

Environmental Security Technology Certification Program (ESTCP)

Final Report



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Non-Thermal, On-Site Decontamination and Destruction of Practice Bombs**

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List of Acronyms

BDU Bomb Dummy Unit

DOD Department of Defense

EOD Explosive Ordnance Disposal

SOP Standard Operating Procedure

UXO Unexploded Ordnance

1 Abstract

The process was evaluated to determine required process variables. Following treatment the clean scrap was evaluated to determine its value. After neutralization, the liquid by-products were analyzed for explosive content, metals, and pH. During operations, gasses evolved were analyzed for hydrogen, carbon monoxide, carbon dioxide, and phosphine.

Overall the process failed to achieve its objectives. The failure was due to the inability of the caustic fluid to penetrate certain blockages of some dropped, but still live, BDU's so that the propellant was not always destroyed. The fact that propellant does remain in some cases means that inspection would be required to verify that the process has worked as intended for each and every BDU treated, which largely negates the projected cost savings from the process, and does not decrease the hazards associated with recycling BDU's over the present practice.

Acknowledgements

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2 Introduction

2.1 Background

The U. S. Armed Forces have been exploring alternative methods of dealing with unexploded ordnance (UXO) practice bombs (such as the BDU-33) that contain spotting charges (such as the CXU-5, MK-4, or KC-4). These spotting charges contain primers, a propelling charge (generally a nitrocellulose base solid propellant), and a smoke charge (containing Titanium Tetrachloride (TiCl₄) or Red Phosphorus (RP)). There are hundreds of thousands of these bombs at bombing ranges throughout the U.S.

The explosive ordnance disposal (EOD) community estimates the dud rate of these bombs to be upwards of 20%. This means that when removing these items from a range, every bomb has to be examined (both visually and by probe) to determine if it has functioned as designed. If it has not functioned, the item must be rendered inert, usually by detonation of a countercharge. Problems with assuring 100% detection and segregation of dud practice bombs, along with problems related to present inerting practices (kickouts, failure to detonate) mean that the costs to deal with these practice bombs are high and are climbing higher as environmental constraints tighten. A new, low cost, safe, and environmentally acceptable method of simultaneously destroying the propelling charge and the TiCl₄ or RP is required.

UXBaseSM, a new process under development by UXB International, Inc. (UXB), uses base hydrolysis (a non-thermal, environmentally safe, cost effective alternative to the “3X” and thermal “5X” methods) to treat and destroy propellant and explosive residues on range-derived scrap and other materials. UXB feels this technology can also be applied to practice bombs.

2.2 Objectives of the Demonstration

The primary objectives of this demonstration were to:

- Validate the UXBaseSM process for the disposal of BDU-33 practice bombs so that the process can be transitioned to deployment;
- Provide data that enables cost comparisons, environmental impact assessments, logistics requirements, and safety analysis.

This test involved the gathering and processing of BDU-33 practice bombs, and the characterization and disposal of by-products (including scrap metal, spent liquids, and any entrained dirt/soil on the practice bombs).

We used the Barry M. Goldwater Range (BMGR) in Arizona, as the venue for this demonstration. The range has a large inventory of dropped BDU's that require disposition.

UXBaseSM is a proven technology for destroying propellants and explosive residue on range-derived ordnance and explosive scrap to a 5X equivalent condition. It generates an easily disposable non-hazardous by-product, and is environmentally friendly, cost-effective, efficient, and safe. This demonstration was the first step in determining the process' applicability for destroying propellant and explosive residue in practice bombs.

2.3 Regulatory Drivers

This project was not driven by existing federal, state, or local regulations, or DOD directives. It was motivated by the need for a safer, more cost-effective, efficient process for simultaneously destroying the propelling charge and $TiCl_4$ / RP in BDU-33s and other munitions.

2.4 Stakeholder/End-User Issues

Stakeholder issues include cost and efficiency drivers as well as environmental issues. The demonstration addressed cost and efficiency concerns by developing a complete cost model and populating that model with actual costs experienced during the demonstration. Environmental issues were addressed by the demonstration as part of the pre-demonstration planning and pre- and post-demonstration experience in terms of actual analysis of waste products produced.

3 Technology Description

3.1 Technology Development and Application

In the UXB process, (Figure 1 & 2) all practice bombs (dud or not) removed from the range are fed to a heated tank containing a solution of sodium hydroxide initially made up to two moles per liter (about 10% by weight). The aluminum in the CXU-5 spotting cartridge reacts with the caustic and dissolves; releasing hydrogen (if the spotting cartridge breaks during loading or unloading, the caustic neutralizes the $TiCl_4$ to non-hazardous titanium dioxide, water, and salt). If the cartridge is a KC-4 type, then the caustic attacks the aluminum case and dissolves it, releasing the RP.

The tank is equipped with a perforated basket for loading and removal of the scrap after treatment. The procedure is to load the practice bombs into the perforated basket while the basket is on the ground. Once loaded, a lift raises the basket. The basket is lowered into the caustic. Once the tank is loaded with scrap items, the mixture is allowed to soak for two to four hours to ensure complete destruction of all energetic material. Alternatively, the mixture can be left until the next day if the loading is performed towards the end of the day. After the hold time has elapsed, the basket will be raised out of the tank and allowed to drain. After draining, the basket is placed into a second tank (containing water) to rinse any remaining caustic from the scrap. The water will be adjusted to a pH between 6 and 9 with hydrochloric (muriatic) acid before the scrap is removed. At the end of the process, the scrap will be water wet, and the water will contain a small amount of sodium chloride (table salt). The scrap will be removed from the tank and each bomb body will be shaken to remove the $TiCl_4$ ampoule (if present), which is recovered and can be recycled as a pure chemical.

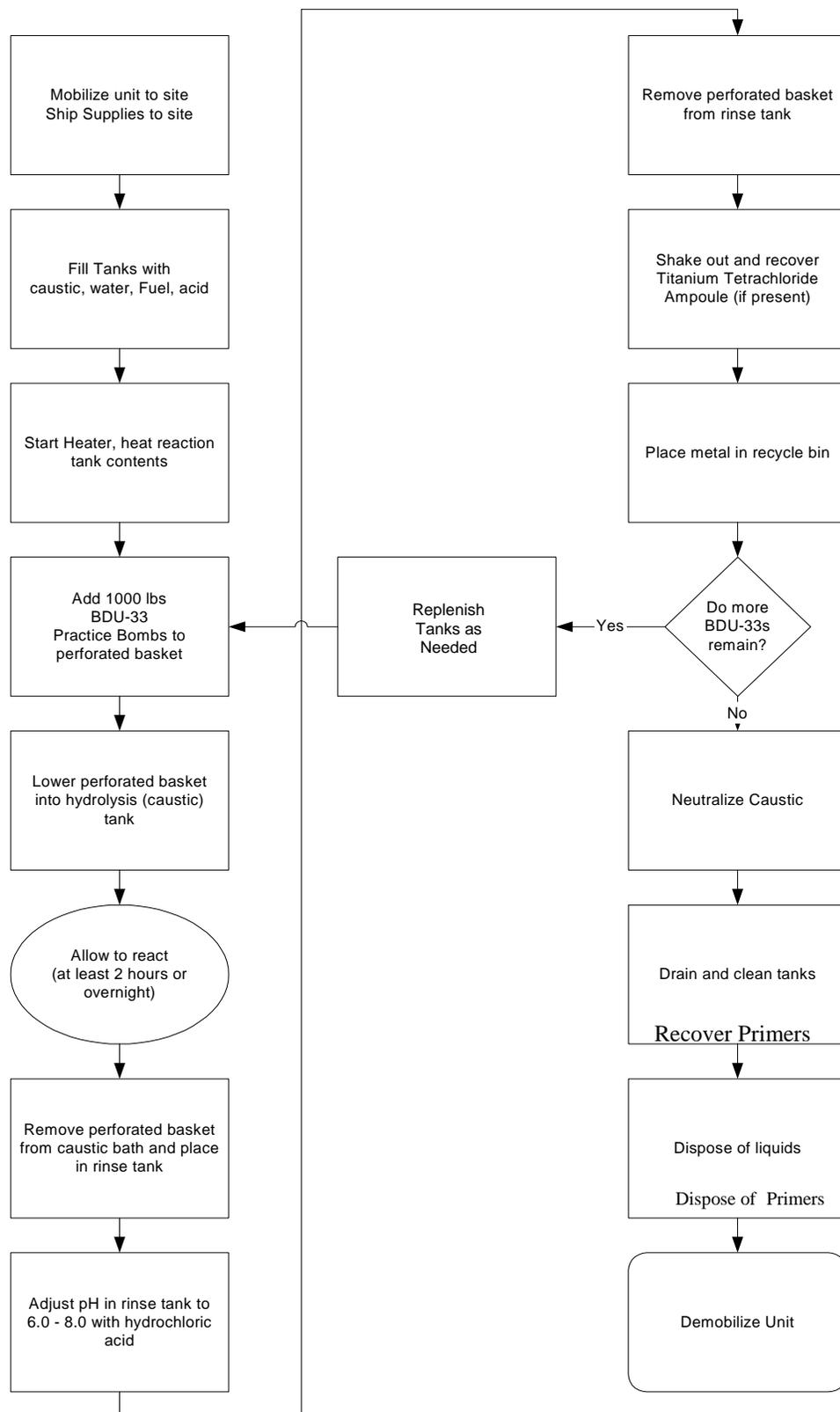
The initial tank fills of hydroxide solution and rinse water are to be re-used for over 100 tons of scrap; each is replenished as required. For this project however, the material was disposed of after four tons as the demonstration was terminated after four tons were processed. If the solutions will be used for approximately 100 tons of practice bombs the solution should be monitored for the buildup of heavy metals. If the heavy metal content is kept below regulatory limits the neutralized solutions may be disposed of as non-hazardous waste. Since the sodium hydroxide solution is used to dissolve the aluminum casings on the spotting charges, aluminum is the predominant metal ion in the solution. The hydroxide solution also hydrolyzes the propellant charges. If the propellant charges (including primer compounds) contain lead, barium, or other heavy metal compounds in their formulation, then these metals will also have to be monitored to keep their level below regulatory limits if the intent is to dispose of the neutralized solution as non-hazardous waste. At the end of the project, both solutions will be neutralized to a pH between 6 and 9 and disposed of as non-hazardous waste. Any remaining non-metallic solid matter (dirt or plastic) will be allowed to dry and will be disposed of as a solid non-hazardous waste. The process is capable of treating all types of metal, plastics, wood, and paper; although aluminum and magnesium are rapidly degraded. Minimal **non-hazardous** solid waste is generated. The technology should be applicable to all sites that contain this UXO, as well as other types of practice bombs.

Figure 1: UXBaseSM Mobile BDU Treatment Unit

See Attachment 1.

The process flow diagram is illustrated in Figure 2.

Figure 2: Process Flow Diagram



Several design criteria required satisfaction when the design for the process and process unit were being finalized. These included:

- Safety. This is the primary design criterion when working with explosive or potentially explosive materials. Over the years several EOD technicians have been injured by BDU-33 practice bombs that were supposed to be inert or expended, but were not. For the unit designed here, safety is achieved by limiting personnel contact with the BDU until after the unit has been processed. Minimal handling of potentially explosive materials is reduced by using mechanical grapples to load the BDU's into the unit.
- Mobility. The process unit must be able to go to where the BDU's are. This achieves several objectives and solves several problems. BDU's of unknown explosive potential cannot be loaded and transported over public roads or rails according to DOT regulations. In addition, multiple handling of BDU's only increases costs and adds to the potential for injury, so this is to be avoided. A mobile process unit allows for processing of the BDU's at each location where needed followed by additional processing of other BDU's at other locations at minimal cost.
- Self contained process unit. Locations where BDU's are present are usually remote and far from any available infrastructure such as electrical power, water, buildings or gas lines. In addition, supplies of fuel may be far away. The process unit must be capable of sustained operations in areas where infrastructure is limited to the presence of unimproved roads into the area.
- Environmental soundness. The objective of these design criteria is to ensure that the environment is protected against issues such as release of chemicals (including explosives, caustics and acids, but not including small amounts of hydrogen gas) into the environment around the process. Verification testing will be performed to validate these criteria. The criteria will also ensure that wastes produced are dealt with in an environmentally responsible manner so that they do not have the potential to injure the environment. In addition these criteria attempt to ensure that the only clue remaining that the process has been active in an area after it leaves will be the footprints of the workers boots.

3.2 Previous Testing of the Technology

Base hydrolysis of energetic materials is a well-known technique for rendering many explosives safe without combustion or detonation. Research has demonstrated hydrolysis of explosives and propellants (including NC, NQ, RDX, HMX, NG, TNT, DNT, and Tetryl among others) at high pH^{ii,iii}. Over the last 40 years, base hydrolysis has been used as a method of decontaminating tools and equipment, and disposing of small amounts of material. Research has shown that at sufficiently high concentrations of hydroxide (over 1 mole/liter), the reaction kinetics become pseudo first order, and thus depend on temperature and concentration of the energetic material being destroyed. Thus, the use of base hydrolysis to destroy and render inert the propelling charge of the CXU-5 or

KC-4 cartridge is an innovative application of well-known principals to the problem of practice bomb disposal.

For the smoke generation chemicals contained in the cartridges, base hydrolysis also destroys $TiCl_4$. $TiCl_4$ reacts vigorously with caustic solutions, yielding titanium dioxide (the white, non-toxic pigment used in modern paints) and hydrochloric acid (HCl), which is subsequently neutralized by the caustic solution to yield water and sodium chloride (table salt)^{iv}.

Our research involved two treatability studies and other experiments on the bench scale -- to ensure that the base hydrolysis method is complete, controllable, and results in a non-hazardous metal byproduct qualified as a 5X material, suitable for recycling. A modification of this process would enable the processing of practice bombs, such as the BDU-33.

Scrap Recovered from a Closed Firing Range in Texas – For the high explosive contaminated scrap metals, UXB used actual scrap removed from a closed range in Texas. Samples of this scrap, in rusted and dirty condition, were contaminated with three different high explosives -- TNT, RDX, and HMX. In each case, the scrap was contaminated by spraying a known concentration solution of the explosive dissolved in acetone onto the scrap items, then allowing the solvent to evaporate. Witness plates, made of mild steel coupons (58 cm²), were also sprayed at the same time to enable verification of the amount of material sprayed on the scrap. The scrap was then placed into a solution of 3 molar NaOH (caustic soda) maintained at a temperature of 80⁰C and allowed to soak for a period of 4 hours. After this time, the heat was turned off, and the materials allowed to soak overnight, simulating field operations. The next morning the scrap was removed and neutralized using hydrochloric acid, and allowed to air dry. After a 24-hour drying time, the dried scrap was extracted with acetone for an additional 24-hours to remove any remaining explosive for analysis. A third party lab^v used a modified EPA SW846 method 8330 to analyze the extract. In all three cases, the extracted scrap items contained no detectable amounts of explosive. The results are summarized in Table 1.

Table 1: Test Results

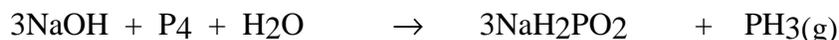
Contaminant	TNT	RDX	HMX
Initial contamination level	479 $\mu\text{g}/\text{cm}^2$	207 $\mu\text{g}/\text{cm}^2$	27 $\mu\text{g}/\text{cm}^2$
Cleaned scrap contamination level	Non-Detect	Non-Detect	Non-Detect

Scrap Solid Propellants and a Propellant Contaminated Sludge at a Commercial Plant in New Jersey -- For the propellant contaminated sludge, a representative sludge sample of 450 grams was placed into the UXB demonstration reactor and allowed to react with 3.7 liters of 3 molar NaOH. The sludge was sampled from a water treatment equalization tank that was located on the grounds of a closed former munitions plant. This sludge was heavily contaminated with energetic material (about 35% of single and double base small arms propellant); dirt and soil made up an additional 15%, and water made up the remainder. The solution was maintained at a temperature of 80⁰C for three hours and then was allowed to cool. After cooling, the material

was neutralized with hydrochloric acid to a pH of 7.0. The liquid was decanted off, leaving a solid residue that was dried and analyzed for explosive content by chemical analysis and explosive character by friction^{vi}, impact^{vii} and flammability^{viii} testing. No explosive or energetic response was noted, to the limit of the instruments. The liquid was also submitted for chemical analysis. The analysis tested for the presence of NC, NG, NQ, TNT, and DNTs, again by EPA method 8330^{ix}. None was detected.

3.2.1 Red Phosphorus

Initial testing with red phosphorus involved producing 500 ml of a 2 molar sodium hydroxide solution in water and placing 10 grams of red phosphorus powder into the solution and heating at 70 °C for 4 hours. No visible changes were made to the red phosphorus at this temperature and duration. This was unexpected, as all references in the literature pertaining to phosphorus reactions with caustics indicated that the reaction would proceed according to:



The phosphine gas thus liberated would then spontaneously ignite on exposure to air. As this did not occur however, there was some question as to why. The experiment was repeated several times to confirm the results eventually increasing the temperature of the caustic solution to 95 °C and soaking for 24 hours. Still the red phosphorus did not hydrolyze as expected. On further investigation it was found that one literature reference from 1911¹ noted that red phosphorus does not react to the action of caustic, even though white and yellow phosphorus (the other phosphorus allotropes) do indeed react as above.

3.2.2 KC-4 Cartridges

A second series of experiments was performed where live KC-4 cartridges were placed in a heated 2 molar solution of NaOH. The temperature was 90 to 95°C, and the reaction was allowed to run to its conclusion, which took about 2 hours. During that time, hydrogen gas was evolved from the solution, as a product from the reaction:



These experiments were successful in that the cartridges were dissolved completely, leaving the red phosphorus as before, and paper and plastic parts (the label and wadding, respectively). All of the propellant grains (a double base material containing nitroglycerine and nitrocellulose) were completely consumed, leaving no solid residue. The primers, however, were not consumed, and were recovered intact. Later testing confirmed that they were still live.

¹ <http://41.1911encyclopedia.org>

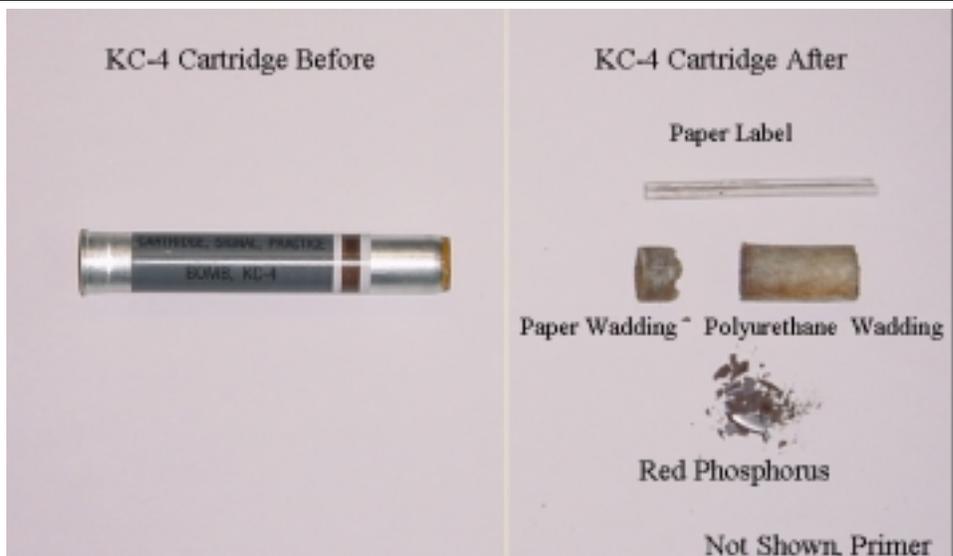


Figure 3: KC-4 Cartridge: Before and After

3.2.3 BDU-33 Practice Bombs with Installed KC-4 Smoke Cartridges

A final series of tests were performed with BDU-33 Practice Bombs with installed KC-4 smoke cartridges. For these tests, the bomb fins were removed, so that the test article would fit into the reaction vessel available in the laboratory. One other modification was that the firing pin actuator for the BDU-33 practice bomb was filed down in order to prevent inadvertent actuation during handling. The reaction vessel was unstirred, as in the design for the larger unit. The bombs with live KC-4 cartridges were placed in a heated 2 molar solution of NaOH. The temperature was 90 to 95°C, and the reaction was allowed to run to its conclusion, which took about 4 hours. During that time, hydrogen gas was evolved from the solution. These experiments were successful in that the cartridges were dissolved completely, leaving the red phosphorus as before, and paper and plastic parts (the label and wadding respectively). All of the propellant grains (a double base material containing nitroglycerine and nitrocellulose) were completely consumed, leaving no solid residue. The primers however were not consumed, and were recovered intact. Later testing confirmed that they were still live. The liquid caustic from several runs was mixed together to make a composite sample that was submitted to an independent laboratory for chemical analysis. The analysis was performed for TCLP metals and explosives by EPA Method SW 8330. All tests came back as non-detect (Appendix D)

3.3 Factors Affecting Cost and Performance

Several factors affect cost and performance. The cost factors include labor costs, chemical costs, disposal costs and mobilization/demobilization costs. Performance factors included the extent of

destruction of the propellants in BDU's that had mud, dirt or rocks blocking the path for caustic to enter the unit, and the number of BDU's that could be processed per load of caustic.

3.4 Advantages and Limitations of the Technology

Advantages of the technology include Safety (both for the operator and downstream personnel), low cost, and mobility. In addition, the process is environmentally sound in that it does not pollute the area where it is used, and does not generate hazardous materials which will have the potential to pollute the environment later.

Disadvantages are that it does not render the BDU shape unrecognizable, and that may be a problem for acceptance by metal recyclers. See section 4.3 for a discussion of alternative processes for processing BDU's.

Based on the field testing, the possibility exists that all of the propellant charge may not be destroyed. Although only 0.4% of the units subjected to the hydrolysis process contained small amounts of residual intact propellant, the potential of propellant ignition once the units dried is still a possibility. The reason that the propellant charges weren't totally destroyed was the inability of the caustic solution to penetrate through the mud, gravel and outer impact debris and contact the spotting cartridge within the practice bomb. Other technologies could be employed to fracture the practice bomb, allowing the caustic unimpeded contact with the aluminum spotting charge casings.

4 Demonstration Design

4.1 Performance Objectives

The performance objectives included the destruction or throughput rate for the BDU's (4 tons per day or more) and complete destruction of the propellant charge in the spotting cartridges. Additionally, the recovery of RP and primers for disposal needed to be demonstrated.

Table 2: Performance Objectives

Type of Performance Objectives	Primary Performance Criteria	Expected Performance (Metric)	Actual Performance Objective Met?
Qualitative	No explosive hazard remaining in treated BDUs	Yes	No
	All primers recovered	Yes	Yes
	All RP recovered	Yes	Yes
Quantitative	Throughput of treated BDUs	4 tons/day	No (2 tons/day)

For the process to be considered a success, there must be no explosive hazard remaining in the treated items, however in two cases (out of 480 units treated or 0.4%) there was some residual

propellant remaining in the BDU after treatment. While the material was not energetic under the UN BAM Fallhammer test (Appendix A) while wet, the potential remains that the material could still be energetic after drying. As this technology depends upon not having to inspect each and every item to assure that destruction of the energetic hazard is complete, any process failure will negate any potential savings that could be achieved with the process.

The production rate of 4 tons per day was also not met. This was also due to the inability of the caustic solution to penetrate through the mud, gravel and other impact debris quickly enough to effect expedient cartridge dissolution. The compacted nature of the debris slowed the wetting time of the caustic significantly and therefore reduced the intended production rate by one half.

This failure to meet the performance objectives is obviously not acceptable for a fielded process, and so modifications to the process would be required. Although outside of the scope of this project, these modifications have been designed. The modifications are more fully described in section 6.3.

4.2 Selecting Test Site(s)

The primary criteria for selecting the Barry M. Goldwater Range (BMGR, Arizona) as the test site was the availability of a significant number of BDU-33 practice bombs that needed to be destroyed. In addition, the range management personnel there were actively interested in furthering the state of the art in BDU disposal, as they stand to get a significant benefit from any cost reductions due to advanced technology.

4.3 Test Site History/Characteristics

The Barry M. Goldwater Range is located approximately 60 miles southwest of Phoenix, AZ, below the town of Gila Bend. The range, situated on 1.7 million acres of withdrawn public land, is utilized by aviation arms of all US military branches of training and limited testing since 1941. Goldwater Range currently supports training for F-16, OA-10, helicopter aviation, and naval carrier based aircraft. The range is also employed by a wide range of allied nations to train pilots of FMS acquired systems as well as their organic tactical aircraft. The range has an aggressive programmatic clearance and maintenance plan established according to AFI 1-212 Volume 1. Active duty military technicians assigned to the 56th Civil Engineer Squadron at Luke AFB provide EOD services. Maintenance services for the range are performed under contract to the 56th FW. 56th RMO Environmental Sciences management is responsible for range residue removal (R3) services. An active contract with Bering Sea Eccotech Co. is in progress to demilitarize and recycle ammunition, explosive, and dangerous article derived metals and trash from the range.

4.4 Present Operations

The Barry M. Goldwater Range has evolved a progressive strategy to mechanically harvest the majority of BDU-33 bombs from their Class A manned range targets. This particular range has dealt with extensive problems from handling and disposing of more of these practice bombs over the years than any other Air Force training range. Past practices have included manual handling,

inspection, and disposal of dud bombs by burning and/or detonation. Past residue disposal practices have included burial in on-range pits, sale through DLA channels, and direct disposal through an on site demilitarization and recycling contract. Goldwater Range's improvements to key handling and demilitarization efforts have removed much of the risk from the processes involved. Any process involving handling unexploded ammunition always involves some risk to people, equipment and the environment, and Goldwater Range is constantly attentive to improving their key processes to ensure safety and maintain environmental compliance.

4.5 Pre-Demonstration Testing and Analysis

No site specific testing was required, as this process is independent of the site location.

4.6 Testing and Evaluation Plan

4.6.1 Demonstration Set-Up and Start-Up

4.6.1.1 Mobilization

4.6.1.1.1 Trailer Setup

The unit was transported from the assembly location in Cordele, Georgia to the test site at BMGR. UXB used Standard Operating Procedures to ready the unit for transport to the demonstration site. A UXB employee drove the unit and a UXB-owned semi-tractor to the site. During transport, the unit did not contain open process fluids – the exception was glycol for the closed loop heat exchangers.

Once at the demonstration site, the unit was transported to the designated work area (an open, level area on the range and BDU stockpile). The unit was blocked and leveled (using the built in air suspension package), and then unhooked from the tractor. The tractor was removed from the demonstration area.

The stairway was placed in a convenient spot to enable trailer access. The operator checked to be certain that the drain plugs on the secondary containment tray were in place and tight.

4.6.1.1.2 Supplies

All supplies (caustic, acid, other consumables) were locally purchased and shipped to the site by the arriving crew. The supplies (which are considered to be Department of Transportation hazardous materials) were delivered by common carrier, thus removing the need for a dedicated hazmat truck and driver.

Water -- The operator filled the water tank with clean water during operations using a delivery truck service. This tank was also filled at the host facilities just before setup with a hose.

Caustic solution (NaOH, 2 moles per liter) -- Caustic solution was packaged in drums and delivered to the site.

Fuel -- Fuel was purchased and the fuel tank filled at a private facility near the site upon arrival, but prior to setup. The amount of fuel in the tank was more than sufficient for the demonstration.

HCl -- Hydrochloric acid was purchased at 37% strength. The solution was delivered in drums to the site.

Site Preparation and Utilities – Site preparation was not required, as the process is designed to not require it. The process is self-contained, and no utilities are required.

4.6.2 Period of Operation – Major Project Milestones

- Lab Scale Measurements Completed – 11-Oct-2002
- Equipment Purchases Completed – 24-Jan-2003
- Write Procedures and Complete Construction – 05-May-2003
- Demonstration Plan Completed – 15-Sep-2003
- System Startup and Testing Completed – 28-Nov-2003
- Demonstration at BMGR Completed – 15-Jul-2005

4.6.3 Scope of Demonstration

UXB traveled to the BMGR and performed 4ea 2000-pound BDU-33 test studies to verify if the process can be used to clean the material to the non-detect level. The runs are further detailed below:

Run Number	Processing time (hrs)	Weight Processed
1	2	2000
2	4	2000
3	overnight	2000
4	overnight	2000

4.6.4 Residuals Handling

At the end of the project, both solutions (the caustic and rinse solutions) were neutralized to a pH between 6 and 9, sampled and disposed of as non-hazardous waste. There was no solid waste generated, and residual metals (treated BDU's) were acceptable for recycling (with the exceptions of two items where propellant was found to be remaining in the cartridges, see section 3.1)

4.6.5 Operating Parameters For The Technology

Preliminary experiments determined that the optimum operating parameters are a process temperature of 70°C or higher (to a maximum of 100°C) and a reaction time of 4 hours. The parameters that were varied for the experiments were the time required for processing and the

batch weight. Three initial runs using a 2000 pound load were made to optimize run time, for 2, 4 hours and two runs were made where the basket contents were allowed to soak overnight, in an attempt to determine if increasing soak time would be helpful in completing destruction of the materials. This attempt resulted in failure however, as in two cases (one in each of the latter two batches) residual propellant was recovered from a treated item.

Samples taken during the process were comprised of two types, liquid and gaseous. The two liquid sample locations were the caustic tank and the rinse tank. Both of the locations were sampled using a grab type sample procured using a dipper. The tanks were sampled at the conclusion of operations (after neutralization of the caustic). Gas samples were taken from the headspace above the caustic tank during each demonstration run (one sample per hour) and were analyzed on site for hydrogen (H₂), carbon monoxide (CO), carbon dioxide (CO₂), and phosphine (PH₃). Table 3 shows the sampling points and frequencies for each experiment.

Table 3: Sampling Points and Frequencies

Sampling Point	Location	Sample Type	Sample Frequency
1	Hydrolysis Tank	Liquid Grab	Once at end of test ²
2	Rinse Tank	Liquid Grab	Once at end of test ³
3	Hydrolysis Tank	Headspace Vapor	Once per hour
4	Hydrolysis Tank	Sludge/mud grab	Once at end of test ⁴
5	Water Tank	Liquid Grab	Once at start of campaign
6	Hydrolysis Tank	Liquid Grab	Once at end of campaign (after neutralization)
7	Rinse Tank	Liquid Grab	Once at end of campaign (after neutralization) ⁵
8	Hydrolysis Tank	Sludge/Mud grab	Once at end of campaign (after neutralization) ⁶

² Combined with sample 6

³ Combined with sample 7

⁴ There was no sludge/mud remaining at end of test

⁵ There was no sludge/mud remaining to sample

⁶ No RP could be found to test.

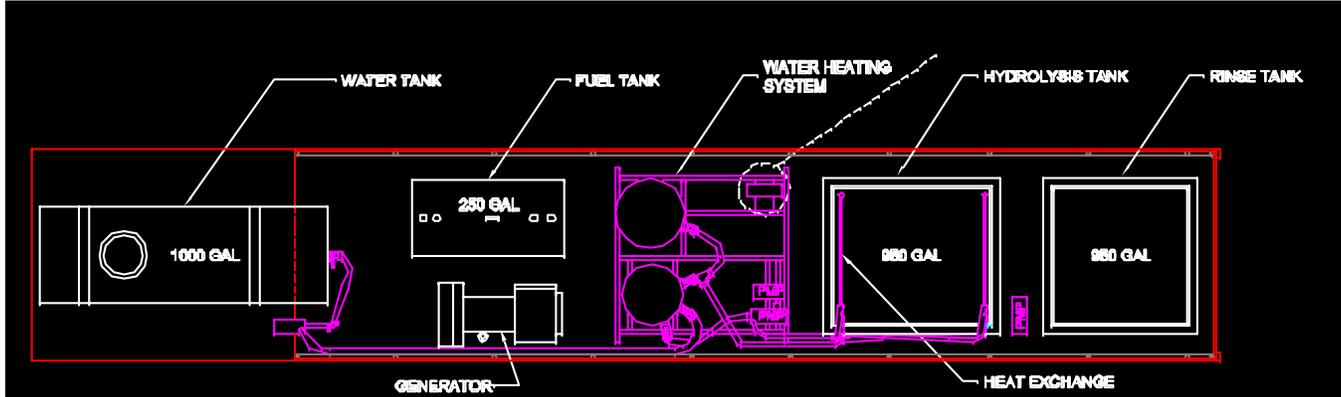


Figure 4: Locations of the Sampling Points

4.6.6 Demobilization

Upon completion of testing, the unit was demobilized and removed from the site. In preparation for demobilization, the neutralized hydrolysis fluids were pumped out of the tanks and removed for offsite disposal by a waste disposal contractor (Clean Harbors). A pump truck with integrated tank was used to remove the wastewater. Following the wastewater removal the tractor was brought back to the site and the hydrolysis unit was hooked up to the tractor. Next the unit was unblocked and removed, along with all remaining supplies. Empty acid and caustic drums were returned to the supplier for their deposit. Treated BDU's were piled in an area designated for the test by BMGR and were left as per their instructions. Rental equipment was returned to the vendor, equipment decontamination was not required. The only other evidence that the process had been there were footprints. Site decontamination and/or restoration were not required.

4.7 Selection of Analytical/Testing Methods

All liquid and solid samples were to be analyzed using EPA Office of Soil and Water (SW) Methodologies, by third party laboratories that are EPA certified. For explosives EPA method SW8330 was used, and for metals a TCLP extraction (SW1311) followed by analysis (SW6010B with analysis for Mercury SW7470). The samples taken from the process tanks indicated in Table 3 were accidentally destroyed before they could be shipped to the laboratory. However, the sample of the combined neutralized process solution was analyzed by Clean Harbors and qualified for disposal as non-hazardous waste. All of these are standard, unmodified stand-alone procedures. Gas samples were analyzed using Dragger tubes for Hydrogen, carbon monoxide, carbon dioxide and phosphine, which are standard, unmodified stand-alone procedures.

4.8 Selection of Analytical Testing Laboratory

The following laboratory was used for analysis of laboratory test results:

GPL Laboratories, LLLP.
202 Perry Parkway
Gaithersburg, MD 20877
Tel: 301.926.6802 ~ Fax: 301.840.1209

Their EPA ID number is MD0016

UXB selected this lab because of its excellent service during prior work, and cost considerations.

5 Performance Assessment

5.1 Performance Criteria

For the test to be considered a success, all of the energetic character of any dud BDU-33 practice bombs processed must be removed. This means that the spotting charge case must be breached by the caustic; the propellant within the spotting charge must be hydrolyzed to a non-hazardous condition (non-reactive to a UN Test Series 3c(a) BAM Fallhammer impact test at 2 kg and 100 cm) if any remains, the primer charge and TiCl₄ smoke ampoule (if present) must be recovered, and any remaining RP must be below reactive levels.

Table 4: Performance Criteria

Performance Criteria	Description	Primary or Secondary	Criteria Satisfied
Residual Contamination level	Detectability of propellant components in BDU or liquid/solid samples	Primary	No
Hazardous Materials	Remaining RP in sludge is below reactive levels (UN test series 3(a)(ii))	Primary	Yes
Hazardous Materials	All live primers are recovered	Primary	Yes
Hazardous Materials	Expend and neutralized caustic is shippable as non-hazardous waste	Secondary	Yes

As Table 4, Performance Criteria, indicates, the residual contamination level criteria for detectability of reactive propellant components in the BDU or liquid/solid samples was not satisfied as residual spotting cartridge propellant was found in two (2) of the 480 units processed (0.4%). The quantity of residual propellant remaining in both units was in gram size quantities.

Performance Confirmation Methods

Table 5: Performance Confirmation Methods

Performance Criteria	Expected Performance Metric (pre demo)	Performance Confirmation Method	Actual (post demo)
PRIMARY CRITERIA (Performance Objectives)(Qualitative)			
Residual Contamination level	<MDL (Method detection limit)	EPA SW8330	Non-detect (criteria Satisfied)
Hazardous materials (RP)	No reaction	UN test series 3(a)(ii)	Non-detect (criteria Satisfied) ⁷
Hazardous Materials (Primers)	No live primers remaining in BDUs	Visual confirmation	Live primers did remain (criteria not satisfied)
Hazardous Materials (Neutralized Caustic)	Neutralized Caustic can be shipped as non-hazardous waste	EPA SW8330, SW1311 SW6010B, SW7470	Neutralized Caustic was shipped as non-hazardous waste

5.2 Data Analysis, Interpretation, and Evaluation

The process was evaluated to determine required process variables. Following treatment the clean scrap was evaluated to determine its value. After neutralization, the liquid by-products were analyzed by Clean Harbors Environmental Services, Inc. for disposal as non-hazardous waste (Appendix E). During operations, gasses evolved were analyzed for hydrogen, carbon monoxide, carbon dioxide and phosphine. All gases were non detectable using the Draeger tube sampling/analysis methodology.

Overall the process failed to achieve its objectives. The failure was due to the inability of the caustic fluid to penetrate certain blockages of some dropped but still live BDU's. Figure 5 below illustrates the problem.

