) to evaluate possible clean-up options for any encountered interferences.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

8.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.

8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.4 MSDS sheets are available for all reagents and standards that have been purchased. These are located on the bookshelves in the Quality Assurance Officer's office.

9.0 Equipment & Supplies

9.1 GC's:

9.1.1 Agilent 6890N- complete with temperature programmable gas chromatograph suitable for split/splitless injection.

9.2 Columns:

9.2.1 Restek Siltek Guard Column (or equivalent): 10 meter x 0.32 mm ID

9.2.2 RTX-CLP or ZB-MR1 (or equivalent): 30 meter x 0.32 mm ID x 0.5 µm film thickness fused silica column.

9.2.3 RTX-CLP II or ZB-MR2 (or equivalent): 30 meters x 0.32 mm ID x 0.5 µm film thickness fused silica column.

9.3 Autosamplers:

9.3.1 Agilent 7683 autosamplers capable of reproducibility from one injection to another, proven by meeting QC and calibration criteria.

9.4 Acquisition Software: HP Chemstation system is interfaced to the GC. The system acquires and stores data throughout the chromatographic program.

9.5 Data Processing Software: Target DB Windows data system is interfaced to the HP Chemstation. The system accepts, processes and stores acquired data.

10.0 Reagents and Standards

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the reagents and standards used for the performance of the method. See **Table 5** for information on standard sources/calibration concentrations.

10.2 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the COA and recorded in the LIMS. The date they are opened is recorded in the LIMS along with their lot number and vendor and given a sequential number. Each standard that is prepared is recorded in the LIMS and given a sequential number. The following are noted in the LIMS: standard makeup, solvent used, date received, date opened, date prepared, expiration date and analyst. Each standard label is completed with the standard number, name, concentration, expiration date, and analyst initials. All stocks and standards are stored in the refrigerator at a temperature of 1°C-4.4°C from the date they are received/prepared. The refrigerator and freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the GC refrigerator temperature logbook.

10.3 List of Reagents:

Hexane - pesticide quality or equivalent.

11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 and table 3-1 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling while soil samples

have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed.

12.0 Quality Control

12.1 Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2 Surrogates - All samples and QC are spiked with surrogates prior to extraction. See **Table 2** for criteria and corrective action.

12.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples to provide accuracy results. It is spiked using an alternate source or lot number than the calibration standards. See **Table 2** for criteria and corrective action.

12.4 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See **Table 2** for criteria and corrective action.

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, if sample is available. If no sample is available, an LCSD must be extracted to provide precision results. See **Table 2** for criteria and corrective action. Some factors that may affect MS/MSD results are:

12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

12.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a non-conformance report to document the problem.

12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

12.6 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

13.0 Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 See Section 14.4 for Calibration details.

14.0 Procedure

14.1 The GC/ECD should be primed by injecting a pesticide standard at 200-500 µg/L and/or PCB standard at 2,500 µg/L, 10 times more concentrated than the mid-level standard. Inject this prior to beginning initial or daily calibration.

14.2 Chromatographic conditions:

14.2.1 ZB MR1/MR2 columns:

GC	ECD3
Purge on	60ml/min at 0.50 min.
Injector/Detector temperature	250/340°C
Column flow	3.0 mL/min
Initial column temperature	100°C for 1.0 minutes
Temperature ramp	35°C/min
Intermediate column temperature	220°C for 0.0 minutes
Second Temperature Ramp	15°C/min
Final Column Temperature	340°C for 2.0 minutes

14.2.2 ZB MR1/MR2 columns:

GC	ECD4
Purge on	60ml/min at 0.50 min.
Injector/Detector temperature	250/350°C
Column flow	3.0 mL/min
Initial column temperature	100°C for 1.0 minutes
Temperature ramp	35°C/min
Intermediate column temperature	220°C for 0.0 minutes
Second Temperature Ramp	15°C/min
Final Column Temperature	340°C for 2.0 minutes

Note: Current gas chromatograph conditions can be confirmed in the corresponding maintenance log.

14.3 Eval Mix – Before pesticide calibration and/or sample analysis, a degradation check standard (evaluation mix) of endrin and 4,4'-DDT must be injected. Degradation of either compound must not exceed 15 percent. See **Table 2** for criteria and corrective action.

14.4 Calibration - (See SW-846 Method 8000B Section 7.4 or Method 8000C Section 9.3).

14.4.1 Initial Calibration - An initial multi-point calibration curve must be prepared in hexane, analyzed and shown to meet the initial calibration criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 5** for standard concentrations/sources and below for makeup of the intermediates. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. For single component pesticides and surrogates, a seven point calibration is injected and analyzed for each analyte of interest. For Toxaphene and Technical Chlordane a single low calibration point standard is analyzed unless they are expected/detected then a six-point calibration is injected and analyzed. Initial calibration for Aroclors may be accomplished by using a six-point curve that contains Aroclors 1016 and 1260. The mixture of these two Aroclors contains many of the peaks represented in the other Aroclor mixtures (1221, 1232, 1242, 1248, 1254, 1262 & 1268). Full calibration is required if they are expected/detected. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

<u>Mix A/B (and Surrogate) Calibration Intermediate Solution</u>: Using a 500µL syringe, 500µL of A/B Mix and 500µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 10 µg/mL standard.*

<u>Mirex (and Surrogate) Calibration Intermediate Solution</u>: Using a 500µL syringe, 100µL of Mirex and 50µL Surrogate are injected into a 10mL volumetric flask

containing approximately 9.5mL hexane and diluted to volume with same to make a 1 µg/mL standard.*

Technical Chlordane (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 100µL of Technical Chlordane and 500µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 10 µg/mL standard.

Toxaphene (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 500µL of Toxaphene and 250µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 50 µg/mL and 5ug/ml standard.*

Aroclor 1016/1260 (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 500µL of Aroclor 1016/1260 and 250µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 50 µg/mL and 5ug/ml standard.*

*After capping and inverting several times, all solutions are transferred into labeled, 12ml, teflon-lined, screw-capped vials and stored in the refrigerator at 4°C or less for up to 6 months. These standards are used to make the calibration curve standards in hexane at the concentrations found in table 5.

14.4.2 Initial Calibration Verification - A second source standard must be prepared in hexane, analyzed and calculated against the initial calibration curve, then shown to meet the ICV criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 5** for standard concentrations/sources. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

14.4.3 Continuing Calibration Verification (CCV) - Every 12 hours (and at the end of the analysis sequence), a CCV must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 5** for standard concentrations/sources. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

14.4.4 RT Windows - Retention time criteria set forth in SW-846 method 8000B Section 7.6 are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program) and at initial calibration using the midpoint standard RTs. If the established retention time window is less than +/-0.03 minutes, the window defaults to +/-0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.

14.5 Samples - Prior to using Method 608, SW-846 8081A, 8081B, 8082, 8082A or CLP (pesticide method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3541, 3546, 3640, 3550, 3580, EPA method 608 or CLP).

14.5.1 Example of a sequence run log:

1-Primer A/B Mix-1000 or Primer PCB-10,000
2- EVAL Mix (Pest only)
3- CCV A/B Mix
4- CCV Toxaphene (single point)
5-CCV Chlordane (single point)
6- CCV PCB 1660
7- Method Blank
8-LCS A/B Mix
9-LCS PCB
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample
15-Sample
16-Sample
17-Sample
18-Sample
19-Sample
20-Sample
21-Sample-MS
22-Sample-MSD
23-Sample
24-Sample
25-Sample
26-Sample
27-Sample
28-Sample
29- CCV A/B Mix
30-CCV PCB

14.6 Data Reduction/Evaluation - Each sample analysis sequence is documented in the run logbook for the instrument. After the sample has been analyzed, the data is processed through the Target DB Windows data system. Quantitative measurements are performed as described in SW-846 8081A Section 7.5.6, and SW-846 8081B Section 11.5.6.1. The following must be checked to determine if the sample will need any reanalysis, cleaning or dilution. Criteria and corrective action are found in Table 2. Formal data evaluation is detailed in SOP QS05 and documented using the Analyst Data Review Checklist (see Appendix). Manual integration guidance is found in SOP QS07.

14.6.1 Analyte concentration after rounding to 3 significant figures must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the mid-range to the top half of the curve.

14.6.2 If the sample shows signs of sulfur contamination in the time range where sulfur compounds elute a sulfur cleanup is required [see SOP-307].

14.6.3 If the sample has extraneous peaks eluting in the chromatogram an acid cleanup is required for PCB samples and may be applicable for certain pesticides, (acid clean-up may be required for all PCB samples, check with your supervisor), [see SOP-308].

14.6.4 Analyte quantitation verification.

14.7 Identification/Quantitation [See SW-846 method 8081A Section 7.6 or method 8082 Sections 7.7-7.9].

14.10.1 Single peak components are identified by retention time on a primary column with confirmation by retention time on a secondary or confirmation column. Which column is used for primary/confirmation is determined by the chromatography in the region of the compound.

14.10.1.1 Due to coelution of certain compounds confirmation for all analytes may not be achieved. The analyst must use experience and judgment to decide if the compound is there. If a call is made, the data should be qualified appropriately.

14.10.1.2 If a compound is outside of its window on one column but in the window on the other column, the analyst will need to use their judgment or seek guidance from the organic lab manager or another experienced analyst to determine if the analyte is present.

14.10.2 Multi-peak components (PCB's, Toxaphene and Technical Chlordane) are identified by pattern recognition using an on scale standard chromatogram to compare to an on scale sample chromatogram enabling the analyst to judge whether the sample pattern matches a standard pattern. Confirmation of multi-peak components is required by the method and may be accomplished in several ways. If the sample is from a source known to contain specific Aroclors then this information may be used as a confirmation. Documentation of this approach must meet the requirements outlined in Sec. 7.7.3 of SW-846 Method 8082. Another approach is to use a column of dissimilar stationary phase and compare the pattern to a known Aroclor standard. Finally if the concentration is high enough GC/MS may be used as confirmation.

A. Generally, five unique peaks representing the full range of the multi-peak component are used in the quantitation of the multi-peak components.

B. Multi-peak components that still have matrix interference after appropriate sample cleanup steps have been taken may need to be hand calculated using peaks that do not have interference. This should be brought to the organic lab manager's attention.

C. Multi-peak components that exhibit a weathered pattern may need to be hand calculated by the analyst. The analyst will need to use peaks that exhibit the full range of weathering. The number of peaks used to quantitate the multi-peak component will depend on the analyst's judgment of what it will take to achieve the truest concentration of the component. This should be brought to the organic lab manager's attention.

14.10.3 Quantitation – Once a compound has been identified qualitatively, the concentration must then be quantitated. Calculations follow in Section 15.0.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Calculate the calibration factor (CF) for each analyte at each concentration as:

$$CF = \frac{\textit{Peak Area (or Height) of the Compound in the Standard}}{\textit{Mass of the Compound Injected (in nanograms)}}$$

15.3 The mean CF is calculated as follows:

$$\text{AvgCF} = \frac{\text{(CF for each standard)}}{\text{N}}$$

15.4 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i-1}^{n} (CFi - \overline{CF})^2}{n-1}}$$

$$RSD = \frac{SD}{CF} \times 100$$

15.5 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\text{\% Drift} = \frac{\text{(Calculated concentration – Theoretical concentration) * 100}}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\text{\% Difference} = \frac{\text{(CCV CF – Average CF) * 100}}{\text{Average CF}}$$

where CCV CF is the calibration factor from the analysis of the verification standard and mean CF is the average calibration factor from the initial calibration.

15.6 Concentration in water samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(\text{A}_x)(V_t)(\text{D})}{(\overline{CF})(\text{V}_i)(V_s)}$$

where:

A_x = Area (or height) of the peak for the analyte in the sample.

V_t = Total volume of the concentrated extract (µL).

D= Dilution factor, if the sample was diluted prior to analysis.
If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.

CF = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

15.7 Concentration in non-aqueous samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_x)(V_t)(D)}{(\overline{CF})(V_i)(W_s)}$$

where:

A_x, V_t, D, and CF are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

16.0 Method Performance

See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

16.1 Method Detection Limit Study or Detection Limit Determination
16.2 Limit of Detection Verification
16.3 Limit of Quantitation or Reporting Limit Verification
16.4 Demonstration of Capability (DOC)
16.5 PT Studies

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

Please see Waste Disposal, SOP QS14 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

21.0 References

21.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Method 8081A, 8081B, 8082, 8082A

21.2 USEPA Code of Federal Regulations, 40, CH 1, PT 136; Method 608, 608.2; APX-B

21.3 USEPA Contract Laboratory Program (CLP) for Organics ILM04.2; ILM04.3

21.4 DOD Quality Systems Manual, Ver. 3/4.1

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.

22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

22.5 Table 5, Calibration Standards

Table1- Detection limits

Analyte (water)	MDL/DL	LOD	LOQ/RL	Units
4,4'-DDD	0.00500	0.0100	0.0200	ug/L
4,4'-DDE	0.00500	0.0100	0.0200	ug/L
4,4'-DDT	0.00500	0.0100	0.0200	ug/L
Aldrin	0.00330	0.0100	0.0200	ug/L
alpha-BHC	0.00330	0.0100	0.0200	ug/L
alpha-Chlordane	0.00330	0.0100	0.0200	ug/L
beta-BHC	0.00330	0.0100	0.0200	ug/L
Chlordane (tech)	0.0170	0.0250	0.0500	ug/L
delta-BHC	0.00330	0.0100	0.0200	ug/L
Dieldrin	0.00500	0.0100	0.0200	ug/L
Endosulfan I	0.00330	0.0100	0.0200	ug/L
Endosulfan II	0.00500	0.0100	0.0200	ug/L
Endosulfan sulfate	0.00500	0.0100	0.0200	ug/L
Endrin	0.00500	0.0100	0.0200	ug/L
Endrin aldehyde	0.00500	0.0100	0.0200	ug/L
Endrin ketone	0.00500	0.0100	0.0200	ug/L
gamma-BHC (Lindane)	0.00330	0.0100	0.0200	ug/L
gamma-Chlordane	0.00330	0.0100	0.0200	ug/L
Heptachlor	0.00330	0.0100	0.0200	ug/L
Heptachlor epoxide	0.00330	0.0100	0.0200	ug/L
Methoxychlor	0.00330	0.0100	0.0200	ug/L
Mirex	0.00330	0.0100	0.0200	ug/L
Toxaphene	0.330	0.667	1.00	ug/L
Aroclor-1016	0.125	0.250	0.500	ug/L
Aroclor-1221	0.125	0.250	0.500	ug/L
Aroclor-1232	0.125	0.250	0.500	ug/L
Aroclor-1242	0.125	0.250	0.500	ug/L
Aroclor-1248	0.125	0.250	0.500	ug/L
Aroclor-1254	0.125	0.250	0.500	ug/L
Aroclor-1260	0.125	0.250	0.500	ug/L
Aroclor-1262	0.125	0.250	0.500	ug/L
Aroclor-1268	0.125	0.250	0.500	ug/L
Analyte (Soil)	**MDL/DL**	**LOD**	**LOQ/RL**	**Units**
4,4'-DDD	0.170	0.340	0.670	ug/Kg
4,4'-DDE	0.170	0.340	0.670	ug/Kg
4,4'-DDT	0.170	0.340	0.670	ug/Kg
Aldrin	0.110	0.340	0.670	ug/Kg
alpha-BHC	0.110	0.340	0.670	ug/Kg
alpha-Chlordane	0.110	0.340	0.670	ug/Kg
beta-BHC	0.110	0.340	0.670	ug/Kg
Chlordane (tech)	0.570	0.850	1.70	ug/Kg
delta-BHC	0.110	0.340	0.670	ug/Kg
Dieldrin	0.170	0.340	0.670	ug/Kg
Endosulfan I	0.110	0.340	0.670	ug/Kg
Endosulfan II	0.170	0.340	0.670	ug/Kg
Endosulfan sulfate	0.170	0.340	0.670	ug/Kg
Endrin	0.170	0.340	0.670	ug/Kg
Endrin aldehyde	0.170	0.340	0.670	ug/Kg

Analyte (Soil)	**MDL/DL**	**LOD**	**LOQ/RL**	**Units**
Endrin ketone	0.170	0.340	0.670	ug/Kg
gamma-BHC (Lindane)	0.110	0.340	0.670	ug/Kg
gamma-Chlordane	0.110	0.340	0.670	ug/Kg
Heptachlor	0.110	0.340	0.670	ug/Kg
Heptachlor epoxide	0.110	0.340	0.670	ug/Kg
Methoxychlor	0.110	0.340	0.670	ug/Kg
Toxaphene	11.0	22.0	33.0	ug/Kg
Aroclor-1016	4.17	8.33	16.7	ug/Kg
Aroclor-1221	4.17	8.33	16.7	ug/Kg
Aroclor-1232	4.17	8.33	16.7	ug/Kg
Aroclor-1242	4.17	8.33	16.7	ug/Kg
Aroclor-1248	4.17	8.33	16.7	ug/Kg
Aroclor-1254	4.17	8.33	16.7	ug/Kg
Aroclor-1260	4.17	8.33	16.7	ug/Kg
Aroclor-1262	4.17	8.33	16.7	ug/Kg
Aroclor-1268	4.17	8.33	16.7	ug/Kg
Analyte (TCLP)	**MDL/DL**	**LOD**	**LOQ/RL**	**Units**
Chlordane (tech)	0.000170	0.000250	0.000500	mg/L
Endrin	0.0000500	0.000100	0.000200	mg/L
gamma-BHC (Lindane)	0.0000330	0.000100	0.000200	mg/L
Heptachlor	0.0000330	0.000100	0.000200	mg/L
Heptachlor epoxide	0.0000330	0.000100	0.000200	mg/L
Methoxychlor	0.0000330	0.000100	0.000200	mg/L
Toxaphene	0.00330	0.00670	0.0100	mg/L

Table 1. Organic Analysis by Gas Chromatography (Methods 8081, 8082)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study. Minimum ±0.030min.	NA.	NA.	
Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 15% for both DDT and Endrin.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 15% for both DDT and Endrin.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin for DoD analyses. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration, if detected. Results may not be quantitated using a single point.

Table 2. Organic Analysis by Gas Chromatography (Methods 8081, 8082) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL;	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate for DoD analyses.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL;	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem, then see SOP QS05. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table 2. Organic Analysis by Gas Chromatography (Methods 8081, 8082) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix.	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Qualify surrogate results on form I.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply qualifier if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 4, Data Reviewers Checklist (Prior to approving data)

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	**8081/8082**

QA/QC Item	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Did the evaluation mix pass criteria?				
2. Does the curve consist of at least five Calibration Standards (six for quadratic curve)?				
3. Is the low standard equal to or below the MRL/LOQ?				
4. Are the %RSD or fit criteria within QC limits for all analytes?				
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 20 samples or every 12 hours and at the end of the sequence?				
2. Are the % differences within QC limits for all analytes?				
D. Sample Analysis				
1. Did the evaluation mix pass criteria?				
2. Are all sample holding times met?				
3. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?				
4. For single peak analytes - are all compounds identified on the primary column confirmed on the secondary column?				
5. For multi-peak analytes - does the pattern of the analyte in the sample match the pattern of the standard?				
6. Are surrogate recoveries within QC limits? (one surrogate both columns)				

ANALYST DATA REVIEW CHECKLIST, cont.

E. QC Samples

1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than the MDLs? _____ _____ _____ _____
2. Is the Laboratory Control Sample and its percent recovery within QC limits? _____ _____ _____ _____
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and is the percent recovery/RPD within QC limits? _____ _____ _____ _____

F. Others

1. Are all nonconformances included and noted? _____ _____ _____ _____
2. Are all calculations checked at the minimum frequency with one example worked out in the space below? _____ _____ _____ _____
3. Did analyst initial/date the appropriate printouts and report sheets? _____ _____ _____ _____
4. Are all sample IDs and units checked for transcription errors? _____ _____ _____ _____
5. Are all manual integrations checked by a second reviewer to verify they were performed correctly? _____ _____ _____ _____

<u>Calculation – one complete calculation from raw area/height to final concentration:</u>

_____

Comments on any "No" response:

_____
_____
_____
_____
_____
_____

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 5 – Standard concentrations/sources
NOTE: All standards are fully documented within the LIMS

	Level 1 (ppb)	Level 2 (ppb)	Level 3 (ppb)	Level 4 (ppb)	Level 5 (ppb)	Level 6 (ppb) MIDPOINT	Level 7 (ppb)	Primary Source (Concentration-ppm)	Secondary Source** (Concentration-ppm)
Single Component Pesticides	1	5	10	25	50	100	200	Restek (200)	Accustandard (1000)
Mirex	1	5	10	25	50	100	200	Accustandard (100)	ChemService (100)
DCB/TCMX	1	5	10	25	50	100	200	Restek (200)	NA
Technical Chlordane*	-	5	10	25	50	100	200	Restek (1000)	Ultra Scientific (5000)
Toxaphene*	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (100)
PCB-1016/PCB-1260	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (1000)
PCB-1221*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1242*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1248*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1254*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1262*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (100)
PCB-1268*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (500)

* - Toxaphene and Technical Chlordane single point at low standard unless detected. PCB calibration 1016/1260 unless other pattern detected.

** - Secondary Source may be from any vendor other than the primary source.

GC/MS VOLATILE NON-AQUEOUS MATRIX EXTRACTION USING SW-846 METHOD 5035 FOR 8260B ANALYSIS

SOP NUMBER: **SOP-225**

REVISION NUMBER: **8**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE MANAGER

EFFECTIVE DATE: **09/24/08**

DATE OF LAST REVIEW: **09/24/08**

GC/MS - VOLATILE
NON - AQUEOUS MATRIX EXTRACTION
USING SW-846 METHOD 5035

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to detail soil sample preparation for EPA method SW-846 5035. Soil samples should be sampled in the field using the EnCore™ sampler then shipped to the lab within 24 hours for preservation, storage and analysis. This SOP should be used in conjunction SOP-202, which details the analytical technique.

