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QUALITY ASSURANCE PROJECT PLAN FOR DEMOLITION OF MINE FILL BUILDINGS NSA CRANE IN

03/11/2010

LAKESHORE ENGINEERING SERVICES INC /TOLTEST INC

QUALITY ASSURANCE PROJECT PLAN
FOR
Demolition of Mine Fill Buildings

Naval Support Activity
300 Highway 361
Crane, Indiana 47522

Submitted to:

Naval Facilities Engineering Command Midwest
Officer in Charge of Construction
Naval Support Activity
300 Highway 361
Crane, Indiana 47522

LES/TolTest JV Project No. 20979.01

EMAC Contract No. N40083-05-D-4015

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March 11, 2010

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LES/TolTest JV hereby certifies that, to the best of its knowledge and belief, the technical data delivered herewith under this contract is complete, accurate, and complies with all requirements of the contract.

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1 PROJECT DESCRIPTION

1.1 INTRODUCTION

LES/TolTest JV has prepared this Quality Assurance Project Plan (QAPP) in accordance with the Indiana Department of Environmental Management's (IDEM) *Risk Integrated System of Closure: Technical Resource Guidance Document,* September 2009. The intent of the QAPP is to provide a complete and detailed description of the location, collection method, type, and number of samples required for field investigation. It also provides a detailed description of all activities, Quality Assurance/Quality Control (QA/QC) procedures, and precautions associated with sample collection and analysis.

1.1.1 Project Background/Site Description

NSA Crane is located in the southern portion of Indiana, approximately 75 miles southwest of Indianapolis and 71 miles northwest of Louisville, Kentucky, immediately east of Crane Village and Burns City (**Figure 1**). NSA Crane encompasses 62,463 acres (approximately 98 square miles), most of which are located in the northern portion of Martin County. Smaller portions of NSA Crane are located in Greene, Daviess, and Lawrence Counties. NSA Crane is located in a rural, sparsely populated area. Most of NSA Crane is forested, and the surrounding area is wooded or farmed land. NSA Crane provides material, technical, and logistical support to the Navy for equipment, shipboard weapons systems, and nonexpendable ordnance items. In addition, NSA Crane supports the Crane Army Ammunition Activity with production, renovation, storage, shipment, demilitarization, and disposal of conventional ammunition.

The Mine Fill area is located in the central portion of NSA Crane as shown on **Figure 1**. Mine Fill A is bordered by Highway 17 and encompasses approximately 40 acres. Mine Fill B is bordered by Highway 18 and is also approximately 40 acres. The Mine Fill A site Latitude and Longitude is approximately 38° 51' 03" N and 86° 49' 25" W respectively. The Mine Fill B site Latitude and Longitude is approximately 38° 50' 33" N and 86° 50' 10" W respectively.

Specifically, Mine Fill A and B were both constructed to support war efforts in 1941. They were designed for producing items requiring the casting of TNT based explosive materials and had melt/pour facilities. Items such as underwater mines, torpedo warheads, anti-submarine projector charges and depth charges were manufactured in the two plants. During the Vietnam conflict, both lines were used for producing low drag bombs ranging from 250 pound (MK 81) design up to the 2000 pound (MK 84) design filled with TNT. Mine Fill B was converted in 1968 to permit the loading of Minol (ammonium nitrate and TNT) explosive into the bombs. Mine Fill B was deactivated completely in the early 1970's in that no cast loading was accomplished beyond that time. The buildings making up the "back side" or starting point for the casting process were used to house ammunition inspection functions that did not deal with exposed explosives. Mine Fill A, North end was also shut down during the early 1970's. One back side building was used to store hazardous materials for a short time during the 1980's.

Explosives (MC) at Mine Fill A and B will include TNT and RDX, which were used as filler for all munitions items loaded during World War II and the Korean War. During the Vietnam War the facilities continued to load TNT, but added Ammonium Nitrate to the mix producing Minol filled bombs. Both lines were shut down after Vietnam and only used for storage which included some hazardous materials. The South end of Mine Fill B was renovated in the mid 1980's and used for casting large "shock charges" which contained HBX (TNT, RDX, & powdered aluminum) and remained in production for some time into the 1990's.

The major sources of contamination at MFB were the wash-down of explosive formulations from building roofs onto the ground surface and exhausts vented from ventilation systems. Also, the therminol boilers located near Buildings 166 and 171 leaked oils containing PCBs. The boilers and

surrounding contaminated soil were removed in 1989, but subsequent soil samples collected near the former locations of the boilers indicated that PCB concentrations were greater than 10 mg/kg in residual soils.

The Mine Fill area was also remediated in 1999, 2000, and 2001 as part of a bioremediation program that included sampling, excavation, on-site treatment through bioremediation (composting) of explosives-contaminated soil, and backfilling of the compost. Beginning in 2008, additional remediation activities were performed in the Mine Fill B area at Solid Waste Management Unit (SWMU) 13. The SWMU 13 project included surface, sub-surface and drainage system remediation of PCB impacted soils and sediments. Approximately 965 tons of TSCA (>50ppm PCB) and 3,460 tons of non-TSCA (<50ppm PCB) materials were eventually removed from the site. In January 2010, closeout sampling activities identified an additional 110 tons of PCB impacted material remaining near Building 171 which has been scheduled for removal in early spring.

The types of analytes measured before and after the various remediation efforts included; volatile organic compounds (VOCs), explosives, metals, and PCBs (Aroclor 1242, 1254, and 1260).

Most recently, work included the demolition of above-ground structures on site. Remaining work includes the removal and disposal of the foundations and drains.

1.1.2 Constituents of Concern

The constituents of concern (COC) for the Mine Fill area are listed below. They are derived from the RFI Phase I Environmental Monitoring Report for Mine Fill B [Halliburton NUS, 1992].

Explosives: PETN; TNT; RDX; HMX; Tetryl; TNB; 1,3-DNB; NB; 4-Am-DNT; 2-Am-DNT; 2,4-DNT; 2,6-DNT; 2-NT; 3-NT; and 4-NT.

PCBs: Aroclor 1242; Aroclor 1254; and Aroclor 1260.

According to the *Interim Measures Report – Removal and Bioremediation of Contaminated Soil from Mine Fill B,* July 2002, all explosive contaminated soil identified during the study had been excavated in order to meet at least industrial cleanup goals. Table 3-3 of the *Interim Measures Report* identifies the areas in which soil was not remediated to below industrial levels due to physical limitations. Soils underneath building foundations and conveyor housings were not sampled.

Explosives are currently the main COC for this project. PCBs are a secondary COC and may be found only under the foundations of buildings 166 and 171.

1.1.3 Scope of Work and Overall Objectives

The following tasks associated with the Mine Fill demolitions will be accomplished under the provisions of the Work Plan and Site Safety and Health Plan (SSHP):

- Removal of concrete tunnel walls, blast walls, and retaining walls associated with the buildings scheduled for demolition.
- Removal of soil berms associated with the items above. This includes sampling, surveying, and capping of industrial contaminated soils with one foot of clay. Residential soils and soils between residential and industrial will be used as backfill material.
- Sampling the soil beneath the foundations of the buildings to be demolished and areas underneath where the conveyor housings were.
- Capping above-industrial soils beneath the building foundations with one foot of suitably impermeable soil material.

1.2 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) establish the type, quality, and quantity of data required to make and defend a particular decision. They are developed using a seven-step process: state the problem, identify the decision, identify inputs to the decision, define the boundaries, develop a decision rule, specify limits on decision errors, and optimize the design for obtaining data.

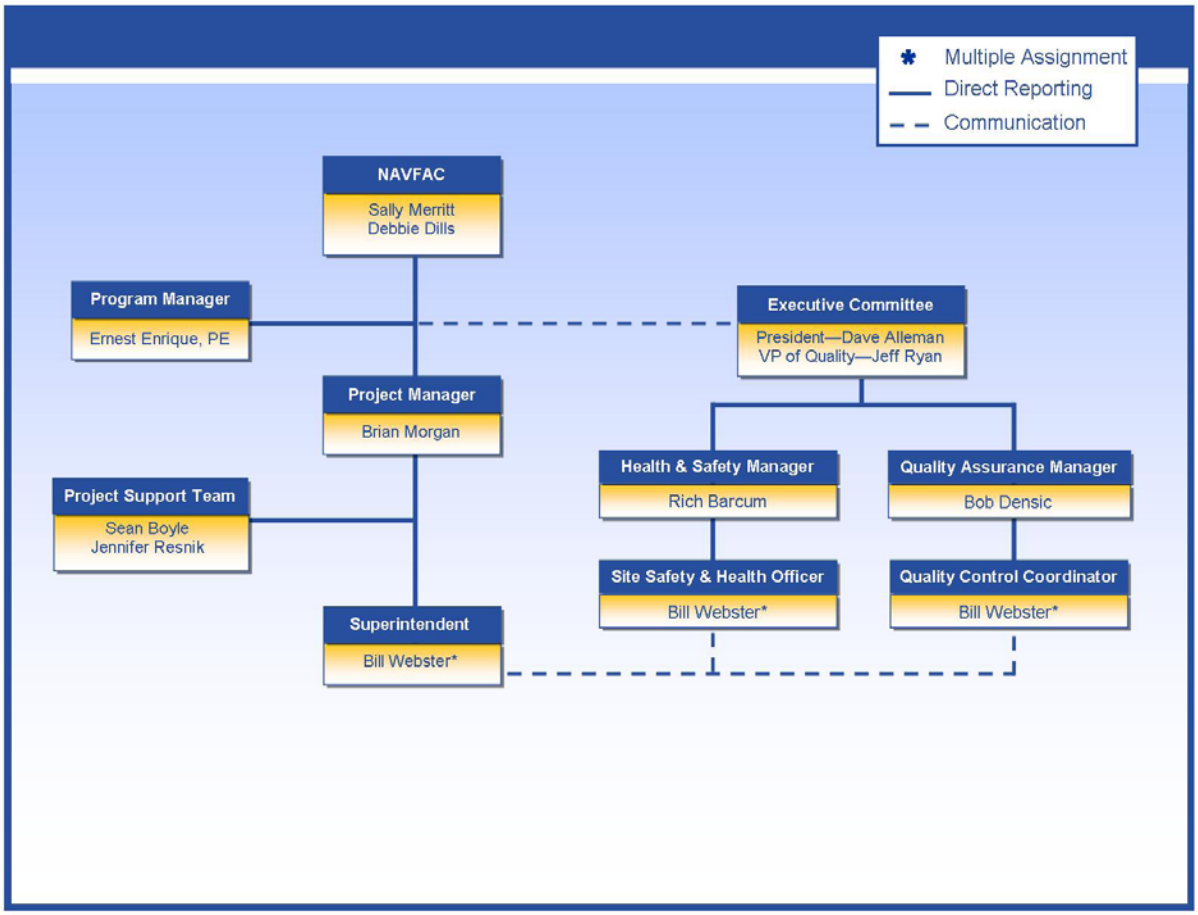
- **State the problem:** Soil beneath the building foundations, drains, and conveyor housings are potentially contaminated with explosives (and PCBs for two buildings). These areas must be sampled after demolition of the foundations to verify which areas are contaminated.
- **Identify the decision:** Soils that are above industrial closure level critera for explosives or PCBs will be capped with a clay soil cap. Soils that are below criteria can be used for backfill.
- **Inputs to the decision**: Soil samples will be collected and compared to Residential and Industrial closure levels for explosives and PCBs—which can be seen in **Table 1-1**. If explosives in soil are at or above 34,000 ppm for HMX, 17 ppm for RDX, and 64 ppm for TNT, they will have to be capped with a clay soil cap. If PCBs in soil are at or above 1 ppm, the soil will have to be capped.
- **Define the boundaries:** It is expected that soil will be contaminated with explosives under the building foundations and under the conveyor housings. It is also expected that all drains associated with building foundations will be contaminated with explosives. PCBs are likely to be found under the foundations of buildings 166 and 171. Decisions on contamination will be made on a building by building basis.
- **Decision rule:** If contaminant levels exceed industrial limits, the associated soil will be capped with one foot of impermeable soil material. Soils under industrial limits will be used as backfill material.
- **Limits on decision errors:** The main source of possible error will come from human sampling error. Quality control samples will be taken and analyzed to check the accuracy and precision of sampling and analyzing techniques. These will include matrix spike/matrix spike duplicate samples, field duplicate samples, equipment blank samples, and trip blank samples. Quality assurance objectives are provided in detail in section 3.
- **Optimize the design:** Soil sampling will be conducted systematically at each sampling location. Each location will have a location-specific grid that will be used to collect one composite sample from approximately every 225 square feet. Sampling procedures are provided in detail in section 4. Therefore, decisions will be able to be made for every 225 sq. ft. section sampled.

2 PROJECT ORGANIZATION AND RESPONSIBILITY

2.1 PROJECT ORGANIZATION CHART

Exhibit 2-1 shows the project organization chart indicating individual assignments for the demolition activities at MFB, NSA Crane. All participants are directly subject to the requirements of this QAPP. The responsibilities of individual positions are briefly described in the following sections.

Exhibit 2-1: Project Organization Chart



2.2 MANAGEMENT/QUALITY ASSURANCE RESPONSIBILITIES

Each of the key personnel presented above has responsibilities and authorities as it relates to their position, to ensure quality and meet contract requirements for project scope, cost, schedule, and communication. Key personnel responsibilities and authorities are provided in *Exhibit 2-2*.

Exhibit 2-2: Key Personnel Responsibilities

Responsibilities	Authorities
Program Manager	
▪ Ensures the physical execution and control of processes are adequate to provide the client with the product, which was contracted ▪ Ensures each project proceeds in accordance with the policies and procedures outlined in the QMS ▪ Oversees and manages overall contract program	▪ Has hiring/firing authority ▪ Has signature authority for the full value of the contract, including change orders ▪ Commits resources at the program level ▪ Has stop work authority for any reason on the project
Project Manager (PM)	
▪ Ensures successful execution of the project and adequate time and resources are available for the proper operation of the quality function ▪ Ensures elements of plans and specifications are satisfied within planned scope, cost, and schedule ▪ Identifies the Definable Features of Work (DFOW), testing, sampling, and includes them on the project schedule ▪ Ensures compliance with Safety and QC requirements, applicable regulations, and codes ▪ Performs submittal and inspection reviews ***Reports to Program Manager***	▪ Supervises and evaluates project staff ▪ Approves deliverables, TO work plans, subcontractor selection, and preparation of TO budgets and schedules ▪ Has stop work authority for unsafe work environment or quality issues ▪ Discuss and prepare contract modifications
Superintendent	
▪ Plans and documents daily/weekly look ahead work schedules and production activities/goals ▪ Ensures execution of site activities are in accordance with approved SOW, work plans, and applicable laws and regulations ▪ Maintains and updates the Project Field Manual ▪ Coordinates site work, including subcontractor activities ▪ Reports immediately to the PM any variance in production rates that would adversely affect cost or schedule ▪ Ensures day-to-day implementation and enforcement of approved safety plan ***Reports to Project Manager***	▪ Sequences work activities to meet project schedule ▪ Signs Daily Reports ▪ Has signature authority to receive, inventory, and store materials and equipment required for TO execution ▪ Has stop work authority for unsafe work environment or quality issues
Quality Assurance (QA) Manager	
▪ Ensures TolTest's QMS is implemented in the contract quality program ▪ Reviews and approves CQCP ▪ Ensures technically qualified and trained QC personnel are assigned to this TO ▪ Provides assistance to QCM ▪ Conducts on-site training if required ▪ Performs subcontractor and supplier evaluations, if necessary ***Reports to Vice President of Quality***	▪ Has stop work authority for unsafe work environment or quality issues

Responsibilities	Authorities
Quality Control Manager (QCM)	
▪ Verifies the project submittals are reviewed and approved as appropriate ▪ Inspects materials and equipment received on-site to assure compliance with contract requirements ▪ Coordinates the tracking and storing of materials on-site ▪ Conducts daily QC inspections of field activities ▪ Implements corrective action for QC deficiencies ▪ Performs and documents the three phase QC process on identified DFOWs ▪ Manages and controls three phase QC process performed by any QC Specialists, laboratory testing or inspection personnel ▪ Verifies QC testing procedures comply with contract requirements ▪ Attends weekly QC/Progress meetings ▪ Enforces compliance with the project CQCP ▪ Enforces correction of non-compliance work ***Reports to TolTest VP through the QA Manager***	▪ Has stop work authority for quality issues including nonconformance to CQCPs and procedures ▪ Signs QC Report
Site Safety & Health Officer (SSHO)	
▪ Conducts safety tailgate briefings ▪ Observes site personnel for symptoms of exposure ▪ Arranges for on-site emergency/first aid care ▪ Directs emergency response program and maintains work zone delineation within the project site ▪ Enforces compliance with the project APP ▪ Assists in conducting daily safety inspections ***Reports to TolTest Health & Safety Director***	▪ Has stop work authority for unsafe work environment, unacceptable safety conditions

2.3 LABORATORY QUALIFICATION REQUIREMENTS

2.3.1 Analytical Chemistry Laboratories

LES/TolTest JV will be responsible for the selection of qualified laboratories capable of performing quality analysis and data. Because the quality of analytical chemistry data must be defensible, only laboratories meeting certain minimum qualification requirements will be utilized. LES/TolTest JV will utilize analytical laboratories that have undergone the laboratory approval process as defined by the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).

The QAPP provides the detailed requirements for analytical sampling, from collection in the field to analysis in the laboratory. Additionally, specific requirements for data deliverables are included in this QAPP.

2.3.2 Test Results

Laboratory test results will be signed by a testing laboratory, project manager or designee authorized to sign certified test results. The laboratory will furnish all test results to the Project Chemist or designee. All test results will contain the following, as a minimum:

- A reference to applicable contract requirements, tests, or analytical procedures used;
- Actual test results; and
- A statement that the item tested or analyzed conforms or fails to conform to specified QC requirements.

3 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results which are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described or referenced in other sections of this QAPP. General quality control limits for accuracy, precision and completeness are provided in **Tables 3-1** and **3-2.**

3.1 PRECISION

3.1.1 Definition

Precision is a measure of the reproducibility of measurements under a given set of conditions. Precision is usually stated in terms of relative standard deviation (RSD) or relative percent difference (RPD). Results of QC samples can be used to calculate the precision of the analytical or sampling process. Field duplicate and replicate samples will be used to determine the precision of the sampling process. Laboratory duplicates, matrix spikes and matrix spike duplicates will be used to determine the precision of the analytical process.

3.1.2 Field Precision Objectives

Field duplicate data will be evaluated to determine potential variability introduced by soil heterogeneity and sampling technique. This evaluation will be performed during data verification, as defined in Section 9.2.2, and summarized in the data assessment. One duplicate sample or reading will be analyzed for at least every 20 samples to qualitatively evaluate precision.

3.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of RPD and RSD for three or more replicate samples. The equations to be used for precision in this project can be found in Section 11. Precision control limits are established by SW-846 methods. Limits for confirmation soil sample precision are summarized in Tables 3-1 through 3-7.

3.2 ACCURACY

3.2.1 Definition

Accuracy is a measure of the bias in a measurement system, and defined as the closeness of the reported value to the true value. Potential sources of error include the sampling process, field contamination, preservation, handling, sample matrix, sample preparation and analytical techniques. The accuracy of a measurement system will be assessed by evaluating the results of quality control samples such as matrix spikes, analytical surrogates and the use of field blanks, trip blanks and equipment rinsate blanks.

3.2.2 Field Accuracy Objectives

For laboratory data evaluation, field blanks, trip blanks and equipment rinsate blank data will be evaluated to identify potential sources of error introduced during sampling. This evaluation will be performed during data validation and summarized in the data assessment. Accuracy for laboratory samples is also assessed through adherence to all sample handling, preservation and holding times. Accuracy for field measurements will be ensured by adhering to established Field SOPs (Appendix A).

3.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of standard matrix spikes, sample matrix spikes (MS) or standard reference materials (SRM) and the determination of percent recoveries. The equation

to be used for accuracy in this project can be found in Section 12. The accuracy control limits are also summarized in Tables 3-1 through 3-2, and may vary based on laboratory performance.

3.3 COMPLETENESS

3.3.1 Definition

Completeness is a measure of the amount of valid data obtained from an analytical data set compared to the amount that was expected to be obtained under normal sampling and analytical conditions.

3.3.2 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all measurements taken in the project. The equation for completeness is presented in Section 12. The laboratory completeness goal for Level C data is 90 percent, which is considered to be attainable using SW-846 and other EPA-approved analytical methods.

3.4 REPRESENTATIVENESS

3.4.1 Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is a qualitative parameter which is controlled by the proper design of the sampling program. Representativeness is addressed by describing sampling techniques and the rationale used to select sampling locations. It will be assessed through the use of samples collected in a manner such that they are equally representative of a given point in space or time. Representativeness will be satisfied by ensuring that this QAPP is followed and that proper sampling techniques and sample preservation procedures (**Table 4-1**) are followed as demonstrated in Section 4.0 of QAPP.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample holding times and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of site conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the RCRA program.

3.5 COMPARABILITY

3.5.1 Definition

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another.

3.5.2 Measures to Ensure Comparability of Field Data

The comparability goal is achieved by using established Field SOPs to collect and analyze representative samples and reporting analytical data in appropriate units. Comparability will be satisfied by ensuring that the QAPP procedures are followed and that proper sampling techniques are used.

3.5.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented per the QAPP. Comparability is also dependent on similar QA objectives.

3.6 LEVEL OF QUALITY CONTROL EFFORT

Field blank, trip blank, method blank, sample duplicate, standard reference materials (SRM), standards matrix spike and sample matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

Field and trip blanks consisting of distilled/deionized water will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Sampling equipment rinsates are collected to ensure sampling equipment is clean and that the potential for cross-contamination has been minimized.

One Field Blank sample will be collected from each source of tap water every month used for equipment decontamination.

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the extraction or digestion and measurement methodology. All matrix spikes are performed in duplicate for organic methods and are hereinafter referred to as matrix spike/ matrix spike duplicate (MS/MSD) samples. One MS/MSD will be collected for every 20 or fewer investigative samples. MS/MSD samples are designated/collected for all analytical methods.

Field duplicates will be collected at a frequency of one sample per 20 investigative samples collected.

4 SAMPLING AND ANALYSIS PLAN

This section describes sampling locations, methods, and rationales for the sampling activities to be conducted in support of the Mine Fill demolition activities.

The equipment and procedures to be used for collecting, handling, preserving, recording, logging, and shipping samples to an analytical laboratory are presented in this section. Soil sampling will generally be performed using trowels and buckets which will be decontaminated after each sample collection and re-used.

Prior to any field activities, the LES/TolTest JV PM will ensure that all field personnel read and understand this QAPP and associated HASP. Each worker or visitor to the site must sign an acknowledgment that he or she has read and understands the HASPs, as applicable.

The FOLs will ensure that all required field equipment for non-health-and-safety-related operations is available and operational, and the SSHOs will ensure that all health-and-safety-related equipment is available and operational.

4.1 SAMPLE PROCEDURES

Since there is already detailed information on COC levels for most of the soil throughout Mine Fill areas, a mixture of systematic and judgmental sampling techniques will be used to sample areas that have not been sampled previously. These areas consist of underneath each building foundation, and the soil that previously had the conveyor lines above them. Composite samples will be collected systemmatically with the use grids, and additional grab samples will be collected from areas that show sign of contamination. Specific areas that will deserve special attention will include sump/drain areas and settling pits. COC's for this sampling event are explosives and PCBs. Explosives are expected throughout, while PCB's are expected near buildings 166 and 171 only.

Survey coordinates will be recorded for each sample location.

4.1.1 Sampling Underneath Foundations

Once the remaining foundations have been removed at each location, surface soil samples will be collected for verification of contamination/no contamination. At each location, sample locations will be designated by measuring out a grid made up of 5 ft x 5 ft subsections. Composite samples will be collected by combining grab-sample increments from no more than nine of these 5 ft x 5 ft subsections—one composite sample will be collected for approximately every 225 square feet per building footprint. Each increment sample will be collected from the ground surface to 6 in below ground surface (bgs). A general example of the grid layout can be seen in **Figure 4** (the grid will be modified for each building according to its dimensions).

Samples from sumps/drains and settling pits will be collected by using a push probe or hand auger to collect samples up to approximately 4 ft bgs. One sample will be collected per sump and settling pit from the depth of the vertical profile that exhibits the most evidence of contamination. If there is no evidence of contamination, the vertical profile will be composited into one sample.

Samples will be analyzed for explosives (USEPA SW-846 method 8330) for all building locations (157, 158, 159, 2715, 3036, 161, 166, 167, 168, 171, 172, 173, 2171, 2179, 2500, 2501, 2502, 2503, 2512, 2514, 2515*)*, and analyzed for explosives and PCB's (USEPA SW-846 method 8082A) at buildings 166 and 171. Buildings listed with their respective estimated number of samples can be seen in **Table 4-2**. All building locations can be seen in **Figure 2** and **Figure 3.**

The increment grab samples will be composited for the required analyses (**Table 4-1**). The grabs will be combined to form one composite sample as follows:

- Place each grab sample into a stainless steel bowl and remove vegetation and large rocks. Homogenize samples by breaking up the soil using a pestle or spoon. Thoroughly mix and homogenize the soil in the bowl. Cone and quarter the soil in the bowl. Collect an equal portion from each quarter and composite to fill an 8-ounce jar for explosives analysis.
- This process will provide a more representative sample as explosives contamination is known to vary over short distances (Crockett and others, 1996).

