DEPARTMENT OF THE NAVY
CRANE DIVISION
NAVAL SURFACE WARFARE CENTER
300 HIGHWAY 361
CRANE, INDIANA 47522-5000

IN REPLY REFER TO

5090
Ser 095/8111
04 JUN 1998

U.S. Environmental Protection Agency, Region V
Waste, Pesticides, & Toxics Division
Waste Management Branch
Illinois, Indiana, and Michigan Section
ATTN: Ms. Carol Witt-Smith (DRP-8J)
77 West Jackson Blvd.
Chicago, IL 60604

Dear Ms. Witt-Smith:

Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) submits the dye water analysis results as enclosure (1). This enclosure reports the analysis conducted on water generated at the Dye Burial Grounds, Solid Waste Management Unit 02/11, currently stored in on-site tanks. Enclosure (2) contains the required certification statement.

NAVSURFWARCENDIV Crane point of contact is Mr. Thomas J. Brent, Code 09510, telephone 812-854-6160.

Sincerely,

Thomas J. Brent, for
JAMES M. HUNSICKER

Encl:
(1) Results of Dye Water Analysis
(2) Certification Statement

Copy to:
ADMINISTRATIVE RECORD (w/2 copies)
COMNAVSEASYSCOM (SEA OOT) (w/o encl)
SOUTHNAVFACENGCOM (Code 1864) (w/o encl)
CODE 4052

DEPARTMENT OF THE NAVY
CRANE DIVISION
NAVAL SURFACE WARFARE CENTER
300 HIGHWAY 361
CRANE, INDIANA 47522-5001

IN REPLY REFER TO

8000/5
Ser 4052/8088
04 JUN [illegible]

MEMORANDUM

From: 4052
To: 095 (T. Brent)

Subj: **ANALYSIS REPORT OF DYES FROM THE CRANE DYE BURIAL GROUNDS, OED/C-98-045**

Ref: (a) QUALITY ASSURANCE PROJECT PLANT (QAPP)
(b) Equipment Operating Procedures of Explosive Sciences Branch

Encl: (1) Results of Dye Water Analysis OED/C-98-045

1. Code 4052 was requested by Code 095 to perform the analysis on nine tanks containing dye water from the Dye Burial Grounds (DGB) at Crane. The samples were assigned the Explosive Sciences Branch Sample Numbers N98-2001 to N98-2075.

2. The samples from the tanks were subjected to the analytical tests described in reference (a). The procedures defined in reference (b) were used as guidelines. A full detailed analysis complete with data sheets is attached as enclosure (1).

3. Code 40 points of contact are Mr. Maroof Qurashi and Mr. Steve Weddle, Code 4052, extensions 4230 and 6049 respectively.

Nancey J. Maegerlein
NANCEY J. MAEGERLEIN

Copy to:
40211

Enclosure (1)

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

Thomas J. Brent, acting

SIGNATURE

Environmental Protection Specialist

TITLE

04 JUN 1998

DATE

Enclosure (2)

DEPARTMENT OF THE NAVY
CRANE DIVISION
NAVAL SURFACE WARFARE CENTER
300 HIGHWAY 361
CRANE, INDIANA 47522-5001

IN REPLY REFER TO

8000/5
Ser 4052/8088
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MEMORANDUM

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Nancey J. Maegerlein
NANCEY J. MAEGERLEIN

Copy to:
40211

RESULTS OF DYE WATER ANALYSIS
FOR
CODE 095

02 June 1998

by

Maroof H. Qurashi
Steven C. Weddle

OED/C 98-045

Approved by:

Nancey J. Maegerlein

Nancey J. Maegerlein
Explosive Sciences Branch
Test and Evaluation Department
Ordnance Engineering Directorate
Naval Surface Warfare Center
Crane Division
Crane, Indiana 47522-5001

Enclosure (1)

Ref: (a) QUALITY ASSURANCE PROJECT PLAN (QAPP)
(b) Equipment Operating Procedures of Explosive Sciences Branch

Appendix A: Data Tables

1. BACKGROUND

The Dye Burial Grounds (DBG), Solid Waste Management Unit (SWMU) 02/11, was the site of disposal of scrap, dyes, and dye contaminated materials from 1940s until 1964. In 1989, the U.S. EPA issued the federal portion of the Final Resource Conservation and Recovery Act (RCRA) permit for Crane Division, Naval Surface Warfare Center, hereafter referred to as NSWC Crane. In 1994, Morrison Knudsen Corporation (MK) was contracted by the Southern Division, Naval Facilities Engineering Command to construct a "RCRA-type" cap at the DBG. This action was part of a voluntary interim measure consistent with the NSWC Crane RCRA permit. During site preparation for the cap in December 1996, a large storm event produced a significant volume of runoff, which was visibly contaminated with a green dye. Approximately 80,000 gallons of contaminated runoff were pumped into twelve (orginally six) temporary storage tanks at the DBG. Since then, another approximately 70,000 gallons of contaminated water has been containerized bringing the total containerized water volume to 150,000 gallons.

