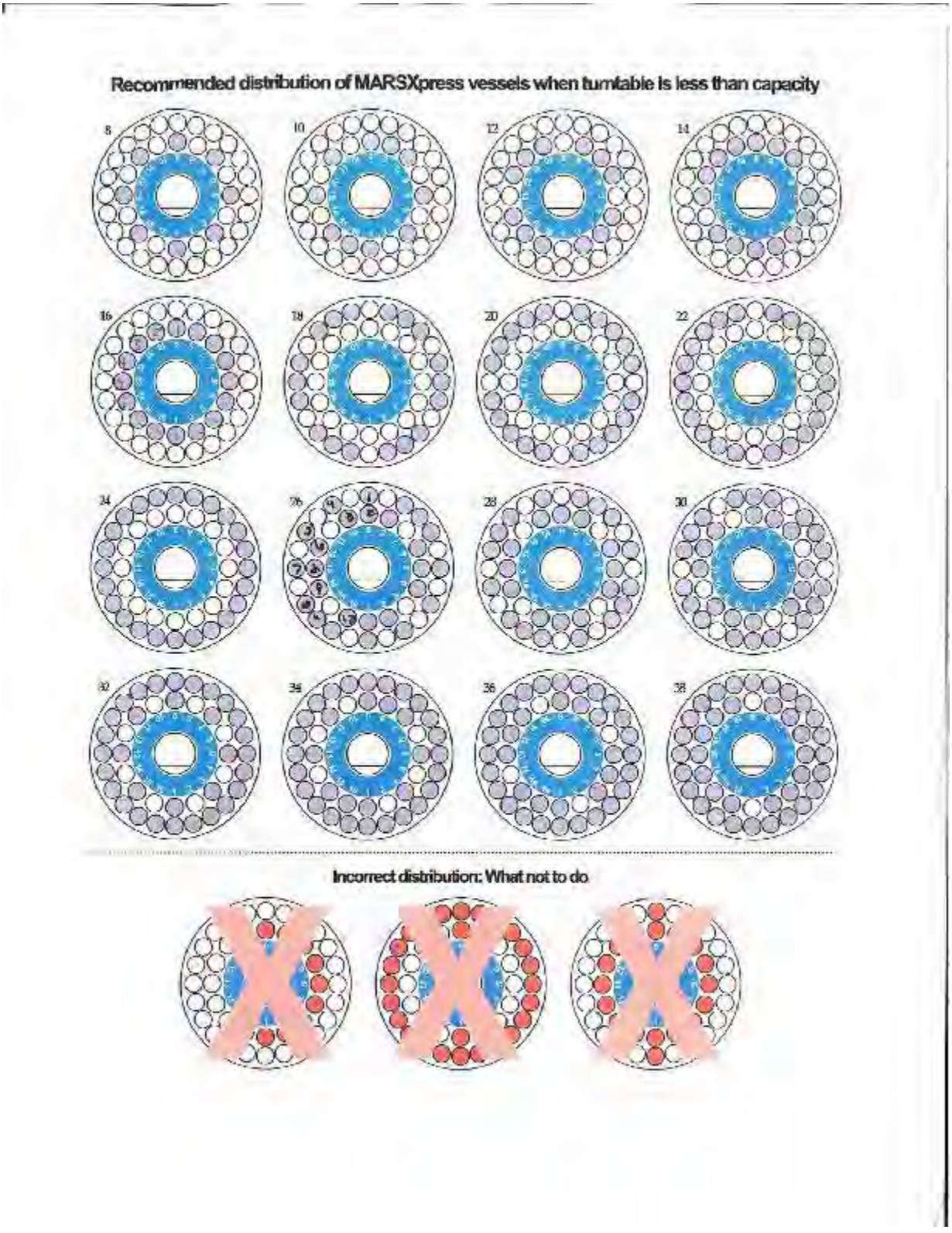
TABLE 1.

Determinative Methods Using Microwave Extraction

Method Description	Determinative Method	SOP
Chlorinated Pesticides	SW-846 8081A SW-846 8081B	DV-GC-0020
Polychlorinated Biphenyls (PCBs)	SW-846 8082 SW-846 8082A	DV-GC-0021
Diesel and Residual Range Organics	SW-846 8015B SW-846 8015C SW-846 8015D NWTPH-Dx AK102 AK103	DV-GC-0027
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM SW-846 8270D SIM	DV-MS-0002
Low-Level NDMA by Isotope Dilution, GC/MS SIM, Large Volume Injection	SW-846 8270C/D SIM	DV-MS-0015

ATTACHMENT 1.

Proper Carousel Loading



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Title: Ultrasonic Extraction of Solid Samples [SW-846 3550B & 3550C]

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Facility Distribution No. _____ Distributed To: _____

1.0 Scope and Application

1.1 This SOP is applicable to the solvent extraction of organic compounds from solid samples, including wipes, using sonication (i.e., ultrasonic extraction). This SOP is based on SW-846 Method 3550B and 3550C.

1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate spiking mixtures and extraction solvents are used.

1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, Concentration of Organic Extracts, for those details.

2.0 Summary of Method

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This mixture is solvent extracted three times using an ultrasonic horn.

3.0 Definitions

Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in “take an aliquot of a sample for testing or analysis.” In the context of this SOP, “aliquot” is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 In order to extract especially wet solids, the initial sample weight might have to be reduced in order to achieve a free-flowing mixture with the sodium sulfate. This can raise the reporting limits and method detection limits.

4.3 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of this SOP (Section 9). Specific selection of reagents may be required to avoid introduction of contaminants.

4.4 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.5 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.6 There are many sources of phthalate contamination in the laboratory. The most common of which are nitrile gloves. The analyst should never touch the inside of glassware with gloves. For the analysis of low-level phthalates by method 8270C SIM, common filter paper can introduce phthalate contamination. Therefore when samples are extracted for this analysis, the Method Comments will instruct the analyst that only glass wool can be used.

4.7 It has been observed that 8270 compounds benzoic acid, 2,4-dinitrophenol, and 4,6-dinitro-2-methylphenol will not recover well if the extract does not drain completely and quickly through the sodium sulfate. Therefore it is very important that a thorough rinse is performed – especially after the 1st sonication. Recoveries will also be improved if the filter paper and funnels used allow for quick drainage. It has been observed that Büchner funnels and glass fiber filter paper will slow drainage.

4.8 It has been observed that 8270 compound Benzidine will not recover well if the filter paper and sodium sulfate are not sufficiently rinsed. Therefore it is very important that a thorough rinse is performed – especially after the 1st sonication.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the

assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection. During operation, the horns will be kept in a sound enclosure inside the fume hood to protect the analyst. If a sound enclosure is not used, then hearing protection is required when within 10 feet of an operating ultrasonic disrupter and the analyst must be in the Hearing Protection Program per DV-HS-0010, Hearing Conservation Program.

5.1.2 Eye protection that satisfies ANSI Z87.1 (as described in the Environmental Health and Safety Manual), laboratory coat, and appropriate gloves must be worn while performing this procedure. Nitrile gloves shall be worn when handling solvents; latex gloves may be worn when handling samples only; and cut resistant gloves shall be worn when washing glassware.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant Poison	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable	50 ppm (TWA)	Prolonged or repeated contact with skin can cause defatting and dermatitis. Contact with eyes can cause redness, tearing, and blurred vision. Exposure can cause lung irritation, chest pain, and edema, which may be fatal.
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Sonicator, at least 300 watts.

6.2 Sonicator horn, ¾ inch

6.3 Balance, >1400-g capacity, accurate to ± 0.1 g, calibrated daily per SOP DV-QA-0014.

6.4 Beakers, 400 mL.

6.5 Media bottles, 250 mL.

6.6 Stainless steel conical funnels

6.7 Ashless cellulose filter paper

6.8 Glass wool - For the analysis of low-level phthalates by method 8270 SIM.

6.9 Pipetter with disposable 1.0-mL tips, calibrated daily per SOP DV-QA-0008.

6.10 Aluminum foil.

6.11 Wooden tongue depressors

6.12 Metal spatulas.

6.13 Solvent dispenser pump.

6.14 Filter flask.

6.15 Vacuum pump.

6.16 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 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.

7.1.1 Methylene chloride – Each lot of solvent is tested following SOP CA-Q-S-001 or CA-Q-W-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.1.2 Acetone - Each lot of solvent is tested following SOP CA-Q-S-001 or CA-Q-W-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.1.3 Hexane - Each lot of solvent is tested following SOP CA-Q-S-001 or CA-Q-W-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.1.4 Baked Sodium Sulfate, 12-60 mesh - QA personnel post the list of approved lots at solvent storage areas. Heat sodium sulfate in a 400°C oven for at least four hours. Cool, covered tightly with foil, and store in tightly closed jars.

7.1.5 Baked Ottawa Sand – Heat Ottawa sand in a 400°C oven for at least four hours.

7.2 Standards

7.2.1 Please reference SOP DV-OP-0020 for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils for Method 8082A	Glass with Teflon-lined lids	30 grams	Cool, $\leq 6^{o}C$	None	SW-846
Wipes for Method 8082A	Glass with Teflon-lined lids	N/A	Cool, $\leq 6^{o}C$	None	SW-846
Soils for all other Methods, including 8082	Glass with Teflon-lined lids	30 grams	Cool, $\leq 6^{o}C$	14 days	SW-846
Wipes for all other Methods, including 8082	Glass with Teflon-lined lids	N/A	Cool, $\leq 6^{o}C$	14 days	SW-846

[1] Exclusive of analysis. Some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require the 14 day holding time for both Methods 8082. The states of Alabama, California, Colorado,

Connecticut, Nevada, New Jersey, Pennsylvania, and Rhode Island require the 14 day holding time for method 8082.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, *Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

9.4.1 One method blank must be processed with each preparation batch.

9.4.2 The method blank for batches of soil samples consists of 30 grams of baked Ottawa sand, which is free of any of the analyte(s) of interest.

9.4.3 TestAmerica Denver typically provides clients with clean filter paper or sterile gauze to use as wipes. In these cases, the laboratory prepares wipe-matrix MBs by spiking clean filter paper or gauze (of the same type that is provided to the client) with the surrogate compounds to be used for analysis. If the client uses a different type of material for the wipes, the client should provide a clean specimen of that material to be used for the MB. If the client does not provide a blank wipe in this case, the laboratory will prepare the MBs from filter paper or gauze, from the laboratory's inventory, spiked with the surrogate compounds.

9.5 Laboratory Control Sample (LCS)

9.5.1 At least one LCS must be processed with each preparation batch. Some projects require two LCSs (LCS and LCSD) in every batch, therefore it is important to check special project instructions for each sample. Specifically, Alaska Methods AK102 and AK103 require an LCS and LCSD.

9.5.2 For soil sample batches, the LCS consists of 30 g of reagent sand to which the analyte(s) of interest are added at a known concentration.

9.5.3 LCSs for wipe-matrix samples are prepared by spiking the compounds of interest and surrogate compounds onto a piece of clean filter paper or sterile gauze. If the client uses a different type of material for the wipes, the client should provide blank wipe material to the laboratory for use in preparing the LCS. If the client does not provide blank wipe material, the laboratory will prepare LCS using clean filter paper or sterile gauze, from the laboratory's inventory, spiked with the compounds of interest and surrogate compounds.

9.5.4 The LCS is carried through the entire analytical procedure just as if it were a sample.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes

have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis. MS/MSDs are not performed on wipe samples.

9.6.2 If insufficient sample volume is available for MS/MSD, an NCM must be written. For SW-846 methods a LCS/LCSD will be required in this case with the exception of work done under the AFCEE program which allows precision to be calculated using LCSs from different batches over the duration of the project.

9.6.3 DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD must be prepared.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

9.8 Sample Duplicates

A sample duplicate is a second aliquot of an environmental sample that is processed with the first aliquot of that sample. Sample duplicates are processed as independent samples within the same batch. The sample and duplicate results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample duplicate precision results are not necessarily representative of the precision for other samples in the batch. Sample duplicates are performed when requested by the client. Sample duplicates do not count towards the 20 sample batch limit.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Specific glassware or equipment positions for the MB and LCS/LCSD are not to be used.

10.4 Critical Procedural Considerations

10.4.1 As stated throughout this SOP, analysts must review the Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

10.4.2 Analysts must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other beaker or media bottle than the designated one should be cleaned or disposed of before coming into contact with the sample. Gloves should never come into contact with the inside of beakers, media jars, or steel funnels.

10.4.3 Sodium sulfate should be kept in closed containers when not in use. It is important to close the container when not actively using the sodium sulfate.

10.5 Sonicator Tuning and Horn Inspection

10.5.1 Every week the sonicator horns are inspected for pitting and the condition is recorded in the Sonicator logbook. The degree of pitting is decided based on the horn's likeness to one of three reference images (attachment: Figure 1).

10.5.1.1 If the condition of the sonicator horn is determined to be poor. The sonicator must be removed from service until the probe is replaced.

10.5.2 If the sonicator is not self-tuning, the sonicator must be tuned once a week or whenever a new horn is installed. Tuning is documented in the sonicator maintenance log.

10.5.3 Starting at a power setting of 1, tune the sonicator so that the output is less than 20%.

10.5.4 Repeat the tune at a power setting of 5 and 10. At each power setting, tune the sonicator so that the output is less than 20%.

10.5.5 If the output is over 20%, consult your supervisor and the manufacturer's manual for troubleshooting help.

10.6 Assemble and clean the glassware immediately before use.

NOTE: Rotate glassware; do not use specific glassware or positions for the MB and LCS/LCSD.

10.6.1 Rinse 400-mL thick-walled beakers with methylene chloride.

NOTE: In order to prevent phthalate contamination, never touch the inside of a beaker with gloves on. When rinsing beakers be sure

to keep gloves away from the mouth of the beaker.

10.6.2 Without gloves on, fold a 18 cm diameter cellulose filter paper in quarters. Open the folds to create a cone. Place the filter paper in the bottom of a conical stainless steel funnel. Place the funnel on a 250-mL media bottle.

NOTE: For low-level phthalate analysis by 8270 SIM, use glass wool in place of filter paper. Be sure not to touch the glass wool with gloves. Check the Method Comments to determine if this is necessary and see Section 4.6.

10.6.3 Place approximately 1 tablespoon of baked sodium sulfate in the funnel. Rinse all surfaces of the funnel, the filter and the sodium sulfate with methylene chloride or acetone/methylene chloride (depending on the extraction solvent, see Section 10.8) so all surfaces of the funnel, filter, and sodium sulfate are rinsed.

NOTE: When preparing glassware for the extraction of wipe samples, sodium sulfate is not necessary and the solvent used in the rinse should be the solvent used in the extraction of the wipe samples. (Normally hexane for methods 8081 and 8082).

10.6.4 Allow the solvent to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional solvent to the rinse if necessary.

10.6.5 Pour the solvent out of the media bottle over the stem of the stainless steel funnel to rinse the funnel stem.

10.6.6 Discard the solvent in the correct waste stream.

10.7 Aliquot Samples

10.7.1 If the sample is a wipe, the sonication can be performed with the wipe in its original container if the original container is large enough. Otherwise, transfer the wipe and any solvent from the original container to a clean beaker.

10.7.2 For each MB and LCS, place a clean wipe into a labeled beaker and proceed to section 10.8.

10.7.3 If the sample is a soil, mix and homogenize samples according to the instructions provided in SOP DV-QA-0023, Subsampling. Use a disposable wooden spatula or a metal spatula that has been rinsed with methylene chloride and dried with a lab tissue.

10.7.4 Break the sample aliquot up into small pieces. The aliquot must not contain particles or clumps bigger than ½ inch in diameter in order to facilitate a complete extraction.

10.7.5 Label a 400-mL beaker with the sample ID, method, and batch number.

10.7.6 Weigh 30 to 33 g of sample into the labeled beaker. Record the weight to the nearest 0.1 g directly into the LIMS or hand record the weight on the benchsheet.

NOTE: Some clients may require the initial aliquot to be adjusted based on the percent moisture of the sample. In those cases, it might be necessary to aliquot more than 33 g of sample. If this is required, the Method Comments will state "Perform Calculation". The laboratory's LIMS (TALS) will calculate the required initial weight of wet sample needed to ensure at least 30 g of dry sample is included in the initial aliquot. In TALS, under the Batch Notes, enter a "1" in the "Perform Calculation" field. TALS will then calculate the required initial weight of wet sample needed under the "Target Amount" field in the Worksheet tab. Weigh out at least that mass of wet sample.

10.7.7 Add approximately 1 tablespoon of baked sodium sulfate to the beaker and mix well. If the sample is especially wet, more sodium sulfate will be needed to ensure the sample is free-flowing. If the sample is extremely wet, the initial sample weight might have to be reduced in order to keep the volume of sample and sodium sulfate in the beaker to a level that the horn can still thoroughly disrupt. Document in an NCM if additional sodium sulfate is added.

10.7.8 For each MB and LCS sample, weigh 30 to 33 g of baked Ottawa sand into labeled beakers. Add 1 tablespoon of baked sodium sulfate to the beaker and mix well. Record a nominal weight of 30 g in the initial volume field, but record the actual weight to the nearest 0.1 g in the notes column.

10.7.9 Cap the beaker tightly with aluminum foil.

10.7.10 Place the beaker on a cart next to the sample container so that a second analyst can check the labels. This is documented on the Organic Extraction Worksheet (See WI-DV-0009).

10.8 Prepare a bottle with a bottle-top dispenser with the appropriate solvent.

10.8.1 Methylene Chloride is used for soil and wipe samples for the following methods:

- SW-846 8015B
- SW-846 8015C
- SW-846 8015D
- Alaska Methods AK102 and AK103
- NWTPH-Dx

- Oklahoma DRO Method

10.8.2 For soil extraction of all other methods, the solvent used is a 1:1 mixture of methylene chloride and acetone.

10.8.3 For wipe samples by method 8081 and 8082, the solvent used is hexane.

10.8.4 For wipe samples by method 8270, the solvent used is a 1:1 mixture of methylene chloride and acetone.

10.9 Add Surrogate, Spikes, and Solvent to Field Samples and all QC samples.

10.9.1 The standards should be allowed to come to room temperature before spiking the samples. Record the ID of the standard and pipettor(s) used on the benchsheet.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-0009 for Surrogate and spike volumes.

10.9.2 Only one batch should be surrogated at a time to ensure the correct standards are used and to ensure the solvent is added as soon as possible to the samples.

10.9.3 Ensure that the sample is free flowing before adding the surrogate standard. If the sample has become hard, gently tap the beaker to break up the solid, or pull back the foil and mix with a wooden spatula if necessary.

10.9.4 Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard to the beaker for each field sample and method blank. Do this by punching a hole in the aluminum foil cap with the pipette tip.

10.9.5 Using a calibrated pipette, add the appropriate volume of the appropriate working spike standard to the beaker for each LCS, LCSD and MS/MSD. Do this by punching a 2nd hole in the aluminum foil cap with the pipette tip.

10.9.6 Immediately after the addition of the spike standard to the LCS, MS, & MSD sample, add approximately 100 mL of the appropriate solvent. Note that the solvent should be added as soon as possible after the addition of the spiking standards to prevent loss of the more volatile extractables. Sufficient solvent should be added so that the solvent level is at least ¾ inch above the solids.

NOTE: When hexane is used as the extraction solvent, use only enough to cover the wipe, i.e., approximately 50 mL. This will help facilitate the concentration of the extract later.

10.10 Rinse the disrupter horn with methylene chloride and wipe down with a clean

laboratory tissue.

10.11 Place the bottom surface of the disrupter horn tip just below the surface of the solvent, but above the sediment layer.

10.12 Sonicate for three minutes, making sure the entire sample is agitated. The output should be set at 10 for the ¾-inch standard horn. The mode switch should be set on pulse, and the percent-duty cycle knob at 50%, for a total process time of 1:30 (1 minute 30 seconds).

10.13 Ensure the filter paper is wet before decanting and filtering occurs. Decant and filter the extract through the prepared stainless steel funnel into the media bottle. Immediately rinse the sodium sulfate in the funnel with at least 50 mL of solvent, ensuring that all sides of the filter paper have been rinsed also. **This is a critical step and must be performed as soon as the extract has drained from the funnel and must be done with at least 50 mL of solvent.**

NOTE: If proper rinsing has occurred, there should **not** be a significant yellow ring of residue (from the spike standards) around the top of the filter paper.

10.14 Repeat the extraction two more times with the appropriate solvent. Each time add sufficient solvent so that the solvent level is at least ¾ inch above the solids. If wipes are being extracted with hexane, then repeat two or more times with additional 50-mL portions of solvent.

10.15 Decant off the solvent after each sonication. After the third and final sonication, pour the entire extract into the funnel. Do not attempt to decant at this step but make every effort to recover all solvent from the beaker. If sufficient room in the media jar exists, rinse the beaker and/or the funnel with an additional 10 to 20 mL of solvent and add the rinse to the funnel.

10.16 Once the solvent has completely drained into the media bottle, dispose of the solid sample and the sodium sulfate into Waste Stream D and cap the media bottle containing the extract with aluminum foil.

10.17 Be sure to rinse the disrupter horn between samples following the procedure in Section 10.10.

10.18 If the extract contains visible solids, it will be necessary to filter the extract again. This filtration can be performed immediately before the concentration step by filtering the extract through another filter paper and funnel directly into the K-D apparatus. If the extract clogs the filter or filtration is extremely slow, the filter and funnel can be placed on a filter flask and a vacuum can be applied.