4.1.1.1 Stormwater Protection Measures

After soil samples have been collected, all open excavations will be covered with reinforced polyethylene sheeting until verification soil sample results have been received. Soil samples will also be collected as soon as possible after a given foundation has been removed to minimize the amount of time excavations are left open.

4.1.2 Sampling Conveyor Lines Soil

Ten composite samples will be collected from each conveyor line (1 conveyor line in MFA and 4 in MFB) from 10 sections approximately 40 ft in length (approx. 250 sq. ft each). Within each 40 ft composite section, composite samples will be made up of increment samples from approximately every 5 feet (8 increment samples per composite section) and collected from 0-6 in bgs. Incremental samples, if applicable, will be taken from areas that visibly show evidence of possible contamination. A general conveyor line sampling diagram can be seen in **Figure 5.** Sample will be composited as described in section 4.1.1 and conveyor line soils will be analyzed for explosives (USEPA SW-846 method 8330).

4.2 QC SAMPLES

QC samples will be collected to ensure that representative and reproducible data are obtained. The types of QC samples that will be used are discussed below. QC samples apply only to samples submitted for off-site analysis by a laboratory.

4.2.1 Equipment Rinsate Blanks

Equipment rinsate blanks (or rinsate blanks) are collected to ensure that sampling equipment is clean and that the potential for cross-contamination has been minimized or prevented. Rinsate blanks are collected from the final reagent grade water rinse used in sample equipment decontamination. Rinsate blanks will be collected and analyzed at a frequency of one per 20 rinses, or at a frequency listed with the sampling procedures, whichever is more stringent. A reduction in the frequency of rinsate blanks may be deemed justifiable by the Project Chemist based on review of analytical results which would need to show no discernable evidence of introduction of cross-contamination due to inadequate equipment cleansing with respect to analysis type.

The rinsate blank is obtained by first decontaminating the sampling device and then pouring reagent grade water over the device. This water is provided by an analytical laboratory. The rinsate blank water is collected directly into the sample bottles. The rinsate blanks are analyzed for the same constituents as the related samples. The results will be used to flag or assess the levels of analyses in the samples and evaluate the effectiveness of sampling equipment decontamination.

4.2.2 Field Duplicate Samples

Field duplicate samples will be collected to allow determination of analytical and sampling precision. One duplicate sample per 20 samples will be collected and submitted for the same analysis as the regular samples. However, one duplicate per 10 samples for explosives will be collected and submitted for analysis. The duplicate sample will be collected simultaneously with the true sample. The sample will be

collected with the same sampling equipment at the same location as the regular sample, but it will be labeled with the next consecutive sample number.

4.2.3 Matrix Spike Samples

Matrix spike/matrix spike duplicates (MS/MSD) samples will be used as further QC checks. These samples will be spiked at the laboratory. The samples will be prepared at a minimum frequency of one MS and MSD for every 20 regular field samples. The samples will allow accuracy to be determined by the recovery rates of compounds (the matrix spike and/or surrogate spike compounds defined in the analytical methods). Precision will also be assessed by comparison of matrix spike duplicate recoveries. The purpose of these laboratory spikes is to monitor any possible matrix effects specific to samples collected from the site. The addition of known concentrations of compounds/ constituents to the sample also monitors extraction/digestion efficiency.

MS/MSD samples will be collected to provide sufficient volume per QC and analytical laboratory requirements. The laboratory will select aliquots that are as homogeneous as possible with respect to one another to avoid precision problems related to sample homogeneity.

4.3 DECONTAMINATION PROCEDURES

4.3.1 Sample Bottles

Sample bottles will be provided by the analytical laboratory. These bottles will arrive at the site in sealed boxes or coolers. Certification of cleanliness will accompany each box of bottles. The exception to this would be if any bottles were pre-preserved by the lab.

If certification is not included or cleanliness of the containers is suspect, U.S. EPA approved cleaning procedures will be carefully conducted by the analytical laboratory. The procedures are as follows:

- *Explosives:* Non-phosphate detergent wash, tap water rinse, deionized water rinse, methanol rinse, final reagent grade water rinse.

4.3.2 Sample Equipment Decontamination

All non-disposable sampling equipment, including stainless steel trowels and spoons, will be decontaminated according to the following procedure and sequence, depending on the contaminants involved:

1) Manual scrub with Alconox and tap water wash
2) Rinse with deionized water
3) Air dry.

Once dry, sampling equipment will be stored in plastic bags to prevent contamination.

4.4 SAMPLE PACKAGING AND SHIPPING

Immediately after collection, samples will be properly labeled, tagged and properly preserved. **Table 4-1** lists the proper container, volume requirements, and preservation needed for the sampling effort. Samples requiring refrigeration for preservation will be immediately placed in coolers packed with ice or ice packs. Proper chain-of-custody documentation will be maintained as discussed in Section 5.0 - Custody Procedures.

Packaging and shipping procedures will vary depending upon sample media, potential contaminant concentration, preservation technique, and sample container. A waterproof ice chest or cooler is suitable for packaging and shipping samples not considered "Dangerous Goods" by the U.S. Department of Transportation (USDOT). The person packaging the samples is responsible for ensuring that the cooler is in suitable condition for shipping. In the event a concern arises regarding

whether a sample shipment is a USDOT "Dangerous Good" or is potentially in violation of international transportation regulations, the Project Manager will be consulted. Sample packaging should ensure that the chance of breakage is minimized and that, in the event a sample container breaks or leaks, it will not impact the integrity of other samples in the shipment. Also, samples should be protected from ice melt. Each sample container should be wrapped in packing material such as bubble wrap or comparable shock-absorbing materials when shipping glass containers to avoid breakage.

Adequate ice, contained in double Ziploc™-type bags or equivalent, must be included with each cooler shipment so that the contents are maintained at 4 $\pm$ 2 degrees Celsius until receipt at the laboratory. A temperature blank will be shipped with each cooler to verify the temperature.

In addition to an adhesive label, sample tags will be tied around the jars, as possible. The tags will include the same information recorded on the label.

A chain-of-custody record, protected from moisture by enclosure in a Ziploc™-type bag, must accompany each cooler shipped. The cooler lid should be secured using packing tape around the outside of the cooler. Custody seals will be signed, dated and affixed to the front, side and back of the cooler. The seals should be placed in such a way that tampering with the cooler lid will result in a ripped seal.

5 CUSTODY PROCEDURES

Custody is one of several factors which is necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under a person's custody if:

- The item is in actual possession of a person; or
- The item is in the view of the person after being in actual possession of the person; or
- The item was in actual physical possession but is locked up to prevent tampering; or
- The item is in a designated and identified secure area.

5.1 FIELD CUSTODY PROCEDURES

Logbooks will provide the means of recording data collecting activities. As such, entries will be described in as much detail as possible so that persons going to the facility could reconstruct a particular situation without reliance on memory.

Logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by the type of activity (i.e., operations, safety, and environmental).

The title page of each logbook will contain the following:

- Person or persons to whom the logbook is assigned,
- Logbook number,
- Project name,
- Subject (Sampling, Waste Management, etc.)
- Site/SWMU name,
- Project start date, and
- End date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered.

Measurements made and samples collected will be recorded. All entries will be made in indelible ink, signed, and dated and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark which is signed and dated by the sampler. Whenever a sample is collected or a measurement is made, a detailed description of the sampling location shall be recorded. All sample locations will be surveyed, with respect to permanent features. The number of the photographs taken, if any, will also be noted. All equipment used to make measurements will be identified, along with the date and results of calibration.

Samples will be collected following the sampling procedures documented in Section 4.0. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description. A detailed description of assigning sample identification numbers is provided in Section 5.1.2.

The sample packaging and shipment procedures summarized in Section 5.1.4 will ensure that the samples will arrive at the laboratory with the chain of custody intact. Chain-of-custody procedures are summarized in Section 5.1.3.

5.1.1 Field Documentation

Logbooks and forms will be used to document all field activities. Information to be entered in the field logbook includes at a minimum:

Operations Logbook:

- Individuals on site
- Operational activities/impacts
- Production information
- Visitors

Safety Logbook:

- Weather conditions
- PPE
- Injuries/incidents/accidents
- Results of air monitoring and personal protective equipment levels

Environmental Logbook:

- Sample identification
- Date and time of sample collection
- Requested analyses, type and number of sample containers, and preservatives
- Results of field screening measurements
- Sample locations and depth (include sketches as necessary)
- Type of sample collected (pond water, decontamination water, soil, grab, etc.)
- Details of QA/QC samples

Notes will be written on sequentially numbered pages with indelible ink. Each page will be initialed and dated when full. Corrections to logbook entries will be made by lining through incorrect entries with a single line and initialing and dating the strike out. At the end of the day, any unused space at the bottom of the last page will be "x-ed" out or lined out, initialed and dated.

5.1.2 Sample Identification

Samples will be labeled, preserved, and properly packaged for shipment to the analytical laboratory. Information on the sample label will include, at a minimum:

- Sample identification number
- Site Name
- Analytical method and type of preservative
- Date and time of sampling

Sample identification numbers will be used to provide a tracking procedure allowing information on a particular sample location to be easily and accurately retrieved. A sample will be labeled with its building number followed by a letter to represent the section that the sample is a composite of. For example: building 171 sample A will be labeled "171-A." This system also ensures that each sample is unique and not confused with any other sample. The Project Manager will maintain a complete list of sample numbers.

All information related to the sample matrix, location, and depth shall be recorded in the logbook by the sampling team. The Field Engineer/Sample Technician is responsible for maintaining a map showing the locations of each sample and keeping a list of which samples are submitted to the laboratory for QC purposes.

The sample number is entered on sample labels and chain-of-custody forms. All sample identification information will be documented in the sampler's logbook.

Samples will be placed in shipping containers for shipment to the laboratory. Custody seals will be affixed to the shipping containers. Information on the custody seal will include the date when the container was sealed and the signature of the sampler or relinquisher.

5.1.3 Chain-of-Custody Procedures

To maintain and document sample possession, chain-of-custody (COC) procedures must be implemented. A COC form will be used to record pertinent information for each sample and the individuals responsible for the sample collection, shipment and receipt.

The subcontracted laboratories will not accept samples collected for analysis without a completed COC form. The COC form shall be initiated in the field by the person collecting the sample, for each sample. Each COC form will be assigned a unique number based on the building number designation. Each sample will be assigned a unique identification number, as described in Section 5.1.2, and that number will be entered on the COC form. In order to be complete, project identification, date and time of sample collection, sample location, requested analyses, turnaround time, and any special instructions must be included on the COC, along with each sample identification. Custody will be relinquished by using the signature blocks at the bottom of the custody form. The original COC form will accompany the shipment, and a copy will be retained by the PM or designee. A signed COC form will be obtained from the laboratory custodian after the samples have been received and their condition checked.

Upon receipt in the laboratory, all samples will proceed through an orderly processing sequence (as defined in the laboratory QA/QC Plan) specifically designed to ensure continuous integrity of both the sample and other information pertinent to the analysis. All samples will be carefully checked and verified for proper COC records, preservation, presence of broken or leaking sample containers, proper label identification, and any associated discrepancies. If any samples arrive leaking or broken, or the custody seal on the shipment coolers is not intact, the Project Manager and Project Chemist will be notified of the problem(s) immediately. If no discrepancies are identified, the sample COC record will be signed, and the samples will subsequently be assigned a unique laboratory identification number by the laboratory for tracking and filing. The laboratory QA system and the use of an internal COC procedure will ensure that the samples are appropriately tracked from receipt through completion of the analytical process.

5.1.4 Handling and Shipping

All samples will be collected in the appropriate containers for each analysis, as detailed in Table 4-1. All sample labels used on sample containers will include, at a minimum, a sample identification number, the date and time of sample collection, site name, and the analytical method requested. The label will adhere to the container, and the writing on it will be in indelible ink. A waterproof metal or equivalent strength

plastic cooler will be used for packaging and shipping samples. Samples will be individually packed with bubble wrap or other cushioning material to prevent breakage during transport. Ice will be placed in the coolers with the samples to maintain shipping temperature at 4 ±2 degrees Celsius. Include a temperature blank with each cooler. The COC will be placed in plastic and affixed to the underside of the cooler lid. Custody seals will be affixed to the front and back of each cooler and covered with clear adhesive tape.

Samples packaging and shipping will vary depending upon sample media, contaminant concentration, preservation technique, and sample container. All samples will be packaged and shipped following all state and federal regulations and will conform with DOT requirements.

5.2 LABORATORY CUSTODY PROCEDURES

Laboratory custody procedures for sample receiving and log-in; sample storage and numbering; tracking during sample preparation and analysis; and storage of data are described in the Laboratory QA Plan. The QA Plan contains standard operating procedures which have been deemed proprietary by the Laboratory.

6 CALIBRATION AND FREQUENCY

This section describes the calibration procedures and the frequency at which these procedures will be performed for both field and laboratory instruments.

6.1 LABORATORY INSTRUMENT CALIBRATION

Laboratory instruments will be calibrated at prescribed intervals and/or as part of daily use. Frequency will be based upon the analytical method, type of equipment, inherent stability, and manufacturer's recommendations. Equipment will be calibrated, whenever possible, using reference standards having known relationships to nationally recognized standards or accepted values of physical constants. If national standards do not exist, the basis for calibration will be documented.

The analytical laboratory will be responsible for the maintenance of laboratory instruments and equipment. Instruments, and the measurements made as part of the analytical methodology, will be as specified in the method, without modification. The laboratory's quality assurance (QA) program ensures that only trained personnel perform routine maintenance on all major instruments and that repairs are performed by trained laboratory personnel or service technicians employed by the instrument manufacturer or representative. Instrument maintenance will be appropriately documented through the use of instrument logs which will be included in the laboratory project file.

7 ANALYTICAL PROCEDURES

This section describes the analytical procedures for all samples collected for analysis.

7.1 LABORATORY ANALYTICAL PROCEDURES

The laboratory will implement project required Standard Operating Procedures (SOPs). These laboratory SOPs for sample preparation, cleanup and analysis are based on the latest SW-846 Revision (U.S. EPA, 1995) and EPA methods for methods of chemical analysis of water and waste (U.S. EPA, 1983) .

The documentation of appropriate method validation for the project target compounds will be submitted in the Full-Scale Completion Report. It includes the criteria for acceptance, rejection or qualification of data.

7.1.1 List of Project Target Compounds and Laboratory Reporting Limits

A listing of project target compounds, cleanup levels, analytical methods, and analytical reporting limits to be used for samples collected during the project are summarized in Table 1-1. Other analytical results will be reported using standard method PQLs as reporting limits.

7.1.2 List of Associated QC Samples

The analytical methods and laboratory SOPs include a QC section which address the minimum QC requirements for the analysis of specific analyte groups. Compounds to be spiked will be representative of the target compounds of interest. A Summary of QC requirements can be found in Attachment 1.

8 INTERNAL QUALITY CONTROL CHECKS

8.1 FIELD QUALITY CONTROL CHECKS

Assessment of field sampling precision and bias will be made by collecting field duplicates and field blanks for laboratory analysis. Collection of the samples will be in accordance with applicable procedures in Section 4.0.

8.2 LABORATORY QUALITY CONTROL CHECKS

The laboratory QC program shall ensure the reliability and validity of the analysis performed at the laboratory. All analytical procedures are documented in writing as SOPs, and each SOP includes a QC section which addresses the minimum QC requirements for the procedure. The internal quality control checks might differ slightly for each individual procedure but in general the QC requirements include the following:

- Method blanks;
- Reagent/preparation blanks (applicable to inorganic analysis);
- Instrument blanks;
- Matrix spikes/matrix spike duplicates;
- Surrogate spikes;
- Analytical spikes (Graphite furnace);
- Laboratory duplicates;
- Laboratory control standards;
- Internal standard areas for GC/MS analysis; control limits;
- Mass tuning for GC/MS analysis; and
- Second, dissimilar column confirmation for GC/EC analysis.

A description of the specific QC requirements is described in laboratory SOPs. QC requirements are provided in **Attachment 1**.

9 DATA REDUCTION, VALIDATION AND REPORTING

9.1 DATA REDUCTION

9.1.1 Field Data Reduction Procedures

Data from direct read instrumentation will be recorded on the daily log immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed and dated by the field sampler, and corrected in a space adjacent to the original (erroneous) entry.

The precision of field duplicate measurements will be determined by calculating the relative percent difference (RPD) between the original and the duplicate measurement. The calculation for RPD is provided in Section 12.0 of this QAPP.

9.1.2 Laboratory Data Reduction Procedures

The following protocol will be used for laboratory data reduction. All raw analytical data will be recorded in numerically identified laboratory notebooks or equivalent forms. These notebooks will be issued only by the Laboratory QA Manager. Data are recorded in this notebook along with other pertinent information, such as the sample identification number and the sample tag number. Other details will also be recorded in the lab notebook, such as the analytical method used (SOP#), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings, and the raw data. Each page of the notebook shall be signed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these notebooks by the Lab QA Manager will be completed prior to final data reporting. (Records of notebook entry inspections are maintained by the Lab QA Manager.)

For this project, the equations that will be employed in reducing data are presented in Section 12. Such formulas make pertinent allowances for matrix type. All calculations will be checked by the Organic Section supervisor at the conclusion of each operating day. Errors will be noted, corrections made, with the original notations crossed out legibly. Analytical results for soil samples shall be calculated and reported on a dry weight basis, and TCLP results will not be matrix spike recovery-corrected.

Quality control data (e.g. laboratory duplicates, surrogates, matrix spikes, and matrix spike duplicates) will be compared to the method acceptance criteria. Data considered to be acceptable will be entered into the laboratory computer system. Data summaries will be sent to the Laboratory QA Manager for review. If approved, data are logged into the project database format. Unacceptable data shall be appropriately qualified in the project report. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits, and any other anomalous conditions encountered during sample analysis. After the Lab QA Manager approves these data, they are considered ready for third party data validation.

9.2 DATA VALIDATION

Data validation procedures shall be performed for both field and laboratory operations as described below:

9.2.1 Procedures Used to Evaluate Field Data

Procedures to evaluate field data for this project primarily include checking for transcription errors and review of logbooks, on the part of field crew members. This task will be the responsibility of the project manager, who will otherwise not participate in making any of the field measurements, or in adding notes, data or other information to the logbook.

9.2.2 Procedures to Validate Laboratory Data

All data will be verified by reviewing analytical laboratory data and associated documentation, including Chain-of-Custody records, sample preservation records, analytical holding times, sample data, and reporting requirements. Verification is a preliminary analytical review to ensure that the samples are preserved, shipped, maintained, and analyzed in accordance with established data quality objectives and standard operation procedures. Data will be reviewed for timeliness of the report, chain-of-custody compliance, accurate sample identifications, compliance to extraction and analytical holding times, and quality of analytical chromatograms. Quality control performed by the laboratory, including method blanks, standard and sample matrix spikes, replicate samples, and surrogate spikes, will be evaluated so that the completeness and correctness of the data report can be addressed.

9.3 DATA REPORTING

Data reporting procedures shall be carried out for field and laboratory operations as indicated below:

9.3.1 Field Data Reporting

Field measurement data will be tabulated and submitted as required. The reports will provide summary tables and graphs of the field measurement data. All field measurement data sheets and logbooks will be retained. Raw data will be identified and included in a separate appendix of the final report. The report will contain a section on QA issues documenting any corrective actions taken during the full-scale operations. Reporting requirements are described in Section 12.0 of this QAPP.

9.3.2 Laboratory Data Reporting

The task of reporting laboratory data begins after the validation activity has been concluded. The Laboratory QA Manager must perform a final review of the report summaries and case narratives to determine whether the report meets project requirements. In addition to the record of chain-of-custody, the report format shall consist of the following:

1. Case Narrative:
   - Date of issuance;
   - Laboratory analysis performed;
   - Any deviations from intended analytical strategy;
   - Laboratory batch number;
   - Numbers of samples and respective matrices;
   - Quality control procedures utilized and also references to the acceptance criteria;
   - Laboratory report contents;
   - Project name and number;
   - Condition of samples 'as-received' including cooler temperature;
   - Discussion of whether or not sample holding times were met;
   - Discussion of technical problems or other observations which may have created analytical difficulties;
   - Discussion of any laboratory quality control checks which failed to meet project criteria; and
   - Signature of the Laboratory QA Manager.
2. Chemistry Data Package

- Case narrative for each analyzed batch of samples;
- Summary page indicating dates of analyses for samples and laboratory quality control checks;
- Cross referencing of laboratory sample number to project sample identification numbers;
- Data qualifiers to be used should be adequately described;
- Sample preparation and analyses for samples ;
- Sample results;
- Raw data for sample results and laboratory quality control samples;
- Results of (dated) initial and continuing calibration checks, and GC/MS tuning results;
- Matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, calibration check compounds, and system performance check compound results; and
- Labeled (and dated) chromatograms/spectra of sample results and laboratory quality control checks.

The data package submitted will be a "CLP-like" data package consisting of all the information presented in a CLP data package. A list of deliverables is provided in Table 9-1.

10 PREVENTATIVE MAINTENANCE

10.1 FIELD INSTRUMENT PREVENTATIVE MAINTENANCE

Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Field instruments will be checked and calibrated daily before use. Calibration checks will be documented in the field logbook. Critical spare parts will be kept on-site to reduce downtime. Backup instruments and equipment will be available on-site or within one day shipment to avoid delays in the field schedule.

10.2 LABORATORY INSTRUMENT PREVENTATIVE MAINTENANCE

As part of their QA/QC program, a routine preventative maintenance program is conducted by the laboratory to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees shall regularly perform routine scheduled maintenance and repair of all instruments, or coordinate with the vendor for the repair of all instruments. All maintenance that is performed shall be documented in the laboratory's operating record.

11 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

11.1 ACCURACY ASSESSMENT

In order to assure the accuracy of the analytical procedures, an environmental sample is randomly selected from each sample shipment received at the laboratory, and spiked with a known amount of the analyte or analyses to be evaluated. In general, a sample spike should be included in every set of 20 samples tested on each instrument. The spike sample is then analyzed. The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. Control charts are plotted for each commonly analyzed compound and kept on instrument, matrix, and analyte specific bases. The percent recovery for a spiked sample is calculated according to the following formula:

$$\%R = \frac{\text{Measured Amount in Spiked Sample - Measured Amount in Sample}}{\text{Known Amount Added}} \times 100$$

11.2 PRECISION ASSESSMENT

11.2.1 Laboratory Data

Spiked samples are prepared by choosing a sample at random from each sample shipment received at the laboratory, dividing the sample into equal aliquots, and then spiking each of the aliquots with a known amount of analyte. The duplicate samples are then included in the analytical sample set. The splitting of the sample allows the analyst to determine the precision of the preparation and analytical techniques associated with the duplicate sample. The relative percent difference (RPD) between the spike and duplicate spike are calculated and plotted. The RPD is calculated according to the following formula:

$$RPD = \frac{|\text{Amount in Spike 1 - Amount in Spike 2}|}{0.5(\text{Amount in Spike 1 + Amount in Spike 2})} \times 100$$

11.3 COMPLETENESS ASSESSMENT

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness} = \frac{\text{(number of valid measurements)}}{\text{(number of measurements planned)}} \times 100$$

12 CORRECTIVE ACTION

12.1 CHANGE CONTROL

Prior to implementation, changes to original documents, procedures, and specifications will require approval by the U.S. Navy and the U.S. EPA representatives. It is the responsibility of project personnel to appropriately record the change and to make the documentation available to the PM. The effect of the change on the project will then be evaluated by the PM and SQCS. A description of the proposed change, along with the results of the evaluation, will be presented to the Navy for approval. Following Navy approval, the change will be documented as a revision to the original document to reflect the work as actually performed.

12.2 FIELD CORRECTIVE ACTION

Nonconforming items and activities are those which do not meet the project requirements, procurement document criteria, or approved work procedures. Nonconformances may be detected and identified by:

- Project Staff - During the performance of field investigation and testing, supervision of subcontractors, and preparation and verification of analyses and design,
- Laboratory Staff - During the preparation for and performance of laboratory testing, calibration of equipment, and quality control activities, and
- Quality Assurance Personnel - During the performance of audits or daily review of field reports or data.

Each nonconformance affecting quality will be documented by the personnel identifying the nonconformance. For this purpose, a standard form (e.g., Nonconformance Report), results of laboratory analysis quality control tests, audit report, internal memorandum, or letter will be used as appropriate. Documentation will include:

- Identification of the individual(s) identifying the nonconformance,
- Cause and description of the nonconformance,
- Method(s) for correcting the nonconformance (corrective action) or description of the variance granted,
- Schedule for completing corrective action, and
- Any required approval signatures.

Documentation will be distributed to the Program Quality Control Manager (PQCM) with copies to the PM and SQCS. It is then the responsibility of the PQCM to approve and initiate resolution for the institution of the necessary corrective action to the nonconformance. Documentation describing the nonconformance and the corrective action implemented will be placed in the project file. Completion of corrective actions for significant nonconformances should be verified by quality assurance personnel as part of future auditing activities.

Any recurring nonconformance should be evaluated by project, laboratory, and/or quality assurance personnel to determine its cause and appropriate changes instituted in project requirements and procedures to prevent future recurrence. When such an evaluation is performed, the results will be documented.