⁷ No RP could be found to test

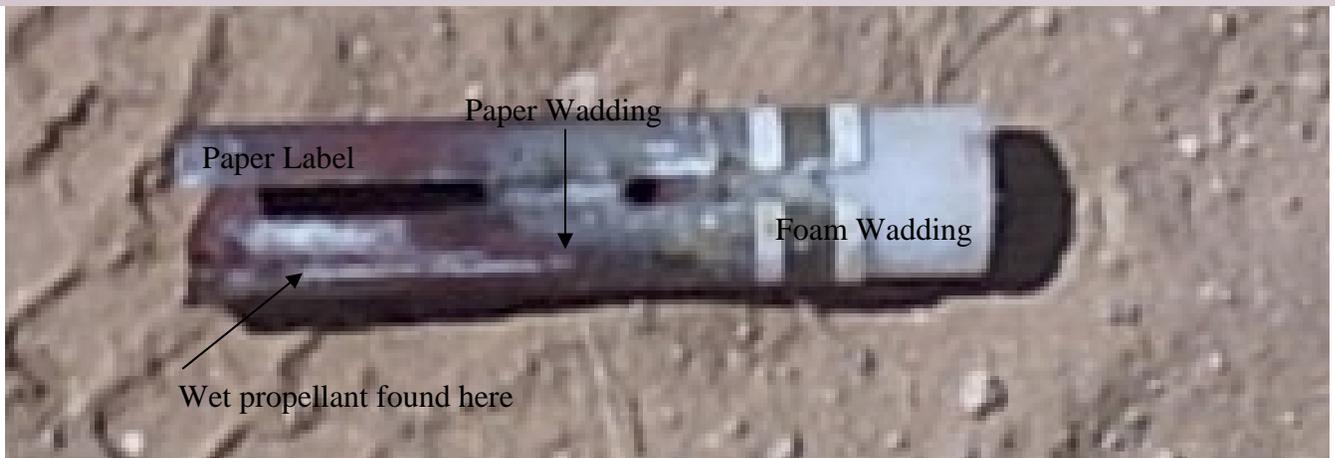


Figure 5: Example of Processed Cartridge Propellant Residue Location

In the upper photo a KC-4 cartridge is shown in as-built configuration. In the lower photo, a KC-4 cartridge is shown after treatment, as it was removed from a treated BDU. As can be seen, the aluminum case has been completely consumed. The primer was recovered, however some wet propellant was also found in the BDU. This wet propellant was not energetic either while wet or after drying, however its presence causes the process to fail its acceptance criteria. The fact that propellant does remain in some cases means that inspection would be required to verify that the process has worked as intended for each and every BDU treated, which largely negates

the projected cost savings from the process, and does not decrease the hazards associated with recycling BDU's over the present practice.

The entire problem with the process is easily overcome however, and can be done in a manner which also enhances the resale value of the residual scrap metal. When this project was first proposed in FY 2001, UXB had not yet developed our LOADS system. The LOADS system, now fully developed and fielded, uses mechanical force to break open the BDU body in the manner of cracking a nut, which accomplishes several objectives. First and foremost, this action separates the BDU body materials from the spotting cartridge. In addition, it renders the body of the BDU unrecognizable as being a munition or munition part so that it will be acceptable by metal recyclers, and thirdly it separates the BDU fins from the body materials so that the different types of metal can be segregated prior to recycling, thus enhancing their resale value. The LOADS system suffers however in that when the BDU body is opened, if the spotting cartridge is live it will fall out intact, thus presenting a disposal problem. The optimum solution then would be to combine the LOADS system with the ESTCP hydrolysis system to develop a complete solution. Spotting cartridges which remain live after removal from the BDU will be placed into the hydrolysis bath and destroyed. As there will be no physical hindrance to prevent the caustic from contacting all parts of the cartridge, destruction will be assured. UXB proposes to try this procedure the next time that the LOADS system is deployed to treat BDUs. In addition, UXB would like to use the hydrolysis system to treat and destroy 40mm grenades, which are aluminum cased and would respond well to treatment via hydrolysis.

6 Cost Assessment

6.1 Cost Reporting

Costs for the process are forecast to be very low, consisting mostly of the labor required to handle the scrap. Chemical costs are minimal, as nearly all of the chemicals are re-used from batch to batch; the only replenishment needed will be to replace and neutralize small amounts of caustic lost from the caustic tank due to drag-out (liquid clinging to the scrap after removal and drainage from the caustic tank). Disposal costs for the process will also be very low, again as all chemicals are re-used, and the scrap will be recycled. Permitting issues will be minimal. The process would also be applicable for decontamination of other range-derived scrap (metal, wood, target materials, etc.), which could be performed at the same time.

The test will also include the monitoring and documentation of all costs involved in the process (Table 6) so that a cost comparison to current BDU-33 disposal practices can be made.

Table 6: Cost Tracking

Cost Category	Sub Category	Costs
Start-Up Costs	Mobilization	\$8,104.68
Capital Costs	Capital Equipment Purchase	\$49,872.01
	Ancillary Equipment Purchase	\$18,658.60
	Modifications	
	Installation	\$23,173.06
	Engineering	\$69,852.00
Operating Costs Direct Environmental Activity Costs	Capital Equipment Rental	\$2,169.50
	Ancillary Equipment Rental	
	Supervision	\$3,392.30
	Site Setup	\$2,211.53
	Processing	\$8,496.63
		(\$17.70/BDU-33 or \$1,699.33/ton)
	Operator Training	\$914.38
	Maintenance	\$2,180.00
	Process Chemicals	\$431.85
	Consumables, Supplies	\$960.35
	Residual Waste Handling	
	Offsite Disposal	\$4,507.95
	Sampling and Analysis	\$527.00
Indirect Environmental Activity Costs	Environmental and Safety Training	\$914.38
	Waste Manifesting	
Demobilization		\$3,279.49
Other		

6.2 Cost Analysis

This section describes the approach for developing an estimated life-cycle cost for the technology. This section includes the following considerations: (1) facility capital cost, (2) startup, mobilization, operations and maintenance, and demobilization costs, and (3) recurring regulatory or institutional oversight costs. The analysis will be performed for an 8-year period.

6.2.1 Cost Comparison

The following scenario quantifies the potential cost savings (Table 7) if using this technology on a hypothetical 100-acre practice bombing range clearance effort (100 tons BDU's).

Table 7: Potential Cost Savings

Task	Present Method	UXBase SM process	
		4 tons/day	2 tons/day
Mobilize/demobilize UXO Techs and equipment to site	\$ 21,550	\$ 11,870	\$ 11,870
Clear Range of UXO	\$ 81,342	\$ 61,164	\$ 61,164
Process BDUs	N/A	\$ 36,088	\$ 72,177
<u>Inspect, countercharge, certify</u>	<u>\$ 32,537</u>	<u>\$ N/A</u>	<u>\$ N/A</u>
Total	\$ 135,429	\$ 114,129	\$ 150,218
	Potential Savings	\$ 21,300	\$ (14,789)

6.2.2 Cost Basis

Basis: Assumes UXB is purchaser/owner/operator of UXBaseSM process equipment. The number of EOD Technicians required on-site is reduced, as detonation of bombs is not required. Clearing and inerting operations can be performed concurrently with UXBaseSM process. This scenario does not include cost savings for concurrent range derived scrap decontamination. Note that this analysis assumes that the process works as designed. If the treated BDUs require inspection and certification after treatment, there are no cost savings and additional costs above the conventional methods would be expected.

6.2.3 Cost Drivers

Major cost drivers are labor costs, chemical costs, and disposal costs.

6.2.4 Life Cycle Costs

Life cycle costing
(4 tons/day)

Capital costs (includes engineering)	\$ 161,555.67
Operating costs (8 years) (Assumes 5 deployments per year, 100 tons BDU each deployment, includes Mob/Demob)	\$2,506,615.20
Total	\$2,668,170.87
Total per year	\$ 333,521.36
less scrap value for metal (2005 rates)	\$ 125,000.00
Grand Total per year	\$ 208,521.36
Total per ton BDU	\$ 2,085.21
Total per BDU	\$ 26.07

An 8-year life cycle was utilized.

7 Implementation Issues

7.1 Environmental Checklist

There are no permits or regulations that impact this technology. In a check of all 50 states, the only state which would require a permit is California, where the DTSC would require a Transportable Treatment Unit (TTU) permit for the unit.

7.2 Other Regulatory Issues

There was no public participation for this demonstration.

7.3 End-User Issues

The end-users for this technology will be two types, Government entities (EOD units) and contractors employing ex-EOD personnel. For EOD units, the technology must be robust, easy to use, and quick. For contractors, the forgoing is true, plus the process must be inexpensive, so that the contractor can make a profit, and/or get enough contracts (work) to justify the capital expenditure required.

From a contractor viewpoint, the largest concern is for efficacy. The process must work, and must work well for the process to be a success. The demonstration showed that the technology did not work well enough in its present configuration for successful implementation. The

UXB International, Inc.

technology can be modified however to address these shortcomings, see section 5.2. The technology that the system is based on is not new, and there are no patents or other intellectual property issues that need to be addressed. All equipment for the unit is available as commercial off the shelf units, except for the caustic and rinse tanks, which are special order items. The unit is built as a full size system, so that scale-up considerations are not required.

Technology transfer will be accomplished by the continued use and demonstration of the unit by UXB after the ESTCP project, and after suitable modifications to the process.

UXB intends to perform a controlled test of the Base Hydrolysis unit with our LOADS process as described in Section 5.2. Once an opportunity for this test is identified UXB will provide ESTCP a work plan and test report on the effort. We anticipate that a project of opportunity will occur sometime during calendar year 2006.

8 References

No papers published to date

9 Points of Contact

Point of Contact Name	Organization Name Address	Phone/Fax/E-Mail	Role in Project
Theodore Rekart	UXB International, Inc. 1715 Pratt Dr., Ste 1300 Blacksburg, VA 24060	Phone: (540) 443-3700 Fax: (540) 443-3790 E-Mail: Ted.Rekart@uxb.com	PM

Signature of Project Lead



June 12, 2006

Theodore E. Rekart

Date

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ⁱ P.C. Dell'Orco, et. al, *Demilitarization of High Explosives*, Progress Report 1995, Joint DOD/DOE Munitions technology Program

ⁱⁱ P.C. Dell'Orco, et al, *Base Hydrolysis and Hydrothermal processing of Composition B-3 Explosive*, Progress Report 1994, Joint DOD/DOE Munitions Technology Program

ⁱⁱⁱ R. Flesner, et al, *Base Hydrolysis and Hydrothermal Processing of PBX-9404*, Industrial and Engineering Chemistry Special Symposium, Atlanta, Georgia, September 1994).

^{iv} UXB Labs

^v GPL Laboratories, Gaithersburg, Maryland

^{vi} UXB Lab BAM Friction Apparatus

^{vii} UXB Lab BAM Fallhammer

^{viii} UXB Lab Modified Micro Flammability Test

^{ix} GPL Labs, Gaithersburg, Maryland

10 Attachments

Attachment 1

UXBaseSM Mobile BDO Treatment Unit

Appendix A:
Analytical Methods Supporting Project

METHOD 7471A

MERCURY IN SOLID OR SEMISOLID WASTE (MANUAL COLD-VAPOR TECHNIQUE)

1.0 SCOPE AND APPLICATION

1.1 Method 7471 is approved for measuring total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution step prior to analysis. If this dissolution procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the solid or semi-solid samples must be prepared according to the procedures discussed in this method.

2.2 Method 7471, a cold-vapor atomic absorption method, is based on the absorption of radiation at the 253.7-nm wavelength by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical instrument detection limit (IDL) for this method is 0.0002 mg/L.

3.0 INTERFERENCES

3.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/Kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.

3.3 Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL). In addition, the dead air space in the BOD bottle must be purged before adding stannous sulfate.

3.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

4.0 APPARATUS AND MATERIALS

4.1 Atomic absorption spectrophotometer or equivalent: Any atomic absorption unit with an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed. Instruments designed specifically for the measurement of mercury using the cold-vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

4.2 Mercury hollow cathode lamp or electrodeless discharge lamp.

4.3 Recorder: Any multirange variable-speed recorder that is compatible with the UV detection system is suitable.

4.4 Absorption cell: Standard spectrophotometer cells 10 cm long with quartz end windows may be used. Suitable cells may be constructed from Plexiglas tubing, 1 in. O.D. x 4.5 in. The ends are ground perpendicular to the longitudinal axis, and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of two 2-in. x 2-in. cards. One-in.-diameter holes are cut in the middle of each card. The cards are then placed over each end of the cell. The cell is then positioned and adjusted vertically and horizontally to give the maximum transmittance.

4.5 Air pump: Any peristaltic pump capable of delivering 1 L/min air may be used. A Masterflex pump with electronic speed control has been found to be satisfactory.

4.6 Flowmeter: Capable of measuring an air flow of 1 L/min.

4.7 Aeration tubing: A straight glass frit with a coarse porosity. Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return.

4.8 Drying tube: 6-in. x 3/4-in.-diameter tube containing 20 g of magnesium perchlorate or a small reading lamp with 60-W bulb which may be used to prevent condensation of moisture inside the cell. The lamp should be positioned to shine on the absorption cell so that the air temperature in the cell is about 10°C above ambient.

4.9 The cold-vapor generator is assembled as shown in Figure 1 of reference 1 or according to the instrument manufacturers instructions. The apparatus shown in Figure 1 is a closed system. An open system, where the mercury vapor is passed through the absorption cell only once, may be used instead of the closed system. Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system either to vent the mercury vapor into an exhaust hood or to pass the vapor through some absorbing medium, such as:

1. equal volumes of 0.1 M KMnO_4 and 10% H_2SO_4 , or
2. 0.25% iodine in a 3% KI solution.

A specially treated charcoal that will adsorb mercury vapor is also available from Barneby and Cheney, East 8th Avenue and North Cassidy Street, Columbus, Ohio 43219, Cat. #580-13 or #580-22.

4.10 Hot plate or equivalent - Adjustable and capable of maintaining a temperature of 90-95°C.

4.11 Graduated cylinder or equivalent.

5.0 REAGENTS

5.1 Reagent Water: Reagent water will be interference free. All references to water in this method refer to reagent water unless otherwise specified.

5.2 Aqua regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO₃.

5.3 Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated sulfuric acid to 1 liter.

5.4 Stannous sulfate: Add 25 g stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should be stirred continuously during use. A 10% solution of stannous chloride can be substituted for stannous sulfate.

5.5 Sodium chloride-hydroxylamine sulfate solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in reagent water and dilute to 100 mL. Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.

5.6 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 mL of reagent water.

5.7 Mercury stock solution: Dissolve 0.1354 g of mercuric chloride in 75 mL of reagent water. Add 10 mL of concentrated nitric acid and adjust the volume to 100.0 mL (1.0 mL = 1.0 mg Hg).

5.8 Mercury working standard: Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 ug/mL. This working standard and the dilution of the stock mercury solutions should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. This acid should be added to the flask, as needed, before adding the aliquot.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and reagent water. Plastic and glass containers are both suitable.

6.3 Non-aqueous samples shall be refrigerated, when possible, and analyzed as soon as possible."

7.0 PROCEDURE

7.1 Sample preparation: Weigh triplicate 0.2-g portions of untreated sample and place in the bottom of a BOD bottle. Add 5 mL of reagent water and 5 mL of aqua regia. Heat 2 min in a water bath at 95°C. Cool; then add 50 mL reagent water and 15 mL potassium permanganate solution to each sample bottle. Mix thoroughly and place in the water bath for 30 min at 95°C. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate.

CAUTION: Do this addition under a hood, as Cl_2 could be evolved. Add 55 mL of reagent water. Treating each bottle individually, add 5 mL of stannous sulfate and immediately attach the bottle to the aeration apparatus. Continue as described under step 7.4.

7.2 An alternate digestion procedure employing an autoclave may also be used. In this method, 5 mL of concentrated H_2SO_4 and 2 mL of concentrated HNO_3 are added to the 0.2 g of sample. Add 5 mL of saturated KMnO_4 solution and cover the bottle with a piece of aluminum foil. The samples are autoclaved at 121°C and 15 lb for 15 min. Cool, dilute to a volume of 100 mL with reagent water, and add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. Purge the dead air space and continue as described under step 7.4. Refer to the caution statement in section 7.1 for the proper protocol in reducing the excess permanganate solution and adding stannous sulfate.

7.3 Standard preparation: Transfer 0.0-, 0.5-, 1.0-, 2.0-, 5.0-, and 10-mL aliquots of the mercury working standard, containing 0-1.0 ug of mercury, to a series of 300-mL BOD bottles or equivalent. Add enough reagent water to each bottle to make a total volume of 10 mL. Add 5 mL of aqua regia and heat 2 min in a water bath at 95°C. Allow the sample to cool; add 50 mL reagent water and 15 mL of KMnO_4 solution to each bottle and return to the water bath for 30 min. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. Add 50 mL of reagent water. Treating each bottle individually, add 5 mL of stannous sulfate solution, immediately attach the bottle to the aeration apparatus, and continue as described in Step 7.4.

7.4 Analysis: At this point, the sample is allowed to stand quietly without manual agitation. The circulating pump, which has previously been adjusted to a rate of 1 L/min, is allowed to run continuously. The absorbance, as exhibited either on the spectrophotometer or the recorder, will increase and reach maximum within 30 sec. As soon as the recorder pen levels off (approximately 1 min), open the bypass valve and continue the aeration until the absorbance returns to its minimum value. Close the bypass valve, remove the fritted tubing from the BOD bottle, and continue the aeration.

7.5 Construct a calibration curve by plotting the absorbances of standards versus micrograms of mercury. Determine the peak height of the unknown from the chart and read the mercury value from the standard curve. Duplicates, spiked samples, and check standards should be routinely analyzed.

7.6 Calculate metal concentrations: (1) by the method of standard additions, (2) from a calibration curve, or (3) directly from the instrument's concentration read-out. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 ug/g dry weight).

8.0 QUALITY CONTROL

8.1 Refer to section 8.0 of Method 7000.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 245.5 of Methods for Chemical Analysis of Water and Wastes.

9.2 The data shown in Table 1 were obtained from records of state and contractor laboratories. The data are intended to show the precision of the combined sample preparation and analysis method.

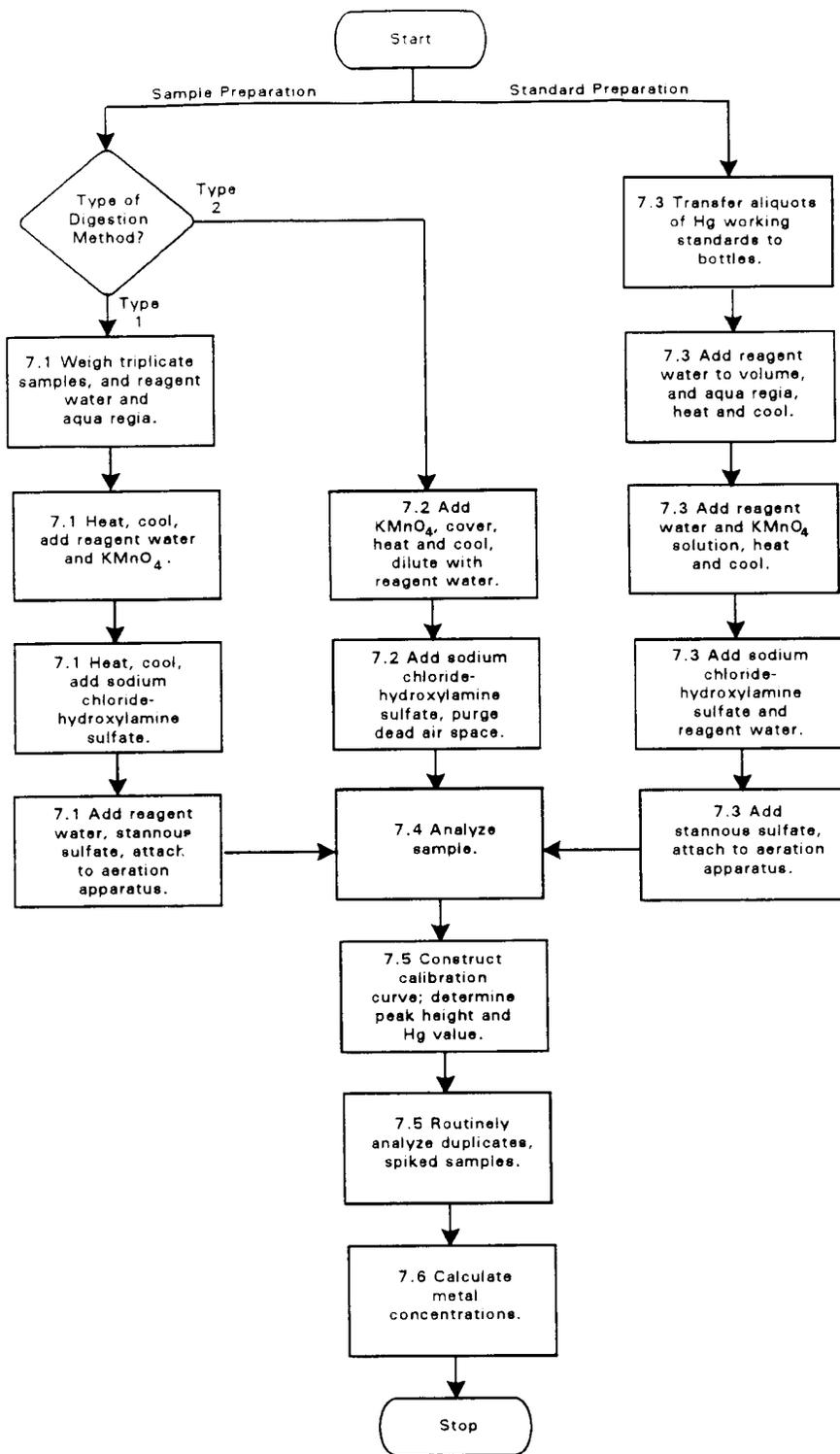
10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 245.5.
2. Gaskill, A., Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.

TABLE 1. METHOD PERFORMANCE DATA

Sample Matrix	Preparation Method	Laboratory Replicates
Emission control dust	Not known	12, 12 ug/g
Wastewater treatment sludge	Not known	0.4, 0.28 ug/g

METHOD 7471A
 MERCURY IN SOLID OR SEMISOLID WASTE (MANUAL COLD-VAPOR TECHNIQUE)



METHOD 7470A

MERCURY IN LIQUID WASTE (MANUAL COLD-VAPOR TECHNIQUE)

1.0 SCOPE AND APPLICATION

1.1 Method 7470 is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. (Method 7470 can also be used for analyzing certain solid and sludge-type wastes; however, Method 7471 is usually the method of choice for these waste types.) All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the liquid samples must be prepared according to the procedure discussed in this method.

2.2 Method 7470, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical detection limit for this method is 0.0002 mg/L.

3.0 INTERFERENCES

3.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

3.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL). In addition, the dead air space in the BOD bottle must be purged before adding stannous sulfate. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

3.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

4.0 APPARATUS AND MATERIALS

4.1 Atomic absorption spectrophotometer or equivalent: Any atomic absorption unit with an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed. Instruments designed specifically for the measurement of mercury using the cold-vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

4.2 Mercury hollow cathode lamp or electrodeless discharge lamp.

4.3 Recorder: Any multirange variable-speed recorder that is compatible with the UV detection system is suitable.

4.4 Absorption cell: Standard spectrophotometer cells 10 cm long with quartz end windows may be used. Suitable cells may be constructed from Plexiglas tubing, 1 in. O.D. x 4.5 in. The ends are ground perpendicular to the longitudinal axis, and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of two 2-in. x 2-in. cards. One-in.-diameter holes are cut in the middle of each card. The cards are then placed over each end of the cell. The cell is then positioned and adjusted vertically and horizontally to give the maximum transmittance.

4.5 Air pump: Any peristaltic pump capable of delivering 1 liter air/min may be used. A Masterflex pump with electronic speed control has been found to be satisfactory.

4.6 Flowmeter: Capable of measuring an air flow of 1 liter/min.

4.7 Aeration tubing: A straight glass frit with a coarse porosity. Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return.

4.8 Drying tube: 6-in. x 3/4-in.-diameter tube containing 20 g of magnesium perchlorate or a small reading lamp with 60-W bulb which may be used to prevent condensation of moisture inside the cell. The lamp should be positioned to shine on the absorption cell so that the air temperature in the cell is about 10°C above ambient.

4.9 The cold-vapor generator is assembled as shown in Figure 1 of reference 1 or according to the instrument manufacturers instructions. The apparatus shown in Figure 1 is a closed system. An open system, where the mercury vapor is passed through the absorption cell only once, may be used instead of the closed system. Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system either to vent the mercury vapor into an exhaust hood or to pass the vapor through some absorbing medium, such as:

1. Equal volumes of 0.1 M KMnO_4 and 10% H_2SO_4 ; or
2. 0.25% Iodine in a 3% KI solution.

A specially treated charcoal that will adsorb mercury vapor is also available from Barnebey and Cheney, East 8th Avenue and North Cassidy Street, Columbus, Ohio 43219, Cat. #580-13 or #580-22.

4.10 Hot plate or equivalent - Adjustable and capable of maintaining a temperature of 90-95°C.

4.11 Graduated cylinder or equivalent.

5.0 REAGENTS

5.1 Reagent Water: Reagent water will be interference free. All references to water in this method will refer to reagent water unless otherwise specified.

5.2 Sulfuric acid (H_2SO_4), concentrated: Reagent grade.

5.3 Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated sulfuric acid to 1.0 liter.

5.4 Nitric acid (HNO_3), concentrated: Reagent grade of low mercury content. If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

5.5 Stannous sulfate: Add 25 g stannous sulfate to 250 mL of 0.5 N H_2SO_4 . This mixture is a suspension and should be stirred continuously during use. (Stannous chloride may be used in place of stannous sulfate.)

5.6 Sodium chloride-hydroxylamine sulfate solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in reagent water and dilute to 100 mL. (Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.)

5.7 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 mL of reagent water.

5.8 Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate in 100 mL of reagent water.

5.9 Stock mercury solution: Dissolve 0.1354 g of mercuric chloride in 75 mL of reagent water. Add 10 mL of concentrated HNO_3 and adjust the volume to 100.0 mL (1 mL = 1 mg Hg). Stock solutions may also be purchased.

5.10 Mercury working standard: Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 ug per mL. This working standard and the dilutions of the stock mercury solution should be prepared fresh daily. Acidity of the working standard should be maintained at

0.15% nitric acid. This acid should be added to the flask, as needed, before addition of the aliquot.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and reagent water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH <2 with HNO₃. The suggested maximum holding times for mercury is 28 days.

6.4 Nonaqueous samples shall be refrigerated, when possible, and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Sample preparation: Transfer 100 mL, or an aliquot diluted to 100 mL, containing <1.0 g of mercury, to a 300-mL BOD bottle or equivalent. Add 5 mL of H₂SO₄ and 2.5 mL of concentrated HNO₃, mixing after each addition. Add 15 mL of potassium permanganate solution to each sample bottle. Sewage samples may require additional permanganate. Ensure that equal amounts of permanganate are added to standards and blanks. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 min. Add 8 mL of potassium persulfate to each bottle and heat for 2 hr in a water bath maintained at 95°C. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. After a delay of at least 30 sec, add 5 mL of stannous sulfate, immediately attach the bottle to the aeration apparatus, and continue as described in Paragraph 7.3.

7.2 Standard preparation: Transfer 0-, 0.5-, 1.0-, 2.0-, 5.0-, and 10.0-mL aliquots of the mercury working standard, containing 0-1.0 ug of mercury, to a series of 300-mL BOD bottles. Add enough reagent water to each bottle to make a total volume of 100 mL. Mix thoroughly and add 5 mL of concentrated H₂SO₄ and 2.5 mL of concentrated HNO₃ to each bottle. Add 15 mL of KMnO₄ solution to each bottle and allow to stand at least 15 min. Add 8 mL of potassium persulfate to each bottle and heat for 2 hr in a water bath maintained at 95°C. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. When the solution has been decolorized, wait 30 sec, add 5 mL of the stannous sulfate solution, immediately attach the bottle to the aeration apparatus, and continue as described in Paragraph 7.3.

7.3 Analysis: At this point the sample is allowed to stand quietly without manual agitation. The circulating pump, which has previously been adjusted to a rate of 1 liter/min, is allowed to run continuously. The absorbance will increase and reach a maximum within 30 sec. As soon as the recorder pen levels off (approximately 1 min), open the bypass valve and continue the aeration until the absorbance returns to its minimum value. Close the bypass

valve, remove the stopper and frit from the BOD bottle, and continue the aeration. Because of instrument variation refer to the manufacturers recommended operating conditions when using this method.

7.4 Construct a calibration curve by plotting the absorbances of standards versus micrograms of mercury. Determine the peak height of the unknown from the chart and read the mercury value from the standard curve. Duplicates, spiked samples, and check standards should be routinely analyzed.

7.5 Calculate metal concentrations (1) by the method of standard additions, or (2) from a calibration curve. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 ug/g dry weight).

8.0 QUALITY CONTROL

8.1 Refer to section 8.0 of Method 7000.

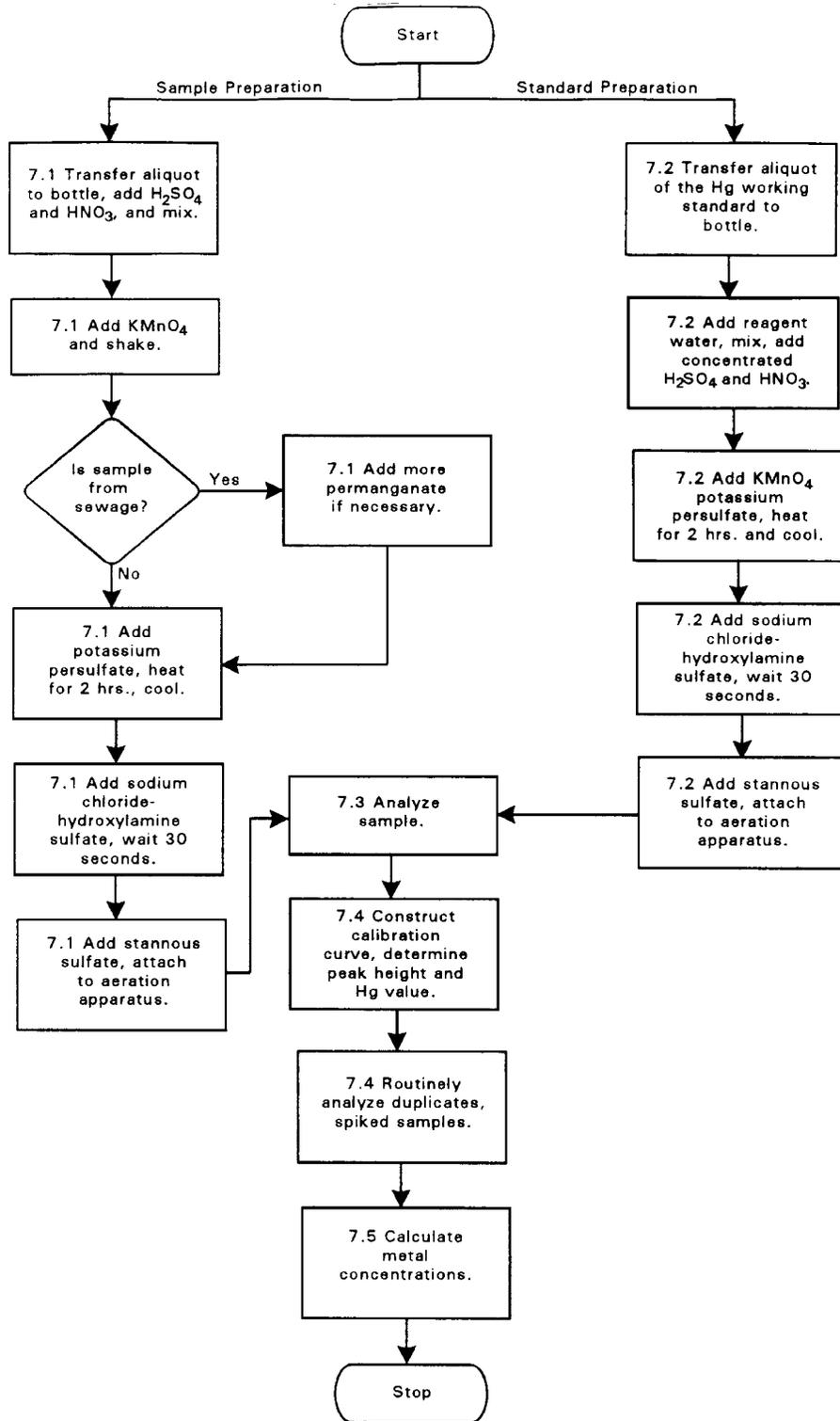
9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 245.1 of Methods for Chemical Analysis of Water and Wastes.

10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 245.1.

METHOD 7470A
 MERCURY IN LIQUID WASTE (MANUAL COLD-VAPOR TECHNIQUE)



METHOD 6010B

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion. Samples which are not digested must either use an internal standard or be matrix matched with the standards. Refer to Chapter Three for the appropriate digestion procedures.

1.2 Table 1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the recommended analytical wavelengths and estimated instrumental detection limits for the elements in clean aqueous matrices. The instrument detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements and matrices other than those listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest (see Section 8.0) is demonstrated.

1.3 Users of the method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using the method for analysis.

1.4 Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g. Chapter Three). When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2 This method describes multielemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used should be as free as possible from spectral interference and should reflect the same change in background

intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 3.0 should also be recognized and appropriate corrections made; tests for their presence are described in Section 8.5. Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

3.0 INTERFERENCES

3.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

3.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

3.1.2 To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

3.1.3 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction **require** the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement

correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

3.1.4 When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects must be evaluated for each individual instrument since the intensities will vary.

3.1.5 Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.

3.1.6 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences (Table 2) as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

3.1.7 Users of sequential instruments must verify the absence of spectral interference by scanning over a range of 0.5 nm centered on the wavelength of interest for several samples. The range for lead, for example, would be from 220.6 to 220.1 nm. This procedure must be repeated whenever a new matrix is to be analyzed and when a new calibration curve using different instrumental conditions is to be prepared. Samples that show an elevated background emission across the range may be background corrected by applying a correction factor equal to the emission adjacent to the line or at two points on either side of the line and interpolating between them. An alternate wavelength that does not exhibit a background shift or spectral overlap may also be used.

3.1.8 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

3.1.9 When interelement corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions. If the correction factors or multivariate correction matrices tested on a daily basis are found to be within the 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at \pm one reporting limit from zero, daily verification is not required. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

3.1.10 When interelement corrections are not used, verification of absence of interferences is required.

3.1.10.1 One method is to use a computer software routine for comparing the determinative data to limits files for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration, (i.e., greater than) the analyte instrument detection limit, or false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

3.1.10.2 Another method is to analyze an Interference Check Solution(s) which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is \geq 20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

3.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate

and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers. The test described in Section 8.5.1 will help determine if a physical interference is present.

3.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

3.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

3.5 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

3.6 The dashes in Table 2 indicate that no measurable interferences were observed even at higher interferant concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

4.0 APPARATUS AND MATERIALS

4.1 Inductively coupled argon plasma emission spectrometer:

4.1.1 Computer-controlled emission spectrometer with background correction.

4.1.2 Radio-frequency generator compliant with FCC regulations.

4.1.3 Optional mass flow controller for argon nebulizer gas supply.

4.1.4 Optional peristaltic pump.

4.1.5 Optional Autosampler.

4.1.6 Argon gas supply - high purity.

4.2 Volumetric flasks of suitable precision and accuracy.

4.3 Volumetric pipets of suitable precision and accuracy.

5.0 REAGENTS

5.1 Reagent or trace metals grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration of the contamination is less than the MDL then the reagent is acceptable.

5.1.1 Hydrochloric acid (conc), HCl.

5.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an appropriately sized beaker.

5.1.3 Nitric acid (conc), HNO₃.

5.1.4 Nitric acid (1:1), HNO₃. Add 500 mL concentrated HNO₃ to 400 mL water and dilute to 1 liter in an appropriately sized beaker.

5.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Refer to Chapter One for a definition of reagent water.

5.3 Standard stock solutions may be purchased or prepared from ultra- high purity grade chemicals or metals (99.99% pure or greater). All salts must be dried for 1 hour at 105°C, unless otherwise specified.

Note: This section does not apply when analyzing samples that have been prepared by Method 3040.

CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly after handling.

Typical stock solution preparation procedures follow. Concentrations are calculated based upon the weight of pure metal added, or with the use of the element fraction and the weight of the metal salt added.

For metals:

$$\text{Concentration (ppm)} = \frac{\text{weight (mg)}}{\text{volume (L)}}$$

For metal salts:

$$\text{Concentration (ppm)} = \frac{\text{weight (mg)} \times \text{mole fraction}}{\text{volume (L)}}$$

5.3.1 Aluminum solution, stock, 1 mL = 1000 µg Al: Dissolve 1.000 g of aluminum metal, weighed accurately to at least four significant figures, in an acid mixture of 4.0 mL of (1:1) HCl and 1.0 mL of concentrated HNO₃ in a beaker. Warm beaker slowly to effect solution. When dissolution is complete, transfer solution quantitatively to a 1-liter flask, add an additional 10.0 mL of (1:1) HCl and dilute to volume with reagent water.

NOTE: Weight of analyte is expressed to four significant figures for consistency with the weights below because rounding to two decimal places can contribute up to 4 % error for some of the compounds.