10.19 Place the extracts in a refrigerator until concentration, ensuring that the extracts in 1:1 methylene chloride:acetone are placed in a flammable rated refrigerator. Document on the benchsheet in which refrigerator the extracts are stored and the total extract count for the batch.

10.20 Handwritten notes on the benchsheet are entered into LIMS, and the transcribed

data must be verified by a second person. This verification is documented on the Organic Extraction Checklist (see WI-DV-009).

10.21 Maintenance

10.21.1 Unless self tuning, the sonicators must be tuned once a week. See Section 10.4.

10.21.2 The probes must be inspected once a week and replaced if excessively worn.

10.22 Troubleshooting

10.22.1 If the sonicator is not working properly, (either not disrupting the soil sufficiently or over-loading) separate the converter from the horn and the horn from the probe. Always use the special wrenches to avoid damaging the parts. Clean all points of contact with either acetone or isopropyl alcohol and then re-assemble and tighten down with the wrenches.

10.22.2 If after following the steps in Section 10.21.1, the sonicator is still not working properly, try to isolate the problem by plugging the converter into a different control box. If the problem goes away, then the control box needs to be sent off for service. If the problem does not go away, proceed to Section 10.21.3.

10.22.3 If after following the steps in Sections 10.21.1 and 10.21.2 the sonicator is still not working properly, then the problem must be in the converter or the horn or probe. Switch the converter to determine if the converter needs to be sent off for repair. If the converter operates properly with a different horn and probe, then the probe needs to be replaced.

11.0 Calibration

Not applicable to this procedure.

12.0 Calculations / Data Reduction

Not Applicable.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs which require the use of Method 8270D, Revision 5. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method, the policies in section 13 of the Environmental Health and Safety Manual for "Waste Management and Pollution Prevention", and the Waste Management procedure, DV-HS-001P.

15.2 Waste Streams Produced By This Method

15.2.1 Methylene chloride – Waste Stream B

15.2.2 Flammable solvent – Waste Stream C

15.2.3 1:1 MeCl2:Acetone – Waste Stream CA

15.2.4 Solid waste/sodium sulfate – Waste Stream D

15.2.5 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3550C Ultrasonic Extraction, Revision 3, February 2007.

16.2 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3550B Ultrasonic Extraction, Revision 3, December 1996.

16.3 Alaska Method AK102, "For the Determination of Diesel Range Organics", Version 04/08/02.

16.4 Alaska Method AK103, "For the Determination of Residual Range Organics", Version 04/08/02.

16.5 Oklahoma Department of Environmental Quality, Methods 8000/8100 (modified) Diesel Range Organics (DRO), October 22, 1997 Rev. 4.1.

16.6 NWTPH-HCID "Hydrocarbon Identification Method for Soil and Water," Manchester Environmental Laboratory, Dept. of Ecology, State of Washington.

17.0 Method Modifications:

17.1 SW-846 Method 3550C Section 11.3.5 calls for all three extractions to be filtered a second time through the same filter. The SOP only requires each extract to be

filtered after each extraction process. The QC has shown great recovery with the use of only the filtration proceeding each extraction.

17.2 SW-846 Method 3550C Section 11.3 instructs that the surrogate and spike compounds should be added to the sample before the sample is mixed with sodium sulfate. This SOP calls for the sample to be mixed thoroughly with sodium sulfate before the surrogate and spike compounds are added. This is done per EPA Memo dated August 5, 2010 titled "Spiking (Prior To vs. After Sample Drying) Issue in SW-846 Organic Extraction Methods."

17.3 SW-846 Method 3550C calls for the use of Büchner funnels and vacuum filtration of all extracts. This SOP calls for the use of conical funnels. This was done to prevent the extract from becoming trapped in the sodium sulfate in the Büchner funnel and specifically to improve the recoveries of benzoic acid, 2,4-dinitrophenol, and 4,6-dinitro-2-methylphenol.

17.4 Oklahoma Department of Environmental Quality method calls for the aliquot not to exceed 20 g. This procedure calls for the soil aliquot to be 30 g to 33 g.

17.5 Oklahoma Department of Environmental Quality DRO method calls for solvent to be added to the sample in a 1:1 ratio (milliliters of solvent to grams of sample). This procedure calls for 100 mL of solvent to be added to 30 g of sample.

17.6 Method from the state of Washington uses a 10 g soil sample that is shaken and processed in a sonic bath. This procedure calls for the soil aliquot to be 30 g to 33 g and is processed directly with a sonicator horn.

17.7 Methods 3550B and 3550C instruct the lab to determine the dry weight of the sample. This is performed according to SOP DV-WC-0023 and is not included in this SOP.

17.8 The medium/high concentration extraction procedure described in Methods 3550B and 3550C is not addressed in this SOP.

17.9 Method AK102 and AK103 calls for samples to be extracted by soxhlet. Valid MDLs and IDOCs have been completed using this procedure, therefore method AK102 and AK103 are listed as a possible determinative methods by this procedure.

18.0 Revision History

Rev 14, dated 04 May 2020

- Annual Technical Review

Rev 13, dated 25 April 2019

- Added section 17.9
- Annual Technical Review

Rev 12, dated 20 November 2018

- Added section 7.1.

Rev 11, dated 5 November 2018

- Revised last sentence in the Copyright Information section.
- Corrected MDL SOP Number in section 13.1.
- Added reference to WI-DV-0009 to section 10.9.1.

Rev 10, dated 31 July 2018

- Revised section 10.5.1 to reflect sonicator horn condition evaluation current practice.
- Added section 10.5.1.1 detailing corrective action required for poor sonicator horn condition.
- Added attachment Figure 1 to reference sonicator horn pitting conditions.

Rev 9, dated 31 March 2018

- Annual Review

Rev 8, dated 31 January 2017

- Added the paragraph to Section 3.0 referencing the QAM for general definitions
- Added Section 4.8 in reference to 8270 compound Benzidine.
- Added the paragraph to Section 6.0 regarding documentation of ID for equipment and pipettes
- Updated Section 9.4 clarifying the use of 1 MB per batch
- Updated Section 9.6.3 to require an LCSD when there is no MS/MSD
- Added note to Section 10.6 to rotate glassware and positions
- Updated Section 10.13 to include the rising of the filter paper in addition to the sodium sulfate.
- Added a note to section 10.13 that yellow ring residue should not be significant if proper rinsing has occurred.
- Updated the language regarding MDLs in Section 13.1 to make it consistent with other SOPs
- Added current Section 13.2 LOQV definition/explanation

Rev 7, dated 31 January 2016

- Annual Technical Review
- Removed “with Teflon lined caps” from Section 6.5
- Updated Section 7.1 to contain verbiage consistent with other SOPs
- Updated Section 9.1 to contain verbiage consistent with other SOPs
- Added “from the laboratory’s inventory” to Sections 9.4.3 & 9.5.3 regarding the filter paper or gauze used for Wipe QC samples
- Added Section 9.6.3 regarding DoD MS/MSD requirements
- Added Section 10.3 instructing to not use specific glassware or equipment positions for MB and LCS/LCSDs.
- Updated the Note in Section 10.6.2 to ensure that glass wool is not touched with gloves for low-level phthalate analysis
- Added the need to NCM when additional sodium sulfate is required to Section 10.7.7
- Added instruction to Section 10.7.8 regarding the recording of weights of QC samples

- Added the need to record the pipettor ID used in Section 10.9.1
- Added Section 10.9.3 to ensure sample is free flowing prior to adding the surrogate standard.
- Added the total process time (1 minute 30 seconds) to Section 10.12
- Removed the use of Teflon lined lids from Section 10.16
- Updated Section 10.19 to place 1:1 methylene chloride: acetone extracts in a flammable rated refrigerator
- Revised Section 13.1 – Method Detection Limit Study (MDL)
- Revised Section 13.2 – Demonstration of Capabilities
- Revised Section 13.3 - Training Requirements
- Archived all revision histories 2010 and earlier

Rev 6, dated 31 January 2015

- Annual Technical Review
- Reformatted SOP
- Revised Section 7.1.4 to remove the requirement to test the sodium sulfate before use. This was done to reflect current practice in CA-Q-S-001 DV-1
- Removed references to low-level NDMA method 8270D_SIM_LL. Soils are extracted by method 3546 instead of 3550C.
- Updated Table 1 with methods listed in this SOP and current analytical SOPs.

Rev 5, dated 15 January 2014

- Expanded footnote for HT table in Section 8
- Updated Section 10.6.1 to match the current TALS method for Low-level NDMA.
- Added Section 10.2
- Added Section 10.20 Maintenance.
- Added Section 10.21 Troubleshooting.
- Table 1 was updated to reflect the current SOPs.
- Revised Sections 9 and 10.1 to reflect current practice.
- Section 9 was revised to state this procedure meets all DoD QSM 5 criteria.
- Section 9.11 was revised to clarify that sample duplicates do not count toward the 20 sample batch limit.

Rev 4, dated 30 November 2012

- Updated Section 8 to indicate per SW-846 Revision 4, soils and wipes for analysis under Method 8082A do not have a holding time.
- Updated Section 9.9 to indicate that the DoD does not require LCSD.
- Updated Section 10.5.7 to indicate that the initial sample weight might have to be reduced for extremely wet samples.
- Section 10.5.5 was revised to remove the requirement to document the extraction date on the extract label.
- Updated Section 15 to include Waste Stream CA.

Rev 3.1, dated 30 November 2011

- Source method review
- Removed references to Method 8070; method no longer active at lab.
- Updated Section 9 to state that MBs and LCSs for wipe samples are created either from filter paper or sterile gauze.

- Added Section 9.11 to include definition and requirements for sample duplicate.
- Added a Note to Section 10.5.6 to describe how to adjust the initial aliquot mass to compensate for percent moisture.
- Updated Section 17 to exclude dry weight determination, high concentration method and Method NWTPH-HCID.
- Updated method references to include NWTPH-HCID.
- Updated SOP references in Table 1 to reflect active SOPs.
- Formatting and grammatical changes throughout

Earlier revision histories have been archived and are available upon request.

19.0 Attachments

Table 1: Determinative Methods Using Ultrasonic Extraction
Figure 1: Sonicator Horn Condition Reference Images.

TABLE 1.

Determinative Methods Using Ultrasonic Extraction

Method Description	Determinative Method	SOP
Diesel Range Organics, Jet Fuels, Motor Oil, Residual Range Organics	SW-846 8015B SW-846 8015C SW-846 8015D Alaska Methods AK102 & AK103 NWTPH-Dx Oklahoma DRO Method	DV-GC-0027
Chlorinated Pesticides	SW-846 8081A SW-846 8081B	DV-GC-0020
Polychlorinated Biphenyls	SW-846 8082 SW-846 8082A	DV-GC-0021
Polynuclear Aromatic Hydrocarbons	SW-846 8310	DV-LC-0009
Semi-volatiles by GC/MS	SW-846 8270C SW-846 8270D	DV-MS-0011 DV-MS-0012
Polynuclear Aromatic Hydrocarbons by GC/MS	SW-846 8270C SIM SW-846 8270D SIM	DV-MS-0002

Figure 1.

Sonicator Horn Condition Reference Images

Condition: good; no further action needed.

Condition: fine; minor pitting, monitor condition.

Condition: poor; heavy pitting, replace horn.

Title: Extraction of Nitroaromatic and Nitroamine Explosive Compounds and Picric Acid from Soil Samples [SW-846 8330A & 8330B]

Approvals (Signature/Date):

Andrew Pepping — 1/29/20 Date Technical Manager	Reed Pottruff — 1/31/20 Date Health & Safety Manager / Coordinator
Roxanne Sullivan — 1/30/20 Date Quality Assurance Manager	Scott Hall — 1/31/20 Date Laboratory Director

Facility Distribution No. _____ Distributed To: _____

1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the extraction of nitroaromatic and nitroamine explosive residues from soil samples. This procedure is based on SW-846 8330A and 8330B, but can also be performed on samples for analysis by method SW-846 8321A.

1.2 This procedure does not describe the analysis of the extracts. For those details, see the following SOPs:

1.2.1 DV-LC-0002, *Analysis of Nitroaromatic and Nitroamine Explosive Compounds by HPLC.*

1.2.2 DV-LC-0010, *Analysis of Nitroaromatic and Nitroamine Explosives Compounds by APCI/LC/MS.*

1.2.3 DV-LC-0025, *Analysis of Picric Acid by LC/MS/MS.*

1.3 Application of 8330A versus 8330B

1.3.1 This procedure is for extraction by either Method 8330A or 8330B. The most important differences in the two source methods are the more rigorous sample collection and preparation measures in 8330B, which are designed to produce more representative results. The more rigorous 8330B process is specifically intended to complement the incremental field sampling process described in Appendix A of method 8330B. If incremental or equivalent systematic sampling processes are not employed in the field, then the extra laboratory homogenization and subsampling effort 8330B requires may add little or no improvement in the overall precision of results.

1.3.2 A larger sample size is used for 8330B (10 g) than is used for 8330A (2 g). A larger sieve size is used for 8330B (10 mesh) than is used for 8330A (30 mesh).

2.0 Summary of Method

Solid samples are air dried to a constant weight and sieved. Soil agglomerates are broken with a mortar and pestle, sieve shaker, or mechanical disaggregator. For samples requiring the more rigorous homogenization techniques found in method 8330B, the analyst employs a ring and puck grinder. The samples are extracted with a 0.1% acetic acid in acetonitrile mixture on a shaker table.

3.0 Definitions

3.1 Definition of terms used in this SOP may be found in the Glossary section of the TestAmerica Denver Quality Assurance Manual (QAM) or SOP DV-QA-003P, *Quality Control Program.*

3.2 **Explosives:** As used in this SOP, the term "explosives" refers specifically to the analytes listed in Table 1. These include compounds that can be readily

detonated with heat, shock, or ignition, such as nitroglycerin, RDX, and TNT. It also includes production by-products and degradation products of true explosives.

3.3 **TALS:** TestAmerica Laboratory Information Management System

3.4 **ISM:** Incremental Sampling Methodology - This is a requirement of method 8330B and describes the technique used to take a 10 g aliquot from a sample in at least 30 increments.

3.5 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in TALS, and is the primary basis of prioritizing work.

3.6 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.

3.7 **Grinding Batch:** A grinding batch is up to 20 samples processed through the same grinding procedure. When using the ring and puck mill, the grinding batch is opened with a grinding LCS and a grinding blank and must be closed after 20 samples or after 3 days, whichever is sooner, due to the expiration of the grinding LCS.

3.8 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client.

3.9 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in TALS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.10 **Aliquot**: A part that is a definite fraction of a whole; as in "take an aliquot of a sample for testing or analysis." In the context of this SOP, "aliquot" is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 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, under the conditions of the analysis, by running method blanks.

4.2 Contamination by carryover can occur when a low-concentration sample is extracted immediately following a high-concentration sample.

4.3 Samples from an ammunition plant or depot usually contain analytes that were deposited via water and leaching and therefore are more uniformly dispersed. Therefore, as per SW-846 8330B Section 11.1.4.2, ring and puck is not necessary.

4.4 Samples from firing ranges and impact zones can contain particles of explosives at a variety of sizes, shapes, and compositions. Therefore the entire sample must be processed through a ring and puck prior to removal of the subsample for analysis. Samples collected at the firing point can contain nitrocellulose fibers. These fibers present a special problem in the grinding step. In order to get the fibers to release the target analytes they must be very finely ground. For these samples only the ring and puck should be used. The client needs to be consulted when selecting a grinding mechanism.

4.5 Tetryl decomposes rapidly with exposure to heat as well as methanol/water solution. All samples expected to contain tetryl should not be exposed to temperatures above room temperature.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

5.2.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; non-disposable gloves must be cleaned immediately. When tightening caps on 40 mL glass vials, cut resistant gloves must be worn.

WARNING: Soil samples with explosive concentrations greater than 2% cannot be accepted by the laboratory unless they have moisture content of 25% or greater. Under no circumstances shall a soil sample with an explosive concentration greater than 10% be accepted by the laboratory.

5.2.1.1 If a sample is expected to have an explosive concentration ≥ 2% (but less than 10%), the EH&S Coordinator and Group Leader shall be notified before any work is performed. Additional safety precautions may be implemented as required due to high concentrations of explosives.

5.2.1.2 Soil samples with high concentrations (between 2 and 10%) of explosives should not be ground using a mortar and pestle. Visual observation of a soil samples is important prior to grinding samples. Any samples containing metal fragments, powders, waxy appearing pieces, or other suspicious material should be brought to the attention of the Group Leader and the EH&S Coordinator before proceeding with the procedure. Bypassing the grinding step and proceeding to solvent dilution is an alternative for samples that are determined to be unsafe to grind.

5.2.2 Anyone working in the grinding room needs to be enrolled in the Hearing Conservation Program. See SOP DV-HS-0010 for details. Personnel operating the grinding equipment are required to wear ear plugs when the equipment is turned on. When standing next to the Humbolt mechanical grinder described in Section 6.1.11 during operation, the decibel levels are above 80 decibels, therefore anyone operating the grinder must be enrolled in the Hearing Conservation Program and wear hearing protection. While the grinder is running, the decibel levels in the room are below 80 decibels, therefore personnel not enrolled in the Hearing Conservation Program can be in the room. Hearing protection is always available to every analyst and they are encouraged to use it.

5.2.3 Operations involving handling samples outside of sealed containers are conducted in ventilation hoods to avoid exposure to dust. Dust masks are available for use, but are optional.

5.2.4 Operations involving the grinding of radioactive samples can be particularly hazardous due to the increased potential for exposure from airborne dust. If a sample is labeled as a "CAT 1", "CAT 2", "CAT 3" or "CAT 4" sample, and requires grinding thru the ring and puck, contact the RSO immediately.

5.3 Primary Materials Used

The following is a list of materials used in this method, which have a serious or significant hazard rating. ***This list does not contain all materials used in the method.*** The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagent and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

MATERIAL [1]	HAZARDS	EXPOSURE LIMIT [2]	SIGNS AND SYMPTOMS OF EXPOSURE
ACETONITRILE	Flammable Poison	40 PPM – TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
METHANOL	Flammable Poison	200 PPM - TWA	A slight irritant to the mucous membranes. Toxic effects are exerted upon the nervous system, particularly the

MATERIAL [1]	HAZARDS	EXPOSURE LIMIT [2]	SIGNS AND SYMPTOMS OF EXPOSURE
	Irritant		optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause the skin to be become dry and cracked. Skin absorption can occur, symptoms may parallel inhalation exposure. Irritant to the eyes.
ACETIC ACID, GLACIAL	Corrosive Poison Flammable Liquid and Vapor	10 PPM - TWA	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur. Can cause serious damage to skin, including redness, pain, and burns. Contact with eyes may cause severe damage followed by loss of sight.
(1) ALWAYS ADD ACID TO WATER TO PREVENT VIOLENT REACTIONS. (2) EXPOSURE LIMIT REFERS TO THE OSHA REGULATORY EXPOSURE LIMIT.			

6.0 Equipment and Supplies

6.1 Equipment

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1.1 Balance, capable of measuring ± 0.01 g. Calibration checked per SOP DV-QA-0014.

6.1.2 Orbital shaker table, capable of maintaining 150 rpm for 18 hours.

6.1.3 Pipettor with disposable 1.0 mL tips, accurate to ± 2%, calibration checked daily in accordance with SOP DV-QA-0008.

6.1.4 Bottle-top pipettor, able to dispense 8 to 20 mL, accurate to ± 2%, calibration checked daily in accordance with SOP DV-QA-0008. If the pipettor does not have a digital display, then the calibration check should be performed whenever the pipette is adjusted.

6.1.5 Ring and Puck - for the grinding of soils per method 8330B

The grinding bowl and puck are cleaned after each use by washing with soap and water with a plastic brush, rinsing with hot tap water, rinsing with DI water, and then rinsing with a 10% acetonitrile solution in acetone. A final wipe down of the bowl and puck while still wet with solvent is done with a Kimwipe (TNT in particular is reported to be prone to adhering to steel surface). In addition, sand blanks are used to monitor potential carry-over for each batch of samples (see Section 9.10.1 for details).

6.1.6 Sample drying systems

6.1.6.1 Trays – "baker's rack" type of stack for the air drying of soils per method 8330B Trays – "baker's rack" type of stack for the air drying of soils per method 8330B

6.1.6.2 Drying tower – custom built tower similar to "baker's rack" type stack for air drying soils, including drying fans and air filters.

6.1.7 Sieves, 10 and 30 mesh – Sieves are cleaned after each use by washing with soap and water and a green plastic brillo pad, (be careful not to damage the mesh), rinsing with hot tap water, rinsing with DI water. Prior to use, the sieves are rinsed with 10% acetonitrile in acetone and wiped with a Kim Wipe. Sieves are allowed to dry in a hood prior to use.

6.1.8 Receiver pans and lids – Receiver pans are cleaned after each use by washing with soap and water, rinsing with hot tap water, rinsing with DI water. Prior to use, the receiver pans are rinsed with a 10% acetonitrile in acetone and wiped dry with a Kim Wipe.

6.1.9 Sieve shaker – used to facilitate the sieving of large sample volumes.

6.1.10 Mortar and pestle – cleaned after each use by washing with soap and water, rinsing with hot tap water, and then rinsing with DI water. Prior to use, the mortars and pestles are rinsed with 10% acetonitrile in acetone and wiped with a Kim Wipe and allowed to dry in a hood prior to use.