12.3 LABORATORY CORRECTIVE ACTION

Corrective action in the laboratory may occur prior to, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, and potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the Laboratory Quality

Assurance (QA) Officer to approve the implementation of corrective action. The laboratory standard operating procedures (SOPs) specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, and automatic reinjection/reanalysis when certain quality control criteria are not met, etc.

The bench chemist will identify the need for corrective action. The Laboratory Project Manager, in consultation with the Laboratory supervisor and staff, will approve the required corrective action to be implemented by the laboratory staff. The Laboratory QA Officer will ensure implementation and documentation of the corrective action. If the nonconformance causes project objectives not to be achieved, it will be necessary to inform all levels of project management to concur with the corrective action.

These corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both Laboratory's corrective action log (signed by analyst, section leader and quality control coordinator), and the narrative data report sent from the laboratory to the Project Chemist. If corrective action does not rectify the situation, the laboratory will contact the TolTest Project Manager.

12.3.1 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

The facility may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required quality assurance objectives (*e.g.*, the holding time for samples is not exceeded, etc.) When the Project Chemist identifies a corrective action situation, it is the Project Manager who will be responsible for approving the implementation of corrective action, including re-sampling, during data assessment. All corrective actions of this type will be documented by the SQCS.

13 QUALITY ASSURANCE REPORTS (POST AUDIT)

Following completion of an audit, the auditors will prepare and submit an audit report to the Project Manager or, as necessary, the Laboratory Manager. This report will serve to notify management of audit results. The report may also be sent to other individuals contacted during the audit and the management of any affected subcontractor.

The report will be prepared as soon as possible (within 30 days) after the audit and contain, as appropriate:

- Date(s) of the audit
- Identification of audit participants
- Identification of activities audited
- Audit results
- Description of items requiring corrective action and, if possible, the means for correction
- Due date for completion of corrective actions and/or audit response
- Means for audit response (i.e., in writing)
- Impact, if any, on project schedule

If corrective action is required in the audit report, the corrective action will be undertaken and completed on schedule unless sufficient evidence can be provided to prove that the action is unnecessary.

The individuals audited will respond in writing to the audit report. The response will clearly state the corrective action taken or planned. If all corrective actions have not been completed prior to issuance of the audit response, a scheduled date for completion must be provided.

TABLES

1-1 RESIDENTIAL AND INDUSTRIAL CLEANUP GOALS FOR CONSTITUENTS OF CONCERN

3-1 QA OBJECTIVES FOR PRECISION, ACCURACY AND COMPLETENESS EXPLOSIVES

3-2 QA OBJECTIVES FOR PRECISION, ACCURACY AND COMPLETENESS PCBs

4-1 ANALYTICAL METHOD REQUIREMENTS

4-2 NUMBER OF SAMPLES PER BUILDING

TABLE 1-1
CLEANUP GOALS FOR SELECT COC's

TABLE 1-1
CLEANUP GOALS FOR SELECT COC's
(mg/kg)

Compound	Residential Cleanup Goal	Industrial Cleanup Goal
HMX	3,300	34,000
RDX	4	17
TNT	15	64
PCBs		1

TABLE 3-1
QUALITY CONTROL OBJECTIVES FOR EXPLOSIVES (Method 8330)

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B)

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).	Flagging criteria are not appropriate.	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.
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	Flagging criteria are not appropriate.	Samples cannot be analyzed without a valid LOD.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If LOQ is not set at the lowest calibration standard, must be verified with each batch. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each. See Box D-14.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. See Box D-14.	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	Flagging criteria are not appropriate.	Samples cannot be analyzed without a valid LOQ.
Soil drying procedure	Each sample and batch LCS.	Laboratory must have a procedure to determine when the sample is dry to constant weight. Record date, time, and ambient temperature on a daily basis while drying samples.	NA.	Flagging criteria are not appropriate.	

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soil sieving procedure	Each sample and batch LCS.	Weigh entire sample. Sieve entire sample with a 10 mesh sieve. Breakup pieces of soil (especially clay) with gloved hands. Do not intentionally include vegetation in the portion of the sample that passes through the sieve unless this is a project specific requirement. Collect and weigh any portion unable to pass through the sieve.	NA.	Flagging criteria are not appropriate.	
Soil grinding procedure	Initial demonstration.	The laboratory must initially demonstrate that the grinding procedure is capable of reducing the particle size to < 75 um by passing representative portions of ground sample through a 200 mesh sieve (ASTM E11).	NA.	Flagging criteria are not appropriate.	
Soil grinding blank	Between each sample.	A grinding blank using clean solid matrix (such as Ottawa sand) must be prepared (e.g., ground and subsampled) and analyzed in the same manner as a field sample. Grinding blanks can be analyzed individually or composited. No target analytes detected greater than 1/2 Reporting Limit (RL).	All blank results must be reported and the affected samples must be flagged accordingly if blank criteria is not met.	If the composite grinding blank exceeds the acceptance criteria, apply B-flag to all samples associated with the grinding composite. If any individual grinding blank is found to exceed the acceptance criteria, apply B-flag to the sample following that blank.	

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soil subsampling process	Each sample, duplicate, and batch LCS.	Entire ground sample is mixed, spread out on a large flat surface (e.g., baking tray), and 30 or more randomly located increments are removed from the entire depth to sum a ~10 g subsample.	NA.	Flagging criteria are not appropriate.	
Soil sample triplicate	At the subsampling step, one sample per batch. Cannot be performed on any type of blank sample.	Three 10 g subsamples are taken from a sample expected to contain the highest levels of explosives within the Quantitation Range of the method. The % RSD (percent relative standard deviation) for results above the RL must not exceed 20%.	Corrective action must be taken if this criterion is not met (e.g., the grinding process should be investigated to ensure that the samples are being reduced to a sufficiently small particle size).	Flagging criteria are not appropriate.	
Aqueous sample preparation	Each sample.	Solid phase extraction (SPE) using resin-based solid phase disks or cartridges is required. The salting-out procedure is not permitted.	NA.	Flagging criteria are not appropriate.	
Initial calibration (ICAL)	Minimum of 5 calibration standards with the lowest standard concentration at or below the RL. Once calibration curve or line is generated, the lowest calibration standard must be re-analyzed.	The apparent signal-to-noise ratio at the RL must be at least 5:1.If linear regression is used, $r \geq 0.995$. If using Internal Standardization, RSD $\leq$ 15%.	Correct problem, then repeat initial calibration.	Flagging criteria are not appropriate.	No samples can be run without a valid initial calibration.Detection by HPLC UV, LC/MS, or LC/MS/MS is allowed.
Second source calibration verification (ICV)	Once after each ICAL, at lowest calibration point, prior to beginning a sample run.	All analyte(s) and surrogates within ± 20% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	Prior to sample analysis, after every ten samples, and at the end of the analysis sequence.	All target analytes and surrogates within ± 20% of the expected value from the ICAL.	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	If reanalysis can not be performed, data must be qualified and explained in the case narrative. Apply Q-flag 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.
Method blank	One per preparatory batch.	No analytes detected > ½ RL and greater than 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 criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis can not 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.
LCS containing all analytes to be reported	One per preparatory batch.	A solid reference material containing all reported analytes must be prepared (e.g., ground and subsampled) and analyzed in exactly the same manner as a field sample. In-house laboratory control limits for the LCS must demonstrate the laboratory's ability to meet the project's MQOs.	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 can not be performed, data must be qualified and explained in the case narrative. Apply Q-flag 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.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. Percent recovery must meet LCS 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 J-flag 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.

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
MSD or sample duplicate	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. Percent recovery must meet LCS limits and relative percent difference (RPD) < 20%.	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 J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Confirmation analysis	When target analytes are detected on the primary column using the UV Detector (HPLC) at concentrations exceeding the Limit of Detection (LOD).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	Report from both columns.	If there is a > 40% RPD between the two column results, data must be J-flagged accordingly.	Confirmation analysis is not needed if LC/MS or LC/MS/MS was used for the primary analysis. Secondary column – Must be capable of resolving (separating) all of the analytes of interest and must have a different retention time order relative to the primary column. Any HPLC column used for confirmation analysis must be able to resolve and quantify all project analytes. Detection by HPLC UV, LC/MS or LC/MS/MS. Calibration and calibration verification acceptance criteria is the same as for the primary analysis.
Results reported between LOD and LOQ	NA.	NA.	NA.	Apply J-flag to all results between LOD and LOQ.	

TABLE 3-2
QUALITY CONTROL OBJECTIVES FOR
PCBs (Method 8081, 8082)

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8130, 8330 and 8330C)

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).	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.
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	NA.	Samples cannot be analyzed without a valid LOD.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If LOQ is not set at the lowest calibration standard, must be verified with each batch. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each. See Box D-14.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. See Box D-14 and Appendix G.	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	NA.	Samples cannot be analyzed without a valid LOQ.
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 72-hour study.	NA.	NA.	

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8130, 8330 and 8330C) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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 for all analytes (ICAL)	Initial calibration 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) r2 ≥ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat initial calibration.	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. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
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.	
Calibration verification: **ICV (second source)** **CCV**	ICV: Immediately following ICAL. 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. GC methods: All project analytes within ± 20% of expected value from the ICAL; HPLC methods: All project analytes within ± 15% of expected value from the ICAL.	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem, then rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	ICV: Flagging criteria are not appropriate. CCV: If reanalysis can not be performed, data must be qualified and explained in the case narrative. Apply Q-flag 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.

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8130, 8330 and 8330C) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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 (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis can not 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. See Box D-3 and Appendix G.	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 can not be performed, data must be qualified and explained in the case narrative. Apply Q-flag 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.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	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 J-flag 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 (see Box D-7).	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 J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TABLE 4-1
ANALYTICAL METHOD REQUIREMENTS

TABLE 4-1 ANALYTICAL METHOD REQUIREMENTS				
Analytes	**Analytical Method[1]**	**Sample Container[2]**	**Preservation**	**Holding Time**
Explosives	8330 (full list)	1 8-ounce glass jar with Teflon-lined lid or brass sleeve	Ice to 4°C	14 days for extraction; analyze within 40 days of extraction
SWMU-specific PCBs per Section 1.4.2.1	8082	1 8-ounce glass jar	Ice to 4°C	14 days for extraction; analyze within 40 days of extraction
Water Analyses (decontamination water)				
Explosives	8330 (full list)	2 1-L amber glass jars	Ice to 4°C	7 days; analyzed within 40 days after extraction
Additional Water Analyses (blanks)				
Explosives	8330 (full list)	2 1-L amber glass jars	Ice to 4°C	7 days; analyze within 40 days after extraction
Notes:				

TABLE 4-2
APPROXIMATE NUMBER OF SAMPLES PER BUILDING

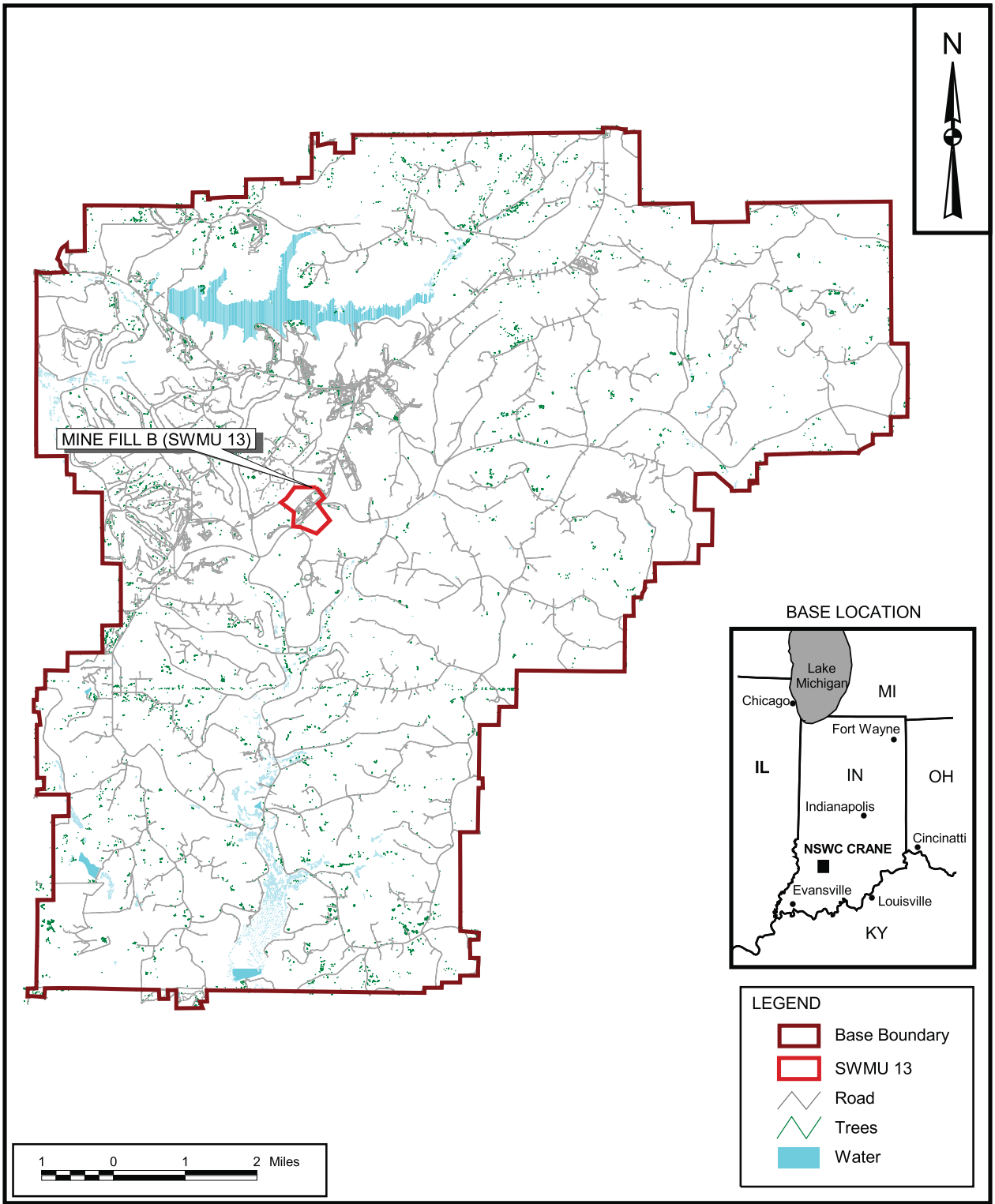
Table 4-2 – Approximate Number of Samples Per Building

Building #	Approx. # of Composite Explosive Samples	Approx. # of Composite PCB Samples
157	3	
158	2	
159	9	
2715	9	
3036	3	
161	5	
166	6	6
167	2	
168	9	
171	6	6
172	2	
173	9	
2171	20	
2179	1	
2500	9	
2501	9	
2502	6	
2503	6	
2512	1	
2514	1	
2515	1	

FIGURES

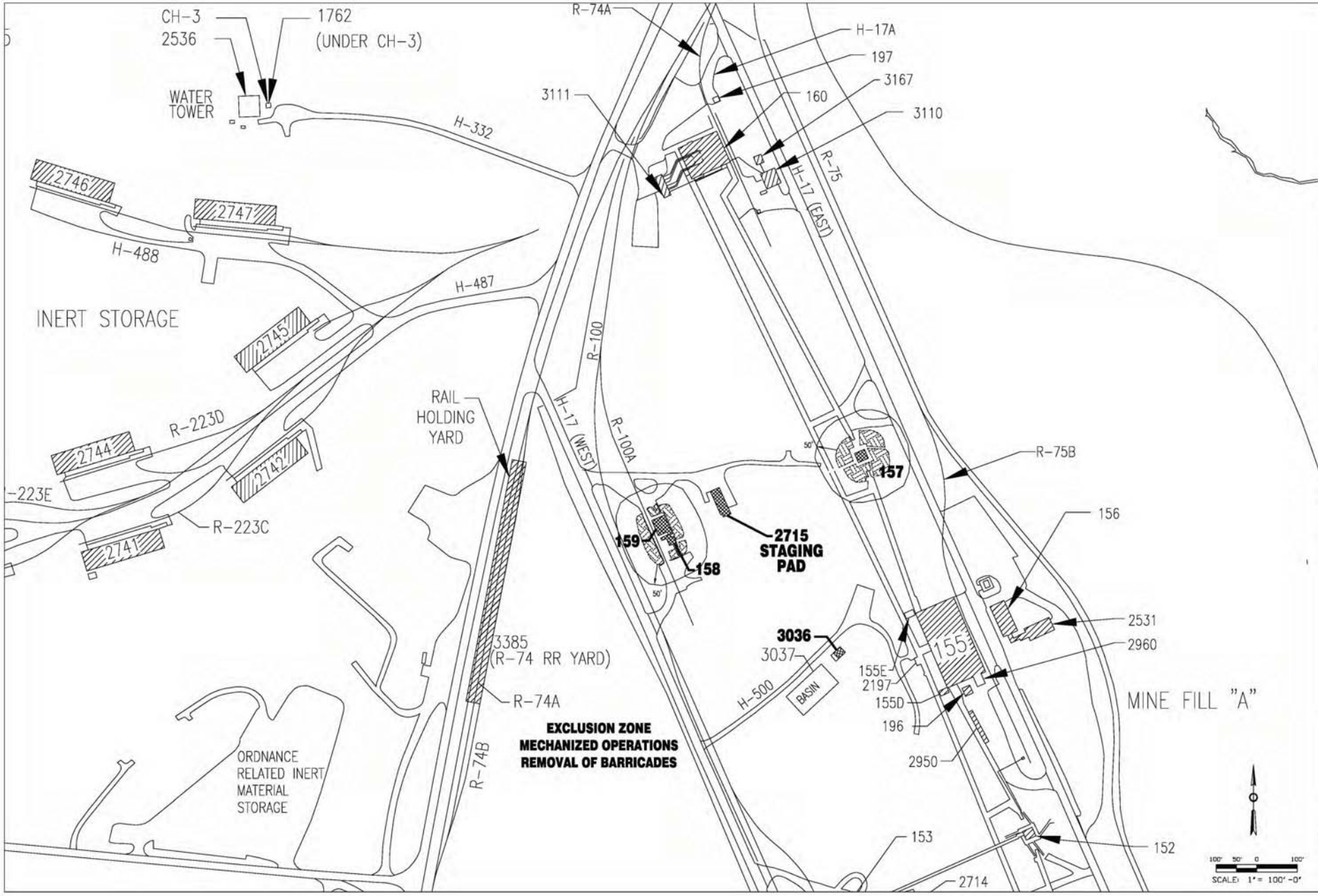
FIGURE 1 SITE LOCATION MAP
FIGURE 2 MINE FILL A BUILDING LOCATIONS
FIGURE 3 MINE FILL B BUILDING LOCATIONS
FIGURE 4 SAMPLING GRID
FIGURE 5 CONVEYOR LINE SAMPLING DIAGRAM

FIGURE 1



DRAWN BY J. LAMEY DATE 8/27/01	Tetra Tech NUS, Inc.	CONTRACT NUMBER 112G00352	OWNER NO. 0020
CHECKED BY T. SMITH DATE 2/16/07	BASE AND SITE LOCATION MAP SWMU 13 - MINE FILL B INTERIM MEASURES WORK PLAN NSWC CRANE CRANE, INDIANA	APPROVED BY ____ DATE ____	
COST/SCHEDULE-AREA		APPROVED BY ____ DATE ____	
SCALE AS NOTED		DRAWING NO. FIGURE 1 - 1	REV 0

P:\GIS\CRANE_NSWC\SURFICIAL.APR SITE LOCATIONS LAYOUT 2/16/07 SP



SATISFACTORY TO: DATE:

REVISIONS

LTR	DESCRIPTION	PREP BY	DATE	DR. CK.
A	OPERATION CHANGES	B. DUZAN	9/23/09	SUBM.
B	NCSSA CHANGES	B. DUZAN	12/02/09	SUBMITTED BY (PWD MANAGER-TRLCD)
				TECHNICAL BRANCH

NAVAL SUPPORT ACTIVITY, CRANE — CRANE, INDIANA

DEMOLITION/DISPOSAL OF SELECTED BUILDINGS IN MINE FILLS A & B

FINAL OPERATIONS OPERATIONS, MINE FILL A

APPROVED — DATE — EFD FOR COMMANDER, NAVFAC

NAVFAC

RECORD DRAWING DATE

CODE I.D. NO. 80091

DRAWING SIZE: D

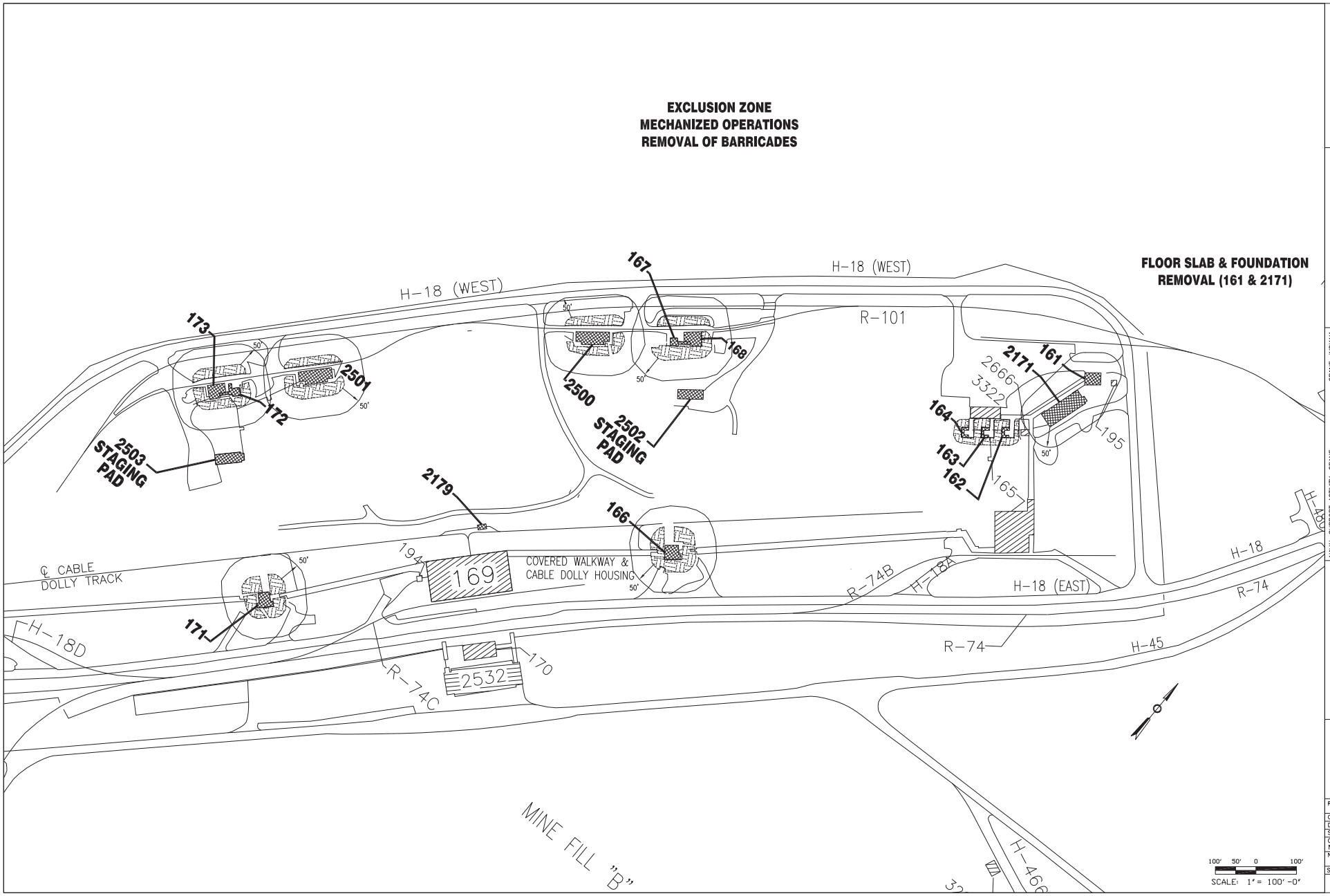
SPEC. NO.06-

CONSTRN. CONTR. NO. N62467-

NAVFAC DRAWING NO. 5/A 16-07

SHEET 1 OF 7

APPENDIX D



SATISFACTORY TO: DATE:

REVISIONS

LTR	DESCRIPTION	PREP BY	DATE	DESIGN	DR B. DUZAN	CHK
A	OPERATION CHANGES	B DUZAN	9/23/09	SUPV	CH ENGR	
B	NOSSA CHANGES	B DUZAN	12/02/09	SUBMITTED BY (PRN MEMBER-TITLE)		DATE
				TECHNICAL BRANCH		
				TPC		