5.3.2 Antimony solution, stock, 1 mL = 1000 µg Sb: Dissolve 2.6673 g K(SbO)C₄H₄O₆ (element fraction Sb = 0.3749), weighed accurately to at least four significant figures, in water, add 10 mL (1:1) HCl, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.3 Arsenic solution, stock, 1 mL = 1000 µg As: Dissolve 1.3203 g of As₂O₃ (element fraction As = 0.7574), weighed accurately to at least four significant figures, in 100 mL of water containing 0.4 g NaOH. Acidify the solution with 2 mL concentrated HNO₃ and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.4 Barium solution, stock, 1 mL = 1000 µg Ba: Dissolve 1.5163 g BaCl₂ (element fraction Ba = 0.6595), dried at 250°C for 2 hours, weighed accurately to at least four significant figures, in 10 mL water with 1 mL (1:1) HCl. Add 10.0 mL (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.5 Beryllium solution, stock, 1 mL = 1000 µg Be: Do not dry. Dissolve 19.6463 g BeSO₄·4H₂O (element fraction Be = 0.0509), weighed accurately to at least four significant figures, in water, add 10.0 mL concentrated HNO₃, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.6 Boron solution, stock, 1 mL = 1000 µg B: Do not dry. Dissolve 5.716 g anhydrous H₃BO₃ (B fraction = 0.1749), weighed accurately to at least four significant figures, in reagent water and dilute in a 1-L volumetric flask with reagent water. Transfer immediately after mixing in a clean polytetrafluoroethylene (PTFE) bottle to minimize any leaching of boron from the glass volumetric container. Use of a non-glass volumetric flask is recommended to avoid boron contamination from glassware.

5.3.7 Cadmium solution, stock, 1 mL = 1000 µg Cd: Dissolve 1.1423 g CdO (element fraction Cd = 0.8754), weighed accurately to at least four significant figures, in a

minimum amount of (1:1) HNO₃. Heat to increase rate of dissolution. Add 10.0 mL concentrated HNO₃ and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.8 Calcium solution, stock, 1 mL = 1000 µg Ca: Suspend 2.4969 g CaCO₃ (element Ca fraction = 0.4005), dried at 180°C for 1 hour before weighing, weighed accurately to at least four significant figures, in water and dissolve cautiously with a minimum amount of (1:1) HNO₃. Add 10.0 mL concentrated HNO₃ and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.9 Chromium solution, stock, 1 mL = 1000 µg Cr: Dissolve 1.9231 g CrO₃ (element fraction Cr = 0.5200), weighed accurately to at least four significant figures, in water. When solution is complete, acidify with 10 mL concentrated HNO₃ and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.10 Cobalt solution, stock, 1 mL = 1000 µg Co: Dissolve 1.00 g of cobalt metal, weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO₃. Add 10.0 mL (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.11 Copper solution, stock, 1 mL = 1000 µg Cu: Dissolve 1.2564 g CuO (element fraction Cu = 0.7989), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO₃. Add 10.0 mL concentrated HNO₃ and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.12 Iron solution, stock, 1 mL = 1000 µg Fe: Dissolve 1.4298 g Fe₂O₃ (element fraction Fe = 0.6994), weighed accurately to at least four significant figures, in a warm mixture of 20 mL (1:1) HCl and 2 mL of concentrated HNO₃. Cool, add an additional 5.0 mL of concentrated HNO₃, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.13 Lead solution, stock, 1 mL = 1000 µg Pb: Dissolve 1.5985 g Pb(NO₃)₂ (element fraction Pb = 0.6256), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO₃. Add 10 mL (1:1) HNO₃ and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.14 Lithium solution, stock, 1 mL = 1000 µg Li: Dissolve 5.3248 g lithium carbonate (element fraction Li = 0.1878), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.15 Magnesium solution, stock, 1 mL = 1000 µg Mg: Dissolve 1.6584 g MgO (element fraction Mg = 0.6030), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO₃. Add 10.0 mL (1:1) concentrated HNO₃ and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.16 Manganese solution, stock, 1 mL = 1000 µg Mn: Dissolve 1.00 g of manganese metal, weighed accurately to at least four significant figures, in acid mixture (10 mL concentrated HCl and 1 mL concentrated HNO₃) and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.17 Mercury solution, stock, 1 mL = 1000 µg Hg: Do not dry, highly toxic element. Dissolve 1.354 g HgCl₂ (Hg fraction = 0.7388) in reagent water. Add 50.0 mL concentrated HNO₃ and dilute to volume in 1-L volumetric flask with reagent water.

5.3.18 Molybdenum solution, stock, 1 mL = 1000 µg Mo: Dissolve 1.7325 g (NH₄)₆Mo₇O₂₄·4H₂O (element fraction Mo = 0.5772), weighed accurately to at least four significant figures, in water and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.19 Nickel solution, stock, 1 mL = 1000 µg Ni: Dissolve 1.00 g of nickel metal, weighed accurately to at least four significant figures, in 10.0 mL hot concentrated HNO₃, cool, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.20 Phosphate solution, stock, 1 mL = 1000 µg P: Dissolve 4.3937 g anhydrous KH₂PO₄ (element fraction P = 0.2276), weighed accurately to at least four significant figures, in water. Dilute to volume in a 1,000 mL volumetric flask with water.

5.3.21 Potassium solution, stock, 1 mL = 1000 µg K: Dissolve 1.9069 g KCl (element fraction K = 0.5244) dried at 110°C, weighed accurately to at least four significant figures, in water, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.22 Selenium solution, stock, 1 mL = 1000 µg Se: Do not dry. Dissolve 1.6332 g H₂SeO₃ (element fraction Se = 0.6123), weighed accurately to at least four significant figures, in water and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.23 Silica solution, stock, 1 mL = 1000 µg SiO₂: Do not dry. Dissolve 2.964 g NH₄SiF₆, weighed accurately to at least four significant figures, in 200 mL (1:20) HCl with heating at 85°C to effect dissolution. Let solution cool and dilute to volume in a 1-L volumetric flask with reagent water.

5.3.24 Silver solution, stock, 1 mL = 1000 µg Ag: Dissolve 1.5748 g AgNO₃ (element fraction Ag = 0.6350), weighed accurately to at least four significant figures, in water and 10 mL concentrated HNO₃. Dilute to volume in a 1,000 mL volumetric flask with water.

5.3.25 Sodium solution, stock, 1 mL = 1000 µg Na: Dissolve 2.5419 g NaCl (element fraction Na = 0.3934), weighed accurately to at least four significant figures, in water. Add 10.0 mL concentrated HNO₃ and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.26 Strontium solution, stock, 1 mL = 1000 µg Sr: Dissolve 2.4154 g of strontium nitrate (Sr(NO₃)₂) (element fraction Sr = 0.4140), weighed accurately to at least four significant figures, in a 1-liter flask containing 10 mL of concentrated HCl and 700 mL of water. Dilute to volume in a 1,000 mL volumetric flask with water.

5.3.27 Thallium solution, stock, 1 mL = 1000 µg Tl: Dissolve 1.3034 g TlNO₃ (element fraction Tl = 0.7672), weighed accurately to at least four significant figures, in water. Add 10.0 mL concentrated HNO₃ and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.28 Tin solution, stock, 1 mL = 1000 µg Sn: Dissolve 1.000 g Sn shot, weighed accurately to at least 4 significant figures, in 200 mL (1:1) HCl with heating to effect dissolution. Let solution cool and dilute with (1:1) HCl in a 1-L volumetric flask.

5.3.29 Vanadium solution, stock, 1 mL = 1000 µg V: Dissolve 2.2957 g NH_4VO_3 (element fraction V = 0.4356), weighed accurately to at least four significant figures, in a minimum amount of concentrated HNO_3 . Heat to increase rate of dissolution. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.30 Zinc solution, stock, 1 mL = 1000 µg Zn: Dissolve 1.2447 g ZnO (element fraction Zn = 0.8034), weighed accurately to at least four significant figures, in a minimum amount of dilute HNO_3 . Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.4 Mixed calibration standard solutions - Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks (see Table 3). Add the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to FEP fluorocarbon or previously unused polyethylene or polypropylene bottles for storage. Fresh mixed standards should be prepared, as needed, with the realization that concentration can change on aging. Some typical calibration standard combinations are listed in Table 3.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.

5.5 Two types of blanks are required for the analysis for samples prepared by any method other than 3040. The calibration blank is used in establishing the analytical curve, and the method blank is used to identify possible contamination resulting from varying amounts of the acids used in the sample processing.

5.5.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial and continuing calibration blank determinations (see Sections 7.3 and 7.4).

5.5.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

5.6 The Initial Calibration Verification (ICV) is prepared by the analyst by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument (see Section 8.6.1 for use).

5.7 The Continuing Calibration Verification (CCV) should be prepared in the same acid matrix using the same standards used for calibration at a concentration near the mid-point of the calibration curve (see Section 8.6.1 for use).

5.8 The interference check solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest, particularly those with known interferences at 0.5 to 1 mg/L. In the absence of measurable analyte, overcorrection could go undetected because a negative value could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure will not be necessary.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material in Chapter Three, Inorganic Analytes, Sections 3.1 through 3.3.

7.0 PROCEDURE

7.1 Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been prefiltered and acidified will not need acid digestion. Samples which are not digested must either use an internal standard or be matrix matched with the standards. Solubilization and digestion procedures are presented in Sample Preparation Methods (Chapter Three, Inorganic Analytes).

7.2 Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration). Operating conditions - The analyst should follow the instructions provided by the instrument manufacturer.

7.2.1 Before using this procedure to analyze samples, there must be data available documenting initial demonstration of performance. The required data document the selection criteria of background correction points; analytical dynamic ranges, the applicable equations, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences. This data must be generated using the same instrument, operating conditions and calibration routine to be used for sample analysis. These documented data must be kept on file and be available for review by the data user or auditor.

7.2.2 Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. Because of differences among various makes and models of spectrometers, specific instrument operating conditions cannot be provided. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality to the program and data user. The analyst should follow the instructions provided by the instrument manufacturer unless other conditions provide similar or better performance for

a task. Operating conditions for aqueous solutions usually vary from 1100 to 1200 watts forward power, 14 to 18 mm viewing height, 15 to 19 liters/min argon coolant flow, 0.6 to 1.5 L/min argon nebulizer flow, 1 to 1.8 mL/min sample pumping rate with a 1 minute preflush time and measurement time near 1 second per wavelength peak for sequential instruments and 10 seconds per sample for simultaneous instruments. For an axial plasma, the conditions will usually vary from 1100-1500 watts forward power, 15-19 liters/min argon coolant flow, 0.6-1.5 L/min argon nebulizer flow, 1-1.8 mL/min sample pumping rate with a 1 minute preflush time and measurement time near 1 second per wavelength peak for sequential instruments and 10 seconds per sample for simultaneous instruments. Reproduction of the Cu/Mn intensity ratio at 324.754 nm and 257.610 nm respectively, by adjusting the argon aerosol flow has been recommended as a way to achieve repeatable interference correction factors.

7.2.3 The plasma operating conditions need to be optimized prior to use of the instrument. This routine is not required on a daily basis, but only when first setting up a new instrument or following a change in operating conditions. The following procedure is recommended or follow manufacturer's recommendations. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure.

7.2.3.1 Ignite the radial plasma and select an appropriate incident RF power. Allow the instrument to become thermally stable before beginning, about 30 to 60 minutes of operation. While aspirating a 1000 ug/L solution of yttrium, follow the instrument manufacturer's instructions and adjust the aerosol carrier gas flow rate through the nebulizer so a definitive blue emission region of the plasma extends approximately from 5 to 20 mm above the top of the load coil. Record the nebulizer gas flow rate or pressure setting for future reference. The yttrium solution can also be used for coarse optical alignment of the torch by observing the overlay of the blue light over the entrance slit to the optical system.

7.2.3.2 After establishing the nebulizer gas flow rate, determine the solution uptake rate of the nebulizer in mL/min by aspirating a known volume of calibration blank for a period of at least three minutes. Divide the volume aspirated by the time in minutes and record the uptake rate; set the peristaltic pump to deliver the rate in a steady even flow.

7.2.3.3 Profile the instrument to align it optically as it will be used during analysis. The following procedure can be used for both horizontal and vertical optimization in the radial mode, but is written for vertical. Aspirate a solution containing 10 ug/L of several selected elements. These elements can be As, Se, Tl or Pb as the least sensitive of the elements and most needing to be optimize or others representing analytical judgement (V, Cr, Cu, Li and Mn are also used with success). Collect intensity data at the wavelength peak for each analyte at 1 mm intervals from 14 to 18 mm above the load coil. (This region of the plasma is referred to as the analytical zone.) Repeat the process using the calibration blank. Determine the net signal to blank intensity ratio for each analyte for each viewing height setting. Choose the height for viewing the plasma that provides the best net intensity ratios for the elements analyzed or the highest intensity ratio for the least

sensitive element. For optimization in the axial mode, follow the instrument manufacturer's instructions.

7.2.3.4 The instrument operating condition finally selected as being optimum should provide the lowest reliable instrument detection limits and method detection limits.

7.2.3.5 If either the instrument operating conditions, such as incident power or nebulizer gas flow rate are changed, or a new torch injector tube with a different orifice internal diameter is installed, the plasma and viewing height should be re-optimized.

7.2.3.6 After completing the initial optimization of operating conditions, but before analyzing samples, the laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description concerning spectral interference and the analytical requirements for background correction in particular are discussed in the section on interferences. Criteria for determining an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls within \pm one reporting limit from zero. The upper control limit is the analyte instrument detection limit. Once established the entire routine must be periodically verified every six months. Only a portion of the correction routine must be verified more frequently or on a daily basis. Initial and periodic verification of the routine should be kept on file. Special cases where continual verification is required are described elsewhere.

7.2.3.7 Before daily calibration and after the instrument warmup period, the nebulizer gas flow rate must be reset to the determined optimized flow. If a mass flow controller is being used, it should be set to the recorded optimized flow rate. In order to maintain valid spectral interelement correction routines the nebulizer gas flow rate should be the same (< 2% change) from day to day.

7.2.4 For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements.

7.2.5 Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

7.2.5.1 Method detection limits must be established for all wavelengths utilized for each type of matrix commonly analyzed. The matrix used for the MDL calculation must contain analytes of known concentrations within 3-5 times the anticipated detection limit. Refer to Chapter One for additional guidance on the performance of MDL studies.

7.2.5.2 Determination of limits using reagent water represent a best case situation and do not represent possible matrix effects of real world samples.

7.2.5.3 If additional confirmation is desired, reanalyze the seven replicate aliquots on two more non consecutive days and again calculate the method detection limit values for each day. An average of the three values for each analyte may provide for a more appropriate estimate. Successful analysis of samples with added analytes or using method of standard additions can give confidence in the method detection limit values determined in reagent water.

7.2.5.4 The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses from a minimum for three, preferably five, different concentration standards across the range. One of these should be near the upper limit of the range. The ranges which may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made should be documented and kept on file. The upper range limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Determined analyte concentrations that are above the upper range limit must be diluted and reanalyzed. The analyst should also be aware that if an interelement correction from an analyte above the linear range exists, a second analyte where the interelement correction has been applied may be inaccurately reported. New dynamic ranges should be determined whenever there is a significant change in instrument response. For those analytes that periodically approach the upper limit, the range should be checked every six months. For those analytes that are known interferences, and are present at above the linear range, the analyst should ensure that the interelement correction has not been inaccurately applied.

NOTE: Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self absorption effects. These curves may be used if the instrument allows; however the effective range must be checked and the second order curve fit should have a correlation coefficient of 0.995 or better. Third order fits are not acceptable. These non-linear response curves should be revalidated and recalculated every six months. These curves are much more sensitive to changes in operating conditions than the linear lines and should be checked whenever there have been moderate equipment changes.

7.2.6 The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

7.3 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 5.4. Flush the system with the calibration blank (Section 5.5.1) between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve must consist of a minimum of a blank and a standard.

7.4 For all analytes and determinations, the laboratory must analyze an ICV (Section 5.6), a calibration blank (Section 5.5.1), and a continuing calibration verification (CCV) (Section 5.7) immediately following daily calibration. A calibration blank and either a calibration verification (CCV) or an ICV must be analyzed after every tenth sample and at the end of the sample run. Analysis of

the check standard and calibration verification must verify that the instrument is within $\pm 10\%$ of calibration with relative standard deviation $< 5\%$ from replicate (minimum of two) integrations. If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV or check standard must be reanalyzed. The analysis data of the calibration blank, check standard, and ICV or CCV must be kept on file with the sample analysis data.

7.5 Rinse the system with the calibration blank solution (Section 5.5.1) before the analysis of each sample. The rinse time will be one minute. Each laboratory may establish a reduction in this rinse time through a suitable demonstration.

7.6 Calculations: If dilutions were performed, the appropriate factors must be applied to sample values. All results should be reported with up to three significant figures.

7.7 The MSA should be used if an interference is suspected or a new matrix is encountered. When the method of standard additions is used, standards are added at one or more levels to portions of a prepared sample. This technique compensates for enhancement or depression of an analyte signal by a matrix. It will not correct for additive interferences, such as contamination, interelement interferences, or baseline shifts. This technique is valid in the linear range when the interference effect is constant over the range, the added analyte responds the same as the endogenous analyte, and the signal is corrected for additive interferences. The simplest version of this technique is the single addition method. This procedure calls for two identical aliquots of the sample solution to be taken. To the first aliquot, a small volume of standard is added; while to the second aliquot, a volume of acid blank is added equal to the standard addition. The sample concentration is calculated by: multiplying the intensity value for the unfortified aliquot by the volume (Liters) and concentration (mg/L or mg/kg) of the standard addition to make the numerator; the difference in intensities for the fortified sample and unfortified sample is multiplied by the volume (Liters) of the sample aliquot for the denominator. The quotient is the sample concentration.

For more than one fortified portion of the prepared sample, linear regression analysis can be applied using a computer or calculator program to obtain the concentration of the sample solution.

NOTE: Refer to Method 7000 for a more detailed discussion of the MSA.

7.8 An alternative to using the method of standard additions is the internal standard technique. Add one or more elements not in the samples and verified not to cause an interelement spectral interference to the samples, standards and blanks; yttrium or scandium are often used. The concentration should be sufficient for optimum precision but not so high as to alter the salt concentration of the matrix. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation. This technique is very useful in overcoming matrix interferences especially in high solids matrices.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection. All quality control measures described in Chapter One should be followed.

8.2 Dilute and reanalyze samples that exceed the linear calibration range or use an alternate, less sensitive line for which quality control data is already established.

8.3 Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank is a volume of reagent water carried through the same preparation process as a sample (refer to Chapter One).

8.4 Analyze matrix spiked duplicate samples at a frequency of one per matrix batch. A matrix duplicate sample is a sample brought through the entire sample preparation and analytical process in duplicate.

8.4.1.1 The relative percent difference between spiked matrix duplicate determinations is to be calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

RPD = relative percent difference.

D_1 = first sample value.

D_2 = second sample value (replicate).

(A control limit of $\pm 20\%$ RPD or within the documented historical acceptance limits for each matrix shall be used for sample values greater than ten times the instrument detection limit.)

8.4.1.2 The spiked sample or spiked duplicate sample recovery is to be within $\pm 25\%$ of the actual value or within the documented historical acceptance limits for each matrix.

8.5 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in Sections 8.5.1 and 8.5.2, will ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

8.5.1 Dilution Test: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:5 dilution should agree within $\pm 10\%$ of the original determination. If not, a chemical or physical interference effect should be suspected.

8.5.2 Post Digestion Spike Addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

CAUTION: If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

8.6 Check the instrument standardization by analyzing appropriate QC samples as follows.

8.6.1 Verify calibration with the Continuing Calibration Verification (CCV) Standard immediately following daily calibration, after every ten samples, and at the end of an analytical run. Check calibration with an ICV following the initial calibration (Section 5.6). At the laboratory's discretion, an ICV may be used in lieu of the continuing calibration verifications. If used in this manner, the ICV should be at a concentration near the mid-point of the calibration curve. Use a calibration blank (Section 5.5.1) immediately following daily calibration, after every 10 samples and at the end of the analytical run.

8.6.1.1 The results of the ICV and CCVs are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument.

8.6.1.2 The results of the check standard are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument.

8.6.1.3 The results of the calibration blank are to agree within three times the IDL. If not, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be performed before continuation of the run.

8.6.2 Verify the interelement and background correction factors at the beginning of each analytical run. Do this by analyzing the interference check sample (Section 5.8). Results should be within $\pm 20\%$ of the true value.

9.0 METHOD PERFORMANCE

9.1 In an EPA round-robin Phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been spiked with various metal concentrates. Table 4 lists the true values, the mean reported values, and the mean percent relative standard deviations.

9.2 Performance data for aqueous solutions and solid samples from a multilaboratory study (9) are provided in Tables 5 and 6.

10.0 REFERENCES

1. Boumans, P.W.J.M. Line Coincidence Tables for Inductively Coupled Plasma Atomic Emission Spectrometry, 2nd Edition. Pergamon Press, Oxford, United Kingdom, 1984.
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TABLE 1
RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Detection Element	Wavelength ^a (nm)	Estimated IDL ^b (µg/L)
Aluminum	308.215	30
Antimony	206.833	21
Arsenic	193.696	35
Barium	455.403	0.87
Beryllium	313.042	0.18
Boron	249.678x2	3.8
Cadmium	226.502	2.3
Calcium	317.933	6.7
Chromium	267.716	4.7
Cobalt	228.616	4.7
Copper	324.754	3.6
Iron	259.940	4.1
Lead	220.353	28
Lithium	670.784	2.8
Magnesium	279.079	20
Manganese	257.610	0.93
Mercury	194.227x2	17
Molybdenum	202.030	5.3
Nickel	231.604x2	10
Phosphorus	213.618	51
Potassium	766.491	See note c
Selenium	196.026	50
Silica (SiO ₂)	251.611	17
Silver	328.068	4.7
Sodium	588.995	19
Strontium	407.771	0.28
Thallium	190.864	27
Tin	189.980x2	17
Titanium	334.941	5.0
Vanadium	292.402	5.0
Zinc	213.856x2	1.2

^aThe wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted (e.g., in the case of an interference) if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference (see Section 3.1). In time, other elements may be added as more information becomes available and as required.

^bThe estimated instrumental detection limits shown are provided as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

^cHighly dependent on operating conditions and plasma position.

TABLE 2
 POTENTIAL INTERFERENCES
 ANALYTE CONCENTRATION EQUIVALENTS ARISING FROM
 INTERFERENCE AT THE 100-mg/L LEVEL^c

Analyte	Wavelength (nm)	Interferant ^{a,b}									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

^a Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L	Mg - 1000 mg/L
Ca - 1000 mg/L	Mn - 200 mg/L
Cr - 200 mg/L	Ti - 200 mg/L
Cu - 200 mg/L	V - 200 mg/L
Fe - 1000 mg/L	

^b The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.

^c Interferences will be affected by background choice and other interferences may be present.

TABLE 3
MIXED STANDARD SOLUTIONS

Solution	Elements
I	Be, Cd, Mn, Pb, Se and Zn
II	Ba, Co, Cu, Fe, and V
III	As, Mo
IV	Al, Ca, Cr, K, Na, Ni, Li, and Sr
V	Ag (see "NOTE" to Section 5.4), Mg, Sb, and Tl
VI	P

TABLE 4. ICP PRECISION AND ACCURACY DATA^a

Element	Sample No. 1				Sample No. 2				Sample No. 3			
	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD ^b (%)	Accuracy ^d (%)	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD ^b (%)	Accuracy ^d (%)	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD ^b (%)	Accuracy ^d (%)
Be	750	733	6.2	98	20	20	9.8	100	180	176	5.2	98
Mn	350	345	2.7	99	15	15	6.7	100	100	99	3.3	99
V	750	749	1.8	100	70	69	2.9	99	170	169	1.1	99
As	200	208	7.5	104	22	19	23	86	60	63	17	105
Cr	150	149	3.8	99	10	10	18	100	50	50	3.3	100
Cu	250	235	5.1	94	11	11	40	100	70	67	7.9	96
Fe	600	594	3.0	99	20	19	15	95	180	178	6.0	99
Al	700	696	5.6	99	60	62	33	103	160	161	13	101
Cd	50	48	12	96	2.5	2.9	16	116	14	13	16	93
Co	700	512	10	73	20	20	4.1	100	120	108	21	90
Ni	250	245	5.8	98	30	28	11	93	60	55	14	92
Pb	250	236	16	94	24	30	32	125	80	80	14	100
Zn	200	201	5.6	100	16	19	45	119	80	82	9.4	102
Se ^c	40	32	21.9	80	6	8.5	42	142	10	8.5	8.3	85

^a Not all elements were analyzed by all laboratories.

^b RSD = relative standard deviation.

^c Results for Se are from two laboratories.

^d Accuracy is expressed as the mean concentration divided by the true concentration times 100.

TABLE 5
ICP-AES PRECISION AND ACCURACY FOR AQUEOUS SOLUTIONS^a

Element	Mean Conc. (mg/L)	N ^b	RSD ^b (%)	Accuracy ^c (%)
Al	14.8	8	6.3	100
Sb	15.1	8	7.7	102
As	14.7	7	6.4	99
Ba	3.66	7	3.1	99
Be	3.78	8	5.8	102
Cd	3.61	8	7.0	97
Ca	15.0	8	7.4	101
Cr	3.75	8	8.2	101
Co	3.52	8	5.9	95
Cu	3.58	8	5.6	97
Fe	14.8	8	5.9	100
Pb	14.4	7	5.9	97
Mg	14.1	8	6.5	96
Mn	3.70	8	4.3	100
Mo	3.70	8	6.9	100
Ni	3.70	7	5.7	100
K	14.1	8	6.6	95
Se	15.3	8	7.5	104
Ag	3.69	6	9.1	100
Na	14.0	8	4.2	95
Tl	15.1	7	8.5	102
V	3.51	8	6.6	95
Zn	3.57	8	8.3	96

^athese performance values are independent of sample preparation because the labs analyzed portions of the same solutions

^bN = Number of measurements for mean and relative standard deviation (RSD).

^cAccuracy is expressed as a percentage of the nominal value for each analyte in acidified, multi-element solutions.

TABLE 6

ICP-AES PRECISION AND BIAS FOR SOLID WASTE DIGESTS^a

Element	Spiked Coal Fly Ash (NIST-SRM 1633a)				Spiked Electroplating Sludge			
	Mean Conc. (mg/L)	N ^b	RSD ^b (%)	Bias ^c (%AAS)	Mean Conc. (mg/L)	N ^b	RSD ^b (%)	Bias ^c (%AAS)
Al	330	8	16	104	127	8	13	110
Sb	3.4	6	73	96	5.3	7	24	120
As	21	8	83	270	5.2	7	8.6	87
Ba	133	8	8.7	101	1.6	8	20	58
Be	4.0	8	57	460	0.9	7	9.9	110
Cd	0.97	6	5.7	101	2.9	7	9.9	90
Ca	87	6	5.6	208	954	7	7.0	97
Cr	2.1	7	36	106	154	7	7.8	93
Co	1.2	6	21	94	1.0	7	11	85
Cu	1.9	6	9.7	118	156	8	7.8	97
Fe	602	8	8.8	102	603	7	5.6	98
Pb	4.6	7	22	94	25	7	5.6	98
Mg	15	8	15	110	35	8	20	84
Mn	1.8	7	14	104	5.9	7	9.6	95
Mo	891	8	19	105	1.4	7	36	110
Ni	1.6	6	8.1	91	9.5	7	9.6	90
K	46	8	4.2	98	51	8	5.8	82
Se	6.4	5	16	73	8.7	7	13	101
Ag	1.4	3	17	140	0.75	7	19	270
Na	20	8	49	130	1380	8	9.8	95
Tl	6.7	4	22	260	5.0	7	20	180
V	1010	5	7.5	100	1.2	6	11	80
Zn	2.2	6	7.6	93	266	7	2.5	101

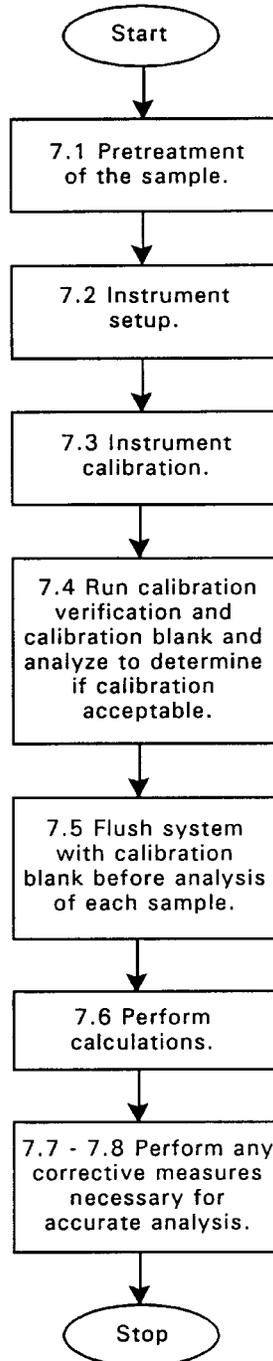
^aThese performance values are independent of sample preparation because the labs analyzed portions of the same digests.

^bN = Number of measurements for mean and relative standard deviation (RSD).

^cBias for the ICP-AES data is expressed as a percentage of atomic absorption spectroscopy (AA) data for the same digests.

METHOD 6010B

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY



METHOD 1311

TOXICITY CHARACTERISTIC LEACHING PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile analytes (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion (see Figure 1) at 30 ± 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessels

4.2.1 Zero-Headspace Extraction Vessel (ZHE). This device is for use only when the waste is being tested for the mobility of volatile analytes (i.e., those listed in Table 1). The ZHE (depicted in Figure 2) allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel (see Section 4.3.1). The vessels shall have an internal volume of 500-600 mL, and be equipped to accommodate a 90-110 mm filter. The devices contain VITON^{®1} O-rings which should be replaced frequently. Suitable ZHE devices known to EPA are identified in Table 3.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see Section 7.3) refers to pounds per square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

¹ VITON[®] is a trademark of Du Pont.

4.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste (see Section 4.3.3). It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Section 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Devices: It is recommended that all filtrations be performed in a hood.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel described in Section 4.2.1 is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 Filter Holder: When the waste is evaluated for other than volatile analytes, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. The type of filter holder used depends on the properties of the material to be filtered (see Section 4.3.3). These devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm (filter holders having an internal capacity of 1.5 L or greater, and equipped to accommodate a 142 mm diameter filter, are recommended). Vacuum filtration can only be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration. Suitable filter holders known to EPA are shown in Table 4.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) may be used only when evaluating the mobility of metals. Borosili-

cate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 μm , or equivalent. Filters known to EPA which meet these specifications are identified in Table 5. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: The meter should be accurate to ± 0.05 units at 25 °C.

4.6 ZHE Extract Collection Devices: TEDLAR^{®2} bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device. The devices listed are recommended for use under the following conditions:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1% of total waste), the TEDLAR[®] bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1% of total waste), the syringe or the TEDLAR[®] bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TEDLAR[®] bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

4.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit (see Section 4.3.2), or other ZHE device).

4.8 Laboratory Balance: Any laboratory balance accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

4.9 Beaker or Erlenmeyer flask, glass, 500 mL.

² TEDLAR[®] is a registered trademark of Du Pont.

4.10 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.

4.11 Magnetic stirrer.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent Water. Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.2.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.2.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.2.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at 90 ± 5 degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Hydrochloric acid (1N), HCl, made from ACS reagent grade.

5.4 Nitric acid (1N), HNO₃, made from ACS reagent grade.

5.5 Sodium hydroxide (1N), NaOH, made from ACS reagent grade.

5.6 Glacial acetic acid, CH₃CH₂OOH, ACS reagent grade.

5.7 Extraction fluid.

5.7.1 Extraction fluid # 1: Add 5.7 mL glacial CH₃CH₂OOH to 500 mL of reagent water (See Section 5.2), add 64.3 mL of 1N NaOH, and dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05 .

5.7.2 Extraction fluid # 2: Dilute 5.7 mL glacial $\text{CH}_3\text{CH}_2\text{OOH}$ with reagent water (See Section 5.2) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05 .

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.8 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 The TCLP may place requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile analyte extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction (see Section 1.4 concerning the use of this extract for volatile organics). If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4 °C. Samples should be opened only immediately prior to extraction).

6.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Section 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Section 8.0 (QA requirements) for acceptable sample and extract holding times.

7.0 PROCEDURE

7.1 Preliminary Evaluations

Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids (Section 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 7.1.2); (3) determination of whether the solid portion of the waste requires particle size reduction (Section 7.1.3); and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 7.1.4).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Section 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

7.1.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.1.1.5 to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Section 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Section 7.1.1.5 or 7.1.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Section 7.1.1.9)}}{\text{Total weight of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2 If the percent solids determined in Section 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Section 7.1.3 to

determine whether the solid material requires particle size reduction or to Section 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Section 7.1.1.9 is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed and to Section 7.3 with a fresh portion of the waste if the volatile TCLP is to be performed.

7.1.2.1 Remove the solid phase and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid phase at 100 ± 20 °C until two successive weighing yield the same value within $\pm 1\%$. Record the final weight.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{(\text{Wt. of dry waste + filter}) - \text{tared wt. of filter}}{\text{Initial wt. of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed, and to Section 7.3 if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP is to be performed, return to the beginning of this Section (7.1) and, with a fresh portion of waste, determine whether particle size reduction is necessary (Section 7.1.3) and determine the appropriate extraction fluid (Section 7.1.4). If only the volatile TCLP is to be performed, see the note in Section 7.1.4.

7.1.3 Determination of whether the waste requires particle size reduction (particle size is reduced during this step): Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^2 , or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken (see Section 7.3.6).

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet

the criteria, sample specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.1.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents (Section 7.2), determine the appropriate fluid (Section 5.7) for the nonvolatiles extraction as follows:

NOTE: TCLP extraction for volatile constituents uses only extraction fluid #1 (Section 5.7.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 7.3.

7.1.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

7.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 7.2.

7.1.4.3 If the pH from Section 7.1.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50 °C, and hold at 50 °C for 10 minutes.

7.1.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 7.2.

7.1.5 If the aliquot of the waste used for the preliminary evaluation (Sections 7.1.1 - 7.1.4) was determined to be 100% solid at Section 7.1.1.1, then it can be used for the Section 7.2 extraction (assuming at least 100 grams remain), and the Section 7.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Section 7.1.1.7, then another aliquot shall be used for the volatile extraction procedure in Section 7.3. The aliquot of the waste subjected to the procedure in Section 7.1.1.7 might be appropriate for use for the Section 7.2 extraction if an adequate amount of solid (as determined by Section 7.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Section 7.2.10 of the nonvolatile TCLP extraction.

7.2 Procedure When Volatiles are not Involved

A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the

solids content of the waste sample (percent solids, See Section 7.1.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (*i.e.*, is 100% solid, see Section 7.1.1), weigh out a subsample of the waste (100 gram minimum) and proceed to Section 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Section 4.4).

NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

7.2.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids (Section 7.1.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5% dry solids (Sections 7.1.1 or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see Section 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at

room temperature, then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.2.5, to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.2.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (See Section 7.2.12) or stored at 4 °C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.9 If the waste contains <0.5% dry solids (see Section 7.1.2), proceed to Section 7.2.13. If the waste contains >0.5% dry solids (see Section 7.1.1 or 7.1.2), and if particle size reduction of the solid was needed in Section 7.1.3, proceed to Section 7.2.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 7.2.11.

7.2.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.1.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid

material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.2.5 or 7.2.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Section 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 ± 2 °C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Section 7.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Section 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the TCLP extract as follows:

7.2.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.2.12 is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.2.12 with the initial liquid phase of the waste obtained in Section 7.2.7. This combined liquid is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Section 7.2.7, is not or may not be compatible with the filtered liquid resulting from Section 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 7.2.14.

7.2.14 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4 °C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to $\pm 0.5\%$), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1) (C_1) + (V_2) (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).

C_1 = The concentration of the analyte of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).

C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

7.3 Procedure When Volatiles are Involved

Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of nonvolatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a

sample from which no additional liquid may be forced out by an applied pressure of 50 psi), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4 °C) to minimize loss of volatiles.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (See Section 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Section 4.6 are recommended for use under the conditions stated in Sections 4.6.1 - 4.6.3.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 7.3, Section 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the waste is 100% solid (see Section 7.1.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Section 7.3.5.

7.3.4 If the waste contains < 0.5% dry solids (Section 7.1.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing \geq 0.5% dry solids (Sections 7.1.1 and/or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For wastes containing < 5% solids (see Section 7.1.1), weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For wastes containing \geq 5% solids (see Section 7.1.1), determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{\text{25}}{\text{percent solids (Section 7.1.1)}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle size reduction of the solid portion of the waste was required in Section 7.1.3, proceed to Section 7.3.6. If particle size reduction was not required in Section 7.1.3, proceed to Section 7.3.7.