6.1.11 Mechanical Disaggregator – Humbolt Manufacturing Part Number H-4199. Used in place of a mortar and pestle to quickly reduce cakes of dry soil. The disaggregator reduces soil agglomerates and sieves the soil through a 10 mesh sieve. The mechanical disaggregator is used to break up soil agglomerates, but it is not an alternative to Ring and Puck. The mechanical disaggregator is cleaned after each sample by removing the hopper. The hopper is washed with soap and water, rinsed with tap water, rinsed with DI water, and then rinsed with 90:10 Acetone:Acetonitrile. The Hopper is then wiped dried with a laboratory tissue. The hammers and body of the disaggregator are cleaned after each sample by rinsing with DI water and wiping dry with a laboratory tissue.

6.2 Supplies

6.2.1 Glass vials, various sizes.

6.2.1.1 Amber glass, 40 mL, with Teflon-lined screw caps for the sonication of soil samples.

6.2.1.2 Amber glass, 8.0 mL, with Teflon-lined screw caps, for the storage of final extracts.

6.2.2 Aluminum foil and aluminum dishes.

6.2.3 Parchment paper

6.2.4 0.2-µm PTFE syringe filters and disposable syringes.

6.2.5 Wooden spatulas – used to lay samples out to dry.

6.2.6 Subsampling tools:

6.2.6.1 Scored paper scoops (TAL-0150 and TAL-0150 LARGE from Commodity Management Services)

6.2.6.2 Plastic sample scoops – square-ended

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

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.

7.1 Ottawa Sand – baked at 400 °C for at least 4 hours.

7.2 Acetonitrile, CH_3CN - HPLC grade (ACN). Each lot is tested per CA-Q-S-001 DV-1.

7.3 Soil Extraction Solvent – approximately 0.1% acetic acid in acetonitrile – Open a new 4-liter bottle of acetonitrile and add 4 mL of acetic acid, then cap and mix. This reagent is given a 1 year expiration date.

7.4 Standards

Please reference SOP DV-OP-0020 for information regarding the surrogate and spike standards used in this procedure.

7.5 Grinding LCS Bulk Material

A standard is purchased in a matrix of -20/+70 Sieved Soil that contains the compounds at the concentrations listed in Table 2. This standard comes packaged in 500 g containers. This standard is stored in a refrigerator at 0 °C to 6 °C and is given a 1 year expiration date. After grinding the ground LCS is stored refrigerated and has a three day expiration date.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Soil samples to be extracted by method 8330A for analysis by method 8330A should be collected in eight-ounce wide mouth jars with Teflon-lined caps. When sampling for DoD projects that must comply with DoD QSM requirements for drying and sieving the entire contents of a soil sample container, a separate container should be used to collect a soil sample for this analysis.

8.2 For soil samples to be extracted by method 8330B for analysis by either method 8330B or method 8321A, it is not uncommon to receive samples of 1 kg or more. Samples may be shipped in wide mouth jars or clean plastic bags.

8.3 Sample extracts must be stored refrigerated in amber glass containers at ≤ 6 °C and not frozen.

8.4 Soil and sediment samples should be air dried at ambient temperature until dry enough to sieve. See Section 10.3 for details. Once the sample is air dried, the sample can be stored at room temperature.

8.5 All soil and sediment samples must be extracted within 14 days of collection and analyzed within 40 days after extraction begins.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils	Glass/ plastic	4 grams (8330A)/up to 1 kg (8330B)	Cool ≤ 6 °C	14 Days	SW846 8330A/B

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the TestAmerica LIMS (TALS) and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 0 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument. See QC Policy DV-QA-003P for further details.

Grinding Batches – A grinding batch is up to 20 samples processed through the same grinding procedure. When using the ring and puck mill, the grinding batch is opened with a grinding LCS and a grinding blank and must be closed after 20 samples or after 3 days, whichever is sooner, due to the expiration of the grinding LCS.

9.4 Method Blank (MB)

A method blank (MB) must be prepared and analyzed with each batch of samples. The MB consists of Ottawa sand with surrogates added. The MB is created at the time of extraction after the samples have been dried, sieved, and ground and is then carried through all extraction and analysis steps. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false-positive data.

9.5 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

One LCS must be analyzed with each batch of samples. The LCS must contain specified analytes of interest and must be carried through the entire analytical procedure. The LCS is prepared by spiking the analytes of interest into Ottawa sand. The LCS is created at the time of sample extraction after the samples have been dried, sieved, and ground. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

NOTE: DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD is not required unless requested by the client.

9.6 Matrix Spike Sample (MS) and Matrix Spike Duplicate (MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. The soil matrix spikes are created at the time of extraction. Spikes and surrogate compounds are added after the sample has been dried, sieved, and ground. One MS/MSD pair must be processed for each preparation batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process.

If insufficient sample volume is available for MS/MSD, a LCSD must be performed and an NCM must be written.

NOTE: DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD is not required unless requested by the client.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, DU, TRL, MS, and MSD) is spiked with surrogate compounds.

9.8 Sample Duplicate (DU)

A duplicate sample is required after ring and puck grinding is performed. A duplicate sample is also required for method 8330B, even if grinding is not performed. A sample duplicate is a second aliquot of one of the samples in the batch. Field blanks cannot be used for duplicate testing. The results for duplicates are reported separately, and cannot be averaged when reporting results. Sample duplicate results are used to evaluate the precision of the method. As such, results should be greater than or equal to the RL for a valid statistical comparison.

9.9 Sample Triplicates (TRL)

A triplicate sample is required after ring and puck is performed. A triplicate sample is also required for method 8330B, even if grinding is not performed. The lab will determine the %RSD as defined below. Results for the %RSD as well as the individual replicate results will be reported to the client. The method suggests that the %RSD for the subsampling error is acceptable if it is < 10%. For DoD QSM 5.1, the %RSD is acceptable if it is < 20% for results above the LOQ.

The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{s}{\overline{C}} \times 100\%$$

Where s is the standard deviation of the average concentration and is calculated as follows:

$$s = \sqrt{\frac{\sum_{i=1}^{n} (C_i - \overline{C})^2}{n-1}}$$

In the event that the laboratory is requested to perform the evaluation of field replicate precision, three field replicates designated by the client will be processed through the entire homogenization and extraction steps. The %RSD for these replicates will be calculated as indicated above and reported to the client.

9.10 Grinding Blank (GB)

9.10.1 Ring and Puck Grinding Blanks

Before each sample is processed through the ring and puck mill, the ring and puck will be cleaned per Section 6.1.5. Then approximately 200 g of Ottawa Sand will be ground. This ground sand will be saved and labeled with the sample ID of the next sample ground with the suffix "blank". After a batch of samples has been processed through the ring and puck, a composite will be generated using sub-aliquots from all blanks ground before the samples. This is done by placing approximately 1 tablespoon of material from each of the individual sample blanks in a clean re-sealable plastic bag. The bag is then sealed and the material is mixed and homogenized by shaking and kneading the bag. A 10 g aliquot is

then removed from the bag and labeled as the batch grinding blank. This composite is extracted and analyzed in the same manner as the field samples.

Corrective Action: If the composite grinding blank results are greater than the acceptance limits, then the individual grinding blanks will be extracted and analyzed to determine when the contamination occurred and exactly which samples were affected. Samples associated with a contaminated grinding blank producing positive results for the same contaminant, must be reprocessed and reanalyzed. If un-ground sample is not available, then the potential carry-over between samples must be described in a non-conformance memo and discussed in the final report case narrative.

9.11 Grinding LCS (LCSSRM)

One Grinding LCS must be ground and analyzed with each batch of samples that are processed through the ring and puck. The Grinding LCS must contain specified analytes of interest and must be carried through the entire analytical procedure. The Grinding LCS is prepared by grinding a 500 g aliquot of the Grinding LCS Bulk Material described in Section 7.5 without having air-dried the material before hand. The Grinding LCS must be ground using the same grinding apparatus (ring and puck) as the samples were ground. The Grinding LCS is used to monitor the effects of the grinding process on the analytes of interest. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

Corrective Action: If the Grinding LCS fails the acceptance criteria, samples associated with the Grinding LCS must be reprocessed and reanalyzed. If un-ground sample is not available, then the results of the grinding LCS must be described in a non-conformance memo and discussed in the final report case narrative.

10.0 Procedure

NOTE: Rotate sieves and any applicable equipment; do **not** use specific sieves or equipment for the MB and LCS/LCSD.

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Dry the Samples – Refer to the Flowchart in Appendix 1 and the batching instructions in Appendix 3.

10.3.1 Check the Method Comments to see if the samples are for a project with the Department of Defense (DoD), if yes, then the entire contents of the sample container must be dried. Check TALS to make sure the client sent more than one container if additional tests are being requested. If additional tests are logged and the client only sent one container, the Project Manager should be notified.

10.3.2 If the sample is logged for method 8330B, or for an ISM method, then the entire contents of the sample container must be dried. Check each sample to make sure the client sent more than one container if additional tests are being requested. If additional tests are logged and the client only sent one container, the Project Manager should be notified.

10.3.3 If the samples do not fall under the descriptions given in Section 10.3.1 or Section 10.3.2 then only a portion of the sample container needs to be dried. In these cases, lay out at least 20 g to dry.

10.3.4 Depending on the sample size, the samples are laid out in aluminum pans, or on large trays lined with aluminum foil to dry. Some clients may request metals analysis on the dried samples. In those cases, samples are laid out on parchment paper.

10.3.5 Spread the samples out in a thin layer to facilitate drying. Use a disposable wooden spatula to break up any clumps and agglomerates.

10.3.6 The tray or pan that the sample is laid out into is labeled with the sample ID. A second analyst checks to make sure that the labels on the tray or pan match the labels on the client sample container to ensure samples are not accidently mixed up. This check is documented in TALS.

10.3.7 Place the samples in a hood or well ventilated area at room temperature. Document in TALS the date and time the samples were laid out to dry. If the samples are very wet, a fan can be used to help facilitate the drying process, but care should be taken so that the air flow is not strong enough to cause cross-contamination between samples. An electronic temperature recording device records the temperature of the room and the data is downloaded weekly.

10.3.8 When the samples appear to be dry enough that they can be sieved without caking, subsample approximately 15 grams into an appropriate weighing vessel and record the exact weight, the date, and the time (see Appendix 5). Set this 15 gram aliquot (still in the weighing vessel) next to the rest of the drying sample. Take care to use an appropriate weighing vessel for the analytical methods requested, as the aliquot removed in this step will still be included in the volume used for ISM (i.e. Do not use an aluminum weigh boat for samples requiring metals analysis).

10.3.9 After 2 hours, reweigh the aliquot in the same weighing vessel and record the exact weight, the date, and the time. If the weight of the sample is within 10% of the previous weight, proceed to Section 10.4.

10.4 Sieve the Samples - Refer to the Flowchart in Appendix 2.

10.4.1 If the client requirements specify a particular sieve size, those instructions take precedence.

10.4.2 If the sample is logged for prep method "8330_P_2g" then a 30 mesh sieve should be used.

10.4.3 If the sample is logged for prep method "8330_Sonc_10g" then a 10 mesh sieve should be used.

10.4.4 Some clients will request metals analyses to be performed on the sieved sample. In those cases, a stainless steel sieve should be used. Brass sieves should be avoided.

10.4.5 Clean the sieves prior to use following the instructions in Section 6.1.7.

10.4.6 Some samples may require the use of a mortar and pestle or a mechanical disaggregator to break up dried clumps. Refer to Sections 6.1.10 and 6.1.11 on how to clean and rinse the mortar and pestles and the mechanical disaggregator before use.

10.4.7 Record the weight of the entire dried sample in the Worksheet tab in TALS. This is a requirement for DoD QSM 5.0 and 5.1.

10.4.8 Sieve the entire dried sample through the appropriate sized sieve. Care must be exercised not to eliminate soil agglomerates during this step. The soil can be broken into small pieces with a gloved hand or another instrument (a wooden spatula for example). If a gloved hand is used, care should be taken to change out gloves in between samples so not to cross- contaminate samples.

10.4.9 Remove large rocks, vegetation, and twigs that do not pass through the sieve. Mosses and other types of fine vegetation should be physically shredded while sieving to release trapped soil and residues. The only materials that should be eliminated by sieving are rocks and vegetation. All soil must be broken up to pass through the sieve.

10.4.10 Place any soil that does not pass through the sieve into a clean mortar. Break up soil agglomerates using the pestle. Or as an alternative use the mechanical disaggregator. Be sure to break up all soil so that it can pass through the sieve. Only extraneous material such as rocks and vegetation should be removed with the sieve. Describe all extraneous material that did not pass through the sieve in an NCM. Document the weight of any material that does not pass through the sieve. Document this weight either in the worksheet section of TALS or in an NCM. Label and retain this material that does not pass through the sieve.

10.4.11 Collect all of the material that passes through the sieve on a clean piece of foil or parchment paper.

10.4.12 An automatic sieve shaker can be used to help facilitate the sieving of samples. A receiver pan is placed under a sieve and the sample is added to the sieve. Then a lid or another receiver pan for a second sample is placed on top. The stack is then clamped inside the sieve shaker for no more than 30 minutes. Inspect the samples to ensure that only extraneous material such as rocks and vegetation should be removed with the sieve. If needed use a mortar and pestle to break up soil agglomerates. Describe all extraneous material that did not pass through the sieve in an NCM. Document the weight of any material that does not pass through the sieve. Document this weight either in the worksheet section of TALS or in an NCM. Label and retain this material that does not pass through the sieve.

10.5 Grind the Samples - Refer to the Flowchart in Appendix 2.

10.5.1 If the samples are <u>not</u> logged with a pre-prep method of "ISM_DD_SI_PM_SS," skip this section and proceed to Section 10.6.

10.5.2 <u>Ring and Puck Grinding</u> *Samples logged for "ISM_DD_SI_PM_SS"*

<u>10.5.2.1</u> See Section 6.1.5 on how to clean the ring and puck dish.

<u>10.5.2.2</u> If the sample is logged for ring and puck grinding, a grinding blank per Section 9.10.1 consisting of baked Ottawa sand will be processed through the ring and puck dish <u>before</u> each sample. These individual blanks will be composited into one grinding blank for the associated samples and will be analyzed in addition to the normal extraction blank.

NOTE: When preparing the grinding blanks, it is not necessary to do five 60-second grinds. One 60-second grind of the Ottawa sand is sufficient.

<u>10.5.2.3</u> After a grinding blank has been processed through a ring and puck dish, that blank is labeled as the blank associated to the next sample processed through that same dish. Do not clean the ring and puck dish after the blank.

<u>10.5.2.4</u> Prepare a grinding LCS per Section 9.11 with every batch. The grinding LCS will be analyzed in addition to the normal extraction LCS.

NOTE: A grinding batch will consist of no more than 20 samples that have been ground within three days of each other. The grinding batch is opened with a grinding LCS and a grinding blank and must be closed after 20 samples or after 3 days, whichever is sooner. A grinding batch must have one Grinding LCS, and at

least one Grinding Blank. If more than one Grinding Blank is prepared, it must be very clear on the benchsheet which individual sample blanks were used to build each Grinding Blank.

10.5.2.5 In a hood, transfer the sample into a clean ring and puck dish. Do not overfill the dish (approximately 300 g of sample can fit in one dish). If needed, grind the sample in 300 g or smaller increments and recombine after all sample has been ground. The entire sample must be ground. Place the dish securely in the holder and close the door on the machine. Grind the sample in five 60-second periods with a one minute cooling time between grinds for a total of 5 minutes of grinding. Remove the dish and in a fume hood, open the lid and inspect the sample. It should be the consistency of flour. The consistency of the material is checked by pinching some between two fingers of a gloved hand and feeling for grit and by looking for any un-ground fibers. If grit is detected or if fibers are observed, additional grinding is needed.

NOTE: During the one-minute cooling time, the dish should be placed in a shallow ice water bath to facilitate cooling. Be sure the bath is shallow enough so that water does not get inside the dish.

10.5.2.6 If the sample reaches a flour-like consistency before all 5 one-minute grinds have been completed, then it might be beneficial to not perform all 5 grinds in order to avoid excessive heat and to avoid packing the sample onto the side of the grinder. If the analyst inspects the sample and it has a flour-like consistency before all 5 grinds are completed, they can make the decision to stop after less than 5 grinds. An NCM should be written to document the deviation from the source method and the reasoning.

NOTE: If multiple 300 g increments are used for grinding and the sample is recombined, it has been shown through Duplicate/Triplicate QC results that the sample is non-homogenous. To re-homogenize the sample, place all volume in to a clean plastic bag, seal, and carefully shake the bag for 1-2 minutes until the sample is thoroughly mixed. Lay out the sample back on the foil/parchment paper. This must be done on all samples regardless if this sample will be used for DU/TRL QC.

10.6 Aliquot the Samples

10.6.1 All aliquots should be taken using a subsampling tool described in section 6.2.6. This is done to ensure that finer sample material does not fall off of the sampling tool, as can happen if a spatula was used instead.

This is particularly necessary when samples are not ground to a consistent grain size using the ring and puck.

10.6.2 **2 Gram Aliquot – Extraction Method "8330_P_2g"** – Remove the cap from a labeled 40 mL amber vial and place the vial on a balance and tare. Spread the entire sample out to a thickness no greater than 1 cm. Use a disposable subsampling tool to build a 2.0 g to 2.2 g aliquot by taking at least five small portions from random locations through the entire thickness of the sample. Record the exact sample weights on the benchsheet and cap the vial with a Teflon™ lined lid. Save the remaining soil for possible re-extraction. Create an LCS and a method blank by placing 2.0 g to 2.2 g of baked Ottawa sand in labeled vials. Record a nominal weight of 2 g in the initial volume field, then record the actual weight to the nearest 0.1 g in the notes column.

10.6.3 **10 Gram Aliquot – Extraction Method "8330_Sonc_10g"** – Remove the cap from a labeled 40 mL amber vial and place the vial on a balance and tare. Spread the entire sample out to a thickness no greater than 1 cm. Use a disposable subsampling tool to build a 10 g to 11 g aliquot by taking at least thirty small portions from random locations through the entire thickness of the sample. Record the exact sample weights on the benchsheet and cap the vial with a Teflon™ lined lid. Save the remaining soil for possible re-extraction. Create a LCS and a method blank by placing 10 g to 11 g of baked Ottowa sand in labeled vials. Record a nominal weight of 10 g in the initial volume field, then record the actual weight to the nearest 0.1 g in the notes column. If the samples were ground create a grinding blank per Section 9.10, and take an aliquot from this composite. Aliquot the grinding LCS as you would a sample.

10.7 Add Surrogate, Spikes, and Solvent to the Samples

10.7.1 Refer to WI-DV-0009 for the correct surrogate and spike standards to use and the correct volume.

10.7.2 The surrogate and spikes standards are kept in a freezer, but should be allowed to come to room temperature before use. Record the ID of the standard and pipette(s) used on the benchsheet.

10.7.3 The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-0009.

10.7.4 Only one batch should be surrogated at a time to ensure the correct standards are used.

10.7.5 Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard to each sample and each QC sample.

10.7.6 Using a calibrated pipette, add the appropriate volume of the appropriate working spike standard to each LCS and MS/MSD.

NOTE: Do not add the spike standard to the grinding LCS. The grinding LCS is created using the material described in Section 7.5 and already contains the analytes of interest.

10.8 Add Extraction Solvent

10.8.1 2 Gram Extraction – Extraction Method "8330_P_2g"

10.8.1.1 Taking into account the volume of surrogate and spike standard added to each sample, bring the extract volume up to 10 mL with the soil extraction solvent described in Section 7.3. Use either a 10 mL Class A graduated cylinder or a bottle top pump that has been calibration checked.

Example: If 0.5 mL of surrogate standard was added to a sample, add exactly 9.5 mL of the soil extraction solvent.

Example: If 0.5 mL of surrogate standard and 0.5 mL of spike standard was added to a LCS, add exactly 9 mL of the soil extraction solvent.

10.8.1.2 Proceed to Section 10.9.

10.8.2 10 Gram Extraction – Extraction Method "8330_Sonc_10g"

10.8.2.1 Taking into account the volume of surrogate and spike standard added to each sample, bring the extract volume up to 20 mL with the soil extraction solvent described in Section 7.3. Use either a 25 mL Class A graduated cylinder or a bottle top pump that has been calibration checked.

Example: If 1 mL of surrogate standard was added to a sample, add exactly 19 mL of the soil extraction solvent.

Example: If 1 mL of surrogate standard and 1 mL of spike standard was added to an LCS, add exactly 18 mL of the soil extraction solvent.

10.9 Extract the Samples

10.9.1 Cap vial with a Teflon-lined cap, vigorously hand shake the vial for one minute, or until all material is well mixed, and place it in a box. Place the box on the platform shaker so that the vials are lying on their side. Set the platform shaker at 150 rpm and allow the samples to be shaken for at least 18 hours. Record the start time on the benchsheet.

10.9.2 After the 18 hour extraction, remove the vials from the shaker table and record the stop time on the benchsheet.

10.9.3 If needed, centrifuge the vial at no more than 2,200 rpm to help separate the solids from the extract. Remove approximately 10 mL of the supernatant solution. Filter the supernatant solution using a 0.2-µm PTFE syringe discarding the first mL into the waste. Filter the remaining supernatant into a labeled 8-mL amber vial.