NAVAL SUPPORT ACTIVITY, CRANE — CRANE, INDIANA

DEMOLITION/DISPOSAL OF SELECTED BUILDINGS IN MINE FILLS A & B

FINAL OPERATIONS, OPERATIONS, MINE FILL B

APPROVED: DATE: ETD FOR COMMANDER, NAVFAC

NAVFAC

RECORD DRAWING DATE

CODE I.D. NO. 80091

DRAWING SIZE: D

SPEC. NO.06-

CONSTRN. CONTR. NO. N62467-

NAVFAC DRAWING NO. S/A 16-07

SHEET 2 OF 7

APPENDIX D

Figure 4-General Sampling Grid

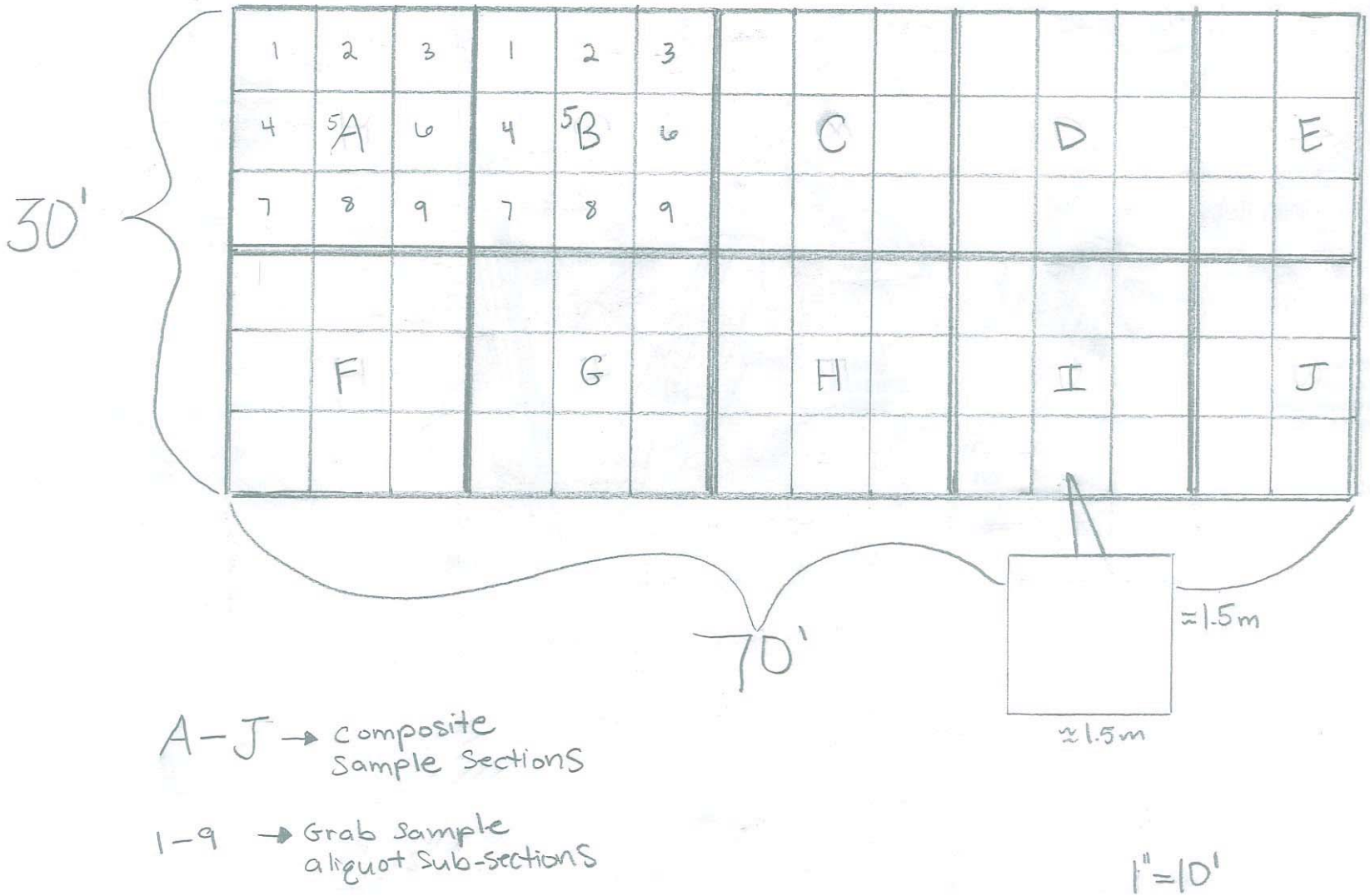
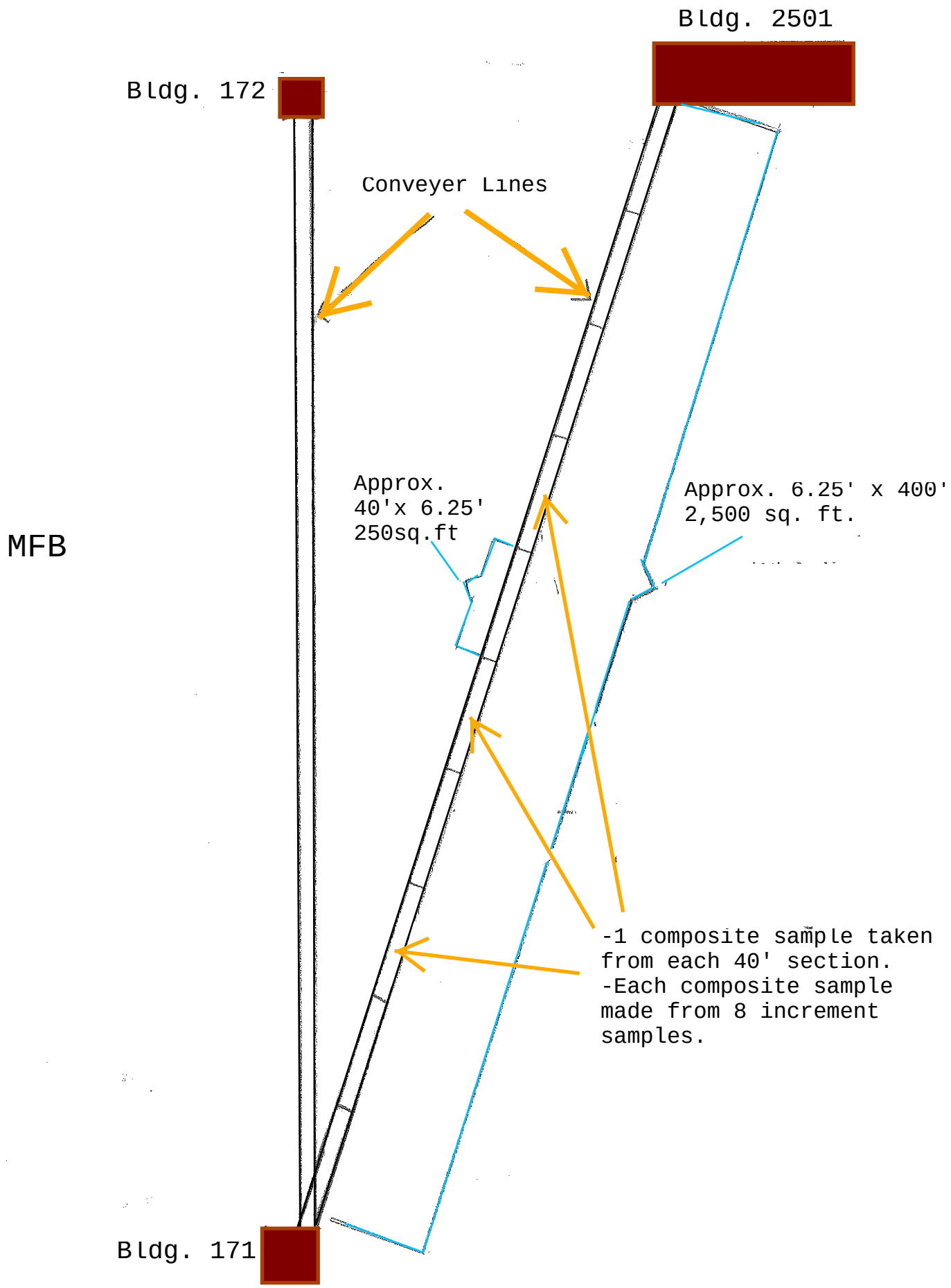


Figure 5 - General Conveyor Line Sampling Diagram



APPENDIX A

FIELD SAMPLING STANDARD OPERATING PROCEDURES

1.0 PURPOSE

The purpose of this procedure is to provide information on the proper decontamination of hand tools and drilling equipment used to perform field investigations to ensure that chemical analysis results reflect the actual concentrations present at sampling locations.

2.0 SCOPE

Hand tools and drilling equipment used in sampling activities must be properly cleaned and decontaminated to minimize the potential for cross-contamination between sampling locations and the transfer of contaminants off site. This procedure addresses the proper decontamination of hand tools and drilling equipment.

3.0 REFERENCES

- U.S. Environmental Protection Agency, December 1987. *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001.
- U.S. Environmental Protection Agency, November 1986. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, EPA SW-846, Third Edition.
- U.S. Environmental Protection Agency, September 1986. Resource Conservation and Recovery Act (RCRA) *Groundwater Monitoring Technical Enforcement Guidance Document*, OSWER-9950.1.

4.0 DEFINITIONS

- **Negative Contamination.** Occurs when the measured concentration of the analyte is artificially reduced due to volatilization, adsorption, and related losses
- **Positive Contamination.** Occurs when the measured concentration of the analyte is artificially raised due to leaching or the introduction of foreign matter
- **Cross Contamination.** A type of positive contamination caused by the introduction of one sample into a second sample during collection or storage

5.0 RESPONSIBILITIES

5.1 Project Manager

The Project Manager is responsible for ensuring that decontamination procedures for all hand tools and drilling equipment are defined prior to the actual field effort. Field personnel should be briefed and trained to execute the task.

5.2 Field Associate

The Field Associate is responsible for ensuring that project-specific plans and the implementation of field investigations are in compliance with this SOP.

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6.0 EQUIPMENT

- Scrub brushes
- Detergent: a laboratory-grade non-phosphate such as Alconox or Liquinox
- Potable (suitable for drinking) water

7.0 PROCEDURE

All equipment involved in field sampling activities will be decontaminated prior to entering the site, before sampling has begun, and prior to leaving the site. All down-hole equipment will be decontaminated between borings. Transport vehicles used on site for personnel and/or equipment will be cleaned prior to leaving the site.

Equipment directly utilized for sampling, such as drilling rigs, augers, backhoes, hand tools, down-hole tools, and transport vehicles will be decontaminated at a designated area using the following procedure:

1. Scrub with potable water and a non-phosphate laboratory-grade detergent.
2. Rinse with potable water.
3. Air dry

8.0 ATTACHMENTS

None

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STANDARD OPERATING PROCEDURE (SOP) 8-3

SOIL SAMPLING (SURFACE SOILS, HAND AUGER AND GRAB SAMPLING)

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1.0 Purpose

The purpose of this procedure is to outline the requirements for sampling surface soils, hand auger sampling, and grab sampling of soils. Soil sampling for environmental projects is normally conducted to define soil types and geological characteristics present in the subsurface and to determine the nature and extent of soil contamination. Soil samples are collected at individual sampling locations across the surface area of investigation as well as at various depths within each sample location to characterize conditions and features.

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2.0 Scope

Soil sampling is potentially applicable to any hazardous waste site. A variety of sampling techniques is available for collection of soil samples. These include grab sampling, split-spoon sampling, collection of hand augured soil samples, Shelby tube sampling, and continuous coring.

Collection of surface samples may require no special sampling equipment or highly detailed procedures. Samples may be obtained on or in the vicinity of a hazardous waste site and should be representative of the nature of local surface soils or sediment.

3.0 References

· U.S. Environmental Protection Agency (EPA), 1987, *A Compendium of Superfund Field Operation Methods.*

4.0 Definitions

Auger Cuttings - soil brought to the surface by the action of the augers as they are drilled into the ground.

Hazardous Waste Sample - medium to high concentration sample (e.g., source material, sludge, leachate) requiring DOT labelling and off-site laboratory handling as a high hazard sample.

5.0 Responsibilities

5.1 Project Manager

The Project Manager is responsible for overall management of field activities and ensuring that appropriate soil sampling procedures are followed.

5.2 Site Geologist

The Site Geologist directly supervises sampling procedures, classifies soil and rock samples, and directs the packaging and sealing of soil and rock samples for shipment to an off-site laboratory. Such duties may also be performed by the on-site geotechnical engineer or Field Associate as required by an individual project.

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6.0 Equipment

The following pieces of equipment may be needed to collect samples:

- Drilling equipment - capable of collecting depth-specific samples
- Stainless steel hand auger, shovel, AMS sampling equipment or post-hole digger
- Teflon® tape
- Teflon® or stainless steel spatula or short, hand-pushed core tube
- Stainless steel bowl or Teflon® mixing bowl
- Organic vapor monitoring device (photoionization detector or organic vapor analyzer)
- Appropriate sampling containers
- Reclosable plastic bags
- Decontamination supplies as specified in TolTest SOP 3-1 and/or the project work plan or sampling plan.
- Field logbook and field sampling forms

7.0 Procedure

The soil sample collection point in the field should be within two feet horizontally of the pre-determined sample location identified in the proposal or work plan. The final location of the sampling point should be defined by surveying or measuring from previously surveyed points following procedures outlined in TolTest SOP 1-6, Site Surveying and Mapping. The accuracy of the soil sampling point location will be determined by the data quality objectives. Sample collection information should be recorded in the field logbook and in the sampling forms.

Surface/air contact may be minimized by placing the sample in an airtight container immediately after collection.

Sampling and sample preparation equipment will be decontaminated in accordance with TolTest SOP 3-3, Decontamination of Manual Sampling and Drilling Equipment, prior to and after each sample is collected unless disposable equipment is used.

If necessary or required, soil sample locations can be permanently identified using a brass surveyor's pin or equivalent permanent marker inscribed with the boring location identification number and placed in a concrete form. Depth-profile sampling must comply with the above requirements. Care must be taken to prevent cross-contamination and misidentification of samples. Vertical depth control tolerances are specified in the TolTest Quality Assurance/Quality Control Manual.

Sampling equipment should be decontaminated by following TolTest SOP 3-1, Decontamination of Ground-Water and Soil Sampling Equipment.

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7.1 Surface Soils Sampling Methods

Samples of surface soils should be collected from the ground surface to a depth of approximately six inches. Samples may be collected using a short, hand-pushed core tube or a stainless steel or polyethylene scoop or shovel. Disposable equipment is preferred, especially if the materials are heavily contaminated. Reused sampling equipment must be decontaminated between uses as describe in TolTest SOP 3-1.

7.2 Hand Augered Sampling Methods

Decontaminated equipment will be used to collect the soil sample. A hand auger consists of a sample bucket attached to the bottom of a length of steel pipe that has a crossbar (handle) at the top. A hole is drilled by turning this crossbar at the same time the operator presses the auger into the ground. The hand auger is driven to the desired depth, which is usually within a few feet of the surface.

Hand-augured samples can be collected directly from the auger bucket or from liners placed within the auger bucket. Liners can be two, four, or six inches in length. The liner for volatile organic analyses is sealed with Teflon® tape and plastic end caps, labeled and sent for analysis.

Geologic data must be maintained on the TolTest Soil Boring Log (see SOP 6-3). Hand auger buckets normally are 6 inches in length. As a result, four individual sample intervals can be obtained over a two-foot section of a boring. Samples from these intervals can be analyzed individually or composited as required by the work plan.

Soil samples that are not to be analyzed for volatile organics are placed in a stainless steel bowl and homogenized with a stainless steel spoon or Teflon® spatula. Large pebbles and cobbles should be removed from the sample. The composite samples for chemical analyses are placed in appropriate containers.

Place the samples on ice in a cooler maintained at 4° Celsius (°C).

Note the sample identification, sample location (provide sketch), sampling time, and sampling personnel in the Field Logbook.

After auguring and sampling activities are completed, the borehole will be filled using soil cuttings, granulated bentonite or concrete as specified in the work plan or proposal. The ground surface will be restored to its original condition. Sampling equipment should be decontaminated following TolTest SOP 3-1, Field Decontamination of Groundwater and Soil Sampling Equipment.

7.3 Grab Sampling Methods

Grab samples are usually obtained from auger cuttings as they are brought to the surface by the continuous flight augers during rotary drilling activities. Careful determination of the sample's depth must be made based on the speed of the augers and visual assessment of previous cuttings.

Soil samples that are not to be analyzed for volatile organics are placed in a stainless steel bowl and homogenized with a stainless steel spoon or Teflon® spatula. Large pebbles and cobbles should be removed from the sample. The composite samples for chemical analyses are placed in appropriate containers.

Place the samples on ice in a cooler maintained at 4°C.

Note the sample identification, sample location (provide sketch), sampling time, and sampling personnel in the Field Logbook.

8.0 Attachments

None.

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STANDARD OPERATING PROCEDURE (SOP) 8-10

PACKAGING AND SHIPMENT OF FIELD SAMPLES

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1.0 Purpose

The purpose of this procedure is to define the requirements necessary to properly package environmental samples and provide the necessary chain-of-custody records used in sample transfers.

2.0 Scope

This procedure applies to the packaging, shipping and documentation of samples being transferred from the field to the laboratory for analysis. Specifically, this document outlines shipping and sample documentation procedures that are in accord with the U.S. Department of Transportation (DOT). This procedure is also applicable to all samples taken from uncontrolled hazardous substance sites for analysis at laboratories away from the site; however, this procedure does not take precedence over region-specific or site-specific requirements for sample transportation and chain-of-custody documentation.

Careful packaging, shipping and documentation are necessary to insure that all samples received are undamaged and authentic. Transportation of environmental samples requires proper selection of shipping method, containerization and container seal materials.

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3.0 References

U.S. Department of Transportation, 1983. *Hazardous Materials Regulations*, 49 CFR 171- 177.

4.0 Definitions

Carrier - A person or firm engaged in the transportation of passengers or property.

Chain-of-custody record form - A printed form that accompanies a sample or group of samples as custody of the sample(s) is transferred from one custodian to the subsequent custodian. Attachment 8-10A shows a TolTest chain-of-custody form. The chain-of-custody record form is a controlled document. One copy of the form must be retained in the project file.

Custodian - The person responsible for the custody of samples at a particular time, until custody is transferred to another person (and so documented), who then becomes custodian. A sample is under

your custody if:

- You possess the sample.
- It is in your view after being in your physical possession.
- It was in your physical possession and then you locked it up to prevent tampering.
- You have designated and identified a secure area to store the sample.

Environmental sample - low concentration sample typically collected off site and not requiring DOT hazardous waste labelling as a high hazard sample.

Hazardous waste sample - medium or high concentration sample (e.g., source material, sludge, leachate) requiring DOT labelling and contract lab handling as a high hazard sample.

Hazardous material - A substance or material in a quantity and form which may pose an unreasonable risk to health and safety or property when transported. Defined and regulated by DOT (49 CFR 173.2) and listed in Attachment 8-10B.

Hazardous waste - Any substance listed in 40 CFR Subpart D (261.30 et seq) or 40 CFR otherwise characterized as ignitable, corrosive, reactive, or EP toxic as specified under Subpart C (261.20 et seq) that would be subject to manifest requirements specified in 40 CFR 262. Defined and regulated by the Environmental Protection Agency (EPA).

Marking - Applying the descriptive name, instructions, cautions, weight, or specification marks or combination thereof required to be placed outside containers of hazardous materials.

Packaging - The assembly of one or more containers and any other components necessary to assure compliance with the minimum packaging requirements of 49 CFR 172, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank car tanks.

Placard - Color-coded, pictorial sign depicting the hazard class symbol and name to be placed on all four sides of a vehicle transporting certain hazardous materials.

Reportable quantity (RQ) - A parenthetical note of the form "(RQ-1000/454)" following an entry in the DOT Hazardous Materials table (49 CFR 172.101) indicates the reportable quantity of the substance in pounds and kilograms. If a spill of that amount or more of the substance occurs during transit or storage, a report must be filed with DOT according to 171.15-17 concerning hazardous material incident reports. If the material spilled is a hazardous waste, a report must always be filed, regardless of the amount, and must include a copy of the manifest. If the RQ notation appears, it must be shown either immediately before or after the proper shipping name on the shipping paper (or manifest). Most shipping papers and manifests will have a column designated "HM" which may be used for this purpose.

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Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the point and time that it was collected.

5.0 Responsibilities

5.1 Project Manager

The Project Manager is responsible for determining that samples are properly packaged and shipped and for determining that the chain-of-custody procedures are implemented from the time the samples are collected to their release to the shippers.

5.2 Field Associate

The Field Associate is responsible for implementing the packaging and shipping requirements and for initiating the chain-of-custody records until they are relinquished to another custodian, to the shipper, or to the carrier.

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6.0 Equipment

- Coder
- Teflon® and nylon strapping tape
- Vermiculite or styrofoam packaging materials
- Bubble pack
- Sampling gloves
- Poly-net
- Reclosable plastic bags
- Permanent felt tip marker
- Pen, black permanent ink
- Shipping coolers
- Ice or blue ice

7.0 Procedure

7.1 Sample Packaging and Shipping

Samples collected for shipment from a site should be classified as either environmental or hazardous material (or waste) samples. In general, environmental samples are collected off site (for example, from streams, ponds, or wells) and are not expected to be grossly contaminated with high levels of hazardous materials. On-site samples (for example, soil, water, and materials from drums or bulk storage tanks,

obviously contaminated ponds, lagoons, pools, and leachates from hazardous waste sites) are considered hazardous. A distinction must be made between the two types of samples in order to:

1. Determine the appropriate procedures for transportation of samples. If there is any doubt, a sample should be considered hazardous and shipped accordingly.

2. Protect the health and safety of laboratory personnel receiving the samples. Special precautions are used at laboratories when samples other than environmental samples are received.

7.2 Environmental Samples

7.2.1 Packaging

1. Environmental samples may be packaged following the procedures outlined in Section 7.4 for samples classified as "flammable liquids" or "flammable solids". Requirements for marking, labeling, and shipping papers do not apply. Environmental samples may also be packaged without being placed inside metal cans as required for flammable liquids or solids.

2. Place the sample container, properly identified and with a sealed lid, in a polyethylene bag and seal the bag.

3. Place the sample in a fiberboard container or cooler which has been lined with a large polyethylene bag and contains ice, blue ice packs or frozen bottled water.

4. Pack with enough noncombustible, absorbent, cushioning materials to minimize the possibility of the container breaking.

5. Seal the large polyethylene bag.

6. Seal or close the outside container.

7.2.2 Marking/Labeling

Sample containers must have a completed sample identification and the outside container must be marked "Environmental Sample." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT markings or labeling are required.

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7.2.3 Shipping Papers

No DOT shipping papers are required. However, the appropriate chain-of-custody forms must be included with the shipment. Core must be taken to obtain and list the correct laboratory address on shipping documents. In addition, the type of shipment method (overnight, second day air, regular mail) must be specified as required by the project.

7.2.4 Transportation

There are no DOT restrictions on mode of transportation.

7.3 Determination of Shipping Classification for Hazardous Material Samples

Samples not determined to be environmental samples, or samples known or expected to contain hazardous materials, must be considered hazardous material samples and transported according to the requirements listed below.

7.3.1 Known Substances

If the substance in the sample is known or can be identified, package, mark label, and ship according to the specific instructions for that material (if it is listed) in the DOT Hazardous Materials Table, 49 CFR 172.101.

The following steps are provided to help in locating a proper shipping name from the Hazardous Materials Table, 48 CFR 172.101.

1. Look first for the chemical or technical name of the material, for example, ethyl alcohol. Note that many chemicals have more than one technical name; for example, perchloroethylene (not listed in 172.101) is also called tetrachloroethylene (listed in 172.101). It may be useful to consult a chemist for all possible technical names a material can have. If your material is not listed by its technical name, then

2. Look for the chemical family name. For example, pentyl alcohol is not listed, but the chemical family name is alcohol, n.o.s. (not otherwise specified). If the chemical family name is not listed, then

3. Look for a generic name based on end use. For example, Paint, n.o.s. or Fireworks, n.o.s. If a generic name based on end use is not listed, then

4. Look for a generic family name based on end use. For example, Drugs, n.o.s. or Cosmetics, n.o.s. Finally if your material is not listed by a generic family name but you suspect or know the material is hazardous because it meets the definition of one or more hazard classes, then

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5. You will have to go to the general hazard class for a proper shipping name. For example, Flammable Liquid, n.o.s., or Oxidizer, n.o.s.

7.3.2 Unknown Substances

For samples of hazardous substances of unknown content, select the appropriate transportation category according to the DOT Hazardous Materials Classification (Attachment 8-10B), a priority system of transportation categories.

The correct shipping classification for an unknown sample is selected through a process of elimination, utilizing Attachment 8-10B. Unless known or demonstrated otherwise (through the use of radiation survey instruments), the sample is considered radioactive and appropriate shipping regulations for "radioactive materials" are followed.

If radioactive material is eliminated, the sample is considered to contain "Poison A" materials (Attachment 8-10C), the next classification on the list. DOT defines "Poison A" as extremely dangerous poisonous gases or liquids of such a nature that a very small amount of gas, or vapor of the liquid, mixed with air is dangerous to life. Most Poison A materials are gases or compressed gases and would not be found in drum-type containers. Liquid Poison A would be found only in closed containers; however, all samples taken from closed drums do not have to be shipped as Poison A, which provides for a "worst case" situation. Based upon information available, a judgement must be made whether a sample from a closed container is a Poison A.