7.3.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Section 7.1.3. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4 °C prior to particle size reduction. The means used to effect particle size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriately altered, proceed to Section 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 7.3.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace

slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Section 7.1.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Section 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% dry solids (see Section 7.1.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Section 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately (See Sections 7.3.13 through 7.3.15) or stored at 4 °C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.3.4 or 7.3.8)}}{100}$$

7.3.12 The following Sections detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (See Section 5.7).

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary agitation apparatus (if it is not already there) and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction occurs) shall be maintained at 23 ± 2 °C during agitation.

7.3.13 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Section 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Section 7.3.9. All extract shall be filtered and collected if the

TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see Sections 4.6 and 7.3.1).

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

7.3.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.3.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Section 7.3.13 and the initial liquid phase (Section 7.3.9) are collectively defined as the TCLP extract.

7.3.15 Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4 °C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1) (C_1) + (V_2) (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).

C_1 = The concentration of the analyte of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).

C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.3.16 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

8.0 QUALITY ASSURANCE

8.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

8.2 A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.2.2 In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2.4 Matrix spike recoveries are calculated by the following formula:

$$\%R (\% \text{Recovery}) = 100 (X_s - X_u)/K$$

where:

X_s = measured value for the spiked sample,

X_u = measured value for the unspiked sample, and

K = known value of the spike in the sample.

8.3 All quality control measures described in the appropriate analytical methods shall be followed.

8.4 The use of internal calibration quantitation methods shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

8.4.1. The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

8.4.2 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The fourth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and

150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. All four aliquots are analyzed.

8.4.3 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependant variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

8.4.4 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject to linear regression of the corrected instrument signals or external-calibration-derived concentrations as the dependant variable versus the independent variable. Derive concentrations for unknowns using the internal calibration curve as if it were an external calibration curve.

8.5 Samples must undergo TCLP extraction within the following time periods:

SAMPLE MAXIMUM HOLDING TIMES [DAYS]				
	From: Field collection	From: TCLP extraction	From: Preparative extraction	
	To: TCLP extraction	To: Preparative extraction	To: Determinative analysis	Total elapsed time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

9.0 METHOD PERFORMANCE

9.1 Ruggedness. Two ruggedness studies have been performed to determine the effect of various perturbations on specific elements of the TCLP protocol. Ruggedness testing determines the sensitivity of small procedural variations which might be expected to occur during routine laboratory application.

9.1.1 Metals - The following conditions were used when leaching a waste for metals analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Extraction time	16 hours vs. 18 hours
Headspace	20% vs. 60%
Buffer #2 acidity	190 meq vs. 210 meq
Acid-washed filters	yes vs. no
Filter type	0.7 μm glass fiber vs. 0.45 μm vs. polycarbonate
Bottle type	borosilicate vs. flint glass

Of the seven method variations examined, acidity of the extraction fluid had the greatest impact on the results. Four of 13 metals from an API separator sludge/electroplating waste (API/EW) mixture and two of three metals from an ammonia lime still bottom waste were extracted at higher levels by the more acidic buffer. Because of the sensitivity to pH changes, the method requires that the extraction fluids be prepared so that the final pH is within ± 0.05 units as specified.

9.1.2 Volatile Organic Compounds - The following conditions were used when leaching a waste for VOC analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Headspace	0% vs. 5%
Buffer #1 acidity	60 meq vs. 80 meq
Method of storing extract	Syringe vs. Tedlar [®] bag
Aliquotting	yes vs. no
Pressure behind piston	0 psi vs. 20 psi

None of the parameters had a significant effect on the results of the ruggedness test.

9.2 Precision. Many TCLP precision (reproducibility) studies have been performed, and have shown that, in general, the precision of the TCLP is comparable to or exceeds that of the EP toxicity test and that method precision is adequate. One of the more significant contributions to poor precision appears to be related to sample homogeneity and inter-laboratory variation (due to the nature of waste materials).

9.2.1 Metals - The results of a multi-laboratory study are shown in Table 6, and indicate that a single analysis of a waste may not be adequate for waste characterization and identification requirements.

9.2.2 Semi-Volatile Organic Compounds - The results of two studies are shown in Tables 7 and 8. Single laboratory precision was excellent with greater than 90 percent of the results exhibiting an RSD less than 25 percent. Over 85 percent of all individual compounds in the multi-laboratory study fell in the RSD range of 20 - 120 percent. Both studies concluded that the TCLP provides adequate precision. It was also determined that the high acetate content of the extraction fluid did not present problems (i.e., column degradation of the gas chromatograph) for the analytical conditions used.

9.2.3 Volatile Organic Compounds - Eleven laboratories participated in a collaborative study of the use of the ZHE with two waste types which were fortified with a mixture of VOCs. The results of the collaborative study are shown in Table 9. Precision results for VOCs tend to occur over a considerable range. However, the range and mean RSD compared very closely to the same collaborative study metals results in Table 6. Blackburn and Show concluded that at the 95% level of significance: 1) recoveries among laboratories were statistically similar, 2) recoveries did not vary significantly between the two sample types, and 3) each laboratory showed the same pattern of recovery for each of the two samples.

10.0 REFERENCES

1. Blackburn, W.B. and Show, I. "Collaborative Study of the Toxicity Characteristics Leaching Procedure (TCLP)." Draft Final Report, Contract No. 68-03-1958, S-Cubed, November 1986.
2. Newcomer, L.R., Blackburn, W.B., Kimmell, T.A. "Performance of the Toxicity Characteristic Leaching Procedure." Wilson Laboratories, S-Cubed, U.S. EPA, December 1986.
3. Williams, L.R., Francis, C.W.; Maskarinec, M.P., Taylor D.R., and Rothman, N. "Single-Laboratory Evaluation of Mobility Procedure for Solid Waste." EMSL, ORNL, S-Cubed, ENSECO.

Table 1.
Volatile Analytes^{1,2}

Compound	CAS No.
Acetone	67-64-1
Benzene	71-43-2
n-Butyl alcohol	71-36-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1,-Trichloroethane	71-55-6
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

¹ When testing for any or all of these analytes, the zero-headspace extractor vessel shall be used instead of the bottle extractor.

² Benzene, carbon tetrachloride, chlorobenzene, chloroform, 1,2-dichloroethane, 1,1-dichloroethylene, methyl ethyl ketone, tetrachloroethylene, and vinyl chloride are toxicity characteristic constituents.

Table 2.
Suitable Rotary Agitation Apparatus¹

Company	Location	Model No.
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel extractor (DC20S)
		8-vessel extractor (DC20)
		12-vessel extractor (DC20B)
		24-vessel extractor (DC24C)
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	2-vessel (3740-2-BRE)
		4-vessel (3740-4-BRE)
		6-vessel (3740-6-BRE)
		8-vessel (3740-8-BRE)
		12-vessel (3740-12-BRE)
24-vessel (3740-24-BRE)		
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	8-vessel (08-00-00)
		4-vessel (04-00-00)
IRA Machine Shop and Laboratory	Santurce, PR (809) 752-4004	8-vessel (011001)
Lars Lande Manufacturing	Whitmore Lake, MI (313) 449-4116	10-vessel (10VRE)
		5-vessel (5VRE)
		6-vessel (6VRE)
Millipore Corp.	Bedford, MA (800) 225-3384	4-ZHE or 4 2-liter bottle extractor (YT310RAHW)

¹ Any device that rotates the extraction vessel in an end-over-end fashion at 30 ± 2 rpm is acceptable.

Table 3.
Suitable Zero-Headspace Extractor Vessels¹

Company	Location	Model No.
Analytical Testing & Consulting Services, Inc.	Warrington, PA (215) 343-4490	C102, Mechanical Pressure Device
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	3745-ZHE, Gas Pressure Device
Lars Lande Manufacturing ²	Whitmore Lake, MI (313) 449-4116	ZHE-11, Gas Pressure Device
Millipore Corporation	Bedford, MA (800) 225-3384	YT30090HW, Gas Pressure Device
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	VOLA-TOX1, Gas Pressure Device
Gelman Science	Ann Arbor, MI (800) 521-1520	15400 Gas Pressure Device

¹ Any device that meets the specifications listed in Section 4.2.1 of the method is suitable.

² This device uses a 110 mm filter.

Table 4.
Suitable Filter Holders¹

Company	Location	Model/ Catalogue No.	Size
Nucleopore Corporation	Pleasanton, CA (800) 882-7711	425910 410400	142 mm 47 mm
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	302400 311400	142 mm 47 mm
Millipore Corporation	Bedford, MA (800) 225-3384	YT30142HW XX1004700	142 mm 47 mm

¹ Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic analytes are of concern. The 142 mm size filter holder is recommended.

Table 5.
Suitable Filter Media¹

Company	Location	Model	Pore Size (µm)
Millipore Corporation	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corporation	Pleasanton, CA (415) 463-2530	211625	0.7
Whatman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	GF75	0.7
Gelman Science	Ann Arbor, MI (800) 521-1520	66256 (90mm) 66257 (142mm)	0.7

¹ Any filter that meets the specifications in Section 4.4 of the Method is suitable.

Table 6. Multi-Laboratory TCLP Metals, Precision

Waste	Extraction Fluid	Metal	\bar{X}	S	%RSD
Ammonia Lime Still	#1	Cadmium	0.053	0.031	60
	#2		0.023		76
Bottoms	#1	Chromium	0.015	0.0014	93
	#2		0.0032		118
	#1	Lead	0.0030	0.0027	90
	#2		0.0032		87
API/EW Mixture	#1	Cadmium	0.0046	0.0028	61
	#2		0.0005		77
	#1	Chromium	0.0561	0.0227	40
	#2		0.105		17
	#1	Lead	0.0031	0.0031	100
	#2		0.0124		110
Fossil Fuel Fly Ash	#1	Cadmium	0.080	0.069	86
	#2		0.093		72
	#1	Chromium	0.017	0.014	85
	#2		0.070		57
	#1	Lead	0.0087	0.0074	85
	#2		0.0457		18
%RSD Range = 17 - 118					
Mean %RSD = 74					

NOTE: \bar{X} = Mean results from 6 - 12 different laboratories
 Units = mg/L
 Extraction Fluid #1 = pH 4.9
 #2 = pH 2.9

Table 7. Single-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	\bar{X}	S	%RSD
Ammonia Lime Still Bottoms	Phenol	#1	19000	2230	11.6
		#2	19400	929	4.8
	2-Methylphenol	#1	2000	297	14.9
		#2	1860	52.9	2.8
	4-Methylphenol	#1	7940	1380	17.4
		#2	7490	200	2.7
	2,4-Dimethylphenol	#1	321	46.8	14.6
		#2	307	45.8	14.9
	Naphthalene	#1	3920	413	10.5
		#2	3827	176	4.6
	2-Methylnaphthalene	#1	290	44.8	15.5
		#2	273	19.3	7.1
	Dibenzofuran	#1	187	22.7	12.1
		#2	187	7.2	3.9
	Acenaphthylene	#1	703	89.2	12.7
		#2	663	20.1	3.0
	Fluorene	#1	151	17.6	11.7
		#2	156	2.1	1.3
	Phenanthrene	#1	241	22.7	9.4
		#2	243	7.9	3.3
Anthracene	#1	33.2	6.19	18.6	
	#2	34.6	1.55	4.5	
Fluoranthrene	#1	25.3	1.8	7.1	
	#2	26.0	1.8	7.1	
API/EW Mixture	Phenol	#1	40.7	13.5	33.0
		#2	19.0	1.76	9.3
	2,4-Dimethylphenol	#1	33.0	9.35	28.3
		#2	43.3	8.61	19.9
	Naphthalene	#1	185	29.4	15.8
		#2	165	24.8	15.0
	2-Methylnaphthalene	#1	265	61.2	23.1
		#2	200	18.9	9.5
%RSD Range = 1 - 33					
Mean %RSD = 12					

NOTE: Units = $\mu\text{g/L}$

Extractions were performed in triplicate

All results were at least 2x the detection limit

Extraction Fluid #1 = pH 4.9

#2 = pH 2.9

Table 8. Multi-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	\bar{X}	S	%RSD
Ammonia Lime Still Bottoms (A)	BNAs	#1	10043	7680	76.5
		#2	10376	6552	63.1
API/EW Mixture (B)	BNAs	#1	1624	675	41.6
		#2	2074	1463	70.5
Fossil Fuel Fly Ash (C)	BNAs	#1	750	175	23.4
		#2	739	342	46.3
Mean %RSD = 54					

NOTE: \bar{X} units = $\mu\text{g/L}$
 \bar{X} = Mean results from 3 - 10 labs
 Extraction Fluid #1 = pH 4.9
 #2 = pH 2.9

%RSD Range for Individual Compounds

A, #1	0 - 113
A, #2	28 - 108
B, #1	20 - 156
B, #2	49 - 128
C, #1	36 - 143
C, #2	61 - 164

Table 9. Multi-Laboratory (11 Labs) VOCs, Precision

Waste	Compound	\bar{X}	S	%RSD
Mine Tailings	Vinyl chloride	6.36	6.36	100
	Methylene chloride	12.1	11.8	98
	Carbon disulfide	5.57	2.83	51
	1,1-Dichloroethene	21.9	27.7	127
	1,1-Dichloroethane	31.4	25.4	81
	Chloroform	46.6	29.2	63
	1,2-Dichloroethane	47.8	33.6	70
	2-Butanone	43.5	36.9	85
	1,1,1-Trichloroethane	20.9	20.9	100
	Carbon tetrachloride	12.0	8.2	68
	Trichloroethene	24.7	21.2	86
	1,1,2-Trichloroethene	19.6	10.9	56
	Benzene	37.9	28.7	76
	1,1,2,2-Tetrachloroethane	34.9	25.6	73
	Toluene	29.3	11.2	38
	Chlorobenzene	35.6	19.3	54
	Ethylbenzene	4.27	2.80	66
	Trichlorofluoromethane	3.82	4.40	115
	Acrylonitrile	76.7	110.8	144
	Ammonia Lime Still Bottoms	Vinyl chloride	5.00	4.71
Methylene chloride		14.3	13.1	92
Carbon disulfide		3.37	2.07	61
1,1-Dichloroethene		52.1	38.8	75
1,1-Dichloroethane		52.8	25.6	49
Chloroform		64.7	28.4	44
1,2-Dichloroethane		43.1	31.5	73
2-Butanone		59.0	39.6	67
1,1,1-Trichloroethane		53.6	40.9	76
Carbon tetrachloride		7.10	6.1	86
Trichloroethene		57.3	34.2	60
1,1,2-Trichloroethene		6.7	4.7	70
Benzene		61.3	26.8	44
1,1,2,2-Tetrachloroethane		3.16	2.1	66
Toluene		69.0	18.5	27
Chlorobenzene		71.8	12.0	17
Ethylbenzene		3.70	2.2	58
Trichlorofluoromethane		4.05	4.8	119
Acrylonitrile		29.4	34.8	118
%RSD Range = 17 - 144				
Mean %RSD = 75				

NOTE: Units = $\mu\text{g/L}$

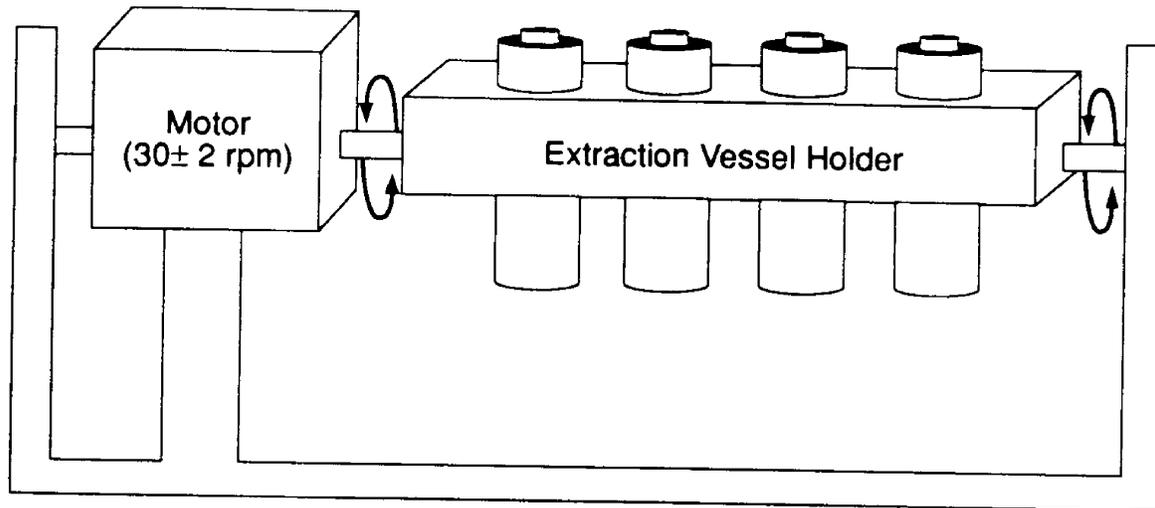


Figure 1. Rotary Agitation Apparatus

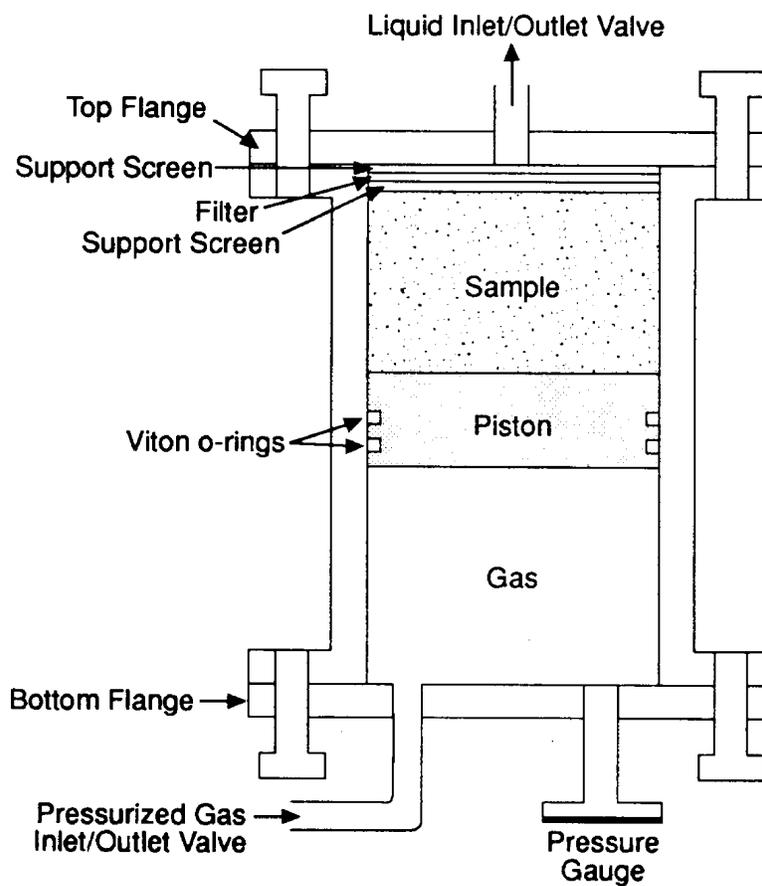
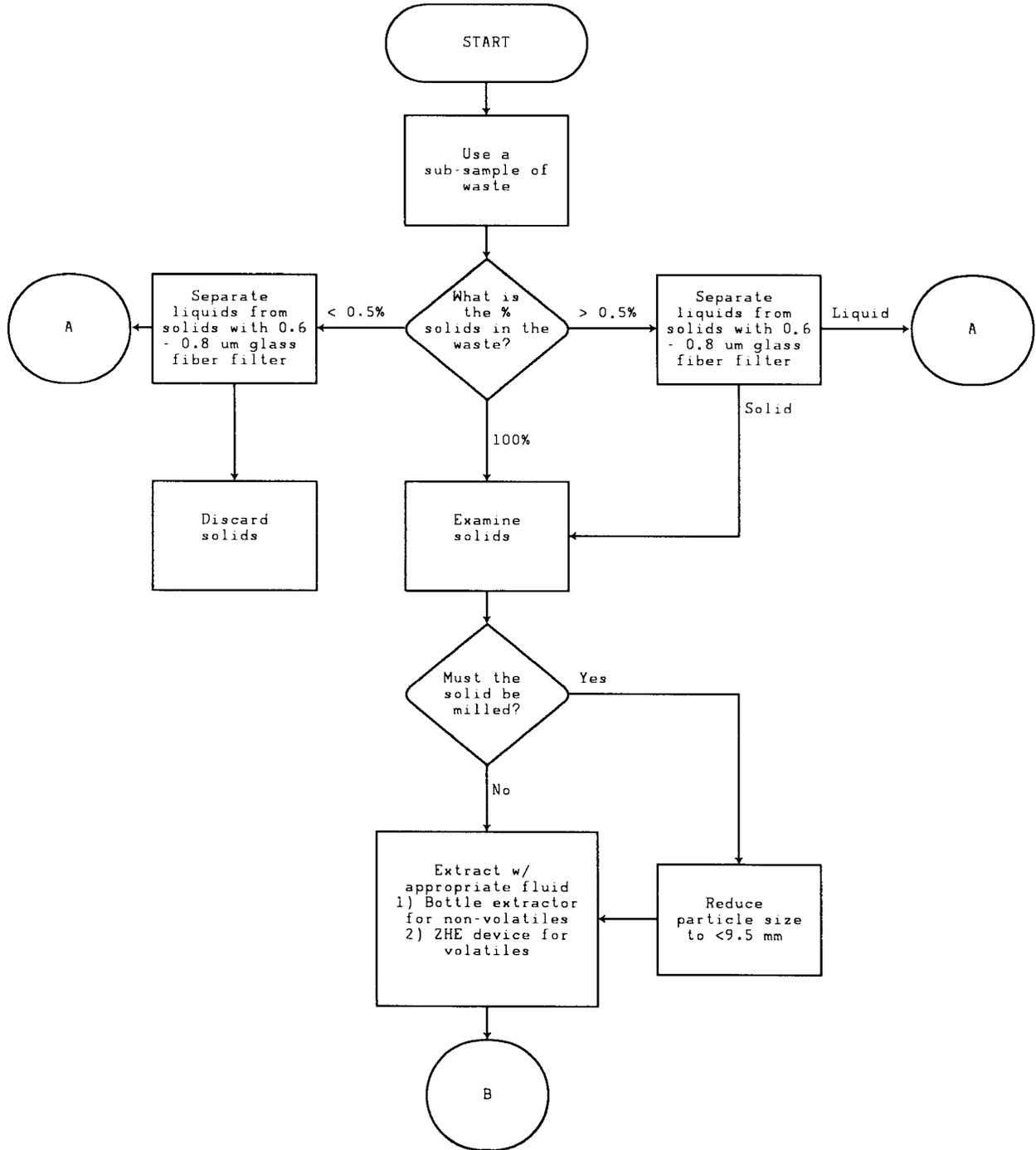


Figure 2. Zero-Headspace Extractor (ZHE)

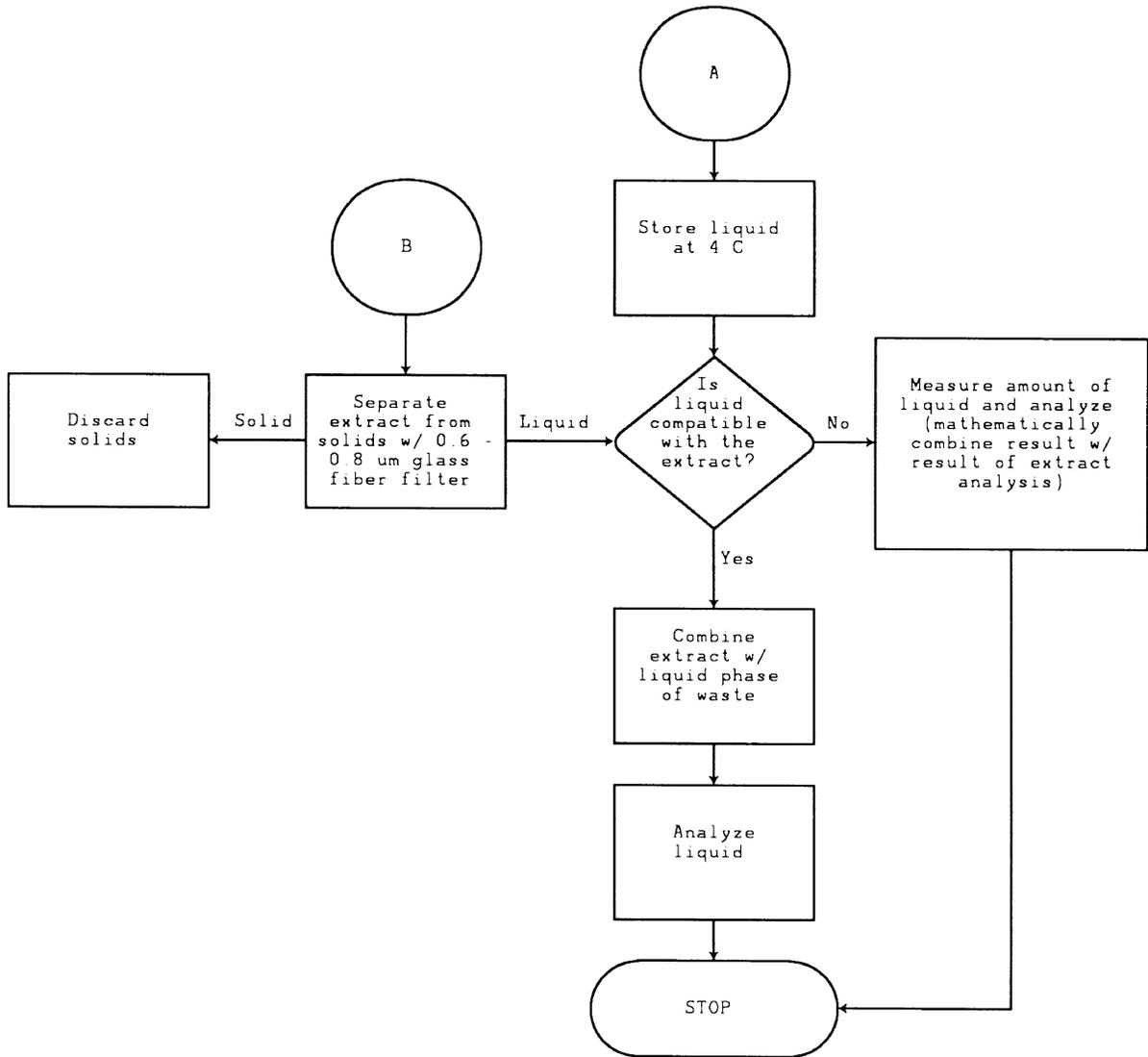
METHOD 1311

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



METHOD 1311 (CONTINUED)

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



METHOD 8330

NITROAROMATICS AND NITRAMINES BY HIGH
PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

1.0 SCOPE AND APPLICATION

1.1 Method 8330 is intended for the trace analysis of explosives residues by high performance liquid chromatography using a UV detector. This method is used to determine the concentration of the following compounds in a water, soil, or sediment matrix:

Compound	Abbreviation	CAS No ^a
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4
1,3-Dinitrobenzene	1,3-DNB	99-65-0
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-45-8
Nitrobenzene	NB	98-95-3
2,4,6-Trinitrotoluene	2,4,6-TNT	118-96-7
4-Amino-2,6-dinitrotoluene	4-Am-DNT	1946-51-0
2-Amino-4, 6-dinitrotoluene	2-Am-DNT	355-72-78-2
2,4-Dinitrotoluene	2,4-DNT	121-14-2
2,6-Dinitrotoluene	2,6-DNT	606-20-2
2-Nitrotoluene	2-NT	88-72-2
3-Nitrotoluene	3-NT	99-08-1
4-Nitrotoluene	4-NT	99-99-0

a Chemical Abstracts Service Registry number

1.2 Method 8330 provides a salting-out extraction procedure for low concentration (parts per trillion, or nanograms per liter) of explosives residues in surface or ground water. Direct injection of diluted and filtered water samples can be used for water samples of higher concentration (See Table 1).

1.3 All of these compounds are either used in the manufacture of explosives or are the degradation products of compounds used for that purpose. When making stock solutions for calibration, treat each explosive compound with caution. See NOTE in Sec. 5.3.1 and Sec. 11 on Safety.

1.4 The estimated quantitation limits (EQLs) of target analytes determined by Method 8330 in water and soil are presented in Table 1.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC, skilled in the interpretation of

chromatograms, and experienced in handling explosive materials. (See Sec. 11.0 on SAFETY.) Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Method 8330 provides high performance liquid chromatographic (HPLC) conditions for the detection of ppb levels of certain explosives residues in water, soil and sediment matrix. Prior to use of this method, appropriate sample preparation techniques must be used.

2.2 Low-Level Salting-out Method With No Evaporation: Aqueous samples of low concentration are extracted by a salting-out extraction procedure with acetonitrile and sodium chloride. The small volume of acetonitrile that remains undissolved above the salt water is drawn off and transferred to a smaller volumetric flask. It is back-extracted by vigorous stirring with a specific volume of salt water. After equilibration, the phases are allowed to separate and the small volume of acetonitrile residing in the narrow neck of the volumetric flask is removed using a Pasteur pipet. The concentrated extract is diluted 1:1 with reagent grade water. An aliquot is separated on a C-18 reverse phase column, determined at 254 nm, and confirmed on a CN reverse phase column.

2.3 High-level Direct Injection Method: Aqueous samples of higher concentration can be diluted 1/1 (v/v) with methanol or acetonitrile, filtered, separated on a C-18 reverse phase column, determine at 254 nm, and confirmed on a CN reverse phase column. If HMX is an important target analyte, methanol is preferred.

2.4 Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath, filtered and chromatographed as in Sec. 2.3.

3.0 INTERFERENCES

3.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences.

3.2 2,4-DNT and 2,6-DNT elute at similar retention times (retention time difference of 0.2 minutes). A large concentration of one isomer may mask the response of the other isomer. If it is not apparent that both isomers are present (or are not detected), an isomeric mixture should be reported.

3.3 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain tetryl should be diluted with acetonitrile prior to filtration and acidified to pH <3. All samples expected to contain tetryl should not be exposed to temperatures above room temperature.

3.4 Degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak. Peak heights rather than peak areas should be used when tetryl is present in concentrations that are significant relative to the concentration of 2,4,6-TNT.

4.0 APPARATUS AND MATERIALS

4.1 HPLC system

4.1.1 HPLC - equipped with a pump capable of achieving 4000 psi, a 100 μ l loop injector and a 254 nm UV detector (Perkin Elmer Series 3, or equivalent). For the low concentration option, the detector must be capable of a stable baseline at 0.001 absorbance units full scale.

4.1.2 Recommended Columns:

4.1.2.1 Primary column: C-18 Reverse phase HPLC column, 25 cm x 4.6 mm (5 μ m), (Supelco LC-18, or equivalent).

4.1.2.2 Secondary column: CN Reverse phase HPLC column, 25 cm x 4.6 mm (5 μ m), (Supelco LC-CN, or equivalent).

4.1.3 Strip chart recorder.

4.1.4 Digital integrator (optional).

4.1.5 Autosampler (optional).

4.2 Other Equipment

4.2.1 Temperature controlled ultrasonic bath.

4.2.2 Vortex mixer.

4.2.3 Balance, \pm 0.0001 g.

4.2.4 Magnetic stirrer with stirring pellets.

4.2.5 Water bath - Heated, with concentric ring cover, capable of temperature control (\pm 5°C). The bath should be used in a hood.

4.2.6 Oven - Forced air, without heating.

4.3 Materials

4.3.1 High pressure injection syringe - 500 μ L, (Hamilton liquid syringe or equivalent).

4.3.2 Disposable cartridge filters - 0.45 μ m Teflon filter.

4.3.3 Pipets - Class A, glass, Appropriate sizes.

4.3.4 Pasteur pipets.

4.3.5 Scintillation Vials - 20 mL, glass.

4.3.6 Vials - 15 mL, glass, Teflon-lined cap.

4.3.7 Vials- 40 mL, glass, Teflon-lined cap.

4.3.8 Disposable syringes - Plastipak, 3 mL and 10 mL or equivalent.

4.3.9 Volumetric flasks - Appropriate sizes with ground glass stoppers, Class A.

NOTE: The 100 mL and 1 L volumetric flasks used for magnetic stirrer extraction must be round.

4.3.10 Vacuum desiccator - Glass.

4.3.11 Mortar and pestle - Steel.

4.3.12 Sieve - 30 mesh.

4.3.13 Graduated cylinders - Appropriate sizes.

4.4 Preparation of Materials

4.4.1 Prepare all materials to be used as described in Chapter 4 for semivolatile organics.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination.

5.1.1 Acetonitrile, CH_3CN - HPLC grade.

5.1.2 Methanol, CH_3OH - HPLC grade.

5.1.3 Calcium chloride, CaCl_2 - Reagent grade. Prepare an aqueous solution of 5 g/L.

5.1.4 Sodium chloride, NaCl , shipped in glass bottles - reagent grade.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Stock Standard Solutions

5.3.1 Dry each solid analyte standard to constant weight in a vacuum desiccator in the dark. Place about 0.100 g (weighed to 0.0001 g) of a single analyte into a 100 mL volumetric flask and dilute to volume with

acetonitrile. Invert flask several times until dissolved. Store in refrigerator at 4°C in the dark. Calculate the concentration of the stock solution from the actual weight used (nominal concentration = 1,000 mg/L). Stock solutions may be used for up to one year.

NOTE: The HMX, RDX, Tetryl, and 2,4,6-TNT are explosives and the neat material should be handled carefully. See SAFETY in Sec. 11 for guidance. HMX, RDX, and Tetryl reference materials are shipped under water. Drying at ambient temperature requires several days. DO NOT DRY AT HEATED TEMPERATURES!

5.4 Intermediate Standards Solutions

5.4.1 If both 2,4-DNT and 2,6-DNT are to be determined, prepare two separate intermediate stock solutions containing (1) HMX, RDX, 1,3,5-TNB, 1,3-DNB, NB, 2,4,6-TNT, and 2,4-DNT and (2) Tetryl, 2,6-DNT, 2-NT, 3-NT, and 4-NT. Intermediate stock standard solutions should be prepared at 1,000 µg/L, in acetonitrile when analyzing soil samples, and in methanol when analyzing aqueous samples.

5.4.2 Dilute the two concentrated intermediate stock solutions, with the appropriate solvent, to prepare intermediate standard solutions that cover the range of 2.5 - 1,000 µg/L. These solutions should be refrigerated on preparation, and may be used for 30 days.

5.4.3 For the low-level method, the analyst must conduct a detection limit study and devise dilution series appropriate to the desired range. Standards for the low level method must be prepared immediately prior to use.

5.5 Working standards

5.5.1 Calibration standards at a minimum of five concentration levels should be prepared through dilution of the intermediate standards solutions by 50% (v/v) with 5 g/L calcium chloride solution (Sec. 5.1.3). These solutions must be refrigerated and stored in the dark, and prepared fresh on the day of calibration.

5.6 Surrogate Spiking Solution

5.6.1 The analyst should monitor the performance of the extraction and analytical system as well as the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and reagent water blank with one or two surrogates (e.g., analytes not expected to be present in the sample).

5.7 Matrix Spiking Solutions

5.7.1 Prepare matrix spiking solutions in methanol such that the concentration in the sample is five times the Estimated Quantitation Limit (Table 1). All target analytes should be included.

5.8 HPLC Mobile Phase

5.8.1 To prepare 1 liter of mobile phase, add 500 mL of methanol to 500 mL of organic-free reagent water.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Follow conventional sampling and sample handling procedures as specified for semivolatile organics in Chapter Four.

6.2 Samples and sample extracts must be stored in the dark at 4°C. Holding times are the same as for semivolatile organics.

7.0 PROCEDURE

7.1 Sample Preparation

7.1.1 Aqueous Samples: It is highly recommended that process waste samples be screened with the high-level method to determine if the low level method (1-50 µg/L) is required. Most groundwater samples will fall into the low level method.

7.1.1.1 Low-Level Method (salting-out extraction)

7.1.1.1.1 Add 251.3 g of sodium chloride to a 1 L volumetric flask (round). Measure out 770 mL of a water sample (using a 1 L graduated cylinder) and transfer it to the volumetric flask containing the salt. Add a stir bar and mix the contents at maximum speed on a magnetic stirrer until the salt is completely dissolved.

7.1.1.1.2 Add 164 mL of acetonitrile (measured with a 250 mL graduated cylinder) while the solution is being stirred and stir for an additional 15 minutes. Turn off the stirrer and allow the phases to separate for 10 minutes.

7.1.1.1.3 Remove the acetonitrile (upper) layer (about 8 mL) with a Pasteur pipet and transfer it to a 100 mL volumetric flask (round). Add 10 mL of fresh acetonitrile to the water sample in the 1 L flask. Again stir the contents of the flask for 15 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract. The inclusion of a few drops of salt water at this point is unimportant.

7.1.1.1.4 Add 84 mL of salt water (325 g NaCl per 1000 mL of reagent water) to the acetonitrile extract in the 100 mL volumetric flask. Add a stir bar and stir the contents on a magnetic stirrer for 15 minutes, followed by 10 minutes for phase separation. Carefully transfer the acetonitrile phase

to a 10 mL graduated cylinder using a Pasteur pipet. At this stage, the amount of water transferred with the acetonitrile must be minimized. The water contains a high concentration of NaCl that produces a large peak at the beginning of the chromatogram, where it could interfere with the HMX determination.