10.9.4 Submit the extract for analysis to the appropriate analytical lab.

10.10 Maintenance

10.10.1 Approximately once a month, the cover on the Ring and Puck should be removed and any dirt should be cleaned up.

10.10.2 When excessive wear is noted, replace the hammers in the Mechanical Disaggregator.

10.10.3 Occasional lubrication of the Ring and Puck clamp is needed.

10.10.4 The o-rings in the Ring and Puck dishes should be replaced when worn.

10.10.5 Every 6 months the centrifuge should be lubricated and tightened.

10.11 Troubleshooting

Low recoveries for Tetryl in the explosives grinding LCS may be indicative of high temperatures during grinding. Review the cooling step noted in Section 10.5.2.5 in order to minimize the effect of the heat generated during the grinding process.

11.0 Method Performance

11.1 Method Detection Limit (MDL)

A valid method detection limit (MDL) study for each analyte of interest must be performed prior to analyzing samples for the first time and verified annually thereafter. Separate soil MDL studies are performed for 8330A using 2 g and 8330B using 10 g of Ottawa sand. Separate soil MDL studies are performed for explosive method 8321A using 2 g of Ottowa sand and 8321A using 10 g of Ottowa sand. An MDL study for picric acid by method 8321A is performed using 10 g of Ottowa sand. An MDL study for explosives by 8321 LC/MS/MS is performed using 10 g of Ottowa sand. The procedure for determining detection

limits is defined in Policy CA-Q-S-006. Quarterly MDLV and LOQV studies are performed for the DoD program (QSM 4.2 and 5.0).

11.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs that specify the requirement. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

11.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

11.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

11.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

11.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

11.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

11.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

11.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

13.0 Waste Management

13.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

13.2 The following waste streams are produced when this method is carried out:

13.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

13.2.2 Flammable solvent waste – Waste Stream C

13.2.3 Solid sample waste - Waste Stream D

13.2.4 Waste soil sample vials - Waste Stream A

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

14.0 References / Cross-References

14.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

14.1.1 Method 8330, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 0, September 1994.

14.1.2 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

14.1.3 Method 8330A, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 1, January 1998.

14.1.4 Method 8330B, Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography, Revision 2, October 2006.

14.2 DoD Environmental Data Quality Workgroup, Frequently Asked Questions (FAQs) Concerning the Implementation of EPA SW-846 Method 8330B, November, 2014.

15.0 Method Modifications:

15.1 Method 8330 prescribes the shelf life for standards as follows:

Standards	Concentration	Shelf Life
Stock standards	1,000,000 µg/L (1,000 ppm)	One year
Intermediate standards	2.5 to 1,000 µg/L	Thirty days
Working standards	1 to 500 µg/L	Daily

This SOP assigns a six-month shelf life to the working level standard based on TestAmerica's experience with these materials. The standards are stored in a freezer.

16.0 Attachments

Table 1. Analyte List
Table 2. Grinding LCS Bulk Material
Appendix 1 Flowchart for Drying Explosive Soils
Appendix 2 Flowchart for Grinding and Sieving Soils
Appendix 3 Instructions for Batching in TALS
Appendix 4 ISM Worksheet
Appendix 5 ISM Constant Weight Worksheet

17.0 Revision History

- Revision 12, dated 01/31/2020
  - Annual Review
- Revision 11, dated 12/12/2018
  - Changed references to WI-DV-009 to correct document ID, WI-DV-0009 throughout SOP.
  - Minor formatting and language corrections throughout.
  - Changed references to "mechanical grinder" to "mechanical disaggregator" throughout SOP, including sections 2.1, 6.1.11, 10.4.6, 10.4.9, 10.10.2. This was done to distinguish more clearly between grinding and soil disaggregation.
  - Added 6.1.6.2 to equipment section to include drying tower.
  - Expanded section 6.2.6 to include new subsampling tool.
  - Updated sections 9.1.2 and 9.9 to reference DoD 5.1 rather than DoD 5.0.
  - Removed note after 10.3.9 which allowed for skipping constant weight analysis for non-DoD samples.
  - Added section 10.4.7 to meet DoD requirement that entire dried sample is weighed prior to sieving.
  - Removed use of grinding stones from section 10.4.11.
  - Removed notes regarding client-specific requirements from sections 10.4.10 and 10.4.12, and incorporated these specific requirements into standard procedures for all samples. These requirements include weighing and retaining material that does not pass through the sieve.
  - Updated sections 10.6.1, 10.6.2, 10.6.3 to reference "subsampling tool" rather than "square-ended scoop."
  - Removed section 10.11 comment regarding securing ring and puck as it no longer requires securing. Added note about troubleshooting low tetryl recoveries.
- Revision 10, dated 31 October 2018
  - Annual Review

- Added 10.3.8 Revision 9, dated 5 October 2017
  - Added wording to 2.1 to clarify that samples are dried to a constant weight.
  - Added 10.3.8 and 10.3.9 and removed 15.2 to include procedure for drying samples to a constant weight.
  - Added Appendix 5
- Revision 8, dated 2 December 2016
  - Added the comment requiring the documentation of equipment IDs to Section 6.1
  - Added Section 6.3 Computer Software and Hardware
  - Added Note to Section 9.5 requiring a LCSD when there is no volume for MS/MSD
  - Added Note to Section 10.0 regarding the rotation of sieves and equipment
  - Updated Section 10.5.2.4 to include the samples should be ground the same length of time as the LCSSRM, renumbered the notes and included a sample grind time exception.
  - Updated Section 10.5.3.5 to include Note 1 from Section 10.5.3.6.
  - Renumbered Note in Section 10.5.3.6
  - Updated Section 10.7.2 to reflect standard SOP
  - Removed the Ball Mill Grinder reference from the entire SOP.
- Revision 7, dated 31 January 2016
  - Annual Technical Review
  - Deleted previous Section 4.3 no longer applied – vegetation and rocks are removed
  - Added Section 4.6 regarding tetryl decomposing with exposure to heat.
  - Added paragraph to Section 7 to contain reagent grade verbiage consistent with other SOPs
  - Section 8.1 – clarified the paragraph to be specifically about method 8330A
  - Added "and not frozen" to section 8.3
  - Revised Section 9.1 to have consistent verbiage and instructions as other SOPs
  - Added Note to Section 9.6 regarding DoD MS/MSD requirements
  - Changed duplicate to triplicate in Section 9.9
  - Clarified instruction to not clean the ring and puck dish after the blank in Section 10.5.3.3
  - Added the weight recording requirements to Sections 10.6.2 & 10.6.3
  - Added "and pipette(s)" to Section 10.7.2
  - Changed the centrifuge speed from 2500 rpm to 2200 rpm and 5mL to 10mL volume of supernatant solution to remove in Section 10.9.3
  - Modified/Rearranged Section 11 to be consistent with other SOPs
  - Removed previous Section 11.1 "Initial Demonstration of Capability"
  - Added current Sections 11.2 "Demonstrations of Capabilities" & 11.3 "Training Requirements"
  - Reformatted Section 14 and added Section 14.2 – reference to DoD Frequently Asked Questions for 8330B
  - Removed references to AFCEE and USACOE throughout document as these programs were incorporated into the DoD program.
  - Removed all 2010 and earlier revision histories
- Revision 6, dated 31 January 2015
  - Annual Technical Review
  - Reformatted SOP.
  - Revised Section 3.7 and Section 9.2 to state that a Ball Mill grinding batch is opened and closed the same day, while a Ring and Puck grinding batch can be open for up to 3 days.

- Revised Section 5.2.2 to give information on the hazards of the Humbolt grinder.
- Revised Section 9.9.1 to give more detail on how the Ring and Puck composite grinding blanks are created.
- Revised Section 10.3.2 and Appendix 1 to state that any sample logged with an ISM method must have the entire sample container dried.
- Revised Section 10.10 to include maintenance on the centrifuge.

- Revision 5, dated 27 January 2014
  - Annual Technical Review
  - Removed Section 1.2.3, DV-LC-0028 no longer performed.
  - Added detail about sieve size to Section 1.3.2.
  - Edited Section 6.1, subsection "Ball Mill" to allow for un-baked sand to be used in the cleaning of the ball mill stones and to allow the use of 1 pint cans. The section was also revised to change the minimum time the stones have to be tumbled during the cleaning process from 3 hours to 2 hours. This was done based on analyst's experience.
  - Edited Section 6.1, subsection "Sieves" to state a brillo pad can be used on the sieves so long as the mesh is not damaged.
  - Updated Section 9.1 to reflect current practice, added a comment stating that this procedure meets DoD QSM 5.0 criteria unless otherwise stated.
  - Removed Acceptance Criteria and Corrective Action information to Section 9. This information can be found in the analytical SOPs.
  - Revised Section 9.6 to state that if there is no volume for a MS/MSD, a LCSD must be performed.
  - Added information to Section 9.9 for DoD acceptance criteria for triplicates.
  - Updated sections 10.1, 10.2 and 11.2 to reflect current practice
  - Added a NOTE in Sections 10.5.2.1 and 10.5.3.6 giving instructions on how to ensure the sample is homogenous after it has been split into separate grinding containers and then later re-combined.
  - Added Section 10.5.3.6 giving guidance on what to do if the sample reaches a flour-like consistency before all 5 grinds have been completed. This was done to avoid over-heating samples and packing the sample against the grinding dish wall.
  - Added Section 10.10 Maintenance and Section 10.11 Troubleshooting per DoD QSM 5.0.
  - Updated Appendix 2 and Appendix 3 to reflect the current method names used in LIMS.
  - Formatting changes throughout
- Revision 4, dated 30 October 2012
  - Annual Technical Review
  - Section 4.6 was added to document the adverse affect headspace in the ball mill can has on the grinding LCS.
  - Section 6.1 and Section 10 were revised to include the description of the Spacer Can in the Ball Mill apparatus.
  - Section 6.1 and Section 10 were revised to include the Mechanical Grinder used as an alternative to mortar and pestle.
  - Section 9.6 was revised to state that LCSDs are not required for DoD work.
  - Section 10 was revised to reference the Explosive Review Checklist in WI-DV-0009.
  - Section 10.3.2 was revised to instruct the analyst to eliminate as much headspace as possible during the Ball Mill grinding step.
  - Appendix 3 was revised to give more detail on the steps taken to ensure all pre-

ground ISM aliquots are taken before the sample is ground. It was also revised to include the use of the Explosive Extraction Checklist in WI-DV-0009.

- Revision 3, dated 10 October 2011
  - The procedure was revised to have the extraction performed by shaker table instead of cooled sonication bath. This was done to increase lab capacity and to create a more rugged extraction.
  - Section 5 was revised to include the requirement that analysts wear cut-resistant gloves when tightening vial caps.
  - Section 7.2 was revised to include the lot approval process for acetonitrile.
  - Sections 7.5 and 9.3 were revised to mandate a 3 day expiration date on the Grinding LCS after it has been ground.
  - Section 9.8 and 9.9 were revised to require a duplicate and triplicate whenever method 8330B is performed, not just when samples are ground.
  - Section 10.2.2 and 10.2.3 were revised to have the analyst use the prep method instead of the pre-prep method to determine sieve size. This is a simpler determination and matches the flow chart in Appendix 2.
  - Section 10.7.3 was revised to change the speed of the centrifuge to prevent the breakage of the extract vials.

- Revision 2, dated 11 January 2011
  - Details about the surrogate and spike standards used in this procedure have been moved to SOP DV-OP-0020.
  - Revised Section 9 to state that duplicates and triplicates are required when ring and puck or ball mill grinding is performed.
  - Revised the procedure to include instructions and details for the laboratory's new LIMS.
  - Revised Section 4 to give more details on the grinding of samples.
  - The procedure was revised to state that samples should be ground on the ball mill for only 8 hours. At that time, the samples should be inspected and only ground longer if required.
  - Added detail in Section 10.1 about the electronic temperature monitoring device that records the temperature of the drying room.
  - Revised the flowcharts to be flowcharts only and not worksheets. All data is now recorded in TALs benchsheets.
  - Added instructions in Appendix 3 on how to batch samples in TALSs
  - Added the option to use an automatic sieve shaker.

Earlier revision histories have been archived and are available upon request.

Table 1. Analyte List

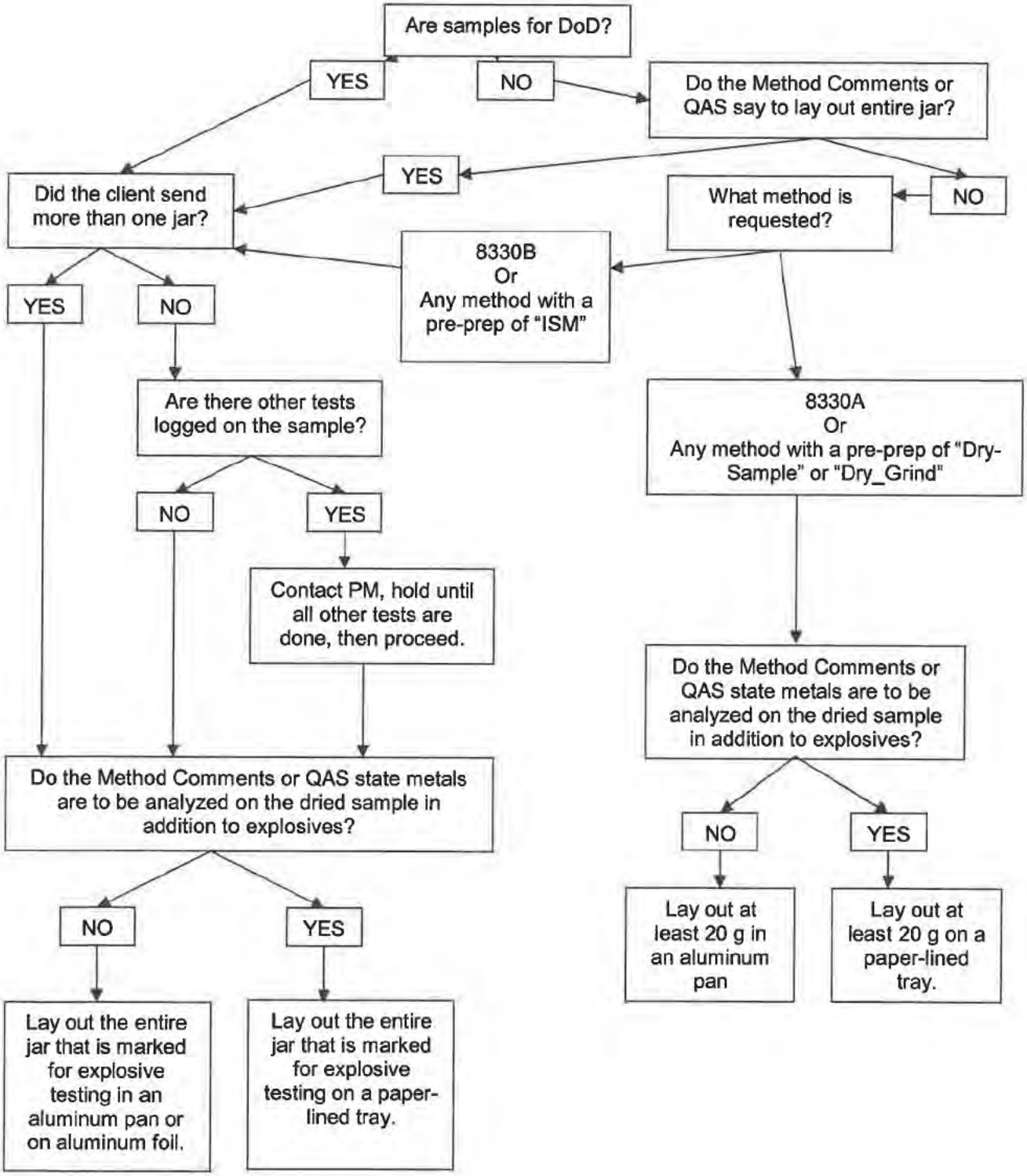
Compound	CAS #	Symbol
Octahydro-1,3,5,7-tetranitro-1,3,5,7,-tetrazocine	2691-41-0	HMX
Hexahydro-1,3,5-trinitro-1,3,5-triazine	121-82-4	RDX
1,3,5-Trinitrobenzene	99-35-4	1,3,5-TNB
1,3-Dinitrobenzene	99-65-0	1,3-DNB
Methyl-2,4,6-trinitrophenyl nitramine	479-45-8	Tetryl
Nitrobenzene	98-95-3	NB
2,4,6-Trinitrobenzene	118-96-7	2,4,6-TNT
4-Amino-2,6-dinitrotoluene	19406-51-0	4-Am-DNT
2-Amino-4,6-dinitrotoluene	35572-78-2	2-Am-DNT
2,6-Dinitrotoluene	606-20-2	2,6-DNT
2,4-Dinitrotoluene	121-14-2	2,4-DNT
2-Nitrotoluene	88-72-2	2-NT
4-Nitrotoluene	99-99-0	4-NT
3-Nitrotoluene	99-08-1	3-NT
Nitroglycerin	55-63-0	NG
PETN	78-11-5	PETN
2,4-Diamino-6-nitrotoluene**	6629-29-4	--
2,6-Diamino-4-nitrotoluene**	59229-75-3	--
Picric Acid	88-89-1	PA
1-Nitroso-3,5-dinitro-hexahydro-1,3,5-triazine**	5755-27-1	MNX
3,5-Dinitroaniline**	618-87-1	3,5-DNA
1,2-Dinitrobenzene (8330 surrogate)	528-29-0	1,2-DNB
Nitrobenzene-d5 (8321 surrogate)	--	NB-d5

** Compounds are only analyzed and spiked upon request.

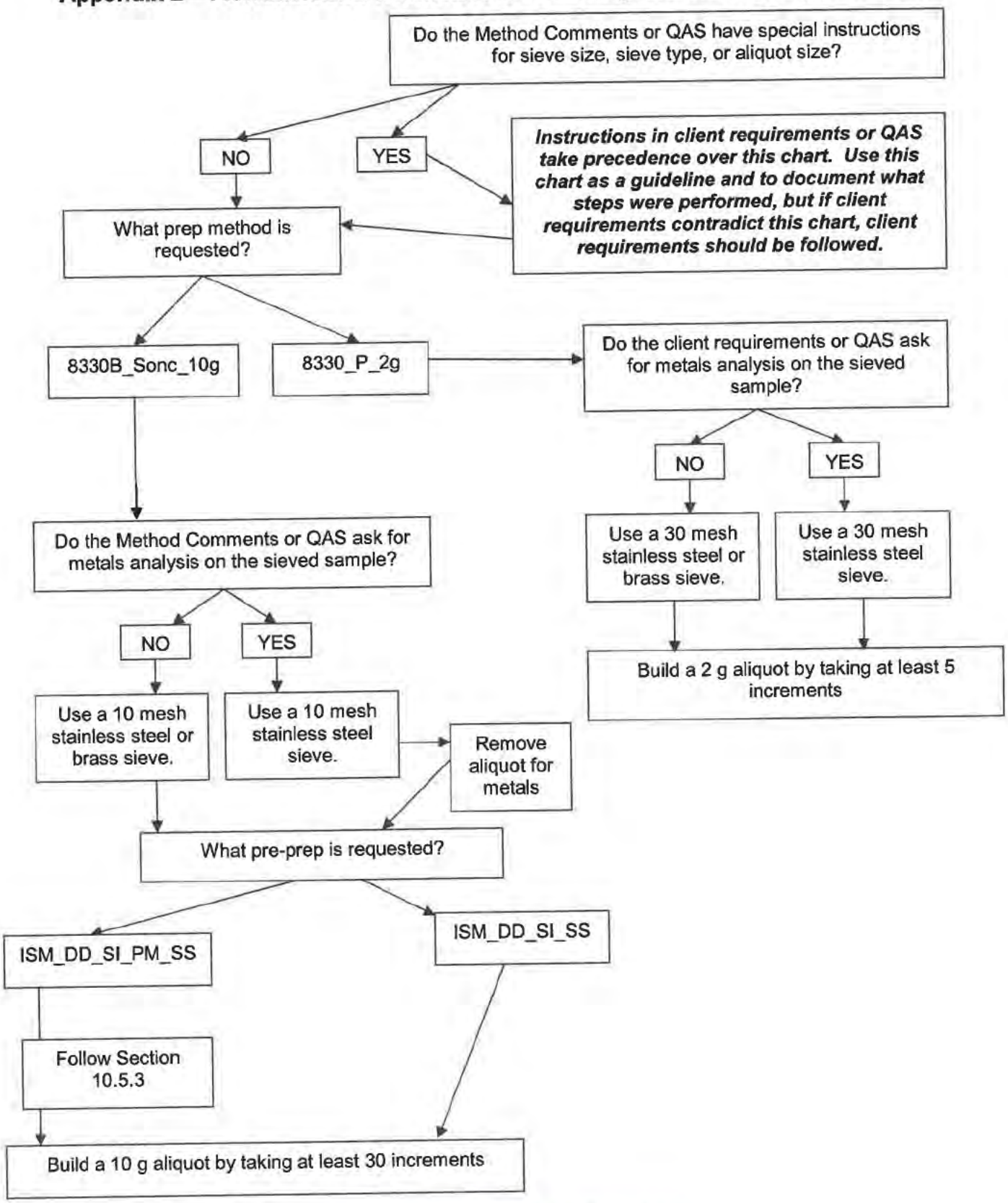
Table 2. Grinding LCS Bulk Material

Compound	Concentration (µg/Kg)
4-Amino-2,6-dinitrotoluene	600
2-Amino-4,6-dinitrotoluene	600
1,3-Dinitrobenzene	600
2,4-Dinitrotoluene	600
2,6-Dinitrotoluene	600
HMX (Octahydro-1,3,5,7-TNTC)	600
1,3,5-Trinitrobenzene	600
Nitrobenzene	600
2-Nitrotoluene	600
3-Nitrotoluene	600
4-Nitrotoluene	600
RDX (Hexahydro-1,3,5-TNTriaz)	600
Tetryl (Methyl-2,4,6-TNPN)	600
Nitroglycerin (Trinitroglycerin)	600
Pentaerythritol tetranitrate (PETN)	600
2,4,6-Trinitrotoluene	600

Appendix 1 – Flowchart and Worksheet for Drying Explosive Soils



Appendix 2 – Flowchart and Worksheet for Grinding and Sieving Explosive Soils



Appendix 3

How to Batch:

ISM_DD_SI_PM_SS (Dry, Disaggregate, Sieve, Ring & Puck, Subsample)
ISM_DD_SI_SS (Dry, Disaggreage, Sieve, Subsample)
Dry_Sample (Dry, Sieve, 2g prep)
Dry_Grind (Dry, Sieve, 2g Prep)

Overview

These five pre-prep methods can be logged in for not just for samples for explosives by 8330A or 8330B and 8321A or 8321B, but also for samples for metals analysis, or perchlorate, or any other method where the client is asking the lab to dry, sieve, and possibly grind the sample before extraction or digestion.