If Poison A is eliminated as a shipment category, the next two classifications are "flammable" or "nonflammable" gases. Since very few gas samples are collected, "flammable liquid" would be the next applicable category. With the elimination of radioactive material, Poison A, flammable gas, and nonflammable gas, the sample can be classified as flammable liquid (or solid) and shipped accordingly. These procedures would also suffice for shipping any other samples classified below flammable liquids in the DOT classification table (Attachment 8-10B). For samples containing unknown materials, categories listed below flammable liquids/solids on Attachment 8-10B are generally not used because showing that these materials are not flammable liquids (or solids) requires flashpoint testing, which may be impractical and possibly dangerous at a site. Thus, unless the sample is known to consist of material listed as less hazardous than flammable liquid (or solid) on Attachment 8-10B, it is considered a flammable liquid (or solid) and shipped as such. For any hazardous material shipment, utilize the shipping checklist as a guideline to ensure that all sample-handling requirements are satisfied.

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7.4 Packaging and Shipping of Samples Classified as Flammable Liquid (or Solid)

7.4.1 Packaging

Applying the word "flammable" to a sample does not imply that it is in fact flammable. The word prescribes the class of packaging according to DOT regulations.

1. Collect the sample in the prescribed container with a nonmetallic, Teflon®-lined screw cap. To prevent leakage, fill container no more than 90% full. If an air space in the sample container would affect sample integrity, place that container within a second container to meet the 90% requirement.

2. Complete the sample label and identification tag and attach it securely to the sample container.

3. Seal the container and place in a 2-ml thick (or thicker) polyethylene bag, one sample per bag. Position identification tag so that it can be read through the bag. Seal the bag.

4. Place the sealed bag inside the metal can and cushion it with enough noncombustible, absorbent material (for example, vermiculite or diatomaceous earth) between the bottom and sides of the can and bag to prevent breakage and absorb any leakage. Pack one bag per can. Use clips, tape, or other positive means to hold can lid securely, tightly and permanently. Mark the can as indicated in Paragraph 1 of Section 7.4.2, below.

5. Place one or more metal cans (or a single 1-gallon bottle) into a strong outside container, such as a cooler or a DOT-approved fiberboard box. Surround the cans with noncombustible, absorbent cushioning material for stability during transport. Mark containers as indicated in Paragraph 2 of Section 7.4.2.

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7.4.2 Marking/Labeling

1. Use abbreviations only where specified. Place the following information, either-hand printed or in label form, on the metal can (or the 1-gallon bottle):

   - Laboratory name and address.
   - "Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325".

Not otherwise specified (n.o.s.) is not used if the flammable liquid (or solid) is identified. Then the name of the specific material is listed before the category (for example, Acetone, Flammable Liquid), followed by its appropriate UN number found in the DOT hazardous materials table (49 CFR 172.101).

Place all information on the outside shipping container as on the inner can (or bottle), specifically:

- Proper shipping name.

- UN or NA number.
- Proper label(s).
- Addressee and sender.

Place the following labels on the outside container: "Cargo Aircraft Only" and "Flammable Liquid" (or "Flammable Solid"). "Dangerous When Wet" label should be used if the solid has not been exposed to a wet environment. "Laboratory Samples" and "THIS SIDE UP" or "THIS END UP" should also be marked on the top of the outside container, and upward-pointing arrows should be placed on all sides of the container.

7.4.3 Shipping Papers

1. Use abbreviations only where specified. Complete the carrier-provided bill of lading and sign the certification statement (if the carrier does not provide one then use the standard industry form, see Attachment 8-10D). Provide the following information in the order listed (one form may be used for more than one exterior container).

   - "Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325).
   - "Limited Quantity" (or "Ltd. Qty.")
   - "Cargo Aircraft Only
   - Net weight (wt) or net volume (vol), just before or just after "Flammable Liquid n.o.s." or "Flammable Solid, n.o.s.," by item, if more than one metal can is inside an exterior container
   - "Laboratory Samples" (if applicable)

2. Include the chain-of-custody record, properly executed in the outside container.

3. "Limited Quantity" or "Flammable Liquid, n.o.s." is limited to one pint per inner container. For "Flammable Solid, n.o.s.", the net weight of the inner container plus the sample should not exceed one pound; total package weight should not exceed 25 pounds.

7.4.4 Transportation

1. Transport unknown hazardous substance samples classified as flammable liquids by rented or common carried truck, railroad, or express overnight package services. Do not transport by any passenger-carrying air transport system, even if they have cargo-only aircraft. DOT regulations permit passenger airline company cargo only aircraft, but difficulties with most of these suggest avoiding them. Instead, ship by airline carriers that only carry cargo.

2. For transport by government-owned vehicle, including aircraft, DOT regulations do not apply. However, procedures described above, with the exception of execution of the bill of lading with certification, should still be used.

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7.5 Packaging and Shipping Of Samples Classified as Poison "A"

This packaging, marking, labeling, and shipping method provides a worst-case procedure for materials classed as Poison A (49 CFR 173.328). In the absence of reliable data that exclude the possibility of the presence of Poison A chemicals or compounds (see Attachment 8-10C), these procedures must be followed.

7.5.1 Packaging

Applying the word "poisonous" to a sample does not imply that it is, in fact, poisonous, or how poisonous. It describes the class of packaging according to DOT regulations.

1. Collect samples in a polyethylene or glass container with an outer diameter narrower than the valve hole on a DOT specification #3A1800 or #3AA1800 metal cylinder. To prevent leakage, fill the container no more than 90% full. Seal the sample container.

2. Complete the sample label and identification tag and attach it securely to the sample container.

3. Attach a string or flexible wire to the neck of the sample container and lower it into the metal cylinder partially filled with noncombustible, absorbent cushioning material (for example, diatomaceous earth or vermiculite). Place only one container in the metal cylinder. Pack with enough absorbing material between the bottom and sides of the sample container and the metal cylinder to prevent breakage and absorb any leakage. After the cushioning material is in place, drop the end of the string into the cylinder valve hole.

4. Replace the valve, torque to 250 ft-lb (for 1-inch opening), and replace valve protector on the metal cylinder, using Teflon® tape.

5. Mark and label the cylinder as described in Paragraph 1 of Section 8.5.2.

6. Place one or more of the cylinders in a DOT-approved outside container.

7. Mark and label the outside container and complete all shipping papers as described below.

7.5.2 Marking/Labeling

1. Use abbreviations only where specified. Place the following information, either hand-printed or in label form, on the side of the cylinder or on a tag wired to the cylinder valve protector.

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- "Poisonous Liquid, n.o.s." or "Poisonous Gas, n.o.s. NA9035".
- Laboratory name and address.
- DOT label "Poisonous Gas" (even if sample is liquid) on the cylinder.

2. Put all information contained on the metal cylinder on the outside container. Print "Laboratory Sample" and "Inside Packages Comply With Prescribed Specifications" on the top and/or front of the outside container. Mark "THIS SIDE UP" on top of the container and place upward-pointing arrows on all four sides.

7.5.3 Shipping Papers

1. Use abbreviations only as specified. Complete a carrier-provided bill of lading and sign the certification statement (if the carrier does not provide one, use a standard industry form, see Attachment 8-10D). Provide the following information in the order listed. One form may be used for more than one exterior container.)
   - "Poisonous Liquid, n.o.s. NA9035."
   - "Limited Quantity" (or "Ltd. Qty.").
   - Net weight (wt) or net volume (vol), just before or after "Poisonous Liquid, n.o.s.", of each cylinder, if more than one is inside the outer container.

2. Include a chain-of-custody record, properly executed, in the container or with the cylinder.

3. Accompany the shipping container to the carrier and, if required, open the outside container(s) for inspection.

7.5.4 Transportation

Transport any unknown hazardous substance samples classified as Poison A only by ground transport or government-owned aircraft. Do not use air cargo, other common-carrier aircraft, or rented aircraft.

7.6 Transport of Investigation and Remediation Wastes

The packaging, marking, labeling, and other shipping requirements will depend on the particular waste to be transported. Examples of wastes which may be generated during the site investigations are decontamination or cleaning solutions, contaminated disposable items, test pit spoils, drilling cuttings or fluids and contaminated monitoring well discharges. Waste materials from remediation include excavation spoils, overpacked drums and discharges from drained lagoons or tanks.

In many cases, wastes generated during site investigations will be disposed of on site. These relatively small volumes of waste will be dealt with as part of the waste to be cleaned up or isolated during remediation. This avenue should be pursued, if feasible, to avoid the inconvenience of transportation and disposal which are disproportionately expensive for small volumes. If such a solution is approved,

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materials should be properly bagged, drummed, covered, buried, or otherwise contained at the end of each day. Those materials which must be transported for treatment, storage, or disposal should be packaged, labeled and marked in accordance with applicable regulations.

Many wastes generated during site investigations and remediation activities will probably be adequately handled under the classification "ORM-E" (i.e., other regulated materials, type E). Types of wastes which would normally fall under this classification are contaminated disposable protective clothing and sampling equipment, spent soapy decontamination solutions and rinses, contaminated drilling cuttings or fluids and contaminated soils excavated during site investigations or remediation.

Spent solvents used for decontamination of sampling equipment (e.g., acetone or methanol) should be referenced by the actual product name. Liquids from drums or tanks should be specified as accurately as possible based on results of lab analysis or reliable records. If the liquid is known to be a solvent, organic liquid, or spent distillation bottoms, it should be referenced by its actual or generic name from the Hazardous Materials Table. In cases requiring emergency actions where the identity of a substance is not accurately know, place the substance in one of the general hazard classes in 49 CFR 173.2. The choice of class should be conservative; that is, use the highest priority class based on available information as described in Section 7.3.2.

The following steps for preparing hazardous materials for shipment were extracted from the "Hazardous Materials Transportation Guide for Shippers" published by the U.S. DOT. References are to CFR Title 49:

1. Determine the Proper Shipping Name

   The shipper must determine the proper shipping name of the materials as listed in the Hazardous Materials Table, 172.101, Column (2).

2. Determine the Hazard Class or Classes

   a. Refer to the Table, 172.101, Column (3) and locate the hazard class of the material or follow the steps described in Section 7.3.2 of this Guideline.

   b. If more than one class is shown for the proper shipping name, determine the proper class by definition.

   c. If the material has more than one hazard, classify the material based on the order of hazards in 173.2.

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3. Select the Proper Identification Number

   a. Refer to the Table, 172.101, Column (3a) and select the Identification Number (ID) that corresponds to the proper shipping name and hazard class.

   b. Enter the ID Number(s) on the shipping papers and display them, as required, on packaging or placards.

4. Determine the Mode(s) of Transport to Ultimate Destination*

   a. As a shipper, you must assure yourself that the shipment complies with the various modal requirements.

   b. The modal requirements may affect the following:
      (1) Packaging
      (2) Quantity per package
      (3) Marking
      (4) Labeling
      (5) Shipping papers
      (6) Certification

   * For example, truck, rail or air.

5. Select the proper label(s) and apply as required

   Required labels are based on the hazard class of the substance to be shipped. No placards are required on vehicles transporting ORM-E substances or limited quantities of any hazardous materials (e.g., hazardous samples as discussed in Section 8.4).

   a. Refer to the Table, 172.101, Column (4) for required label(s).

   b. For details in labeling refer to:
      (1) Additional Labels, 172.402
      (2) Location of Labels, 172.406
      (3) Packaging (Mixed or Consolidated), 172.404(a) and (b)
      (4) Packages Containing Samples, 172.402(h)
      (5) Radioactive Materials, 172.403
      (6) Authorized Label Modification, 172.405

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6. Determine and Select the Proper Packaging

   a. Refer to the Table, 172.101, Column (5a) for exceptions and Column (5b) for authorized Packaging. Consider the following when selecting an authorized container: Quantity per package; Cushioning material, if required; Proper closure and reinforcement.

   b. If packaged by a prior shipper, make sure the packaging is correct and in proper condition for transportation.

7. Mark the Packaging (Including Overpacks)

   a. Apply the required marking (172.300); Proper shipping name and number, when required (172.301); Name and address of Consignee and Consignor (172306).

   b. For details and other required markings, see 172.300 through 172.338.

8. Prepare Shipping Papers

   a. The basic requirements for preparing shipping papers include: proper shipping name; hazard class; ID number; total quantity; shipper's certification.

   b. Make all entries on the shipping papers, using the information required, and in proper sequence 172.202.

   c. For additional requirements, see 172.200 through 172.205.

9. Certification

   a. Each shipper must certify, by printing (manually or mechanically) on the shipping papers, that the materials being offered for shipment are properly classified, described, packaged, marked, and labeled, and are in proper conditions for transportation according to the applicable DOT Regulations (172.204).

10. Loading, Blocking, and Bracing

    When loading hazardous materials into the transport vehicle or freight container, each package must be loaded, blocked, and braced in accordance with the requirements for the mode of transport.

    a. If the shipper loads the freight container or transport vehicle, the shipper is responsible for the proper loading, blocking, and bracing of the materials. The packages must be properly labeled as to the right side up and samples must be packed to avoid damage in case of overturning.

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b. If carrier personnel do the loading, the carrier is responsible.

11. Determine the Proper Placard(s)

Each person who offers hazardous materials for transportation must determine that the placarding requirements have been met.

a. For highway, unless the vehicle is already correctly placarded, the shipper must provide the required placard(s) and required identification number(s) (172.506).

b. For rail, if loaded by the shipper, the shipper must placard the rail car if placards are required. (172.508)

c. For air and water shipments, the shipper has the responsibility to apply the proper placards.

12. Hazardous Waste/Hazardous Substance

a. If the material is classed as a hazardous waste or hazardous substance, most of the above steps will be applicable.
b. Pertinent Environmental Protection Agency Regulations are found in the Code of Federal Regulations, Title 40, Part 262.

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7.7 Chain-of-Custody Guidelines

The term "chain-of-custody" refers to procedures which ensure that evidence presented in a court of law is what it is represented to be. The chain-of-custody procedures track the evidence from the time and place it is first obtained to the courtroom. These procedures also provide an auditable trail for the evidence as it is moved and/or passes from the custody of one individual to another. In addition, procedures for consistent and detailed records facilitate the admission of evidence under Rule 803(b) of the Federal Rules of Evidence (P.L. 93-575).

Chain-of-custody procedures, record keeping, and documentation are an important part of the overall management control of the samples. Regulatory agencies must be able to provide the chain of possession and custody of any samples that are offered for evidence, or that form the basis of analytical test results introduced as evidence. Written procedures must be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed.

7.7.1 Sample Identification

The following information shall be written in the sample log book when in-situ measurements or samples for laboratory analysis are collected:

- project code;
- station number;
- location of station;
- date and time of measurement;
- samples used (if any);
- field observations (include date and time);
- level of personnel protection (if required);
- equipment used to make physical measurements and collect samples; and
- calibration data for equipment used.

Measurements and observations shall be recorded using black, waterproof ink

7.7.2 Sample Label

Samples, other than in-situ measurements, are removed and transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often divided into portions, depending upon the analyses to be performed. Each portion is preserved in accordance with the project work plan or proposal. Each sample container is identified by a sample label.

Sample labels are provided by the laboratory or container manufacturer. The sampler fills out the following information on the sample label:

- Project number

- Sample number - The unique sample number identifying this sample

- Date - A six-digit number indicating the month, day, and year of sample collection; e.g., 12/21/95

- Time - A four digit number indicating the 24-hour time of collection (for example: 0954 is 9:54 a.m., and 1629 is 4:29 p.m.)

- Medium - Water, Soil, Sediment, Sludge, Leachate, etc.

- Sample type - Grab or Composite

- Preservation - Type, quantity, and concentration of preservative added

- Analysis - Analyses requested as part of the investigation. Both name and method number must be listed (e.g., BTEX/8020)

- Sampled by - Name of the sampler

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- Lab # - The receiving laboratory assigns the lab # to the sample label (this number is not to be used for on-site analyses)

- Remarks - If for contract lab analysis, include the contract lab case of SAS number, and contract lab sample number from the traffic report, SAS Packing List, or Dioxin Shipment Record. Also, pertinent observations of the sampler (e.g., sequence number for sequential samples).

7.7.3 Chain-of Custody Procedures

After collection, separation, identification, and preservation, the sample is maintained under chain-of-custody procedures until it is in the custody of the analytical laboratory and has been stored or disposed.

Field Custody Procedures:

1. Samples are collected as described in the project work plan or proposal. Care must be taken to record precisely the sample location and to ensure that the sample number on the label exactly matches those numbers on the sample log sheet and the chain-of-custody record.

2. The person undertaking the actual sampling in the field is responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

3. When photographs are taken of the sampling as part of the documentation procedure, the name of the photographer, date, time, site location, and site description are entered sequentially in the site logbook as photos are taken. Once developed, the photographic prints shall be serially numbered, corresponding to the logbook descriptions.

4. Sample labels shall be completed for each sample, using waterproof ink unless prohibited by weather conditions, i.e., a logbook notation would explain that a pencil was used to fill out the sample label because a ballpoint pen would not function in freezing weather.

7.7.4 Transfer of Custody and Shipment

Samples are accompanied by the chain-of-custody record form (Attachment 8-10A). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory. The chain-of-custody record is filled out as follows:

1. Enter header information (project number and name, Contract Lab case No. or SAS No.). For each station number, enter date, time, composite/grab, station location, number of containers, analytical parameters.

2. Sign, date, and enter the time under "Relinquished by" entry.

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3. Make sure that the person receiving the sample signs for the "Received by" entry, or enter the name of the carrier (e.g., UPS, Federal Express) under "Received by". Receiving laboratory will sign "Received for Laboratory by" on the lower line and enter the date and time.

4. Enter the bill-of-lading or Federal Express airbill number under "Remarks or Reason for Change of Custody", if appropriate.

5. Place the original (top, signed copy) of the chain-of-custody record form in the appropriate sample shipping package. Retain a copy with field records.

6. Shipping containers should be secured to ensure samples have not been disturbed during transport by using nylon strapping tape and EPA custody seals. The custody seals should be placed on the containers so that they cannot be opened without breaking the seal.

7. Complete other carrier-required shipping papers.

   The custody record is completed using black waterproof ink. Any corrections are made by drawing a line through and initialing and dating the change, then entering the correct information. Erasures are not permitted.

   Common carriers will usually not accept responsibility for handling chain-of-custody record forms; this necessitates packing the record in the sample container (enclosed with the other documentation is a plastic zip-lock bag). As long as custody forms are sealed inside the sample container and the custody seals are intact, commercial carriers are not required to sign off on the custody form.

   The laboratory representative who accepts the incoming sample shipment signs and dates the chain-of-custody record, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal log books and custody records throughout sample preparation and analysis.

7.7.5 Receipt for Samples Form

Whenever samples are split with a private party or government agency, a separate Receipt for Samples Record Form (see Attachment 8-10F) is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the party or agency shall require the signature of a representative of the appropriate party acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Received by" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time. This form must be completed and a copy given to the owner, operator, or agent-in-charge even if the offer for split samples is declined. The original is retained by the Project Manager.

8.0 Attachments

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8-10A Chain-of-Custody Record Form and Chain-of-Custody Seal

8-10B DOT Hazardous Materials Classification (49 CFR 1732)

8-10C DOT List of Class "A" Poison (49 CFR 172.101) and Hazardous Material Shipping Checklist

8-10D Standard Industry Certification Form

8-10E Sample Label

8-10F Receipt For Samples Form

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STANDARD OPERATING PROCEDURE (SOP) 8-16

CHAIN-OF-CUSTODY PREPARATION

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1.0 Purpose

The purpose of this procedure is to establish a standard procedure for preparation of chain-of-custody forms.

The purpose of document control is to assure that all documents for a specific project are accounted for. Chain-of-custody procedures are an integral part of this document control. The purpose of the chain-of-custody form is to assure that the samples collected in the field are being identified properly and the original integrity of a sample is not destroyed by poor collecting and/or handling techniques.

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2.0 Scope

This procedure defines the procedures to prepare and complete the chain-of-custody form.

3.0 References

- U.S. Environmental Protection Agency, 1979. *Methods for Chemical Analyses of Water and Wastes,* EPA 600/4-79-020.
- National Well Water Association, 1986. *RCRA Ground-Water Monitoring Technical Guidance Document,* NWWA/EPA Series.

4.0 Definitions

None

5.0 Responsibilities

5.1 <u>Project Manager</u>

The Project Manager is responsible for ensuring that field personnel know the chain-of-custody

procedures.

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5.2 Field Associate

The Field Associate is responsible for ensuring the chain-of-custody form is properly prepared.

6.0 Equipment

None

7.0 Procedure

To maintain and document sample possession, chain-of-custody procedures are followed. Using these procedures and documentation gives a reasonable assurance that 1) the sample was handled properly and/or 2) if suspected of being improperly handled, witnesses are available who can disclose the information needed to prove or disprove improper handling. Writing chain-of-custody procedures facilitates the admission of evidence under Rule 803(6) of the Federal Rules of Evidence (P.L. 93-575). Evidentiary foundation is recognized by most courts as fulfilling the requirement of sample identification which shows that the original sample:

1. Was not exchanged with another sample
2. Was not contaminated or tampered with
3. Was the one tested?
4. Was the same sample from which the analytical results are derived?

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7.1 Uses of the Chain of Custody Form

Different situations in which the chain-of-custody is used are the following:

- If the person who sampled is transferring the sample(s) to another person; or
- If the laboratory is transferring the sample(s) to another outside laboratory; or
- If the person who sampled is transferring the sample(s) to the laboratory.

7.2 Chain-of-Custody Preparation Procedure

Instructions for preparation of the TolTest, Inc. chain-of-custody form is as follows:

1. Enter the following information in the appropriate sections:
   - TolTest project number or the client's identification number
   - TolTest division or the client's name
   - Purchase order number (not required for TolTest projects)
   - TolTest project manager or the person responsible for the project

- Telephone number where the project manager can be reached
- Turnaround time required in business days (standard is 10 business days)

2. PRINT the sampler's name.

3. Obtain the sampler's signature.

4. Complete the sample information sections. For the type of sample, use the following abbreviations:
   - A = auger cuttings
   - AR = Air rotary cuttings
   - C = composite
   - G = grab
   - S = split-spoon
   - ST = Shelby tube

5. Specify the analysis required and the method number, if known.

6. Note whether preservatives were added to any sample container.

7. Indicate any remarks concerning the samples.

8. Obtain the signature of the person transferring the samples.

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8.0 Attachments

8-16A TolTest Chain-of-Custody Form

procedures.

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5.2 Field Associate

The Field Associate is responsible for ensuring the chain-of-custody form is properly prepared.

6.0 Equipment

None

7.0 Procedure

To maintain and document sample possession, chain-of-custody procedures are followed. Using these procedures and documentation gives a reasonable assurance that 1) the sample was handled properly and/or 2) if suspected of being improperly handled, witnesses are available who can disclose the information needed to prove or disprove improper handling. Writing chain-of-custody procedures facilitates the admission of evidence under Rule 803(6) of the Federal Rules of Evidence (P.L. 93-575). Evidentiary foundation is recognized by most courts as fulfilling the requirement of sample identification which shows that the original sample:

1. Was not exchanged with another sample
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1. Enter the following information in the appropriate sections:
   - TolTest project number or the client's identification number
   - TolTest division or the client's name
   - Purchase order number (not required for TolTest projects)
   - TolTest project manager or the person responsible for the project

- Telephone number where the project manager can be reached
- Turnaround time required in business days (standard is 10 business days)

2. PRINT the sampler's name.

3. Obtain the sampler's signature.

4. Complete the sample information sections. For the type of sample, use the following abbreviations:
   - A = auger cuttings
   - AR = Air rotary cuttings
   - C = composite
   - G = grab
   - S = split-spoon
   - ST = Shelby tube

5. Specify the analysis required and the method number, if known.

6. Note whether preservatives were added to any sample container.

7. Indicate any remarks concerning the samples.

8. Obtain the signature of the person transferring the samples.

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8.0 Attachments

8-16A TolTest Chain-of-Custody Form

ATTACHMENT 1

APPENDIX D OF DoD QUALITY SYSTEMS MANUAL – ESSENTIAL QUALITY CONTROL REQUIREMENTS

Appendix D – Essential Quality Control Requirements

The quality control protocols specified by the laboratory's method manual (5.4.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals and/or the Laboratory Quality Manual.

All quality control measures shall be assessed and evaluated on an on-going basis and quality control acceptance criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

The requirements from the body of Chapter 5, e.g., 5.9.2, apply to all types of testing. The specific manner in which they are implemented is detailed in each of the sections of this Appendix, i.e., chemical testing, W.E.T. testing, microbiology testing, radiochemical testing and air testing.

D.1 Chemical Testing

D.1.1 Positive and Negative Controls

D.1.1.1 Negative Control – Method Performance

a) Purpose: The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps. The method blank shall be processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure. Procedures shall be in place to determine if a method blank is contaminated. Any affected samples associated with a contaminated method blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

b) Frequency: The method blank shall be analyzed at a minimum of 1 per preparation batch. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

c) Composition: The method blank shall consist of a quality system matrix that is similar to the associated samples and is known to be free of the analytes of interest.

d) Evaluation Criteria and Corrective Action: While the goal is to have no detectable contaminants, each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. The source of contamination shall be investigated and measures taken to minimize or eliminate the problem and affected samples reprocessed or data shall be appropriately qualified if:

    1) The concentration of a targeted analyte in the blank is at or above the reporting limit as established by the test method or by regulation, AND is greater than 1/10 of the amount measured in any sample.

    2) The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.