2.0 SUMMARY

Samples are collected in EnCores or Terracore vials. EnCore samples have to be prepped within 48 hrs of collection. Terracores are shipped already prepared.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

EnCores are prepped within 48 hrs of collection in sodium bisulfate and refrigerated at 4°C or in reagent water and frozen. Terracores are prepped in the field in sodium bisulfate or water. Sodium bisulfate Terracores are refrigerated at 4°C and those prepped in reagent water are frozen. Holding Time is 14 days

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Sample vials can be a source of contamination. Vials should be checked for contamination before use. Samples can be contaminated during sample prep. Prep blanks should be prepared at the same time as the samples to check for contamination.

5.0 EQUIPMENT AND MATERIALS

- Sample Containers – 40mL VOA vials with low bleed septa. Available from ESS (Part No. PC0040-0300 pack of 72), alternate sources are possible but must be checked for contaminants before use. ESS also supplies pre-prepped vials with the preservative and stirbar (Part No. PC4039-5035 pack of 72).
- Varian Archon 51 position programmable autosampler, or equiv.
- Top-loading balance – capable of accurately weighing to 0.01g.

- 1-10 mL Adjustable Dispenser, Model 400 Series, Oxford pipettor. Available from Oxford (Part No. 8885-040009).
- Spatula, stainless steel – narrow enough to fit into a sample vial.
- Magnetic stirring bars – PTFE- or glass-coated, of the appropriate size to fit the sample vials. Available from A. Daigger (Part No. WX22782A, case of 50).
- EnCore™ sampler – (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent. Necessary for field sampling crew.
- Terracore Vials- Available from QEC.
- Balance weights – used to calibrate the balance.
- Labels.

6.0 REAGENTS

- Reagent Water - Reagent water is NANO PURE WATER from source in the instrument lab, which is then purged with helium before use.
- Methanol, CH_3OH – purge-and-trap quality, or equivalent. Store away from other solvents.
- Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- Sodium bisulfate solution – Prepare by adding 200 grams of $NaHSO_4$ (ACS reagent grade, or equivalent) to 1000 milliliters of helium-purged reagent water. Record the vendor and lot number of the $NaHSO_4$ in the Standards and Reagents Logbook. Each standard/reagent that is prepared is recorded in the logbook and given a sequential number. The label is completed with the standard/reagent number, name, preparation date, expiration date, solvent and analyst initials. The solution should be discarded after six months or sooner if it shows signs of contamination.

7.0 SAMPLE COLLECTION

As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of volatile compounds. Always wear gloves whenever handling the tared sample vials. Several techniques may be used to perform the transfer of the sample to the relatively narrow opening of the low concentration soil vial such as the EnCore™ sampler, a cut off disposable plastic syringe, or a stainless steel spatula. We prefer to use the EnCore™ sampler.

7.1 The EnCore™ sampler is both a sampler and a container for low-level and high level soils. It is designed to collect an average weight with the exact weight to be

determined in the lab. It is disposable and is also designed to have zero headspace. The EnCore™ sampler will require the field personnel to get the sample to the laboratory within 24-36 hours of collection. The laboratory needs to be contacted prior to sample collection to ensure that all necessary containers (with or without preservative) are available and that the proper sampling technique is used.

7.2 All low-level soil samples must be collected in duplicate to allow the laboratory an additional sample for reanalysis. A third sample should be collected for preparation of a high-level sample. This sample would be prepared at the same time as the "low-level" sample. (Some projects may not require the "low-level" detection limits, in this case only the high level sample preparation would be required.) A fourth sample may be collected to enable the laboratory to perform a pretest on the soil to determine if the soil sample contains carbonate minerals that will effervesce upon contact with the acidic sodium bisulfate preservative solution in the low concentration sample vial. The additional soil samples must be collected from the same soil stratum or the same section of solid waste being sampled and within close proximity to the location from which the original sample was collected. Additional bulk samples should be collected for screening and dry weight determination without the preservative. Note: If the low-level sample cannot be preserved with sodium bisulfate, the remaining low-level sample aliquot(s) is(are) transferred to a pre-weighed vial containing 5 mL of reagent water. The sample in the unpreserved vial must either be analyzed immediately (within 48 hours of collection) or frozen within the 48 hour time frame and then analyzed within the 14 day holding time.

8.0 PROCEDURE

8.1 Log-in personnel will log the samples in, place them in the Soil walk-in cooler assigned for volatile sample storage and notify the Organic Lab Manager that samples are in-house for 5035 preparation.

8.2 The Organic Lab Manager or designee will determine the amount of time remaining on the 48 hour EnCore™ holding time and assign the task of preserving the samples.

8.3 Samples received from the field should be designated for low-level, high-level or % solids/screening (this fraction should be in a regular soil jar, if it is not, it will require transfer to a VOA vial). Each low-level and high-level sample must be preserved appropriately as follows:

8.3.1 Organize the VOA vials required and label them with the sample number, date and LOW or HIGH for either low-level or high-level preservation. The LOW level VOA vials should have gray caps and septa if using the ESS brand.

8.3.2 Get the samples from the Hobart assigned for volatile sample storage and log them out.

8.3.3 Enter the sample numbers in the soil sample preparation logbook and add a sample preparation/storage blank to the book for each level being prepared (HIGH/LOW). There must be a line in the logbook for each sample vial being prepared (i.e. if there are 2 low-level samples and 1 high-level sample, the sample number should be listed in the logbook 3 times- use a,b,c to designate each vial associated with the same sample).

8.3.4 Using an adjustable Oxford pipettor, add 5 mL P&T methanol to each of the vials marked HIGH. Then record the vendor & lot number of methanol and the exact volume of methanol added to each sample in the sample preparation logbook. If the vial is not to be used immediately, weigh the vial to the nearest 0.01g and record the weight on the vial. The vial weight must be verified to be within ±0.01g of this value before using for sample preparation.

8.3.5 For each of the vials marked LOW, add 5 mL of sodium bisulfate or reagent water if frozen and record the reagent number in the sample preparation logbook. Add a magnetic stir bar to each vial. If pre-prepped vials from ESS (or equivalent) are used, this step is unnecessary but the lot number and the pre-prepped status must be recorded in the preparation log.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and place low concentration samples in vials that contain 5ml water and a stir bar. This sample must be frozen in a slanted position until analysis or analyzed within 48 hours of sampling. Notify the Organic Lab Manager if this occurs, note this in the sample preparation logbook and generate a CAR to document the problem.

8.3.6 Place the vial (LOW/HIGH) on the top-loading balance, tare the vial then extrude the sample into the vial and record the weight of the sample in the sample preparation logbook. Make sure the lip of the vial does not have any soil on it, which might cause a leak, cap the vial tightly and mark the weight on the sample label.

8.3.7 Place the preserved samples in a box, return them to the Hobart assigned for volatile sample storage and log them back in.

9.0 ANALYSIS

9.1 Samples are analyzed by USEPA SW-846 methods 5035/8260B (low-level) using the Archon 51 position autosampler in conjunction with the GC/MS or 5030B/8260B (high-level) using any purge and trap instrument in conjunction with the GC/MS. For method 5035, the prepared low-level vials are placed in the Archon autosampler. The autosampler is programmed to add the appropriate internals and surrogates to each sample. Use of the autosampler is covered in the owners manual. Calibration of the analytical instrument with subsequent analysis of the samples is covered under SOP-202.

9.2 Determination of % Dry Weight – Weigh 5-10 grams of the sample from the bulk jar used for dry weight analysis in a tared crucible or aluminum pan. Dry overnight at 105°C. Allow to cool in a dessicator before weighing. Calculate % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

10. HEALTH,SAFETY,WASTE MANAGEMENTAND POLLUTION PREVENTION

10.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.

10.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

10.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves across from the Quality Assurance Officers cube.

10.4 Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

REFERENCES

1.Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 5035.

DEFINITIONS

Refer to SOP-431 for common environmental laboratory definitions.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

SOP #: 239 **REV #: 2** **EFFECTIVE DATE: 07/21/09**
DATE OF LAST REVIEW: 07/21/09

PERCHLORATE IN WATER, SOIL, AND SOLID WASTE USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/ ELECTROSPRAY IONIZATION/ MASS SPECTROMETRY BY SW846 METHOD 6850

APPROVED SIGNATURES:

LAB DIRECTOR: _____ **Date:** 8/11/09

DEPT. MGR: _____ **Date:** 8.11.09

QA OFFICER: Randy N. Ward **Date:** 8-11-09

1. SCOPE AND APPLICATION

1.1. This SOP is used for the Analysis of Perchlorate in Water, Soils, and Solid Wastes using High Performance Liquid Chromatography / Electrospray Ionization/Mass Spectrometry (HPLC / ESI / MS).

1.2. This method is for the determination of perchlorate in groundwater, surface water, soil, and sediment using high performance liquid chromatography (HPLC) coupled with electrospray ionization (ESI) mass spectrometry (MS).

1.3. The Reporting Limits (RLs) are highly matrix-dependent and are listed below:

Matrix	Reporting Limits	Units
Solids	2.0	µg/KG
Aqueous	0.2	µg/L

2. METHOD SUMMARY

Solids are first extracted prior to analysis with reagent water using an ultrasonic bath. Water samples are not extracted. Extracts and water samples are filtered, if needed. An appropriate volume of the sample or sample extract is introduced into a HPLC/MS instrument. Perchlorate is separated by HPLC from the sample matrix, using negative electrospray ionization and detected by MS using mass-to-charge (m/z) using mass-to-charge ratios 99, 101, and 107. Quantitation is performed using m/z 99 and internal standard calibration. Isotopically-labeled perchlorate (Cl18O4-), m/z 107, serves as the internal standard. The 99/101 isotopic ratio reflects the isotopic ratio of naturally occurring 35Cl/37Cl and is used for additional confirmation of perchlorate identification.

3. DEFINITIONS

3.1. **Preparation Batch**: a group of 20 or fewer samples of the same matrix prepared together on the same day.

3.2. **Analytical Batch**: a group of 20 or fewer samples analyzed together.

3.3. **Internal Standards** – Internal standards are analytes which are similar to the analytes of interest but which are not found in the samples. The chosen internal standards are used to calibrate the instrument's response.

3.4. **Continuing Calibration Verification (CCV):** standard analyzed at a routine frequency throughout the sequence to establish the instrument is meeting calibration criteria.

3.5. **Independent Calibration Verification (ICV):** standard analyzed from a source that is different than that used to calibrate the instrument. The ICV is a check on the purity and accuracy of the calibration standards and can be used in preparing matrix spike solutions for spiking of QA/QC samples.

3.6. **Relative Percent Difference (RPD):** measure of the relative difference between two values.

3.7. **Method Detection Limit (MDL) -** also called LoD (Limit of Detection): a statistically derived value representing the lowest level of target analyte that may be measured by the instrument with 99% confidence that the value is greater than zero.

3.8. **Reporting Limit (RL):** The minimum amount of a target analyte that can be measured and reported quantitatively. The RL is equivalent to Practical Quantitation Level (PQL), reporting limit, and Limit of Quantitation (LoQ).

3.9. **QA/QC Samples**: Samples added to a sample preparation batch, or an analytical batch to provide quality assurance checks on the analysis.

3.9.1. **Duplicate Sample (DUP):** a laboratory duplicate. The duplicate sample is a separate sample aliquot that is processed in an identical manner as the sample proper.

3.9.2. **Laboratory Control Sample (LCS):** a method blank that has been fortified with the analyte of interest. Used to verify laboratory performance. The LCS is prepared in a similar matrix as that of the samples and is prepared in Ottawa sand for soil and DI water for water matrix.

3.9.3. **Method Blank (MB):** a laboratory blank that is used to check that reagents and equipment are free of target analytes. The Method Blank follows the same preparative techniques as samples and is prepared in Ottawa sand for soil and DI water for water matrix. Unless stated in project specific data quality objectives, reagents are considered free of target analyte contamination if the level detected in the Method Blank is less than the ½ RL.

3.9.4. **Matrix Spike Sample (MS):** a duplicate sample aliquot that has been fortified with the analyte of interest. Used to observe the effects of the sample matrix on target analyte recovery.
3.9.5. **Matrix Spike Sample/ Matrix Spike Duplicate Sample (MS/MSD):** matrix

spike samples prepared in duplicate. It is used to evaluate method precision and matrix effects.

4. INTERFERENCES

4.1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.2. Sample extracts with any visible particulates should be filtered through a minimum 0.45-µm nominal pore size membrane or frit to remove particulates and prevent damage to the instrument, columns and flow systems. Filters specifically designed for HPLC applications should be used.

4.3. Hydrogen sulfate ion H34SO4-, formed from a minor sulfur isotope, is commonly present in samples. H34SO4- elutes before perchlorate but at high concentrations can tail into the retention time of the perchlorate peak and elevate its baseline at m/z 99. Quantitation of perchlorate based on m/z 83 and 89 avoids this potential interference from H34SO4-.

4.4. Glassware must be scrupulously cleaned. Glassware should be rinsed as soon as possible after use with the last solvent used. This should then be followed by detergent washing with hot water and rinses with tap water and organic-free reagent water. After washing, the glassware should then be drained, dried, and heated in a laboratory oven at 130°C for two to three hours before reuse. Solvent rinses with acetonitrile may be substituted for the oven heating. After drying and cooling, glassware should then be stored in a clean environment to prevent accumulation of dust or other contaminants.

5. SAFETY

5.1. The toxicity or carcinogenicity of each reagent used in this procedure may not be precisely defined. Material safety data sheets (MSDS) are available on each solvent and standard and should be reviewed as part of employee training.

5.2. Personal protective clothing (safety glasses, gloves and lab coat) are recommended when prepping samples, handling standards, or performing maintenance on pressurized systems.

5.3. The proper use of syringes and glass pipettes should be part of employee training. Care should be taken to avoid personal injury as a result of improper handling techniques.
5.4. Refer to the Safety Manual for further discussion of general safety procedures and information.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Water samples should be collected in purchased, pre-cleaned 125-mL polyethylene bottles.

6.2. For solids, collect samples in purchased clean, 4-oz amber glass jars.

6.3. Prepare and analyze samples and extracts within 28 days of collection.

6.4. Care should be taken to avoid temperature extremes during shipment and storage

6.6. Samples will be refrigerated to 0-6°C upon sample login.

7. APPARATUS AND EQUIPMENT

7.1. Volumetric flasks, 100-mL and other sizes as needed.

7.2. Automatic precision pipetters and disposable tips - 10 mL capacity and gas tight syringes.

7.3. Disposable autosampler vials, 2mL capacity.

7.4. Disposable plastic centrifuge tubes - 15 mL.

7.5. Disposable plastic micro-beakers.

7.6. Sample bottles - polyethylene or glass of sufficient volume to allow replicate analyses.

7.7. Disposable PTFE 0.45-µm, surfactant-free, membrane syringe filters.

7.8. SPE Cartridges – C18 (2000 mg) chromatography columns or equivalent for extract solution cleanup.

7.9. High Performance Liquid Chromatograph/Mass Spectrometer - An analytical system combining a high performance liquid chromatograph for separation of the sample components, electrospray ionization interface and mass spectrometer for fragmentation and detection of sample components.
7.9.1. High Performance Liquid Chromatograph (Agilent 1200) – The instrument contains a programmable solvent delivery system and all required accessories including injection loop, pump, analytical columns, autosampler and UV detector.

7.9.2. Electrospray ionization (ESI) source – The ESI source generates gas phase ions of perchlorate from the liquid phase.

7.9.3. Mass Spectrometer – A quadrupole mass analyzer, Agilent 6140 LC/MS quadrupole mass spectrometer. Instrument tuning and instrument conditions are based on the manufacturer's instructions. Tuning is not required prior to daily analyses. Tuning parameters are available from the instrument manufacturer, along with mass tuning solutions and instructions on how to optimize the mass spectrometer.

7.9.4. Analytical column – 4 mm x 250 mm Metrohm Metrosep A Supp 5 column (cat# 6.1006.530) or equivalent. Guard column- Metrohm Metrosep A Supp 4/5 S-Guard(cat# 6.1006.540).

7.9.5. Data system capable of performing analyte signal acquisition, peak integration, instrument calibration and analyte quantification.

7.10. Analytical balance, capable of ±0.0001 g accuracy.

7.11. Centrifuge - capable of 5000 rpm and/or generating g-force of such strength as needed for providing adequate separation of a solid suspension and thus yielding a clear supernatant for sample injection and analysis.

7.12. Ultrasonic Bath
7.13. Vortex Mixer

8. PREVENTIVE MAINTENANCE

8.1. A maintenance log will be kept documenting maintenance performed on the analytical system. Log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements (defined as any non-consumable part essential to operation), phone logs from technical support contacts, a description of the maintenance performed, and the analyst's initials.

8.2. Any major maintenance performed on the instrument must be recorded in a maintenance log and must include the date, the person performing , the type and the outcome of the maintenance.

8.3. Routine maintenance such as changing columns, changing guard columns or inlet filters is noted in the run log.

9. REAGENTS, AND CONSUMABLE MATERIALS

9.1. Reagents

9.1.1. Acetonitrile – HPLC grade or equivalent

9.1.2. Ammonium Formate – Reagent grade

9.1.3. Ammonium Carbonate – Reagent grade

9.1.4. Ammonium Hydroxide – Reagent grade

9.1.5. DI water – MODULAB Research Grade Water System

10. STANDARDS PREPARATION

10.1. All Standards for this analysis are prepared in water from the MODULAB system.

10.2. The initial calibration curve given is subject to variation due to detection levels needed. A minimum of five calibration standards is recommended as well as a blank standard. The lowest concentration level shall be at the reporting level and at least three times the MDL. The remaining levels should define the working linear range of the analytical system.

10.3. Vendors and vendors' products are sometimes listed for the ease of the analyst using this SOP, but products and purchased concentrations are examples only and subject to change at any time. All purchased standards are certified by the vendor. Certificates of Analysis are kept in the department. Purchased standards are routinely checked against an independent source for both analyte identification and analyte concentration.

10.4. Stock standard solution (1000 mg/L ClO4-) - This solution is purchased commercially as a certified standard. Stock standards may be stored at room temperature for a period of up to 12 months. Expiration dates should be clearly specified on the label.

10.5. Intermediate Standard (10 mg/L ClO4-) – Dilute 1.0 ml of 1000 mg/L standard solution to 100 mL with DI water. Working standard may be stored at room temperature for up to 12 months. Expiration dates should be clearly specified on the label.

10.6 Intermediate Standard (0.1 mg/L ClO4-) – Dilute 1.0ml of 10 mg/L standard solution to 100 mL with DI water. Working standard may be stored at room temperature for up to 12 months. Expiration dates should be clearly specified on the label.

10.7. Calibration standards – Prepare by adding various amounts of the 0.1 mg/L intermediate standard solution to DI water and dilute to 10 ml. Spike each calibration standard with 50uL of internal standard spiking solution (1.0 mg/L). The final internal standard concentration is exactly the same (5.0 µg/L Cl18O4-) for each calibration standard.

Calibration Concentration(ug/L)	Volume (uL) of 0.1 mg/L ClO4- working std	Final Volume (mL)
0.20	20	10
0.50	50	10
1.00	100	10
2.00	200	10
5.00	500	10
10.0	1000	10

10.6. Internal standard spiking solution (1000 µg/L Cl18O4-) –Purchased commercially. Refrigerate. Expires per manufacturer's indications or 12 months if no other indication is given.

10.6.1. Each standard and sample is spiked with 50 µL of internal standard spiking solution per 10 mL of sample or standard to obtain a final internal standard concentration of 5 µg/L Cl18O4-.

10.7. HPLC Mobile Phase – Prepare by dissolving 0.946 grams of ammonium formate and 0.721 grams of ammonium carbonate in 250 ml of DI water, add 700 mL acetonitrile, 10 ml of ammonium hydroxide, and bring up to 1 liter with DI water.

10.8. Second Source Standard – The second source standard is purchased at 1000 mg/L from a different manufacturer than the Stock standard solution. Make a 10 mg/L standard by diluting 1 ml to 100 ml of DI water. Expires per manufacturer's indications or 12 months if no other indication is given.

10.9. Independent Calibration Verification Standard (ICV): The ICV is prepared from the second source standard at a final concentration of 5.0 ug/L.

10.10. Continuing Calibration Verification Standard (CCV): The CCV is prepared from the 0.1 mg/L intermediate standard solution at a final concentration of 5.0 ug/L.

10.11. LODV(Limit of Detection Verification, 0.2 ug/L) – A standard prepared at a concentration approximately 2 times the MDL.

10.12. LCS/MS Spiking Solution (0.1 mg/L) – Dilute 1.0 mL of a 10 mg/L Second Source Perchlorate Standard to 100 mL with DI water. Use this to spike the MS and the LCS. Store at room temperature. Expires in 6 months.

10.13. Laboratory Control Sample (LCS) (0.2 ug/L) – Dilute 20 uL of 0.1 mg/L LCS/MS solution to 10 mL DI water. Process as a sample.

10.14. Interference Check Sample (ICS) (0.2 ug/L) –

10.14.1. ICS Matrix preparation - 5000 mg/L each of chloride, sulfate, carbonate by dissolving 8.240g NaCl +7.395g Na2SO4 +5.294g Na2CO3 in DI and diluting to 1000ml.

10.14.2. ICS – prepared upon use – add 1.0 mL of ICS matrix to 8.0 mL HPLC water. Add 20 uL of 0.1 mg/L perchlorate standard (second source = LCS/MS Spiking Solution) and 50 uL internal standard. Prepare and process as a sample.