If one sample is logged in for 8330B and 6010B and 6020B and 7471A and all of these methods have the pre-prep of ISM_DD_SI_SS, the sample will show up on the backlog 4 times, (once for each analytical method). This would happen if the client wants us to dry, sieve, and perform ISM for each of these methods.

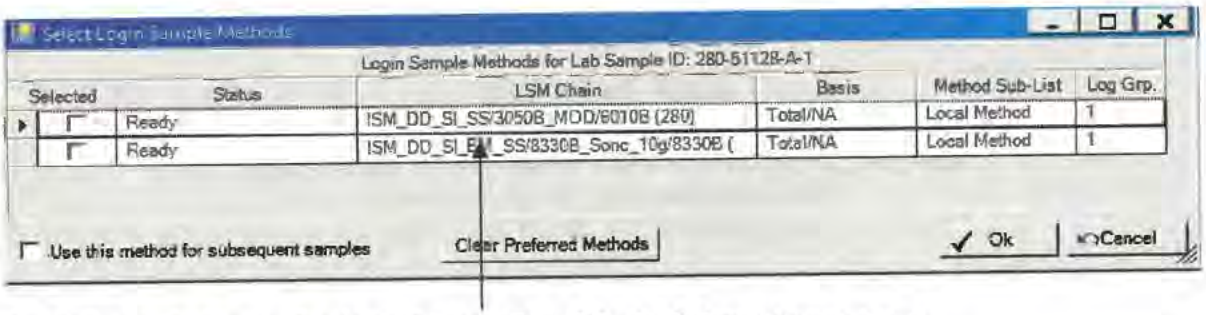
If one sample is logged in for 8330B with a pre-prep of ISM_DD_SI_PM_SS and the same sample is logged in for method 6010B with a pre-prep of ISM_DD_SI_SS, that means that the client wants us to dry, sieve the sample, perform ISM for method 6010B, then ring and puck and perform ISM for 8330B.

We will use a different status to indicate where the samples are.

- A status of "Batched" means the samples have been laid out to dry.
- A status of "Scheduled" on ISM_DD_SI_PM_SS means the samples have been laid out to dry, but possibly need ISM performed before grinding.
- A status of "Partial" means the samples have been sieved.
- A status of "2nd Level Review" on methods ISM_DD_SI_SS, Dry_Sample, or Dry_Grind means that the aliquots have been taken and someone has checked your work.
- A status of "2nd Level Review" on ISM_DD_SI_PM_SS means samples have completed the grinding.

Steps for Samples logged for <u>both</u> ISM_DD_SI_SS <u>and</u> Ring & Puck

1. Run the Dry/Sieve/Grind/ISM backlog. This backlog will only have samples that are logged in for these five pre-preps. This backlog is sorted by sample ID so that if a sample is logged in for ISM_DD_SI_SS for metals and ISM_DD_SI_PM_SS for explosives, you can easily see that the sample needs both preps.
2. Batch the samples under the ISM_DD_SI_SS method.
   NOTE: Do not put samples in the same batch that require different sieve sizes.
3. Scan your samples into the batch. A window will appear called "Select Login Sample Methods".



4. Select only the methods that have ISM_DD_SI_SS as the pre-prep.
    a. You can only batch ISM samples under a ISM_DD_SI_SS batch.
    b. You can only batch ring and puck samples under a ISM_DD_SI_PM_SS batch.
    c. You can batch "Dry_Grind" and "Dry Sample" samples under the same batch.
5. Save the batch and print the benchsheet.
6. Print labels. Print one label for each method logged on each sample. Write on the label the analytical method.
7. Lay out the samples to dry. Place all of the labels on the sample tray for the sample. Also label the tray "Grinding Needed" if the sample is logged for Ring and Puck.
8. Set the Ring and Puck Methods to "Scheduled" in the backlog. Do not batch them at this time.
9. Sieve the samples and take the required ISM aliquots. Whenever possible, aliquot the samples directly in the digestion cup for metals, or microwave tube or beaker for organics and record the aliquot masses in the ISM worksheet in Appendix 4. Place the ISM aliquots on the tray with the sample so a 2nd analyst can perform a label check. Document these steps on the TALS batch sheet
10. The Notes field in the Worksheets tab can be used to document if there was rocks or vegetation that did not go thru the sieve. Write NCMs for any samples that contained rocks or vegetation that was removed from the sample.
11. In the Worksheet tab, you can record the weight of the sample before and after drying and the weight of the sample that went through the sieve and the weight of the sample that did not go through the sieve. These measurements are not normally required, so they only need to be performed if client requested.
12. Have a 2nd analyst review the ISM_DD_SI_SS batch to ensure all required ISM aliquots have been performed. Take the ISM_DD_SI_SS batch to 2nd level review.
13. Now the samples are ready to be ground by Ring and Puck.
14. As the samples are ground, add them to the ISM_DD_SI_PM_SS batch.
15. As you add samples to the grinding batch, watch the LSM window to ensure that all ISM_DD_SI_SS methods are at 2nd level review. If there are ISM_DD_SI_SS methods that are not at 2nd level review, perform ISM on the sample for the requested methods before grinding the sample.
16. Take the samples to 2nd level review.
17. Return all empty containers to the walk-in refrigerator using ICOC. Any left-over dried and ground material is stored in the walk-in refrigerator on the same shelf as the original client containers.

Steps for Samples logged for Ring & Puck ONLY. No ISM_DD_SI_SS methods logged.

1. Run the Dry/Sieve/Grind/ISM backlog. This backlog will only have samples that are logged in for these five pre-preps. This backlog is sorted by sample ID so that if a sample is logged in for ISM_DD_SI_SS for metals and ISM_DD_SI_PM_SS for explosives, you can easily see that the sample needs both preps.

2. Pull the samples from the walk-in cooler and take custody of the samples. Take note of what shelf the sample came from.
3. Batch the samples and print out labels. Then remove the samples from the batch to place them back on the backlog.
4. Lay the samples out on parchment or foil. Label each tray with the sample ID and the grinding method (Ring & Puck)
5. Document the date and time the samples were laid out to dry. Document if the samples were laid out on parchment or foil. Document that a label check was performed.
6. Set the samples to Scheduled in the backlog to show that they are laid out to dry.
7. Once the samples are dry enough to sieve, sieve the samples and document what sieve size on the Explosive Review Checklist.
8. Open the batch with a grinding LCS. As the samples are ground, add them back to the original ISM_DD_SI_PM_SS batch. There can only be 20 field samples in each batch,. Ring and puck batches can be open for up to 3 days.
9. As you add samples to the grinding batch, watch the LSM window to ensure that the samples do NOT require any non-ground aliquots.
10. Take the samples to 2nd level review.
11. Return all empty containers to the walk-in refrigerator using ICOC. Any left-over dried and ground material is stored in the walk-in refrigerator on the same shelf as the original client containers.

Steps for Samples logged for <u>only</u> ISM_DD_SI_SS, Dry_Grind, or Dry_Sample. No grinding methods logged.

1. Run the Dry/Sieve/Grind/ISM backlog. This backlog will only have samples that are logged in for these five pre-preps. This backlog is sorted by sample ID so that if a sample is logged in for ISM_DD_SI_SS for metals and ISM_DD_SI_PM_SS for explosives, you can easily see that the sample needs both preps.
2. Batch the samples under the pre-prep method logged. Do not put samples in the same batch that are logged for different pre-prep methods. Do not put samples in the same batch that require different sieve sizes.
3. Scan your samples into the batch. If your samples are logged in for more than one of these three methods, a window will appear called "Select Login Sample Methods".
4. Select only the methods that have ISM_DD_SI_SS as the pre-prep.
    a. You can only batch ISM samples under a ISM_DD_SI_SS batch.
    b. You can batch "Dry_Grind" and "Dry Sample" samples under the same batch.

   If the LSM window shows methods with pre-preps of ISM_DD_SI_PM_SS or ISM_DD_SI_BM_SS, then stop and follow the instructions above under the header "Steps for Samples logged for <u>both</u> ISM_DD_SI_PM_SS and Ring & Puck.
5. Save the batch and print the benchsheet.
6. Print labels. Print one label for each method logged on each sample. Write on the label the analytical method.
7. Lay out the samples to dry. Place all of the labels on the sample tray for the sample.
8. Sieve the samples and take the required aliquots. Whenever possible, aliquot the samples directly in the digestion cup for metals, or microwave tube or beaker for organics and record the aliquot masses in the ISM worksheet in Appendix 4. Place the aliquots on the tray with the sample so a 2nd analyst can perform a label check.
9. The Notes field in the Worksheets tab can be used to document if there was rocks or vegetation that did not go thru the sieve. Write NCMs for any samples that contained rocks or vegetation that was removed from the sample.
10. In the Worksheet tab, you can record the weight of the sample before and after drying and the weight of the sample that went through the sieve and the weight of the sample that did not go

through the sieve. These measurements are not normally required, so they only need to be performed if client requested.

11. Have a 2nd analyst review the batch to ensure all required aliquots have been performed. Take the batch to 2nd level review.
12. Return all empty containers to the walk-in refrigerator using ICOC. Any left-over dried and ground material is stored in the walk-in refrigerator on the same shelf as the original client containers.

Appendix 4

ISM Worksheet

G:/QA/Edit/FORMS/Organic Prep Forms/MASTER ISM Spreadsheet_Rev1

ISM BATCH:

Use this spreadsheet to document alquot weights when aliquotting into digestion or extraction vessels. If aliquoting into a temporary vessel, no need to document the exact weight because the sample aliquot will be transferred and weighed at the time of analysis.

Login	Sample	Method --->											
			(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
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Appendix 5

ISM Constant Weight Worksheet

Located: \\tafs\Lab2\Denver\Admin\QA\Edit\FORMS\Organic Prep Forms

DV-F-0070-10-03-2017-Rev 0

ISM Batch Number:

Use this spreadsheet to document drying samples to a constant weight.

			Analyst Initial / Date/time 1	Analyst Initial / Date/time 2	Dry?		Analyst Initial / Date/time 3	Dry?	
Login	Sample	Tare weight (weight of weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 1, 2	Proceed with ISM?	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 2, 3	Proceed with ISM?
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
					#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!
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10/3/2017

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Title: ACID DIGESTION OF SOLIDS [Method EPA 3050B]

Approvals (Signature/Date):

Doug Gomer 3/2/20
Doug Gomer Date
Technical Specialist

Reed Pottruff 3/2/2020
Reed Pottruff Date
Health & Safety Manager / Coordinator

Roxanne Sullivan 3/2/20
Roxanne Sullivan Date
Quality Assurance Manager

Scott Hall 3/2/20
Scott Hall Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 This is a strong acid digestion procedure for the preparation of sediments, sludge, soils, and other types of solid materials by EPA Method 3050B for analysis by inductively coupled plasma atomic emission spectroscopy (ICP) or inductively coupled plasma-mass spectrometry (ICP/MS).

1.2 Method 3050B is designed to determine the concentration of "environmentally available" metals, and is not a true "total metals" digestion (see discussion below). The procedure is used primarily for hazardous waste characterization and other Resource Conservation and Recovery Act (RCRA) compliance testing.

1.3 The elements approved for Method 3050B are shown in Table I. The source method also mentions that other elements may be prepared by the method if the quality control requirements are met. The complete list of elements routinely included in this procedure by TestAmerica Denver is shown in Table II.

1.4 If sample preparation utilizing the Incremental Sampling Method is required, see SOP DV-OP-0013 for the procedure required prior to acid digestion for metals incorporating this procedure.

2.0 Summary of Method

A representative 1 to 2 gram portion of sample is digested with two cycles of nitric acid additions, followed by hydrogen peroxide digestion. For ICP analysis, the sample is also refluxed with hydrochloric acid. The resulting solution is filtered and diluted to 100 mL with reagent water. For the Incremental Sampling Method, 10 g of sample is used and brought to a final volume of 500 ml.

3.0 Definitions

3.1 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

3.2 **Total Metals -** Although Method 3050B is often referred to as a "total metals" digestion, it is important to understand that there are many compounds formed from these elements that are not efficiently dissolved using this digestion procedure. It is more accurately termed a strong acid digestion procedure. The limitations are discussed further in Section 4 (Interferences) below. The method itself states, "This method is not a total digestion technique for most samples." There are a variety of total digestion procedures used for metal assay, geochemical analysis, etc., that involve more vigorous digestions than 3050B.

3.3 **Preparation Batch -** A group of up to 20 samples that are of the same matrix and are processed together using the same lots of reagents and standards. The minimum QC elements in a batch are outlined in Section 9.

3.4 **Reagent Water –** Water that is free of the analytes of interest. In the Metals group, reagent water is obtained from a Barnstead E-Pure water purification system.

3.5 Other quality control terminology used in this procedure is based on SW-846, and is defined in the glossary section of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*.

4.0 Interferences

4.1 There are common compounds formed by the elements of interest (e.g., barium sulfate, beryllium oxide, silicon dioxide, crystalline silicates, titanium dioxide, etc.) that are not efficiently dissolved using this EPA approved procedure.

4.2 Silicon or silica are occasionally requested as part of the Method 3050B digestion. However, this digestion will include only acid-soluble silicon, and will not dissolve crystalline silica. The analysis is for silicon, but the final result is sometimes expressed as silica rather than silicon.

4.3 Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP-MS procedure that includes HCl as the final digestion acid. See Section 11.12 of this SOP.

4.4 Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., powdered gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. See Attachment 1 for more information regarding contaminant control.

4.5 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

4.6 For critical low-level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.7 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrix materials may not be digested using these methods if they are not soluble in acids. If physical interferences are present, they should be documented.

4.8 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals or conversion of metals to insoluble forms. For example, antimony is easily lost by volatilization from hydrochloric media. If this occurs the sample must be re-prepared.

4.9 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.10 Samples Requiring Additional Digestion Reagents

A few examples of types of samples that might require additional digestion reagents follow. It is very important to note situations where samples are not

behaving normally. However, do not assume that adding additional reagents will be acceptable for the project, even if it is obvious that the digestion will be incomplete without it. The situation must be discussed with the project manager and documented in a Nonconformance Memo (NCM), whether or not the variations suggested in the following examples are approved.

4.10.1 Samples with high organic content may require additional nitric acid and/or hydrogen peroxide for a thorough digestion, but these oxidizing reagents should be added very carefully to avoid violent reactions.

4.10.2 Samples with high concentrations of metal in the elemental form or refractory oxides may require additional hydrochloric acid for a thorough digestion. As an example, blasting sand used to remove paint from the hull of ships typically consists of 30% cupric oxide. Following 3050B exactly will produce results as low as 0.1% without additional hydrochloric acid. Increasing the volume of hydrochloric acid can produce results approaching the true copper concentration. Samples that appear to have nonstandard matrices or have visible metal particles should be documented in an NCM.

4.10.3 Highly alkaline materials may require larger volumes of acid than specified in this procedure.

4.10.4 If the use of extra digestion reagents is approved, the same volume of reagents must be added to all field samples and QC samples in the batch. Usually the method blank results will not be elevated. To ensure that the QC sample results accurately reflect sample results, the QC samples must be treated exactly like the samples.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Samples that contain high concentrations of carbonates or organic materials or samples that are at elevated pH can react violently when acids are added. If any solid sample appears to be a chemical substance rather than an environmental sample, consult with the group supervisor or the Project Manager (PM) before adding acid.

5.3.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and

reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Hydrogen Peroxide, H_2O_2	Oxidizer Corrosive Poison	1 ppm TWA 1.4 mg/m^3 TWA 75 ppm IDLH	Contact with other materials may cause fire. Eye contact may result in permanent eye damage. Causes eye and skin burns. Corrosive: May cause severe respiratory tract irritation. Harmful if swallowed, may cause digestive tract irritation or burns.
Nitric Acid, HNO_3	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid, HCl	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

(1) Always add acid to water to prevent violent reactions.

(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Instrumentation

6.1.1 Top-loading balance capable of accurately weighing to the nearest 0.01 grams.

Note: Balances are serviced annually and the accuracy checked daily using three standard masses. See SOP DV-QA-0014 for details.

6.1.2 Digestion "Hot Block" or equivalent heating device capable of maintaining a temperature of 90-95 °C. The Hot Block temperature must be monitored separately for each unit. The temperature of each Hot Block is checked by placing a calibrated thermometer through a cap on a digestion tube that is partially filled with water. The water in the tube must be high enough to cover the thermometer past the minimum immersion line. The temperature is directly recorded in the Batch Information area in the TestAmerica LIMS (TALS).

6.2 Supplies

6.2.1 Thermometers (non-mercury liquid filled or digital) that cover a temperature range including 80-110 °C with clearly visible 1 °C increments.

Note: Thermometers are calibrated before use and periodically as described in SOP DV-QA-0001.

6.2.2 Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use. See SOP DV-QA-0008.

6.2.3 Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

6.2.4 Whatman 541 (acid washed) filter paper, or equivalent.

6.2.5 Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters, No. 6973-2504, for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

6.2.6 Syringes or equivalent filtration apparatus.

6.2.7 Disposable plastic funnels.

6.2.8 Disposable wooden spatulas for subsampling.

6.2.9 Centrifuge, capable of at least 2,000 rpm.

6.2.10 Graduated cylinders, 100 mL and 500 mL, capable of ± 3% accuracy.

6.2.11 Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

Note: Mechanical pipettes are calibrated before use as described in SOP DV-QA-0008.

6.2.12 Class A volumetric flasks.

6.2.13 pH indicator strips (pH range 0 – 6).

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

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.

7.1 Reagent water – Millipore DI system or equivalent, 10-18.2 megohm-cm. See SOP DV-QA-0026 for daily water monitoring procedure.

7.2 Nitric acid (HNO_3), concentrated - Trace metal grade or better.

7.3 Nitric acid (HNO_3), 5% - Add 50 mL of concentrated HNO_3 to approximately 900 mL of reagent water and dilute to 1 liter.

7.4 Hydrochloric acid (HCl), concentrated - Trace metal grade or better.

7.5 30% Hydrogen peroxide (H_2O_2) - Reagent grade used for ICP analysis.

7.6 30% Hydrogen peroxide (H_2O_2) – Ultra pure used for ICP-MS analysis.

7.7 Glass beads, ≤ 1 mm diameter, washed with aqua regia (for DoD projects).

7.8 Standards

7.8.1 All standards must be NIST traceable. Unless purchased directly from NIST, the accuracy of each standard is verified before the initial use, as described in SOP DV-QA-0015.

7.8.2 Storage and Shelf Life of Metal Standards

7.8.2.1 Standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. They are stored at room temperature.

7.8.2.2 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.8.3 LCS and MS Spike Solutions

7.8.3.1 ICP and ICP/MS spike solutions are purchased as custom-made solutions from a commercial vendor at ready-to-use concentrations. No further dilutions are needed.

7.8.3.2 If a non-routine element is required that is not contained in the custom-made solution, single-element solutions from a commercial vendor may also be used.

7.8.3.3 Intermediate standards prepared in the laboratory may be used for spiking as long as the procedures for standard recording and verification outlined in SOP DV-QA-0015 are followed.

Typical LCS and MS/MSD spike standard concentrations are shown below.Analysis	Standard	Elements	Conc. (mg/L)
ICP	Spike Mix #1	As, Se, Si, Sn, Tl Ba, Be, Cd, Co, Cr, Li, Mn, Mo, Ni, Pb, Sr, Ti, V	200 100
ICP	Spike Mix #2	Ca, K, Mg, Na Al, Fe	5000 1000
ICP	Spike Mix # 3	P S U W	2000 1000 200 100
ICP	Spike Mix # 4	Bi B, Th Zn, Zr	200 100 50

Typical LCS and MS/MSD spike standard concentrations are shown below.Analysis	Standard	Elements	Conc. (mg/L)
ICP	Spike Mix # 4B	Ag	5
ICP	Spike Mix # 5	Sb	100
ICP-MS	MS CALSTD-1	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn Sr, Ti, Tl, V	100
ICP-MS	ICP CALSTD-2	Al, Ca, Fe, K, Mg, Na	2000
ICP-MS	MS Spike 2	Ag, Sr, Th, U, W, Zn	20

Note: ICP or ICP-MS digestions may select different combinations of spikes in order to satisfy client requests. All spikes used for sample digestion will be recorded in the Reagent module in TALS.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2 Soil samples do not require chemical preservation, but are stored at ≤6 °C until the time of analysis.