    3) When a blank is determined to be contaminated, the cause must be investigated and measures taken to minimize or eliminate the problem. Samples associated with a contaminated blank shall be evaluated as to the best corrective action for the samples (e.g., reprocessing or data qualifying codes). In all cases the corrective action must be documented.

Positive and Negative Controls: Evaluation Criteria for Blanks (Requirment)

For DoD samples, the method blank will be considered to be contaminated if:

- The concentration of any target analyte in the blank exceeds 1/2 the reporting limit **and** is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
- The concentration of any common laboratory contaminant exceeds the LOQ; or
- The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

D-1

D.1.1.2 Positive Control – Method Performance

D.1.1.2.1 Laboratory Control Sample (LCS)

a) Purpose: The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria, indicates that the analytical system is "out of control". Any affected samples associated with an out of control LCS shall be reprocessed for re-analysis or the results reported with appropriate data qualifying codes.

b) Frequency: The LCS shall be analyzed at a minimum of 1 per preparation batch. Exceptions would be for those analytes for which no spiking solutions are available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

c) Composition: The LCS is a quality system matrix, known to be free of analytes of interest, spiked with known and verified concentrations of analytes. *Note:* the matrix spike may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. Alternatively the LCS may consist of a media containing known and verified concentrations of analytes or as Certified Reference Material (CRM). All analyte concentrations shall be within the calibration range of the methods. The following shall be used in choosing components for the spike mixtures:

The components to be spiked shall be as specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components the laboratory shall spike per the following:

For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected should be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2-year period.

1) For methods that include 1–10 targets, spike all components;
2) For methods that include 11–20 targets, spike at least 10 or 80%, whichever is greater;
3) For methods with more than 20 targets, spike at least 16 components.

Positive and Negative Controls: LCS Spiking Compounds (Requirement)

- All target analytes must be spiked in the LCS. Target analytes are identified by the client on a project-specific basis. This may require the preparation of multiple LCSs to avoid interferences.
- The concentration of the spiked compounds shall be at the project-specific concentration of concern. If this is not specified, it shall be at or below the midpoint of the calibration curve.

D-2

d) Evaluation Criteria and Corrective Action: The results of the individual batch LCS are calculated in percent recovery or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation.

The individual LCS is compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits or utilize client specified assessment criteria.

Positive and Negative Controls: LCS Control Limits (Requirement)

A laboratory shall establish in-house limits that:

- Are statistically-derived using scientifically valid and documented procedures;
- Meet the limits specified by the project or as stated in the method, if available;
- Are updated on an annual basis, or as stated in the method, and re-established after major changes in the analytical process (e.g., new instrumentation, new analyst);
- Are based on at least 30 data points generated under the same analytical process;
- Do not exclude failed LCS recovery data and statistical outliers from the calculation, unless there is a documented and scientifically valid reason (e.g., bad LCS standard, leaking purging cell);

Control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. Control charts shall be maintained and used to detect trends and prevent out-of-control conditions. Control limits shall be continually monitored for shifts in mean recovery, changes in standard deviation, and development of trends.

D-3

A LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be “out of control” shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.

e) If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.

Positive and Negative Controls: LCS Marginal Exceedance (ME) Limits (Requirement)

The marginal exceedance limit is four (4) standard deviations around the mean.

D-4

The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than 11 analytes.

Positive and Negative Controls: Target Analytes (Requirement)

DoD does not allow any target analyte to exceed its LCS control limits, even marginally. It is inappropriate to control batch acceptance on poor-performing analytes.

D-5

The number of allowable marginal exceedances is as follows:

1) >90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit;
2) 71–90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit;
3) 51–70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit;
4) 31–50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit;
5) 11–30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit;
6) <11 analytes in LCS, no analytes allowed in ME of the LCS control limit;

Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken. Laboratories must have a written procedure to monitor the application of marginal exceedance allowance to the LCS to ensure random behavior.

Positive and Negative Controls: Random Marginal Exceedance (Clarification)

DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior.

D-6

D.1.1.3 Sample Specific Controls

The laboratory must document procedures for determining the effect of the sample matrix on method performance. These procedures relate to the analyses of matrix specific Quality Control (QC) samples and are designed as data quality indicators for a specific sample using the designated test method. These controls alone are not used to judge laboratory performance.

Examples of matrix specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD); sample duplicates; and surrogate spikes. The laboratory shall have procedures in place for tracking, managing, and handling matrix specific QC criteria including spiking appropriate components at appropriate concentrations, calculating recoveries and relative percent difference, evaluating and reporting results based on performance of the QC samples.

D.1.1.3.1 Matrix Spike; Matrix Spike Duplicates

a) Purpose: Matrix specific QC samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these controls is sample/matrix specific and would not normally be used to determine the validity of the entire batch.

b) Frequency: The frequency of the analysis of matrix specific samples shall be determined as part of a systematic planning process (e.g., Data Quality Objectives) or as specified by the test method.

Positive and Negative Controls: MS/MSD Frequency (Requirement)

Each preparation batch of samples must contain an associated MS and MSD (or sample duplicate, see Box D-11) using the same matrix collected for the specific DoD project. The requirements for MS/MSD are not applicable to all methods (e.g.,

asbestos, certain air-testing samples, classic chemistry, and industrial hygiene samples). If adequate sample material is not available, then the lack of MS/MSDs shall be noted in the case narrative. Additional MS/MSDs may be required on a project-specific basis.

D-7

c) Composition: The components to be spiked shall be as specified by the mandated test method. Any permit specified analytes, as specified by regulation or client requested analytes shall also be included. If there are no specified components, the laboratory shall spike per the following:

For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria for choosing the number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2-year period.

1) For methods that include 1–10 targets, spike all components;
2) For methods that include 11–20 targets, spike at least 10 or 80%, whichever is greater;
3) For methods with more than 20 targets, spike at least 16 components.

Positive and Negative Controls: MS/MSD Spiking Compounds (Requirement)

The MS and MSD must be spiked with all target analytes. The concentration of the spiked compounds shall be at or below the midpoint of the calibration range or at the appropriate concentration of concern.

(Guidance) Multiple spiked samples may need to be prepared to avoid interferences.

D-8

d) Evaluation Criteria and Corrective Action: The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R), relative percent difference (RPD), or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation for %R, RPD or other statistical treatment used.

Positive and Negative Control: Calculation of Relative Percent Difference (RPD) (Requirement)

For DoD, relative percent difference (RPD) between original and duplicate analyses must be calculated as follows:

$$RPD = \frac{|C_O - C_D|}{\frac{C_O + C_D}{2}} \times 100\%$$

where C_O and C_D are the concentrations of the original and duplicate, respectively.

D-9

The results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix spike results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

Positive and Negative Controls: MS/MSD Acceptance Criteria (Requirement)

The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the LCS.

D-10

D.1.1.3.2 Matrix Duplicates

a) Purpose: Matrix duplicates are defined as replicate aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the precision of the results for the specific sample using the selected method. The matrix duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.

b) Frequency: The frequency of the analysis of matrix duplicates may be determined as part of a systematic planning process (e.g., Data Quality Objectives) or as specified by the mandated test method.

Positive and Negative Controls: Sample duplicate Frequency (Requirement)

If the known concentration of concern is greater than five times the LOQ, a sample duplicate may be analyzed in place of the MSD. A matrix spike is still required (see Box D-8). Duplicate analysis should be performed at a minimum frequency of once per preparatory batch per matrix type.

D-11

c) Composition: Matrix duplicates are performed on replicate aliquots of actual samples. The composition is usually not known.

d) Evaluation Criteria and Corrective Action: The results from matrix duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD) or another statistical treatment (e.g., absolute differences). The laboratory shall document the calculation for relative percent difference or other statistical treatments.

Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix duplicates results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

D.1.1.3.3 Surrogate Spikes

a) Purpose: Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Added prior to sample preparation/extraction, they provide a measure of recovery for every sample matrix.

b) Frequency: Except where the matrix precludes its use or when not commercially available, surrogate compounds must be added to all samples, standards, and blanks for all appropriate test methods.

c) Composition: Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method or MQO. They are often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of select compounds.

d) Evaluation Criteria and Corrective Action: The results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory should determine internal criteria and document the method used to establish the limits. Surrogates outside the acceptance criteria must be evaluated for the effect indicated for the individual sample results. The appropriate corrective action may be guided by the data quality objectives or other site specific requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria should include appropriate data qualifiers.

Positive and Negative Controls: Surrogate Spike Acceptance Criteria (Requirement)

Surrogate spike results shall be compared with project-specific acceptance criteria specified by the client. If project-specific criteria are not available, the laboratory shall compare the results with its in-house criteria.

D-12

D.1.2 Limit of Detection and Limit of Quantitation

All procedures used must be documented. Documentation must include the quality system matrix type. All supporting data must be retained.

D.1.2.1 Limit of Detection (LOD)

The laboratory shall utilize a test method that provides an LOD that is appropriate and relevant for the intended use of the data. An LOD is not required for a test method when test results are not reported outside of the calibration range. LODs shall be determined by the protocol in the mandated test method or applicable regulation. If the protocol for determining LODs is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

Limit of Detection (LOD): Determination and Verification (Requirement)

A laboratory shall establish a detection limit (DL) using a scientifically valid and documented procedure for each suite of analyte-matrix-method, including surrogates. The detection limit shall be used to determine the LOD for each analyte and matrix as well as for all preparatory and cleanup methods routinely used on samples, as follows:

After each detection limit determination, the laboratory must immediately establish the LOD by spiking a quality system matrix at approximately two to three times the detection limit (for a single-analyte standard) or one to four times the detection limit (for a multi-analyte standard). This spike concentration establishes the LOD. It is specific to each combination of analyte, matrix, method (including sample preparation), and instrument configuration. The LOD must be verified quarterly. The following requirements apply to the initial detection limit/LOD determinations and to the quarterly LOD verifications.

- The apparent signal to noise ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second-column confirmation, or pattern recognition.) For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentrations.
- If a laboratory uses multiple instruments for a given method the LOD must be verified on each.
- If the LOD verification fails, then the laboratory must repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.
- The laboratory shall maintain documentation for all detection limit determinations and LOD verifications.

D-13

a) The LOD shall be initially determined for the compounds of interest in each test method in a quality system matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the LOD must be determined in the quality system matrix of interest (see definition of matrix).

b) LOD must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

c) The laboratory must have established procedures to relate LOD with LOQ.

d) The LOD must be verified annually for each quality system matrix, method and analyte according to the procedure specified in C.3.

D.1.2.2 Limit of Quantitation (LOQ)

a) Any established LOQ must be above the LOD.

b) The LOQ must be verified annually for each quality system matrix, method and analyte according to the procedure specified in C.3. Alternatively, the annual LOQ verification is not required if the LOD is reevaluated or verified according to D.1.2.d above.

Limit of Quantitation (LOQ): Establishment and Verification of LOQ (Requirement)

For DoD projects, the LOQ must be set at the lowest calibration standard or the laboratory must run a LOQ verification sample with each batch. All LOQ verification samples must go through sample preparation. At a minimum the LOQ must be verified quarterly.

The laboratory procedure for establishing the LOQ must empirically demonstrate precision and bias at the LOQ. The LOQ and associated precision and bias must meet client requirements and must be reported. If the method is modified, precision and bias at the new LOQ must be demonstrated and reported.

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D.1.3 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.1.4 Quality of Standards and Reagents

a) The source of standards shall comply with Section 5.6.3.

b) Reagent Quality, Water Quality and Checks:

1) Reagents – In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.

2) Water – The quality of water sources shall be monitored and documented and shall meet requirements.

Quality of Standards and Reagents: Water Quality in Method SOPs (Requirement)

The quality (e.g., purity) specifications for all standards and reagents (including water) shall be documented or referenced in SOPs.

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3) The laboratory will verify the concentration of titrants in accordance with written laboratory procedures.

D.1.5 Selectivity

a) The laboratory shall evaluate selectivity by following the checks established within the method, which may include mass spectral tuning, second column confirmation, ICP inter-element interference checks, chromatography retention time windows, sample blanks, spectrochemical absorption or fluorescence profiles, co-precipitation evaluations, and electrode response factors.

b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

Selectivity: Analyte Confirmation (Requirement)

When reporting data for methods that require analyte confirmation using a secondary column or detector, project-specific reporting requirements shall be followed. If project-specific requirements have not been specified, follow the reporting requirements in the method. If the method does not include reporting requirements, then report the results from the primary column or detector, unless there is a scientifically valid and documented reason for not doing so.

Results that are unconfirmed, or for which confirmation was not performed, shall be identified in the test report, using appropriate data qualifier flags, and explained in the narrative. The laboratory shall use method-specified acceptance criteria for analyte confirmation. If method-specific criteria do not exist, the laboratory shall develop acceptance criteria and document them in the SOP.

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The laboratory shall document acceptance criteria for mass spectral tuning.

D.1.6 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) Glassware Cleaning – Glassware shall be cleaned to meet the sensitivity of the test method.

Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

D.2 Toxicity Testing

These standards apply to laboratories measuring the toxicity and/or bioaccumulation of contaminants in effluents (whole effluent toxicity or WET), receiving waters, sediments, elutriates, leachates and soils. In addition to the essential quality control standards described below, some methods may have additional or other requirements based on factors such as the type of quality system matrix evaluated.

D.2.1 Positive and Negative Controls

a) Positive Control – Reference toxicant tests demonstrate a laboratory's ability to obtain consistent results with the test method and evaluate the overall health and sensitivity of test organisms over time.

1) The laboratory must demonstrate its ability to obtain consistent results with standard reference toxicants (SRT) and complete an initial Demonstration of Capability (DOC) in order to attain accreditation in toxicity testing methods.

i) An initial DOC shall consist of five or more acceptable SRT tests for each test method, species and endpoint with different batches of organisms. Appropriate negative controls (water, sediment, or soil) shall be tested at the frequency and duration specified in the test method. Initial DOCs shall be prepared in accordance with the requirements of Appendix C.

ii) Initial DOC is established by maintenance of SRT test results on control charts. A laboratory shall record the control performance and statistical endpoints (such as NOEC or ECp) for each method species and endpoint on control charts. Initial DOC

is established where 95% of the test results required in D.2.1a)1)i) fall within the control limits established in accordance with D.2.1a)1)iii) and meet test acceptability criteria (TAC). The laboratory shall evaluate precision (i.e., coefficient of variation, CV) or sensitivity (i.e., statistical minimum significant difference, SMSD) measures (see D.2.1a)1)iv)) for these tests against method specific or (lacking the former) laboratory-derived criteria to determine validity of the initial DOC.

iii) For endpoints that are point estimates (ICp, ECp) control charts are constructed by plotting the cumulative mean and the control limits which consist of the upper and lower 95% confidence limits (+/- 2 standard deviations). In case of highly variable point estimates which exceed method-specific criteria the control chart limits are adjusted accordingly. For endpoints from hypothesis tests (NOEC, NOAEC) the values are plotted directly and the control limits consist of one concentration interval above and below the concentration representing the central tendency (i.e., the mode).

iv) For endpoints that are point estimates the cumulative mean CV is calculated and for endpoints from hypothesis tests, the SMSD is calculated. These values are maintained on a control chart.

2) Ongoing laboratory performance shall be demonstrated by routine SRT testing for each test method and species and endpoint in accordance with the minimum frequency requirements specified in D.2.1.a.3.

i) Intralaboratory precision is determined on an ongoing basis through the use of control charts as established in D.2.1.a) 1) ii. The control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC50 for acute tests, or as appropriate hypothesis test values, such as the NOEC or NOAEC, over time within a laboratory.

ii) After initial laboratory DOC is determined, the control limits and CV for an individual test method endpoints, and species shall be adjusted as additional test results are obtained. After 20 data points are collected for a test method and species, the control chart is maintained using only the last 20 data points, i.e., each successive mean value and control limit is calculated using only the last 20 values.

iii) Control chart limits are expected to be exceeded occasionally regardless of how well a laboratory performs. Acceptance limits for point estimates (ICp, ECp) which are based on 95% confidence limits should theoretically be exceeded for one in twenty tests. Depending on the dilution factor and test sensitivity, control charts based on hypothesis test values (NOEC, NOAEC) may be expected to be exceeded on a similar frequency. Test results which fall outside of control chart limits at a frequency of 5% or less, or which fall just outside control chart limits (especially in the case of highly proficient laboratories which may develop relatively narrow acceptance limits over time), are not rejected de facto. Such data are evaluated in comparison with control chart characteristics including the width of the acceptance limits and the degree of departure of the value from acceptance limits.

iv) Laboratories shall develop acceptance/rejection policies, consistent with the test methods, for SRT data which considers source of test organisms, the direction of deviation, test dilution factor, test sensitivity (for hypothesis test values), testing frequency, out-of-control test frequency, relative width of acceptance limits, inter-test CV, and degree of difference between test results and acceptance limits.

v) In the case of reference toxicant data which fails to meet control chart acceptance criteria, the test data are examined for defects, corrective action taken, and the test repeated if necessary, using a different batch of organisms or the data is qualified.

3) The frequency of ongoing laboratory reference toxicant testing shall be as follows unless the method specifically requires less frequent SRT tests (e.g., sediment tests):
    i) For test methods conducted at a frequency of monthly or greater, SRT tests shall be conducted at an ongoing frequency of monthly.
    ii) For test methods and species commonly used in the laboratory, but which are tested at a frequency of less than monthly, SRT tests shall be conducted concurrently with the environmental test.
    iii) If the test organisms are obtained from an outside source the sensitivity of each batch of organisms received from a supplier shall be determined via a concurrent SRT test unless the supplier can provide control chart data for the last five SRT tests using the same SRT and test conditions. Supplied SRT data may not be older than six months.
    iv) The DOC for an analyst shall be consistent with 5.2.6.c)3) but the frequency need not exceed the method specified requirements and D.2.1.a)3).
4) These standards do not currently specify a particular reference toxicant and dilution series however, if the state or permitting authority identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements. All reference toxicant tests conducted for a given test method and species must use the same reference toxicant, test concentrations, dilution water and data analysis methods. A dilution factor of 0.5x or greater shall be used for both acute and chronic tests.
5) The reference toxicant tests shall be conducted following the same procedures as the environmental toxicity tests for which the precision is being evaluated, unless otherwise specified in the test method (for example, 10-day sediment tests employ 96-h water-only reference toxicant tests). The test duration, laboratory dilution water, feeding, organism age, range and density, test volumes, renewal frequency, water quality measurements, and the number of test concentrations, replicates and organisms per replicate shall be the same as specified for the environmental toxicity test.

b) Negative Control – Control, Brine Control, Control Sediment, Control Soil or Dilution Water
    1) The standards for the use, type and frequency of testing of negative controls are specified by the test methods and by permit or regulation and shall be followed. A negative control is included with each test to evaluate test performance and the health and sensitivity of the specific batch of organisms.
    2) Appropriate additional negative controls shall be included when sample adjustments (for example addition of thiosulfate for dechlorination) or solvent carriers are used in the test.
    3) Test Acceptability Criteria (TAC) – The test acceptability criteria specified in the test method must be achieved for both the reference toxicant and the effluent or environmental sample toxicity test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity tests.

D.2.2 Variability and/or Reproducibility

Intralaboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described in item D.2.1.a above.

D.2.3 Accuracy

This principle is not applicable to Toxicity Testing.

D.2.4 Test Sensitivity

a) The SMSD shall be calculated according to the formula specified by the test method and reported with the test results.

b) Point estimates: (LCp, ICp, or ECp) – Confidence intervals shall be reported as a measure of the precision around the point estimate value, when the calculation is possible.

c) The SMSD shall be calculated and reported for only hypothesis test values, such as the NOEC or NOAEC.

D.2.5 Selection of Appropriate Statistical Analysis Methods

a) If required, methods of data analysis and endpoints are specified by language in the regulation, permit or the test method.

b) Dose Response Curves – The data shall be plotted in the form of a curve relating the dose of the chemical or concentration of sample to cumulative percentage of test organisms demonstrating a response such as death. Evaluation criteria shall be established for interpretation of concentration or dose response curves.

D.2.5.2 Selection and Use of Reagents and Standards

a) The grade of all reagents used in toxicity tests is specified in the test method except the reference standard. All reference standards shall be prepared from chemicals which are analytical reagent grade or better. The preparation of all standards and reference toxicants shall be documented.

b) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH or specific conductance, shall comply with the standards outlined in Section 5.6.3.

c) Only reagent-grade water collected from distillation or deionization units is used to prepare reagents.

D.2.6 Selectivity

This principle is not applicable. The selectivity of the test is specified by permit or regulation.

D.2.7 Constant and Consistent Test Conditions

a) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid cross-contamination.

b) Laboratory space must be adequate for the types and numbers of tests performed. The building must provide adequate cooling, heating and illumination for conducting testing and culturing; hot and cold running water must be available for cleaning equipment.

c) Air used for aeration of test solutions, dilution waters and cultures must be free of oil and fumes.

d) The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s)) and the names(s) of the taxonomic expert(s) must be kept on file at the laboratory. When organisms are obtained from an outside source the supplier must provide this same information.

e) Instruments used for routine support measurements of chemical and physical parameters such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, ammonia and weight shall be calibrated, and/or standardized per manufacturer's instructions. As these are support measurements, only the calibration and verification requirements specified at 5.5.2.1 apply. All measurements and calibrations shall be documented.

f) Test temperature shall be maintained as specified for the test method. Temperature control equipment must be adequate to maintain the required test temperature(s). The average daily temperature of the test solutions must be maintained within the method specified range. The minimum frequency of measurement shall be once per 24 hour period. The test temperature for continuous-flow toxicity tests shall be recorded and monitored continuously. Where electronic data loggers are used, temperature shall be monitored at a frequency sufficient to capture temporal variations of the environmental control system.

g) Reagent grade water, prepared by any combination of distillation, reverse osmosis, ion exchange, activated carbon and particle filtration, shall meet the method specified requirements.

h) The quality of the standard dilution water used for testing or culturing must be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. Water used for culturing and testing shall be analyzed for toxic metals and organics whenever the minimum acceptability criteria for control survival, growth or reproduction are not met and no other cause, such as contaminated glassware or poor stock, can be identified. It is recognized that the analyte lists of some methods manuals may not include all potential toxicants, are based on estimates of chemical toxicity available at the time of publication and may specify detection limits which are not achievable in all matrices. However, for those analytes not listed, or for which the measured concentration or limit of detection is greater than the method-specified limit, the laboratory must demonstrate that the analyte at the measured concentration or reported limit of detection does not exceed one tenth the expected chronic value for the most sensitive species tested and/or cultured. The expected chronic value is based on professional judgment and the best available scientific data. The "U.S. EPA Ambient Water Quality Criteria Documents" and the EPA AQUIRE database provide guidance and data on acceptability and toxicity of individual metals and organic compounds.

i) The quality of the food used for testing or culturing must be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. The laboratory shall have written procedures for the evaluation of food acceptance.

j) A subset of organisms used in bioaccumulation tests must be analyzed at the start of the test (baseline) for the target compounds to be measured in the bioaccumulation tests.

k) Test chamber size and test solution volume shall be as specified in the test method. All test chambers used in a test must be identical.

l) Test organisms shall be fed the quantity and type food or nutrients specified in the test method. They shall also be fed at the intervals specified in the test methods.

m) All organisms in a test must be from the same source. Where available certified seeds are used for soil tests.

n) All organisms used in tests, or used as broodstock to produce neonate test organisms (for example cladocerans and larval fish), must appear healthy, show no signs of stress or disease and exhibit acceptable survival (90% or greater) during the 24 hour period immediately preceding use in tests.

o) All materials used for test chambers, culture tanks, tubing, etc. and coming in contact with test samples, solutions, control water, sediment or soil or food must be non-toxic and cleaned as described in the test methods. Materials must not reduce or add to sample toxicity. Appropriate materials for use in toxicity testing and culturing are described in the referenced manuals.

p) Light intensity shall be maintained as specified in the methods manuals. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the test methods and shall be documented at least quarterly. For algal and plant tests, the light intensity shall be measured and recorded at the start of each test.

q) The health and culturing conditions of all organisms used for testing shall be documented by the testing laboratory. Such documentation shall include culture conditions (e.g., salinity, hardness, temperature, pH) and observations of any stress, disease or mortality. When organisms are obtained from an outside source, the laboratory shall obtain written documentation of these water quality parameters and biological observations for each lot of organism received. These observations shall adequately address the 24 hour time period referenced in item D.2.8.n. above. The laboratory shall also record each of these observations and water quality parameters upon the arrival of the organisms at the testing laboratory.

r) Age and the age range of the test organisms must be as specified in the test method. Supporting information, such as hatch dates and times, times of brood releases and metrics (for example, chironomid head capsule width) shall be documented.

s) The maximum holding time of effluents (elapsed time from sample collection to first use in a test) shall not exceed 36 hours; samples may be used for renewal up to 72 hours after first use except as prescribed by and approved by the regulatory agency having authority for program oversight.

t) All samples shall be chilled to 0 to 6°C during or immediately after collection (see requirements in section 5.8.3.1) except as prescribed by the method and approved by the regulatory agency having authority for program oversight.

u) Organisms used in a given test must be from the same batch.

v) All tests shall have the minimum number of replicates per treatment as prescribed by the method.

w) The control population of *Ceriodaphnia* in chronic effluent or receiving water tests shall contain no more than 20% males.

x) The culturing of *C. dubia* shall be adequate such that blocking by parentage can be established.

y) Dissolved oxygen and pH in aquatic tests shall be within acceptable range at test initiation and aeration (minimal) is provided to tests if, and only if, acceptable dissolved oxygen concentrations cannot be otherwise maintained or if specified by the test method.

z) Test soils or sediments must be within the geochemical tolerance range of the test organism.

aa) An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests (see test conditions and test acceptability criteria specified for each test method). The acceptability of the test shall depend on the experience and professional judgment of the technical director and the permitting authority.