The present report is part of a phased investigation to determine the nature and extent of migration of dye related contaminants at the DBG, and to establish the appropriate cleanup actions. The analytical work is done according to the Quality Assurance Project Plan (QAPP), reference (a).

2. INTRODUCTION

Twenty-three (23) bottles (one gallon each), two bottles (2 liter each), 42 bottles (500 milliliters each) and 41 bottles (40 milliliters each) of samples were collected from nine holding tanks and submitted to the Crane Explosive Sciences Branch (ESB) Laboratory in Building 2707. The samples were assigned lab numbers and logged into the Laboratory data system. Initially, nine hundred milliliters from each preliminary dye water sample was evaporated using nitrogen purge at room temperature. The evaporite was subjected to Thin Layer Chromatography (TLC) to determine what dyes were present. The Explosive Sciences Laboratory has samples of dyes dating back over 40 years. These dyes were used in production at Crane. We found that the green dye in the water samples is Disodium fluorescein (Uranine). The blue dye components match with the blue dye described in Mil-D-16627 (BuOrd) used for projectile spotting. The following series of analytical tests were performed on the tank water samples:

a. Toxicity data analysis using Microtox.
b. Explosives analysis for Octogen (HMX), Cyclonite (RDX), 2,4,6-Trinitrotoluene (TNT), Nitroglycerin (NG), Nitroguanidine

(NQ), 2-Amino-4,6-dinitro toluene (2-Am-DNT), 1,3-Dinitrobenzene (1,3-DNB), 2,4-Dinitrotoluene (2,4-DNT), Nitrobenzene (NB), 1,3,5-Trinitrobenzene (1,3,5-TNB), 4-Amino-2,6-Dinitrotoluene (4-Am-DNT), 2,6-Dinitrotoluene (2,6-DNT), 2-Nitrotoluene (2-NT), 3-Nitrotoluene (3-NT), 4-Nitrotoluene (4-NT), and Tetryl using High Pressure Liquid Chromatography (HPLC) EPA Method 8330.

c. Analysis for Disodium Fluorescein(Uranine) using HPLC.

d. Total Suspended Solids (TSS) using EPA Method 160.2.

e. Anions analysis using Ion Exchange Chromatography (IEC).

f. Ammonia-Nitrogen analysis determined. potentiometrically using an Ion Selective Electrode per EPA Method 350.3.

g. Total Organic Carbon (TOC) analyzed using Dohrmann DC-80 TOC system using EPA Method 415.2.

h. Volatile Organic Compounds (VOC) using EPA Method 8240.

i. Evaporite analysis using Fourier Transform/Infrared Spectroscopy (FTIR).

j. Blue dye analysis using UltraViolet/Visible (UV/Vis) Spectrophotometer.

k. Thin Layer Chromatography (TLC).

l. Scanning Electron Microscopy (SEM) Energy Dispersive X-Ray Analysis of the evaporite.

See reference (a) for further details on each of the methods. This report contains information on the types of analyses performed, results of the work done on the dyes, and interpretation of the results.

3. ANALYTICAL TEST PROCEDURES

The following paragraphs contain the types of analytical evaluations performed and a summary of the procedure performed on each sample:

a. **Toxicity/Microtox:** This test is performed using the Microbics-M-500 Analyzer per ASTM D-5660-95. This procedure assesses the Microbial Detoxification of chemically contaminated Water and Soil. In this procedure living organisms are exposed to a chemical, substance or sample under controlled conditions to determine if exposure causes any effect to the organisms. Toxic responses are the consequences of interference with biochemical reactions. The light produced by the organism is tied to the metabolism of the cell. Some toxicants can enter the cell membrane faster than others. This toxicity test uses Luminescent Marine Bacterium. Toxicity is measured as toxic inhibition of bacterial light output. The results are reported in terms of inhibitory concentration (IC) or effective concentration (EC). It is the calculated concentration of sample required to produce a specific quantitative and qualitative inhibition. EC50 is where gamma=1 or light loss/light remaining 50/50=1.

b. **Explosives in Dye Water**: High Pressure Liquid Chromatography (HPLC) Method 8330 of EPA manual SW-846 is used to analyze for Nitroaromatics and Nitroamines. Nine hundred milliliters (ml) of each sample were evaporated to dryness using

nitrogen purge at room temperature. A 2 ml aliquot of Acetonitrile (ACN) was added to each dry sample beaker. The evaporite was dispersed in solvent and transfered to a 10 ml volumetric flask. The second 2 ml aliquot of ACN is added to each beaker to continue removing the evaporite. Repeat the process until 10 ml of ACN has been used to clean all the surfaces of the beaker. Nitroguanidine, Nitroglycerine, HMX, RDX, 1,3,5-TNB, 1,3-DNB, TETRYL, NB, 2,4,6-TNT, 4-AM-DNT, 2-AM-DNT, 2,4-DNT, 2,6-DNT, 2-NT, 3-NT, and 4-NT are the explosives involved in this analysis. The HPLC system was established using the following parameters:

Chromatographic Column: C-18 Alltech
Detector: Ultraviolet at 254 nanometers Wavelength
Mobile Phase: Isocratic, 50% Methanol, 50% Deionized Water
Test Data: Maxima Software
Flow Rate: 1.0 milliliter per minute
Injection volume: 10 microliters

c. **Analysis for Disodium Fluorescein (Uranine) Green Dye:** HPLC was used to analyze the Disodium Fluorescein (Uranine) in the dye water samples. Nine hundred milliters of each sample is evaporated to dryness using nitrogen purge at room temperature. The evaporite was extracted with methanol to a 5 ml volume. Dye standards were also prepared in methanol. The following parameters were used for this analysis:

Chromatographic Column: C-18 Alltech
Detector: Ultraviolet at 254 nanometers Wavelength
Mobile Phase: Isocratic, 50% Methanol, 50% Deionized Water and a Tetrabutylammonium Phosphate, 0.5 molar, Ph 7.5
Test Data: Maxima Software
Flow Rate: 1.0 milliliter per minute
Injection volume: 10 microliters

d. **Total Suspended Solids (TSS):** TSS was performed in accordance with EPA Method # 160.2. This method is applicable to drinking, surface, and saline waters in addition to domestic and industrial wastes. The practical range of determination is 4 parts per million (ppm) to 20,000 ppm. Four hundred milliliters of sample was filtered through glass fiber filters. The residue plus filter was dried at 103 to 105 degrees Centigrade to a constant weight. The TSS value is determined by the difference in gross (sample filtered weight) and tare (empty filter) weight.

e. **Ion Exchange Chromatography(IEC):** IEC uses a Waters Ion Exchange Chromatography (IEC) system to analyze for seven different anions in the DBG samples. The seven anions analyzed were: Fluoride, Chloride, Nitrite, Bromide, Nitrate, Phosphate, and Sulfate. The following parameters were used for this analysis:

Mobile phase: 0.038 Molar Borate-Gluconate mixture with 50 milliliters Acetonitrile and 20 milliliters of Butyl Alcohol per liter.

Sample/standard filtration: 0.45um particulate filters and then prewashed with a Sep Pack C18.

Pump: Waters 590

Detector: Waters 430 Conductivity

Injection unit: Shimadzu SIL 9 Auto Injector

Test Data: Maxima Software.

f. **AMMONIA-NITROGEN:** Ammonia-nitrogen was analyzed using EPA Method 350.3, Nitrogen, Ammonia (Potentiometric, Ion Selective Electrode). This method of analysis is applicable to the measurement of ammonia-nitrogen in drinking, surface and saline waters in addition to domestic and industrial wastes. The procedure covers the range from 0.03 to 1400 mg NH3-N/L. Color and turbidity have no effect on the measurements, thus, distillation may not be necessary. Ammonia is determined potentiometrically using an ion selective ammonia electrode anode and a pH meter having an expanded millivolt scale or a specific ion meter. The ammonia electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an ammonium chloride internal solution. Ammonia in the sample diffuses through the membrane and alters the pH of the internal solution, which is sensed by a pH electrode. There was no special sample preparation involved. One hundred milliliters of sample was used to get readings from the electrometer.

g. **TOTAL ORGANIC CARBON (TOC):** TOC analysis of the dye water samples was performed using EPA Method 415.2 Organic Carbon, Total (low level) (UV promoted, persulfate oxidation). This method covers the determination of total organic carbon in drinking water and other water containing carbonaceous matter which is either soluble or has a particle size of 0.2 mm or less or a non-homogenized sample. The Dohrmann DC-80 TOC System was used to analyze for TOCs in the Dye water samples. The samples were filtered, adjusted to pH 2-3 and then analyzed. Homogenizing the sample can cause loss of purgeable organic carbon, thus yielding low results. The total organic carbon measured by this procedure is the sum of the purgeable organic carbon and the nonpurgeable organic carbon.

h. **VOLATILE ORGANIC Compounds (VOC):** The method used was EPA method 8240. An inert gas is bubbled through a 5 milliliter water sample contained in a specially designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a solvent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are them detected with a mass spectrometer.