10.15. MS/MSD (0.2 ug/L) – Add 20 uL of 0.1 mg/L spiking solution to 10 mL of water sample. Process as a sample.

10.16. The LCS for Soil (+2.0 ug/kg) - Add 20 uL of 0.1 mg/L spiking solution to 1 g of Ottawa sand or soil sample. Process as a sample.

10.17. Mass Calibration Standard – a proprietary mix from Agilent. Use, store, and discard as per manufacturer's instructions.

10.18. Tune Verification Standard – 100 ug/L each of perchlorate and internal standard

11. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

12. PROCEDURE

12.1. Be sure the analysts performing the test have a current IDOC or CDOC (see section 19). Be sure the instrument has a current MDL study (see section 14).

12.2. Sample Preparation - Check the conductivity of the samples(soils after sample prep) to be analyzed to see if they need to be diluted to avoid interference from common suppressors.

12.2.1. Water Sample Preparation
12.2.1.1. Dispense 10.0 mL of sample into a 15 mL disposable centrifuge tube.

12.2.1.2. Add 50 µL of internal standard spiking solution (1.0 mg/L) to the sample tube, cap and shake until mixed well. The final concentration of internal standard in the sample must be exactly the same as that in the calibration standards
12.2.1.3. If needed, filter the sample solution using a plastic syringe fitted with a 0.45-µm PTFE membrane filter. Dispense the sample into an autosampler vial for analysis. If any of the samples in the prep batch are filtered, filter the entire prep batch, including QC.

12.2.2. Solid Sample Preparation

12.2.2.1. Weigh 1 g of solid sample, recording the weight to 0.01 g into a 15 mL disposable centrifuge tube.

12.2.2.2. Add 10 mL of DI water, 50 µL of internal standard spiking solution to the sample tube. Vortex the mixture, followed by sonication (ultrasonic bath) for a minimum of 10 minutes, followed by additional vortexing.

12.2.2.3. Centrifuge the sample for 5 minutes, if necessary, to separate the solids from the extract solution.

12.2.2.4. If needed, filter the supernatant extract solution using a plastic syringe fitted with a 0.45-µm PTFE membrane filter. Dispense the extract sample into an autosampler vial for analysis. If any of the samples in the prep batch are filtered, filter the entire prep batch, including QC. NOTE: The final concentration of internal standard in the sample extract must be exactly the same as that in the calibration standards.

12.2.2.5. If large quantities of organic contaminants are not believed to be present in the solid sample extract (i.e. supernatant extract is relatively clear and not highly colored), proceed to instrument analysis. If necessary, however, a cleanup step using a C18 column may be performed to remove organic contaminants from the supernatant extract solution.

12.2.2.5.1. Activate the C18 cartridge column by pushing approximately 5 mL of methanol through the column, followed by 5 mL of DI water. A flow rate of approximately 0.5 mL/min is recommended. Care should be taken not to let the column become dry.

12.2.2.5.2. Pull approximately 6 mL of the supernatant extract solution through the activated column. Use vacuum, if necessary.

12.2.2.5.3. Discard the first 2 mL of eluted sample extract. The initially eluted sample extract is discarded because it is diluted by the solution remaining in the column following the activation process.

12.2.2.5.4. Collect the remaining eluted sample extract (approximately 4 mL) in a clean container. Filter the sample extract using a plastic syringe fitted with a 0.45-µm membrane filter. Dispense the extract sample into an autosampler vial for analysis. Quantitative recovery of the eluted extract sample is not necessary because there is no dilution or concentration of the sample.

13. INSTRUMENT SET-UP

13.3.1. Instrument conditions for analysis are set to optimize response and separation of the analytes. Analysts are advised to adjust the HPLC systems to particular analytical needs. Once optimized, the same conditions are to be used for analysis of all standards, client samples, and QC samples. The current system uses the conditions in Table 1. Actual instrument conditions are optimized and recorded in the instrument daily run log.

13.3.2. Mass Calibration –

• Frequency – Upon instrument setup and as needed (QC failures, large ion mass deviations, major instrument maintenance, tune won't meet criteria)
• Acceptance Criteria – the calibration range must bracket the ion masses of interest without greatly exceeding the range. The most recent mass calibration must be used for an analytical run and the same mass calibration must be used for all data files in an analytical run. Mass calibration must be verified by acquiring a full scan mass spectrum of a perchlorate standard (200ug/L). Perchlorate ions should be within ±0.3 *m/z* of mass 83, 85 and 89.
• Corrective action – Correct the problem and recalibrate if the mass calibration fails.

13.3.3. Retention Time

13.3.3.1. Due to the use of the internal standard, formal retention time studies are not needed. The retention time varies due to sample matrix conductivity,

however the internal standard retention time varies at the same rate. It is the relative retention time between the sample and the internal standard which is most critical and must be monitored (see section 12). The retention times of the sample and internal standard are summarized and included in the report.

13.3.4. Interference Threshold study –

• Frequency – at initial setup and when major changes occur in the method's operating procedures (addition of cleanup procedure, column changes, mobile phase changes).

• Acceptance Criteria – Measure the threshold of common suppressors (chloride, sulfate, carbonate, bicarbonate) that can be present in the system without affecting the quantitation of perchlorate. The threshold is the concentration of the common suppressors where perchlorate recovery of a 0.5 ug/L standard falls outside an 85-115% window. Analyze successively higher conductivity standards with the perchlorate concentration at 0.5 ug/L. Presently the recovery is good up to 18,000 us/cm conductivity.

13.4. Initial Calibration

13.4.1. Start up entire HPLC system and allow baseline response to settle.

13.4.2. All injections of standards and samples and associated QC are to be the same. 100 uL injections are used.

13.4.3. The first sample through the system should be an instrument blank. This blank is simply an injection of DI water and internal standard. As the first injection of the day, this helps to further settle the baseline and equilibrate the system.

13.4.4. A pre-curve blank is run to check for system and reagent cleanliness. Any potentially interfering peaks should be noted. This blank is prepared alongside the calibration standards.

13.4.5. Process a minimum of 6 calibration standards and a blank (as described in Section 9). The lowest calibration standard must be at (or below) the reporting limit and the standard at the reporting limit must be at least three times the MDL.

13.4.6. Analyte responses are tabulated and recorded in the integration software.

13.4.7. Calibration Curve –

13.4.7.1. Response Factor - To obtain the average response factor, calculate the RF (as below) for each calibration point. Calculate the mean of the RFs to use for sample calculation. Calculate the Standard Deviation of the RFs

and the Percent Relative Standard Deviation of the RFs to evaluate the linearity. The limit for the calibration to use average response factor for quantitation is 20% RSD. For a linear curve the minimum correlation is 0.995.

$$RF = \frac{(As)(Cis)}{(Ais)(Cs)}$$

Where:
As = Area of the characteristic quantitation ion for compound .
Ais = Area of the characteristic quantitation ion for the specified internal standard.
Cs = The concentration of the compound added (ppb).
Cis = The concentration of the specified internal standard (ppb).
Calculate standard deviation (SD) and the percent relative standard deviations (%RSD) for each analyte from the mean with:

$$SD = \sqrt{\sum_{I=1}^{N} \frac{(\overline{RFi} - RF)^2}{N - 1}}$$

where:
RFi = RF for each of the calibration levels
RF = mean of initial RFs
N = Number of RF values (i.e., 6)

$$\%RSD = \frac{(SD)}{(Avg\ RF)} X\ 100.$$

where:
RSD = relative standard deviation.
RF = mean of 6 initial RFs for a compound.
SD = standard deviation of average RFs for a compound

13.4.8. The concentration corresponding to the absolute value of the calibration curve's Y-intercept must be ≤ LOD.

13.4.9. All of the ICAL criteria must be acceptable before samples may be analyzed.

13.4.10. Routinely, after the high point of the curve, a blank is analyzed to prove that there is no carry-over from the autosampler. It should be noted if there are any target hits above ½ of the RL.

13.4.11. Verify the calibration by analyzing an independent calibration verification

standard. Acceptance criteria for the ICV is 85-115% recovery.

13.4.12. Samples immediately following an initial calibration and ICV may skip the system blank and the opening CCV. Set up the rest of the sequence to comply with the frequency requirements of the QC listed in section 14.

13.5. Daily Sample Analysis

13.5.1. When an ICAL is not run on the day of sample analysis, run blanks initially to warm up the system. Do not begin the sequence until the blanks are less than ½ the RL. Analyze the CCV. Set up the rest of the sequence to comply with the frequency requirements of the QC listed in section 14.

13.6. Evaluation of Sample Analysis

13.6.1. Examine solution concentrations of target analytes in the samples. If the concentration is greater than the high calibration standard, reanalyze the sample at a dilution. As a general rule, dilutions should be performed to keep target analytes in the upper half of the calibration range.

13.6.2. Check for possible carryover. Pay particular attention to situations where samples containing low levels of target analyte were analyzed one or two injections after samples containing levels of target analyte near or over the high end of the calibration range. Reanalyze as needed to confirm.

13.6.3. Check peak integrations. Where possible, all integrations should be performed consistent with integration of the corresponding calibration standards.

13.6.4. Monitor the retention times of the ions in samples and QC. The retention times of the ClO3- m/z 99 peak and the IS Cl18O3- m/z 107 peak should not vary by more than 0.2 min from each other.

13.6.5. Monitor the mass ratio between ions 99/101. The ion ratio should fall within the established window of 2.3-3.8. If criteria not met, the sample(s) must be repeated. Reextract using cleanup procedures or alternative techniques to confirm the presence of perchlorate such as post spikes or dilutions to reduce the interference. Qualify the data and discuss in the case narrative as necessary.

14. QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

14.1. Tune verification – Verification is performed after Mass Calibration and before a new calibration curve. The quantitation ions must be within ±0.3 *m/z* of actual.

14.2. Initial Calibration Criteria: Calibration criteria are given in section 13.

14.3. Method Blank

• Frequency - Process one MB with each preparation batch of 20 or fewer samples.
The MB should be carried through all stages of sample preparation and analysis.
• Acceptance Criteria - The result of the MB must be less than ½ the reporting limit.
• Corrective Action - If it is not, determine the source of the contamination and reprep and reanalyze the associated samples.

14.4. Reagent blanks

• Frequency – prior to calibration and after each batch is analyzed. When perchlorate concentrations in the samples are over the linear range, any samples which follow the overrange sample will be repeated if the sample concentrations were greater than ½ the RL.
• Acceptance Criteria – must be less than ½ RL.
• Corrective Action – reanalyze reagent blanks until no carryover is observed (<1/2 RL). Reanalyze all samples processed since the contaminated blank. If the closing reagent blank fails, reanalyze all samples since the last clean reagent blank.

14.5. MS

• Frequency - Prepare one MS (or MS/MSD pair) with each batch of 20 or fewer samples of the same matrix.
• Acceptance Criteria - The limit for the water and soil is 80-120% of the true value.
• Corrective Action - If recovery of MS fails on a sample batch the samples are acceptable only if the LCS meets acceptance criteria. If the LCS passes, but the MS fails, report the MS recoveries and note outliers in the case narrative.

14.6. LCS –

• Frequency - Prepare one LCS with each batch of 20 or fewer samples of the same matrix.
• Acceptance Criteria (recovery) - The limit for the water and soil LCS is 80-120%.
• Corrective Action - If the LCS fails on a sample batch, reprep and reanalyze the sample batch. If a duplicate LCS was run with the batch and one LCS was slightly out, but the other LCS was in, the data is accepted.

14.7. DUP/MSD Precision –

• Frequency - Precision for each prep batch of 20 or fewer samples of the same matrix must be established by either analyzing a sample in duplicate (DUP) or analyzing a MS in duplicate (MSD).
• Acceptance Criteria – (for all samples above the reporting limit), the RPD value between samples or MS/MSD results must not exceed 15%.
• Corrective Action - examine the chromatograms and benchsheets for potential matrix interferences. Reanalyze the pair if deemed appropriate. The outlying RPD should be mentioned in the Case Narrative so that data may be flagged appropriately.

14.8. The ICV is discussed in Section 13.

14.9. CCV

• Frequency – bracketing every 10 or fewer samples
• Acceptance Criteria - All samples must be bracketed by an acceptable CCV. The acceptance limit is 15% difference relative to the true value.
• Corrective Action - If the CCV does not meet acceptance criteria, correct the problem and achieve an acceptable CCV. Recalibration may be necessary. All samples bound by the unacceptable CCV must be reanalyzed with acceptable QC.

14.10. Internal standard area response verification

• Frequency – The IS is added to every sample, spiked sample, standard, instrument blank, and method blank
• Acceptance Criteria - The internal standard area counts for each sample and QC standard must be monitored throughout the analysis and the IS area must be within ±50% of the average of the internal standard area counts of the ICAL standards.
• Corrective Action - If the internal standard area counts exceed ± 50% of the average of the internal standard area counts of the ICAL standards, rerun the sample at increasing dilutions until the acceptance criteria are met. If the criteria cannot be met with dilution, interference is suspected and the sample must be reprepared using additional pretreatment steps. Thoroughly document corrective action in the case narrative.

14.11. Internal standard and Target retention times

• Frequency – IS is added to every sample, spiked sample, standard, instrument blank, and method blank.
• Acceptance Criteria – The RRT (relative retention time) must be 0.98-1.02. Calculate the RRT in a sample by dividing the retention time of the perchlorate ion by the retention time of the internal standard.
• Corrective Action – Thoroughly document in the case narrative.

14.12. MDL – see section 16.

14.13. LODV(Limit of Detection Verification 0.2 ug/L)

• Frequency – before and directly after every analytical batch of samples. It may be run more frequently to reduce reanalysis rate.
• Acceptance criteria – the recovery must be within 30% of the true value. Exception – any samples with concentrations above the RL may be reported.
• Corrective Action – if there are samples below the RL, correct the problem, obtain a compliant LODV and reanalyze affected samples.

14.14. ICS (Interference Check Standard)

• Frequency – one ICS is extracted with every batch of 20 or fewer samples. At least one ICS must be analyzed daily.
• Acceptance Criteria – the recovery must be within 30% of the true value.
• Corrective action – Correct the problem and reanalyze all samples associated with a failed ICS.

15. DATA REDUCTION AND REPORTING

15.1. Identify perchlorate in the sample chromatograms

15.1.1. Compare the retention times of the ClO4-, m/z 99, peak and the internal standard Cl18O4-, m/z 107, peak. The retention times should not vary by more than 0.2 minutes from each other. The relative retention time (Retention time of perchlorate ion divided by the retention time of the internal standard) must be 0.98 – 1.02.

15.1.2. Evaluate the relative abundances of the m/z 99 and 101 ions in the chromatogram. To confirm the presence of perchlorate, the 99/101 peak area counts ratio should be 2.3-3.8. Repeat the samples if ratio criteria not met or flag the data as estimated and discuss in the case narrative.

15.2. Calculate the perchlorate concentration for each sample using the internal standard calibration. Use m/z 99 and 107 from the internal standard for quantitation. Calculate the concentration of each of the sample matrices as follows:

15.2.1. Water Samples

Concentration (µg/L) = *(C)(D)*

Where:
C = Concentration from the calibration curve in µg/L.
D = Dilution factor, if needed

15.2.2. Solid Samples

$$\text{Concentration (µg/kg ClO4-)} = \frac{(C)(V)(D)}{M}$$

Where:
C = Concentration in extract (µg/L ClO4-)
V = Volume of sample extract used for analysis (mL)
D = Dilution factor (if needed)
M = Mass of initial sample extracted (g)

15.3 Results are adjusted for soil samples based on Dry Weight.

15.4 Adjustments to the Reporting Limit - RLs are elevated for insufficient sample volume, matrix interference, and dilutions.

16. METHOD PERFORMANCE

16.1. MDL study

16.1.1. Design - The MDL study must be performed in the matrix of interest using a standard at a concentration that is 1 to 10 times the estimated MDL value. The standard is taken through the entire preparation and analysis procedure.

16.1.2. Frequency - A full MDL study must be conducted at initial setup and subsequently once per 12 month period and when major changes occur in the method's operating procedures (addition of cleanup procedures, column changes, mobile phase changes). If no changes have been made to the method, quarterly MDL verification checks (see below) may be performed in lieu of the yearly MDL study.

16.1.3. Acceptance criteria – The study must produce an MDL value which is no more than 10 times lower than the spike level (10x rule). The calculated MDL must be 3 times less than the reporting limit. The MDL must be verified by an acceptable MDL verification check (see below) prior to the analysis of samples.

16.1.4. Corrective Action

16.1.4.1. If the MDL study does not meet the 10x rule, adjust the spike

concentration and repeat the study.

16.1.4.2. If the MDL is not 3 times less than the reporting limit, correct the problem and repeat the MDL study or raise the reporting limit accordingly.

16.1.4.3. If the MDL verification check fails, see below.

16.2. MDL verifications checks

16.2.1. Concentration – the MDL verification check is a low-level spike at approximately 2 times the MDL taken through the entire preparation process.

16.2.2. Frequency – with each new MDL study prior to the analysis of samples and quarterly.

16.2.3. Acceptance criteria – must produce a signal at least 3 times the instrument's noise level and meet the isotope ratio criteria of 2.3 to 3.8. Recovery should be within 30% of its true value.

16.2.4. Corrective action – re-perform the MDL study. Perform maintenance, if needed. The MDL verification check must pass before samples are analyzed.

16.3. Demonstration of Capability is performed upon instrument set-up , whenever a new analyst begins independent analysis, and annually thereafter.

17. WASTE MANAGEMENT AND POLLUTION PREVENTION

Standards are prepared on an as-needed basis in small quantities and minimum sample volumes are used during analysis. All expired standards are disposed of as hazardous and hazardous samples are disposed of either as acidic aqueous waste or as hazardous aqueous waste depending on the analytes concentration.

18. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

If data is produced that is out of control, the samples are to be re-analyzed with in-control QA whenever possible. See corrective actions in Section 14 of this SOP.

19. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, a Corrective Action Report must be generated and the problem addressed in the case narrative.

20. REFERENCES

a. EPA SW-846 Perchlorate in Water, Soils and Solid Wastes Using High Performance Liquid Chromatography/Electrospray Ionization/ Mass Spectrometry. Method 6850. January 2007.

b. DoD Perchlorate Handbook, Revision 1, Change 1, August 2007.

21. TABLES & ATTACHMENTS

a. Table 1, Instrument Settings
b. Table 2, Data Checklist

TABLE 1 INSTRUMENT SETTINGS

Autosampler: Injection Volume: 100 µL

Analytical Column: Column Temperature: 35 °C

HPLC Pump
Flow Rate: 0.4 mL/min
Run Time: 13.0 min
Mobile Phase:
0.946g Ammonium Formate, 0.721g Ammonium Carbonate dissolved in 250 ml DI water, 700 ml Acetonitrile, 10 ml Ammonium Hydroxide, bring to one liter with DI water.

Mass Spectrometer
Ionization Mode: Electrospray
Polarity: Negative

SIM Parameters:

SIM Ion	Fragmentor	Gain (EMV)	Dwell
99	120 V	10	995 msec
101			995 msec
107			995 msec

Spray Chamber:

Gas Temperature	Drying Gas (N2)	Nebulizer Pressure
350 °C	12.0 L/min	50 psig

Capillary voltage:
1700V Negative

TABLE 2 Data Checklist

Empirical Laboratories

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: Perchlorate 6850

QA/QC Item	Yes	No	NA	Second Level Review
1. Has the instrument been tuned and has a tune verification check been analyzed and within ± 0.3 *m/z* perchlorate ions.				
2. Are the % RSDs within 20% or 0.995 corr. and minimum response factors within limits for the initial calibration?				
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and within 15 %?				
4. Does the Continuing Calibration Standard (CCV) meet ± 15% and the LODV meet ± 30% and analyzed at the desired frequency?				
5. Is the Method Blank and System Blanks run at the desired frequency and is its concentration for perchlorate < ½ RL?				
6. Are the LCS, MS, MSD within 80-120% recovery and RPD within 15% and run at the desired frequency?				
7. Are the retention times of the ions 99 and 107 within 0.2 min and RRT within 0.98-1.02				
8. Are all sample holding times met, perchlorate within calibration range, IS response within 50% of the average calibration response ?				
9. Is the ICS analyzed at the desired frequency and within 30%?				

Comments on any "No" response:

_____

_____

_____

Data flags needed? _____

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 302 **REVISION #: 17** **EFFECTIVE DATE: 042610**

PESTICIDE/PCBs
AQUEOUS MATRIX EXTRACTION
FOR EPA METHOD 608/608.2 AND
SW846 METHOD 8081/8082
USING SW846 METHOD 3510C

APPROVALS:

Lab Director: _____ Date: 4/27/10

Data Quality Manager: _____ Date: 4/27/10

Section Supervisor: _____ Date: 4/27/10

Changes Summary

Revision Date: 042610

- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

1.0 Identification of the Test Method

1.1 This SOP is compliant with SW-846 Method 3510C and Method 608/608.2

2.0 Applicable Matrix or Matrices

2.1 This SOP is applicable to aqueous samples

3.0 Detection Limit

Not Applicable to this SOP

4.0 Scope of Application, including components to be analyzed

Not Applicable to this SOP

5.0 Summary of the Test Method

5.1 Aqueous samples are extracted with methylene chloride. The extracts are dried through sodium sulfate and concentrated and exchanged to hexane.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 "Technical/Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2 Additional definitions specific to this SOP are listed below:

6.2.1 PCBs- polychlorinated biphenyls

6.2.2 Pest- pesticides

6.2.3 TCMX- tetrachloro-m-xylene

7.0 Interferences

7.1 Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.

7.2 Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.