D.3 Microbiology Testing

These standards apply to laboratories undertaking microbiological analysis of environmental samples. Microbiological testing refers to and includes the detection, isolation, enumeration, or identification of microorganisms and/or their metabolites, or determination of the presence or absence of growth in materials and media.

D.3.1 Sterility Checks and Blanks, Positive and Negative Controls

a) Sterility Checks and Blanks

The laboratory shall demonstrate that the filtration equipment and filters, sample containers, media and reagents have not been contaminated through improper handling or preparation, inadequate sterilization, or environmental exposure.

1) A sterility blank shall be analyzed for each lot of pre-prepared, ready-to-use medium (including chromofluorogenic reagent) and for each batch of medium prepared in the laboratory. This shall be done prior to first use of the medium.

2) For filtration technique, the laboratory shall conduct one beginning and one ending sterility check for each laboratory sterilized filtration unit used in a filtration series. The filtration series may include single or multiple filtration units, which have been sterilized prior to beginning the series. For pre-sterilized single use funnels a sterility check shall be performed on one funnel per lot. The filtration series is considered ended when more than 30 minutes elapses between successive filtrations. During a filtration series, filter funnels must be rinsed with three 20-30 ml portions of sterile rinse water after each sample filtration. In addition, laboratories must insert a sterility blank after every 10 samples or sanitize filtration units by UV light after each sample filtration.

3) For pour plate technique, sterility blanks of the medium shall be made by pouring, at a minimum, one uninoculated plate for each lot of pre-prepared, ready-to-use media and for each batch of medium prepared in the laboratory.

4) Sterility checks on sample containers shall be performed on at least one container for each lot of purchased, pre-sterilized containers. For containers prepared and sterilized in the

laboratory, a sterility check shall be performed on one container per sterilized batch with non-selective growth media.

5) A sterility blank shall be performed on each batch of dilution water prepared in the laboratory and on each batch of pre-prepared, ready-to-use dilution water with non-selective growth media.
6) At least one filter from each new lot of membrane filters shall be checked for sterility with non-selective growth media.

b) Positive Controls

Positive culture controls demonstrate that the medium can support the growth of the target organism(s), and that the medium produces the specified or expected reaction to the target organism(s).

1) Each pre-prepared, ready-to-use lot of medium (including chromofluorogenic reagent) and each batch of medium prepared in the laboratory shall be tested with at least one pure culture of a known positive reaction. This shall be done prior to first use of the medium.

c) Negative Controls

Negative culture controls demonstrate that the medium does not support the growth of non-target organisms or does not demonstrate the typical positive reaction of the target organism(s).

Each pre-prepared, ready-to-use lot of selective medium (including chromofluorogenic reagent) and each batch of selective medium prepared in the laboratory shall be analyzed with one or more known negative culture controls, i.e., non-target organisms, as appropriate to the method. This shall be done prior to first use of the medium.

D.3.2 Test Variability/Reproducibility

For test methods that specify colony counts such as membrane filter or plated media, duplicate counts shall be performed monthly on one positive sample, for each month that the test is performed. If the lab has two or more analysts, each analyst shall count typical colonies on the same plate. Counts must be within 10% difference to be acceptable. In a laboratory with only one microbiology analyst, the same plate shall be counted twice by the analyst, with no more than 5% difference between the counts.

D.3.3 Method Evaluation

a) Laboratories are required to demonstrate proficiency with the test method prior to first use. This shall be achieved by comparison to a method already approved for use in the laboratory, or by analyzing a minimum of ten spiked samples whose quality system matrix is representative of those normally submitted to the laboratory, or by analyzing and passing one proficiency test series provided by an approved proficiency sample provider. The laboratory shall maintain this documentation as long as the method is in use and for at least 5 years past the date of last use.
b) Laboratories shall participate in the Proficiency Test programs identified by NELAP (4.1.5.k or 5.9.1). The results of these analyses shall be used to evaluate the ability of the laboratory to produce acceptable data.

D.3.4 Test Performance

a) All growth and recovery media must be checked to assure that the target organism(s) respond in an acceptable and predictable manner (see D.3.1.b).
b) To ensure that analysis results are accurate, target organism identity shall be verified as specified in the method, e.g., by use of the completed test, or by use of secondary verification tests such as a catalase test.

D.3.5 Data Reduction

The calculations, data reduction and statistical interpretations specified by each test method shall be followed.

D.3.6 Quality of Standards, Reagents and Media

The laboratory shall ensure that the quality of the reagents and media used is appropriate for the test concerned.

a) Culture media may be prepared from commercial dehydrated powders or may be purchased ready to use. Media may be prepared by the laboratory from basic ingredients when commercial media are not available or when it can be demonstrated that commercial media do not provide adequate results. Media prepared by the laboratory from basic ingredients must be tested for performance (e.g., for selectivity, sensitivity, sterility, growth promotion, growth inhibition) prior to first use. Detailed testing criteria information must be defined in either the laboratory's test methods, SOPs, Quality Manual, or similar documentation.

b) Reagents, commercial dehydrated powders and media shall be used within the shelf-life of the product and shall be documented according to 5.6.4.

c) Distilled water, deionized water or reverse-osmosis produced water free from bactericidal and inhibitory substances shall be used in the preparation of media, solutions and buffers. The quality of the water shall be monitored for chlorine residual, specific conductance, and heterotrophic bacteria plate count monthly (when in use), when maintenance is performed on the water treatment system, or at startup after a period of disuse longer than one month.

d) Analysis for metals and the Bacteriological Water Quality Test (to determine presence of toxic agents or growth promoting substances) shall be performed annually. Results of these analyses shall meet the specifications of the required method and records of analyses shall be maintained for five years. (An exception to performing the Bacteriological Water Quality Test shall be given to laboratories that can supply documentation to show that their water source meets the criteria, as specified by the method, for Type I or Type II reagent water.)

e) Media, solutions and reagents shall be prepared, used and stored according to a documented procedure following the manufacturer's instructions or the test method. Documentation for media prepared in the laboratory shall include date of preparation, preparer's initials, type and amount of media prepared, manufacturer and lot number, final pH of the media, and expiration date. Documentation for media purchased pre-prepared, ready-to-use shall include manufacturer, lot number, type and amount of media received, date of receipt, expiration date of the media, and pH of the media.

D.3.7 Selectivity

a) In order to ensure identity and traceability, reference cultures used for positive and negative controls shall be obtained from a recognized national collection, organization, or manufacturer recognized by the NELAP Accrediting Authority. Microorganisms may be single use preparations or cultures maintained by documented procedures that demonstrate the continued purity and viability of the organism.

    1) Reference cultures may be revived (if freeze-dried) or transferred from slants and subcultured once to provide reference stocks. The reference stocks shall be preserved by a technique which maintains the characteristics of the strains. Reference stocks shall be used to prepare working stocks for routine work. If reference stocks have been thawed, they must not be re-frozen and re-used.

    2) Working stocks shall not be sequentially cultured more than five times and shall not be subcultured to replace reference stocks.

D.3.8 Constant and Consistent Test Conditions

a) Laboratory Facilities

Floors and work surfaces shall be non-absorbent and easy to clean and disinfect. Work surfaces shall be adequately sealed. Laboratories shall provide sufficient storage space, and shall be clean and free from dust accumulation. Plants, food, and drink shall be prohibited from the laboratory work area.

b) Laboratory Equipment

1) Temperature Measuring Devices

Temperature measuring devices such as liquid-in-glass thermometers, thermocouples, and platinum resistance thermometers used in incubators, autoclaves and other equipment shall be the appropriate quality to meet specification(s) in the test method. The graduation of the temperature measuring devices must be appropriate for the required accuracy of measurement and they shall be calibrated to national or international standards for temperature (see 5.6.2.2.2) [see 5.6.2.2]. Calibration shall be done at least annually.

2) Autoclaves

i) The performance of each autoclave shall be initially evaluated by establishing its functional properties and performance, for example heat distribution characteristics with respect to typical uses. Autoclaves shall meet specified temperature tolerances. Pressure cookers shall not be used for sterilization of growth media.

ii) Demonstration of sterilization temperature shall be provided by use of continuous temperature recording device or by use of a maximum registering thermometer with every cycle. Appropriate biological indicators shall be used once per month to determine effective sterilization. Temperature sensitive tape shall be used with the contents of each autoclave run to indicate that the autoclave contents have been processed.

iii) Records of autoclave operations shall be maintained for every cycle. Records shall include: date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time (may be recorded as time in and time out) and analyst's initials.

iv) Autoclave maintenance, either internally or by service contract, shall be performed annually and shall include a pressure check and calibration of temperature device. Records of the maintenance shall be maintained in equipment logs.

v) The autoclave mechanical timing device shall be checked quarterly against a stopwatch and the actual time elapsed documented.

3) Volumetric Equipment

Volumetric equipment shall be calibrated as follows:

i) equipment with movable parts such as automatic dispensers, dispensers/diluters, and mechanical hand pipettes shall be verified for accuracy quarterly.

ii) equipment such as filter funnels, bottles, non-class A glassware, and other marked containers shall be calibrated once per lot prior to first use.

iii) the volume of the disposable volumetric equipment such as sample bottles, disposable pipettes, and micropipette tips shall be checked once per lot.

4) UV Instruments

UV instruments, used for sanitization, shall be tested quarterly for effectiveness with an appropriate UV light meter or by plate count agar spread plates. Replace bulbs if output is less than 70% of original for light tests or if count reduction is less than 99% for a plate containing 200 to 300 organisms.

5) Conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments shall be calibrated according to the method specified requirements (see Section 5.5.2.1.d).
6) Incubators, Water Baths, Ovens
    i) The stability and uniformity of temperature distribution and time required after test sample addition to re-establish equilibrium conditions in incubators and water baths shall be established. Temperature of incubators and water baths shall be documented twice daily, at least four hours apart, on each day of use.
    ii) Ovens used for sterilization shall be checked for sterilization effectiveness monthly with appropriate biological indicators. Records shall be maintained for each cycle that include date, cycle time, temperature, contents and analyst's initials.
7) Labware (Glassware and Plasticware)
    i) The laboratory shall have a documented procedure for washing labware, if applicable. Detergents designed for laboratory use must be used.
    ii) Glassware shall be made of borosilicate or other non-corrosive material, free of chips and cracks, and shall have readable measurement marks.
    iii) Labware that is washed and reused shall be tested for possible presence of residues which may inhibit or promote growth of microorganisms by performing the Inhibitory Residue Test annually, and each time the lab changes the lot of detergent or washing procedures.
    iv) Washed labware shall be tested at least once daily, each day of washing, for possible acid or alkaline residue by testing at least one piece of labware with a suitable pH indicator such as bromothymol blue. Records of tests shall be maintained.

D.4 Radiochemical Testing

These standards apply to laboratories undertaking the examination of environmental samples by radiochemical analysis. These procedures for radiochemical analysis may involve some form of chemical separation followed by detection of the radioactive decay of analyte (or indicative daughters) and tracer isotopes where used. For the purpose of these standards procedures for the determination of radioactive isotopes by mass spectrometry (e.g., ICP-MS or TIMS) or optical (e.g., KPA) techniques are not addressed herein.

D.4.1 Negative and Positive Controls

a) Negative Controls
    1) Method Blank – Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The method blank result shall be assessed against the specific acceptance criteria [see 5.4.1.2.b)18] specified in the laboratory method manual [see 5.4.1.2]. When the specified method blank acceptance criteria is not met the specified corrective action and contingencies [see 5.4.1.2.b) 19 and 20] shall be followed and results reported with appropriate data qualifying codes. The occurrence of a failed method blank acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a].
    2) In the case of gamma spectrometry, generally a non-destructive analysis, a method blank shall be prepared using a calibrated counting geometry similar to that used for the samples. The container of the appropriate geometry can be empty or filled to similar volume to partially simulate gamma attenuation due to a sample matrix.
    3) There shall be no subtraction of the required method blank [see D.4.1.a)1] result from the sample results in the associated preparation or analytical batch unless permitted by method or program. This does not preclude the application of any correction factor (e.g.,

instrument background, analyte presence in tracer, reagent impurities, peak overlap, etc.) to all analyzed samples, both program/project submitted and internal quality control samples. However, these correction factors shall not depend on the required method blank result in the associated analytical batch.

4) The method blank sample shall be prepared with similar aliquot size to that of the routine samples for analysis and the method blank result and acceptance criteria [5.4.1.2.b)18] shall be calculated in a manner that compensates for sample results based upon differing aliquot size.

b) Positive Controls

1) Laboratory Control Samples – Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The laboratory control sample result shall be assessed against the specific acceptance criteria [see 5.4.1.2.b)18] specified in the laboratory method manual [see 5.4.1.2]. When the specified laboratory control sample acceptance criteria is not met the specified corrective action and contingencies [see 5.4.1.2.b)19 and 20] shall be followed. The occurrence of a failed laboratory control sample acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a].

2) Matrix Spike – Shall be performed at a frequency of one per preparation batch for those methods which include a chemical separation process without the use of an internal standard or carrier, and where there is sufficient sample to do so. Although gross alpha, gross beta and tritium measurements do not involve a chemical separation process, matrix spikes shall be performed for these analyses on aqueous samples. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The matrix spike result shall be assessed against the specific acceptance criteria [see 5.4.1.2.b)18] specified in the laboratory method manual [see 5.4.1.2]. When the specified matrix spike acceptance criteria is not met, the specified corrective action and contingencies [see 5.4.1.2.b)19 and 20] shall be followed. The occurrence of a failed matrix spike acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a]. The lack of sufficient sample aliquot size to perform a matrix spike shall be noted in the laboratory report.

3) The activity of the laboratory control sample shall: (1) be at least 5 times the limit of detection and (2) at a level comparable to that of routine samples when such information is available if the sample activities are expected to exceed 5 times the limit of detection.

4) The activity of the matrix spike analytes(s) shall be greater than 5 times the limit of detection.

5) The laboratory standards used to prepare the laboratory control sample and matrix spike shall be from a source independent of the laboratory standards used for instrument calibration and must meet the requirements for reference standards provided in D.4.7.a).

6) The matrix spike shall be prepared by adding a known activity of target analyte after subsampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.). Where a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope (e.g., plutonium, Pu 238 and Pu 239, using alpha spectrometry), only one of the analyte isotopes need be included in the laboratory control or matrix spike sample at the indicated activity level. However, where more than one analyte isotope is present above the specified limit of detection each shall be assessed against the specified acceptance criteria.

7) Where gamma spectrometry is used to identify and quantitate more than one analyte isotope the laboratory control sample and matrix spike shall contain isotopes that represent the low (e.g., americium-241), medium (e.g., cesium-137) and high (e.g., cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples the isotopes need

not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

8) The laboratory control sample shall be prepared with similar aliquot size to that of the routine samples for analyses.

c) Other Controls

1) Tracer – For those methods that utilize a tracer (i.e., internal standard) each sample result shall have an associated tracer recovery calculated and reported. The tracer shall be added to the sample after subsampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method. The tracer recovery for each sample result shall be one of the quality control measures to be used to assess the associated sample result acceptance. The tracer recovery shall be assessed against the specific acceptance criteria [see 5.4.1.2.b) 18] specified in the laboratory method manual [see 5.4.1.2]. When the specified tracer recovery acceptance criteria is not met the specified corrective action and contingencies [see 5.4.1.2.b) 19 and 20] shall be followed. The occurrence of a failed tracer recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a].

2) Carrier – For those methods that utilize a carrier for recovery determination, each sample shall have an associated carrier recovery calculated and reported. The carrier shall be added to the sample after subsampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method. The carrier recovery for each sample shall be one of the quality control measures to be used to assess the associated sample result acceptance. The carrier recovery shall be assessed against the specific acceptance criteria [see 5.4.1.2.b) 18] specified in the laboratory method manual [see 5.4.1.2]. When the specified carrier recovery acceptance criteria is not met the specified corrective action and contingencies [see 5.4.1.2.b) 19 and 20] shall be followed. The occurrence of a failed carrier recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a].

D.4.2 Analytical Variability/Reproducibility

a) Replicate – Shall be performed at a frequency of one per preparation batch where there is sufficient sample to do so. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The replicate result shall be assessed against the specific acceptance criteria [see 5.4.1.2.b) 18] specified in the laboratory method manual [see 5.4.1.2]. When the specified replicate acceptance criteria is not met the specified corrective action and contingencies [see 5.4.1.2.b) 19 and 20] shall be followed. The occurrence of a failed replicate acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a].

b) For low level samples (less than approximately three times the limit of detection) the laboratory may analyze duplicate laboratory control samples or a replicate matrix spike (matrix spike and a matrix spike duplicate) to determine reproducibility within a preparation batch.

D.4.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Initial Demonstration of Capability – (section 5.4.2.2 and Appendix C) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type (e.g., different detection technique), personnel or method.

b) Proficiency Test Samples – The results of such analysis (4.1.5.k and 5.9.1) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.4.4 Radiation Measurement Instrumentation

Because of the stability and response nature of modern radiation measurement instrumentation, it is not typically necessary to verify calibrate of these systems each day of use. However, verification of calibration is required as outlined in (b) below. This section addresses those practices that are necessary for proper calibration and those requirements of section 5.5.2.2 (Instrument Calibrations) that are not applicable to some types of radiation measurement instrumentation.

a) Instrument Calibration

   1) Given that activity detection efficiency is independent of sample activity at all but extreme activity levels, the requirements of subsections f, h and i of 5.5.2.2.1 are not applicable to radiochemical method calibrations except mass attenuation in gas-proportional counting and sample quench in liquid scintillation counting. Radiation measurement instruments are subject to calibration prior to initial use, when the instrument is placed back in service after malfunctioning and the instrument's response has changed as determined by a performance check or when the instrument's response exceeds predetermined acceptance criteria for the instrument quality control.
   2) Instrument calibration shall be performed with reference standards as defined in section D.4.7a. The standards shall have the same general characteristics (i.e., geometry, homogeneity, density, etc.) as the associated samples.
   3) The frequency of calibration shall be addressed in the laboratory method manual [see 5.4.1.2.b)13] if not specified in the method. A specific frequency (e.g., monthly) or observations from the associated control or tolerance chart, as the basis for calibration shall be specified.

b) Continuing Instrument Calibration Verification (Performance Checks)

   Performance checks shall be performed using appropriate check sources and monitored with control charts or tolerance charts to ensure that the instrument is operating properly and that the detector response has not significantly changed and therefore the instrument calibration has not changed. The same check source used in the preparation of the tolerance chart or control chart at the time of calibration shall be used in the calibration verification of the instrument. The check sources must provide adequate counting statistics for a relatively short count time and the source should be sealed or encapsulated to prevent loss of activity and contamination of the instrument and laboratory personnel.

   1) For gamma spectroscopy systems, the performance checks for efficiency and energy calibration shall be performed on a day of use basis along with performance checks on peak resolution.
   2) For alpha spectroscopy systems, the performance check for energy calibration shall be performed on a weekly basis and the performance check for counting efficiency shall be performed on at least a monthly basis.
   3) For gas-proportional and liquid scintillation counters, the performance check for counting efficiency shall be performed on a day of use basis. For batches of samples that uninterruptedly count for more than a day a performance check can be performed at the beginning and end of the batch as long as this time interval is no greater than one week. Verification of instrument calibration does not directly verify secondary calibrations, e.g., the mass efficiency curve or the quench curve.
   4) For scintillation counters the calibration verification for counting efficiency shall be performed on a day of use basis.

c) Background Measurement

   Background measurements shall be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory maintains its capability to meet required data quality

objectives. These values may be subtracted from the total measured activity in the determination of the sample activity.

1) For gamma spectroscopy systems, background measurements shall be performed on at least a monthly basis.
2) For alpha spectroscopy systems, background measurements shall be performed on at least a monthly basis.
3) For gas-proportional counters background measurements shall be performed at least on a weekly basis.
4) For scintillation counters, background measurements shall be performed each day of use.

d) Instrument Contamination Monitoring

The laboratory shall have a written procedure for monitoring radiation measurement instrumentation for radioactive contamination. The procedure shall indicate the frequency of the monitoring and shall indicate criteria, which initiates corrective action.

D.4.5 Minimum Detectable Activity (MDA)/Minimum Detectable Concentration (MDC)/Lower Level of Detection (LLD)

a) Must be determined prior to sample analysis and must be redetermined each time there is a significant change in the test method or instrument type.

b) The procedures employed must be documented and consistent with mandated method or regulation.

D.4.6 Data Reduction

a) Refer to Section 5.4.7.2, "Computers and Electronic Data Related Requirements," of this document.

b) Measurement Uncertainties – Each result shall be reported with the associated measurement uncertainty. The procedures for determining the measurement uncertainty must be documented and be consistent with mandated method and regulation.

D.4.7 Quality of Standards and Reagents

a) The quality control program shall establish and maintain provisions for radionuclide standards.

1) Reference standards that are used in a radiochemical laboratory shall be obtained from the National Institute of Standards and Technology (NIST), or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.
2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22 - 1995, Section 8, Certificates.
3) Laboratories should consult with the supplier if the lab's verification of the activity of the reference traceable standard indicates a noticeable deviation from the certified value. The laboratory shall not use a value other than the decay corrected certified value. The laboratory shall have a written procedure for handling, storing and establishment of expiration dates for reference standards.

b) All reagents used shall be analytical reagent grade or better.

D.4.8 Constant and Consistent Test Conditions

The laboratory shall maintain a radiological control program that addresses analytical radiological control. The program shall address the procedures for segregating samples with potentially widely varying levels of radioactivity. The radiological control program shall explicitly define how low level and

high level samples will be identified, segregated and processed in order to prevent sample cross-contamination. The radiological control program shall include the measures taken to monitor and evaluate background activity or contamination on an ongoing basis.

D.5 Air Testing

These standards shall apply to samples that are submitted to a laboratory for the purpose of analysis. They do not apply to field activities such as source air emission measurements or the use of continuous analysis devices.

D.5.1 Negative and Positive Controls

a) Negative Controls
   1) Method Blanks – Shall be performed at a frequency of at least one (1) per batch of twenty (20) environmental samples or less per sample preparation method. The results of the method blank analysis shall be used to evaluate the contribution of the laboratory provided sampling media and analytical sample preparation procedures to the amount of analyte found in each sample. If the method blank result is greater than the limit of quantitation and contributes greater than 10% of the total amount of analyte found in the sample, the source of the contamination must be investigated and measures taken to eliminate the source of contamination. If contamination is found, the data shall be qualified in the report.
   2) Collection Efficiency – Sampling trains consisting of multiple sections (e.g., filters, sorbent tubes, impingers) that are received intact by the laboratory, shall be separated into “front” and “back” sections if required by the client. Each section shall be processed and analyzed separately and the analytical results reported separately.

b) Positive Controls
   1) Laboratory Control Sample (LCS) – Shall be analyzed at a rate of at least one (1) per batch of twenty (20) or fewer samples per sample preparation method for each analyte. If a spiking solution is not available, a calibration solution, whose concentration approximates that of the samples, shall be included in each batch and with each lot of media. If a calibration solution must be used for the LCS, the client will be notified prior to the start of analysis. The concentration of the LCS shall be relevant to the intended use of the data and either at a regulatory limit or below it.

c) Surrogates – Shall be used as required by the test method or if requested by the client.

d) Matrix spike – Shall be used as required by the test method, or if requested by the client.

D.5.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates – Shall be analyzed at a minimum of 1 in 20 samples per sample batch. The laboratory shall document their procedure to select the use of appropriate types of spikes and duplicates. The selected samples(s) shall be rotated among client samples so that various sample matrix problems may be noted and/or addressed. Poor performance in the spikes and duplicates may indicate a problem with the sample composition and shall be reported to the client.

D.5.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Capability – (Sections 5.2.6 and 5.4.2.2) shall be performed prior to the analysis of any samples and with a significant change in instrument type, personnel, quality system matrix, or test method.

b) Calibration – Calibration protocols specified in Section 5.5.2 shall be followed.

c) Proficiency Test Samples – The results of such analyses (4.1.5.k or 5.9.1) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.5.4 Limit of Detection

The requirements of D.1.2.1 shall apply.

D.5.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.5.6 Quality of Standards and Reagents

a) The source of standards shall comply with Section 5.6.3.
b) The purity of each analyte standard and each reagent shall be documented by the laboratory through certificates of analyses from the manufacturer/vendor, manufacturer/vendor specifications, and/or independent analysis.
c) In methods where the purity of reagents is not specified, analytical reagent grade or higher quality, if available, shall be used.

D.5.7 Selectivity

The laboratory shall develop and document acceptance criteria for test method selectivity such as absolute and relative retention times, wavelength assignments, mass spectral library quality of match, and mass spectral tuning.