i. **FOURIER TRANSFORM INFRARED (FT/IR):** In this application, a Potassium Bromide dispersion technique was performed. FT/IR analysis was performed on the evaporite. A small amount of evaporite (approximately 0.7 milligrams) is blended with Potassium Bromide (approximately 100 milligrams) and formed into a translucent pellet. A Potassium Bromide pellet is used as a blank. Sample scans are cross-matched verses standard material scans for identification.

j. **UV/VISIBLE ANALYSIS OF BLUE DYES:** A UV/VIS Spectrophotometer was use to quantitate the Blue Dye constituent of the water samples from the Dye Burial Grounds. Samples were evaporated from 900 milliliters to 10 milliliters for analysis. Acetic acid was added (one drop per 10 milliliters) to samples and standards to inhibit the Uranine dye present and enhance the Blue dye present for analysis. Standards were prepared in methanol. A Perkin & Elmer Model 552 UV/VIS spectrophotometer measured absorbance at 408 nm for the Blue Dye. The Blue Dye Mil-D-16627 is only 50 percent dye so quantitation was performed accordingly. A solution concentration of blue dye was determined by plotting absorbance verses concentration. The absorbance point was then plotted on the standard line. The concentration and dilution factors were calculated to give the final concentration of Blue Dye in the original tank solutions.

k. **THIN LAYER CHROMATOGRAPHY (TLC):** Nine hundred milliliters of sample was evaporated to dryness using nitrogen purge. Dry extracts were spotted on a silica gel TLC plate. A mixture of organic solvents was used to develop the TLC plate and solvent polarity was adjusted to get better separation. The developed plate was examined under visible and ultraviolet lamps to determine the number of dye components and their color characteristics. Preliminary analyses by the Explosive Sciences Branch determined the presence of only two dyes (Blue Dye Mil-D-16627 and Disodium Fluorescein (Uranine)).

l. **SCANNING ELECTRON MICROSCOPE (SEM) ENERGY DISPERSIVE X-RAY ANALYSIS:** This technique provides qualitative information on the chemical composition of a material. SEM was used to detect elemental constituents in the residue from the evaporated Dye Burial Ground water. The solid sample requires no additional preparation to be analyzed. The sample is placed on copper, aluminum or carbon tape for analysis. The sample is then placed in the SEM chamber and the chamber is evacuated. The sample is analyzed at a minimum of 20 keV for elemental Identification. Data is acquired to obtain 3000 to 30,000 counts for identification.

4. RESULTS AND DISCUSSION

Based on the work described above, the following findings are made:

a. **Microtox/Toxicity:** All samples were tested as received and

were found to be Non-Toxic. EC50 was used as an expression of toxicity. A 100 ppm Phenol standard shows 17.4924 ppm EC50 value at a 95% confidence level. A standard containing 3.5 ppm of blue dye showed no toxicity. Another standard containing 7.5 ppm of Disodium Fluorescein (Uranine) showed no toxicity. The EC50 value was higher than the concentration value of the standards. Also, the EC50 value was higher than the highest concentration of the samples. The sample was concentrated until some toxicity was determined. Nine hundred milliliters of sample concentrated to 10 milliliters (factor of 90:1) gave an EC50 value to be 0.22 ppm, a concentration factor of 450:1 gave a 3.4 ppm EC50 value.

b. **Explosives in Dye Water:** Appendix A contains the details of explosives analyzed. All samples showed explosives below detection limit, 0.001 ppm.

c. **Disodium Fluorescein in Dye Water:** Disodium Fluorescein (Uranine) Dye was found to be below the detection limits of 10 ppb for the HPLC. A spiked sample showed 1 ppm and a composite spike detected 1.5 ppm. Appendix A shows the results of Disodium Fluorescein found in the Dye Water samples.

d. **Total Suspended Solids:** Appendix A contains the results of all tanks. This method followed EPA Method number 160.2. The analysis showed all samples to be less than 4 ppm with the exception of sample DBGFT 8 121797 (Tank # S1885 Composite). This sample contained 8.50 ppm suspended solids.

Spiked sample	87.8 percent recovery
NPDES permit	10.0 ppm monthly ave. (For TSS)

e. **Ion Exchange Chromatography:** Ion Exchange Chromatography was used to test for seven anions. IEC analysis showed chloride in the range 0.9 to 6.9 ppm and sulfate in the range 24 to 178 ppm present in the water samples. Spiked recoveries for each anion were calculated for each spiked sample. Unfortunately, the Fluoride spike peak was obscured by the injection peak. But, at a concentration of Fluoride above 1.5 ppm, interference is insignificant (ref. CR4052 EOP 0010). Here is a list of the spike recoveries.

Anion	Ave. Spike Recovery (percent)
Chloride	82.5
Nitrite	89.8
Bromide	91.9
Nitrate	95.6
Phosphate	77.3
Sulfate	89.6

f. **Ammonia-Nitrogen:** The results of the Ammonia-Nitrogen Analysis found the samples to have between 0.03 ppm and 0.037 ppm Ammonia-Nitrogen. A spiked sample produced 105.72 percent

recovery. The Ammonia-Nitrogen detection limit is 0.03 ppm.