7.3 Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

9.0 Equipment and Supplies

9.1 Separatory Funnel – 2L with Teflon stopcock
9.2 Beaker – 250mL or 400mL
9.3 Drying/Chromatographic column – 20mm I.D. x 300mm
9.4 Filter funnel
9.5 Turbo-Vap evaporation tube – 200mL tube made by Zymark or equivalent
9.6 Metal rack – capable of holding six glass evaporation tubes
9.7 Turbo-Vap Evaporator – heated and capable of temperature control (±5˚C); the bath should be vented into a hood
9.8 Vials, 10mL glass with Teflon-lined screw cap
9.9 pH indicator paper – wide range (1.0-12.0)
9.10 Syringe – 1mL
9.11 Graduated cylinder – 1000mL, 500mL, and 100mL, glass, Class A
9.12 Pasteur pipette – length 9”
9.13 Pasteur pipette bulb
9.14 Labels – Avery
9.15 Teflon Bottles – 500mL
9.16 Volumetric Flasks – 500mL, 100mL, 50mL, and 10mL, glass, Class A
9.17 Ring Stand – 3-prong
9.18 Burette clamp – double
9.19 Aluminum foil – heavy duty
9.20 Nitrogen tank – equipped with pressure regulator
9.21 Boiling chips – Teflon
9.22 Glass Wool – Roving, 9989 purchased from Fisher #11-388 or equivalent

10.0 Reagents and Standards

10.1 Reagents

10.1.1 Reagent water – Reagent water is gathered in a carboy from source in the instrument lab daily.

10.1.2 Sodium Sulfate – Granular, anhydrous, trace pure 10-60 mesh purchased in 200lb bulk fiber drum from Fisher #S415-200lb or equivalent. Place an aliquot in a 1500mL heavy-duty Pyrex beaker and bake in muffle furnace at 400˚C for a minimum of 4 hours. Remove and cool in open air and place in designated “Baked Sodium Sulfate” container at room temperature.

10.1.3 Sulfuric Acid Solution (1:1) – Slowly add 500mL concentrated Sulfuric Acid, purchased from Fisher #A300C-212 or equivalent, to 500mL of reagent water in a 1000mL Teflon container. This mixture will get very warm. Let stand until cool.

10.1.4 Sodium Hydroxide Solution (10N) – Weigh 800g NaOH, purchased in a fiber drum from Tennessee Reagents #2-31825-25lb or equivalent, into a 2000mL volumetric flask and add approximately 1000mL of reagent water. Swirl until pellets are mostly dissolved. Add a stir bar and place on stir plate. This mixture will get very hot. Continue to add reagent water while mixture is being stirred until a final volume of 2000mL is attained. Let stand until cool. Transfer to 1000mL Teflon containers.

10.1.5 Methylene Chloride - purchased from Fisher #D151-4 or equivalent. **Please see SOP 336 before handling this solvent in our laboratory.**

10.1.6 Hexane – suitable for gas chromatography, purchased from Fisher #H303-4

10.2 Standards – The extraction analyst makes up surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.

10.2.1 TCMX/DCB (2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl) – Surrogate solution is prepared, with a final concentration of 0.5ug/mL, by diluting a stock solution (purchased from Restek #32000) in acetone. This solution is named "Pesticide Surrogate for Extractions 500ppb" and expires 6 months after the date it is made. Use 1.0mL of this solution per 1000mL of aqueous sample.

10.2.2 PCB Spiking Solution – For all standard extractions, a mixture of 1016/1260 is prepared and used. The stock standards (purchased by Accustandard 1016 #APP-9-158-10X and 1260 #C260S-H-10X) are diluted in acetone to a final concentration of 5ug?mL. This solution is named "PCB 1660 LCS for Extractions 5ppm" and expires 6 months after the date it is made. Use 1.0mL of this solution per 1000mL of aqueous sample. The Laboratory Director and/or Organic Manager will determine if another PCB mixture is necessary, such as 1242, 1258, or 1254.

10.2.3 Pesticide Spiking Solution – A spiking solution, with a final concentration of 1ug/mL, is prepared by making a dilution of the Pesticide AB ICV Intermediate (this is made in-house by GC operators) in acetone. This solution is named "Pesticide AB LCS for Extractions 1.0ppm" and expires 2 weeks after the date it is made. Use 1.0mL of this solution per 1000mL of aqueous sample. For 608 samples, 1 out of every 10 samples must be spiked

10.2.4 TCLP- When necessary to set up a TCLP, in addition to setting up the sample, two matrix spikes must be set up and should include the following:

A. TCLP Spike 1 – This matrix spike must include a solution containing Chlordane at a concentration of 100ug/mL and Toxaphene at a concentration 10ug/mL. Both compounds are diluted in acetone from stock standards purchased from reputable vendors (Chlordane from Ultra Scientific #EPA-1086, Toxaphene from AccuStandard #P-0935-H). This solution is named "Tox/Chlor LCS for Extractions 10-100ppm" and expires 6 months from the date it is made. Add 1.0mL of leachate.

B. TCLP Spike 2 – This matrix spike must include the Pesticide Spiking Solution known as "Pesticide AB LCS for Extractions 10ppm." Add 1.0mL of this solution per 100mL of leachate.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 Aqueous samples have a hold time of 7 days from the date of sampling.

12.0 Quality Control

12.1 Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

13.0 Calibration and Standardization

Not applicable to this SOP

14.0 Procedure

14.1 All waters have a seven-day holding time counted from the day they are sampled. Determine the samples necessary to extract from the following (Note: never extract samples of unknown origin without discussion with supervisor):

- 14.1.1 Each day the extractions group leader will generate a sample backlog using LIMS
- 14.1.2 This backlog is used to determine extraction priorities based n hold times and due dates.
- 14.1.3 Samples requiring RUSH turn around time may be logged in throughout the day, which will require immediate attention. Sample receiving personnel will generally communicate this need.
- 14.1.4 Samples are placed in LIMS "batches" based on parameter and extracted accordingly.

14.2 Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass amber jars and have a Teflon lid.

14.3 Find out if any special dilutions need to be made for client. Routine procedures for difficult matrices are listed below:

- 14.3.1 Sludge – use only 100mL and dilute to 1000mL with reagent water
- 14.3.2 TCLP Extract – use only 100mL for the sample and dilute to 1000mL with reagent water. There must be two matrix spikes of 100mL as well that are also diluted to 1000mL with reagent water.
- 14.3.3 Bad Matrix – e.g. a liquid that is partially sediment. See Organics Supervisor to find out what dilution, if any, should be made.
- 14.3.4 NPDES client – Samples for method 608/608.2 are checked by login to make sure the pH of the sample is in the range of 5.0-9.0. If the sample is not in this range, extraction personnel will be notified. At that time, it is the responsibility of the extraction lab to adjust the pH of the sample to the appropriate range (pH of 5-9 using NaOH solution or Sulfuric Acid, as necessary) or to extract the sample within 72 hours of sampling. If a pH adjustment is made, the details of the adjustment must be recorded on the sample COC and in LIMS. Set up one full list matrix spike for every ten samples.

14.4 Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume.

14.5 Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH on the bench sheet and later, in LIMS.

14.6 Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and a LCS must be processed with each set of samples. If the sample is a TCLP, blank fluid may be provided along with the extracted TCLP sample(s). Follow instructions for TCLP in section 14.3.2 of this SOP. Process a matrix spike and matrix spike duplicate on aqueous samples if requested by client. If not, a LCSD must be processed.

14.7 Rinse separatory funnels with methanol and discard of waste according to SOP QS14.

14.8 Pour samples into separatory funnel, placing the label from the sample bottle on the designated separatory to ensure proper identification. Use Avery labels to properly identify method blank, LCS, LCSD, any TCLPs, and TCLP spikes. If a sample requires both Pesticide and PCB analysis, a Pesticide LCS/MS/MSD (if client specified) or LCS/LCSD and a PCS LCS/MS/MSD (if client specified) or LCS/LCSD must be processed to satisfy QC requirements for the batch.

14.8.1 Due to limited volume received, it is usually necessary to use 500mL of sample to do a matrix spike so that a matrix spike duplicate can also be extracted. If only one sample container is provided for spiking purposes, use a 500mL glass cylinder to measure out half of the sample for extraction. Add half of the normal amount of spiking solution and half of the normal amount of surrogate.

14.9 Add 50mL of methylene chloride to the empty sample container, swirl, and pour into the designated separatory funnel.

14.10 Using the 1L glass graduated cylinder marked “DIH20 WATER ONLY” measure 1L of reagent water from the carboy and transfer it to the designated separatory funnels for method blank, LCS, and LCSD.

14.11 Add 50mL of methylene chloride to the method blank, LCS, and LCSD.

14.12 Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set the surrogate/spike out at least ten minutes before use to allow it to warm to room temperature.

14.13 Using the 1.0mL glass syringe marked “TCMX/DCB” surrogate, add 1.0mL of TCMX/DCB surrogate to each sample, method blank, and spike. A second analyst must verify that the surrogate has been added. Enter the ID# of the standard, amount, and the initials of the analysts on the LIMS generated bench sheet and later in LIMS.

14.14 Determine if the sample will require a Pesticide spike, PCB spike, or both and proceed as follows:

14.14.1 Pesticide and PCB – Refer to 14.8 for instructions on how to determine QC requirements. To all Pesticide QC, add 1.0mL of Pesticide AB LCS with a glass syringe dedicated for that particular spike. To all PCB QC, add 1.0mL of PCB 1660 LCS using a glass syringe dedicated for that particular spike.

14.14.2 Pesticide only – To all Pesticide QC, add 1.0mL of Pesticide AB LCS with a glass syringe dedicated for that particular spike.

14.14.3 PCB only – To all PCB QC, add 1.0mL of PCB 1660 LCS with a glass syringe dedicated for that particular spike. 1660 is the standard PCB that we analyze for, if client specifies another PCB the extraction analyst will need to prepare another spike mix accordingly.

14.14.4 Enter the LIMS generated spike mix ID#, amount added, and the initials of the extraction and verifying analysts on the bench sheet and, later, in LIMS.

14.15 If the pH is not within 5.0-9.0 range, it must be adjusted using either the NaOH solution or Sulfuric Acid solution. If a pH adjustment is made, the details of the adjustment must be recorded in LIMS.

14.16 Seal and shake the separatory funnel vigorously for 3 minutes in the shaker apparatus with the stopcock open.

14.16.1 Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.

14.17 Allow the sample to set for a few minutes, if needed, after it has been shaken. It will separate into two layers with the solvent layer on the bottom.

14.17.1 If it forms an emulsion (thick, cloudy, viscous mixture that you cannot see through), drain what you believe to be 50mL into a 250mL centrifuge bottle.

14.17.2 Save and drain into this centrifuge bottle until the extraction is complete.

14.17.3 The emulsion must be centrifuged at 2500rpm for a good separation of the water from solvent.

14.18 Drain solvent layer into an appropriately labeled 250mL beaker.

14.19 Following steps 14.16 through 14.18, extract two more times with 40mL of methylene chloride combining all solvent extracts into the same appropriately labeled 250mL beaker.

14.20 Prepare a sample vial tray with 12mL vials and vial labels printed from LIMS. These labels contain the sample number, client name, initial/final volume, parameter, and date extracted.

14.21 Remove any water layer from the extract in the beaker or centrifuge bottle, by either or both of the following two methods.

14.21.1 Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not solvent. Discard this layer.

14.21.2 Use the smallest amount possible of Sodium Sulfate by sprinkling the top layer until it hardens, separates, and drops to the bottom.

14.22 Turbo-Vap Concentration

14.22.1 Rinse a Turbo-Vap tube and arrange it underneath a methylene chloride rinsed sodium sulfate filled filter funnel.

14.22.2 Using a sharpie, label the Turbo-Vap with the sample IDs

14.22.3 Pour the extract through the filter funnel into the appropriately labeled Turbo-Vap tube.

14.22.4 Rinse the beaker three times with methylene chloride and pour through funnel.

14.22.5 Rinse the filter funnel with methylene chloride once more and allow the funnels to sit until there is no more solvent dripping.

14.22.6 For solvent exchange purposes, add 50mL of hexane to each tube. Total volume in the Turbo-Vap tube should not exceed 200mL to avoid splattering

on the lid of the Turbo-Vap. If there is a large volume of methylene chloride extract, allow the sample to condense in Turbo-Vap until 75mL-100mL are left in the turbo tube.

14.22.7 Adjust pressure of nitrogen gas tank to >30psi, making sure that the tank has 200psi or more on the main valve.

14.22.8 Record the water bath temperature in the logbook located beside the TurboVap, making sure that it is 40˚C-50˚C.

14.22.9 Place turbo-vap tube in the Turbo-Vap. Be sure to push the tube down so the tip slides into the sensor well.

14.22.10 Close the lid and push corresponding well light to start concentration.

14.23 For PCBs Only – Some wastewater samples will form a gel like substance when the hexane is concentrated. Proceed with these samples as follows:

14.23.1 Add just enough methylene chloride to make the gel go back into solution

14.23.2 Acid clean the extract and reconcentrate.

14.23.3 Exchange with hexane again

14.23.4 If gel forms again, add enough methylene chloride to get gel back into solution

14.23.5 Transfer to a suitable container and record the final volume on the label and on bench sheet. Make sure to note the percentage of methylene chloride in sample.

14.24 When the samples reach a volume of 3mL-5mL, remove the tube from the batch

14.25 Hold the sample vial and tube in one hand at ~45˚ angle and 9" Pasteur pipette equipped with a latex bulb in the other.

14.26 Draw up sample and transfer into appropriately labeled 12mL sample vial. Be careful not to spill a drop during transfer.

14.27 Add 2-3mL of hexane to the tube and rinse several times using the pipette. Transfer this rinsate to sample vial and bring sample up to 10mL with hexane and cover the extract with a Teflon-sealed screw cap.

14.28 Take sample batch to GC Hobart sample refrigerator and log the sample numbers, analyst initials, and the date and time the samples were placed into the Hobart in the sample logbook located beside the refrigerator.

14.29 Transfer handwritten extraction details from bench sheet to LIMS and archive bench sheet for future reference.

15.0 Data Analysis and Calculations

Not applicable to this SOP

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to independently extracting samples and yearly thereafter. The analyst must prepare 4 LCS samples. The data is calculated for accuracy and precision requirements.

17.0 Pollution Prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Not applicable to this SOP

19.0 Contingencies for Handling out-of-control or unacceptable data

Not applicable to this SOP

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

21.1 *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition

21.2 40 CFR, Method 608

22.0 Tables, Diagrams, Flowcharts, and Validation Data

Not applicable to this SOP.

SOP Title:

BNA & Pesticide/PCBs & TPH NON-AQUEOUS MATRIX (MICROWAVE EXTRACTION) USING SW-846 METHOD 3546

SOP NUMBER: SOP-343

REVISION NUMBER: 0

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: 08/01/09

DATE OF LAST REVIEW: 08/01/09

BNA & Pesticide/PCB & TPH NON-AQUEOUS MATRIX
(Microwave Extraction)
Using SW846 METHOD 3546

1. SCOPE AND APPLICATION

a. This SOP describes the extraction of BNAs, pesticides/PCBs, and TPHs from soil, sediment, sludges and waste solids by an automated method (3546).

2. SUMMARY

a. Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Microwave extractor for BNAs, Pesticides/PCBs, or TPHs. The extracts are then concentrated by a Turbo Vap concentrator.

3. INTERFERENCES

a. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
b. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
c. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

APPARATUS AND MATERIALS

d. Stainless Steel spatula
e. Microwave extractor unit with 40 position carousel, electronic components, and ample ventilation
f. Microwave extraction Teflon tubes, capacity approximately 75mL
g. Suitable Teflon cap and screw-top lid
h. Drying column (Chromatographic column) – 20mm I.D. x 300mm
i. Vial – 2mL clear with Teflon-lined screw cap
j. Vial – 12mL clear with Teflon-lined screw cap
k. Syringe – 1mL, 500uL
l. Pasteur pipet – 9" length
m. Pasteur pipet bulb
n. Labels – Dymo
o. Aluminum foil – heavy duty
p. Nitrogen tank – equipped with pressure regulator
q. TurboVap Concentrator with 200mL concentrator tubes
r. Teflon funnels for pouring off
s. Balance – capable of weighing to 0.1grams
t. Aluminum pie pans for mixing samples
u. Filter paper – 185mm

4. REAGENTS

a. Sodium Sulfate (Na2SO4) – Granular, anhydrous, trace pure 10-60 mesh (purchased in bulk containers from Fisher #S415-10S or equivalent)
b. Methylene Chloride (Please read SOP – 336 before handling this solvent in our laboratory) (Dichloromethane) – suitable for spectrophotometry and gas chromatography (Fisher #D151-4 or equivalent)
c. Hexane – suitable for spectrophotmetry and gas chromatography (Fisher #H303-4)
d. Surrogate/Spike Solutions – Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes or if the initial concentration of stock is different than that listed below:
    i. **BNA Surrogate (100ug/mL)** – The base neutral and acid surrogates are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0ug/mL BN Surrogate spiking solution.)**
    ii. **BNA Spiking Solution #1 & #2 (100 ug/mL)** – The base neutral and acid spiking solutions are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor with same compounds as for calibration. Use 0.5 mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0 ug/mL PAH spiking solution.) The BNA Spiking solutions contain all targets that are calibrated for GC/MS. DOD QSM requires all targets to be spiked in the LCS and MS/MSD.**
    iii. **TCMX/DCB (2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl) Surrogate solution is** prepared in acetone by making a cut on stock purchased from a reputable vendor. 0.5mL at 0.5 ug/mL of this solution is added per 15g of non-aqueous sample.
    iv. **PCB Spiking Solution** – Arochlor 1016/1260 or the PCB of choice (1242, 1248, 1254, or 1260 are the most common) is prepared in acetone at a concentration of 5.0ug/mL. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Use 0.5mL per 15.0g of non-aqueous sample.
    v. **Pesticide Spiking Solution** – A spiking solution is prepared at 1.0 ug/mL. Use 0.5mL per 15g of non-aqueous sample.
    vi. **TPH Surrogate** – Surrogate solution is prepared in acetone by diluting stock ortho-terphenyl standard to a final concentration of 20 ug/mL. Use 1mL per 15 grams of sample.
    vii. **TPH Spike** – A spiking solution is prepared by extractions analyst that has a concentration of 1000 ug/mL in acetone.

5. SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

a. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a Teflon-lined cap.
b. Samples are preserved by cooling to 4°C.
c. Holding time is 14 days from collection date to extraction.

6. PROCEDURE

a. All soils have a 14-day holding time counted from the day they are sampled. Determine the samples necessary to extract using the following information (DO NOT extract samples for which you have no information.):
    i. Each day a backlog is generated in ELEMENT providing all relevant sample information, including samples numbers and respective analysis required.
    ii. Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
    iii. Check the backlog throughout the day to re-evaluate priority if needed.
b. Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a microwave extraction is truly necessary.
c. Get twice the number of aluminum pie pans to prepare the number of samples you have plus any additional spikes of LCSs and a method blank. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples). Using the LIMS, create a batch of samples and print off sample labels. The LIMS will create a unique batch sequence number.
d. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan.
e. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering process should be performed as follows:

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container

f. Place an aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the Extraction calibration/temperature logbook. Using a spatula, transfer the **appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}**, of a representative sample to the nearest 0.1 gram. Normally 10 or 15g sample weights are used. Record this amount on your label. Put your label on the side of the 400-mL beaker. For spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc.

g. Add ~ 15 grams of sodium sulfate to the aluminum pie pan. Using a spatula and/or a glass rod, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the spatula or glass rod from the mixed sample, leave behind all the sample possible. Cover the aluminum pie pan with foil and continue to weigh up the remaining samples. For the method blank and LCS, weigh up 15 grams of sodium sulfate. The matrix used for the method blank and LCS must be free of the analytes of interest and processed through the same analytical steps as the samples.

h. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by watching and signing off on bench sheet.

NOTE: Surrogate and spike should be added just prior to extraction.

i. Using the 1-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. **(For low level PAHs use 1.0 ml of the 1.0 µg/mL BN Surrogate spiking solution.)** or using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 0.5 mL of TCMX/DCB surrogate to each sample, blank and spike. TPH samples will need 1.0 mL of appropriate.

   For the BNA sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe marked Base Neutral Acid Spiking to add 0.5 mL of the Base Neutral Acid Spiking solution. **(For low level PAHs use 1.0 ml of the 1.0µg/mL PAH spiking solution.)**
   For Pest/PCB samples, determine if the sample will require a Pesticide Spike and/or a PCB Spike. Proceed as follows:
   Pesticide and PCB - set up two LCS's – one for Pesticide getting an AB MIX spike and one for PCB, which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.
   Pesticide only – To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.
   PCB only - To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB. Add 20 grams of Na2SO4.

j. Place a Teflon cap and Teflon screw top on the Teflon microwave tube. Using the cap tightener station, tighten the caps and invert sample to insure proper mixing and check for leaks in cap.

k. Place microwave tubes in microwave carousel making sure they are in order and spaced evenly throughout the carousel to insure proper heating while in microwave.

l. Place microwave carousel in microwave making sure the carousel is properly lined up with the turning mechanism.

m. Choose saved program option based on total number of samples to extract and begin process by pressing the start button. The program is set to EPA method 3546 specifications.