D.5.8 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.
b) The laboratory shall document that all sampling equipment, containers and media used or supplied by the laboratory meet required test method criteria.
c) If supplied or used by the laboratory, procedures for field equipment decontamination shall be developed and their use documented.
d) The laboratory shall have a documented program for the calibration and verification of sampling equipment such as pumps, meter boxes, critical orifices, flow measurement devices and continuous analyzers, if these equipment are used or supplied by the laboratory.

D.6 Asbestos Testing

These standards apply to laboratories undertaking the examination of asbestos samples. These standards are organized by analytical technique including transmission electron microscopy (TEM) for the analysis of water, wastewater, air, and bulk samples; phase contrast microscopy (PCM) for analysis of workplace air; and polarized light microscopy (PLM) for analysis of bulk samples. These procedures for asbestos analysis involve sample preparation followed by detection of asbestos. If NIST SRMs specified below are unavailable, the laboratory may substitute an equivalent reference material with a certificate of analysis.

D.6.1 Negative Controls

D.6.1.1 Transmission Electron Microscopy

D.6.1.1.1 Water and Wastewater

a) Blank determinations shall be made prior to sample collection. When using polyethylene bottles, one bottle from each batch, or a minimum of one from each 24 shall be tested for background level. When using glass bottles, four bottles from each 24 shall be tested. An acceptable bottle blank level is defined as ≤ 0.01 MFL > 10 µm. (EPA /600/R-94/134, Method 100.2, Section 8.2)
b) A process blank sample consisting of fiber-free water shall be run before the first field sample. The quantity of water shall be ≥ 10 mL for a 25-mm diameter filter and ≥ 50 mL for a 47-mm diameter filter. (EPA /600/R-94/134, Method 100.2, Section 11.8)

D.6.1.1.2 Air

a) A blank filter shall be prepared with each set of samples. A blank filter shall be left uncovered during preparation of the sample set and a wedge from that blank filter shall be prepared alongside wedges from the sample filters. At minimum, the blank filter shall be analyzed for each 20 samples analyzed. (40 CFR Part 763, Appendix A to Subpart E (AHERA), Table 1)

b) Maximum contamination on a single blank filter shall be no more than 53 structures/mm2. Maximum average contamination for all blank filters shall be no more than 18 structures/mm2. (AHERA, III.F.2)

D.6.1.1.3 Bulk Samples

a) Contamination checks using asbestos-free material, such as the glass fiber blank in SRM 1866 (Page C-3, NIST Handbook 150-3, August 1994) shall be performed at a frequency of 1 for every 20 samples analyzed. The detection of asbestos at a concentration exceeding 0.1% will require an investigation to detect and remove the source of the asbestos contamination.

b) The laboratory must maintain a list of non-asbestos fibers that can be confused with asbestos (Section 7.5, Page C-8, NIST Handbook 150-3, August 1994). The list must include crystallographic and/or chemical properties that disqualify each fiber being identified as asbestos (Section 2.5.5.2.1 Identification, Page 54, EPA/600/R-93/116).

c) The laboratory should have a set of reference asbestos materials from which a set of reference diffraction and X-ray spectra have been developed.

D.6.1.2 Phase Contrast Microscopy

At least two (2) field blanks (or 10% of the total samples, whichever is greater) shall be submitted for analysis with each set of samples. Field blanks shall be handled in a manner representative of actual handling of associated samples in the set with a single exception that air shall not be drawn through the blank sample. A blank cassette shall be opened for approximately thirty (30) seconds at the same time other cassettes are opened just prior to analysis. Results from field blank samples shall be used in the calculation to determine final airborne fiber concentration. The identity of blank filters should be unknown to the counter until all counts have been completed. If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

D.6.1.3 Polarized Light Microscopy

a) Friable Materials – At least one blank slide must be prepared daily or with every 50 samples analyzed, whichever is less. This is prepared by mounting a subsample of an isotropic verified non-ACM (e.g., fiberglass in SRM 1866) in a drop of immersion oil (nD should reflect usage of various nDs) on a clean slide, rubbing preparation tools (forceps, dissecting needles, etc.) in the mount and placing a clean coverslip on the drop. The entire area under the coverslip must be scanned to detect any asbestos contamination. A similar check must be made after every 20 uses of each piece of homogenization equipment. An isotropic verified non-ACM must be homogenized in the clean equipment, a slide prepared with the material and the slide scanned for asbestos contamination. (This can be substituted for the blank slide mentioned in this section.)

b) Non-Friable Materials – At least one non-ACM non-friable material must be prepared and analyzed with every 20 samples analyzed. This non-ACM must go through the full preparation and analysis regimen for the type of analysis being performed.

D.6.2 Test Variability/Reproducibility

D.6.2.1 Transmission Electron Microscopy

Quality assurance analyses shall be performed regularly covering all time periods, instruments, tasks, and personnel. The selection of samples shall be random and samples of special interest may be included in the selection of samples for quality assurance analyses. When possible, the checks on personnel performance shall be executed without their prior knowledge. A disproportionate number of analyses shall not be performed prior to internal or external audits. It is recommended that a

laboratory initially be at 100% quality control (all samples reanalyzed). The proportion of quality control samples can later be lowered gradually, as control indicates, to a minimum of 10%.

D.6.2.1.1 Water and Wastewater

All analyses must be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings. Quality assurance analyses shall not be postponed during periods of heavy workloads. The total number of QA samples and blanks must be greater than or equal to 10% of the total sample workload. Precision of analyses is related to concentration, as gleaned from interlaboratory proficiency testing. Relative standard deviations (RSD) for amphibole asbestos decreased from 50% at 0.8 MFL to 25% at 7 MFL in interlaboratory proficiency testing, while RSD for chrysotile was higher, 50% at 6 MFL.

a) Replicate – A second, independent analysis shall be performed on the same grids but on different grid openings than used in the original analysis of a sample. Results shall be within 1.5X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (EPA /600/R-94/134, Method 100.2, Table 2)
b) Duplicate – A second aliquot of sample shall be filtered through a second filter, prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (EPA /600/R-94/134, Method 100.2, Table 2)
c) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings used in the original analysis of a sample. The two sets of results shall be compared according to Turner and Steel (NISTIR 5351). This shall be performed at a frequency of 1 per 20 samples. Qualified analysts must maintain an average of ≥ 80% true positives, ≤ 20% false negatives, and ≤ 10% false positives.

D.6.2.1.2 Air

All analyses must be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings.

The laboratory and TEM analysts must obtain mean analytical results on NIST SRM 1876b so that trimmed mean values fall within 80% of the lower limit and 110% of the upper limit of the 95% confidence limits as published on the certificate. These limits are derived from the allowable false positives and false negatives given in Section D.6.2.1.2c, Verified Analysis, below. SRM 1876b shall be analyzed a minimum of once per year by each TEM analyst.

The laboratory must have documentation demonstrating that TEM analysts correctly classify at least 90% of both bundles and single fibrils of asbestos structures greater than or equal to 1 µm in length in known standard materials traceable to NIST, such as NIST bulk asbestos SRM 1866.

Interlaboratory analyses shall be performed to detect laboratory bias. The frequency of interlaboratory verified analysis must correspond to a minimum of 1 per 200 grid square analyses for clients.

If more than 1 TEM is used for asbestos analysis, intermicroscope analyses must be performed to detect instrument bias.

a) Replicate – A second, independent analysis shall be performed in accordance with Section D.6.2.1.1.a. (AHERA, Table III)
b) Duplicate – A second wedge from a sample filter shall be prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (AHERA, Table III)
c) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings in accordance with Section D.6.2.1.1.c. (AHERA, Table III)

D.6.2.1.3 Bulk Samples

Determination of precision and accuracy should follow guidelines in NISTIR 5951, Guide for Quality Control on the Qualitative and Quantitative Analysis of Bulk Asbestos Samples: Version 1. Because bulk samples with low (<10%) asbestos content are the most problematic, a laboratory's quality control program should focus on such samples. At least 30% of a laboratory's QC analyses shall be performed on samples containing from 1% to 10% asbestos.

a) Intra-Analyst Precision – At least 1 out of 50 samples must be reanalyzed by the same analyst. For single analyst laboratories, at least 1 out of every 10 samples must be reanalyzed by the same analyst.
b) Inter-Analyst Precision – At least 1 out of 15 samples must be reanalyzed by another analyst. Inter-analyst results will require additional reanalysis, possibly including another analyst, to resolve discrepancies when classification (ACM vs.non-ACM) errors occur, when asbestos identification errors occur, or when inter-analyst precision is found to be unacceptable.
c) Inter-Laboratory Precision – The laboratory must participate in round robin testing with at least one other laboratory. Samples must be sent to this other lab at least four times per year. These samples must be samples previously analyzed as QC samples. Results of these analyses must be assessed in accordance with QC requirements. As a minimum, the QC requirements must address misclassifications (false positives, false negatives) and misidentification of asbestos types.

D.6.2.2 Phase Contrast Microscopy

a) Inter-Laboratory Precision – Each laboratory analyzing air samples for compliance determination shall implement an inter-laboratory quality assurance program that as a minimum includes participation of at least two (2) other independent laboratories. Each laboratory shall participate in round robin testing at least once every six (6) months with at least all the other laboratories in its inter-laboratory quality assurance group. Each laboratory shall submit slides typical of its own workload for use in this program. The round robin shall be designed and results analyzed using appropriate statistical methodology. Results of this QA program shall be posted in each laboratory to keep the microscopists informed.
b) Intra- and Inter-Analyst Precision – Each analyst shall select and count a prepared slide from a "reference slide library" on each day on which air counts are performed. Reference slides shall be prepared using well-behaved samples taken from the laboratory workload. Fiber densities shall cover the entire range routinely analyzed by the laboratory. These slides shall be counted by all analysts to establish an original standard deviation and corresponding limits of acceptability. Results from the daily reference sample analysis shall be compared to the statistically derived acceptance limits using a control chart or a database. It is recommended that the labels on the reference slides be periodically changed so that the analysts do not become familiar with the samples. Intra- and inter-analyst precision may be estimated from blind recounts on reference samples. Inter-analyst precision shall be posted in each laboratory to keep the microscopists informed.

D.6.2.3 Polarized Light Microscopy

Refer to Section D.6.2.1.3.

D.6.3 Other Quality Control Measures

D.6.3.1 Transmission Electron Microscopy

D.6.3.1.1 Water and Wastewater

a) Filter preparations shall be made from all six asbestos types from NIST SRMs 1866 and 1867. These preparations shall have concentrations between 1 and 20 structures (> 10 µm) per 0.01 mm2. One of these preparations shall be analyzed independently at a frequency of 1 per 100 samples analyzed. Results shall be evaluated as verified asbestos analysis in accordance with Turner and Steel (NISTIR 5351).

b) NIST SRM 1876b must be analyzed annually by each analyst. Results shall be evaluated in accordance with limits published for that SRM. Comment: This SRM is not strictly appropriate for waterborne asbestos but analysts can demonstrate general TEM asbestos competence by producing results within the published limits of this (the only recognized TEM counting standard) SRM.

D.6.3.1.2 Air

Filter preparations shall be made from all six asbestos types in accordance with Section D.6.3.1.1.a.

NIST SRM 1876b must be analyzed annually in accordance with Section D.6.3.1.1.b.

D.6.3.1.3 Bulk Samples

All analysts must be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite). Standards for the six asbestos types listed are available from NIST (SRMs 1866 and 1867). These materials can also be used as identification standards for AEM (Section 3.2.1 Qualitative Analysis, Page 57, EPA/600/R-93/116).

D.6.3.2 Phase Contrast Microscopy

a) Test for Non-Random Fiber Distribution – Blind recounts by the same analyst shall be performed on 10% of the filters counted. A person other than the counter should re-label slides before the second count. A test for type II error (NIOSH 7400, Issue 2, 15 August 1994, Section 13) shall be performed to determine whether a pair of counts by the same analyst on the same slide should be rejected due to non-random fiber distribution. If a pair of counts is rejected by this test, the remaining samples in the set shall be recounted and the new counts shall be tested against first counts. All rejected paired counts shall be discarded. It shall not be necessary to use this statistic on blank recounts.

b) All individuals performing airborne fiber analysis must have taken the NIOSH Fiber Counting Course for sampling and evaluating airborne asbestos dust or an equivalent course.

c) All laboratories shall participate in a national sample testing scheme such as the Proficiency Analytical Testing (PAT) program or the Asbestos Analysts Registry (AAR) program, both sponsored by the American Industrial Hygiene Association (AIHA), or equivalent.

D.6.3.3 Polarized Light Microscopy

a) Friable Materials – Because accuracy cannot be determined by reanalysis of routine field samples, at least 1 out of 100 samples must be a standard or reference sample that has been routinely resubmitted to determine analyst's precision and accuracy. A set of these samples should be accumulated from proficiency testing samples with predetermined weight compositions or from standards generated with weighed quantities of asbestos and other bulk materials (Perkins and Harvey, 1993; Parekh et al., 1992; Webber et al., 1982). At least half of the reference samples submitted for this QC must contain between 1 and 10% asbestos.

b) Non-Friable Materials – At least 1 out of 100 samples must be a verified quantitative standard that has routinely been resubmitted to determine analyst precision and accuracy.

D.6.4 Method Evaluation

In order to ensure the accuracy of reported results, the following procedures shall be in place:

a) Demonstration of Capability – (Refer to Sections 5.2.6 and 5.4.2.2) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, or method.

b) Performance Audits – (Refer to Sections 4.1.5.k and 5.9.1) The results of such analyses shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.6.5 Asbestos Calibration

Refer to methods referenced in the following sections for specific equipment requirements.

D.6.5.1 Transmission Electron Microscopy

AEM (Analytical Electron Microscopy) equipment requirements will not be discussed in this document.

D.6.5.1.1 Water and Wastewater

All calibrations listed below (unless otherwise noted) must be performed under the same analytical conditions used for routine asbestos analysis and must be recorded in a notebook and include date and analyst's signature. Frequencies stated below may be reduced to "before next use" if no samples are analyzed after the last calibration period has expired. Likewise, frequencies may have to be increased following non-routine maintenance or unacceptable calibration performance.

a) Magnification Calibration – Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 10,000 and 20,000x. A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification. Calibration data must be displayed on control charts that show trends over time. (EPA /600/R-94/134, Method 100.2, Section 10.1)

b) Camera Constant – The camera length of the TEM in the Selected Area Electron Diffraction (SAED) mode must be calibrated before SAED patterns of unknown samples are observed. The diffraction specimen must be at the eucentric position for this calibration. This calibration shall allow accurate (< 10% variation) measurement of layer-line spacings on the medium used for routine measurement, i.e., the phosphor screen or camera film. This must also allow accurate (< 5% variation) measurement of zone axis SAED patterns on permanent media, e.g., film. Calibrations shall be performed monthly to establish the stability of the camera constant (EPA /600/R-94/134, Method 100.2, Section 10.2). Where non-asbestiform minerals may be expected (e.g., winchite, richterite, industrial talc, vermiculite, etc.), an internal camera constant standard such as gold, shall be deposited and measured on each sample to facilitate accurate indexing of zone axis SAED patterns. In such cases, layer line analysis alone shall not be used. Calibration data must be displayed on control charts that show trends over time.

c) Spot Size – The diameter of the smallest beam spot at crossover must be less than 250 nm as calibrated quarterly. Calibration data must be displayed on control charts that show trends over time. (EPA /600/R-94/134, Method 100.2, Section 10.3)

d) Beam Dose – The beam dose shall be calibrated so that beam damage to chrysotile is minimized, specifically so that an electron diffraction pattern from a single fibril ≥ 1 µm in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least 15 seconds.

e) EDXA System

   1) The x-ray energy vs. channel number for the EDXA system shall be calibrated to within 20 eV for at least two peaks between 0.7 keV and 10 keV. One peak shall be from the low end (0.7 keV to 2 keV) and the other peak from the high end (7 keV to 10 keV) of this range. The calibration of the x-ray energy shall be checked prior to each analysis of samples and recalibrated if out of the specified range.

   2) The ability of the system to resolve the Na Kα line from the Cu L line shall be confirmed quarterly by obtaining a spectrum from the NIST SRM 1866 crocidolite sample on a copper grid.

   3) The k-factors for elements found in asbestos (Na, Mg, Al, Si, Ca, and Fe) relative to Si shall be calibrated semiannually, or anytime the detector geometry may be altered. NIST SRM 2063a shall be used for Mg, Si, Ca, Fe, while k-factors for Na and Al may be obtained from suitable materials such as albite, kaersutite, or NIST SRM 99a. The k-factors shall be determined to a precision (2s) within 10% relative to the mean value obtained for Mg, Al, Si, Ca, and Fe, and within 20% relative to the mean value obtained for Na. The k-factor relative to Si for Na shall be between 1.0 and 4.0, for Mg and Fe shall be between 1.0 and 2.0, and

for Al and Ca shall be between 1.0 and 1.75. The k-factor for Mg relative to Fe shall be 1.5 or less. Calibration data must be displayed on control charts that show trends over time.

4) The detector resolution shall be checked quarterly to ensure a full-width half-maximum resolution of < 175 eV at Mn Kα (5.90 keV). Calibration data must be displayed on control charts that show trends over time.
5) The portions of a grid in a specimen holder for which abnormal x-ray spectra are generated under routine asbestos analysis conditions shall be determined and these areas shall be avoided in asbestos analysis.
6) The sensitivity of the detector for collecting x-rays from small volumes shall be documented quarterly by collecting resolvable Mg and Si peaks from a unit fibril of NIST SRM 1866 chrysotile.

f) Low Temperature Asher – The low temperature asher shall be calibrated quarterly by determining a calibration curve for the weight vs. ashing time of collapsed mixed-cellulose-ester (MCE) filters. Calibration data must be displayed on control charts that show trends over time.
g) Grid Openings – The magnification of the grid opening measurement system shall be calibrated using an appropriate standard at a frequency of 20 openings/20 grids/lot of 1000 or 1 opening/sample. The variation in the calibration measurements (2s) is <5% of the mean calibration value.

D.6.5.1.2 Air

All calibrations must be performed in accordance with Section D.6.5.1.1, with the exception of magnification. Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 15,000 to 20,000x (AHERA, III.G.1.c). A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification.

D.6.5.1.3 Bulk Samples

All calibrations must be performed in accordance with Section D.6.5.1.2.

D.6.5.2 Phase Contrast Microscopy

a) At least once daily, the analyst shall use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric.
b) The phase-shift limit of detection of the microscope shall be checked monthly or after modification or relocation using an HSE/NPL phase-contrast test slide for each analyst/microscope combination (refer to NIOSH 7400, Issue 2, 15 August 1994, Section 10b). This procedure assures that the minimum detectable fiber diameter (< ca. 0.25 µm) for this microscope is achieved.
c) Prior to ordering the Walton-Beckett graticule, calibration, in accordance with NIOSH 7400, Issue 2, 15 August 1994, Appendix A, shall be performed to obtain a counting area 100 µm in diameter at the image plane. The diameter, dc (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule. The field diameter (D) shall be verified (or checked), to a tolerance of 100 µm ± 2 µm, with a stage micrometer upon receipt of the graticule from the manufacturer. When changes (zoom adjustment, disassembly, replacement, etc.) occur in the eyepiece-objective-reticle combination, field diameter must be re-measured (or re-calibrated) to determine field area (mm2). Re-calibration of field diameter shall also be required when there is a change in interpupillary distance (i.e., change in analyst). Acceptable range for field area shall be 0.00754 mm2 to 0.00817 mm2. The actual field area shall be documented and used.

D.6.5.3 Polarized Light Microscopy

a) Microscope Alignment – To accurately measure the required optical properties, a properly aligned polarized light microscope (PLM) shall be utilized. The PLM shall be aligned before each use. (Section 2.2.5.2.3, EPA/600/R-93/116, July 1993)

b) Refractive Index Liquids – Series of nD = 1.49 through 1.72 in intervals less than or equal to 0.005. Refractive index liquids for dispersion staining, high-dispersion series 1.550, 1.605, 1.680. The accurate measurement of the refractive index (RI) of a substance requires the use of calibrated refractive index liquids. These liquids shall be calibrated at first use and semiannually, or next use, whichever is less frequent, to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or RI glass beads.

D.6.6 Analytical Sensitivity

D.6.6.1 Transmission Electron Microscopy

D.6.6.1.1 Water and Wastewater

An analytical sensitivity of 200,000 fibers per liter (0.2 MFL) is required for each sample analyzed (EPA /600/R-94/134, Method 100.2, Section 1.6). Analytical sensitivity is defined as the waterborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the fraction of the filter sampled and the dilution factor (if applicable).

D.6.6.1.2 Air

An analytical sensitivity of 0.005 structures/cm^2 is required for each sample analyzed. Analytical sensitivity is defined as the airborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the effective surface area of the filter, the filter area analyzed, and the volume of air sampled (AHERA, Table I).

D.6.6.1.3 Bulk Samples

a) The range is dependent on the type of bulk material being analyzed. The sensitivity may be as low as 0.0001% depending on the extent to which interfering materials can be removed during the preparation of AEM specimens. (Section 2.5.2 Range, Page 51, EPA/600/R-93/116)

b) There should be an error rate of less than 1% on the qualitative analysis for samples that contain chrysotile, amosite, and crocidolite. A slightly higher error rate may occur for samples that contain anthophyllite, actinolite, and tremolite, as it can be difficult to distinguish among the three types. (Section 3, Page 10, NIST Handbook 150-3, August 1994)

D.6.6.2 Phase Contrast Microscopy

The normal quantitative working range of the test method is 0.04 to 0.5 fiber/cm^2 for a 1000 L air sample. An ideal counting range on the filter shall be 100 to 1300 fibers/mm^2. The limit of detection (LOD) is estimated to be 5.5 fibers per 100 fields or 7 fibers/mm^2. The LOD in fiber/cm^2 will depend on sample volume and quantity of interfering dust but shall be <0.01 fiber/cm^2 for atmospheres free of interferences. (NIOSH 7400, Issue 2, 15 August 1994)

D.6.6.3 Polarized Light Microscopy

The laboratory shall utilize a test method that provides a limit of detection that is appropriate and relevant for the intended use of the data. Limit of detection shall be determined by the protocol in the test method or applicable regulation.

D.6.7 Data Reduction

D.6.7.1 Transmission Electron Microscopy

D.6.7.1.1 Water and Wastewater

a) The concentration of asbestos in a given sample must be calculated in accordance with EPA /600/R-94/134, Method 100.2, Section 12.1. Refer to Section 5.4.7.2, “Computers and Electronic Data Related Requirements”, of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample (EPA /600/R-94/134, Method 100.2, Section 12.2.2).

D.6.7.1.2 Air

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized, e.g., AHERA. Refer to Section 5.4.7.2, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

D.6.7.1.3 Bulk Samples

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993). Refer to Section 5.4.7.2, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – Proficiency testing for floor tiles analyzed by TEM following careful gravimetric reduction (New York ELAP Certification Manual Item 198.4) has revealed an interlaboratory standard deviation of approximately 20% for residues containing 70% or more asbestos. Standard deviations range from 20% to 60% for residues with lower asbestos content.

D.6.7.2 Phase Contrast Microscopy

a) Airborne fiber concentration in a given sample must be calculated in accordance with NIOSH 7400, Issue 2, 15 August 1994, Sections 20 and 21. Refer to Section 5.4.7.2, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the intra-laboratory and inter-laboratory relative standard deviation with each set of results. (NIOSH 7400, Issue 2, 15 August 1994)

c) Fiber counts above 1300 fibers/mm2 and fiber counts from samples with > 50% of the filter area covered with particulate should be reported as "uncountable" or "probably biased". Other fiber counts outside the 100–1300 fibers/mm2 range should be reported as having "greater than optimal variability" and as being "probably biased".

D.6.7.3 Polarized Light Microscopy

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993). Refer to Section 5.4.7.2, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Method Uncertainties – Precision and accuracy must be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonable expanded errors (refer to EPA/600/R-93/116, July 1993, Table 2-1) should be generated for different concentrations of asbestos.

D.6.8 Quality of Standards and Reagents

D.6.8.1 Transmission Electron Microscopy

a) The quality control program shall establish and maintain provisions for asbestos standards.

   1) Reference standards that are used in an asbestos laboratory shall be obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers who participate in supplying NIST standards or NIST traceable asbestos. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.

   2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22-1995, Section 8, Certificates.

b) All reagents used shall be analytical reagent grade or better.

c) The laboratory shall have mineral fibers or data from mineral fibers that will allow differentiating asbestos from at least the following "look-alikes": fibrous talc, sepiolite, wollastonite, attapulgite

(palygorskite), halloysite, vermiculite scrolls, antigorite, lizardite, pyroxenes, hornblende, richterite, winchite, or any other asbestiform minerals that are suspected as being present in the sample.

D.6.8.2 Phase Contrast Microscopy

Standards of known concentration have not been developed for this testing method. Routine workload samples that have been statistically validated and national proficiency testing samples such as PAT and AAR samples available from the AIHA may be utilized as reference samples (refer to Section D.6.2.2b) to standardize the optical system and analyst. All other testing reagents and devices (HSE/NPL test slide and Walton-Beckett Graticule) shall conform to the specifications of the method (refer to NIOSH 7400, Issue 2, 15 August 1994).

D.6.8.3 Polarized Light Microscopy

Refer to Section D.6.8.1.

D.6.9 Constant and Consistent Test Conditions

The laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.