7.1.1.1.5 Add an additional 1.0 mL of acetonitrile to the 100 mL volumetric flask. Again stir the contents of the flask for 15 minutes, followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract in the 10 mL graduated cylinder (transfer to a 25 mL graduated cylinder if the volume exceeds 5 mL). Record the total volume of acetonitrile extract to the nearest 0.1 mL. (Use this as the volume of total extract [V_t] in the calculation of concentration after converting to μL). The resulting extract, about 5 - 6 mL, is then diluted 1:1 with organic-free reagent water (with pH <3 if tetryl is a suspected analyte) prior to analysis.

7.1.1.1.6 If the diluted extract is turbid, filter it through a 0.45 - μm Teflon filter using a disposable syringe. Discard the first 0.5 mL of filtrate, and retain the remainder in a Teflon-capped vial for RP-HPLC analysis as in Sec. 7.4.

7.1.1.2 High-Level Method

7.1.1.2.1 Sample filtration: Place a 5 mL aliquot of each water sample in a scintillation vial, add 5 mL of acetonitrile, shake thoroughly, and filter through a 0.45- μm Teflon filter using a disposable syringe. Discard the first 3 mL of filtrate, and retain the remainder in a Teflon-capped vial for RP-HPLC analysis as in Sec. 7.4. HMX quantitation can be improved with the use of methanol rather than acetonitrile for dilution before filtration.

7.1.2 Soil and Sediment Samples

7.1.2.1 Sample homogenization: Dry soil samples in air at room temperature or colder to a constant weight, being careful not to expose the samples to direct sunlight. Grind and homogenize the dried sample thoroughly in an acetonitrile-rinsed mortar to pass a 30 mesh sieve.

NOTE: Soil samples should be screened by Method 8515 prior to grinding in a mortar and pestle (See Safety Sec. 11.2).

7.1.2.2 Sample extraction

7.1.2.2.1 Place a 2.0 g subsample of each soil sample in a 15 mL glass vial. Add 10.0 mL of acetonitrile, cap with

Teflon-lined cap, vortex swirl for one minute, and place in a cooled ultrasonic bath for 18 hours.

7.1.2.2.2 After sonication, allow sample to settle for 30 minutes. Remove 5.0 mL of supernatant, and combine with 5.0 mL of calcium chloride solution (Sec. 5.1.3) in a 20 mL vial. Shake, and let stand for 15 minutes.

7.1.2.2.3 Place supernatant in a disposable syringe and filter through a 0.45- μ m Teflon filter. Discard first 3 mL and retain remainder in a Teflon-capped vial for RP-HPLC analysis as in Sec. 7.4.

7.2 Chromatographic Conditions (Recommended)

Primary Column: C-18 reverse phase HPLC column, 25-cm x 4.6-mm, 5 μ m, (Supelco LC-18 or equivalent).

Secondary Column: CN reverse phase HPLC column, 25-cm x 4.6-mm, 5 μ m, (Supelco LC-CN or equivalent).

Mobile Phase: 50/50 (v/v) methanol/organic-free reagent water.

Flow Rate: 1.5 mL/min

Injection volume: 100- μ L

UV Detector: 254 nm

7.3 Calibration of HPLC

7.3.1 All electronic equipment is allowed to warm up for 30 minutes. During this period, at least 15 void volumes of mobile phase are passed through the column (approximately 20 min at 1.5 mL/min) and continued until the baseline is level at the UV detector's greatest sensitivity.

7.3.2 Initial Calibration. Injections of each calibration standard over the concentration range of interest are made sequentially into the HPLC in random order. Peak heights or peak areas are obtained for each analyte. Experience indicates that a linear calibration curve with zero intercept is appropriate for each analyte. Therefore, a response factor for each analyte can be taken as the slope of the best-fit regression line.

7.3.3 Daily Calibration. Analyze midpoint calibration standards, at a minimum, at the beginning of the day, singly at the midpoint of the run, and singly after the last sample of the day (assuming a sample group of 10 samples or less). Obtain the response factor for each analyte from the mean peak heights or peak areas and compare it with the response factor obtained for the initial calibration. The mean response factor for the

daily calibration must agree within $\pm 15\%$ of the response factor of the initial calibration. The same criteria is required for subsequent standard responses compared to the mean response of the triplicate standards beginning the day. If this criterion is not met, a new initial calibration must be obtained.

7.4 HPLC Analysis

7.4.1 Analyze the samples using the chromatographic conditions given in Sec. 7.2. All positive measurements observed on the C-18 column must be confirmed by injection onto the CN column.

7.4.2 Follow Sec. 7.0 in Method 8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence. If column temperature control is not employed, special care must be taken to ensure that temperature shifts do not cause peak misidentification.

7.4.3 Table 2 summarizes the estimated retention times on both C-18 and CN columns for a number of analytes analyzable using this method. An example of the separation achieved by Column 1 is shown in Figure 1.

7.4.4 Record the resulting peak sizes in peak heights or area units. The use of peak heights is recommended to improve reproducibility of low level samples.

7.4.5 Calculation of concentration is covered in Sec. 7.0 of Method 8000.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures. Quality control to validate sample extraction is covered in Method 3500.

8.2 Quality control required to validate the HPLC system operation is found in Method 8000, Sec. 8.0.

8.3 Prior to preparation of stock solutions, acetonitrile, methanol, and water blanks should be run to determine possible interferences with analyte peaks. If the acetonitrile, methanol, or water blanks show contamination, a different batch should be used.

9.0 METHOD PERFORMANCE

9.1 Table 3 presents the single laboratory precision based on data from the analysis of blind duplicates of four spiked soil samples and four field contaminated samples analyzed by seven laboratories.

9.2 Table 4 presents the multilaboratory error based on data from the analysis of blind duplicates of four spiked soil samples and four field contaminated samples analyzed by seven laboratories.

9.3 Table 5 presents the multilaboratory variance of the high concentration method for water based on data from nine laboratories.

9.4 Table 6 presents multilaboratory recovery data from the analysis of spiked soil samples by seven laboratories.

9.5 Table 7 presents a comparison of method accuracy for soil and aqueous samples (high concentration method).

9.6 Table 8 contains precision and accuracy data for the salting-out extraction method.

10.0 REFERENCES

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residues in soil. Part II: Further development and ruggedness testing. USA Cold Regions Research and Engineering Laboratory, CRREL Report 88-8.

8. Leggett, D.C., T.F. Jenkins and P.H. Miyares (1990) Salting-out solvent extraction for preconcentration of neutral polar organic solutes from water. *Analytical Chemistry*, 62: 1355-1356.
9. Miyares, P.H. and T.F. Jenkins (1990) Salting-out solvent extraction for determining low levels of nitroaromatics and nitramines in water. USA Cold Regions Research and Engineering Laboratory, Special Report 90-30.

11.0 SAFETY

11.1 Standard precautionary measures used for handling other organic compounds should be sufficient for the safe handling of the analytes targeted by Method 8330. The only extra caution that should be taken is when handling the analytical standard neat material for the explosives themselves and in rare cases where soil or waste samples are highly contaminated with the explosives. Follow the note for drying the neat materials at ambient temperatures.

11.2 It is advisable to screen soil or waste samples using Method 8515 to determine whether high concentrations of explosives are present. Soil samples as high as 2% 2,4,6-TNT have been safely ground. Samples containing higher concentrations should not be ground in the mortar and pestle. Method 8515 is for 2,4,6-TNT, however, the other nitroaromatics will also cause a color to be developed and provide a rough estimation of their concentrations. 2,4,6-TNT is the analyte most often detected in high concentrations in soil samples. Visual observation of a soil sample is also important when the sample is taken from a site expected to contain explosives. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

TABLE 1
ESTIMATED QUANTITATION LIMITS

Compounds	Water ($\mu\text{g/L}$)		Soil (mg/kg)
	Low-Level	High-Level	
HMX	-	13.0	2.2
RDX	0.84	14.0	1.0
1,3,5-TNB	0.26	7.3	0.25
1,3-DNB	0.11	4.0	0.25
Tetryl	-	4.0	0.65
NB	-	6.4	0.26
2,4,6-TNT	0.11	6.9	0.25
4-Am-DNT	0.060	-	-
2-Am-DNT	0.035	-	-
2,6-DNT	0.31	9.4	0.26
2,4-DNT	0.020	5.7	0.25
2-NT	-	12.0	0.25
4-NT	-	8.5	0.25
3-NT	-	7.9	0.25

TABLE 2
RETENTION TIMES AND CAPACITY FACTORS ON LC-18 AND LC-CN COLUMNS

Compound	Retention time (min)		Capacity factor (k)*	
	LC-18	LC-CN	LC-18	LC-CN
HMX	2.44	8.35	0.49	2.52
RDX	3.73	6.15	1.27	1.59
1,3,5-TNB	5.11	4.05	2.12	0.71
1,3-DNB	6.16	4.18	2.76	0.76
Tetryl	6.93	7.36	3.23	2.11
NB	7.23	3.81	3.41	0.61
2,4,6-TNT	8.42	5.00	4.13	1.11
4-Am-DNT	8.88	5.10	4.41	1.15
2-Am-DNT	9.12	5.65	4.56	1.38
2,6-DNT	9.82	4.61	4.99	0.95
2,4-DNT	10.05	4.87	5.13	1.05
2-NT	12.26	4.37	6.48	0.84
4-NT	13.26	4.41	7.09	0.86
3-NT	14.23	4.45	7.68	0.88

* Capacity factors are based on an unretained peak for nitrate at 1.71 min on LC-18 and at 2.00 min on LC-CN.

TABLE 3
SINGLE LABORATORY PRECISION OF METHOD FOR SOIL SAMPLES

	Spiked Soils			Field-Contaminated Soils		
	Mean Conc. (mg/kg)	SD	%RSD	Mean Conc. (mg/kg)	SD	%RSD
HMX	46	1.7	3.7	14	1.8	12.8
				153	21.6	14.1
RDX	60	1.4	2.3	104	12	11.5
				877	29.6	3.4
1,3,5-TNB	8.6	0.4	4.6	2.8	0.2	7.1
	46	1.9	4.1	72	6.0	8.3
1,3-DNB	3.5	0.14	4.0	1.1	0.11	9.8
Tetryl	17	3.1	17.9	2.3	0.41	18.0
2,4,6-TNT	40	1.4	3.5	7.0	0.61	9.0
				669	55	8.2
2,4-DNT	5.0	0.17	3.4	1.0	0.44	42.3

TABLE 4
MULTILABORATORY ERROR OF METHOD FOR SOIL SAMPLES

	Spiked Soils			Field-Contaminated Soils		
	Mean Conc. (mg/kg)	SD	%RSD	Mean Conc. (mg/kg)	SD	%RSD
HMX	46	2.6	5.7	14	3.7	26.0
				153	37.3	24.0
RDX	60	2.6	4.4	104	17.4	17.0
				877	67.3	7.7
1,3,5-TNB	8.6	0.61	7.1	2.8	0.23	8.2
	46	2.97	6.5	72	8.8	12.2
1,3-DNB	3.5	0.24	6.9	1.1	0.16	14.5
Tetryl	17	5.22	30.7	2.3	0.49	21.3
2,4,6-TNT	40	1.88	4.7	7.0	1.27	18.0
				669	63.4	9.5
2,4-DNT	5.0	0.22	4.4	1.0	0.74	74.0

TABLE 5
MULTILABORATORY VARIANCE OF METHOD FOR WATER SAMPLES^a

Compounds	Mean Conc. (µg/L)	SD	%RSD
HMX	203	14.8	7.3
RDX	274	20.8	7.6
2,4-DNT	107	7.7	7.2
2,4,6-TNT	107	11.1	10.4

^a Nine Laboratories

TABLE 6
MULTILABORATORY RECOVERY DATA FOR SPIKED SOIL SAMPLES

Laboratory	Concentration ($\mu\text{g/g}$)						
	HMX	RDX	1,3,5-TNB	1,3-DNB	Tetryl	2,4,6-TNT	2,4-DNT
1	44.97	48.78	48.99	49.94	32.48	49.73	51.05
3	50.25	48.50	45.85	45.96	47.91	46.25	48.37
4	42.40	44.00	43.40	49.50	31.60	53.50	50.90
5	46.50	48.40	46.90	48.80	32.10	55.80	49.60
6	56.20	55.00	41.60	46.30	13.20	56.80	45.70
7	41.50	41.50	38.00	44.50	2.60	36.00	43.50
8	52.70	52.20	48.00	48.30	44.80	51.30	49.10
True Conc	50.35	50.20	50.15	50.05	50.35	50.65	50.05
Mean	47.79	48.34	44.68	47.67	29.24	49.91	48.32
Std Dev	5.46	4.57	3.91	2.09	16.24	7.11	2.78
% RSD	11.42	9.45	8.75	4.39	55.53	14.26	5.76
% Diff*	5.08	3.71	10.91	4.76	41.93	1.46	3.46
Mean % Recovery	95	96	89	95	58	98	96

* Between true value and mean determined value.

TABLE 7
 COMPARISON OF METHOD ACCURACY FOR SOIL AND AQUEOUS SAMPLES
 (HIGH CONCENTRATION METHOD)

Analyte	Recovery (%)	
	Soil Method*	Aqueous Method**
2,4-DNT	96.0	98.6
2,4,6-TNT	96.8	94.4
RDX	96.8	99.6
HMX	95.4	95.5

* Taken from Bauer et al. (1989), Reference 1.

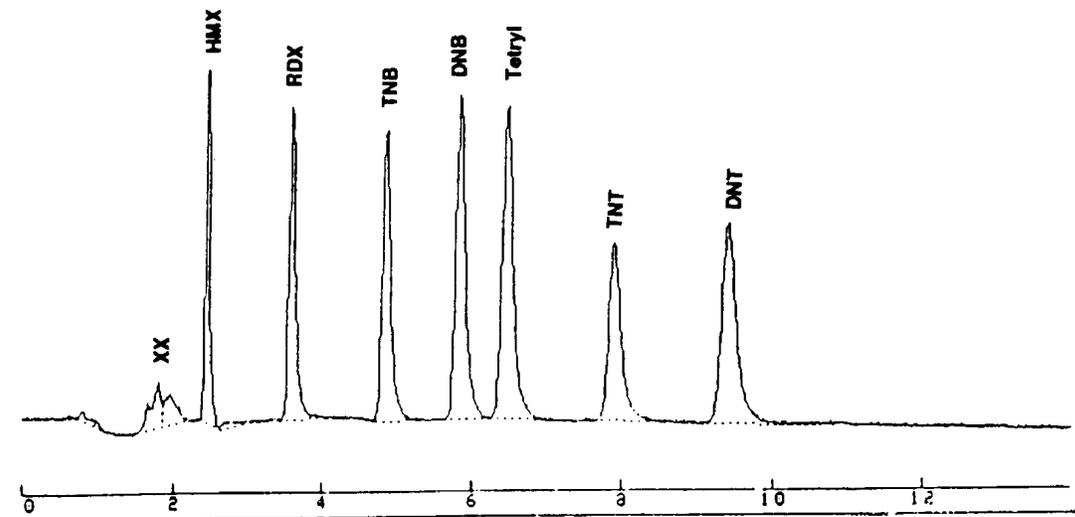
** Taken from Jenkins et al. (1984), Reference 3.

TABLE 8
PRECISION AND ACCURACY DATA FOR THE SALTING-OUT EXTRACTION METHOD

Analyte	No. of Samples ¹	Precision (% RSD)	Ave. Recovery (%)	Conc. Range (µg/L)
HMX	20	10.5	106	0-1.14
RDX	20	8.7	106	0-1.04
1,3,5-TNB	20	7.6	119	0-0.82
1,3-DNB	20	6.6	102	0-1.04
Tetryl	20	16.4	93	0-0.93
2,4,6-TNT	20	7.6	105	0-0.98
2-Am-DNT	20	9.1	102	0-1.04
2,4-DNT	20	5.8	101	0-1.01
1,2-NT	20	9.1	102	0-1.07
1,4-NT	20	18.1	96	0-1.06
1,3-NT	20	12.4	97	0-1.23

¹Reagent water

**EXPLOSIVES ON A
C18 COLUMN**



**EXPLOSIVES ON A
CN COLUMN**

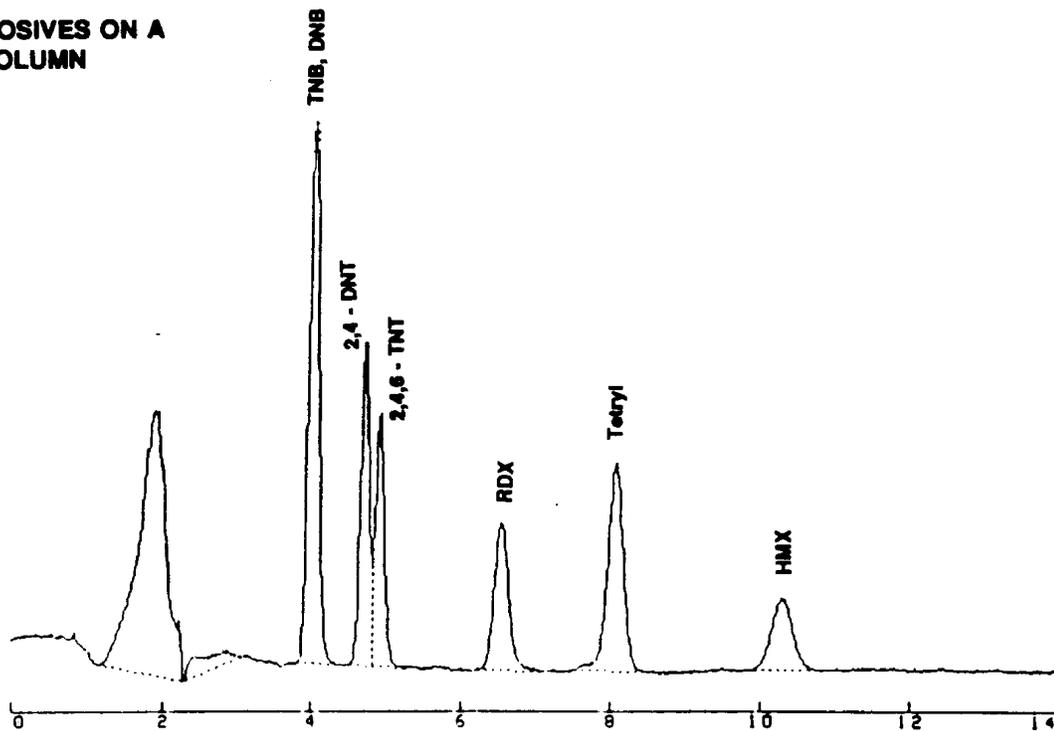
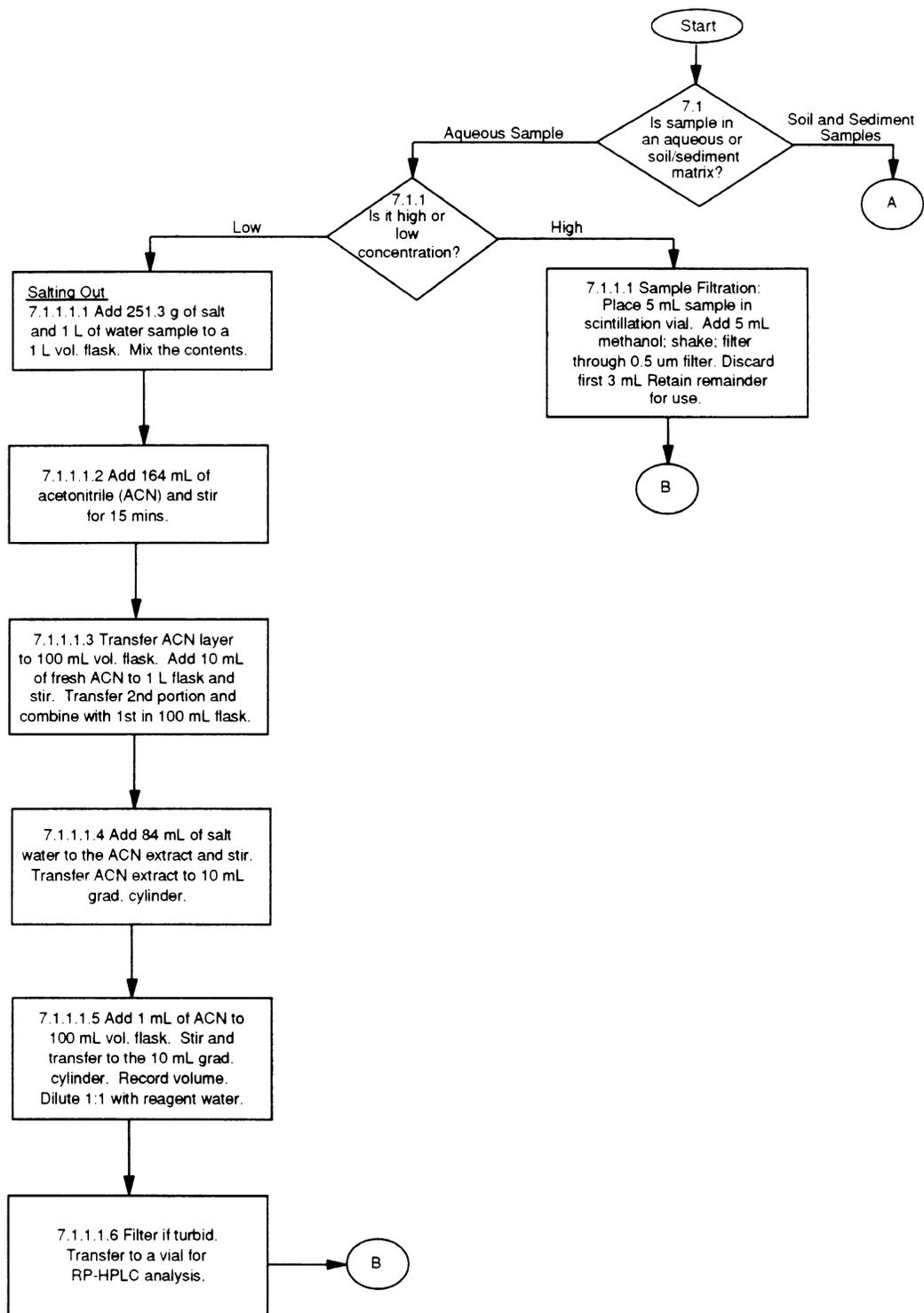
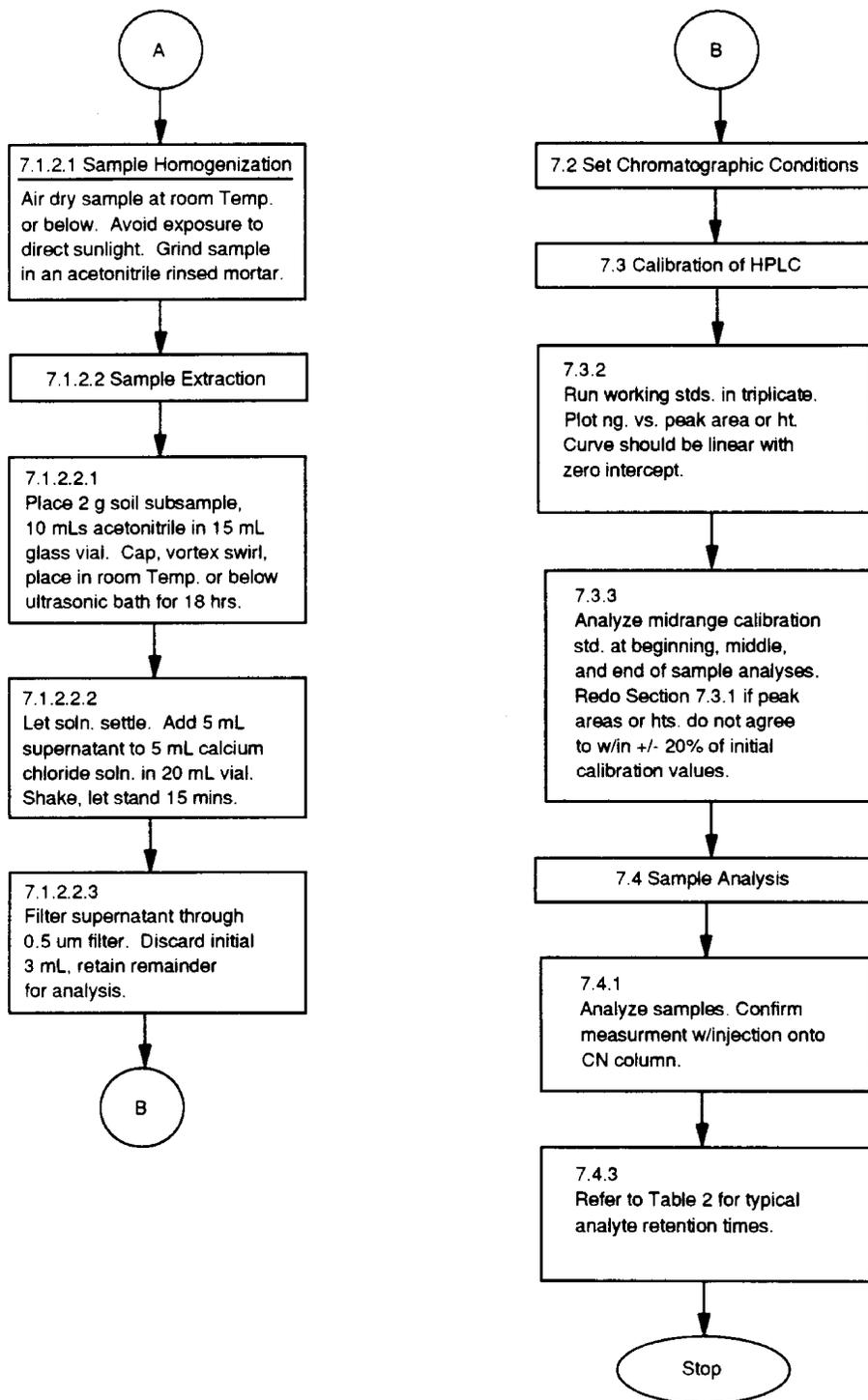


FIGURE 1
CHROMATOGRAMS FOR COLUMNS DESCRIBED IN Sec. 4.1.2.
COURTESY OF U.S. ARMY CORPS OF ENGINEERS, OMAHA, NE.

METHOD 8330
 NITROAROMATICS AND NITRAMINES BY HIGH
 PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)





Appendix B:

Quality Control Plan

Quality Control Plan

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Quality Control Plan

1 PURPOSE

The purpose of this Quality Control Plan is to specify the policies and procedures for conducting quality management in the successful accomplishment of all assigned task orders under the scope of work for this scrap processing action.

This Quality Control Program applies to all UXB team members involved in the execution of this delivery order.

2 QUALITY POLICIES AND OBJECTIVES

2.1 Quality Policies

All of UXB's services are consistent with the public good and will meet all applicable laws and regulations. Quality management is applied through all phases of the project until the Final Report is accepted. The Quality Program emphasis is placed on preventive actions.

All UXB employees and team members are empowered to and expected to identify and evaluate potential quality problem areas, and are encouraged to recommend solutions and corrective actions. The Delivery Order tasks are thoroughly reviewed by UXB personnel to assure all of the client's requirements are met and the appropriate UXB Quality Program elements are applied. All UXB personnel associated with the completion of the Delivery Order are provided with all the information necessary to accomplish their assigned tasks in a safe, responsible, and cost-efficient manner. All UXB employees and team members are held accountable for the quality of their work.

UXB employees and team members are provided with a Contracting Officer approved Work/Safety and Health Plan prior to the performance of any UXO-related activities on the project site. UXB provides and maintains an inspection system acceptable to the client. UXB takes timely corrective and preventive actions on any complaint or quality defect from an audit of operations. UXB's Quality Program is effective and economical, as the Quality Program is developed with input from both the technical and administrative sectors within UXB.

2.2 Quality Objectives

To provide quality services which consistently meet or exceed the expectations of the client, during the administration of any UXB Delivery Order.

UXB staffs all project sites with the best qualified, trained, and available personnel, based upon their knowledge and experience with the type of operations and hazards expected to be encountered. The minimum qualifications delineated in OT-025 are required for all UXB personnel involved in UXO-related activities.

To maintain a high degree of day-to-day quality awareness in all UXB employees and team members through UXB's management commitment to provide a high quality product for the client.

3 DEFINITIONS

Chemical Agent: A chemical agent listed in Appendix B (AR 50-6) that is intended for use in military operations to kill, seriously injure, or incapacitate a person through its physiological properties. Excluded from consideration are industrial chemicals, riot control agents, chemical herbicides, smoke, and flame.

Customer/Client: ESTCP Program Office

Explosive Ordnance Disposal (EOD) Personnel: Active duty military EOD personnel.

Government Representative: An on-site Government employee with specified responsibilities and authority.

Hazardous Toxic Radiological Waste (HTRW): Waste or media (i.e. air, water, soil, etc.) contaminated with chemicals or compounds that have been determined to be harmful to human health and the environment and are regulated by Federal and State Law.

Inert Ordnance: Inert ordnance is an item which has functioned as designed leaving an inert carrier, an item manufactured inert to serve a specific training purpose, or fragments from UXO.

Nonconformance:

A minor nonconformance is not likely to materially reduce the usability of the services. It is generally a departure from the approved procedures that have little bearing on the end-product.

A major nonconformance is likely to result in failure of the services or to materially reduce the usability of the end-product.

A critical nonconformance is likely to result in hazardous or unsafe conditions for individuals using or depending upon the services.

Ordnance and Explosives (OE): OE is anything designed to cause damage to people or material through explosive force, incendiary action, or toxic effects. This includes bombs; warheads; missiles; artillery; mortars; rockets; small arms ammunition; anti-personal and antitank mines; demolition charges; pyrotechnics; grenades; torpedoes and depth charges; high explosives and propellants; depleted uranium rounds; military chemical agents; and all similar and related items or components explosive in nature or otherwise designed to cause damage to people or material (fuzes, boosters, bursters, or rocket motors).

Quality Assurance: The procedures by which the Government fulfills its responsibility to be certain that QC is functioning and the specific product is realized.

Quality Conformance Inspection (QCI): Normal inspections/audits conducted by authorized UXB personnel during the accomplishment of the organization's mission to determine conformance to contract requirements.

Quality Control (QC): The UXB system to manage, control, and document the activities to comply with the contract requirements.

Quality Defect: A nonconformance issue with published policy and/or a contractual requirement that requires corrective action(s).

Quality Management: All those control and assurance activities instituted to safely and effectively accomplish the assigned mission.

Recovered Chemical Warfare Material (RCWM): As defined in EP 1110-1-18, CWM is an item configured as a munition containing a chemical substance that is intended to kill, seriously injure, or incapacitate a person through its physiological effects.

Root Cause: The basic reason for an undesirable condition or problem which, if eliminated or corrected, would have prevented it from existing or occurring.

Stop-Work-Authority: The right and obligation to stop all work when serious quality or safety concerns arise.

Supplier: The organization that provides a product or a service to the purchaser. When used in the Quality Systems definition of U. S. Government contracts, the term supplier denotes the contractor (UXB).

Unexploded Ordnance (UXO): As defined in EP 1110-1-18, UXO are military munitions that have been primed, fuzed, armed, or otherwise prepared for action, and have been fired, dropped, launched, projected, or placed in such a manner as to constitute a hazard to operations, installation, personnel, or material and remain unexploded either by malfunction, design, or any other cause.

UXO Personnel: Former EOD personnel provided by UXB.

4 ASSOCIATED MATERIAL

- DACA87-97-R-0006
- OT-005-01, Work Plan
- OT-015, Accident/Incident Report
- OT-025, Personnel Qualifications
- OT-030, Site Specific Removal Report
- OT-040, Disposal Feasibility Letter Report
- OT-045, Report/Minutes, Record of Meeting
- OT-055, Telephone Conversations/Correspondence Records
- OT-060, Conventional Explosives Safety Plan (ESS)
- OT-080, Monthly Status Report
- OT-085, Weekly Status Report
- Safety Concepts and Basic Considerations for Unexploded Ordnance Operations, USAESCH, February 16, 1996.
- Work Standards for Ordnance Response, USAESCH, October 25, 1994. [30 July 1996?]
- DoD Manual 4160.21.M Defense Reutilization and Disposal Manual
- DoD 6055.9 Std. DoD Ammunition and Explosive Safety Standard
- AR 200-1 Environmental Protection and Enhancement
- AR 385-10, The Army Safety Program
- AR 385-40, Accident Reporting and Records with USACE Supplement
- AR 385-63, Policies and Procedures for Firing Ammunition for Training, Target Practice and Combat.
- AR 385-64, Ammunition and Explosive Safety Standards
- Government-Furnished Technical Manual 60-series publications.
- MIL-I-45208A, Inspection System Requirements, dated July 24, 1981
- EM 385-1-1, CE Safety and Health Requirements Manual
- USAESCH Quality Management Plan for Ordnance and Explosives Program, Chapter 5
- UXB International, Inc., Quality Management Plan, UXBQUAL-1-96
- Root Cause Analysis, A Tool for Total Quality Management by Wilson, Dell, and Anderson

5 RESPONSIBILITIES

5.1 UXB Responsibilities

- UXB has the sole responsibility for the control of product quality and for offering products/services that conform to the contractual requirements.
- Ensure that the corporate quality policy and program is understood, implemented and maintained at all levels within the UXB team.
- Ensure that vendor and subcontractors have and maintain a quality control system acceptable to the Client.
- Maintain documented evidence that the services conform to the contract requirements. This includes the quality program procedures and processes, and the specified end product
- Provide and maintain a quality control inspection system that is acceptable to the client.
- Assure the Quality Program provides for the prevention and ready detection of quality defects, and for timely and positive corrective action. The evidence of quality performance and/or non-conformance shall be documented and readily available to the client and included in the project final report.
- Ensure the Quality Program provide for an equipment maintenance program which addresses 1) preventive maintenance, 2) routine repair and adjustment and, 3) emergency repair.
- Provide effective management for quality by assuring personnel performing quality functions has sufficient, well-defined responsibilities including stop-work authority, and the organizational freedom to identify and evaluate quality problems and to initiate, recommend or provide solutions.
- Provide a training program for all on-site personnel and those personnel that perform quality functions.

5.2 UXB Director of Quality Responsibilities

- Execution of and compliance with the Quality Management Program
- Delegation of the authority to administer the on-site quality control function to the on-site Quality Control Personnel.
- Meet or exceed the minimum qualifications for QC personnel.
- Issue a Certificate of Training documenting that training of the on-site QC personnel has been completed

5.3 UXB On-Site LCPM/SUXOS Responsibilities

- Assure that all personnel are aware of the tasks to be performed and the expected quality standard to which the task is to be performed.
- Coordinate with the UXB Director of Quality to accommodate the Quality Program activities when Quality Control Personnel are not on site.
- Immediately address customer complaints received verbally, by written correspondence. The UXB LCPM/SUXOS will conduct an investigation to analyze the complaint and

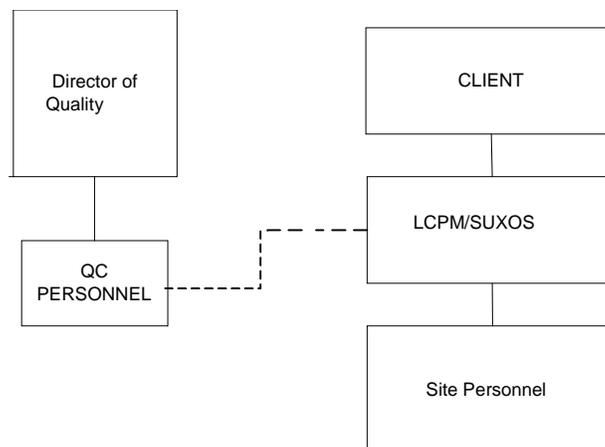
assure corrective action has been initiated. The corrective action will address the root cause and the controls applied to assure effectiveness. The LCPM/SUXOS will document the complaint and investigation on the current revision of the Customer Action Request (CAR) (UXB Form 11-00.1-1).

- Assure that all personnel are aware of the tasks to be performed and the expected quality standard to which the task is to be performed.
- Coordinate with the Quality Control Personnel when on site to accommodate the Quality Program activities.

5.4 UXB Quality Control Personnel Responsibilities

A Quality Control Supervisor will be assigned to the projects that warrant additional quality control personnel. If no Quality Control Supervisor is assigned, the Quality Control Specialist assumes the responsibilities of the Quality Control Supervisor. UXB will not have a full time QC Personnel on the project, but will dual-hat the UXOSO. This individual reports directly to the Director of Quality, but is a member of the on-site team. He will coordinate his scheduled activities with the LCPM/SUXOS. Figure 1 illustrates the UXB on-site QC organization.

Figure 1: UXB On-Site Quality Control Organization



This coordination and lateral communication are critical for proper implementation of the Quality Control Program. When on site QC personnel responsibilities are as follows:

- Assure that all personnel are aware of the tasks to be performed and the expected quality standard to which the task is to be performed.
- Conduct compliance audits on all site operations to assure the objectives of the project are being accomplished in accordance with the approved Work Plan. The Quality Control Inspection Schedule outlines the audits to be performed.
- Brief the on-site personnel, during site -specific training on the importance of quality work and review of the quality policies and objectives stated in Section 3 of this program. The briefing is aimed at ensuring that all site personnel understand the UXB Quality Policies and Objectives.
- Establish, operate, and maintain a site for daily instrument operational checks.
- Prepare the Daily QCI Report.