   For 1-15 samples:
   Max power: 800W 100%
   Ramp time: 15:00
   Control temperature (in Celsius): 110
   Hold time: 10:00
   Cool down: 5:00

For 16-40 samples:
Max power: 1600W 100%
Ramp time: 15:00
Control temperature (in Celsius): 110
Hold time: 10:00
Cool down: 5:00

n. Allow samples to cool in the carousel for an additional 30 minutes before attempting to handle the extracts.
o. Transfer the extract to a pre-rinsed turbo vap tube by first passing through a funnel with P4filter paper sodium sulfate. All tubes and funnels should be pre-rinsed with Methylene Chloride. After pouring the extract into the turbo, rinse the microwave tube 3 times with Methylene Chloride and transfer the rinsate to the turbo. Finally, rinse the funnel with an adequate amount of Methylene Chloride using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest.
p. Now concentrate the extract to 1.0mL using the turbovap concentrator.
    i. **Turbo-Vap Operation:** Adjust the pressure of nitrogen gas tank to 50 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 50-55°C. The pressure target range should be about 20-25 psi.
    ii. Place the turbo vap tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
    iii. When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
q. BNA and TPH samples need to be concentrated to ~1.0mL while Pesticides and PCB should be concentrated to ~5.0mL in turbo vap. Using clean solvent, rinse turbo with Pasteur pipet and bring sample to volume in sample vial.

7. DOCUMENTATION OF CAPABILITY (DOC)

a. Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

8. WASTE MANAGEMENT AND POLLUTION PREVENTION

a. Please see Waste Disposal SOP-405 for the proper disposal of waste generated from this area.
b. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

9. METHOD PERFORMANCE

a. Refer to SOP-201 SOP-211 and SOP-219 for method performance.

10. REFERENCES

a. EPA Methods SW-846, Method 3546

11. DEFINITIONS

a. Refer to SOP-431 for definitions.

12. HEALTH AND SAFETY

a. Wear appropriate personal protection equipment when working with chemicals or samples.
b. Use the lab hoods when working with solvents.
c. Use caution when mixing strong acids or bases. Solutions will become extremely hot when mixing with water. Avoid splashing these solutions so they won't come in contact with the skin or eyes. If this happens, flush with lots of water. Contact your supervisor if serious and medical attention is needed.

LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE STANDARD OPERATING PROCEDURES

SOP NUMBER: **SOP-404**

REVISION NUMBER: **13**

APPROVED BY:

SECTION MANAGER

TECHNICAL DIRECTOR

EFFECTIVE DATE: **06/29/09**

DATE OF LAST REVIEW : **06/29/09**

LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

This SOP lists in as much detail as possible our daily procedures for sample receiving, log in and storage of laboratory samples. Keep in mind that there may be project specific requirements that are more strict or different than our routine procedures. In these instances, the project specific requirements must be met and followed. Although a few project specific requirements are detailed in this SOP, i.e. USACE certification issues, not every situation can be addressed. If there is ever any uncertainty on what procedures must be followed, please see the Testing Coordinator immediately. If ever in doubt, always go with the more stringent requirements. This document will constantly be reviewed and revised as necessary.

SAMPLE ACCEPTANCE CRITERIA

A sample may be rejected for compliance purposes if it does not meet the following criteria. Analyses may only proceed after notification and approval to proceed from the client or from the laboratory manager.

1. Sample must be properly preserved and in the proper container for the requested analysis.
2. Sample integrity must be maintained. The container shall be intact without cracks, leaks, or broken seals.
3. Adequate sample volume must be received for the requested analysis, including volume for any requested QA/QC (MS/MSD).
4. The sample ID on the bottle label must match the sample ID listed on the chain of custody.
5. The sample container label and the chain of custody must be completed with indelible ink. The sample label must be intact and list all necessary information; to include: sample date, sample time, sampler, and sample ID/location. The chain of custody shall also indicate sample date and time, requested analyses, and all necessary client information.
6. Sample temperature must be less than 6°C or received on ice.
7. Sample must be within holding time for the requested analysis.

These issues are discussed in more detail below under the "Sample Receiving" section of this document.

I. Sample Receiving

A. Samples are received at the Empirical Laboratories on 621 Mainstream Drive, Suite 270 Nashville, TN 37228.

1. The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Empirical Laboratories Sample Receiving (SR) area loading dock in back of the laboratory. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we do pick up our

coolers at the FedEx location and transport them back directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client.

2. Some coolers and/or samples may be received directly by Empirical Laboratories Sample Receiving personnel. If samples are hand delivered by the client make sure that necessary paperwork is included and that you sign and date the chain of custody, as well as record the temperature of the samples on the chain of custody as well. If the *Empirical Laboratories* ***Chain of Custody [Attachment II]*** is used the white and yellow copy of the chain of custody is retained and the pink copy must be given to the client.

B. When going through the required steps for Sample Receiving and Sample Log In, keep in mind that a ***Corrective Action Report (CAR) for Sample Receiving [Attachment III]*** must be completed to document any problems, discrepancies, project changes, etc. encountered during the process. This includes but is not limited to incorrect sample containers, improper preservatives [chemical and temperature], chain of custody discrepancies, sample descriptions, etc. A CAR may be completed just to keep a record of a situation, which is not actually "out of compliance."

1. Make sure that all information on the CAR is stated clearly and very detailed. Many times it is necessary to refer to these documents a year or more after they were completed. Document all correspondence including name, date, company and response.

2. The CAR forms must be numbered starting with No. 001 at the beginning of the year (e.g. 01-001). No two forms should have the same number. All CARs must be forwarded to the Project Manager and/or receiving manager for approval and distribution. THIS MUST BE DONE ASAP OF WHEN THE PROBLEM/SITUATION IS DISCOVERED.

C. Visually inspect all coolers for tampering, custody seals present and intact (if applicable) leakage, etc. If a cooler has been damaged beyond repair, unpack the samples and discard the cooler as to not reuse it. If you suspect a cooler may be damaged or is extremely dirty this cooler must not be reused. If coolers were sent by Federal Express, examine the Federal Express airbills for the number of packages in the shipment and make sure that all the packages (coolers, boxes etc.) in a group have been received. If there are any problems the Project Manager must be contacted immediately. If anything looks unusual, take the time to check it out and document the situation and findings.

D. Open each cooler in order to quickly inspect the contents and to locate the chain of custody. Sample Receiving personnel should wear the following personal protection equipment: gloves, safety glasses and a laboratory coat. All coolers received from projects with the **US Army Corps of Engineering (USACE) and AFCEE** projects should be opened under the hood in the sample storage room. Sign then list the date and time received on the chain of custody. The time received <u>must</u> reflect the actual time the samples were received even though they may be logged into the system at a later time. Samples received on Saturday may be processed on the following Monday morning, or samples received late in the day during the week may be processed the next morning. All cooler(s) <u>must</u> be opened, examined for

leakage, breakage etc., the temperature measured and the chain of custody signed and dated to reflect the actual date and time which they were received. The samples must be delivered to the appropriate analytical department or put in cold storage as soon as possible.

1. Attach any shipping receipts, work orders, etc. to the chain of custody.

2. If a chain of custody or other paperwork is not sent, the client must be contacted and the samples temporarily placed on hold in cold storage. In some instances the log-in person may complete a chain of custody. The required information may be found on the sample containers or it may be necessary to call the client to get the missing information (i.e. sample ID, collection date and time, etc.). Note on the chain of custody that it was completed by laboratory personnel and record the name of the person with whom you spoke. All attempts to encourage our customers to complete a chain of custody or submit written information for samples must be made.

3. Project specific paperwork may be required. For all projects, a ***Cooler Receipt Form [Attachment IV]*** must be completed for each cooler received. Sample receiving personnel must begin completing this form as soon as a cooler is received and complete this form as samples go through the log in process.

E. The temperature of each cooler or set of samples must be measured as quickly as possible using a thermometer with 0.1°C increments. This thermometer must be calibrated against a NIST certified thermometer once a year and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometer must be tagged with the unique identification number of SR#1 and serial #; (Sample Receiving #1), the date calibrated and the correction factor. This information must also be recorded in a bound notebook. Only this thermometer can be used for recording the temperature of sample coolers upon receipt.

1. To measure the temperature, open the temperature control blank if supplied, point the IR thermometer at the liquid surface, wait 30 seconds for temperature to stabilize. Read the temperature to the nearest 0.1 °C. The corrected value temperature must also be recorded on the chain of custody. (This value will also be recorded into the LIMS at a later point.). All regulatory compliance samples received from North Carolina that do not meet the temperature requirement will be segregated and the client will be notified of the non-compliance. The samples will not be analyzed until we receive client notification to proceed with analyses.

2. If the temperature exceeds 6°C for any sample, the Project Manager or Sample Receiving personnel must contact the client immediately. There may be tighter temperature control limits for specific project requirements. The customer must make the decision to either continue with the analyses or resample. Make sure the client is aware that if the samples are analyzed, the following qualifier is normally included on the final report: "The shipping cooler temperature exceeded 6°C upon receipt to Empirical Laboratories. This may have an impact on the analytical results. The concentration may be considered as

estimated." Not all samples for the project will be flagged, just those samples received above 6°C.

Many times we are not able to get in touch with the client quickly and the best judgment on how to handle the samples must be made after discussion with the Testing Coordinator and/or Laboratory Director or Technical Director. The samples may still need to go through the log in process although it may be eventually determined that the samples will not be analyzed or the samples may temporarily be placed on hold and not logged in. Above all do not allow the samples to set out at room temperature for an extended period of time while waiting for a decision. **A CAR outlining the problem and all correspondence must be completed.**

The only exceptions to the 6°C rule are:

a. Water samples for all Metals, (except Chrome 6+ and mercury) that have been preserved with HNO_3 to a pH of ≤ 2. *Keep in mind that non-aqueous sample for Metals must be cooled.*

b. Samples for Fluoride, Chloride and Bromide.

c. Waste/Product samples for all parameters.

d. Samples generated in the Aquatic Toxicology laboratories and brought directly to Sample Receiving after they are collected. Sample receiving personnel should place these in cold storage as soon as possible.

e. Samples collected locally by Empirical Laboratories personnel or local customers that hand deliver their samples. In some instances these samples may not have had time to cool down; however, these samples should have been placed on ice in an attempt to cool them to the proper temperature. This exception is only applicable if the samples were collected the same day as the laboratory receives them. It should be noted if samples are "Received On Ice" (ROI).

f. Samples that are received on ice and it is evident that the client made a good faith attempt to properly cool the samples.

F. If several coolers are received at once, they must be inspected to determine the order in which the samples should be unpacked and logged in. The following priorities should be given:

1. Any analyses, which have a 24-72 hour holding, time. It is the log-in person's responsibility to notify the department manager or section group leader of such samples via e-mail and verbally.

2. Any sample which has almost exceeded its' holding time. (Especially watch for this with waters organic extractions, Solids and Sulfides, all of which have only 7 days). A list of parameters and holding times is posted in the log-in room.

   a. If a sample is received already out of holding time, the project manager must be contacted. The sample can be analyzed at the client's request, but it will be qualified on the final report as being analyzed out of holding time. The project manager must inform you of the client's need.

   b. If a sample is received with limited holding time remaining for any parameter it may be necessary to contact the project manager so that he/she can contact the client. If the sample has to be analyzed on a rush basis to meet the holding time a rush charge may apply. Also it may not be possible to analyze the sample within the holding time due to sample load, etc. A CAR must be completed.

3. Samples requiring rush turnaround.

   a. If sample(s) require 24-hour turnaround they will take first priority. Other rush requests also have high priority.

   b. The Project Manager and/or Section Manager must be contacted for approval concerning any unscheduled rush requests.

G. Unpack all samples from the cooler. If there are any known or suspected hazards this must be done under a hood. All coolers from USACE projects should be unpacked under a hood. It may be necessary to rinse off the outside of the containers in the sink and/or wipe them off with a paper towel.

   1. Visually inspect them for tampering and custody seals (if applicable). Sort and inventory the samples against the chain of custody by arranging them in the same order as they are listed on the chain of custody. Normally samples are assigned log numbers in the same order as they are listed on the chain of custody but for certain projects or situations it is acceptable to arrange them in a manner which will make them easiest to log in.

   2. Check for leakage as this could compromise the sample integrity. If any spillage occurred in the cooler make sure this is noted. Also list all the other samples in the cooler as cross contamination could occur. A CAR must be completed and the Project Manager and/or the customer may need to be notified in these situations. It may be necessary to resample.

H. Check the chain of custody information against the information recorded on the containers. If these do not agree, contact appropriate person (s) - Project Manager, sampler, client, etc. All problems must be documented with a CAR.

1. If major changes are made on the chain of custody received from an engineering job, then the PE should submit written confirmation of these changes or make the corrections and initial them directly on the chain of custody.

2. Any error found on the chain of custody must be marked through with one line, initialed and dated and the correction written in.

I. Note any unusual requests, methodology, hazards (known or suspected) to the Project Manager and/or Laboratory Section Manager or analysts before the samples are actually logged in. Keep notes of any problems (improper containers, preservatives, temperature, or descriptions, etc.) A CAR must be completed and the analyst or manager should be notified immediately. If ever in doubt, fill one out!

II. Sample Log In

A. After samples have been unpacked, sorted and reviewed, they are then ready to be assigned log numbers and continue through the log in process. Make sure that the parameters for the samples are clearly marked on the chain of custody. If we prepared the sample kits there should be a sample kit work order form. Contact the Project Manager if there are any questions, problems, etc.

B. Assign a work-order and sample number to each individual sample and record it on each sample container and the chain of custody.

1. All containers with the same description must have the same sample number even if they have different preservatives and require different tests. However, each different fraction (bottle type and/or preservative) should be designated with a letter (A, B, C, etc.)

2. Grab and composite samples from the same sample location must be considered as separate samples. It may be necessary to use "grab" or "composite" as part of the sample description to distinguish between the samples. Only assign different log numbers to them if the parameters are clearly marked as grab and as composite. Do not assume that VOC must be analyzed from grab samples so therefore the client must have taken a grab sample.

3. Sample numbers must begin with 001 at the beginning of each year (e.g. 0101001).

C. Check the following items and record this information on the cooler receipt form to further ensure sample integrity. A CAR must be completed if any of the following requirements are not met and it may be necessary to contact the client. We can perform the analyses in most cases and will do so with the client's approval, however the results may be qualified in some manner on the final report.

Preserving sample integrity throughout the log in procedure must be one of our section's top priorities. This includes not only ensuring that the proper chemical preservatives have been added but also that the samples are received and maintained at the proper temperature. ***When samples are unpacked they must be placed in cold storage within two hours even if they have not been through the entire log in procedure.*** All samples for NPDES compliance monitoring from North Carolina will be stored at a temperature range of 1.0 to 4.4°C. All other NPDES samples will be stored at 4.0 ± 2.0°C. On the days we receive a large volume of samples, or are short handed, etc., we may not be able to completely log in all samples until late in the day or even the next day. Samples should not set out at room temperature if there is a delay. The samples must temporarily be placed in cold storage until you are able to complete the log in procedure. This should also be done when we take lunch breaks.

[Make sure the VOC containers are not temporarily stored in a non designated VOC only storage area.]

1. Determine if the samples were received at the proper temperature. (See section IC)

2. The sample descriptions on the bottle should match those on the chain of custody. (See section 1H)

3. Check to determine if the proper chemical preservatives were added to adjust the sample to the correct pH. All regulatory compliance samples received from North Carolina that do not meet the preservation requirement will be segregated and the client will be notified of non-compliance. The samples will not be analyzed until notification to proceed with analyses is received from the client. A list of parameters and the required chemical preservatives is posted in the log-in room. The verification of this preservation will be recorded on the Cooler Receipt Form for all projects. If Empirical Laboratories prepared and shipped out the sample containers they will have been pre-preserved unless instructed otherwise by the client. Complete traceability of the preservatives used to pre-preserve the sample containers and to preserve samples in the log-in area is required. A bound notebook must be used to trace this information and must include the following: Lot #, Type of preservative, Date Prepped, Amount and Analyst Name. This information must also be labeled on each container, re-pipetter, etc. that the preservative is stored in. Each lot of HNO3 used for Metals preservation must be tested prior to using them for preservation. These analyses are kept on file.

   a. The pH of each container (except VOA vials) which requires pH preservation must be checked. Do not open and check the pH of VOA vials in sample receiving/log-in.

   b. The pH of preserved samples is checked and confirmed using pH narrow range indicator paper. When the client request pH analysis on samples and they must be reported and measured for pH using the narrow range paper, rather then a pH meter, the accuracy of each batch of indicator paper must be calibrated to the nearest tenth versus certified pH buffer and recorded into a bound logbook in accordance with SW846 method 9041A pH Paper method.

c. When taking the pH reading, DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot and dispose of this volume after the pH is taken. For some samples (wastes) the indicator paper may not be accurate due to interferences. The observation of the appropriate color change is a strong indication that no interferences have occurred. If it appears as if there is interference, the pH must be measured using the pH meter. [See SOP ATSD-187 pH, Electrometric.]

4. The following guidelines must be followed to check pH preservation:

a. Water samples for Cyanide analyses must be preserved to a pH of >12 with NaOH upon collection. If the pH of these samples is between 11.0-12.0 upon receipt, and the samples are at the proper temperature and not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to ≥12.0 unless project/client specific requirements are to contact the client first.

b. Water samples for Metals analyses must be preserved to a pH of ≤2.0 with HNO_3 upon collection. If the pH of these samples is between 2.0-.3.0 upon receipt, and the samples are not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to ≤2.0. unless project/client specific requirements are to contact the client first.

c. Samples requiring analyses which are preserved with H_2SO_4 (i.e., Nitrogen compounds, Total Phenolics, Oil and Grease, Total Phosphorus, etc.) can be accepted up to a pH of 2.5 without a CAR, however the sample should be adjusted ≤2.0 unless project/client specific requirements are to contact the client first. Samples for sulfide analysis must have a pH >9.

d. If a sample is not properly preserved, log-in personnel must either do the following:

- To meet project specific requirements, including all USACE projects, the client must be notified before preserving or adding additional preservative to the sample unless otherwise instructed. If the client instructs us to add chemical preservatives to a sample, complete traceability of the preservatives used is required (See section IIC, #3). A CAR must be completed.

- For other projects it may be acceptable to preserve the sample accordingly before the sample is placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.

- All metals samples preserved upon receipt must be held 24 hours before proceeding with analysis. These samples must be CAR generated and the client notified to see if the lab is to proceed with analysis.

e. In some instances it may not be possible to adjust the sample to the proper pH due to matrix problems which cause excessive foaming or require an unusually large amount of acid. Do not continue to add acid if a few mL's of acid does not lower the pH. Notify the Project Manager, Metals Manager and/or analyst. They will make the decision if the sample will be diluted, not analyzed, etc. A CAR must be completed in these situations. Make sure you note on the container and in the LIMS notes that the sample is not at the proper pH as well as any useful information (i.e., foaming, strong odor, etc.).

f. A CAR may not be required for samples generated in the Aquatic Toxicology Laboratories and brought directly to Sample Receiving after they are collected but before they are preserved. Log-in personnel must preserve the samples accordingly before they are placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.

5. Check to make sure samples are in proper containers and that there is adequate volume for all the parameters requested and no leakage.

6. If VOA vials are present, each vial must be inverted and checked for head space. "Pea-sized" bubbles (i.e. bubbles not exceeding 1/4 inch or 6 mm in diameter) are acceptable and should be noted, however, a CAR is not required. Large bubbles or head space is not acceptable and a CAR must be completed. If this occurs, the client must be contacted. The samples can be analyzed with their approval, however the report will be qualified and the data may be questionable. All VOA vials will be preserved with $Na_2S_2O_3$ (0.2g) when chlorine is known to be, or suspected to be present.

7. All pesticide samples to be analyzed by method 608 will be checked by the sample receiving personnel for the correct pH range of 5.0 to 9.0. The pH of the sample(s) will be communicated via E-mail to the Section Manager and appropriate analyst.

8. All chlorinated effluent samples received for Cyanide must be checked for residual chlorine. The one liter sample container should initially contain 1 to 2g/L of Ascorbic Acid. Potassium Iodide starch indicator paper will be used for detecting the presence of residual chlorine. DO NOT PUT THE TEST PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot, neutralize, test and dispose of this volume after the sample is checked. If the test paper turns blue, the sample must be treated for residual chlorine. Add Ascorbic Acid, approximately 0.6g at a time and recheck the sample until there is no residual chlorine present. If the sample required this treatment this information must be included in the LIMS notes. This must be done by log-in personnel before leaving the receiving area. It may be necessary to notify the Inorganic Manager and/or analyst.

9. Be aware of holding time requirements. (See section 1D)

D. Once sample containers have been numbered, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A ***Sample Receiving Custody and Disposal Form [Attachment VII]*** must be completed each day. Samples should not leave the log-in area until this has been completed. *[see IIC; it may be necessary to temporarily store samples in cold storage until the samples can be second checked, the amount of time that the samples are at room temperature must be minimized as much as possible.]* The original is to remain in Sample Receiving until the samples are disposed of. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:

1. Client and Log #s
2. Date/Time Unpacked
3. Logged In/Numbered By (Initials)
4. 2nd Checked By (Initials)
5. Date/Time Placed in Cold Storage
6. Storage Area (Walk In, VOC Cooler, Quarantined Soils, Quarantined-VOC, Other)
7. Disposed of By/Date
8. Method of Disposal

E. Notify the proper analyst if samples have been logged in for analyses which have a 24-48 hour holding time or if a 1-2 day turnaround has been requested. The log number and description on sample (s) must be second checked before it is released to the analyst. (The analyst can second check the sample, but must initial the custody form.)

III. Sample Storage

A. After samples have been correctly logged in they are then transferred to one of the following cold storage areas and arranged in numerical order by the assigned log in/LIMS sample number. ***Note that aqueous VOC samples must be segregated from all other samples.***

1. The Hobart refrigerator in the MS Lab: All aqueous VOC's must be stored in this refrigerator. Storage blanks consisting of organic free water from the laboratory may be required for specific projects. These will be analyzed for VOCs only. ***Storage blanks are required for all DOD projects.***

2. Walk In Refrigerator: All aqueous samples for all analyses must be stored in this refrigerator.

3. Soil Walk-In Refrigerator: All quarantined and non-quarantined soil samples for all analyses must be stored in this refrigerator.