The NSWC NPDES permit for waste treatment is 1.5 ppm (summer) and 3.5 ppm (winter) as the monthly average limit for Ammonia as Nitrogen.

g. **Total Organic Carbon (TOC):** Total Organic Carbon (TOC) analysis of the samples as received found TOC levels between 1.8 ppm and 4.1 ppm. TOC was evaluated using three 10 ppm standards. A five ppm standard was used for spiking.

Standard (ppm)	Results (ppm)
10	9.995
10	9.993
10	10.04

Spike (ppm)	Percent Recovery
5	94.3

h. **Volatile Organic Compound (VOC):** Volatile Organic Compounds (VOC) analysis was not performed on the samples for several reasons:

(1) TOC results did not indicate any additional organics that were found in the background samples. TOC value is due to biological components in ground water.

(2) FT/IR analysis of the evaporite showed no indicatioon of organics.

(3) Previous testing found no additional VOC's.

i. **Fourier Transform Infrared (FT/IR):** Analysis of the evaporite was performed and FT/IR scans found calcium sulfate as the major constituent of the dye residue. No organic was detected in the FT/IR scans.

j. **Ultraviolet/Visible Spectrometry:** Analysis for the Blue Dye Mil D-16627 showed less than 1 ppm in the samples. The UV/VIS spectrophotometer detection limit was 0.02 - 1.00 ppm for this analysis.

k. **Thin Layer Chromatography (TLC):** TLC determined the presence of a Blue Dye Mil D-16627 and a Green Dye (Disodium Fluorescein). No other dyes were detected.

l. **Scanning Electron Microscope (SEM) Energy Dispersive X-Ray Analysis:** SEM elemental analysis confirmed the presence of calcium and sulfur in the evaporite from the Dye Burial Ground Water. SEM analysis also found Sodium, Magnesium, Silicon, Chloride, Iron, Oxygen and Aluminum in the Dye water residue. These elemental constituents are normally found in soil and probably tank corrosion products.

5. RESULTS AND DISCUSSION PER TANK

Tank # 1001MEA: In summary, analysis for heavy metals and toxicity determined the water to be non-toxic. Based on EPA regulations and chemical analyses(HPLC Explosives, HPLC Uranine Dye, TSS, IEC, Ammonia-Nitrogen, TOC, FT/IR, UV/VIS Blue Dye, and SEM), the tank could be processed through waste treatment (i.e. carbon tower).

Tank # GS505EA: In summary, analysis for heavy metals and toxicity determined the water to be non-toxic. Based on EPA regulations and chemical analyses(HPLC Explosives, HPLC Uranine Dye, TSS, IEC, Ammonia-Nitrogen, TOC, FT/IR, UV/VIS Blue Dye, and SEM), the tank could be processed through waste treatment (i.e. carbon tower).

Tank # GS522EA: In summary, analysis for heavy metals and toxicity determined the water to be non-toxic. Based on EPA regulations and chemical analyses(HPLC Explosives, HPLC Uranine Dye, TSS, IEC, Ammonia-Nitrogen, TOC, FT/IR, UV/VIS Blue Dye, and SEM), the tank could be processed through waste treatment (i.e. carbon tower).

Tank # GS650NEA: In summary, analysis for heavy metals and toxicity determined the water to be non-toxic. Based on EPA regulations and chemical analyses(HPLC Explosives, HPLC Uranine Dye, TSS, IEC, Ammonia-Nitrogen, TOC, FT/IR, UV/VIS Blue Dye, and SEM), the tank could be processed through waste treatment (i.e. carbon tower).

Tank # GS88EA: In summary, analysis for heavy metals and toxicity determined the water to be non-toxic. Based on EPA regulations and chemical analyses(HPLC Explosives, HPLC Uranine Dye, TSS, IEC, Ammonia-Nitrogen, TOC, FT/IR, UV/VIS Blue Dye, and SEM), the tank could be processed through waste treatment (i.e. carbon tower).

Tank # S1647SD: In summary, analysis for heavy metals and toxicity determined the water to be non-toxic. Based on EPA regulations and chemical analyses(HPLC Explosives, HPLC Uranine Dye, TSS, IEC, Ammonia-Nitrogen, TOC, FT/IR, UV/VIS Blue Dye, and SEM), the tank could be processed through waste treatment (i.e. carbon tower).

Tank # 1885EA: In summary, analysis for heavy metals and toxicity determined the water to be non-toxic. Based on EPA regulations and chemical analyses(HPLC Explosives, HPLC Uranine Dye, TSS, IEC, Ammonia-Nitrogen, TOC, FT/IR, UV/VIS Blue Dye, and SEM), the tank could be processed through waste treatment (i.e. carbon tower).