Matrix	Sample Container	Min. Sample Size	Preservation[1]	Holding Time[2]	Reference
Soils	Glass	3 grams	Cool ≤ 6 °C	180 Days	N/A

[1] Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration. Samples which will be used to aliquot for both analyses must be refrigerated.

[2] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Preparation batches may consist of up to 20 field samples. Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.3 Minimum QC Requirements

Each preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. Note that some programs require an unspiked duplicate sample in place of or in addition to the duplicate matrix spike. Be sure to check special instructions in TALS. If clients specify specific samples for the MS and MSD, the batch may contain multiple MS/MSD pairs.

9.3.1 Method Blank (MB)

One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. Soil method blanks are prepared by taking 5 mL or 5 g of reagent water through the procedure described in Section 11. Add 1.0 g of prewashed glass beads to the blank if required by the client to better simulate a solid matrix.

The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

Acceptance Criteria: Criteria for the acceptance of blanks are contained within the individual analytical method SOPs.

Corrective Action: If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be re-digested and reanalyzed.

9.3.2 Laboratory Control Sample (LCS)

One aqueous LCS must be processed with each preparation batch. The LCS contains reagent water that is spiked with all the analytes of interest and is carried through the entire analytical procedure. A duplicate LCS (LCSD) must be prepared when there is insufficient sample volume to perform an MS/MSD. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Add 1.0 g of prewashed glass beads to the LCS if required by the client to better simulate a solid matrix.

The spike solutions described in Section 7.8.3 are used to prepare LCSs as follows:

- Routine ICP: Add 1.0 mL of spike
- DoD ICP: Add 1.0 mL of spike to 1.0 g of glass beads
- Routine ICP-MS: Add 1.0 mL of spike
- DoD ICP-MS: Add 1.0 mL of spike to 1.0 g of glass beads

The resulting spike concentrations for each element are given in Table 2 and Table 3.

Incremental Sampling Method LCSs are spiked with 5 ml of spike.

Acceptance Criteria: Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs.

Corrective Action: When LCS results fail to meet control limits, the LCS and all associated samples in the batch must be re-prepared and reanalyzed.

9.3.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a second aliquot of a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a third aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Samples identified as field blanks cannot be used for MS/MSD analysis. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process.

The spike solution described in Section 7.7.3 is also used to prepare matrix spikes, as follows:

- ICP: Add 1.0 mL of spike
- ICP-MS: Add 1.0 mL of spike

The resulting spike concentrations for each element are given in Tables II through IV. Incremental Sampling Method MS/MSD pairs are spiked with 5 ml of spike.

NOTE 1: The spike must be added after the sample aliquot but before any reagents.

NOTE 2: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD pair is not available, an LCS and LCSD are used to measure precision.

10.0 Calibration

Not applicable. This SOP addresses sample preparation only for subsequent ICP or ICP/MS analysis. Calibration of the measurement system is covered in the SOPs for the determinative methods.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed

must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Custody

11.3.1 Samples are transferred from the Sample Control group to the Metals group and the transfer is documented using the Sample Transfer function of the Internal Chain of Custody in TALS (see SOP DV-QA-0003 for details).

11.3.2 Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be done in a manner to ensure connection with the proper sample.

11.4 Subsampling

11.4.1 It is not acceptable to simply collect 1.0 g off of the top of the sample. Samples must be mixed and incrementally subsampled to obtain a representative portion. At a minimum, mix by stirring with a disposable wooden spatula. If there is insufficient room in the sample container to allow for proper mixing, refer to SOP DV-QA-0023, *Subsampling*, for directions.

11.4.2 Select at least three incremental subsamples from different locations in the original sample and place them in a tared 50 mL digestion tube. The final sample weight should be between 1.0 and 1.5 g. Record the weight to the nearest 0.01 g.

11.4.3 Measure additional aliquots for QC samples required in the batch and spike as required (see Section 9 for details).

NOTE: When adding glass beads to the Method Blank and LCS digestion tubes, the nominal weight must be entered into the Initial Amount field in TALS. The true weight of glass beads should be recorded in the Notes field on the Worksheet tab in the preparation batch.

11.5 Incremental Sampling Method Digestion

For the Incremental Sampling Method approximately 10 g of sample is weighed out by the Organic Prep group following the procedure described in SOP DV-OP-0013. This pre-weighed sample is then delivered to the Metals group for digestion and analysis. The sample weight is recorded on the ISM Worksheet and attached to the incremental sampling batch in TALS. The pre-weighed aliquots are delivered in 125 mL digestion tubes which are ready for spike standards and reagents to be added. The Method 3050B digestion reagents are increased 5x to maintain the same proportions as are used for a 1-2 gram sample. When required, 10 g of glass beads are added to the Method Blank and LCS prior to digestion.

11.6 Initial Digestion Cycle with 1:1 Nitric Acid

11.6.1 Add approximately 5 mL of reagent water to each digestion tube.

11.6.2 Add 5 mL of concentrated HNO_3.

11.6.3 After all of the acid has been added to the preparation batch, gently swirl the samples to mix and then place the sample rack on the Hot Block.

11.6.4 Place a ribbed cover on each tube.

11.6.5 Heat samples to 90-95 °C, and reflux for 15 minutes without boiling.

NOTE: **DO NOT ALLOW SAMPLES TO BOIL OR GO DRY** during any part of the digestion. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.6.6 Remove the samples from the Hot Block and allow them to cool before proceeding with the next step.

11.6.7 Record the start time, starting temperature, end time, and ending temperature in TALS.

11.7 Second Digestion Cycle Using Concentrated Nitric Acid

11.7.1 Add 5 mL of concentrated HNO_3, and replace the ribbed cover.

11.7.2 Place samples back on the Hot Block and reflux at 90-95 °C for 30 minutes. Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.

11.7.3 If brown fumes are observed, this means that material in the sample is actively being oxidized. There may not be enough HNO_3 acid to complete the oxidation, and there could be violent reaction of the sample with peroxide in the third digestion step. For that reason, it is necessary to repeat the previous two steps until no more fumes are evolved.

11.7.4 Heat the samples at 90-95 °C for 2 hours.

11.7.5 Allow the samples to thoroughly cool before proceeding.

11.8 Third Digestion Cycle Using Hydrogen Peroxide

11.8.1 Add 2 mL of reagent water to each tube.

11.8.2 Add 3 mL of 30% H_2O_2 a few drops at a time. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.8.3 Replace the ribbed cover and heat samples until effervescence subsides.

11.8.4 Allow the samples to cool.

11.8.5 Continue adding 30% H_2O_2 in 1 mL increments with warming until effervescence is minimal or sample appearance is unchanged. If additional peroxide is added to a sample then it must also be added to the method blank and LCS.

NOTE: Do not add more than a total of 10 mL of 30% H_2O_2. If 10 mL have been added and the samples are still vigorously effervescing, document the situation with an NCM and continue with the digestion.

11.8.6 Heat the samples at 90-95 °C for 2 hours.

11.8.7 Allow the samples to cool.

11.8.8 If samples will be analyzed by ICP, continue on with the fourth digestion step using HCl in Section 11.8. If the samples will be analyzed by ICP-MS, skip the HCl digestion step and go to step 11.10.

11.9 Fourth Digestion Cycle for ICP Using Concentrated Hydrochloric Acid

11.9.1 If the samples are being prepared for ICP analysis, add 10 mL of concentrated HCl to the samples in the digestion tubes and cover with ribbed covers.

11.9.2 Reflux for an additional 15 minutes without boiling.

11.9.3 Allow the samples to cool.

11.10 Separating Undigested Solids from the Digestion Solution

11.10.1 Filter samples through Whatman 541 or equivalent fiber filters into a graduated 125 mL digestion tube whose accuracy is documented to be better than ± 3%.

NOTE: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.10.2 For samples digested by the Incremental Sampling Method use a 500 mL poly bottle that has been measured after measuring out 500 mL of DI water from a graduated cylinder.

11.10.3 Wash the original digestion tube and ribbed cover with reagent water to ensure quantitative transfer of all of the digestion solution into the new digestion tube.

11.10.4 Rinse the funnel and filter paper with reagent water to ensure complete sample transfer into the new digestion tube.

11.10.5 Re-volume sample to 100 mL with reagent water. This must be done volumetrically, rather than by weight. Record the final volume in TALS.

For Multi-Incremental samples the final volume is 500 mL.

11.11 Documentation and Record Management

The following information must be recorded for each preparation batch. This information is directly entered into TALS.

- Initial sample weight and final digestion volume
- Preparation analyst and date
- Identification of all reagents and standards
- Identification of all measuring and test equipment used (e.g., balances, thermometers, pipettes)
- Glass beads lot number
- Filter paper lot number
- Digestion tube lot number
- Hot Block ID number
- Fume Hood ID number

11.12 Alternate Antimony Preparation for Analysis by ICP-MS

11.12.1 Weigh out 1.0-1.5 g soil samples according to the procedure in Section 11.3.

11.12.2 Add approximately 5 mL of reagent water to each digestion tube.

11.12.3 Spike the LCS, LCSD, MS, and MSD with 1.0 mL of the MS spike 2 standard.

11.12.4 Add 2.5 mL concentrated HNO_3 and 2.5 mL concentrated HCl to each sample and batch QC.

11.12.5 Cover each tube with a watch glass and reflux on hot block at 90-95 °C for 15 minutes.

11.12.6 Filter through Whatman 541 or equivalent filter paper into a new 125 mL digestion tube while still hot.

11.12.7 Rinse the filter and funnel with 1.25 ml of hot (~95 °C) concentrated HCl.

11.12.8 Rinse three times with hot (~95 °C) reagent water (5 mL rinses.)

11.12.9 Place the filter paper and soil residue back into the original sample

digestion vessel. Add 2.5 mL concentrated HCl, cover and reflux on the hot block for 20 minutes or until paper dissolves.

11.12.10 Filter through a fresh filter into the original filtrate. Rinse three times with reagent water (5 mL rinses).

11.12.11 Bring to final volume of 100 mL with reagent water.

12.0 Calculations / Data Reduction

Not applicable. Calculations of final results are described in the determinative analytical SOPs.

13.0 Method Performance

13.1 Method Detection Limit (MDL)

An MDL must be determined for each analyte/matrix prior to the analysis of any samples. See the SOPs for the determinative analysis methods for details.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.3 Training Requirements

The group leader or supervisor is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the

required experience. A new analyst must be working under supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered in the batch record (View Batch Information)until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Program*.

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Aqueous Acidic (Metals) - Corrosive – Waste Stream J

15.2.2 Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996; Method 3050B.

16.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

16.3 Department of Defense Quality Systems Manual for Environmental Laboratories Version 5.0, July 2013.

17.0 Method Modifications:

Item	Method	Modification

1	3050B	Method 3050B Section 7.1 states that a 1-2 g aliquot is to be used. The amount specified by TestAmerica Denver in this procedure is limited to 1-1.5 g in order to prevent increased instrument maintenance and sample reruns due to dilutions.

18.0 Figures, Tables, and Attachments

Table 1: Method 3050B Approved Analyte List for ICP/ICP-MS

Table 2: Soil LCS and MS/MSD Spikes for ICP

Table 3: Soil LCS and MS/MSD Spikes for ICP-MS

Attachment 1: Contamination Control Guidelines

19.0 Revision History

- Revision 14 dated 3 March 2020
  - Updated copyright information
- Revision 13 dated 31 January 2020
  - Annual Review
  - Updated spiking standard concentrations in Sections 7.8.3.3, Tables 2 and 3
- Revision 12 dated 31 October 2019
  - Annual Review
- Revision 11 dated 31 October 2018
  - Annual Review
  - Updated copyright information
- Revision 10 dated 31 October 2017
  - Annual Review
- Revision 9 dated 31 October 2016
  - Annual Review
  - Update the temperature heating range to 90-95°C where stated in the SOP
  - Removed the reference to AFCEE throughout SOP
  - Added current section 3.1 – reference to QAM for general definitions
  - Restructured and renumbered section 6.0
  - Added initial paragraph to section 6.0 regarding the documentation of equipment IDs
  - Revised the current sections 6.1 and 6.2 to reflect consistent verbiage and formatting as other SOPs
  - Added current section 7.6 Ultra Pure Peroxide reference
  - Added current footnote 1 to the section 8 table regarding soil preservation
  - Re-numbered previous footnote 1 to be footnote 2 to the section 8 table

- Updated section 9.1 and subsections to reflect current practices and verbiage
- Re-numbered Notes in section 9.3.3 to be Note 1 and Note 2
- Added LCSD required when an MS/MSD is not available to sections 9.3.2 and 9.3.3 Note 2
- Renumbered and updated section 11.1 and 11.2 to reflect current practices and verbiage
- Added current section 11.2
- Updated section 13.2 to reflect current practices and verbiage
- Added Strontium to Table 3
- Removed Titanium and Zirconium from Table 3

- Revision 8 dated 31 October 2015
  - Annual Review
  - Edited Sections 9.5.1 and 9.5.2 to clarify glass bead requirement
  - Added definition of reagent water
  - Updated Section 11.6.4 and 11.7.6 to reflect current practice
  - Removed Method exception 1 regarding method blank limits as it no longer applies
  - Added detail to training requirements for new analysts Section 13.3
  - Added note to Section 9.5.3 regarding precision requirements
  - Added note to Section 11.3 regarding recording of glass bead weights

- Revision 7 dated 31 March 2015
  - Annual Review
  - In Section 11.7.8 the section referenced was updated to 11.8
  - Updated spike standard name to MS spike 2 in Section 11.11.3
  - Formatting and grammar corrections throughout
  - Section 6.4 removed reference to calibrating digestion tubes
  - Section 6.6 changed name of filter paper to match current practice
  - Section 6.14 added to define computer systems used
  - Sections 7.7.3.1 and 7.7.3.2 combined
  - New Sections 7.7.3.2 - 7.7.3.4 added to define spikes used
  - Table of spike names and concentrations added to Section 7.7.3.4
  - Changed LIMS to TALS throughout
  - Section 8.2 changed storage temperature to ≤6 °C
  - Deleted Section 9.3, duplicated in 13.2
  - Added new Section 9.3 to address federal requirements
  - Rewrote Section 9.5
  - Changed Sections 9.6 – 9.8 to be subsections of the new 9.5
  - Rewrote Section 11.2.1
  - Removed method modification 2 because it referred to the analytical SOP
  - Created new method modification 2 explaining the 1-1.5 g sample aliquot
  - Section 11.3.2 changed required sample aliquot to 1-1.5 g to help avoid targeting
  - Rewrote Section 11.4 to define and explain the Incremental Sampling Method
  - Added new Section 11.5.3 to explain sample mixing
  - Section 11.7.5 added language to note regarding samples that require more than 10 mL of H2O2
  - Added detail into Sections 11.9.1 – 11.9.5
  - Folded Section 11.10.1 into 11.10

- Rewrote list of data to be entered into TALS in Section 11.10
- Rewrote Section 13.2 to match boilerplate
- Deleted flowcharts Figures 1 and 2
- Corrected element list in Table 2

- Revision 6 dated 31 March 2014
  - Annual Review
  - Formatting changes throughout document
  - Added to Section 11.7.5 to add additional peroxide to QC if added to samples
  - Updated section number in text to 11.8 in section 11.7.8
  - Added references for DoD QSM
  - Removed Attachment 2

- Revision 5 dated 04 March 2013
  - Section 7.7.3.1 Added DoD to the glass beads requirement
  - Section 11.11.2 Added that 5ml of water is added to the samples
  - Section 11.11.3 Changed spike name to 200.8 Cal-2
  - Updated spike level to 1.0ml in Table 3
  - Updated work instructions to current revision.
  - Formatting changes throughout document

- Revision 4 dated 3 February 2012
  - Changed references of Multi-Incremental Sampling to Incremental Sampling Method throughout document
  - Section 2.0 Added reference to Incremental Sampling Method
  - Section 6.4 Added 50 mL digestion tubes
  - Added introductory statement to section 7.0 regarding reagent purity
  - Section 7.1 Updated acceptable criteria for the reagent water
  - Section 9.7.2 Added LCS Incremental Sampling Method spike amounts
  - Section 9.8.2 Added MS/MSD Incremental Sampling Method spike amounts
  - Section 11.4 Updated sample amount for Incremental Sampling Method to 1 10g aliquot
  - Section 11.9 Added Incremental Sampling Method final volume

- Revision 3.5, dated 24 August 2011
  - A note has been added to section 9.8.3 for the addition of the LCS/MS spike before reagents.

Earlier revision histories have been archived and are available upon request.

Table 1.

Method 3050B Approved Analyte List for ICP/ICP-MS

Element	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

Table 2.

Soil LCS and MS/MSD Spikes for ICP

ELEMENT	Stock Standard (mg/L)	Sample Spike (mg/kg)	Final Digested Solution (mg/L)
Aluminum	1,000	1,000	10.0
Antimony	100	100	1.0
Arsenic	200	200	2.0
Barium	200	200	2.0
Beryllium	100	100	1.0
Bismuth	200	200	2
Boron	100	100	1.0
Cadmium	100	100	1.0
Calcium	5,000	5,000	50
Chromium	100	100	1.0
Cobalt	100	100	1.0
Copper	100	100	1.0
Iron	1,000	1,000	10.0
Lead	100	100	1.0
Lithium	100	100	1.0
Magnesium	5,000	5,000	50
Manganese	100	100	1.0
Molybdenum	100	100	1.0
Nickel	100	100	1.0
Phosphorous	2,000	2,000	20
Potassium	5,000	5,000	50
Selenium	200	200	2.0
Silicon	200	200	2.0
Silver	5	5	0.05
Sodium	5,000	5,000	50
Strontium	100	100	1
Sulfur	1,000	1,000	10
Thallium	200	200	2
Thorium	100	100	1.0
Tin	200	200	2.0
Titanium	100	100	1.0
Uranium	200	200	2.0
Vanadium	100	100	1.0
Zinc	50	50	0.50
Zirconium	50	50	0.50

NOTE: Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

Table 3.

Soil LCS and MS/MSD Spikes for ICP-MS

ELEMENT	Stock Standard (mg/L)	Sample Spike (mg/kg)	Final Digested Solution (µg/L)
Aluminum	400	400	4,000
Antimony	20	20	200
Arsenic	20	20	200
Barium	20	20	200
Beryllium	20	20	200
Cadmium	20	20	200
Chromium	20	20	200
Cobalt	20	20	200
Copper	20	20	200
Iron	400	400	4,000
Lead	20	20	200
Manganese	20	20	200
Molybdenum	20	20	200
Nickel	20	20	200
Selenium	20	20	200
Silver	20	20	200
Strontium	40	40	400
Thallium	20	20	200
Thorium	20	20	200
Tin	20	20	200
Tungsten	20	20	200
Uranium	20	20	200
Vanadium	20	20	200
Zinc	20	20	200

NOTE: Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

Attachment 1

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with 5% HNO_3 according to the procedure described in SOP DV-IP-0005.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered should not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

- Yellow pipette tips and volumetric caps can sometimes contain cadmium.
- Some sample cups have been found to contain lead or cobalt.
- New glassware can be a source of silica and boron.
- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Latex gloves contain over 500 ppb of zinc.

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1.0 Scope and Application

1.1 This is an electrometric procedure for measuring pH in solid samples. This method is applicable to the analysis of soils, sediments, sludges, or non-aqueous liquids. It does not apply to multiphase wastes where the aqueous phase constitutes more than 20% of the sample. See DV-WC-0031 *Manual and Automated pH* for the determination of pH in multiphase wastes.

1.2 A detection limit (MDL) for pH has not been defined, however, for reporting purposes this laboratory uses 0.1 pH units as the RL and MDL.

1.3 This method is applicable to all ranges of pH.

2.0 Summary of Method

The sample is mixed with reagent water. The pH meter, glass electrode, and reference electrode (or single combination electrode) are standardized against five reference buffer solutions of known pH bracketing the pH expected to be found in the sample. The sample measurement is made by immersing the electrodes into the sample solution and taking a reading from the meter.

3.0 Definitions

3.1 **pH** - At a given temperature, the intensity of the acidic or basic character of a solution is indicated by pH or hydrogen ion activity. Because of ionic interactions in all but very dilute solutions, it is necessary to use the “activity” of an ion and not its molar concentration. The use of the term pH assumes that the activity of the hydrogen ion is being considered. The approximate *equivalence* to molarity can be presumed only in very dilute solutions. A logarithmic scale is used to accommodate the wide range of ionic activities.

3.2 Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P *Quality Control Program* for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 The pH response of most glass electrodes is imperfect at both ends of the scale. The indicated pH value of highly alkaline solutions, as measured with the glass electrode, will be too low. The indicated pH value of salts and strong acids, which have a pH less than 1, will often be higher than the true pH value. Interferences can be minimized by the selection of the proper electrodes for these conditions. For example, sodium may interfere at pH > 10, and is controlled by using a “low sodium error” electrode.