B. Quarantined soils are those quarantined by the US Department of Agriculture. These soil samples must be segregated from other soil samples during storage. A separate disposal log must be maintained for these soils including the location, date and quantity of the soil received and processed. Soil residues from quarantined samples must be treated according to regulations after testing (see Sample Disposal SOP). Quarantined soils are defined as:

1. Soil taken from much of the southeastern US and parts of New York and Maryland at a depth of three feet or less. *Soils from three feet or more are not regulated provided they are stored separately.* A map of the regulated areas in the United States entitled ***Soil Movement Regulations [Attachment VIII]*** is posted in the log-in room.

2. All soils taken from foreign sources, US. Territories and Hawaii.

NOTE: All soils are treated as quarantined soils and are disposed of in accordance with USDA regulations. Above for information purposes only.

C. All samples must be stored in one of the three refrigerators detailed above with the following exceptions:

1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)

2. Highly contaminated waste or product type samples that could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Project Manager for other options.

D. The temperature of each sample refrigerator must be monitored and recorded each day by Wet Chem personnel by the following method. A Mercury thermometer or digital min/max thermometer with 1° increments must be used. Each thermometer must be calibrated against a NIST certified thermometer once a year **(digital thermometers quarterly)** and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometers must be tagged with a unique identification, the date calibrated and the correction factor.

The tolerance range for all refrigerators is 1 to 6°C. This range and the range using the corrected reading must be posted on the outside of each cooler. If the temperature exceeds this range, corrective action measures must be put in place immediately. A CAR must be completed specifically noting the date and time the problem was discovered. The Project Manager, Laboratory Director and Technical Director will be notified in order to assess the situation. It may be necessary to put a service call in to the refrigeration repair service.

E. All personnel removing samples from any refrigerator must sign them in and out. This is done by completing the ***Sample Custody Form [Attachment IX]*** which is attached to the door of each refrigerator. These completed forms are kept on file [see section III, #4F]

F. The water walk in refrigerator in the sample room is the largest refrigerator and stores a large majority of the samples. A back up compressor is hooked into the system and scheduled to automatically come on if the main compressor fails. There is a digital min/max thermometer, which monitors the temperature 7 days a week. This thermometer will be calibrated quarterly against the NIST thermometer.

G. As stated above the temperatures for all refrigerators that samples are stored are checked each day Monday-Friday and monitored seven days a week with min/max thermometers. Pay close attention to these readings and watch for signs of possible problems.

H. A temperature maintenance record book is kept for each refrigerator.

I. Samples must be held for a minimum of 30 days after the final report unless specified otherwise. For USACE projects, samples must be held for a minimum of 60 days after the final report unless otherwise specified. See SOP ATSD 405 entitled Analytical Laboratory Waste Disposal SOP for guidance on disposal of samples.

IV. Laboratory Information Management System (LIMS)

A. Log the sample information into the LIMS for each sample. Every attempt should be made to get every sample logged into the LIMS by the end of the day. All information entered should be clearly stated and recorded on the COC provided. After opening the main menu of the LIMS, select the 'Work Orders' tab from the 'Sample Control' drop down menu. Now click on the 'Add' button to create a new Work Order. You will see the following:

1. ***Client*:**

   Select the client I.D. by clicking on the pull-down and choosing from the client list. This list is in alphabetical order. If the desired client is not on the list, a new client must be created by the project manager or I.T. director.

2. ***Projects*:**

   Click on 'Projects' and choose the project I.D. The projects will be client specific. After the project is chosen the "project information" areas should fill in. The 'Project Name,' 'Project Number,' 'TAT,' 'Client Project Manager,' 'Lab Project Manager,' and 'Comments' information should also appear. If there are no applicable project choices, a project must be created by the project manager or I.T. director. There are two types of projects:

a. Internal -- Empirical Laboratories projects;

b. External -- direct laboratory clients.

3. ***Comments:***

This area is to be used to note any information from the project manager for all work orders of this project. It can also be used to list any work order specific notes; this includes but is not limited to information concerning rush turnaround, deliverables or other QC requirements, analyte concentrations, safety issues, quarantined soils, CAR #s, preservation or matrix problems, etc.

4. ***Received By:***

Enter the name of the person who received the samples.

5. ***Logged In By:***

Enter the name of the person who logged in the samples.

6. ***Received:***

Enter the date and time received separated by a space and using military time.
Example: *08/02/2008 08:30*

7. ***Project/Package Date Due:***

After the date and time received have been entered, the date due for both of these fields will be calculated. If this information is not correct or needs to be amended later, check with the project manager before doing so.

8. ***Shipping Containers:***

Click on the 'Coolers' button and enter the temperature and condition upon receipt. If more than one cooler was received, each cooler must be assigned a different name. For example, if these came in by dedicated courier, enter the last four numbers of the Tracking Number as the name. After all of a cooler's information has been entered (received on ice, where custody seals present, preservation confirmed, COC/container labels agree, sample containers in-tact) click the 'Save' button. If more than one cooler was received, click the 'Add' button and repeat the process above, then click 'Done' after all the coolers' info has been saved.

9. ***COC Number:***

If an identifiable COC number is listed, record that ID here.

10. ***Shipped By:***

Enter the courier used to deliver the samples. If the samples were picked up by a lab employee or dropped of by the client/representative, enter 'Hand-Delivered.'

After these items have been completed, click 'Save,' then the 'Samples' button to continue. To begin entering information for a sample, click the 'Add' button on the bottom of the Samples screen.

11. ***Sample Name:***

a. Only abbreviate if description is too long for the spaces allotted in the LIMS. This information should come directly from the chain of custody. The sample ID entered into the LIMS will be the sample ID on the final report.

b. If no sample ID is provided, or is indistinguishable from other samples listed, contact the project manager to ascertain distinction in the samples. Include date as part of the description if this is the only way to differentiate the samples.

c. When logging in trip blanks that do not have an ID assigned by the client, list them as "Trip Blank # _____". This information should be on the containers. A log book must be kept in the sample kit room which lists all trip blanks and the date they were filled. This will ensure consistency with the descriptions for trip blanks. Make sure you record the trip blank on the chain of custody if it is not listed.

12. ***Collection Date:***

Enter the date and time the sample was collected. You must use military time and separate by a space. Often the time collected is not given. Although this is a sampling requirement, this information may not be crucial unless a parameter with a short holding time or a data deliverables package is required. In the event that a sample collection time is not listed on the COC or the sample container, a default time of 00:00 can be used temporarily until client verification. Once verified, then the correct sample collection time must be input into LIMS. If the COC and sample containers do not list a collection date and time, a CAR must be generated. All attempts should be made to get all our clients to supply this information.

13. ***Lab/Report Matrix:***

Click on pull down and select matrix. Many times it is difficult to discern the matrix if it is not specified on the COC, and log-in personnel must use their best judgment with

regard to analytes/methods requested. Keep in mind that the detection limits and units on the LIMS reports are linked to the matrix. In some cases it may be necessary to ask the Section Managers about the matrix selection. Log-in may do a dilution test to distinguish water samples from oil samples if the COC does not clarify a sample matrix if need be.

14. ***Sample Type:***

This is used to differentiate between special types of samples (i.e. Field Duplicates, Equipment Blanks, Trip Blanks, etc.). If there is no definite way to determine that a sample should be classified as something else, then "SAMP-Client Sample" will be selected as the sample type. Do not list a sample as anything other than a Client Sample unless noted on the COC of are instructed by the client to do so.

15. ***Container:***

Click on the drop down list and select the appropriate bottle type. If multiple bottles are received for the same sample, then move down to the next line and select all other containers as required. Repeat this process until all containers for the sample are listed. As each container is entered, an individual number is assigned to it by the LIMS system. This number is also listed on the container labels that are printed from the LIMS, and is placed on the corresponding bottle for container tracking purposes.

16. ***pH*** (Container Preservative)***:***

Use this to document the pH check information taken during sample unpacking. If no preservative was used, then nothing is required in this field.

17. ***Comments:***

Enter any information that is applicable at the sample level.

18. ***Field Analysis:***

Click on field analysis tab and enter field information when provided.

19. ***Work Analyses:***

Select all parameters requested for the sample from this list.

a. If the required test code is not listed, and the sample matrix is not a contributing factor, click the Work Analyses tab to open the All Analyses list. When selecting from this expanded list, be careful to select the proper method as all methods available for the current matrix will be selectable.

b. If any analyses are selected from the All Analyses list, the Project Manager in charge should be notified so that the correctness of methods and pricing can be checked and updated as needed.

c. All preparation codes for analytes are entered and stored by the system independently of the test codes selected, except in the cases of Dry Weight analysis, and TCLP/SPLP preparation (tumbling). In the case of the TCLP/SPLP prep codes, these are entered alongside the other required analyses automatically by the LIMS when a TCLP/SPLP analyte is selected. As for Dry Weight, it is required for all solids testing except in the cases of TCLP/SPLP analysis, Explosives only analysis, and/or any pure product/non-soil based sample when specified by the client.

20. ***Analyses Comments:***

These comments should be used for any notes that only apply to that particular test code.

21. ***RTAT:***

If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.

22. ***Save:***

Once all applicable information is entered for a sample, click the save button. At this time the LIMS applies the Laboratory Sample ID to the sample. This is a four part ID code composed of the following:

a. A 2-digit numeral of the year. Example (**08**11248-06)

b. A 2-digit numeral of the month. Example (08**11**248-06)

c. A 3-digit numeral of the work order number. This number reset to 001 at the beginning of each month. Example (0811**248**-06)

d. A 2-digit numeral of the sample number separated by a dash. Example (0811248-**06**). This number is different for each sample in a work order, and a single work order cannot contain more than 99 samples. If more sample numbers are needed, a new work order number will have to be assigned to the applicable set of sample.

23. ***Add/Edit/Copy:***

Use these selections to add more samples to the work order, or to change existing information prior to label printing.

Once all the tests have been selected and all samples have been added in the work order, a work order summary and all container labels are printed. Labels are checked for accuracy against the containers while being labeled. At this point log-in of this group of samples is complete.

B. After log-in of a work order is complete, the COC can then be scanned into the system, attached to the work order on the Work Order screen, and the work order can be updated to Available status so as to be seen by the analysts.

V. Daily Follow Up for Sample Receiving/Log In

A. Wipe out the inside of coolers and return all Empirical Laboratories coolers to the sample kit room. Discard any coolers that are cracked, broken or filthy.

B. If any samples were received for RUSH turnaround, then a ***RUSH SHEET [Attachment XII]*** must be completed and distributed to all laboratory personnel via e-mail. If ever in doubt of which analysts should be notified, pass them out to everyone. Always give copies to the Laboratory Director, Administrative Assistant and Section Managers. It may be necessary to send out two RUSH sheets per day (one around mid-day and the other at the end of the day).

C. Complete any required CARs.

D. At the end of the day organize all paperwork received and generated for the day. The following should be given to the Project Manager (section supervisor):

1. The original chains of custody and yellow original or copy of each. The CRF will accompany the COC for the project.

2. Any information (letters, regulatory limits, etc.) from a client which was received with any samples.

3. All CARs.

4. LIMS sample receiving logs.

5. Copies of any RUSH sheets which have been distributed

6. Sample Receiving Custody and Disposal Form.

7. Cooler receipt form.

E. All the above information from the day will be reviewed as soon as possible.

1. All LIMS logs must be 2nd checked by a different person than the person entering the information into the LIMS. Each set of logs must be initialed dated by the person 2nd checking. These will be kept on file at the Project Manager desk.

2. If any corrections or changes are required, all laboratory personnel will be notified by distributing a ***Sample Log Change Form [Attachment XIII]*** through email distribution. A ***Sample Log Change Form*** by the project manager will also be sent out if a client adds or deletes any parameters, changes sample IDs, etc.

F. The Testing Coordinator will distribute the following after they have been through the 2nd QA check:

1. Copies of the LIMS receiving reports to necessary laboratory personnel.

2. Original (white copy) chains of custody are given to the Project Manager. These will be sent with the final report to the client.

3. Finalized/approved CARs must be sent to the:

a. Organic Manager

b. Inorganic Manager

c. Laboratory Manager

d. Laboratory Director {optional}

e. Quality Assurance Officer

f. Administrative Assistant

g. Client {optional}

4. Copies of any project/sample specific information to the Section Manager and analysts.

G. Information will be filed as follows:

1. Chains of custody:

a. Original (white copy) is returned to the customer with the final report along with the CRF.

b. Pink copies should be retained by the sampler.

2. CARs

a. CARs can be found at V:\LAB\log-in\login (year)\logcar (year).

3. Sample Change Forms and RUSH Sheets

a. Sample Change Forms are distributed by email.

b. RUSH Sheets are found at V:\LAB\login\Rushsheets

4. At the end of each year, files for that year are boxed and archived. Make sure files are labeled properly and place them in banker's boxes. Complete a storage box file form with as much detailed information as possible. The Laboratory Administrative Assistant will label and number the boxes and incorporate the storage boxes into the laboratory file archive system. Boxes containing files from Sample Receiving are kept on site for 1-2 years and then may be moved to off site storage upon release from the Project Manager.

VI. Miscellaneous

A. All projects which require deliverables or other QC requirements should be listed in the notes section of the LIMS.

B. If samples are received from a new client or a new job number that is not in the LIMS, a new client code must be set up. This information should be on the chain of custody or it may be necessary to contact the customer if the information is incomplete.

C. Samples from the Aquatic Toxicity Laboratory (ATL) are logged into the LIMS for billing and long-term tracking purposes. The receiving information and proper assignment of tests are reviewed by the ATL Manager. The samples are then logged in by ATL personnel.

D. A flow chart outlining sample receiving and the flow of data, reporting and invoicing is attached as ***Attachment XIV.***

E. A ***Telephone Conversation Log [Attachment XV]*** may be required to document information and may be attached to or used as a CAR.

F. All log books used in the Sample Receiving and Sample Storage Areas are numbered. The following log books are presently maintained. All log books must be "Z"ed out. The Testing Coordinator will review the log books each week to check for completeness.

Log Book ID	**Log Book Description**
LI014	Trip Blank Prep Log Book
LI009	Tracking of VOC Trip Blanks Shipped
LI011	Quarantined Soil Treatment Log Book
LI012	Acid Neutralization Log Book
LI013	Sample Receiving and Disposal Log Book
LI010	Kit Room Preservation Preparation Log Book

Attachments to SOP 404

II	Chain of Custody Record
III	Corrective Action Report for Sample Receiving/Log In
IV	Cooler Receipt Form
V	List of Short Holding Time (Immediate-72 hrs.) Parameters
VII	Sample Receiving Custody and Disposal Form
VIII	Map of Quarantined Soil Areas in the US.
IX	Laboratory Sample Custody Form for Walk In Refrigerator
X	Container Codes for the LIMS
XI	Routine NPDES Clients
XII	RUSH Sheet
XIII	Sample Log Change Form (Green Sheet)
XIV	Flow Chart, Laboratory Sample Tracking System

[Attachments I and VI were removed during the editing process and not added to the SOP.]

EMPIRICAL LABORATORIES, LLC - CHAIN OF CUSTODY RECORD

SHIP TO: 227 French Landing Drive, Suite 550 ♦ Nashville, TN 37228 ♦ 615-345-1115 ♦ (fax) 615-846-5426

Send Results to:	Send Invoice to:	Analysis Requirements:	Lab Use Only:
Name _____	Name _____		VOA Headspace Y N NA
Company _____	Company _____		Field Filtered Y N NA
Address _____	Address _____		Correct Containers Y N NA
City _____	City _____		Discrepancies Y N NA
State, Zip _____	State, Zip _____		Cust. Seals Intact Y N NA
Phone _____	Phone _____		Containers Intact Y N NA
Fax _____	Fax _____		
E-mail _____	E-mail _____		Airbill #: _____
Project No./Name:	**Sampler's (Signature):**		CAR #: _____

Lab Use Only Lab #	Date/Time Sampled	Sample Description	Sample Matrix														Comments	No. of Bottles	*Lab Use Only* Containers/Pres.

Sample Kit Prep'd by: (Signature)	Date/Time	Received By: (Signature)
Relinquished by: (Signature)	Date/Time	Received By: (Signature)
Relinquished by: (Signature)	Date/Time	Received By: (Signature)
Received for Laboratory by: (Signature)	Date/Time	*Temperature*

REMARKS:

Details:

Page _____ of _____

Cooler No. _____ of _____

Date Shipped _____

Shipped By _____

Turnaround _____

Distribution: Original and yellow copies accompany sample shipment to laboratory; Pink retained by samplers.

Attachment III

CAR 09-

EMPIRICAL LABORATORIES

CORRECTIVE ACTION REPORT FOR SAMPLE RECEIVING/LOG-IN

Date Completed:

Form Completed By:

Date Samples Received:

Parameter(s):

Client/Job #:

Samples:

Problem(s):

Action Taken:

Action Taken to Prevent Reoccurrence of this Problem:

Approval of Section Leader: _____

Distributed to:

EMPIRICAL LABORATORIES
COOLER RECEIPT FORM

LIMS Number: _____ Number of Coolers: _____ of _____

Client: _____ Project: _____

Date/Time Received: Date cooler(s) opened:

Opened By (print): (signature): _____

Circle response below as appropriate

1. How did the samples arrive?: FedEx UPS DHL Hand Delivered

EL Courier Other: _____

If applicable, enter airbill number here: _____

2. Were custody seals on outside of cooler(s)? ..... Yes No

How many: _____ Seal date: _____ Seal Initials: _____

3. Were custody seals unbroken and intact at the date and time of arrival? ..... Yes No N/A

4. Were custody papers sealed in a plastic bag included in the sample cooler? ..... Yes No N/A

5. Were custody papers filled out properly (ink, signed, etc.)? ..... Yes No N/A

6. Did you sign custody papers in the appropriate place for acceptance? ..... Yes No N/A

7. Was project identifiable from custody papers? ..... Yes No N/A

8. If required, was enough ice present in the cooler(s)? ..... Yes No N/A

Type of Coolant: WET DRY BLUE NONE Temperature of Samples upon Receipt: _____

Dates samples were logged-in:

9. Initial this form to acknowledge login of sample(s): (Name): **WILLIAM SCHWAB** (Initial): _____

10. Were all bottle lids intact and sealed tightly? ..... Yes No N/A

11. Did all bottles arrive unbroken? ..... Yes No N/A

12. Was all required bottle label information complete? ..... Yes No N/A

13. Did all bottle labels agree with custody papers? ..... Yes No N/A

14. Were correct containers used for the analyses indicated? ..... Yes No N/A

15. Were preservative levels correct in all applicable sample containers? ..... Yes No N/A

16. Was residual chlorine present in any applicable sample containers? ..... Yes No N/A

17. Was sufficient amount of sample sent for the analyses required? ..... Yes No N/A

18. Was headspace present in any included VOA vials? ..... Yes No N/A

If Non-Conformance issues were present, list by sample ID: _____

_____ CAR#: _____

Short Holding Time Parameters

(Immediate-72 hours)

Parameter	Holding Time
pH	Immediate [a]
Sulfite	Immediate [a]
Temperature	Immediate [a]
Residual Chlorine	Immediate [a]
Coliform (Fecal and Total) RCRA/WW	6 hours
Hexavalent Chromium (Cr +6)	24 hours
Odor	24 hours
Coliform (Fecal and Total) *Drinking Water only*	30 hours
BOD	48 hours
Color	48 hours
Settleable Solids	48 hours
MBAS	48 hours
Orthophosphate	48 hours
Turbidity	48 hours
Nitrite	48 hours
Flashpoint	72 hours [b]

[a] Immediate generally means within 15 minutes of sample collection.

[b] This is an internal holding time. The method does not specify a holding time.