- Perform Quality Control Inspections.
- Maintain all required Quality Program Records
- Complete Part Three of CAR to verify corrective/preventive actions taken for the complaint/non-conformance have in fact addressed the issues. Schedules future QCI to monitor and ensure the CAR solution is effective.
- Initiate action to prevent occurrence of quality non-conformities relating to the provided services.
- Identify and record any quality problems relating to the services.
- Initiate, recommend or provide solutions through the compliance channel.
- Verifies implementation of solutions.
- Control further actions of non-conforming services until the unsatisfactory conditions have been corrected.

5.5 UXB UXO Safety Officer (UXOSO) Responsibilities

- Verifies that all required safety procedures are followed.
- Reports to the Director of Quality any safety program non-compliance.
- Conduct daily tailgate safety meetings.
- Conduct weekly supervisor safety meetings.
- Provide visitors general and specific hazards training.

5.6 UXB Site Team Members

- To perform all work in accordance with all the safety and health procedures.
- To perform all work with the highest standard of quality possible.
- To stop-work when operations are not being performed in accordance with established safety practices and/or the UXB Quality Program.
- Maintain qualifications current for the tasks being performed.
- Keep Field Supervision and Project Management informed of changing field conditions which could affect the safety and quality of the work being performed.

6 PROCEDURE

6.1 Quality Control Program

UXB's Quality Control (QC) Program has been established at the direction of the President. The responsibility for the development, integration and execution of the Quality Control Program resides with the Director of Quality. The authority to administer the on-site Quality Control (QC) Program is delegated to the on-site QC personnel.

- Approval authority for the Quality Control Program resides with the following UXB personnel:
 - President
 - Director, Environmental and Explosive Operations

- Director, AEDA & Technical Services
- Director of Quality
- Changes to the Quality Control Program are restricted to the approval authorities or their designees.
- The Quality Control Program is a key component of the Work Plan.

6.2 Work Plan

Prior to commencing development of the Work Plan, the UXB management team reviews the delivery order to assure the requirements are concisely defined and a clear understanding of the scope of work between UXB and the Client.

The Work Plan is the product of the Technical Services Group, which describes the process and procedures that will be used by UXB to complete the tasks specified in the delivery order.

Upon receipt of the delivery order, the Technical Services Group devotes the necessary resources to develop the Work Plan in accordance with the requirements of OT-005-11, for their customer, UXB's Environmental and Explosive Operations Sector.

The Work Plan is developed using the UXB Project Management Procedures. The design will comply with all of the applicable requirements of the references in Section 5 of this document.

The Work Plan generated by the UXB Technical Services Group is reviewed, at the appropriate phases, by the following:

- Life Cycle Project Manager/Senior UXO Supervisor assigned to the project
- Director, Environmental and Explosive Operations
- Certified Industrial Hygienist
- Safety/Training Manager
- Director of Quality

The Work Plan is used by on-site supervision to brief and train their team members at the project site.

6.3 Preventive Maintenance

The Work Plan will include a maintenance program for all site equipment that addresses: 1) preventive maintenance; 2) routine repair and adjustment; and 3) emergency repair.

Preventive maintenance includes scheduled or unscheduled maintenance performed by appropriate personnel, i.e., an operator, in an effort to maintain the equipment in a satisfactory operating condition. The LCPM/SUXOS's include before, during, and after maintenance checks, which are documented.

6.4 Property Accountability

When Government Furnished Property (GFP) is provided and Contractor Acquired Property (CAP) is purchased, the accountability for this equipment is controlled with the appropriate UXB Project Management procedures. The property administration fulfills the requirements of FAR Subpart 45.5. These procedures include the following:

- Examination upon receipt to detect damage in transit.

- Inspection for completeness and proper type
- Periodic inspection and precautions to assure adequate storage to guard against damage and theft.
- Function testing to determine satisfactory operation.

6.5 Quality Control Procedures

Quality Control Inspections (QCI) are normal inspections and audits conducted by authorized UXB personnel during the accomplishment of the organization's mission to determine conformance to contract requirements. Performance of QCI is addressed in the UXB Quality Control procedures. Attachment 1 to this document lists the Quality Control Inspection (QCI) Schedule. This QCI schedule includes all QCIs required by OT-005-11, Section 10.2.1, which are:

- Equipment Calibration audits
- Property accountability audits
- UXO-related audits
- Search Effectiveness (SE) audits
- Equipment operator maintenance audits
- Personal Protective Equipment (PPE) audits

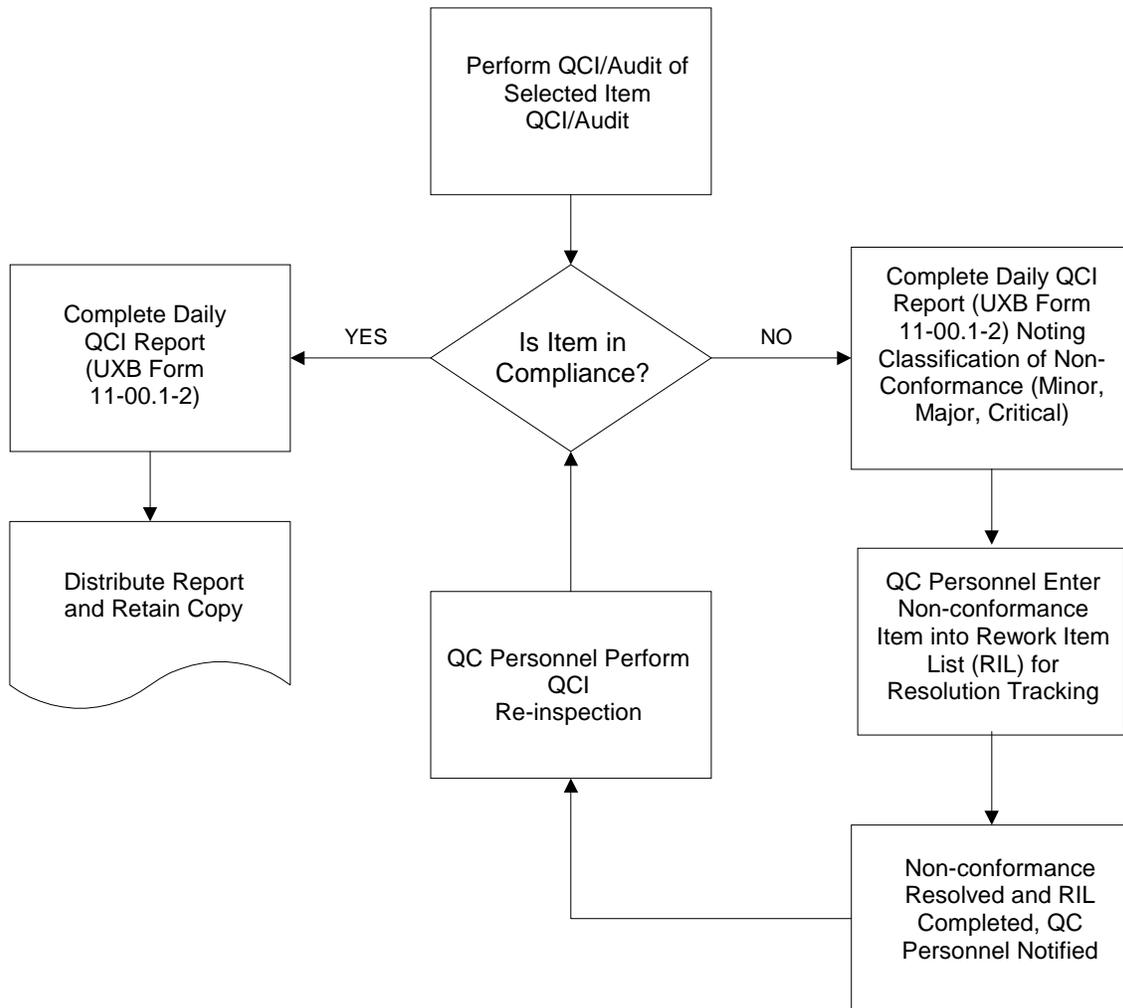
Not all QCIs apply to all projects.

Attachment 2 outlines the UXB quality control requirements for this scrap processing project. Additional quality control procedures are covered in Appendix F, which applies only to JPJ personnel and procedures.

QCI conducted on Hazard Assessment and Risk Analysis will be assessed in terms of hazard severity and accident probability and assigned a risk assessment code (RAC). The QCI will verify that a RAC has been completed by the on-site management, and that it is a valid RAC. The guidelines for RAC are contained in Attachment 3.

QCI Documentation is completed for QCIs found to be in compliance, those non-compliant and for re-inspection of non-conformance. The QCI findings are documented on the Daily QCI Report (UXB Form 11-00.1-2) Figure 2 illustrates the QCI/Audit procedure flowchart.

Figure 2: Quality Control Inspection (QCI)/Audit Flowchart



- The QCI Report is prepared daily in an electronic transmission format. The form documents the daily QCI activities performed by the UXB QC staff and the daily QA activities which were performed by the Client (USAESCH).
- Format is the same as UXB Form 11-00.1-2
- Prepared in Microsoft Word
- Submitted at the end of each workday.
- Distribution is as follows:
 - Richmond H. Dugger, III, Ph.D. – President, UXB International, Inc.
 - Client - as directed
- Daily QCI Reports will categorize non-conformant tasks as minor, major or critical.
- When a non-conformance occurs, the QC personnel record the non-conformance item on the Rework Item List (RIL) (UXB Form 11-00.1-4). The RIL is used by UXB Team

members to document the resolution of the non-conformance item. Upon completion of the rework item QC personnel are notified so that the item may be re-inspected for conformance and proper resolution.

- The Daily QCI Report will indicate all re-inspection nonconformances. Nonconformances have to be corrected by on-site management. Inaction by the on-site management will result in another QCI being performed, reported on the Daily QCI Report, and another Rework List Item generated.
- Once resolution has been achieved for the non-conformance on the RIL, the RIL will be signed off and completed, and QC personnel notified that the non-conformance has been corrected.
- If re-inspection indicates the non-conformance has been corrected, The completed Rework Item List will be retained and filed with the Daily QCI Report which noted the non-conformance

Customer Action Request (CAR) - Customer complaints are immediately addressed. The complaint may be by verbal comment, written correspondence, or a HND Form 948. The UXB LCPM/SUXOS will conduct an investigation to analyze the complaint and assure corrective action has been initiated. The corrective action(s) will address the root cause, as well as the application of controls to ensure the corrective action(s) effectiveness.

- The UXB LCPM/SUXOS will use the Customer Action Request (CAR) (UXB Form 11-00.1-1) to document the complaint or nonconformance and the investigation. The investigation will entail studying the symptoms of the problem and determining the root cause(s). Instructions for completing the form are on the reverse side of the form.
- The action on the CAR is not complete until QC Personnel have completed Part Three. The corrective/preventive actions must be adequate to prevent reoccurrence and the USAESCH must be satisfied with these actions.
- QC Personnel will schedule a future QCI to address the issues in the CAR to ensure that the corrective/preventive actions were successful.
- The Director of Quality will maintain a CAR log, which accounts for all CARs and indicates each CAR's status.

6.6 Quality Control Records

Quality Control records for all on-site activities are maintained on-site and available for USAESCH review. The QC Specialist establishes and maintains the following files:

- Daily QCI Report File - may be either hard copy or on a computer disk.
- CAR File - A two-part file containing active and inactive CAR's.

The Director of Quality will maintain a CAR log, which accounts for all CARs and indicates each CAR's status.

6.7 Quality Control Audits

The routine Quality Control Audits to be performed are listed in Attachment 1. This attachment lists the definable work breakdown number, feature of work, the auditable activity, the property of that activity to be audited, the QC phase and the audit frequency. The routine audits correspond to the QCIs and are documented as such.

6.8 Quality Control Training

The UXB Director of Quality will have the necessary experience in UXO related tasks to conduct the program. The Director of Quality exceeds the experience and training requirements of the QC Personnel.

The UXB QC personnel will meet the minimum qualifications as outlined in the OT-025, Personnel Qualifications.

Quality Control Training will be conducted on-site to all personnel of the UXB team by the Quality Control Personnel to meet the requirements of the Work Plan.

Safety Training for all site personnel will be conducted by the SSHO in accordance with the Work Plan. This training will consist of the following as a minimum:

- Daily tailgate safety meetings.
- Weekly Supervisor safety meetings.
- Visitor training for general and specific hazards training, as required.

7 DISTRIBUTION

In addition to being a component of the Work Plan, this QC Program is distributed to the following:

- President UXB
- Director, Environmental and Explosive Operations
- Director, AEDA & Technical Services
- Director of Quality
- Project Life Cycle Manager/Senior UXO Supervisor
- Project QC Personnel
- Project Bulletin Board (if any)

Attachment 1 – Quality Control Inspection (QCI) Schedule

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
01	Project Management				
	Administrative				
	Licenses/ATF/blasters/transport	PP/IP	O	Major	Verify all licenses and permits were obtained and current.
	Personnel Qualifications				
	Completeness	PP/IP	O	Major	Verify all required qualifications are complete.
	Currentness	IP/FP	M	Major	Check that all qualifications are up to date and current.
	Communications				
	System	PP/IP	O	Major	Review system meets requirements of contract.
	System	FP	D	Major	Verify system is present and functional.
	EMR Calculations	PP	O	Major	Confirm EMR calculations were performed and documented.
	Operational Checks	IP/FP	D	Major	Verify daily operational checks are being performed.
	System Redundancy	IP/FP	D	Major	Confirm system redundancy exists.
	Maintenance	FP	W	Major	Check that required maintenance is being performed.
	Management of GFP/CAP/PPP				
	Initial Receipt Inspection	PP	O	Minor	Review receipt inspection documentation for completeness.

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
	Maintenance & Testing	FP	W	Minor	Review and check maintenance and testing documents.
	Secured Storage	FP	W	Minor	Verify secured storage is intact and security is functional.
	Inventory	FP	W	Major	Confirm equipment inventories are being maintained properly.
	Energy Conservation	FP	M	Minor	Check that energy conservation is being practiced.
	Administration				
	Approved WP	PP/IP	O	Minor	Verify that an approved and current Work Plan is onsite.
	HR materials	PP/IP	O/M	Minor	Verify all HR materials are posted IAW HR Compliance Posting Requirements Checklist
	Field Cost Accounting	FP	W	Major	Examine cost accounting and budget for proper controls.
02	Design Development				
	Contract review	PP	O	Minor	Check that proper contract review was performed.
	Delivery Order review	PP	O	Minor	Verify DO review prior to start of Work Plan design.
	Regulatory review	IP	O	Minor	Confirm that all regulatory reviews are completed.
	Site visit	IP	O	Minor	Verify site visit provided information needed for design.
	Concept development	FP	O	Minor	Review design concept for technical and economic

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
					soundness.
	60% Review	FP	O	Minor	Verify 60% review was completed with proper approvals.
	100% Review	FP	O	Minor	Verify 100% review was completed with proper approvals.
03	Location Survey and Mapping				
	Equipment Calibrations	PP/IP	O	Major	Examine calibration records for completeness and currency.
	Marking of Clearance boundaries	FP	W	Major	Ensure established boundaries meet specified distances.
	Grid establishment				
	Markers	FP	W	Minor	Verify proper marker type, material and placement method.
	Designations	FP	W	Minor	Confirm markers are properly designated.
	Final drawings				
	Clearance boundaries/exceptions	FP	O	Minor	Check drawings for indication of boundaries and exceptions.
	Grids	FP	O	Minor	Verify grids are properly depicted on all drawings and maps.
	OE	FP	O	Minor	Confirm that all OE is properly located and identified.

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
04	Site Preparation				
	Environmental Controls (if required)	IP/FP	D	Major	Verify environmental controls are correct and functional.
	Brush/Vegetation Removal				
	Size Specification	FP	W	Major	Verify size of brush to be cut is IAW the Work Plan.
	Completion	FP	W	Minor	Confirm brush removal from each grid is complete.
	Disposition	FP	W	Minor	Check for proper disposition of brush removed from grids.
05	OE Surface Clearance				
	SEP Validation Tests	IP/FP	O/D	Major	Audit SEP using Attachment 2
	OE Clearance	FP	D	Critical	Audit OE clearance using Attachment 2
06	Geophysical Detection and Analysis				
	Instrumentation				
	Calibration	IP/FP	O/D	Major	Verify current calibration documentation for all instruments.
	Operation & Maintenance	FP	D	Minor	Check daily operation and maintenance forms for

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
					completeness.
	Data Collection				
	File Management	FP	W	Minor	Review data file management for to assure proper documentation.
	Data Analysis				
	Software selection/documentation	PP	O	Minor	Verify documentation and selection are IAW Work Plan
	Software verification	IP	O	Minor	Verify software verification documentation is completed.
	File management	FP	W	Minor	Confirm data handling, tracking and documentation are accurate.
07	OE Subsurface Clearance				
	Grid sheets	FP	W	Minor	Verify grid sheets properly indicate anomalies.
	Anomaly Reacquisition	FP	W	Major	Confirm anomaly reacquisition is accurate and complete.
	Anomaly Excavation (manual)				
	Procedure	FP	D	Major	Verify excavation procedure is being followed.
	Anomaly Excavation Log	FP	D	Minor	Confirm the excavation log is being properly completed.
	Anomaly Excavation				

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
	(mechanical)				
	Operator training	IP	O	Minor	Review heavy equipment training is current for operators.
	Procedure	FP	D	Major	Verify excavation procedure is being followed.
	Maintenance	FP	W	Minor	Audit heavy equipment maintenance IAW UXB form 1.0046.
	OE Assessment	FP	E	Major	Ensure OE assessments are performed for each OE identified.
	OE Removal	FP	D	Critical	Verify proper OE removal IAW the Work Plan.
08	OE Disposition				
	OE assessment	FP	E	Major	Ensure OE assessments are performed for each OE identified.
	Disposition evaluation				
	Fragmentation radius	FP	E	Major	Verify fragmentation calculations for each disposition.
	Environmental impacts	FP	E	Minor	Verify environmental impacts minimized for each disposition.
	Protective works requirements	FP	E	Minor	Verify proper protective works in place for each disposition.
	Disposition activity				
	Notifications	FP	E	Major	Confirm notifications are performed prior to a disposition action.

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
	Work Plan Compliance	FP	E	Major	Ensure disposition activity is performed IAW Work Plan.
	Documentation	FP	E	Major	Verify documentation for each disposition is properly completed.
	Backfill & stabilization	FP	W	Minor	Verify backfill and stabilization are accomplished IAW Work Plan.
09	Explosives Management				
	Demolition Supervisor				
	Training	PP/IP	O	Major	Check licenses and training are current and IAW Work Plan.
	Storage				
	Facility	PP/IP	O	Major	Verify facility meets all Work Plan requirements.
	Security	FP	D	Critical	Verify security is functional and meets all requirements.
	Fire protection				
	Fire extinguishers	IP/FP	O/W	Major	Confirm fire extinguisher type, size, mounting and serviceability.
	Fire Breaks	IP/FP	O/W	Major	Determine if fire breaks are present and intact IAW requirements.
	Inventory management	IP/FP	O/D	Critical	Audit inventory documentation, UXB forms 1.0032 & 1.0039

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
	Transportation – driver				
	Training/License	PP/IP	O	Major	Verify driver training and licenses are current and valid.
	Transportation – vehicle				
	Licensed vehicle	IP/FP	O/E	Major	Verify vehicle is licensed.
	Inspected	IP/FP	O/E	Major	Audit vehicle inspection documents, UXB forms 1.0038 & 1.0041
	Placards	IP/FP	O/E	Major	Verify placarding is correct and being used when required.
	Segregation	FP	E	Major	Confirm segregation is occurring and properly monitored.
10	Inert OE and OE Related Scrap				
	Facility	IP	O	Major	Verify scrap storage facility is proper and security measures intact.
	Segregation	FP	E	Major	Confirm scrap segregation is occurring and properly monitored.
	Transportation	FP	E	Major	Confirm scrap is being transported IAW Work Plan Requirements
	Disposition	FP	E	Major	Verify UXO-related scrap disposal IAW Work Plan & DRMO.
	Documentation	FP	E	Critical	Verify DD Form 1348-1 properly completed IAW DoD

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
					4160.21-M.
11	Environmental				
	Regulatory review	PP	O	Major	Verify Regulatory review has occurred & is properly documented.
	Environmental survey review	PP	O	Minor	Verify environmental survey review is properly documented
	Environmental assessments				
	Wetlands	PP/IP	O	Major	Verify wetlands assessment is completed and documented.
	Flora/fauna	PP/IP	O	Major	Verify flora/fauna assessments are completed and documented.
	Cultural resource assessment				
	Historical properties	PP/IP	O	Major	Confirm historical properties review is complete and documented.
	Archaeological sites/features	PP/IP	O	Major	Verify archaeological assessments are complete and documented.
	SPCC Plan	IP/FP	O/W	Major	Check if SPCC plan is in place and implementable.
	Solid waste control and disposal	IP/FP	O/W	Major	Verify solid waste disposal and control procedures are being used.
	Sanitary waste control and	IP/FP	O/W	Major	Verify sanitary waste and disposal is occurring IAW the

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
	disposal				Work Plan.
12	Quality Control				
	Coordination & Mutual Understanding Meeting	I/P	O	Major	Review SOW, Work Plan, QA/QC programs, and AHA. Examine work areas, discuss protocols and QCIs/audits. Document attendees.
13	Health and Safety				
	Records				
	Medical Surveillance	PP/IP/FP	O/O/M	Major	Audit medical surveillance records for completeness & currency.
	Daily Safety Briefing	FP	W	Major	Audit safety meeting documentation UXB form 1.0023.
	Weekly Supervisor Safety Mtg.	FP	W	Major	Audit safety meeting documentation UXB form 1.0023.
	Visitor Briefings	FP	W	Major	Audit site visitor documentation UXB form 1.0012.
	OSHA 200 log	FP	M	Major	Review and verify proper OSHA 200 log keeping.
	Accident & Incident reports	FP	E	Major	Verify timely and complete preparation of reports/notifications.
	Safety Inspections	FP	M	Major	Audit UXB form 1.0024 and observe safety inspection.

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
	PPE				
	Work Plan compliance	FP	W	Major	Verify implementation of PPE requirements and its proper usage.
	Emergency Response				
	Telephone numbers	IP/FP	O/W	Major	Verify telephone numbers are current and properly documented.
	Medical support locations	IP/FP	O/W	Major	Verify locations are identified and directions for each are available.
	Evacuation routes	IP/FP	O/W	Major	Verify evacuation routes are identified and documented.
	Onsite medical supplies	IP/FP	O/W	Major	Verify supplies are replenished and locations are suitable.
	Fire protection				
	Fire extinguishers	IP/FP	O/W	Major	Verify number, type, size, location and serviceability.
	Accident Hazard Analysis				
	Completeness	IP	O	Major	Verify AHA is complete IAW Work Plan requirements and SOW.
	Currentness	FP	W	Major	Verify AHA is updated to include Work Plan SOW revisions.
	MSDSs				
	Completeness	IP	O	Major	Confirm MSDS are current and readily accessible to

Number	Definable Feature of Work with Auditable Function	QC Phase	Frequency of Audit	Degree of Non-compliance	Audit Procedure
					personnel.
14	Training				
	Site specific				
	Records	PP/IP/F P	O/O/M	Major	Verify required personnel training documentation is completed.
	Content compliance	PP/IP	E	Major	Review training lesson plans/classes for required content.
	Hazard communication				
	Records	IP	E	Major	Verify documentation of communication of hazards to personnel.
	Explosives Training	PP/IP	O	Major	Verify completeness & currency of explosives training.

Frequency

Daily
W = Weekly
M = Monthly

O = Once
E = Each occurrence

QC Phases

IP = Initial Phase
PP = Preparatory Phase
FP = Follow-up Phase

D =

Attachment 2

UXB Quality Control Procedures

1. UXOQCS will be responsible for monitoring the inspection of OE scrap prior to its placement in the transport/storage containers.
2. The QCS will ensure that all scrap is inspected (100%) and that all BDUs that cannot be visually inspected will be probed to ensure that there are no explosives present.
3. The QCS will ensure that all storage/transport containers are locked at the end of each work day. SUXOS will retain keys for containers which are not ready for shipment.
4. The QCS will sign, with the SUXOS, the certification statement for the scrap in the transport/storage containers.
5. The QCS will inspect a minimum of 10% of each days inspected quantity of scrap. He may be assisted by the UXO team, depending on volume and type of scrap. This inspection may be conducted during the day as part of the processing of the scrap.
6. If a piece of scrap which was initially declared free of explosive hazard is found to contain such a hazard, the UXO technician who originally inspected the piece of scrap will be removed from the operation and retrained by the SUXOS and UXOQCS in the proper inspection technique. All scrap which may have been inspected by this UXO technician that day will be re-inspected to insure that it is free of explosive hazard.
7. The QCS will retain the keys to the transport/storage containers once they are full and awaiting transport to JPJ for processing (ready for shipment). The QCS will also retain a copy of the certification statement for each full container.
8. Once all scrap has been inspected and either placed into a transport/storage container for shipment or transferred to local authorities for disposal, the QCS will travel to the JPJ processing plant and will observe the final processing of the scrap. He will obtain the final processing documents from JPJ and transport them to UXB for inclusion in the final report.

Attachment 3

Risk Assessment Code (RAC) Guidelines

Hazard Severity

Accident Probability

<p>Category: I Description: Catastrophic Definition: Death or permanent total disability, system loss, major property damage.</p>	<p>Description: Frequent Level: A Individual item: Likely to occur frequently in life of system, item, facility, etc. Fleet or inventory: Continuously experienced.</p>
<p>Category: II Description: Critical Definition: Permanent partial disability or temporary disability in excess of 3 months, major system damage, significant property damage.</p>	<p>Description: Probable Level: B Individual Item: Will occur several times in life of item. Fleet or inventory: Will occur frequently.</p>
<p>Category: III Description: Marginal Definition: Minor injury, lost workday accident, or compensable injury or illness; minor system damage; minor property damage.</p>	<p>Description: Occasional Level: C Individual Item: Likely to occur sometime in life of item. Fleet or inventory: Will occur several times.</p>
<p>Category: IV Description: Negligible Definition: First aid or minor supportive medical treatment, minor system impairment.</p>	<p>Description: Remote Level: D Individual Item: Unlikely but possible to occur in life of item. Fleet or inventory: Unlikely, but can reasonably be expected to occur.</p>
	<p>Description: Improbable Level: E Individual Item: So unlikely it can be assumed occurrence may not be experienced. Fleet or inventory: Unlikely to occur, but possible.</p>

Risk Assessment Code Matrix (AR 385-10)						
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Hazard Severity	Accident Probability					
		A	B	C	D	E
I	1	1	2	3	5	
II	1	2	3	4	5	
III	2	3	4	5	5	
IV	3	4	5	5	5	

Attachment 4
Daily QCI Report

DAILY QUALITY CONTROL INSPECTION REPORT

PROJECT:
CONTRACT:
DATE:
QC SPECIALIST:

PART ONE - UXB QC

TASK INSPECTED ⁽¹⁾	RESULTS ⁽²⁾	COMMENTS ⁽³⁾
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PART TWO - USAESCH QA

TASK INSPECTED	RESULTS ⁽²⁾	COMMENTS ⁽³⁾
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¹ From QCI Schedule

² C - Conforms, N - Nonconformance, MI - Minor, MA - Major, CR - Critical, i.e., N-Minor

³ Briefly describe Nonconformance(s);

Appendix C:
Site-Specific Health and Safety Plan

Site-Specific Safety and Health Plan

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List of Acronyms

ACGIH	American Conference of Government Industrial Hygienists
BMGR	Barry M. Goldwater Range
CFR	Code of Federal Regulations
CSHP	Corporate Safety and Health Plan
CTD	Cumulative Trauma Disorder
CWM	Chemical Warfare Materiel
HBV	Hepatitis B Virus
HCV	Hepatitis C Virus
HIV	Human Immunodeficiency Virus
HTRW	Hazardous, Toxic, and Radiological Waste
IDHL	Immediately Dangerous to Life or Health
OE	Ordnance and Explosives
OSHA	Occupational Health and Safety Administration
PELs	Permissible Exposure Levels
PPE	Personal Protective Equipment
QC	Quality Control
SOPs	Standard Operating Procedures
SOW	Scope of Work
SSHP	Site-Specific Safety and Health Plan
SUXOS	Senior UXO Supervisor
TLVs	Threshold Limit Values
UXB	UXB International, Inc.
UXO	Unexploded Ordnance
UXOSO	UXO Safety Officer
WBGT	Wet-Bulb Globe Temperature

Site-Specific Safety and Health Plan

1 UXB CORPORATE SAFETY AND HEALTH PLAN

UXB places the highest priority on a safe working environment. To substantiate its commitment, UXB has developed and implemented a Corporate Safety and Health Plan (CSHP) that provides general safety and health guidance to:

- Qualified individuals performing Demilitarization Services
- Qualified individuals performing Ordnance and Explosive (OE) operations
- Qualified individuals performing Hazardous, Toxic, and Radiological Waste (HTRW) operations
- Approved visitors to a site potentially containing OE or HTRW

UXB's CSHP complies with federal, state, and local laws, statutes, directives, and ordinances that relate to worker safety and health, and the protection of the environment, including:

- 29 Code of Federal Regulations (CFR) 1910.120 (Occupational Safety and Health Administration (OSHA) standards)
- 29 CFR 1926.65 (OSHA standards)

UXB's CSHP was most recently updated in August 2000. The Contracting Officer may request a copy for review from UXB's Operations Director.

2 SITE-SPECIFIC SAFETY AND HEALTH PLAN (SSHP)

This SSHP is tailored to the working environment anticipated at Barry M Goldwater Range (BMGR), considering the level of effort required to accomplish the tasks as required for the proposed work site and the anticipated hazards associated with on-site operations. The SSHP describes the procedures and control measures to protect personnel from these hazards. Adherence to these procedures will significantly reduce, but not eliminate, the potential for occupational injury and illness.

Should operational circumstances substantially differ from those described and/or anticipated, operations shall be temporarily terminated until the suspect hazards are evaluated and appropriate health, safety, and operational precautions are implemented.

A SSHP is required on sites regulated by 29 CFR 1910.120(b)(4), 29 CFR 1926.65(b)(4), and ER 385-1-92. This SSHP complies with the requirements of:

- CFR 1910.120 (OSHA - Hazardous Waste Operations and Emergency Response)
- ER 385-1-92 (U.S. Army Corps of Engineers' Safety and Occupational Health Document for Hazardous, Toxic, and Radioactive Waste (HTRW) and Ordnance and Explosive (OE) Activities)
- EM 385-1-1 (U.S. Army Corps of Engineers' Safety and Health Requirements Manual)

2.1 General

UXB is assigning fully qualified personnel versed in the operational and safety aspects of the HYDROLYSIS PROCESS. The UXO Technicians assigned will implement the on-site elements of the SSHP. All authorized visitors to the site during the HYDROLYSIS PROCESS operations will also adhere to the guidelines of this SSHP.

2.1.1 Qualifications and Responsibilities

Direction and administration of our safety program is a corporate responsibility; implementation is the overall and direct responsibility of the HYDROLYSIS PROCESS Operations Director, who will provide the guidance, support, and resources necessary to promote compliance with all aspects of the SSHP. All assigned personnel are trained, qualified, and experienced. This section outlines the safety-related qualifications and responsibilities of key personnel. The operational responsibilities for each person are listed in the Technical Management Plan.

2.1.1.1 Senior UXO Supervisor (SUXOS)

Our proposed SUXOS is a graduate of an approved EOD school, and meets all requirements of this position. He reports to the HYDROLYSIS PROCESS Operations Director, and his safety and health responsibilities include:

- Ensure compliance with the on-site elements of the SSHP
- Recommend changes to PPE levels (as site conditions warrant)
- Evaluate potential safety problems and implement safety-related corrective actions
- Conduct Quality Control Inspections on the processed BDU-33's
- Conduct safety inspections, document findings, and implement corrective actions
- Investigate and report accidents/incidents as soon as possible
- Maintain the daily safety log

Provide and document employee training regarding site specific hazards

2.1.1.2 UXO Technician

The other assigned UXO Technician will be a graduate of approved EOD schools and have all appropriate specialized training to operate on the HYDROLYSIS PROCESS.

Reporting to the Senior UXO Supervisor, the UXO Technician has stop work authority to resolve and correct unsafe conditions. The safety and health responsibilities for this position include:

- Maintaining continuous communications with Supervisor(s) during HYDROLYSIS PROCESS operations
- Conduct Quality Control Inspections on the processed BDU-33's
- Ensuring team compliance with all safety and work-related SOPs
- Coordinating with the SUXOS to ensure all site safety considerations are being enforced

2.2 Site Description and Contamination Characterization

2.2.1 Site Description

The work site is adjacent to an active range and training area used for the firing of air delivered and projected ordnance with both practice (inert) and live (explosive) charges. For the purpose of this operation, the only ordnance to be processed will be BDU-33's.

2.2.2 Contamination Characterization

Based upon a review of the available information, personnel can expect to encounter the hazards identified in Table 1.

Table 1: On-Site Hazards

Hazard	Concentration on Range	Media	On-Site Location	Quantity Fired
Ordnance				
BDU-33 Practice Bombs	Unknown	Consolidated	Unknown	Unknown
Chemical				
Documents indicate that there is no CWM, however there are hazardous or toxic waste (HTW) hazards associated with this operation, and there are process chemical hazards				
Radiological				
Available documentation indicates that there are no radiological hazards associated with this site.				
Biological				
Snakes (Sonoran Coral Snake, Rattlesnakes)				
Insects (Ticks, Bees, Wasps, Hornets and Mosquitoes)				
Spiders (Black Widow and Brown, Recluse)				

2.3 Hazard Analysis and Risk Assessment

All known or potential ordnance, chemical, biological, and radiological hazards that may pose a threat to the health and safety of site workers have, to the extent possible, been identified and the risk of exposure assessed to ensure workers are adequately informed and protected. Emphasis has been placed on identifying situations and tasks that have known or may create Immediately Dangerous to Life or Health (IDLH) conditions, or any other condition with the potential for serious safety or health effects.

The overall hazards and risks associated with participation in operations on this site are **Moderate-Low**. The anticipated hazards and the risks are discussed in this section and summarized in Table 3-4. This table ensures compliance with the revised OSHA standard 29 CFR 1910.132, which requires assessment of tasks and operational hazards where personal protective equipment (PPE) is required to protect personnel.

Evaluation of work site characteristics and hazards is an ongoing process, and on-site personnel will play a major role in continuing this evaluation throughout the duration of this evolution. All

site workers must be vigilant in identifying and informing their supervisor of hazards. This section will be modified whenever new hazards are encountered. This section:

- Identifies the physical, safety, and biological hazards for each task/site operation
- Identifies, specifies, and justifies action levels for engineering controls and practice controls, emergency evacuation, and prevention/minimization of public exposure

2.3.1 Operational Site

Table 2 lists the task-specific hazards.

Table 2: Task-Specific Hazards

Tasks	Hazards												
	Physical				Safety			Biological					
	Heat/Cold Stress	Thunderstorms/Lightning	Exertion	Repetitive Motion	UXO	Slips, Trips, and Falls	Cuts	Wildlife	Snakes	Insects	Spiders	Rodents	Poison Ivy/Oak/Sumac
BASE HYDROLYSIS Operations	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	

2.3.2 On-Site Hazards and Preventative Measures

2.3.2.1 Physical Hazards

2.3.2.1.1 Heat/Cold Stress

Heat/Cold Stress disorders and monitoring procedures are detailed in Section 2.13.

2.3.2.1.2 Tornado

A tornado appears as a rotating, funnel-shaped cloud that spins like a top – striking the ground with winds that can exceed 200 miles per hour. While they can occur at any time of the year, they appear most often in the late afternoons of April, May, and June.

A “Tornado Watch” means that tornadoes are possible. Stay tuned to area radio/television reports and keep a watch on the sky. A “Tornado Warning” means that a tornado has been sighted; take immediate shelter. When a tornado is sighted:

- Take immediate shelter and stay away from windows, doors, and outside walls
- Go to the lowest interior level of a building and seek shelter under a sturdy table
- If in a vehicle, immediately exit and seek more substantial shelter

- If there is no shelter nearby, lie flat in the nearest ditch, ravine, or culvert with your hands shielding your head

2.3.2.1.3 Thunderstorm

Severe thunderstorms are one of the most common natural hazards

Large hail can cause serious injury, so avoid the outdoors while a storm is in progress. Shelter vehicles in order to prevent costly damage.

Flash floods often occur without warning following heavy upstream rainfall. Drainage canals, streambeds, canyons, or areas downstream from a dam are potential flood areas. Monitor current weather conditions and review evacuation plans. Roads and trails that parallel existing drainage systems may be swept away by floodwaters. When a flash flood warning is issued, or you realize a flash flood is coming, go to high ground immediately, don't drive through already flooded areas, and do not attempt to cross a flowing stream on foot where the water is above your knees.

2.3.2.1.4 Lightning

Electrical storms occur during the spring, summer, and fall. Since the storms are often fast moving, field personnel should watch for indications of electrical storms (forecasts should be covered in the morning safety briefing).