Tank # S47ST: In summary, analysis for heavy metals and toxicity determined the water to be non-toxic. Based on EPA regulations and chemical analyses(HPLC Explosives, HPLC Uranine Dye, TSS, IEC, Ammonia-Nitrogen, TOC, FT/IR, UV/VIS Blue Dye, and SEM), the tank could be processed through waste treatment (i.e. carbon

tower).

Tank # S879NSD: In summary, analysis for heavy metals and toxicity determined the water to be non-toxic. Based on EPA regulations and chemical analyses(HPLC Explosives, HPLC Uranine Dye, TSS, IEC, Ammonia-Nitrogen, TOC, FT/IR, UV/VIS Blue Dye, and SEM), the tank could be processed through waste treatment (i.e. carbon tower).

DATA TABLES

Appendix A

DETECTION LIMITS TABLE

Analytical Test	Detection Limits
1. Microtox/Toxicity	0.22 PPM (lower limit)
2. Explosives	0.001 PPM (lower limit)
3. Disodium fluorescein	10 PPB (lower limit)
4. Total Suspended Solids: (TSS)	4 PPM
5. Anions	0.01 PPM (lower limit)
6. Ammonia-Nitrogen	0.03 PPM (lower limit)
7. Total Organic Carbon (TOC)	0.1 PPM
8. Blue Dye	0.01-1.00 PPM

Spike and Spike Recovery Data for Blue Dye

Tank number	Spiked sample PPM in Solution	Percent recovery
GS88EA(TOP)	0.16	74.37
GS505EA(TOP)	0.19	
GS505EA(BOT)	0.17	
GS650NEA(COMP)	0.23	124.2

Spike Sample Recovery Data for Total Suspended Solids (TSS)

Spike Sample	recovery
composite	87.8%

Toxicity values in concentrated sample

Concentrated sample	0.22 EC Value
concentrated composite sample	3.4 EC Value

Spike Sample Recovery Data for Explosives in Dye water

composite spike sample recovery 90.0%

Spike Sample Recovery Data for Disodium fuorescein in Dye water

spike sample	1 PPM	86 % recovery
Composite sample spike	1.5 PPM	75 % recovery

Spike Sample Recovery Data for Anions in Dye Water

Anions	Average Spike Recovery %
Chloride	82.50
Nitrite	89.80
Bromide	91.90
Nitrate	95.60
Phosphate	77.30
Sulphate	89.60

Spike Sample Recovery Data for Ammonia-Nitrogen in Dye Water

Spike sample	105.72% recovery

Spike Sample Recovery Data for Total Organic Carbon (TOC) in Dye Water

5 PPM Spike sample	94.30% recovery

Total Organic Carbon Background Analysis (5-28-98)

Total Organic Carbon (TOC) Analysis of Dye Burial Ground Background sample (N98-2075, collected 5-22-98, NSWC02/11-09, Turkey Creek Tributary on H-58)

Calibration Adjusted Average 9.990

Standard #1 TOC 9.743

Calibration Adjusted Average 9.996

Standard #2 TOC 10.10

Sample	**TOC (ppm)**
N98-2075 #1	1.846
N98-2075 #2	1.961
N98-2075 spiked at (5 ppm)	5.540
Blank	0.535

Average of two N98-2075 samples = 1.904 ppm for TOC

TANK # 1001MEA

All units measured in parts per million (ppm)

TESTS	TOP	BOTTOM	COMP
TOC			2.0
VOC			N/A
AMMONIA			0.033
IEC - FLUORIDE			BDL
IEC - CHLORIDE			3.6
IEC - NITRITE			BDL
IEC - BROMIDE			BDL
IEC - NITRATE			BDL
IEC - PHOSPHATE			BDL
IEC - SULPHATE			133
HPLC - HMX			BDL
HPLC - RDX			BDL
HPLC -TNT			BDL
HPLC - FLUORESCEIN			BDL
TSS			<4.0
FT/IR			CALCIUM SULFATE
SEM			Na, Mg, Si, Cl, Fe, Ca, S, O
TLC			N/A
UV MIL-D-16627 (BLUE DYE)			0.24
VISUAL			GREEN
TOXICITY			NON-TOXIC

TANK # GS505EA

All units measured in parts per million (ppm)