Attachment VII

Empirical Laboratories, LLC

SAMPLE RECEIVING CUSTODY AND DISPOSAL FORM

Date Samples Received _____

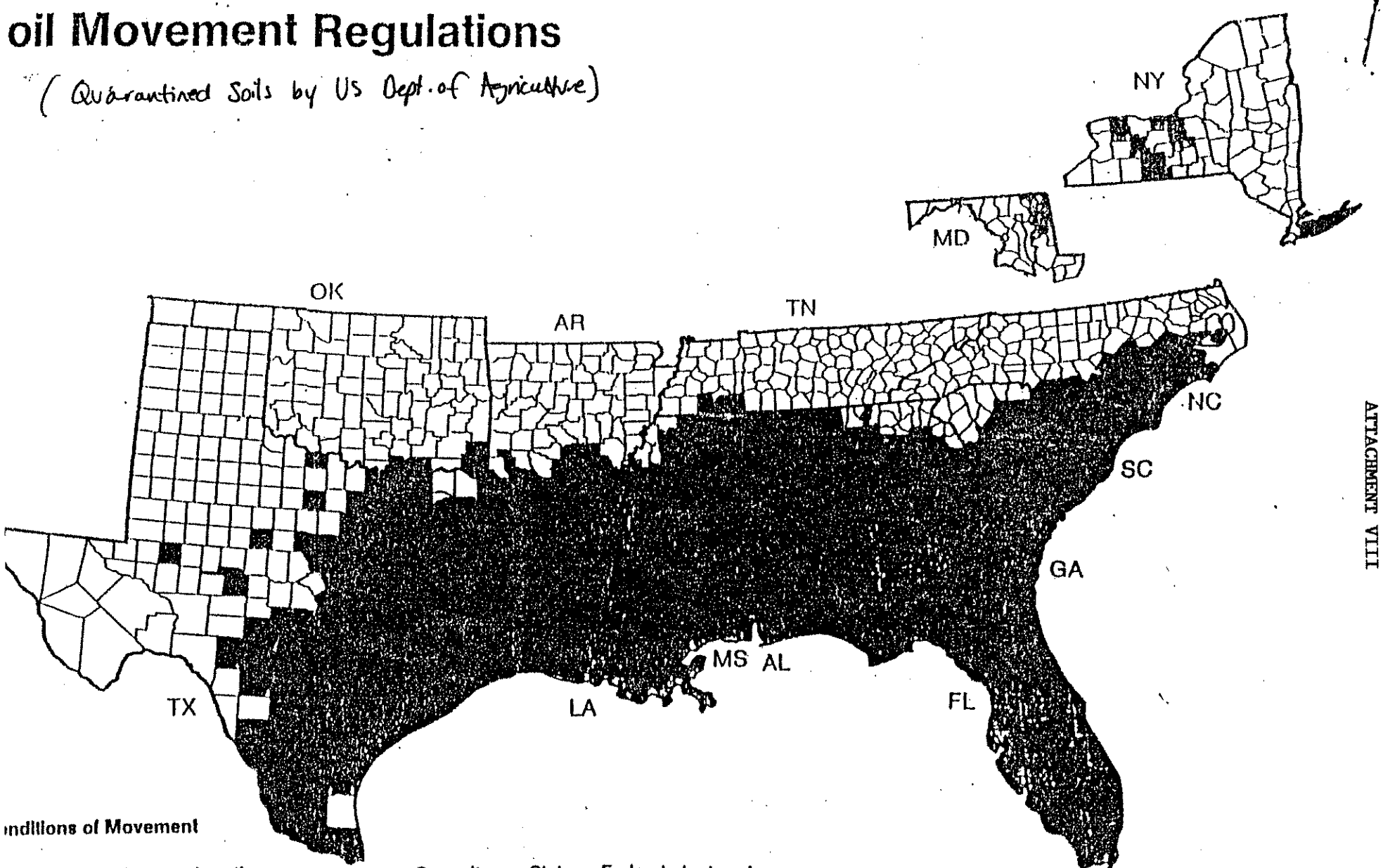
Client	Log #'s	Date Unpacked	Logged In/ Numbered By	2nd Checked By *	Placed in Storage By	Date Placed in Storage	Storage Area (1)	Disposed of By/Date	Method of Disposal

(1) W=Walk in Refrigerator
HV= Hobart Refrigerator, VOC samples only
Q= Refrigerator for Quarantined Soil Samples
QV= Refrigerator for Quarantined Soil, VOC samples only
O=Other (Must Specify)

***= MUST HAVE INITIALS OF THE PERSON WHO SECOND CHECKED AND MUST BE FILLED IN BY THAT PERSON IN INK .**

oil Movement Regulations

(Quarantined Soils by US Dept. of Agriculture)



ındıtıons of Movement

ıstrıctıons are Imposed on the move-
ınt of regulated articles from a regulated
ɜa as follows:
ɔm red areas into or through white
ɜas. Movement within red areas may be
gulated.

Consult your State or Federal plant protection inspector or your county agent for assistance regarding exact areas under regulation and requirements for moving regulated articles.

Regulated Area

ATTACHMENT VIII

Empirical Laboratories

EMPIRICAL LABORATORIES, LLC
LABORATORY SAMPLE CUSTODY FORM
WALK-IN REFRIGERATOR

Sample Log # (s)	Time/Date/Initials Removed	Time/Date/Initials Returned (Note if all Sample Used)	Notes/ Comments	Task Performed

Preservatives		Types of Container	
NI	*HNO3*	**A**	*1 LITER - PLASTIC*
NF	*HNO3 (Filtered)*	**B**	*500 mL - PLASTIC*
SU	*H2SO4*	**C**	*250 mL - PLASTIC*
SH	*NaOH*	**D**	*120 mL - PLASTIC*
ZN	*ZnAC / NaOH*	**EN**	*ENCORE PAK*
HY	*HCl*	**F**	*1 LITER - GLASS CLEAR WIDE MOUTH*
		G	*1 LITER - GLASS CLEAR BOSTON ROUND*
		H	*1 LITER - GLASS AMBER*
		I	*250 ml. - AMBER*
		J	*VOA VIALS - (40 ml.)*
		K	*500 ml. - (16 oz)*
		L	*250 ml. - (8 oz)*
		M	*125 ml. - (4 oz)*
		N	*60 ml. - (2 oz)*
		O	*OTHER*
		P	*PLASTIC BAG -1 Gallon*

ATTACHMENT XI

ROUTINE NPDES CLIENTS (Page 1 of 2)

ALCAN Ingot and Recycling
Amoco Oil
Armstrong (Pirelli)
Elf Atochem-Carrollton, KY
Auburn Hosiery Mill
Autostyle

Bando Manufacturing
Bowers Ink
Bowling Green Municipalities (City of)
BP Oil
Bremner, Inc.
Brentwood, City of
Brown Printing Central

Cargill Steel and Wire
Clarksville Products

E. I. Dupont
Eaton and Olsen
Emhart Pop Rivets

Fleet Design
Franklin, City of

Gatlinburg

Hennessey Co. (Coats)
H.I.S. Laundry
H.K. Bell (City of Hopkinsville Landfill Monthly Monitoring)
Hoover

International Paper
J. S. Technos

Ken Koat
King Industries

Lannom Tannery
Leonard Plating

:al Plate, Inc.
Morflex, Inc.

Nashville Wire
Norandal USA Inc.

Oak Ridge, City of

Plymouth Tube
Prime Colorants

RMI

Shared Hospital Services
Snap On Tools
Springfield, City of
Special Metals
te Industries

Tennessee Dickel Distillery
Tullahoma, City of

UCAR, Clarksville

Valmore Leather

Westvaco (Mayfield Creek Up/Down)
Woodbury

Revised 9/10/96

Date Distributed 1/01/02

RUSH

DATE RECEIVED	CLIENT	LOG # (S)	PARAMETERS	E-MAIL COPY DUE (DATE / TO)	FAX COPY DUE (DATE / TO)	*FINAL COPY DUE (DATE / TO)

*ALL FINAL REPORTS SHOULD BE DELIVERED TO TERESA WILLIAMS OR HERBIE JOHNSON THREE DAYS PRIOR TO FINAL DUE DATE TO CLIENT.

ATTACHMENT XII

ATTACHMENT XIII

96-0001

SAMPLE LOG CHANGE FORM

DATE:

TO:

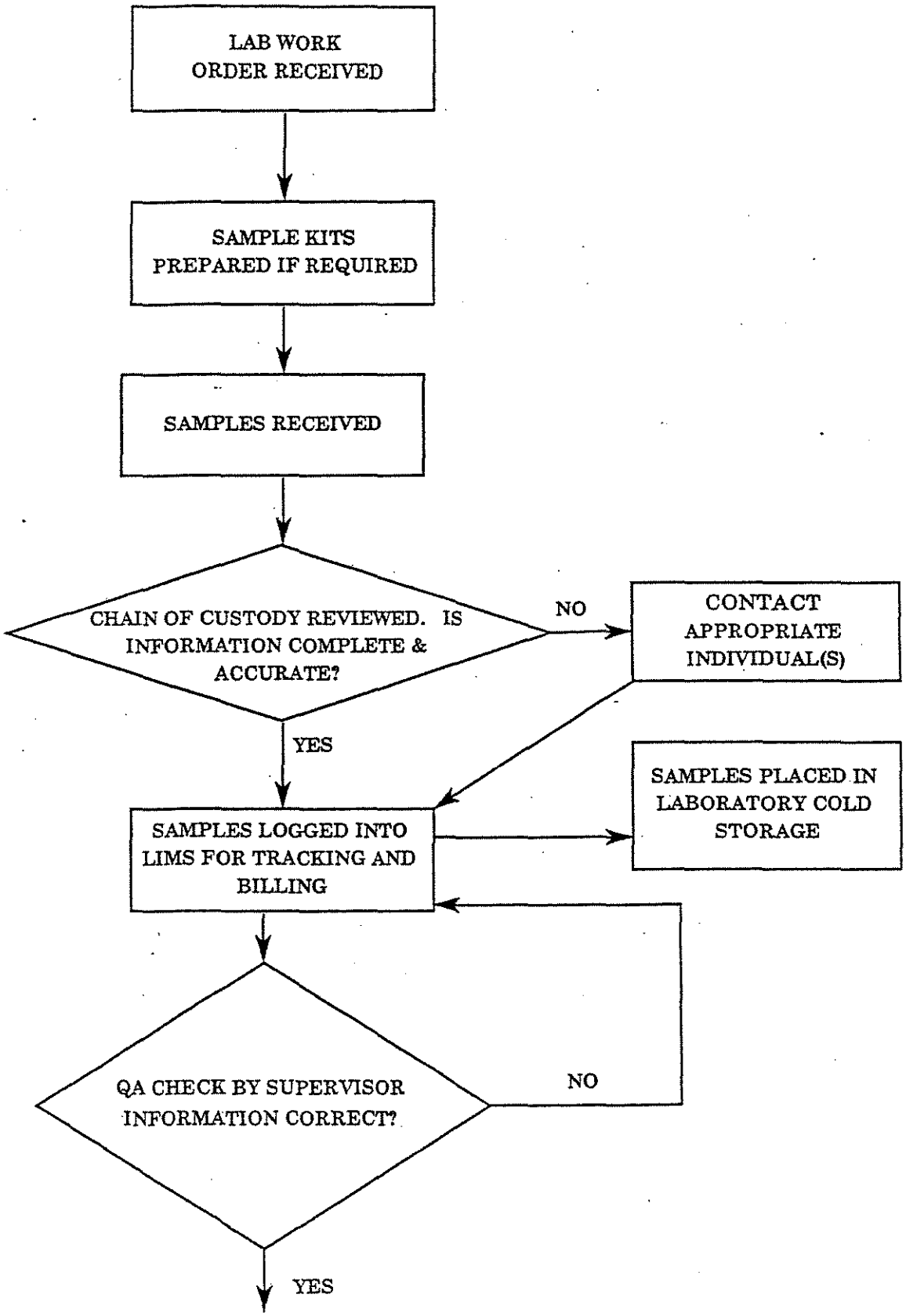
SAMPLE #(S):

CLIENT:

Changes:

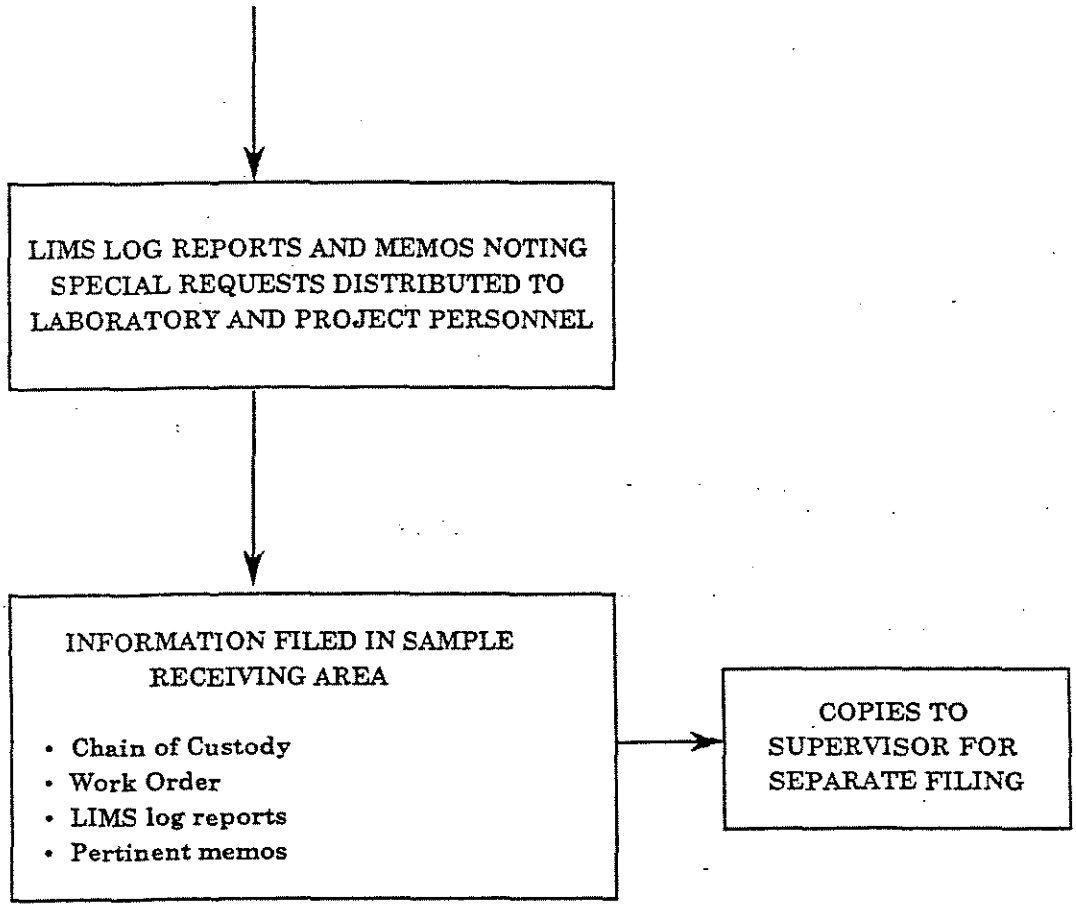
ATTACHMENT XIV

LABORATORY SAMPLE TRACKING SYSTEM
SAMPLE RECEIVING



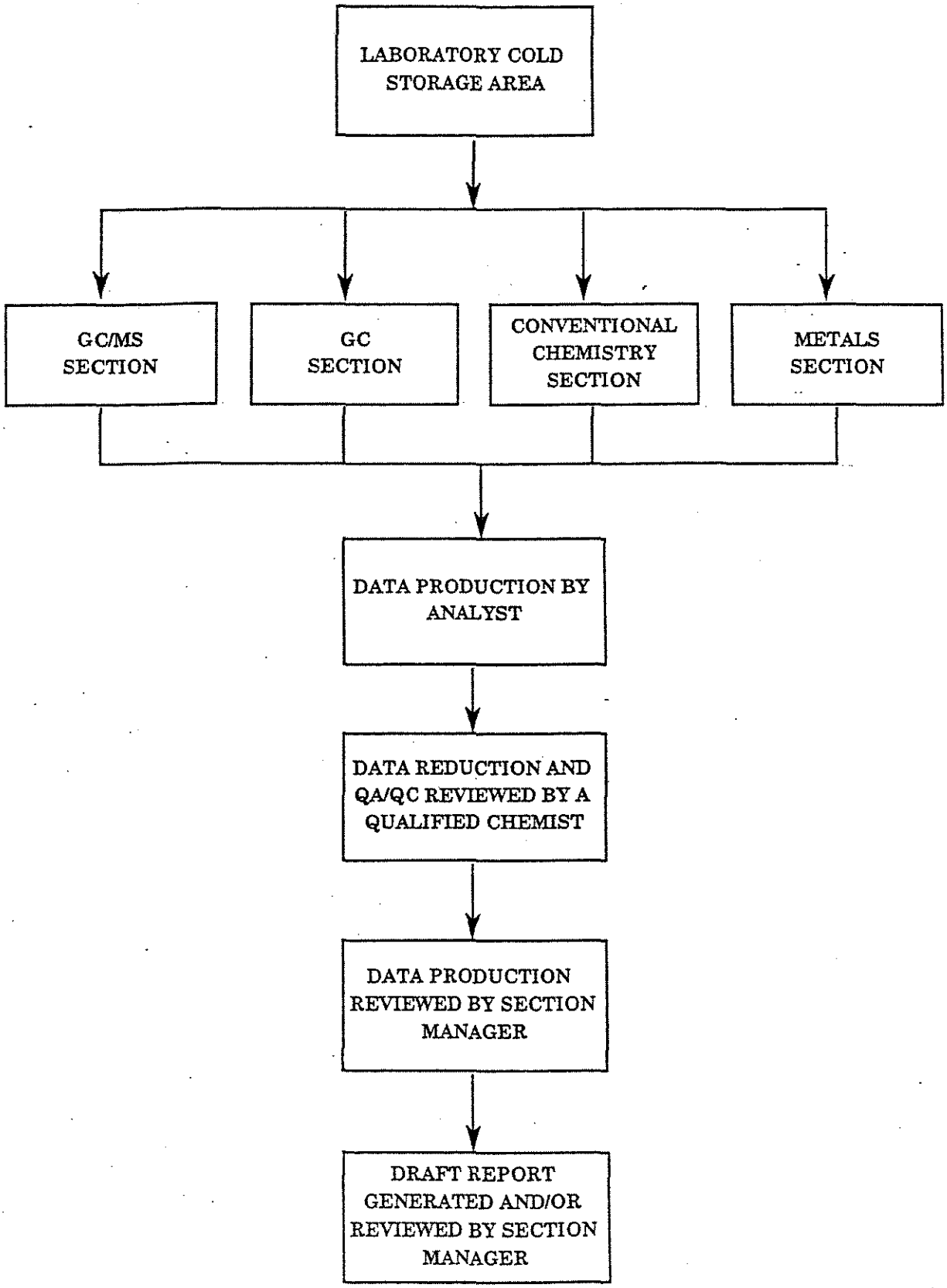
L:\QAM\SAMPTRAK.AF2

LABORATORY SAMPLE TRACKING SYSTEM
SAMPLE RECEIVING (continued)

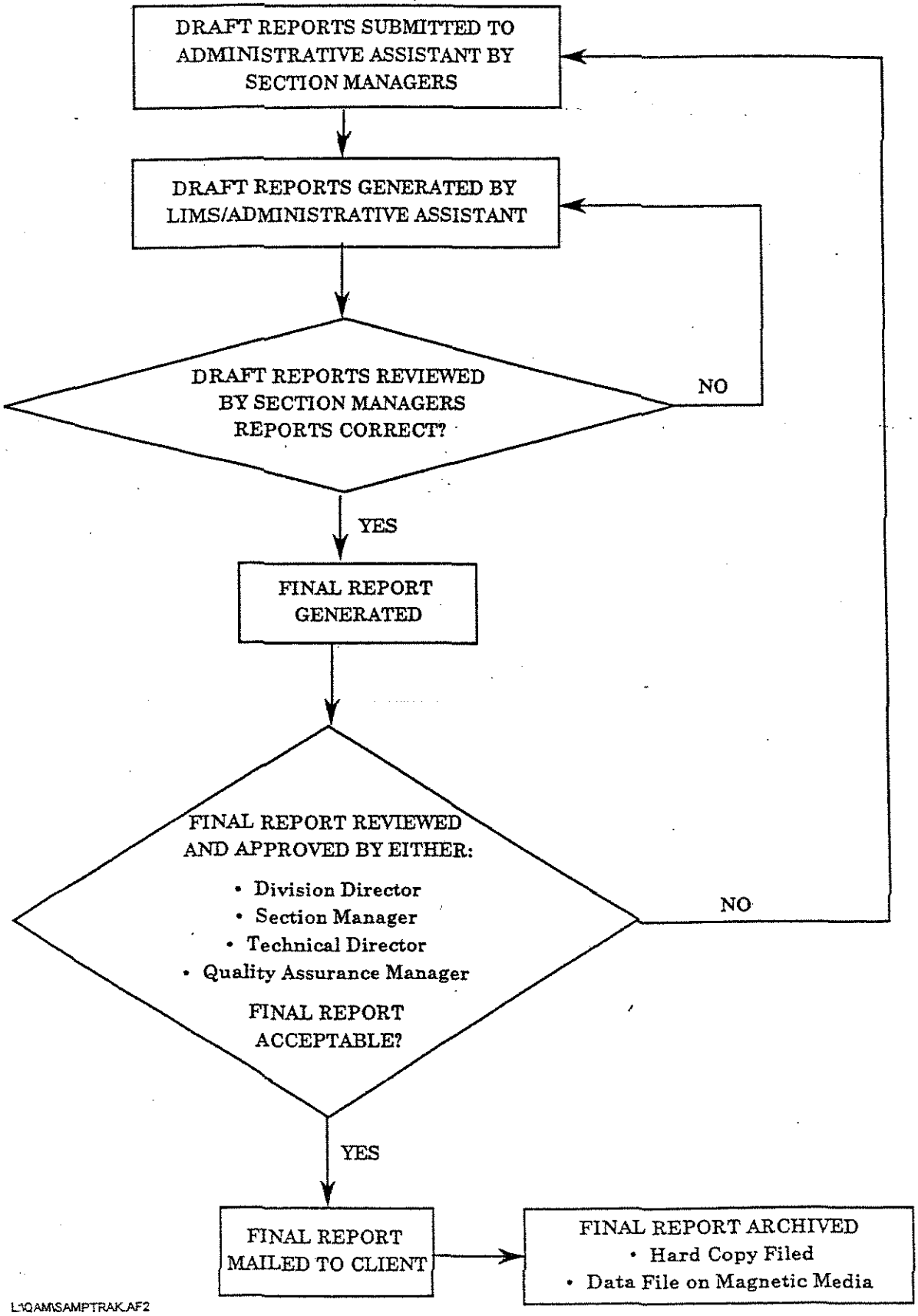


ATTACHMENT XIV (Continued)