The distance to an electrical storm can be estimated by observing the interval between the lightning flash and the sound of the thunder. Since sound travels approximately 1,100 feet per second, an interval of 5 seconds corresponds to a storm distance of approximately 1-mile.

If caught in the open by an electrical storm, all personnel will immediately seek shelter in their vehicle and proceed to the site office for further instructions. In the event that their vehicle is inaccessible, personnel should follow these rules:

- Move to a topographically low area, away from tall objects and conductors (trees, transformers, fences, pipelines, power lines, metal sheds) and wait for the storm to leave the area.
- If you feel your hair stand on end (an indicator that lightning is about to strike), drop to your knees and bend forward, putting your hands on your knees. Do not lie flat on the ground.

2.3.2.1.5 Exertion

The Senior UXO Supervisor will establish a work/rest regimen that is conducive to on-site conditions. Personnel are to adhere to this regimen.

2.3.2.1.6 Repetitive Motion

Repetitive motion injuries, or--more specifically--occupationally related motion disorders, are now common problems. Numerous terms, including cumulative trauma disorder, overuse syndrome, repetitive stress injury, and repetition strain injury; have been used to describe this disorder. In the United States, cumulative trauma disorder (CTD) is the preferred label.

Many different symptoms can arise from the accumulation of small injuries or stresses to the body. CTD is not so much a disease as it is a response to excessive demands that we place on our bodies - without giving them adequate time to recover between. CTD results from a combination of muscle tension, repetitive motion, over use, and incorrect motion.

Forward head posture is one of the leading risk factors for CTD. It is also known as slouching, or rounded-shoulders. In this posture, the weight of the head is no longer being carried directly over the spine. Instead, the head is moved forward ahead of the spine. This mechanically increases the weight of the head on the neck by about 300 percent. The rounding forward of the shoulders produces a stretch weakness of the posture muscles of the upper back which must also work to hold the head upright. Therefore, the load on these muscles is dramatically increased while their strength is significantly decreased. This can lead to several chronic muscle problems in the upper back.

When the upper back is rounded forward, the head must bend backward slightly on the neck to restore level vision. This can cause pressure to the soft tissues at the base of the skull and can lead to chronic headaches.

The forward posture of the head on the neck causes the muscles under the chin to be pulled tight, which can pull the jaw back, causing pressure and irritation in the joint where the jaw attaches to the skull. Forward head posture also moves the shoulder joint from the side of the body toward the front of the body, causing pressure on the shoulder joint tendons, risking shoulder tendonitis. Finally, this position of the head on the neck can cause the lateral neck muscles to shorten, causing pressure on the nerves and blood vessels passing through these muscles on their way to the working arm.

Another commonly overlooked but very significant CTD risk factor is lack of upper extremity support. Some tasks require the holding of arms up slightly all day, fighting gravity. This is hard work for the neck and shoulder muscles and magnifies the stresses described for forward head posture. One of the best ways to reduce excessive work fatigue and forward head posture stresses is to arrange the work so that there is some space available for the arms to rest, even if only for a split second. This can greatly reduce the physical work of sustained posture.

Posture plays a significant role in CTD. Slouching with the spine or leaning with the head puts the body out of balance and causes the limbs to be stretched or bent awkwardly. The neutral spine position starts with the three natural curves of the spine - the inward curve of the neck (cervical) region, the outward curve of the mid back (thoracic) region, and the inward curve of the lower back (lumbar) region. Too much bending (flexing) or straightening (extension) in either the cervical or the lumbar region takes the spine out of its neutral position and increases the risk of injury.

In summary, to avoid CTD, exercise the following precautions:

- Instead of leaning with the head, rotate the upper body forward at the hips.
- Instead of slouching, rotate the upper body forward at the hips.
- Instead of bending or lifting with a flexed lumbar spine, rotate the upper body forward at the hips.
- Take breaks as required
- Don't be a statue
- Avoid caffeine and tobacco

2.3.2.2 Safety Hazards

2.3.2.2.1 Slips, Trips, and Falls

Protruding objects, careless movements, and placement of materials on pathways can cause slips, trips, and falls. To prevent these injuries:

- Be alert to obstacles in your path
- Maintain proper footing
- Remove tripping hazards, when encountered
- Maintain three points of contact when working on elevated equipment

2.3.2.3 Cuts

Personnel shall wear, inspect, and maintain designated PPE equipment and supplies. Personnel shall ensure cutting devices are properly stored when not in use.

The following shall be used to control minor bleeding:

- Wear surgical gloves or some other barrier, place sterile gauze/cloth over the bleeding area and apply direct pressure. Do not removed blood-soaked bandage; add more to the top.
- If bleeding persists, elevate the area to help reduce blood flow and continue applying direct pressure to the bleeding area.
- If bleeding continues, apply pressure at a pressure point (brachial or femoral) and continue with direct pressure over the wound.
- Seek medical attention.

2.3.2.4 Biological Hazards

2.3.2.4.1 Snakes and Reptiles

Rattlesnakes, Arizona Coral and Massasauga snakes are indigenous this area, and each are venomous. Venoms are complex poisons that vary greatly in composition and potency among species. These snakes share some common physical characteristics – including triangular shaped heads, a facial pit, elliptical pupils, and foldable fangs.

A venomous snakebite is usually characterized by pain and swelling at the site of the bite, and general skin discoloration. The manifestations of the bite include general weakness, rapid pulse, nausea and vomiting, and shortness of breath.

First Aid – Get the victim to the hospital and professional medical care. Meanwhile, the following first aid measures should be taken:

- Calm and reassure the patient. Keep the patient as still as possible, and do not allow him to run or move about unnecessarily.
- Remove any constricting items, such as jewelry, from the affected limb.
- When practical, immobilize the affected limb at approximately heart level.
- The victim should remain in a comfortable prone position, and body temperature should be stabilized to minimize shock. Do not delay evacuation.
- The bitten area may be washed with soap and water, and blotted dry with sterile gauze. Dressings and bandages may be applied.
- Transport to a medical facility for evaluation and treatment. Do not delay evacuation

Prevention of Snakebite—The best snakebite treatment is to avoid being bitten. The following suggestions will help in this process:

- Watch where you sit, and where you put your hands and feet.
- Avoid rock piles, stacks of old boards, and brush in wooded areas. If movement is necessary, use a remote means to initially relocate the material. Prior to entering a heavily wooded or brush area, look and listen carefully.
- Never handle "dead" snakes; they may not be completely dead.
- Do not attempt to capture or kill snakes.

Identification of Poisonous Snakebites--

- Severe burning pain at the bite site
- Two small puncture wounds about one-half inch apart (some cases may have only one fang mark)
- Swelling (happens within 5 minutes and can involve an entire extremity)
- Discoloration and blood-filled blisters possibly developing in 6 to 10 hours
- In severe cases, nausea, vomiting, sweating, and weakness

If someone is bitten--

- Get the victim and bystanders away from the snake. Snakes have been known to bite more than once. Pit vipers can strike about one-half their body length. Be careful around a decapitated snake head - head reactions can persist for 20 minutes or more.
- Keep the victim quite. If possible, carry the victim or have the victim walk very slowly to help.
- Gently wash the bitten area with soap and water.
- If you are more than one hour from a medical facility with antivenin or if the snake was large and the victim's skin is swelling rapidly, immediately apply suction with the Extractor™ (from Sawyer Products). It does not require cutting.
- Seek medical attention immediately. This is the most important thing to do for the victim.

Nonpoisonous snakebite actions--

- Gently clean the bite site with soap and water
- Care for the bite as you would a minor wound
- Seek medical advice

2.3.2.4.2 Spiders

Use extreme caution when lifting manually moving debris, handling BDU-33's, etc., since spiders may typically be found in these areas.

Black Widow Spider (Latrodectus Mactans)

Description – Varies from dark brown to black in color. Body is ¼ inch wide and overall size is 1-½ inches with legs extended. Female has a red or yellow hourglass marking on the underside of the abdomen. Only the female is poisonous.

Signs and Symptoms—Determining whether a Black Widow Spider has bitten a person is difficult. The victim will experience:

- A sharp pinprick of the spider's bite may be felt, although some victims are not even aware of the bite. In no more than 15 minutes, a dull, numbing pain develops in the bitten area.
- A faint red bite mark appears.
- Muscle stiffness and cramps—usually affecting the abdomen when the bite is in the lower part of the body or legs, and affecting the shoulders, back, or chest when the bite is on the upper body or arms.
- Headache, chills, fever, heavy sweating, dizziness, nausea, vomiting, and severe abdominal pain.

First Aid

- If possible, catch the spider to confirm its identity. Even if the body is crushed, save it for identification.
- Clean the bitten area with soap and water or rubbing alcohol. Do not apply a constricting band because the black widow venom's action is swift; there is little to be gained by trying to slow absorption with a constriction band.
- To relieve pain, place an ice pack over the bite.
- Keep the victim quiet and monitor breathing
- Seek immediate medical attention.

Brown or Violin Spider (*Lox Osceles Reclusa*)

The brown spider (sometimes referred to as the Brown Recluse) is light tan to brown with a violin-shaped figure on its back. With legs extended it is approximately the size of a quarter.

Signs and Symptoms

- The initial pain may be slight enough to be overlooked
- A blister at the bite site, along with redness and swelling, appears after several hours
- Pain, which may remain mild but can become severe, develops within two to eight hours at the bite sight
- Fever, weakness, vomiting, joint pain, and a rash may occur
- An ulcer forms within a week; gangrene may develop in some cases

First Aid

- If possible, catch the spider to confirm its identity. Even if the body is crushed, save it
- Clean the bitten area with soap and water or rubbing alcohol
- To relieve pain, place an ice pack over the bite
- Keep the victim quiet and monitor breathing

- Seek immediate medical attention

2.3.2.5 Ticks

Lyme Disease is caused by a bacterium, which may be transmitted by the bite of a tick. Ticks carrying Lyme Disease may be found throughout the U. S. living in grassy and wooded areas, and feeding on mammals such as mice, shrews, birds, raccoons, opossums, deer, and humans. Not all ticks are infected with the bacterium. Lyme Disease is transmitted by deer ticks that are very small, about the size of the period at the end of this sentence. Wood ticks or dog ticks are larger, about the size of a capital O. When an infected tick bites, the bacterium is passed into the bloodstream of the host, where it multiplies. If detected early, Lyme Disease can be treated with antibiotics.

Remove ticks with small tweezers. Do not squeeze the tick's body. Grasp it where the mouthparts enter the skin and tug gently, but firmly, until it releases its hold on the skin. Save the tick in a jar labeled with the date, body location of the bite, and the place where it may have been acquired. Wipe the bite with antiseptic and seek medical attention as soon as possible.

The illness typically occurs in the summer months and is characterized by a slowly expanding red rash that develops a few days to a few weeks after the bite of an infected tick. Flu-like symptoms, headache, stiff neck, fever, muscle aches, and/or general malaise can accompany the illness. At this stage, treatment by a physician is usually effective; but if left alone, these early symptoms may disappear and problems that are more serious may follow. A second stage symptom may be a partial facial paralysis, or palsy, on one side of the face. The most common late symptom of the untreated disease is arthritis; other problems include meningitis, neurological, and cardiac abnormalities. NOTE: some people do not get the characteristic rash but progress directly to the later manifestations. Treatment of follow-on symptoms is more difficult than early symptoms and is not always successful.

Rocky Mountain Spotted Fever is another tick borne disease. Nearly all cases of infection occur in the spring and summer, generally several days after exposure to infected ticks. The onset of illness is abrupt and often accompanied by high fever, headache, chills, and severe weakness. After the fourth day of fever, victims develop a spotted pink rash that usually starts on the hands and feet and gradually extends to most of the body. Early detection and treatment significantly reduces the severity of illness. The disease responds to antibiotic therapy with tetracycline or chloramphenicol.

Precautions:

- Wear long pants and long sleeved shirts that fit tightly at the ankles and wrists; tape cuffs if necessary.
- Wear light colored clothing so ticks can be easily spotted.
- Tick repellents should be used. (Recommend the application of vapor active repellent such as "DEET" to any exposed skin surface [except eyes and lips] and application of a repellent spray such as permethrin to field clothing). Apply and use following manufacturer instructions.
- Inspect clothing frequently while in tick habitat.
- Inspect head and body thoroughly when you return from the field.
- Remove any ticks by tugging with tweezers. Do not squeeze or crush the tick.

NOTE: There is a commercially available vaccine to prevent Lyme disease. Individual who are interested in this option should consult their physician.

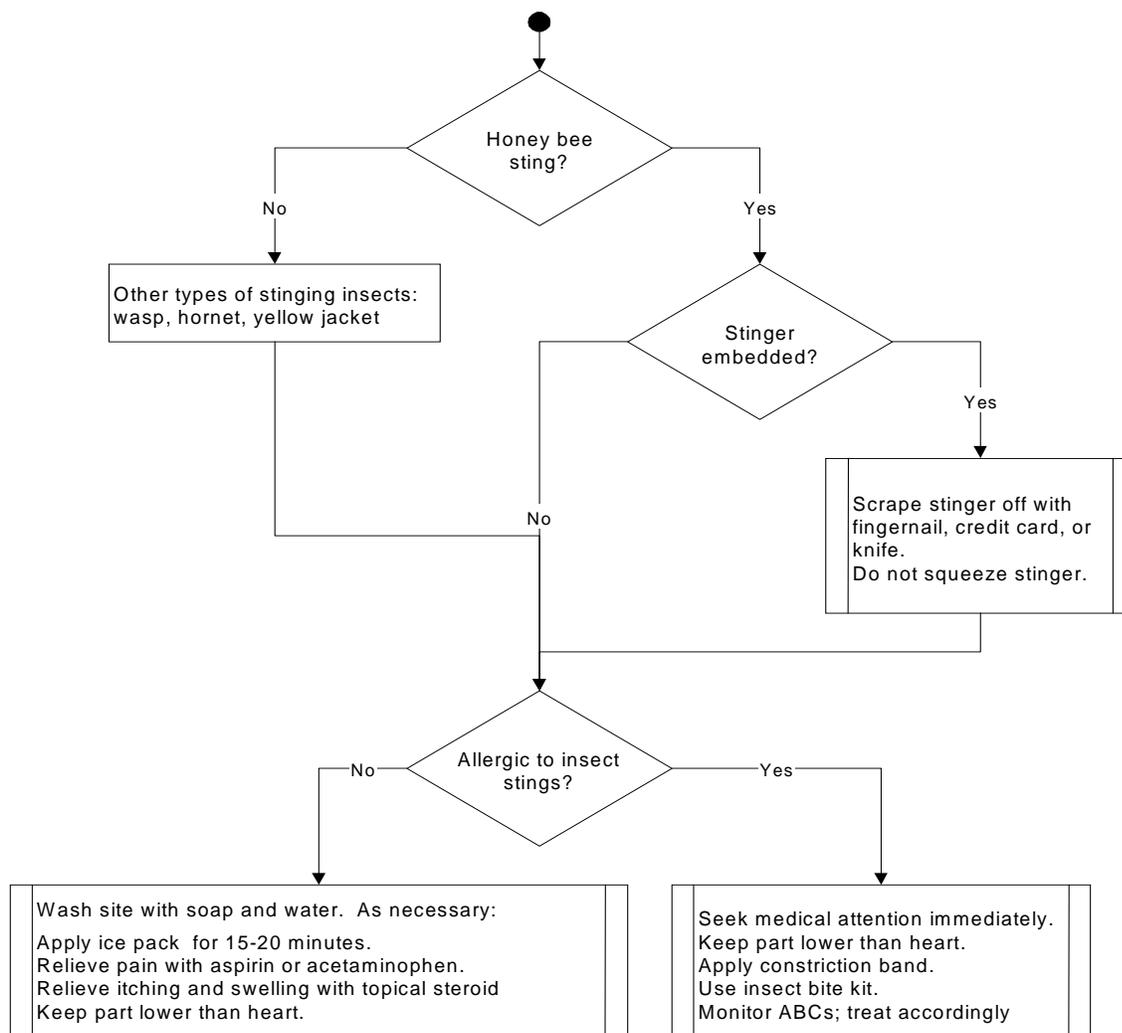
2.3.2.6 Bees, Wasps, and Hornets

Symptoms of an insect sting are normally a sharp, immediate pain in the body part stung.

Site personnel will comply with the following work practices:

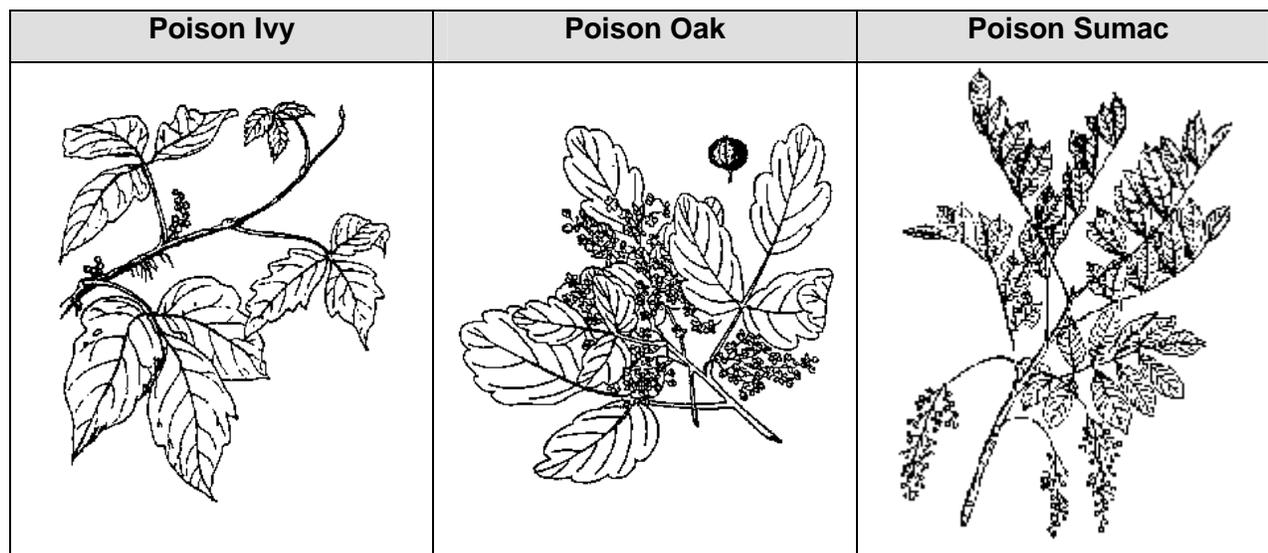
- Personnel with a known hypersensitivity to bee, wasp, or hornet stings will inform the UXO Safety Officer of this condition prior to performing site activities
- Personnel with a known hypersensitivity will keep emergency medication in their possession
- All personnel will remain vigilant for the presence of these stinging insects. Discovered nests will be flagged and their location reported to other site personnel
- If stung, immediately inform the SUXOS to receive treatment (Figure 1).

Figure 1: Decision Diagram for Stings from Flying Insects



2.3.2.7 Poison Ivy/Poison Oak/Poison Sumac

Poison Ivy, Poison Oak, and Poison Sumac (Figure 2) may cause severe skin reaction on contact. The plants are poisonous to the touch, frequently producing severe inflammation and blisters. These are woody shrubs or vines, and the leaves are arranged in an alternate pattern on the vine. Poison Ivy may be a climbing vine or a non-climbing shrub. The plant leaves have three serrated-edge pointed leaflets, the middle leaflet has a much longer stalk than the two side ones. The leaves vary in size from 8 to 55mm in length. They vary in color from reddish in the spring, green in the summer, and become various shades of yellow, orange, or red in the autumn. Poison Oak also may be a climbing vine or shrub but is a very large plant. It also has leaves grouped as three leaflets and changes color like Poison Ivy. The leaflets are similar in shape to the leaves of oak trees. Poison Sumac is a tall smooth stemmed shrub or bush whose stems have rows of six to twelve leaflets arranged in pairs with an additional single leaflet at the end of the midrib. It has small yellowish green flowers in clusters that mature into green fruits that hang in loose clusters 10 - 30 cm in length.

Figure 2: Poison Ivy/Poison Oak/Poison Sumac

Avoid contact with these plants. If contact is made, the majority of skin reactions are allergic in nature, characterized by headache, fever, itching, redness, and rash. Ordinarily, the rash begins within a few hours after exposure, but may be delayed for 24 to 48 hours. If you suspect you have contacted these plants, immediately wash the contact area with soap and water.

The reaction to these plants is caused by contact with the plant's colorless or yellowish oil called urushiol. The plant oil is released when the plant stem or leaves are cut or crushed. The plant oil is invisible and sticky and may be carried on tools, animal fur, clothing, or in the smoke of a burning plant. Suspected contaminated clothing and/or equipment should be kept separate and washed to avoid transporting the oil offsite and spreading the contamination. Once the oil has bonded to the skin it cannot be removed but the reaction may be treated with non-prescription preparations. Severe reactions will require a physician's care.

2.4 Engineering Controls and Practice Controls

2.4.1 Emergency Evacuation

In the unlikely event that an emergency evacuation is required, notification will be communicated by radio or one long steady blast for 30 seconds or more on an air horn, which indicates an evacuation is imminent. UXB personnel will immediately assemble at the entry point to the site and identify themselves for accountability purposes and proceed as directed by the SUXOS.

2.4.2 Prevention/Minimization of Public Exposure

The HYDROLYSIS PROCESS team will report unauthorized personnel entering the work area to the Senior UXO Supervisor, who in turn will request assistance from Base security in removing the unauthorized personnel from the area. HYDROLYSIS PROCESS operations will halt until unauthorized personnel have vacated the work area.

2.5 Training

In compliance with 29 CFR 1910.120(e), all HYDROLYSIS PROCESS personnel have receive safety and health indoctrination, site-specific training, and continuing training to enable them to perform their assigned tasks safely and efficiently (Table 3). Applicable training certificates and documentation for site-specific attendance will be maintained on-site.

2.6 Personal Protective Equipment (PPE)

This section addresses: 29 CFR 1910.120(g)(5), 25 CFR 1910.134, and 29 CFR 1910.132.

Personnel will be physically able, medically qualified, and trained to wear the PPE required at the project site. Personnel will be proficient in proper PPE wear, maintenance, inspection, and testing. They must be knowledgeable of the PPE limitations and reduced performance levels.

Protective equipment must be maintained in a serviceable condition at all times. Defective equipment will not be used. Before being stored or reissued to another person, equipment must be cleaned, disinfected, inspected, and repaired (if necessary).

2.6.1 Task/Operation-Specific Minimum PPE Levels

PPE will be selected to reduce contaminant levels below published occupational exposure limits, e.g., OSHA Permissible Exposure Levels (PELs) or ACGIH Threshold Limit Values (TLVs). The goal is to keep potential exposure levels as low as reasonable achievable. If operations proceed as planned, the concentration of contaminants will always be less than PEL. As a conservative measure, PPE shall be worn at any time there is a potential for exposures. The following criteria were evaluated in selecting appropriate PPE levels.

- Routes of Potential Exposure
- Inhalation, Skin Absorption and Ingestion
- Eye and Skin Contact
- Performance and Durability of PPE Materials
- Task-Specific Conditions

The minimum levels of protection for each task associated with this project are identified in Table 4. If site conditions change, the PPE levels will be re-evaluated.

Table 3: Training Requirements for On-Site Personnel

Training/Content	Duration	Frequency	Personnel Categories					
			UXO Supervisors	UXO Specialists	Technical	Operations Director	Operational Support	Visitors
Health and Safety for Hazardous Waste Operations and Emergency Response -- 29 CFR 1910.120	40-Hours	Once	✓	✓	✓	✓	✓	
Excavations, Fall Protection, 29 CFR 1926 Subparts B, P, and M	1-Hour	Mobilization	✓	✓	✓	✓	✓	✓
Health and Safety for Hazardous Waste Operations and Emergency Response -- 29 CFR 1910.120 Refresher	8-Hours	Annual	✓	✓	✓	✓	✓	
Site-Specific SSHP, Responsibilities, Hazards, PPE, Safe Work Practices & Equipment Use, Medical Surveillance, Decontamination, Emergency Response, UXO Refresher	2-Hours	Mobilization	✓	✓	✓	✓	✓	
Supervisor's Health and Safety for Hazardous Waste Operations and Emergency Response -- 29 CFR 1910.120	8-Hours	Once	✓					
Hazard Communication 29 CFR 1910.1200 - Hazards of materials used/encountered	1-Hour	Mobilization	✓	✓	✓	✓	✓	
Hearing Conservation 29 CFR 1910.95 - Physical/psychological effects of noise levels - Noise exposure limits - Selection/use/limitations of protection (As mandated by exposure)	1-Hour	Mobilization	✓	✓	✓	✓	✓	
Visitor - Operational Activities & Hazards - Boundaries of Work Area and Entry/Exit - Emergency Evacuation & Assembly Points - PPE	15-Minutes	Once per visit						✓
Morning Safety Meetings - Potential hazards and risks - Encounters with hazardous materials to date	15-Minutes	Daily, Prior to Operations	✓	✓	✓	✓	✓	
Supervisor's Safety Meeting	30-Minutes	Weekly	✓					

Table 4: Task Specific PPE Levels

Tasks	PPE Level	Coveralls/Pants	Work Boots	Work Gloves	Protective Eye Ware	Hearing Protection	Hard Hat	Chemical Resistant Coveralls	Full Face Respirator
Setup and Teardown Operations, BDU handling	D	√	√	√	√	√			
Area of Forklift Ops.	D	√	√	√	√		√		
Caustic/Acid Ops.	D	√	√	√		√		√	√

2.6.1.1 Upgrade/Downgrade Criteria

If site conditions change and warrant an upgrade, personnel will stop work, evacuate the area, and the UXOSO (in conjunction with UXB's UXO Safety Manager and in consultation with the Government) will direct the required PPE upgrade. There will be no downgrade from Level D.

2.6.1.2 On-Site Emergency and First Aid Equipment

The following emergency and first aid equipment will be immediately available in the event of an emergency:

- **Emergency and First Aid Kits** – will be located at each operational area and in the office; per the U.S. Army Corps of Engineers' Safety and Health Requirements Manual (EM 385-1-1). There will be one first aid kit for every 25 persons on-site. The SUXOS will maintain one kit at all times. Kits will be inspected on a weekly basis, and missing components immediately replaced.
- **Fire Extinguishers** – Classification 2-A:10-B:C fire extinguishers (5 pound) will be maintained in the personnel transport vehicles at each range.
- **Eye and Face Protection (ANSI Z87.1)** – When mandated by PPE requirements, personnel must wear appropriate eye and face protection; personnel requiring lenses will wear eyeglasses with approved corrective lenses, goggles over normal eyeglasses, or goggles fitted with corrective lenses, or full face shields over eyeglasses.
- **Head Protection (ANSI Z89.1)** – Personnel working in or visiting a hardhat area will be issued and required to wear protective headgear. Hard hats will not be required during ordnance operations unless head injury is possible. Hard hats will be inspected daily for signs of cracks, dents, or other defects that may reduce effectiveness.
- **Emergency Eye Washes** – will comply with ANSI Z-358.1

- **Emergency Use Respirators** -- will not be required.
- **Spill Control Materials and Equipment** – Caustic and Acid spill kits.

2.7 Medical Surveillance

All UXB personnel participate in UXB's medical surveillance program, in accordance with 29 CFR 1910.120(f). This program requires that medical examinations be performed:

- Prior to assignment
- At least once per year, within 365 days
- At permanent termination from UXB employment
- As soon as possible after notification of exposure or development of symptoms indicating an exposure
- As site conditions warrant

The examinations include a baseline health assessment consisting of:

- Medical and occupational history review
- Blood Tests for contaminants of interest
- Urine Tests for contaminants of interest
- Baseline cholinesterase levels (for CWM sites)
- Electrocardiogram
- Pulmonary function tests – including the ability to use respiratory equipment AND to operate with surface supplied SCUBA air for diving operations (as required)
- Chest x-ray
- General physical examination (including hearing and vision)

Biological exposure monitoring is not required, as this project site is not located in an HTRW environment.

It is anticipated that there will be no potential occupational exposures during this project, and respirators will not be required.

A written medical opinion from the examining physician as to the fitness to perform the required work shall be made available to the Contracting Officer, upon request.

2.8 Environmental and Personal Monitoring

2.8.1 Meteorological Monitoring

Meteorological monitoring will consist of the SUXOS turning to a local radio station and/or calling the weather station for weather reports and forecasts.

2.8.2 Corrective Actions

The SUXOS will administer corrective actions, when necessary, as directed by UXB's UXO Safety Manager.

2.9 Site Control

This section outlines the requirements needed to protect on-site personnel, the environment, and the general public from task hazards and exposures to hazardous conditions. The standard operating procedures (SOPs), engineering controls, and work practices that site personnel will implement to eliminate or reduce the risks of exposure to site hazards will be closely adhered to at all times. The SUXOS will assess and reassess the effectiveness of these procedures as each task is conducted and if new site information becomes available. If it becomes necessary, the, SUXOS and UXB's UXO Safety Manager will amend the control procedures.

2.9.1 Work Zone Delineation

An exclusion zone will be established around each work area.

2.9.2 Site Access Controls

The project site is a restricted area.

Roads will be marked and blocked (if necessary) to prevent unauthorized personnel from entering the HYDROLYSIS PROCESS operational site.

2.10 Personnel and Equipment Decontamination

Decontamination may be required for this project. In the event that personnel in PPE require decontamination they will be decontaminated using clear water from the on board water tank. Personnel not in proper PPE that are exposed to chemical hazards will be treated as an emergency. Equipment will be decontaminated using clear water also.

2.11 Emergency Response and Contingency Procedures

Personnel must be prepared to immediately respond to emergency situations, and all required equipment must be readily available, in proper working order, and ready for use. To ensure rapid, effective response to an emergency, these emergency responses and contingency procedures (developed in accordance with 29 CFR 1910.120) shall be implemented prior to and during operations.

2.11.1 Pre-Emergency Planning and Procedures

The following will ensure appropriate pre-emergency planning and procedures are in place:

- Prior to the HYDROLYSIS PROCESS Operations all HYDROLYSIS PROCESS personnel will be briefed on:
 - Location of the nearest means of off-site communications
 - Evacuation routes
 - Assembly points
 - Location of maps depicting the route to the hospital and maintained in the designated emergency vehicle and all personnel vehicles
 - Emergency telephone numbers First Aid and PPE equipment shall be readily available

2.11.2 Communications

Communications consist of a combination of hand-held radios and cellular telephones.

2.11.3 Emergency Contacts

Table 5 is a list of Emergency Telephone Numbers that will be posted on the site and available for all personnel in case of an emergency.

Table 5: Emergency Telephone Numbers

AGENCY	NAME	TELEPHONE NUMBER
Fire Department Police and BMGR Security Ambulance/Rescue Service Hospital	West Valley Hospital 13677 W. McDowell Rd. Goodyear, AZ 85338	911 911 911 623-882-1500
National Capitol Poison Control Center		(800) 222-1222
UXB International, Inc. Main Number Sector Director Director, AEDA Services Director, Demil Services Operations Director	Ted Rekart Harley Heaton John Stine John Kierepka	(703) 724-9600 (703) 724-9647 (703) 724-9646 (256) 772-8263 (703) 395-0129
Army Operations Center To report discovery of CWM		(703) 697-0218 (703) 697-5690

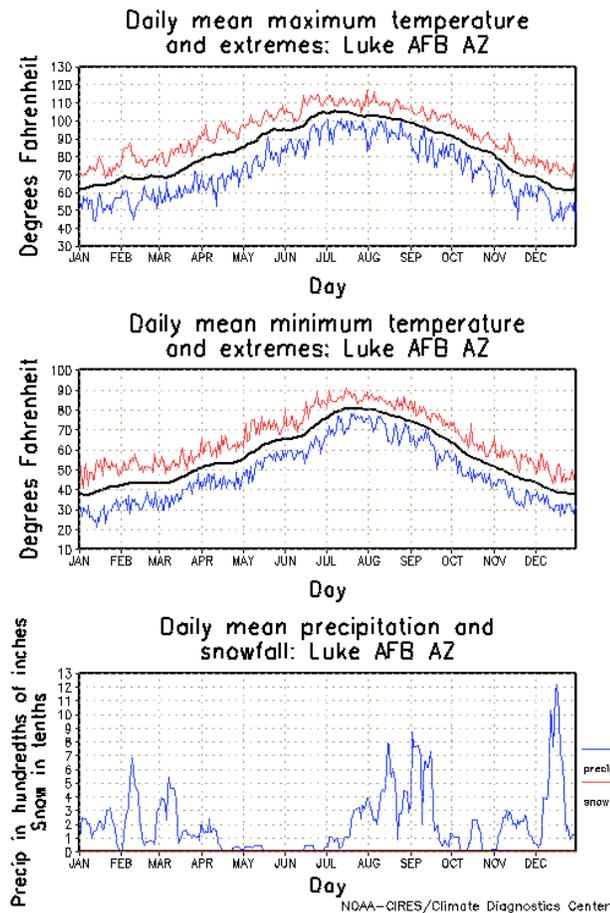
2.11.3.1 Emergency Recognition and Prevention

At the morning safety meetings, personnel will be briefed on new developments, tasks, hazards, hazard recognition, and appropriate prevention procedures. These meetings will include:

- Tasks to be performed
- Specific hazards that may be encountered, including their effects, how to recognize symptoms or monitor them, concentration limits, or other danger signals
- Additional hazards as a direct result of site activities, and prevention and control techniques
- Emergency procedures

2.11.3.2 Prevailing Weather Conditions

Figure 3 provides annual climate information for BMGR.

Figure 3: BMGR Climate Information

2.11.3.3 Emergency Actions

2.11.3.4 Site Evacuation

The SUXOS will determine the need to evacuate the work area based on the following criteria:

- Change in site conditions that would require an upgrade of PPE
- Fire and/or explosion

2.11.3.5 Emergency Alerting Procedures/Employee Alarm System

Emergency notification will be conducted via hand-held radios or air horns.

2.11.3.6 Emergency PPE and Equipment

Each UXB vehicle, and operational site will be supplied with at least one 2A:10B:C fire extinguisher (5 pounds). The site will also be equipped with emergency eyewash, safety shower, and spill control/neutralization kits for acids and caustics

2.11.4 Medical Treatment for Injured Personnel

The on-site UXB personnel will provide minor medical treatment. If MEDIVAC is required, it will be coordinated through Range Control and the West Valley Hospital ER.

2.11.5 Material Safety Data Sheets

Material Safety and Data Sheets for each hazardous substance anticipated to be encountered on-site will be kept on-site. These shall be accessible to all on-site personnel at all times.

2.12 Spill Containment

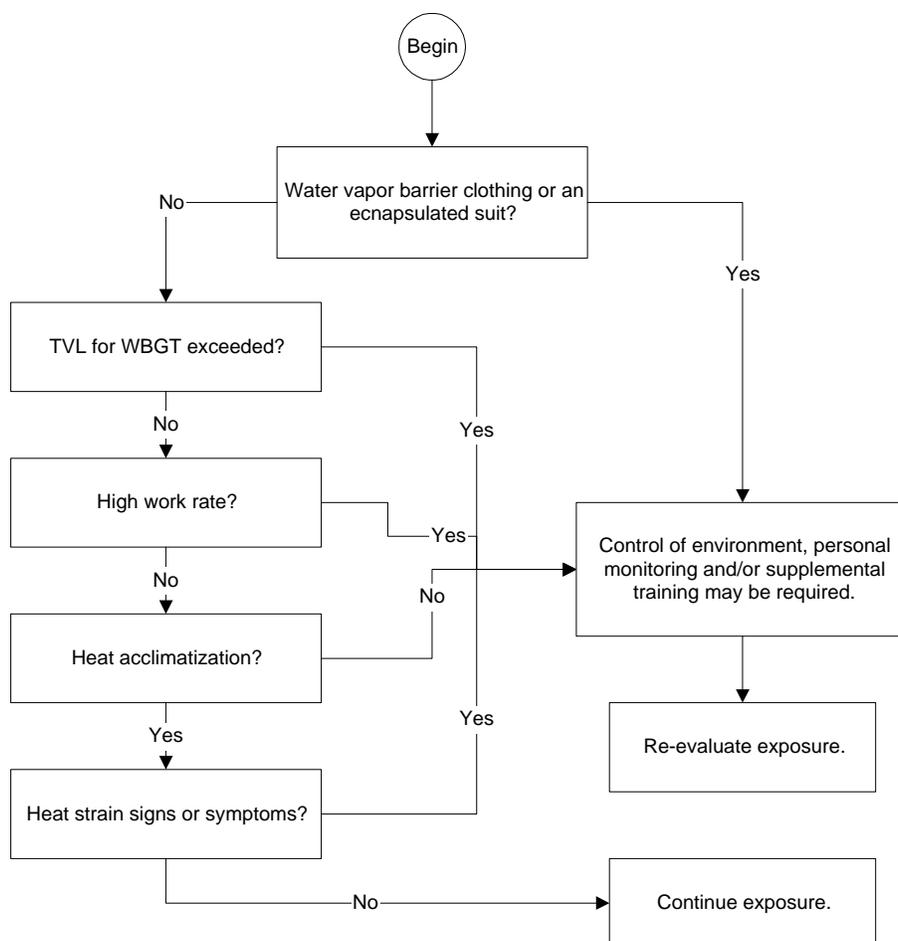
UXB will primarily fuel and repair all vehicles off-site. Bulk fuel for the generator and heating unit will be stored on-board the hydrolysis unit and may be re-supplied by truck.