4.2 Temperature fluctuations will cause measurement errors.

4.3 Coatings of oil and particulate matter may impair electrode response.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

Eye protection that satisfies ANSI Z87.1 (as per the Corporate Environmental Health and Safety Manual), laboratory coat, and nitrile or latex gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4 Primary Materials Used

There are no materials used in this method that have a serious or significant hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 pH Meter with temperature compensation ability. Details of the pH meter and electrode currently in use can be found at R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision).

6.1.2 Glass electrode with reference electrode. A calomel, silver-silver chloride or other reference electrode of constant potential may be used; or use a combination electrode that incorporates both measuring and reference functions.

6.1.3 Analytical balance capable of weighing to the nearest 0.1 gram. The balance is checked for accuracy each day it is used in accordance with SOP DV-QA-0014 *Selecting and Using Balances*.

6.1.4 Shaker table.

6.2 Supplies

6.2.1 40 dram vials with snap cap or a container large enough to hold sample and cover electrodes.

6.2.2 Glass wool, if oily wastes are to be tested.

6.2.3 50 mL graduated cylinder

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 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.

7.2 **Reagent water**: Water with a resistivity of 1 Megohm-cm or greater. The Eurofins TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

7.3 **pH Buffers: 2, 4, 7, 10 and 12:** Use commercially available solutions that have been validated by comparison to NIST standards. The solution aliquots used to calibrate the pH meter must be replenished each day of use.

7.4 **ICV Buffer Solution:** A pH 7 buffer solution from a second source provider, obtained commercially and traceable to NIST standards. The ICV solution aliquot used must be replenished each day of use.

7.5 **Laboratory Control Sample (LCS) Solution:** The LCS solution must be certified for pH and is commercially available. The ICV pH 7 buffer from Section 7.4 is normally used as the LCS. Due to the nature of pH determination, a solid matrix is not used.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation [1]	Holding Time [2,3]	Reference
Solid	4 oz. glass or plastic	50 g	Cool, ≤ 6 °C	None	SW-846

[1] 40 CFR Part 136.3 and SW-846 indicate no preservation is required for pH. It is intended by both programs that the samples be analyzed in the field. Eurofins TestAmerica Denver typically refrigerates these samples because the aliquot tested is taken from a sample container that is used for other tests that do require refrigeration.

[2] pH is intended to be a field measurement and samples are to be analyzed immediately per SW-846. The laboratory attempts to measure pH as soon as possible upon receipt. All laboratory analyzed samples are flagged as out of hold.

[3] Samples **must** be analyzed the same day that the extraction is performed.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the Eurofins TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver Policy DV-QA-003P *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver Policy DV-QA-024P *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM unless otherwise stated. Any deviation or exceptions from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 *Non-*

Conformance and Corrective Action System. This is in addition to the corrective actions described in the following sections.

9.2 Sample QC - The following quality control samples are prepared with each batch of samples.

9.2.1 Laboratory Control Sample (LCS/LCSD)

One LCS/LCSD is required with each batch of samples processed, not to exceed 20 samples. See Section 7.5.

Acceptance Criteria: The LCS must be within ± 0.05 pH units of the true value.

Corrective Action: If the LCS is not within the control limits, rerun all associated samples.

9.2.2 Duplicate Samples

One duplicate sample must be analyzed with each batch of samples processed not to exceed 20 samples.

Acceptance Criteria: The two results should agree within ± 0.10 pH units.

Corrective Action: If the difference is greater than ± 0.10 repeat the analysis. If the difference still exceeds the control limit the data will be flagged as outside of the limit.

9.2.3 Method blanks and matrix spikes are not applicable to pH.

9.3 Instrument QC

9.3.1 Initial Calibration Verification

Record the expected pH, manufacturer, and lot number of the verification buffer used for a second source pH 7.0 buffer solution. Analyze the second source pH 7 buffer solution.

Acceptance Criteria: The second source ICV buffer solution should read within ± 0.05 pH units of the true value.

Corrective Action: If this criterion is not met, the problem should be identified, corrected, and the meter recalibrated.

9.3.2 Low and High Calibration Verification

A pH 2.0 buffer and 12.0 buffer check is required after the ICV and prior to analytical samples. These buffers are the same pH buffer solutions used in the initial calibration.

Acceptance Criteria: The pH buffer checks must be within ± 0.05 units of the true value.

Corrective Action: If this criterion is not met, the problem should be identified, corrected, and the meter recalibrated.

9.3.3 Continuing Calibration Verification

A pH 7.0 buffer check is required after every 10 or fewer samples and at the end of the run. The CCV is the same pH 7 buffer solution used in the initial calibration.

Acceptance Criteria: The pH buffer checks must be within ± 0.05 units of the true value.

Corrective Action: If the pH 7.0 buffer check is outside of the control limits, rerun all samples since the last acceptable pH 7.0 buffer check.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 *Non-Conformance and Corrective Action System*. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective described.

10.3 Sample Preparation

10.3.1 Weigh a minimum of 40 g of sample into a 40 dram vial, add 40 mL reagent water using a graduated cylinder and cap the vial. A sample which is not a soil but another material may possibly react violently on the addition of water. In such cases, add the water to the solid in a hood. If the sample shows any signs of heat or gas evolution, do not cap the vial as pressure may build up

10.3.1.1 If the samples are oily, filter through glass wool to remove oil. Retain the aqueous phase for analysis.

10.3.1.2 If the sample is hygroscopic and absorbs all the reagent water, add an additional 20 mL of reagent water to the extraction vessel and mix to incorporate. Note the increased water volume on the benchsheet.

10.3.1.3 If the sample is a waste and additional water is needed, add an additional 40 mL of reagent water.

10.3.2 Mix on shaker table for 5 minutes.

10.3.3 Let the sample settle undisturbed for a minimum of one hour.

10.4 Calibration

10.4.1 Follow the operating instructions supplied by the manufacturer of the pH meter. See WI-DV-0034 *pH Meter Calibration* for more information concerning meter calibration.

10.4.2 Record the instrument ID, pH probe ID, thermometer probe ID, and reagent IDs in the batch record in TALS.

10.4.3 When using the Thermo Five Star pH meter, all results are to be temperature corrected to 25 °C using the Automatic Temperature Compensation function available with the instrument.

NOTE: Methods 9045C and 9045D state: "The sample temperatures must be within ± 2 °C of the calibrated buffers or temperature corrected." All samples are automatically corrected for temperature by the instrument.

10.4.4 Calibrate the pH meter using five buffers at pH 2.0 (Low Check), 4.0, 7.0, 10.0 and 12.0 (High Check). The buffers should be fresh for each day of use.

10.4.4.1 Pour a small amount of the buffer solutions into separate disposable medicine cups, adding enough solution to cover the bulb at the bottom of the pH electrode. Add a stirring bar to each cup and place on a stirring plate. Turn on the plate stir function at a low level.

10.4.4.2 Allow the pH to stabilize and record the reading in the "Initial Cal Reading" box in the pH probe calibration logbook.

10.4.4.3 Follow the instructions for calibration as described in WI-DV-0034 *pH Meter Calibration*.

10.4.4.4 After each calibration point has been saved, allow the probe to remain in the buffer until the pH reading stabilizes. Record this reading in the "Final Cal Reading" box in the pH probe calibration logbook before moving to the next buffer solution.

10.4.4.5 The reading of the buffer solutions must be within ± 0.05 pH units of the certified buffer solution values. If they are not, correct the reading to reflect the true pH of the buffer (ex. 1.68 changes to 2.00).

10.4.4.6 After the calibration is complete, record the slope in the instrument logbook. The source methods do not provide criteria for acceptance of the slope. The Orion 5 Star pH meter manual sets the acceptable slope range from 85% to 115%. If the slope falls outside this range, maintenance is required (see Sections 10.6 and 10.7).

10.4.4.7 Record the manufacturer and lot number of the buffers used in the pH probe calibration logbook. See Attachment 1.

10.4.5 Verify the calibration using a buffer solution (ICV). See Sections 7.4 and 9.3.1. Also verify with the Low and High Checks. See section 9.3.2.

10.4.5.1 Record the pH, manufacturer, and lot number of the verification buffer used.

10.4.5.2 The reading of the buffer solution should be within ± 0.05 pH units of the true value. If this criterion is not met, the problem should be identified, corrected, and the meter recalibrated.

NOTE: Internal standard calibration is not an appropriate technique for the determination of pH.

10.5 Sample Analysis

10.5.1 Samples must be analyzed on the day they are prepared.

10.5.2 Analyze one LCS and one sample duplicate per batch of 20 samples.

10.5.3 Insert the electrode into the aqueous layer just far enough to cover the electrode bulb and junction. Do not allow the electrode to come into direct contact with oil or soil.

10.5.4 Allow the reading to stabilize.

10.5.5 The pH reading, temperature, and time are recorded directly in TALS. See Attachment 2.

NOTE: Methods 9045C and 9045D require the sample temperature to be reported with each pH result. All sample temperatures are recorded on the instrument raw data. TALS reports the pH as pH adjusted to 25 °C to account for the temperature correction performed by the instrument.

10.5.6 Rinse the electrodes well between measurements.

10.5.7 A pH 7.0 buffer check (CCV) is required after every 10 or fewer samples (excluding the LCS/LCSD) and at the end of the run. See Section 9.3.3.

10.5.8 Record the balance ID, pH meter ID, the pH probe ID and the pH thermometer ID in the batch record.

10.5.9 Follow the instructions supplied with the electrodes for storage after use. Record daily maintenance in the pH Calibration and Maintenance Log. See Attachment 1.

10.6 Troubleshooting

10.6.1 Slow response or a wavering response is indicative of a dirty or oil-coated pH probe or that the probe is not properly connected to the meter. Samples high in dissolved CO_2 can cause the pH to change as the sample is stirred.

10.6.2 No temperature displayed may be a result of the temperature probe not being properly connected to the meter.

10.6.3 Using plastic disposable beakers and a magnetic stir plate and stir bar may generate static electricity that could affect stability. Turn off the stir plate, unplug and allow to sit for a few minutes.

10.7 Maintenance

10.7.1 Clean the electrode as needed following manufacturer's instructions.

10.7.2 See Section 20 of the Denver Quality Assurance Manual for maintenance procedures.

11.0 Calculations / Data Reduction

11.1 There are no calculations. This is a direct reading method. Data are manually entered in TALS at time of measurement.

11.2 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 *Data Review* for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

There is no MDL study for pH. The laboratory reports samples to the nearest 0.1 pH units and uses this increment as the MDL for reporting purposes.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration. The pH 7 Buffer solution (Section 7.5) is typically used.

12.2.2 The pH of each aliquot must be within 0.05 units of the true value.

12.2.3 If the analyte does not meet the acceptance criteria, the test must be repeated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Further details concerning demonstrations of proficiency are described in DV-QA-0024 *Training*.

12.3 Training Requirements

The group leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use, has the required experience, and has successfully analyzed initial demonstration samples (see SOP DV-QA-0024 *Training* for details).

13.0 Pollution Control

13.1 It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

13.2 This method does not contain any specific modifications that serve to prevent or minimize pollution.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P *Waste Management Plan*.

14.2 The following waste streams are produce when this method is carried out:

14.2.1 Acidic sample waste generated by the analysis – Aqueous Acidic (F).

14.2.2 Alkaline sample waste generated by the analysis – Aqueous Alkaline (E).

14.2.3 Exhausted soil samples utilized in the analysis – Soils (S)

14.2.4 Exhausted acidic and/or alkaline buffer solutions utilized in the analysis and expired standards and reagents – Contact the Waste Coordinator for guidance.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 9045D, "Soil and Waste pH", Revision 4, November 2004.

15.1.2 Method 9045C, "Soil and Waste pH", SW-846, Revision 3, 1995.

15.2 "Soil pH (Hydrogen Ion Activity)", Methods of Soil Analysis, Second Edition, American Society of Agronomy, 1982.

16.0 Method Modifications

Item	Method	Modification
1	9045C/9045D	Temperature is not reported with the pH result. Sample pH is reported as pH adj. to 25°C.
2	9045C/9045D	Sample size used in this SOP is 40 g solid sample to 40 mL reagent water. This is the same proportion, though larger, than that stated in the source methods. The larger sample size helps ensure a more representative result. The source methods further state that additional dilutions may be made if the sample is hygroscopic. Additional water is added to the sample rather than starting with a new sample aliquot.
3	9045C/9045D	The source method has conflicting statements regarding use of additional dilutions for waste and starting with new aliquot and added double the volume of water. The laboratory adds additional reagent water starting with sample to water ratio of 1:3 rather than 1:2.

17.0 Attachments

Attachment 1: Example pH Calibration and Maintenance Log
Attachment 2: Example TALS Benchsheet

18.0 Revision History

This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files and available upon request.

- Revision 18, dated 30 July 2021
  - Annual Review and Method Review
  - Updated copyright information
  - Removed QSM versions and instead referenced SOP DV-QA-024P QA/QC Requirements for Federal Programs for information about DoD QSMs
  - Changed TestAmerica to Eurofins TestAmerica throughout
  - Added reference to Attachment 2 to section 10.5.5
  - Changed section 10.4.4.5 to say to correct the reading to reflect the true pH of the buffer
  - Added to section 10.5.3 to not allow the electrode to come into direct contact with oil or soil
  - Added section 9.3.2 with criteria for the Low and High Check.
  - Added information about the Low and High Check to section 10.4.5.
  - Updated language and formatting throughout

- Revision 17, dated 30 June 2020
  - Annual Review
  - Updated copyright information

Attachment 1.

Example pH Calibration and Maintenance Log

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Denver

Calibration and Maintenance Log

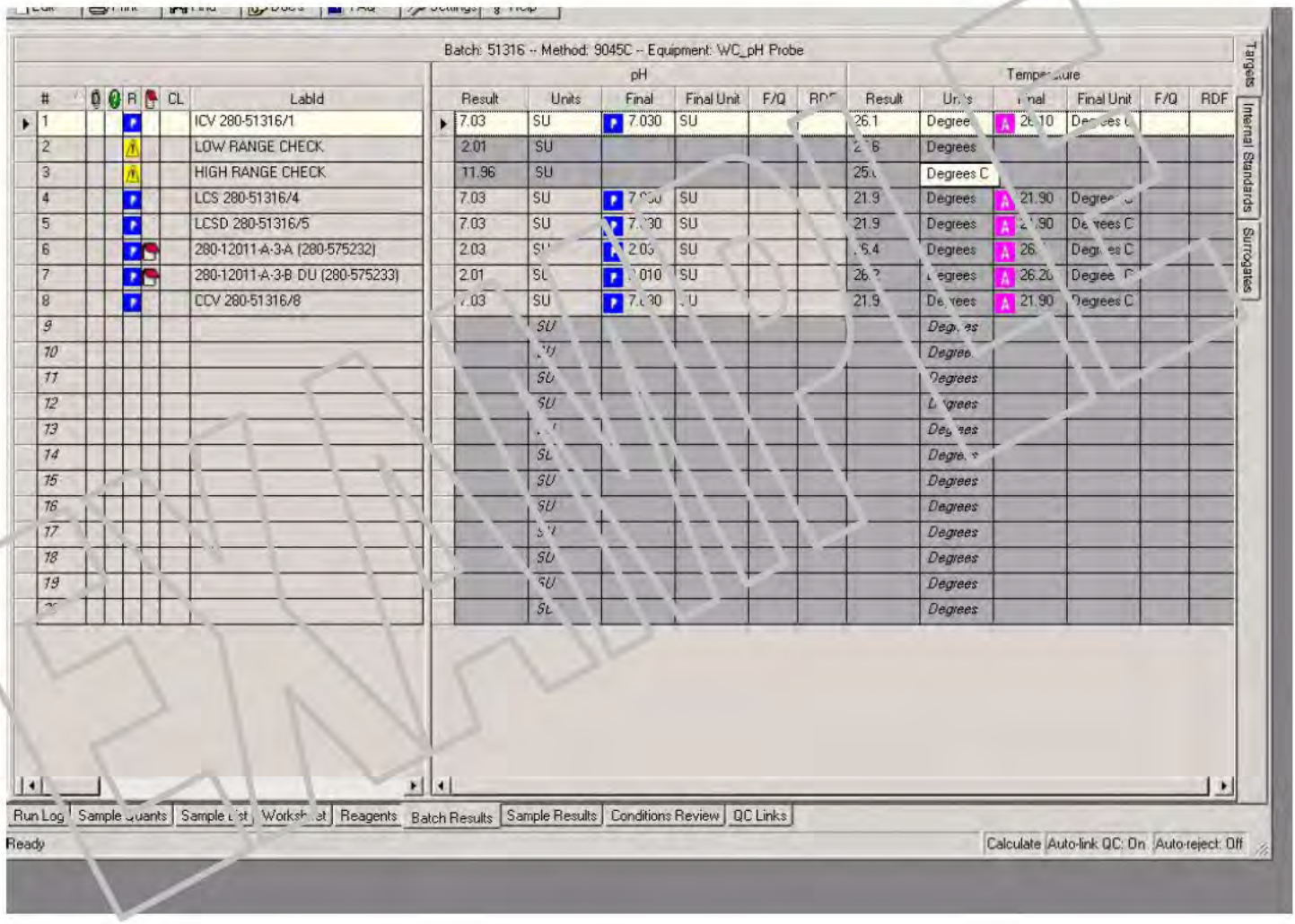
Wet Chemistry / pH Probe

Daily Maintenance	Day	Sat	Sun	Mon	Tue	Wed	Thu	Fri
No maintenance required when instrument is not in use.	Date/Time:							
	Analyst:							
1) Inspect the probe for scratches or cracks.								
2) Probe solution refilled.								
3) Store probe in storage solution.								
4) Wipe off apparatus and clean up any spills.								
Calibration Standards:	2.0 Buffer Lot #: Expiration Date:							
	2.0 Buffer Calibration Reading							
	4.0 Buffer Lot #: Expiration Date:							
	4.0 Buffer Calibration Reading							
	7.0 Buffer Lot #: Expiration Date:							
	7.0 Buffer Calibration Reading							
	10.0 Buffer Lot #: Expiration Date:							
	10.0 Buffer Calibration Reading							
	12.0 Buffer Lot #: Expiration Date:							
	12.0 Buffer Calibration Reading							
Calibration Slope:								
Instrument Removed From Service: Y / N	Initials:	Date:	Return to Service with Passing Calibration				Initials:	Date:

Additional Maintenance/Comments:

Attachment 2.

Example TALS Benchsheet



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Electronic Copy Only

Title: Percent Moisture in Soils and Wastes

[ASTM D2216, CLP ILM05.3, SW 3550C]

Approvals (Signature/Date):

Connie Jewell — 10/29/19 — Date
Technical Manager

Reed Pottruff — 10/29/19 — Date
Health & Safety Manager / Coordinator

Roxanne Sullivan — 10/29/19 — Date
Quality Assurance Manager

Scott Hall — 10/31/19 — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

- **Scope and Application**

.1 This standard operating procedure (SOP) provides instructions for determining percent moisture (or percent solids) in samples and is based on CLP ILM05.3, ASTM D 2216-05, and SW-846 Method 3550C.

.2 This method is applicable to soils and sludges. Wipe samples do not require % moisture. Please see Group leader or Project Manager before proceeding with % moisture for any other unusual matrices For the determination of total solids, total suspended solids, total dissolved solids, volatile solids, volatile dissolved solids, and volatile suspended solids, refer to SOP DV-WC-0064.

.3 The practical range for the determination of percent moisture is from 0.4% to 99.6%, based on the uncertainty of the top-loading balance used.

- **Summary of Method**

.1 The percent moisture of a solid or semi-solid sample is determined gravimetrically by weighing a 15 g aliquot of the homogenized "wet" sample, letting the sample dry in an oven at 100 ± 5 °C for a minimum of 12 hours and weighing the remaining "dry" residue.

.2 The moisture lost by the sample is calculated as the difference between the "wet" mass and the "dry" mass. Percent moisture and percent solids are calculated relative to the original "wet" sample mass.

- **Definitions**

.1 Percent Moisture: The amount of water in a solid or semi-solid material that is lost as a result of drying the material in an oven at a defined temperature, typically 100 ºC, and expressed as a percent of the original sample mass. The moisture determined in this manner is not necessarily the total water content of a material. For example, drying a sample at 100 ºC will not remove waters of hydration.

.2 Percent Solids: The amount of solid in a solid or semi-solid material that is retained after drying the material in an oven at a defined temperature, typically 100 ºC, and expressed as a percent of the original sample mass. The solid determined in this manner is not necessarily the total solid content of a material. For example, drying a sample at 100 ºC will not remove waters of hydration.

.3 Refer to the Glossary of the *TestAmerica Denver Quality Assurance Manual* (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

- **Interferences**

.1 The principal source of error for this method is failure to obtain a representative sample. Non-representative particulates, e.g., rocks or leaves in a soil sample, should be excluded from the sample if it is determined that their inclusion is not

desired in the final result. Any observations of odd sample matrices or exclusions of sample fractions must be recorded by the analyst.

.2 Positive biases may result from, among other things, evaporative losses, loss of water of crystallization, and loss of volatile organic matter during drying.

.3 Negative biases may result from the presence of significant amounts of oil and grease, as well as the absorption of moisture after drying.

- **Safety**

.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

.3 Specific Safety Concerns or Requirements

.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

.3.2 Be careful when placing samples into and taking samples out of the drying oven (100 ºC). Wear insulated gloves or use tongs when handling hot weighing dishes.

.4 Primary Materials Used

There are no materials used in this method that have a serious or significant hazard rating. **Note: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

- **Equipment and Supplies**

.1 Aluminum weigh pans, disposable.