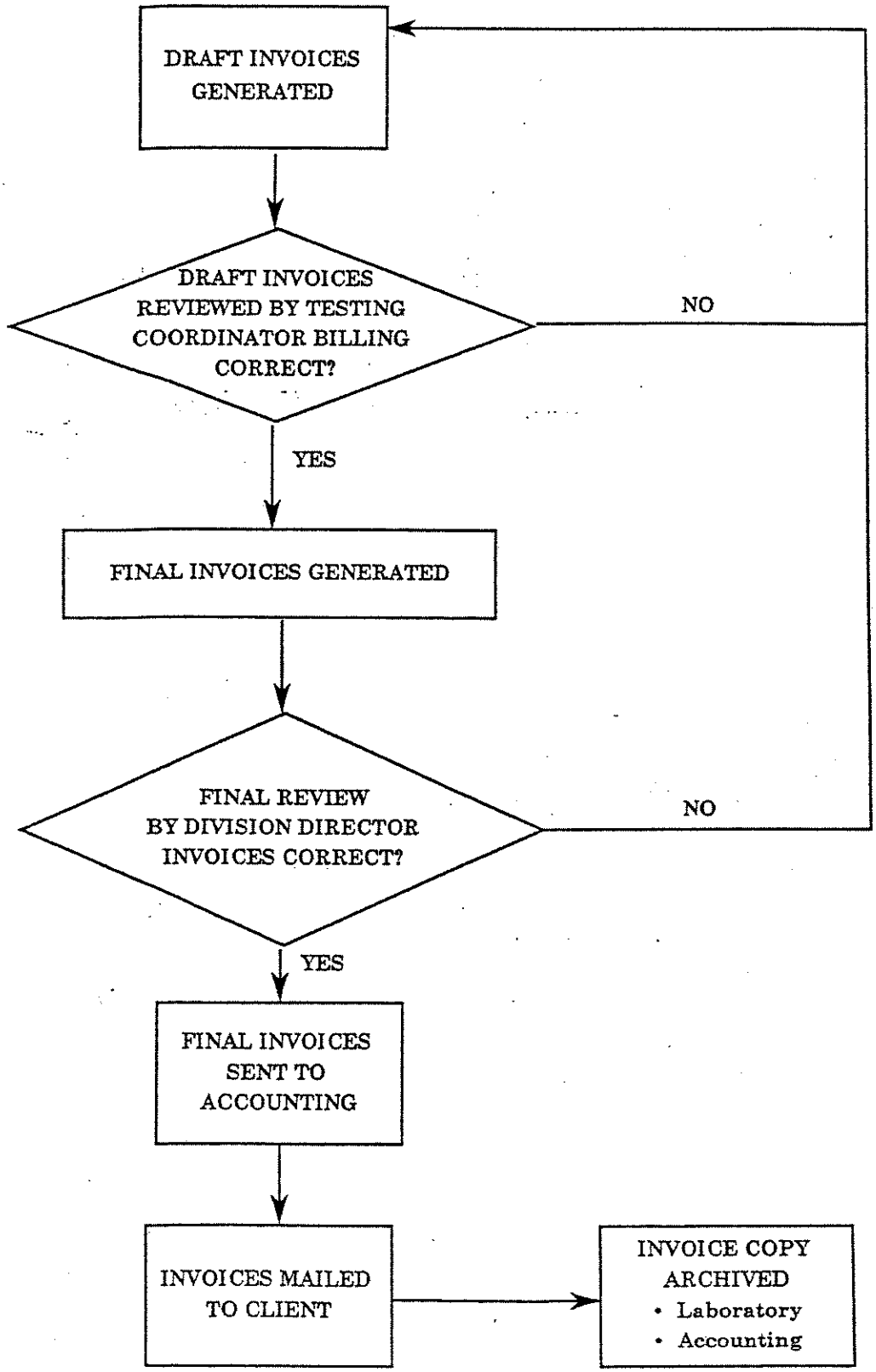
LABORATORY SAMPLE TRACKING SYSTEM
DATA PRODUCTION AND REVIEW



LABORATORY SAMPLE TRACKING SYSTEM
DRAFT AND FINAL REPORT



LABORATORY SAMPLE TRACKING SYSTEM
INVOICING



DISTRIBUTION/TRAINING LOG

LABORATORY SAMPLE RECEIVING, LOGIN AND STORAGE STANDARD OPERATING PROCEDURES

SOP NUMBER: **SOP-404**

REVISION NUMBER: **13**

RECEIVED BY/DATE:

W. Schwab

F. Rivers

~~R. Townsend~~

Signature above signifies acknowledgement of responsibility to know and follow the contents of this document. It also signifies receipt of training covering all new aspects of the SOP.

TRAINED BY:

EFFECTIVE DATE: **06/29/09**

PLEASE COLLECT OLD SOPs AND RETURN WITH SIGNED FORM TO QAO

ANALYTICAL LABORATORY WASTE DISPOSAL

SOP NUMBER: **SOP-405**

REVISION NUMBER: **5**

APPROVED BY:

LAB DIRECTOR

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: **06/23/09**

DATE OF LAST REVIEW: **06/23/09**

Analytical Laboratory Waste Disposal
Standard Operating Procedure

I. SCOPE AND APPLICATION:

Empirical Laboratories, LLC laboratory waste includes excess client sample waste and waste that are generated while performing an array of analytical services, some of which are hazardous. These wastes must be disposed of in a manner that is safe, cost efficient and in accordance with hazardous waste regulations.

A. Wastes can be broken down into the following categories:

1. Unused portions of actual samples received from outside clients.

   a. Unused aliquots of completed water samples.

   b. Unused aliquots of completed non-aqueous samples.

2. Soils from quarantined areas

3. All other soils, sediments, building debris, wipes etc.

4. Hazardous waste generated within the laboratory as part of numerous analytical procedures.

II. SUMMARY OF PROCEDURES:

A. There are four options for disposing of unused sample portions:

1. Return completed samples and any generated waste from these samples to the client.

2. Throw the sample away after confirming that it is non-hazardous.

3. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year.

4. Treat the sample to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

B. There are two options for disposing of laboratory generated waste:

1. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year. The waste must be stored properly until the waste is transported off site.

For example: Solvent waste must be stored in the vented flammable cabinet.

2. Treat the waste to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

III. EQUIPMENT/APPARATUS:

A. Proper safety equipment in good working condition. This includes gloves, lab coat and safety glasses/goggles (voluntary use of cartridge respirator allowed see area manager or QAO).

B. USDOT approved drums for storing and shipping hazardous waste.

C. Fume hood vented outside the building.

D. Flammable storage cabinet which is vented to the outside

IV. PROCEDURE

Waste disposal is done under the management and coordination of the Sample Receiving Manager, Section Managers and the Health and Safety Officer.

A. Disposal of completed aqueous samples:

Completed samples are kept in cold storage for approximately three weeks after the final report has been mailed. Engineering support projects involving CLP work, litigation cases etc. may be saved for longer than three weeks at the request of the project manager.

No samples should be disposed of without approval from the responsible area manager or analyst. **At this point the area manager and/or analyst will communicate information about samples deemed as hazardous.**

1. The majority of the water samples (ground, surface and drinking) is non-hazardous and is disposed of by pouring them down the sink.

a. This must be done under the hooded area located near the sink in sample receiving. Make sure that the sash is closed far enough to produce sufficient ventilation. The tap water should be turned on to supply copious wash for sample disposal.

b. Proper safety equipment **must** be used including safety glasses (face shield if necessary), lab coat and gloves.

c. **be alert to potential problems: for example, separate Cyanide waste from acid waste. Neutralize acid waste that will be poured down the drain and don't mix waste/samples thought to contain Cyanide with samples that are acidified. Also, look for things such as phase separation, odd color, odor etc. Check with the area manager or Health and Safety Officer before disposing of any questionable samples.**

d. Tap water must be running during the time samples are poured out and for approximately 10 minutes after so sufficient flushing and dilution takes place.

e. All containers must be rinsed out, all identifying markings defaced or removed, and thrown into the trash.

f. All samples disposed of in this manner must be documented in the bound disposal log.

2. If water samples are hazardous (known or suspected), one of the following steps must be taken.

a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**

b. Treat the sample to make it non-hazardous. One example of this is if the sample is highly corrosive, the pH may be adjusted.

c. Store the sample properly until either a sealed drum or lab pack is sent out.

d. All samples disposed of in this manner must be documented in the bound disposal log.

B. Disposal of completed non-aqueous samples:

The majority of non-aqueous samples are soils or sediments, although there may also be building debris, wipes, oils, and occasionally product type samples.

1. If samples are non-hazardous they must have all identifying markings defaced or removed, and thrown into the trash. On specific projects we may also opt to return the unused portions to the client even if they are non-hazardous.

2. If non-aqueous samples are hazardous (known or suspected), one of the following steps must be taken.

a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**

b. Store the sample properly until a lab pack is sent out.

3. Soil samples taken at a depth of three feet or less from areas, which have been quarantined by the US Department of Agriculture (USDA), must first be treated at the laboratory to prevent the spread of any plant pests. The USDA has detailed proper treatment procedures of which we use the following:

a. The sample is heated to 180°C(356°F)in a vented oven for two hours.
b. After the heating the samples are placed close to a hood to cool and are marked as being ready for disposal.

4. Once the samples have undergone treatment they can then be disposed of by one of the procedures for non-aqueous samples. **All samples disposed of in this manner must be documented in the bound disposal logbook with the following information:**

a. Client
b. Sample #s

c. Date(s) treated
d. Treatment method used

C. Disposal of laboratory generated waste:

Generated waste is stored outside the building, inside the caged fence until a waste pick up occurs. This area must be maintained properly.

1. Waste handling and disposal within each laboratory section:

Each laboratory analyst and section manager is responsible to assure that **handling** operations within their area are being followed according to the laboratory requirement.

a. General Chemistry/Inorganic

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, **(>2% by volume) and** dilute mercury waste (mercury, chemical oxygen demand, total kjeldahl nitrogen and chloride analyses waste) are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry**.
- Dilute acid waste **(<2% by volume or less)** are neutralized using concentrated amounts of sodium hydroxide and poured down a sink drain within hooded ventilation with copious amounts of tap water. The amounts of acid waste treated along with the amount of sodium hydroxide used to neutralize the acid waste, is then recorded into an acid waste neutralization log book that is kept in sample receiving.
- **All other non-hazardous sample waste, reagents and standards are poured down the drain with copious amounts of tap water.**

b. Metals

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed

below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, aqueous sample waste digestates and old unused calibration standards **(>2% by volume)** are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence.
- Non-aqueous sample digestate wastes are decanted off the soil/solid samples into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Rinse the soil/solid with tap water several times and discard the first rinsate into the Acid Satellite Waste drum and the sequential rinsates decant down an acid drain with copious amounts of tap-water.**
- **Throw the soil/solids in the trash once the acid has been rinsed free.**
- **Cr6 digestates as with all concentrated metal/acid waste are poured into the Acid Satellite Waste drum.**

c. Organic Extraction Laboratory Area

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the organic manager or the safety officer.

- Concentrated acid waste is discarded into the Acid Satellite Waste located outside the back of the building inside the caged fence.
- Non-chlorinated solvent waste (Acetone, Ether, Hexane, and Methanol ....etc...) pour into the Non-Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.
- Chlorinated solvent waste (Methylene Chloride, Chloroform, chlorinated standard and spike waste) pour into the Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.

****Note: Laboratory generated solvent waste is transferred to the appropriate Satellite Solvent Waste Drum (chlorinated or non-chlorinated) weekly or as deemed necessary. Disposal of solvent waste is done under the direction of the organic laboratory manager. These drums are located outside the back of the building inside the caged fence and only authorized laboratory staff are allowed to add waste solvent to these drums. The date of addition to the drum, type and quantity of solvent is entered into the '*Organic Solvent Waste Logbook*' located on the shelf next to the drums.**

- **Aqueous sample waste from extracted samples (once the extraction solvent has been removed) is poured down the drain and flush with copious amount of tap water.**
- Non-aqueous sample waste and sodium sulfate waste is dumped into a waste container under an extraction laboratory hood and left overnight or until the solvent is evaporated and then the waste is discarded into the trash.

d. Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Laboratory

- Autosampler vials are discarded into the appropriately labeled box located in the GC/HPLC Laboratory.

PCB Box – all samples/standards
Pesticide Box – all samples/standards
Herbicide Box – all samples/standards
8330 Box – all samples/standards
Methylene Chloride Box- all samples/standards that contain methylene chloride (Diesel Range Organics, DRO)

- Sample and spike extract vials are separated according to the contents in the vial. **Acid cleaned extracts** are combined into a separatory funnel and the acid layer separated from the solvent. The acid portion is discarded into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. The solvent waste is discarded into the appropriate solvent waste bottle (chlorinated/non-chlorinated waste) located in the hood in the organic extraction laboratory.

Unused stock and working standards are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

e. Gas Chromatography/Mass Spectrometry

- Volatile sample, standard and reagent waste

Waste from the instrument - Aqueous sample waste is collected in waste bottles via waste lines from the instrument. The bottles are emptied into buckets and poured down the drain(pH is < 2% by volume). A small amount of methanol used to clean glassware is also dumped into the bucket and poured down the drain. While disposing of sample waste always run the cold tap water 10-15 minutes. Non-aqueous waste from sample analyses is retained and dispose of in the same manner as the unused sample. Unused sample is held for sample disposal by the sample receiving area, see A and B listed above.

Standards - Unused stock and working standards are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

In conjunction with section managers, the sample receiving area disposes of solid sample waste and unused aqueous and solid samples see procedures A and B listed above.

- Semivolatile sample and standard waste disposal

Methylene chloride waste solvent and standard waste in vials are poured into the chlorinated waste bottle in the hood in the organic extraction laboratory. The empty vials are rinsed with solvent and the solvent poured into the waste solvent bottle. The vials with labels removed are discarded into the glassware waste disposal container.

Auto sampler vials are collected in buckets and stored under the hood in the organic extraction laboratory. **Periodically the vials are consolidated in lab packs for disposal by a licensed waste disposal company**.

f. Bioassay Laboratory

- Aqueous sample waste and a small amount of methanol are poured down the drain with copious amounts of tap water. Larger amounts of methanol used for glassware cleaning are collected in beakers and evaporated at room temperature.

- Hazardous or product samples are returned to the client.

D. Consolidation of satellite waste for contractor disposal:

In conjunction with the Safety Officer, the sample receiving supervisor is responsible to coordinate waste disposal operations with outside waste disposal contractors.

1. Solvent waste from the areas discussed above is periodically consolidated into two drums located outside the back of the building inside the caged fence (c. *Organic Extraction Laboratory Area*, * **Note**). A drum designated either chlorinated or non-chlorinated solvent waste is available to receive the appropriate solvent waste. When the drums become full (fluid surface six inches below the top of the drum), an authorized hazardous waste contractor will be scheduled to remove them to proper waste disposal.

2. The Acid Satellite Waste drum is also disposed through the authorized hazardous waste contractor once the drum is full to the level of six inches below the top of the drum.

3. Consolidated autosampler and standard vials are periodically Lab-Packed in drums and disposed through the authorized hazardous waste contractor.

4. The Laboratory Health and Safety Officer will administer the Waste Disposal Program and maintain current information to track quantities of waste generated and stored on-site.

It is the continuous objective of our laboratory to find ways to decrease the amount of waste generated.

DISTRIBUTION/TRAINING LOG

ANALYTICAL LABORATORY WASTE DISPOSAL

SOP NUMBER: **SOP-405**

REVISION NUMBER: **5**

RECEIVED BY/DATE:

W. Schwab	WS
R. Townsend	Russell Townsend RET
F. Rivers	FR
J. Holliman	J.H.
B. DeVille	Betty DeVille
A. Monteiro	
B. Richard	

Signature above signifies acknowledgement of responsibility to know and follow the contents of this document. It also signifies receipt of training covering all new aspects of the SOP.

TRAINED BY: Randy H. Wood

EFFECTIVE DATE: **06/23/09**

PLEASE COLLECT OLD SOPs AND RETURN WITH SIGNED FORM TO QAO

STANDARD OPERATING PROCEDURE (SOP) FOR LABORATORY SAMPLE STORAGE, SECURE AREAS AND SAMPLE CUSTODY

SOP NUMBER: **SOP-410**

REVISION NUMBER: **7**

APPROVED BY:

SECTION MANAGER

TECHNICAL DIRECTOR

EFFECTIVE DATE: **06/23/09**

DATE OF LAST REVIEW: **06/23/09**

STANDARD OPERATING PROCEDURE (SOP) FOR LABORATORY SAMPLE STORAGE, SECURE AREAS AND SAMPLE CUSTODY

Empirical Laboratories, LLC is located at 621 Mainstream Dr. suite 270 Nashville, TN 37228 on the first floor. This building is locked and monitored by an alarm system after normal business hours. No unauthorized personnel are permitted within the facility without a proper escort and a visitor's badge. During non business hours, all doors to the building are locked and secured by an alarm system. All front and back doors are locked and only Empirical Laboratories, LLC personnel have a key to access the building. Upon unlocking the door and entering into the laboratory, then the employee is to deactivate the alarm system using the assigned 4 digit alarm code assigned to them by Human Resources. Each employee is assigned their own designated alarm code, with no code being assigned twice. There is a buzzer at the door to Login to allow entry for sample and supply deliveries.

The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Shipping/Sample Receiving (SR) area in back of the building. The laboratory is located close to Federal Express (FedEx) distribution station; therefore we pick up our coolers at the FedEx location daily and transport them directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client. The SR personnel must not leave any packages/cooler without authorized receipt from laboratory personnel. Samples must be accompanied by some type of chain of custody record. Sample receiving personnel sign, and list the date and time received on the chain of custody. The time received must reflect the actual time or validation date and time of receipt for the samples although they may be placed in cold storage and logged into the system at a later time. The method of delivery is listed on the cooler receipt form(CRF). The tracking # (if available) is attached to the chain of custody.

Once sample containers have been assigned a laboratory ID number, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A Cooler Receipt Form also must be completed to accompany the cohesive Chain of Custody. Samples should not leave the log-in area until this has been completed. Log-in is also responsible for maintaining a Sample Receiving Custody and Disposal Form for samples received. This form is to be filled out before the actual disposing of any

sample in house. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:

- Client and Log #s
- Date/Time Unpacked
- Logged In/Numbered By (Initials)
- 2nd Checked By (Initials)
- Date/Time Placed in Cold Storage
- Storage Area (Walk In, Blue Air-VOCs, Quarantined Soils, Quarantined-VOC, Other)
- Disposed of By/Date
- Method of Disposal

Original samples are stored in following areas of the laboratory.

1. Hobart Refrigerator in the VOC lab: All water VOCs must be stored in this refrigerator.

2. Water Walk In Refrigerator: All waters for all analyses except VOCs must be stored in this refrigerator.

3. Soil Walk in Refrigerator for all soils.

4. VOC Dry Storage Rack: All water VOCs that have exceeded double holding time can be stored on this rack. These samples are stored here segregated alone to ensure no cross contamination occurs between VOC samples and other non-VOC aqueous samples.

All soils are treated as quarantined.

All samples must be stored in one of the four locations detailed above with the following exceptions:

1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)

2. Highly contaminated waste or product type samples which could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Testing Coordinator for other options.

Any person removing samples from the storage areas listed above, must sign them out on a laboratory custody sheet (attached). The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the samples are returned and signed back into the appropriate storage area. It must be noted if the entire sample volume was used and that the container was discarded.

Sample extracts and digestates are stored in the following areas:

1. All metals digestates are stored in the metals instrument laboratory. The transfer from the digestion analysts to the ICAP analysts is documented in the metals digestion log book.

2. Non - ZHE TCLP extracts are returned to the refrigerator in which the original samples are stored. For ZHE samples, the extract is returned to the refrigerator in which the original VOC sample containers are stored.

3. Extracts from medium level VOC analyses are also stored in the Soil Walk – in or VOC sample freezer in the VOC Lab.

4. All Organic extracts are stored in a Beverage Air side by side refrigerator in the organic extraction laboratory.

The generation of all sample extracts/digests and their movement through the laboratory will also be tracked on a laboratory custody sheet or in a log book. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the extracts are returned and signed back into the appropriate storage area. The metals digestates are not removed from the metals instrument laboratory.

After the analytical results have been reported, the original samples, sample extracts, and digestates will remain in secure storage until they are disposed of in accordance with the Waste Disposal Standard Operating Procedure. Samples will be held for a minimum of 30 days after the final report unless specified otherwise. Sample extracts and digestates are held for a minimum of 60 days after the final report unless project specific requirements state otherwise. See SOP No. 405 entitled Laboratory Waste Disposal SOP for guidance on disposal of samples.

The following personnel as of June 23rd, 2009 have access to all sample storage areas:

James Dalton	Herbie Johnson
Ashley Bester	Dahae Kim
Roger Burr	Dustin Lynch
Janice Shilling	Marcia McGinnity
Rick Davis	Badeen Mekael
Jessica Sales	Antonio Montiero
Betty DeVille	Kelienne Verdier
Amanda Fei	Gino Moore
Kendra Gentry	Lorraine Norohna
Jason Goodman	Melanie Sams
Sonya Gordon	Brian Richard
Gwen Hallquist	Franklin Rivers
Veronica Mullen	William Schwab
William Lancaster	Russell Townsend
Jade Holliman	Christy Thompson
John Hughes	Renee Vogel
Karu Huka	Randy Ward
Randy Ward	Marcia McGinnity

In the event that an employee is terminated, the supervisor is responsible for collecting the employee's keys.

For additional information see SOP No. 404 entitled Laboratory Sample Receiving, Log-In and Storage.

DISTRIBUTION/TRAINING LOG

STANDARD OPERATING PROCEDURE (SOP) FOR LABORATORY SAMPLE STORAGE, SECURE AREAS AND SAMPLE CUSTODY

SOP NUMBER: **SOP-410**

REVISION NUMBER: 7

RECEIVED BY/DATE:

W. Schwab WS

F. Rivers FR

R. Townsend Russell Townsend RET

Signature above signifies acknowledgement of responsibility to know and follow the contents of this document. It also signifies receipt of training covering all new aspects of the SOP.

TRAINED BY: Randy H. Ward

EFFECTIVE DATE: 06/23/09

PLEASE COLLECT OLD SOPs AND RETURN WITH SIGNED FORM TO QAO