2.13 Heat/Cold Stress Monitoring

2.13.1 Heat Stress Monitoring

Heat stress and heat strain must be assessed in evaluating worker safety and health, using the decision tree diagram shown at Figure 4. Heat stress is the net load on the body with contributions from both metabolic heat production and external environmental factors including temperature, humidity, radiant heat transfer, and air movement, as they are affected by clothing.

Figure 4: Evaluating Heat Stress and Strain



Threshold Limit Values (TLVs) specified in Table 6 refer to heat stress conditions under which it is believed that nearly all heat acclimatized, adequately hydrated, un-medicated, healthy

workers wearing light-weight summer clothing may be repeatedly exposed without adverse health effects. The work areas are assumed to have at least some air movement. When the Wet Bulb Globe thermometer (WBGT) values in Table 6 must be exceeded, the guidelines in Table 7 must then be followed.

Table 6: TLVs for Heat Exposure

Hourly Activity	Work Rates		
	Light	Moderate	Heavy
100% Work	30.0°C/86.0°F	26.5°C/80.0°F	25.0°C/77.0°F
75% Work - 25% Rest	30.5°C/87.0°F	28.0°C/82.5°F	26.0°C/79.0°F
50% Work - 50% Rest	31.5°C/89.0°F	29.5°C/85.0°F	28.0°C/82.5°F
25% Work - 75% Rest	32.0°C/89.5°F	31.0°C/88.0°F	30.0°C/86.0°F
Temperature values in WBGT Refer to Table 3-9 for work rates. Work and rest environments are assumed to be the same. If different, hourly time-weighted average WBGTs must be calculated.			

2.13.1.1 Physical Barriers to Heat Loss

Evaporation of sweat from a person’s skin can be an important means of dissipating heat from the body. Also, the free movement of cool, dry air over the skin’s surface optimizes heat loss. Water vapor impermeable or thermally insulating clothing, encapsulating suits, and similar convective and evaporative barriers can severely restrict heat loss and produce life-threatening heat strain, even when the ambient air temperature, radiant heat, and humidity are low. Whenever workers wear such restrictive clothing, it is essential that extra caution be exercised. Since such impervious clothing will not be worn during this ordnance and explosive survey, this step of the heat stress and strain decision tree can be skipped.

Table 7: Guidelines for Heat Exposure Limits

Always monitor signs and symptoms of heat-stressed workers (refer to Table 10). When WBGT-TLV criteria (Table 6) are exceeded or water vapor impermeable clothing is worn, discontinue any environmentally induced or activity-induced heat stress for a person when any one of the following indicators is exhibited.
Sustained heart rate is greater than 160 beats per minute for those under 35 years of age Sustained heart rate is greater than 140 beats per minute for those 35 years of age or older Complaints of sudden and severe fatigue, nausea, dizziness, lightheadedness, or fainting Periods of inexplicable irritability, malaise, or flu-like symptoms Sweating stops and the skin becomes hot and dry
<i>Good physical fitness and adequate hydration are the first lines of defense against heat strain</i>

2.13.2 Wet-Bulb Globe Temperature (WBGT)

Since measurement of deep body temperature is impractical for monitoring the workers' heat load; the measurement of environmental factors is required to correlate with deep body temperature and other physiological responses to heat. The Wet Bulb Globe Temperature (WBGT) Index is the simplest and most suitable technique to measure the environmental factors.

Measurement of WBGT offers a useful, first-order index of heat stress by assessing the net effects of dry air temperature, radiant heat transfer, and ambient humidity. It may not, however, sufficiently reflect the effects of air movement on convective heat transfer or evaporative heat loss (typically major avenues of heat loss) and it does not by itself account for heat produced by physical work, often a major source of heat strain.

WBGT values are calculated: $WBGT = 0.7 NWB + 0.2 GT + 0.1 DB$

WBGT = wet bulb globe temperature index

NWB = natural wet-bulb temperature

GT = globe temperature

DB = dry-bulb temperature

The WBGT requires the use of a black globe thermometer, a natural (static) wet-bulb thermometer, and a dry-bulb thermometer.

- The wick of the natural wet-bulb thermometer should be kept wet with distilled water for at least ½ hour before the temperature reading is made. The wick should extend over the bulb of the thermometer, covering the stem about one additional bulb length.
- The dry bulb thermometer must be shielded from the sun and the other radiant surfaces of the environmental without restricting the airflow around the bulb.
- The globe thermometer should be exposed at least 25 minutes before it is read.
- A stand should be used to suspend the three thermometers so that they do not restrict free airflow around the bulbs, and the wet-bulb and globe thermometers are not shaded.

Acceptable WBGT exposures depend on the rate of metabolic heat production and the balance of work and rest periods.

2.13.3 Work Rate

As shown in Table 8, physical activity can impose high rates of heat production. It is commonly the major source of heat strain, even when there is little or no environmental heat stress. The metabolic heat resulting from such activity can be an especially serious threat for someone wearing water-vapor-barrier or heat-insulating clothing, or an encapsulated suit. Even in cool and dry ambient conditions, high work rates or wearing such clothing requires that the guidelines in Table 8 be followed.

Table 8: Examples of Metabolic Heat Production

Work Rates	Activity	Watts
Resting	Sitting quietly	130
	Sitting with moderate arm movements	150
Light	Sitting with moderate arm and leg movements	175
	Using table saw	215
	Standing with light work at machine or bench and some walking about	220
	Standing with light work at machine or bench while using mostly arms	250
	Replacing tires	255
	Standing with moderate work at machine or bench and some walking about	260
Moderate	Walking about with moderate lifting or pushing	360
	Carpenter sawing by hand	475
	Shoveling dry sand	480
	Heavy assembly work on a non-continuous basis	500
Heavy	Intermittent heavy lifting with pushing or pulling (pick and shovel work)	520
	Shoveling wet sand	535

2.13.4 Heat Acclimatization

Periodic physical activity performed under heat stress conditions normally induces physiological changes, including those in Table 9, that allow a worker to withstand heat stresses with reduced heat strain. Heat acclimatization is acquired gradually over weeks of continued physical activity under heat stress conditions. Its loss begins when the activity under heat stress conditions is discontinued.

Table 9: Heat Acclimatization Benefits

Someone with heat acclimatization exposed to environmentally or activity-induced heat-stress has:
Improved productivity and safety
More stable and better regulated blood pressure which promotes lower pulse rates, less fatigue, improved alertness, and focus of attention
More finely tuned sweating reflexes with increased sweat production rate at lower electrolyte concentrations, when needed
Fewer and less severe signs and symptoms of heat strain for the same level of heat stress

2.13.5 Heat Strain

Heat Strain manifests itself in four disorders – Heat Stroke, Heat Exhaustion, Heat Cramps, and Sunburn. The symptoms, treatments, warnings, and prevention of each of these disorders and a work/rest schedule are noted in Table 10.

Table 10: Heat Strain Monitoring

HEAT STRAIN (In descending order of severity)	
<p>HEAT STROKE (Sun Stroke) results from the disruption of the body's heat regulating system, producing excessively high body temperature. Can be caused by continuous exposure to high temperatures for as little as three hours. RAPID COOLING IS URGENT TO PREVENT DEATH.</p>	
<p>SYMPTOMS:</p>	<ul style="list-style-type: none"> • Extremely high body temperature, often 106^o Fahrenheit (F) or above • Skin is red, hot, and dry; sweating is absent • Pulse is rapid and strong • Convulsion or collapse is possible • Delirium, disorientation, or unconsciousness is possible
<p>EMERGENCY TREATMENT:</p>	
<ul style="list-style-type: none"> • Immediately call for emergency help • Place person in an air-conditioned room, vehicle, or in a shaded area (at a minimum) • COOL THE PERSON RAPIDLY -- remove the person's clothing, bathe the body in cool water, and massage continuously (submerge in cool water, or at least administer a sponge bath). Fans or air currents (such as hand fanning) also assist in the cooling process. • When the back of the hand held against the victim's cheek indicates normal skin temperature, or when internal body temperature reaches 102°F or less, discontinue the cooling process and wrap the person in a blanket to prevent shock. • Victim may sip liquids 	
<p>WARNINGS:</p>	<ul style="list-style-type: none"> • Do not administer alcoholic beverages or stimulants (coffee, tea) • If the body temperature begins to rise again, repeat the cooling process
<p>PREVENTION:</p>	<ul style="list-style-type: none"> • Protect the body from radiant heat • Break the workday into short work/rest periods • Drink enough fluids to replace those lost by sweating
<p>HEAT EXHAUSTION (Heat Prostration or Heat Collapse) results when the circulatory system fails to sufficiently compensate for increased blood flow demands imposed by the need to cool the body and dehydration caused by profuse sweating. Failure to provide quick treatment can lead to heat stroke.</p>	
<p>SYMPTOMS:</p>	<ul style="list-style-type: none"> • Body temperature is normal or slightly elevated • Skin is clammy and pale, and there is a moist, profuse sweating • Pulse may be weak with low blood pressure • Victim is tired and weak • Victim may complain of dizziness or giddiness, and fainting is possible • Possible muscle cramps • Possible nausea or vomiting • Mental state is generally rational
<p>EMERGENCY TREATMENT:</p>	
<ul style="list-style-type: none"> • Place person in an air conditioned room, vehicle, or in a shaded area (at a minimum) • Have person lie down; elevate feet 8 - 12 inches 	

HEAT STRAIN (In descending order of severity)	
	<ul style="list-style-type: none"> • Loosen tight fitting clothing • If conscious, have victim sip a glass of electrolyte replacement solution (Gatorade, ERG, or Squincher). Repeat ever 15 minutes to 1 hour; stop fluids if vomiting occurs. • If symptoms persist or return, immediately summon medical assistance
	<p>PREVENTION:</p> <ul style="list-style-type: none"> • Schedule frequent rest periods • Drink electrolyte liquids every 15 minutes to 1 hour to replace body fluids
<p>HEAT CRAMPS are muscle pains and spasms caused by loss of electrolytes (due to sweating over an extended period). Not life threatening, but heat cramps may hinder work or cause a potential hazardous situation.</p>	
	<p>SYMPTOMS:</p> <ul style="list-style-type: none"> • Painful muscle cramps and spasms • Heavy sweating, vomiting, and/or convulsions • Normal, or near normal, pulse and blood pressure • Irrational behavior
	<p>EMERGENCY/MEDICAL TREATMENT:</p> <ul style="list-style-type: none"> • Quiet rest in a cool, shaded area • Gently massage affected area • If person is not vomiting, give electrolyte fluids every 15 minutes to 1 hour • If symptoms persist, victim should be transported to the nearest medical facility.
	<p>PREVENTION:</p> <ul style="list-style-type: none"> • Salt food more heavily than normal • Drink electrolyte solutions • Eat salty foods during heavy sweat-producing activities
	<p>WARNING: If on a low sodium diet or taking diuretics, consult your physician regarding the replacement of salts prior to field activities. Be sure to explain any problems to the Senior UXO Supervisor.</p>
<p>SUNBURN is the most common of the heat disorders. It is usually a first-degree burn of the epidermis (first layer of skin), and the affects may not be noticed or felt until several hours after exposure.</p>	
	<p>SYMPTOMS:</p> <ul style="list-style-type: none"> • Skin redness • Swelling • Pain • Blisters, nausea, vomiting, chills (in severe cases)
	<p>EMERGENCY/MEDICAL TREATMENT:</p> <ul style="list-style-type: none"> • Put cold water on the burned area as quickly as possible • Submerge severe burns in cold water or soak with wet cloths • Elevate burned limbs • Do not break a blister (this increases the chance of infection)
	<p>PREVENTION:</p> <ul style="list-style-type: none"> • Cover exposed parts of the body • Gradually expose the skin to the sun for 20 minute intervals per day; extending the time as the skin builds its own natural protection in the form of a tan • Use sun lotion, sun block, and sun shields (reapply every hour, even on cloudy days)
HEAT STRESS MONITORING AND WORK/REST CYCLE	
<p>The following will be implemented during field activities when the temperature exceeds 70°F. The Senior UXO Supervisor will determine the appropriate work/rest schedule and ensure all personnel take the appropriate breaks; additional breaks will be approved, as necessary.</p> <p><u>Measure Heart Rate:</u> To be conducted for 30 seconds as early as possible during the rest cycle. The rate should not exceed 110 beats per minute for most people. If the heart rate is higher during the next rest period, the following cycle should be shortened by 33 percent. The reduction of the work cycle will continue until the pulse rate reaches 110 beats per minutes.</p> <p><u>Measure Body Temperature</u> orally with a clinical thermometer as early as possible in the resting period. If oral</p>	

HEAT STRAIN (In descending order of severity)	
	<p>temperature (OT) at the beginning of the rest period exceeds 99.40°F, the worker will be prohibited from continuing work until the OT is maintained below 99.4°F (37.4°C). The next work cycle will be reduced by 33% if the oral temperature exceeds 99.68 °F (37.6°C).</p> <p><u>Establish Work/Rest Schedule:</u> The SUXOS will monitor personnel every two hours at temperatures above 70°F, and every hour at temperatures above 90°F. Cool drinks will be available for consumption during rest periods.</p>

2.13.6 Optimizing Safety and Health

People who are least endangered while working in heat stress conditions are young and appropriately clothed, in good general health and physical condition, not obese, and adequately hydrated with electrolyte concentrations in normal ranges. To help assure that safe and healthy conditions are maintained, the guidelines in Table 11 should be followed.

Table 11: Guidelines for Optimizing Safety and Health

For people working in hot conditions:
Encourage drinking small volumes (approximately 1 cup) of cool or tepid water about every 20 minutes
Provide work settings with good ventilation and shielding from radiant heat sources
Assure co-worker observation to detect signs and symptoms of heat strain
Pay extra attention to those who take medications that compromise normal cardiovascular, blood pressure, body temperature regulation, renal, or sweat gland functions
<i>Never ignore anyone’s signs or symptoms of heat strain</i>

2.13.7 Cold Stress Monitoring

Cold Stress disorders include Hypothermia, Frostbite, and Chilblain. The symptoms, treatment, and prevention of these disorders, plus work/rest cycles are detailed in Table 12.

Table 12: Cold Stress Monitoring

COLD STRESS (In descending order of severity)											
<p>HYPOTHERMIA is characterized by a significant loss of body heat. Moderate cases may exhibit the first 7 symptoms below. Severe cases are indicated with extremely cold skin; loss of consciousness; faint pulse; and shallow, infrequent, or apparently absent respiration – death may result.</p>											
SYMPTOMS:	<table style="width: 100%; border: none;"> <tr> <td style="width: 50%;">1. Severe shivering</td> <td style="width: 50%;">6. Repeated falling</td> </tr> <tr> <td>2. Abnormal behavior</td> <td>7. Inability to walk</td> </tr> <tr> <td>3. Slowing of movements</td> <td>8. Collapse</td> </tr> <tr> <td>4. Stumbling</td> <td>9. Stupor</td> </tr> <tr> <td>5. Weakness</td> <td>10. Unconsciousness</td> </tr> </table>	1. Severe shivering	6. Repeated falling	2. Abnormal behavior	7. Inability to walk	3. Slowing of movements	8. Collapse	4. Stumbling	9. Stupor	5. Weakness	10. Unconsciousness
1. Severe shivering	6. Repeated falling										
2. Abnormal behavior	7. Inability to walk										
3. Slowing of movements	8. Collapse										
4. Stumbling	9. Stupor										
5. Weakness	10. Unconsciousness										
EMERGENCY/MEDICAL TREATMENT:	<ul style="list-style-type: none"> • A severely shivering worker shall immediately terminate work and exposure to cold • Seek qualified medical help immediately • Remove the victim from the hypothermia-producing environment 										

COLD STRESS (In descending order of severity)	
	<ul style="list-style-type: none"> • Keep handling to a minimum; DO NOT rub or massage the victim • Cover the victim lightly with blankets; plastic may be used for further insulation. DO NOT cover face. • If victim is conscious, administer hot drinks, encourage activity (walking while wrapped in a blanket), and DO NOT administer any form of sedative, tranquilizer, or analgesic (pain reliever), as they may facilitate further heat loss.
	PREVENTION: Wear insulated garments in a layered fashion.
FROSTBITE includes localized injury resulting from exposure to cold temperatures, and includes several degrees of severity.	
	SYMPTOMS: <ul style="list-style-type: none"> • <u>frost nip or incipient frostbite</u> is a sudden blanching or whitening of the skin • <u>superficial frostbite</u> has a waxy or white appearance and is firm to the touch, but the tissue beneath is resilient • <u>deep frostbite</u> is an extremely serious condition in which tissues are cold, pale, and solid.
	EMERGENCY/MEDICAL TREATMENT: DO NOT rub effected part with snow. Slow re-warming in water at 103° - 105°F. Give warm nutritious drinks (no alcohol). Victim should not smoke.
	PREVENTION: Wear insulated garments in a layered fashion.
CHILBLAIN is an inflammation caused by exposure to cold moisture.	
	SYMPTOMS: <ul style="list-style-type: none"> <li style="width: 50%;">• Localized itching <li style="width: 50%;">• Severe spasms <li style="width: 50%;">• Swelling <li style="width: 50%;">• Pain <li style="width: 50%;">• Inflammation of fingers, toes, or ears
	EMERGENCY/MEDICAL TREATMENT: Warm gradually by placing parts in lukewarm water or with warm hands. Do not rub. Place victim in warm but not hot room. Give warm nutritious drinks (no alcohol).
	PREVENTION: Wear insulated garments in a layered fashion.
COLD STRESS PREVENTION AND WORK/REST CYCLE	
	<ul style="list-style-type: none"> • A work/rest regime and a heated shelter shall be provided, as needed. A change of clothing for each worker shall be on hand. Warm, non-alcoholic drinks (avoid caffeine) and soup will be available, if required. • Use the heated shelter at regular intervals when temperatures below 20°F or equivalent wind chill are present. Frequency of breaks is dependent on environmental conditions and each worker's needs. • When entering shelter, remove outer layer of clothing and loosen remaining clothing to permit sweat evaporation. Do not return to work with wet work clothing. • Heavy shivering, frost nip, excessive fatigue, drowsiness, irritability, or euphoria indicates an immediate return to the heated shelter. • The equivalent chill temperature should be used when estimating the combined cooling effect of wind and low air temperatures on exposed skin or when determining clothing insulation requirements to maintain the deep body core temperature. • If conditions are below 10°F or an equivalent wind chill, the following shall apply: Work under constant supervision and the buddy system .

COLD STRESS (In descending order of severity)	
	Reduce work rate to avoid heavy sweating Instruct workers in warming procedures, first aid, clothing, eating/drinking, and recognition of signs and symptoms of impending frostbite, hypothermia, or excessive cooling without shivering.

The ACGIH cold stress standard shall be the minimum protocols for cold stress. The cooling power of wind on exposed flesh is depicted in Table 14. The TLVs Work/Warm-up schedule for four-hour shifts is given in Table 13.

Table 13: Work/Warm-up Schedule for Four Hour Shift

Air Temp. Sunny Sky		No Noticeable Wind		5 mph Wind		10 mph Wind		15 mph Wind		20 mph Wind	
		Max. Work Period	No. of Breaks								
C (approx.)	F (approx.)										
-26 to -28	-15 to -19	Norm.	1	Norm	1	75 min	2	55 min	3	40 min	4
-29 to -31	-20 to -24	Norm.	1	75 min	2	55 min	3	40 min	4	30 min	5
-32 to -34	-25 to -29	75 min	2	55 min	3	40 min	4	30 min	5	Non-emergency work should cease	
-35 to -37	-30 to -34	55 min	3	40 min	4	30 min	5	Non-emergency work should cease			
-38 to -39	-35 to -39	40 min	4	30 min	5	Non-emergency work should cease					
-40 to -42	-40 to -44	30 min	5	Non-emergency work should cease		Non-emergency work should cease					
-43 & below	-45 & below	Non-emergency work should cease									

Notes for Table: Schedule applies to any 4-hour work period with moderate to heavy work activity, with warm-up periods of ten (10) minutes in a warm location and with an extended break (e.g., lunch) at the end of the 4-hour work period in a warm location. For Light-to-Moderate Work (limited physical movement): apply the schedule on step lower. For example, at -35C (-30F) with no noticeable wind (Step 4), a worker at a job with little physical movement should have a maximum work period of 40 minutes with 4 breaks in a 4-hour period (Step 5).

The following is suggested as a guide for estimating wind velocity if accurate information is not available: 5 mph: light flag moves -- 10 mph: light flag fully extended -- 15 mph: raises newspaper sheet -- 20 mph: blowing and drifting snow

If only the wind chill cooling rate is available, a rough rule of thumb for applying it rather than the temperature and wind velocity factors given above would be: 1) special warm-up breaks should be initiated at a wind chill cooling rate of about 1750 W/m²; 2) all non-emergency work should have ceased at or before a wind chill of 2250 W/m². In general, the warm up schedule provided above slightly under-compensates for the wind at the warmer temperatures, assuming acclimatization and clothing appropriate for winter work. On the other hand, the chart slightly over-compensates for the actual temperatures in the colder ranges because windy conditions rarely prevail at extremely low temperatures. TLVs apply only for workers in dry clothing.

Table 14: Cooling Power of Wind on Exposed Flesh Expressed as Equivalent Temperature

Estimated Windspeed (in mph)	Actual Temperature Reading (° F)											
	50	40	30	20	10	0	-10	-20	-30	-40	-50	-60
Equivalent Chill Temperature (° F)												
calm	50	40	30	20	10	0	-10	-20	-30	-40	-50	-60
5	48	37	27	16	6	-5	-15	-26	-36	-47	-57	-68
10	40	28	16	4	-9	-24	-33	-46	-58	-70	-83	-95
15	36	22	9	-5	-18	-32	-45	-58	-72	-85	-99	-112
20	32	18	4	-10	-25	-39	-53	-67	-82	-96	-110	-121
25	30	16	0	-15	-29	-44	-59	-74	-88	-104	-118	-133
30	28	13	-2	-18	-33	-48	-63	-79	-94	-109	-125	-140
35	27	11	-4	-20	-35	-51	-67	-82	-98	-113	-129	-145
40	26	10	-6	-21	-37	-53	-69	-85	-100	-116	-132	-148
Windspeeds greater than 40 mph have little additional effect.	Little Danger In < 1 hr with dry skin. Maximum danger of false sense of security.			Increasing Danger Danger from freezing of exposed flesh within one minute.				Great Danger Flesh may freeze within 30 seconds.				
	Trench foot and immersion foot may occur at any point on this chart.											

2.14 Standing Operating Procedures, Engineering Controls, and Work Practices

Standard Operating Procedures (SOPs) are designed to protect personnel, prevent accidents, minimize hazards, and take action to correct hazards. As applicable to their on-site responsibilities, personnel shall review, be familiar with, and adhere to approved standard operating procedures. At a minimum, standard operating procedures (SOP), engineering controls, and work practices shall be adhered to.

2.14.1 General Safety Precautions

- Do not apply cosmetics, eat, drink, chew gum or tobacco, or smoke in work areas
- Do not conduct operations without approved operating procedures and proper supervision
- Do not become careless because of familiarity with range residue

- Do not conduct HYDROLYSIS PROCESS operations during electrical, sand, dust, or snow storms
- Avoid contact with suspect chemical hazards
- Be aware of monitoring equipment, wind direction, nearest water source, evacuation routes, emergency communication, and notification procedures
- Keep a first aid kit and fire extinguisher readily available while in the work area
- In accordance with 385-1-1, working within the swinging arc of a backhoe is considered a hardhat area, and appropriate headgear will be worn.
- All personnel should be aware of the potential for slips, trips, and falls; always be aware of the placement of your feet. When working off the ground on large pieces of equipment, maintain at least three points of contact with the equipment.
- Alert team personnel of a suspected or potentially dangerous situation. If an alarm sounds, a strong nauseating odor is detected, or you see fire or smoke, all personnel are to evacuate the site and notify the Senior UXO Supervisor.
- UXB-generated waste will be controlled and held for disposal.
- Operational activities will only be conducted during daylight hours.

2.14.2 Buddy System

The buddy system requires work crews consisting of teams of at least two, and is required during all operations. Team members will maintain visual contact with each other and be alert for signs of a medical emergency, such as:

- Changes in complexion or skin color
- Changes in coordination or demeanor
- Excessive salivation and pupillary response
- Changes in speech pattern
- Mention of headaches, dizziness, blurred vision
- Nausea, cramps
- Irritation of eyes, skin, or respiratory tract

2.14.3 Smoking Restrictions

Smoking is prohibited in the work areas. Upon mobilization, the Senior UXO Supervisor will designate a smoking area.

2.14.4 Illumination

All operations will be performed during daylight hours; illumination will not be necessary.

2.14.5 Sanitation

2.14.5.1 Potable Water

- UXB will provide workers and visitors with an adequate supply of potable water approved by federal, state, or local authorities.

- Disposable cups will be provided for all sources of drinking water; no common drinking cups will be used.
- A waste receptacle for soiled cups will be supplied.
- Potable drinking water containers will be labeled as such and not used for any other purpose. Potable drinking water containers will be cleaned and sanitized daily, containing only ice and water, and will be fitted with a tight fitting lid and tap.

2.14.5.2 Non-Potable Water

- Non-potable water may be used for washing equipment and general housekeeping/cleaning, provided the water does not contain substances that may pose a threat to the employees' health.
- There will not be any cross-connection, open or potential, between systems furnishing potable and non-potable water.
- All outlets for non-potable water will be conspicuously posted “**CAUTION - Water unfit for drinking, washing, or cooking.**”

2.14.6 Daily Safety Inspections

Will be accomplished in accordance with requirements established in Table 6-16

2.14.7 Toilet Facilities

- Portable toilet facilities will be located near the work areas.

2.14.8 Temporary Sleeping Quarters

- UXB will provide per diem eligible employees (per Joint Travel Regulations) with temporary worker sleeping quarters at a local hotel, motel, or other suitable habitable quarters.

2.14.9 Washing Facilities

- Water in portable containers and a means of drying hands or sanitary wipes will be provided at the work site.

2.14.10 Housekeeping

- The HYDROLYSIS PROCESS work site must be left clean and orderly at the end of the day.

2.14.11 Personal Hygiene

- An adequate supply of water, soap, or sanitary wipes will be provided to allow personnel to wash hands and face prior to leaving the project site.
- Personnel will be reminded to wash prior to eating or drinking in the work area.

2.14.12 Safety Meetings

- Safety meetings will be held at the start of each day's activities and any time changed site conditions warrant.
- All visitors to the site must receive a safety briefing covering the site hazards and procedures to follow in case of emergency.

2.14.13 Equipment Safety

- Equipment safety will be in accordance with the most current revision of the USACE Safety and Health Requirements Manual (EM 385-1-1).

2.14.14 Motor Vehicles and Trailers

- Equipment safety will be in accordance with the most current revision of the USACE Safety and Health Requirements Manual (EM 385-1-1).

2.14.15 Hand Tools

Section 13 (Hand and Power Tools) of the USACE Safety and Health Requirements Manual (EM 385-1-1) shall be adhered to in selecting and using hand tools:

- Hand tools will be used, inspected, and maintained in accordance with the manufacturer's specifications
- Tools will be inspected, tested, in good repair, and equipped with all safety devices to assure they are in safe operating condition prior to use
- Only non-sparking tools will be used in locations where sources of ignition may cause a fire or explosion
- Protective clothing recommended for use while using the equipment will be made available and used by the operator
- Throwing tools from one person to another is not permitted

2.14.16 Bloodborne Pathogens

Bloodborne Pathogens are pathogenic microorganisms that are present in human blood and can cause disease in humans. These pathogens include, but are not limited to, Hepatitis B Virus (HBV), Hepatitis C Virus (HCV), and Human Immunodeficiency Virus (HIV). Personnel administering care must have successfully completed bloodborne pathogens training in accordance with Table 6-3 and must implement exposure control measures.

Personal Protective Equipment is the first line of defense against bloodborne pathogens. The following protective equipment will be available on-site for personnel administering first aid.

- Surgical Gloves - must be worn when hand contact with blood or other body fluids is possible or the care provider has non-intact skin areas on their hands.
- Masks/Eye Protection/Face Shields - will be worn when splashes, sprays, or droplets of blood or body fluids are likely to occur and contaminate the eyes, nose, or mouth of the care provider.
- Coveralls/Jacket - will be donned if the possibility exists for contamination of the body of the caregiver.

To insure that equipment is used effectively, employees will adhere to the following practices when using personal protective equipment:

- Any garments penetrated by blood or other suspect infectious materials are to be removed immediately, or as soon as feasible.
- All personal protective clothing/equipment shall be removed prior to leaving the site and placed in a suitable container for decontamination and/or disposal.
- Disposable gloves are to be replaced as soon as practical after contamination or if they are torn, punctured, or otherwise lose their ability to function as an "exposure barrier."
- Potential exposure to the body of the caregiver will require donning a coat or coveralls to provide protection.

2.15 Logs, Reports, and Record Keeping

Table 15 lists the safety-related logs, reports, and records that will be maintained during on-site operations, the criteria, and the personnel responsible for maintaining the information.

2.15.1 Accident Reporting

All on-site and off-site work related accidents, regardless of severity, will be reported to UXB headquarters, investigated, and analyzed. The UXB Director of Environmental & Explosive Operations and Senior UXB staff members (selected by UXB's President) will investigate the accident, analyze the cause, and identify the corrective action to be implemented to prevent similar occurrences.

Employees are responsible for immediately reporting all injuries, occupational illnesses, abnormal conditions, or disorders caused by exposure to environmental factors associated with the work site. The Senior UXO Supervisor will document the accident; and provide medical treatment as appropriate. Within 24-hours, the Senior UXO Supervisor will notify UXB Headquarters.

UXB's Employee Injury/Property Damage Report Form (UXB Form 10-04-2) will be completed and forwarded to UXB Headquarters within two working days of the injury.

All accidents will be reported to site supervision, who will evaluate the situation and forward necessary reports to the appropriate agencies. The following reporting criteria will be used:

- Death (regardless of the length of time between the injury and death)
- One or more lost workdays
- Restriction of work or motion
- Loss of consciousness
- Transfer to another job site
- Medical treatment required (other than first aid)

The UXB SUXOS will maintain records on all safety and health issues and assure reportable accident and incident reports are submitted in a timely manner – Spot Report (UXB Form 11-00.1-3). Injury and illness records will be maintained at UXB's Human Resources Department in Ashburn, Virginia.

Table 15: Safety-Related Record-Keeping Requirements

Reporting Requirement	Criteria	Responsible Personnel
Site-Specific Training	Specialized Training Form (UXB Form 10-00.1-1) Work Plan Acknowledgment Form (UXB Form 03-00)	SUXOS
Daily Safety Inspections	Written weekly report to UXB Operations Director to identify: Task/area inspected Safety topics at Morning Safety Meetings Findings Corrective actions Safety Inspection Log (UXB Form 10-00.1-2)	SUXOS
Employee/Visitor	Site Visitor's Log (UXB Form 02-06-3)	SUXOS
Medical Surveillance	Certificates and Records	SUXOS
Personal Exposure Monitoring	In accordance with applicable OSHA standards, 29 CFR 1904, 1910, and 1926	SUXOS
Accidents	Accident Investigation Report (UXB Form 10-04-2)	SUXOS
Morning Safety	Conducted Daily Safety Meeting Attendance Log (UXB Form 10-02-1)	SUXOS
Visitor Safety Briefings	Safety Meeting Attendance Log (UXB Form 10-02-1)	SUXOS
Equipment Maintenance	Heavy Equipment Inspection Log (UXB Form 02-04.2-1) Daily Truck Inspection Log (UXB Form 12-00.1-1)	HYDROLYSIS PROCESS Engineering Tech
Daily Activities	Official record of operational activities, will contain, as a minimum, the following information: Date and start and stop times Daily weather conditions Personnel on-site, company, and title or classification Record of Site Safety Meeting Proposed and actual work activities Equipment use (type and length of time) Injuries, incidents, or any other safety and health-related issues or situations, including as many facts concerning the accident/incident as possible Official communications, written and verbal	SUXOS

Appendix D:

Test Results:

Laboratory Tests of BDU-33 Practice Bombs with Installed KC-4 Smoke Cartridges

Analytical Report For 306076

for

UXB International

Project Manager : Harley Heaton

Project Name : CAD/Hydrolysis

July 8, 2003

GPL

Laboratories

**GPL Laboratories, LLLP Certifies that the test results meet all requirements of the
NELAC Standards unless otherwise noted.**

**Reviewed by,
Project Manager**

**Approved by,
Laboratory Director**

**202 Perry Parkway Gaithersburg, MD 20877 Phone (301) 926-6802 Fax: (301) 840-1209
www.gplab.com**

Summary of Analytical Results

Client ID: BDUHYD060903
GPL ID: 306076-001-001-1/1
Matrix: WATER
Date Collected: 06/11/2003
Date Received: 06/11/2003

Prep Method: SW3010A
Prep Date: 06/16/2003
Prep Time: 00:00
Prep Batch: 60886

Analytical Method: SW6010B
Date Analyzed: 06/17/2003
Time Analyzed: 19:49
Analysis Batch: 14842

Parameter	Result	Rep Limit	Units	Qualifier	D.F.
Arsenic	BQL	800	ug/L	U	40
Barium	BQL	4000	ug/L	U	40
Cadmium	BQL	240	ug/L	U	40
Chromium	BQL	200	ug/L	U	40
Lead	BQL	400	ug/L	U	40
Selenium	BQL	800	ug/L	U	40
Silver	BQL	120	ug/L	U	40

Summary of Analytical Results

Client ID: BDUHYD060903
GPL ID: 306076-001-001-1/1
Matrix: WATER
Date Collected: 06/11/2003
Date Received: 06/11/2003

Prep Method: SW7470A_DIG
Prep Date: 06/13/2003
Prep Time: 15:30
Prep Batch: 60867

Analytical Method: SW7471A_TCLP
Date Analyzed: 06/16/2003
Time Analyzed: 11:40
Analysis Batch: 14842

Parameter	Result	Rep Limit	Units	Qualifier	D.F.
Mercury	BQL	8	ug/L	U	40

Summary of Analytical Results

Client ID: BDUHYD060903
GPL ID: 306076-001-001-1/1
Matrix: WATER
Date Collected: 06/11/2003
Date Received: 06/11/2003

Prep Method: EXT_SW8330
Prep Date: 06/20/2003
Prep Time: 00:00
Prep Batch: 61003

Analytical Method: SW8330
Date Analyzed: 07/06/2003
Time Analyzed: 10:22
Analysis Batch: 61351

Parameter	Result	Rep Limit	Units	Qualifier	D.F.
1,3,5-Trinitrobenzene	BQL	0.26	ug/L	U	1
1,3-Dinitrobenzene	BQL	0.26	ug/L	U	1
2,4,6-Trinitrotoluene	BQL	0.26	ug/L	U	1
2,4-Dinitrotoluene	BQL	0.26	ug/L	U	1
2,6-Dinitrotoluene	BQL	0.26	ug/L	U	1
2-Amino-4,6-Dinitrotoluene	BQL	0.26	ug/L	U	1
4-Amino-2,6-Dinitrotoluene	BQL	0.26	ug/L	U	1
HMX	BQL	0.52	ug/L	U	1
Nitrobenzene	BQL	0.26	ug/L	U	1
RDX	BQL	0.52	ug/L	U	1
Tetryl	BQL	0.52	ug/L	U	1
m-Nitrotoluene	BQL	0.52	ug/L	U	1
o-Nitrotoluene	BQL	0.52	ug/L	U	1
p-Nitrotoluene	BQL	0.52	ug/L	U	1

GPL LABORATORIES, LLP
ANALYTICAL RESULTS

Project Name : CAD/Hydrolysis

Date Printed July 8, 2003

GPL ID	Client ID
306076-001-001-1/1	BDUHYD060903

Appendix E:

Test Results for the Disposal of Neutralized Process Solutions



Clean Harbors San Jose, LLC.
1021 Berryessa Rd.
SAN JOSE, CA 95133

DATE RECEIVED: 06/14/05
DATE TESTED: 06/14/2005
REPORTED: 6/17/2005

Generator: Luke AFB Barry M Goldwater Range
Customer: UXB International Inc.
Profile: CH99734B

ANALYTICAL REPORT

ANALYTE	RESULTS	UNIT	METHOD NO.
Layer	2		
Solid, wt/wt (wet)	18	%	
Color	brown		
PH	9.43	STD. Unit	EPA 150.1
Alkalinity	4,000 0.08N	mg Caco ₃ /L Normality	EPA 305.1
Density	1.10		
Ignitability	Negative		
Water Compatibility	pass		ASTM D5058-90
Radiation	Negative		Geiger meter
Cyanide Screen	Negative		ASTM D5049-90
Sulfide Screen	Negative		ASTM D4978-89
Ammonia	ND		

* This results is for process Tank sample, not for rinse sample

ND: NOT DETECTED

NOTE: This analysis is intended for use by Clean Harbors employees
for the purpose of determining waste acceptability. No other claims are made or implied.

Roger Lo

ROGER LO (Lab supervisor)