TESTS	TOP	BOTTOM	COMP
TOC	2.98	3.338	
VOC	N/A	N/A	
AMMONIA	0.030	0.030	
IEC - FLUORIDE	BDL	BDL	
IEC - CHLORIDE	0.9	0.9	
IEC - NITRITE	BDL	BDL	
IEC - BROMIDE	BDL	BDL	
IEC - NITRATE	BDL	BDL	
IEC - PHOSPHATE	BDL	BDL	
IEC - SULPHATE	30	31	
HPLC - HMX	BDL	BDL	
HPLC - RDX	BDL	BDL	
HPLC -TNT	BDL	BDL	
HPLC - FLUORESCEIN	BDL	BDL	
TSS	<4.0	<4.0	
FT/IR	CALCIUM SULFATE	CALCIUM SULFATE	
SEM	Na, Mg, Al, Si, Fe, Ca, S, O	Na, Mg, Al, Si, Fe, CA, S, O	
TLC	N/A	N/A	
UV MIL-D-16627 (BLUE DYE)	0.12	0.09	0.11 (AVE)
VISUAL	BROWN	BROWN	
TOXICITY	NON-TOXIC	NON-TOXIC	

TANK # GS522EA
All units measured in parts per million (ppm)

TESTS	TOP	BOTTOM	COMP
TOC	2.4	2.7	
VOC	N/A	N/A	
AMMONIA	0.034	0.032	
IEC - FLUORIDE	BDL	BDL	
IEC - CHLORIDE	3.3	3.3	
IEC - NITRITE	BDL	BDL	
IEC - BROMIDE	BDL	BDL	
IEC - NITRATE	BDL	BDL	
IEC - PHOSPHATE	BDL	BDL	
IEC - SULPHATE	103	104	
HPLC - HMX	BDL	BDL	
HPLC - RDX	BDL	BDL	
HPLC -TNT	BDL	BDL	
HPLC - FLUORESCEIN	BDL	BDL	
TSS	<4.0	<4.0	
FT/IR	CALCIUM SULFATE	CALCIUM SULFATE	
SEM	Na, Mg, Al, Si, Cl, Ca, S, O	Na, Mg, Al, Si, Cl, Fe, Ca, S, O	
TLC	N/A	N/A	
UV MIL-D-16627 (BLUE DYE)	0.24	0.22	0.23 (AVE)
VISUAL	GREEN	GREEN	
TOXICITY	NON-TOXIC	NON-TOXIC	

TANK # GS650NEA
All units measured in parts per million (ppm)

TESTS	TOP	BOTTOM	COMP
TOC			2.497
VOC			N/A
AMMONIA			0.031
IEC - FLUORIDE			BDL
IEC - CHLORIDE			3.5
IEC - NITRITE			BDL
IEC - BROMIDE			BDL
IEC - NITRATE			BDL
IEC - PHOSPHATE			BDL
IEC - SULPHATE			162
HPLC - HMX			BDL
HPLC - RDX			BDL
HPLC -TNT			BDL
HPLC - FLUORESCEIN			BDL
TSS			<4.0
FT/IR			CALCIUM SULFATE
SEM			Mg, Al, Si, Cl, Ca, S, O
TLC			N/A
UV MIL-D-16627 (BLUE DYE)			0.19
VISUAL			GREEN
TOXICITY			NON-TOXIC

TANK # GS88EA

All units measured in parts per million (ppm)

TESTS	TOP	BOTTOM	COMP
TOC	1.9	1.9	
VOC	N/A	N/A	
AMMONIA	0.034	0.033	
IEC - FLUORIDE	BDL	BDL	
IEC - CHLORIDE	1.6	1.8	
IEC - NITRITE	BDL	BDL	
IEC - BROMIDE	BDL	BDL	
IEC - NITRATE	BDL	BDL	
IEC - PHOSPHATE	BDL	BDL	
IEC - SULPHATE	24	24	
HPLC - HMX	BDL	BDL	
HPLC - RDX	BDL	BDL	
HPLC -TNT	BDL	BDL	
HPLC - FLUORESCEIN	BDL	BDL	
TSS	<4.0	<4.0	
FT/IR	CALCIUM SULFATE	CALCIUM SULFATE	
SEM	Fe, Mg, Si, Cl, Ca, S, O	a, Mg, Al, Si, Cl, Fe, Ca, S, O	
TLC	N/A	N/A	
UV MIL-D-16627 (BLUE DYE)	0.08	0.1	0.13 (AVE)
VISUAL	BROWN	BROWN	
TOXICITY	NON-TOXIC	NON-TOXIC	

TANK # S1647SD

All units measured in parts per million (ppm)

TESTS	TOP	BOTTOM	COMP
TOC			3.5
VOC			N/A
AMMONIA			0.034
IEC - FLUORIDE			BDL
IEC - CHLORIDE			5.4
IEC - NITRITE			BDL
IEC - BROMIDE			BDL
IEC - NITRATE			BDL
IEC - PHOSPHATE			BDL
IEC - SULPHATE			178
HPLC - HMX			BDL
HPLC - RDX			BDL
HPLC -TNT			BDL
HPLC - FLUORESCEIN			BDL
TSS			<4.0
FT/IR			CALCIUM SULFATE
SEM			Na, Mg, Al, Si, Cl, Mn, Ca, S,
TLC			N/A
UV MIL-D-16627 (BLUE DYE)			0.28
VISUAL			GREEN
TOXICITY			NON-TOXIC

TANK # S1885EA

All units measured in parts per million (ppm)

TESTS	TOP	BOTTOM	COMP
TOC			3.096
VOC			N/A
AMMONIA			0.037
IEC - FLUORIDE			BDL
IEC - CHLORIDE			3.3
IEC - NITRITE			BDL
IEC - BROMIDE			BDL
IEC - NITRATE			BDL
IEC - PHOSPHATE			BDL
IEC - SULPHATE			119
HPLC - HMX			BDL
HPLC - RDX			BDL
HPLC -TNT			BDL
HPLC - FLUORESCEIN			BDL
TSS			8.5
FT/IR			CALCIUM SULFATE
SEM			Na, Mg, Al, Si, Cl, Fe, Ca, S, O
TLC			N/A
UV MIL-D-16627 (BLUE DYE)			0.15
VISUAL			GREEN
TOXICITY			NON-TOXIC

TANK # S47ST

All units measured in parts per million (ppm)

TESTS	TOP	BOTTOM	COMP
TOC	3.7	4.1	
VOC	N/A	N/A	
AMMONIA	0.034	0.036	
IEC - FLUORIDE	BDL	BDL	
IEC - CHLORIDE	5.7	5.7	
IEC - NITRITE	BDL	BDL	
IEC - BROMIDE	BDL	BDL	
IEC - NITRATE	BDL	BDL	
IEC - PHOSPHATE	BDL	BDL	
IEC - SULPHATE	168	172	
HPLC - HMX	BDL	BDL	
HPLC - RDX	BDL	BDL	
HPLC -TNT	BDL	BDL	
HPLC - FLUORESCEIN	BDL	BDL	
TSS	<4.0	<4.0	
FT/IR	CALCIUM SULFATE	CALCIUM SULFATE	
SEM	Na, Mg, Si, Cl, Al, Ca, S, O	Na, Mg, Al, Si, Cl, Ca, S, O	
TLC	N/A	N/A	
UV MIL-D-16627 (BLUE DYE)	0.24	0.21	0.23 (AVE)
VISUAL	GREEN	GREEN	
TOXICITY	NON-TOXIC	NON-TOXIC	

TANK # S879NSD

All units measured in parts per million (ppm)

TESTS	TOP	BOTTOM	COMP
TOC	1.848	2.115	
VOC	N/A	N/A	
AMMONIA	0.032	0.030	
IEC - FLUORIDE	BDL	BDL	
IEC - CHLORIDE	4.0	3.9	
IEC - NITRITE	BDL	BDL	
IEC - BROMIDE	BDL	BDL	
IEC - NITRATE	BDL	BDL	
IEC - PHOSPHATE	BDL	BDL	
IEC - SULPHATE	75	76	
HPLC - HMX	BDL	BDL	
HPLC - RDX	BDL	BDL	
HPLC -TNT	BDL	BDL	
HPLC - FLUORESCEIN	BDL	BDL	
TSS	<4.0	<4.0	
FT/IR	CALCIUM SULFATE	CALCIUM SULFATE	
SEM	Na, Mg, Al, Si, Cl, Ca, S, O	Na, Mg, Si, Cl, Fe, Ca, S, O	
TLC	NN/A	N/A	
UV MIL-D-16627 (BLUE DYE)	0.11	0.13	0.12 (AVE)
VISUAL	GREEN	GREEN	
TOXICITY	NON-TOXIC	NON-TOXIC	

YB

8000/5
Ser 4052/8088

MEMORANDUM

From: 4052
To: 095 (T. Brent)

Subj: **ANALYSIS REPORT OF DYES FROM THE CRANE DYE BURIAL GROUNDS, OED/C-98-045**

Ref: (a) QUALITY ASSURANCE PROJECT PLANT (QAPP)
(b) Equipment Operating Procedures of Explosive Sciences Branch

Encl: (1) Results of Dye Water Analysis OED/C-98-045

1. Code 4052 was requested by Code 095 to perform the analysis on nine tanks containing dye water from the Dye Burial Grounds (DGB) at Crane. The samples were assigned the Explosive Sciences Branch Sample Numbers N98-2001 to N98-2074.
N98-2075

2. The samples from the tanks were subjected to the analytical tests described in reference (a). The procedures defined in reference (b) were used as guidelines. A full detailed analysis complete with data sheets is attached as enclosure (1).

3. Code 40 points of contact are Mr. Maroof Qurashi and Mr. Steve Weddle, Code 4052, extensions 4230 and 6049 respectively.

NANCEY J. MAEGERLEIN

Copy to:
40211