.2 Disposable wooden spatula.

.3 Top-loading balance, 0.01 g readability.

.4 Drying oven, capable of maintaining a temperature of 100 ± 5 °C.

.5 Insulated gloves or tongs.

.6 Desiccator.

.7 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents and Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

- **Reagents and Standards**

DRIERITE desiccant (anhydrous calcium sulfate): Available from commercial sources.

- **Sample Collection, Preservation, Shipment and Storage**

.1 Samples are to be collected in a glass or plastic bottle with a tight fitting cap and refrigerated to ≤ 6 °C.

.2 There is no regulatory holding time for this parameter; however, analysis should begin as soon as possible.

- **Quality Control**

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

.1.1 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential

trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

- **QC Samples**

.1.1 A method blank is not applicable to percent moisture determinations and is not required by the source methods.

.1.2 Laboratory control samples are not applicable to percent moisture determinations and are not required by the source methods.

.1.3 One sample duplicate is required with each batch of 20 or fewer samples.

Acceptance Criteria: When the moisture content of the sample is greater than 10%, the relative percent difference (RPD) between the sample and sample duplicate must be ≤ 20%.

Corrective Action: If the RPD for the duplicate samples exceeds the established limit, then the sample must be reanalyzed, if any sample is remaining. If no sample is remaining, then the data must be appropriately flagged.

.1.4 Matrix spikes and matrix spike duplicates are not applicable to percent moisture determinations and are not required by the source methods.

- **Procedure**

.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

.3 Calibration

.3.1 The top loading balance is calibrated annually by an outside vendor as described in SOP DV-QA-0014.

.3.2 Verify the accuracy of the balance daily before use as described in SOP DV-QA-0014. Daily calibration verifications are recorded in a balance logbook.

.4 Sample Analysis

.4.1 Perform all weighings as quickly as possible. Wet samples lose mass through evaporation of water. Dried samples can be very hygroscopic and absorb moisture from the air.

.4.2 Mass measurements are electronically captured for direct entry into TALS. The TALS Worksheet performs all calculations. If necessary, the data may be directly entered manually into the Worksheet tab in TALS. Weights should not be recorded on secondary sheets for later entry.

.4.3 For each sample in the batch, label the aluminum weigh pan with the sample number. Label an additional pan for the sample duplicate.

.4.4 Weigh each pan on the balance to determine the mass (tare weight) of the pan. Record the tare weight to the nearest 0.01 g for each pan. Collect all of the pans for each batch onto a single aluminum tray.

.4.5 Homogenize the sample by stirring and pulverizing any large chunks.

.4.6 Place a 13 - 17 g representative subsample into the weigh pan. Record the "wet" sample mass as displayed by the balance.

.4.7 Note any observations of the sample condition on the benchsheet (e.g., “rocky”).

.4.8 Carefully place the tray of pans into the drying oven that has been pre-heated to 100 ± 5 ºC. Allow the samples to dry for at least 12 hours. Record the time the samples were placed into the oven, oven temperature when the samples were placed in the oven (observed temperature and corrected temperature), the time they were removed (to document the drying time), and the temperature of the oven (observed temperature and corrected temperature). Also record the thermometer ID and balance ID. (See Attachment 1 for example entries in the Batch Editor.)

NOTE: Samples may be dried for less than 12 hours if it can be demonstrated that a constant mass is obtained. In this case, data must be recorded for a minimum of two repetitive weigh/dry/desiccate/weigh cycles with at least 1 hour of drying time in each cycle. Constant mass is defined as a loss in mass of no greater than 0.01 g between the starting weight and the final weight of the last cycle.

.4.9 At the end of the drying time, remove the tray of pans from the oven and immediately place it in a desiccator. Record the time samples are placed in the desiccator. Allow the samples to cool to room temperature (at least 1 hour). Record the time the samples are removed from the desicator.

NOTE: The desiccant is blue in color when dry. When the indicator color changes to purplish-pink, the desiccant must be replaced.

.4.10 If the sample appears oily, generate an NCM to document the anomaly: "Results for sample (indicate sample number) may be inaccurate - sample appeared oily after drying."

.4.11 Weigh the dried sample and record the mass to the nearest 0.01 g (see Attachment 2).

.5 Troubleshooting and Maintenance

.5.1 DRIERITE in the desiccators should be changed when it turns purplish-pink.

.5.2 If the oven temperature cannot be maintained within range, notify facility maintenance.

- **Calculations / Data Reduction**

.1 Raw data are entered directly into TALS and calculations are performed by the Worksheet tab in the analytical batch. The following calculations represent those performed by TALS and can be used to manually verify these calculations.

.2 Calculation of Solids Fraction

The fraction of solids in the original sample is used to correct the concentration of other measured analytes in a wet sample for the dry sample weight. The fraction of solids in a sample is calculated as follows:

$$\text{solids fraction} = \frac{D - T}{W - T} \qquad \textit{Equation 1}$$

Where:

D = Gross weight (mass) of the dried sample and the weigh pan (grams).
T = Tare weight (mass) of the weigh pan (grams)
W = Gross weight (mass) of the wet sample and the weigh pan (grams).

.3 Calculation for Percent Solids

The percent solids in a sample is calculated by multiplying the fraction of solids by 100%, as follows:

$$\%\,\text{solids} = \frac{D - T}{W - T} \times 100\% \qquad \textit{Equation 2}$$

.4 Calculation for Percent Moisture

TALS uses the percent moisture value to correct reported wet-weight analyte concentrations to a dry-weight basis. The percent moisture in a sample is calculated by subtracting the percent solids from 100%, as follows:

$$\%\,\text{moisture} = 100\% - \left\lfloor \frac{D-T}{W-T} \times 100\% \right\rfloor \qquad \textit{Equation 3}$$

.5 The relative percent difference is calculated for the sample and sample duplicate determinations using the formula:

$$\%\,RPD = \frac{R_1 - R_2}{(R_1 + R_2)/2}\, x\, 100\% \qquad \textit{Equation 4}$$

Where R_1 and R_2 are the calculated % moisture results for the sample and sample duplicate.

.6 When reporting chemical concentrations on a dry-weight basis, divide the wet weight concentration by the fraction of solids in the sample, as follows:

$$\text{Dry Weight Concentration} = \frac{\text{Wet Weight Concentration}}{\text{Solids Fraction}} \qquad \textit{Equation 5}$$

.7 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for a copy of the checklist and for more detail on the review process.

- **Method Performance**

.1 Method Detection Limit Study (MDL)

An initial method detection limit study is not performed for percent moisture. The lower limit of quantitation is dictated by the accuracy and sensitivity of the balance used. This test uses a top loading balance with a readability of 0.01 g. The balance must pass daily calibration verification within ± 0.02 g for weights less than 20 g. If the daily calibration verification control limits represent the maximum "noise" level of the balance, then three times this level, or 0.06 g, would be a reasonable lower limit of quantitation. This translates to a practical measurement range of 0.4 to 99.6 % moisture.

.2 Demonstration of Capabilities

.2.1 Since a spiked aliquot is not appropriate for this procedure, initial and continuing demonstration of capability is documented by collecting data for a completed batch. An acceptable IDOC is determined by meeting any method required batch QC.

.2.2 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024, *Training*.

.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

- **Pollution Control**

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

- **Waste Management**

.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.

.2 The following waste streams are produced when this method is carried out:

.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

.2.2 Solid Sample Waste – Waste Stream S

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

- **References / Cross-References**

.1 USEPA Contract Laboratory Program Statement of Work for Inorganic Analytes, Multi-Media, Multi-Concentration, ILMO 5.3, March 2004, Exhibit D, Section 1.6, *Percent Solids Determination Procedure*.

.2 ASTM D 2216, *Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass*, ASTM International, March 1, 2005.

.3 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3550C, *Ultrasonic Extraction*, Revision 3, December 1996.

- **Method Modifications:**

Item	Method	Modification
1	ILMO5.3 ASTM D 2216 3550C	ILMO5.3 and 3550C specify a sample aliquot of 5 to 10 grams. ASTM D 2216 lists a minimum sample size of 20 grams, but allows discretion for the use of smaller aliquots. The 15-gram aliquot specified in this SOP is appropriate for the size of the weigh pan used.
2	ILMO5.3, ASTM D 2216 3550C	ILMO5.3 specifies a drying temperature of 103 to 105 ºC. ASTM D 2216 specifies a drying temperature of 110 ± 5 ºC. Method 3550C states a drying temperature of 105°C. These temperature specifications are based on the boiling point of water at sea level, which is 100 ºC. The boiling point of water at the altitude of the laboratory location is 95 ºC. Therefore, the acceptable drying temperature range for this SOP has been set at 100 ± 5 ºC to provide results comparable to performing this method at sea level. This is justified by the discussion in ASTM D 2216, which notes that materials containing significant amounts of hydrated water (e.g., gypsum) may present a special problem as these materials can slowly dehydrate at the standard drying temperature of 110 ºC and at very low relative humidity.
3	ILMO5.3	ILMO5.3 allows a single weighing of the dried sample, but only if the sample is dried for a period of at least 12 hours and no longer than 24 hours. If the sample is dried for a period of time less than 12 hours, then it must be demonstrated that a constant weight has been achieved by repeating the drying, cooling, and weighing cycle at least twice. This SOP prescribes a drying time of at least 12 hours. Samples may be left in the ovens over the weekend. This is because the determination of moisture content is not affected by longer drying periods.
4	ILMO5.3, ASTM D 2216	Because of the sample aliquot size and the type of balance used, the practical range of the method is limited to 0.4 to 99.6 weight percent.
5	ILMO5.3, ASTM D 2216	Aluminum weigh dishes are used instead of porcelain crucibles as a matter of convenience. The porcelain dish is not necessary, because the determination of volatile solids, which requires heating in a muffle furnace at 550 ºC is not included in the scope of this SOP.
6	ILMO5.3	Method ILM05.3 requires the use of a lid. The lid is not necessary, because the determination of volatile solids in not included in the scope of this SOP. This is consistent with ASTM D 2216 which states the lid is optional.
7	3550C	Method 3550C states to weigh the sample for dry weight determination immediately following the weighing of the aliquot for extraction. In this laboratory, the dry weight determination is done separately from the preparation of that solid sample for any other tests. The dry weight determined is used for dry weight correction of all tests on that sample. The aliquot used for the determination of dry weight is taken from a container that is clearly marked with the client and lab sample IDs for that particular suite of tests. Depending upon the clients' practices, the sample for dry weight may or may not come from the same container used for one of the other tests for that sample..

- **Attachments**

  Attachment 1: Example Batch Editor Screen
  Attachment 2: Example Gravimetric Calculation Benchsheet

- **Revision History**

  - Revision 11, dated 31 October 2019
    Annual Review
  - Revision 10, dated 31 October 2018
    Annual Review
  - Revision 9, dated 31 October 2017
    - Added statement to section 2.1 about different matrices.
  - Revision 8, dated 31 January 2017
    - Annual Review
  - Revision 7, dated 29 February 2016
    - Annual Review
    - Corrections for structure and grammar throughout
    - Removed pre-2011 revision history
    - Added new Section 3.2, definition of percent solids
    - Removed reference to hazard table in Section 5.4
    - Added language to Section 10.4.2 for clarity
    - Reworded Section 10.4.8 for clarity
    - Changed Section 10.4.9 to align with standard SOP language
    - Changed LIMS to TALS throughout

  - Revision 6, dated 28 February 2015
    - Revised section 10.4.6 to specify range of weights for sample
    - Revised Section 10.4.8 to list all items to be documented in the batch record (observed and corrected temperatures both in an out of oven, time in and out of oven)
    - Revised section 10.4.9 to include recording of times in and out of the desiccator
    - Added new Attachment 1 to provide example of documentation of equipment, oven temperatures, and times.
    - Annual Technical Review

  - Revision 5, dated 28 February 2014
    - Added Section 10.5
    - Annual technical review

  - Revision 4, dated 28 February 2013
    - Added Method 3550C as a reference and throughout SOP as needed
    - Added section 3.2
    - Revised section 9.1, 10.1, and 10.2 to reflect current practice
    - Revised section 10.4.2 to reflect that electronic transfer of weights to the LIMS is the preferred practice for recording data
    - Moved section on data review from section 10 to 11.7 for consistency with other documents
    - Revised section 12.2 to describe the appropriate documentation of an IDOC for a non-spiked test
    - Revised Method modifications 1 and 2 to include Method 3550C
    - Added Method Modification 7 to address determination of percent moisture as an

independent test from other preparations of that sample for analysis
  - Annual technical review
  - Formatting and grammatical changes throughout

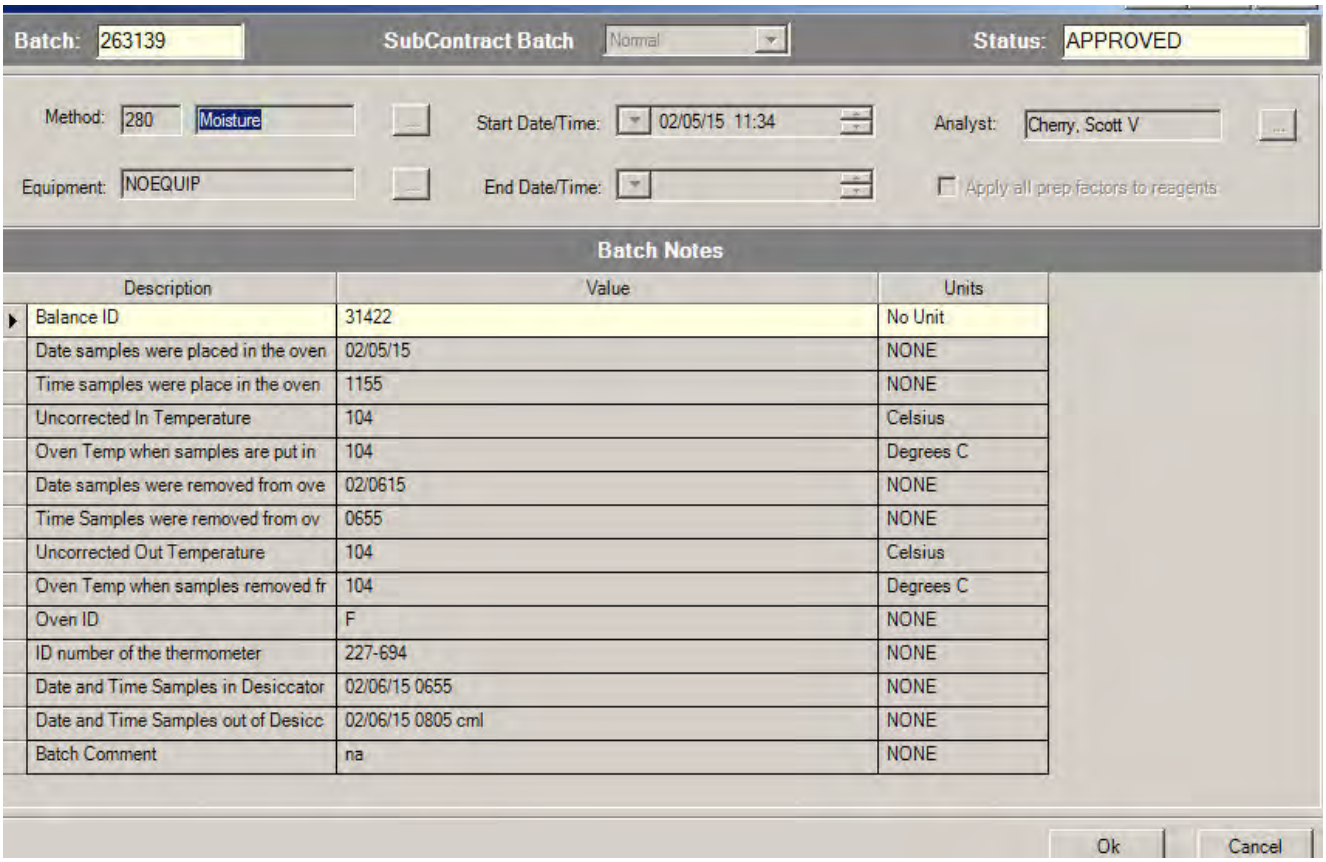
- Revision 3.4, dated 28 February 2012
  - Source method review
  - Added gloves and disposable spatulas to supplies list.
  - Added calculation for % RPD
  - Removed statement that percent moisture of a sample is calculated and entered into the LIMS as raw data are input into the LIMS and result calculated by the LIMS
  - Removed Method EPA 160.3 from references and method modifications. (Previously removed in Revision 3.)
  - Revised method modifications to address sample size, drying temperature and optional use of lid per ASTM D 2216.
  - Removed Attachment 2 and inserted discussion of data review process in section 10.5 with reference to data review SOP and form.

- Revision 3.3, dated 04 March 2011
  - Annual Technical Review.
  - Updated Attachments 1 & 2

Earlier revision histories have been archived and are available upon request

Attachment 1.

Example Batch Editor Screen



Batch: 263139 SubContract Batch Normal Status: APPROVED

Method: 280 Moisture Start Date/Time: 02/05/15 11:34 Analyst: Cherry, Scott V

Equipment: NOEQUIP End Date/Time: Apply all prep factors to reagents

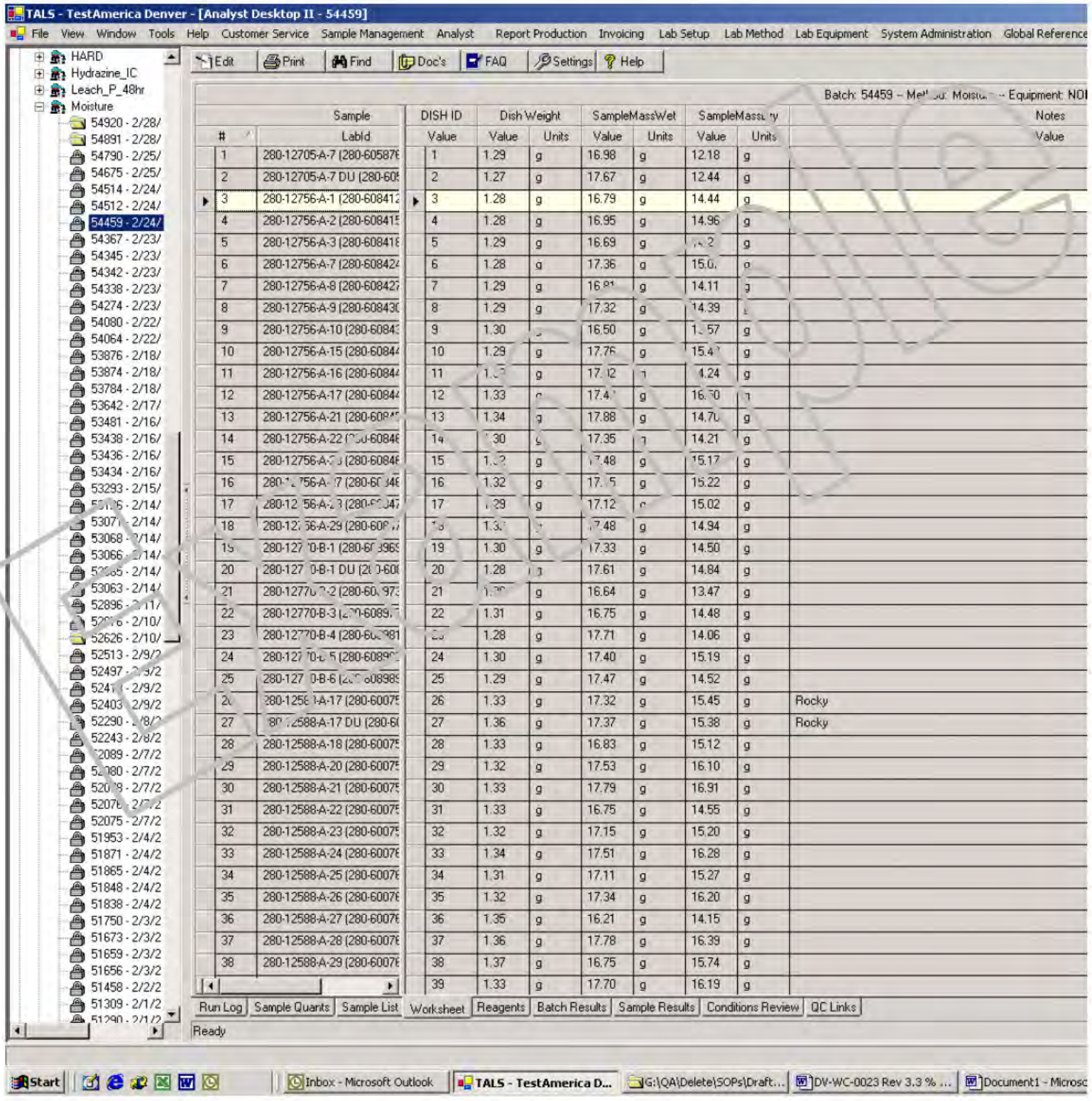
Batch Notes

Description	Value	Units
Balance ID	31422	No Unit
Date samples were placed in the oven	02/05/15	NONE
Time samples were place in the oven	1155	NONE
Uncorrected In Temperature	104	Celsius
Oven Temp when samples are put in	104	Degrees C
Date samples were removed from ove	02/0615	NONE
Time Samples were removed from ov	0655	NONE
Uncorrected Out Temperature	104	Celsius
Oven Temp when samples removed fr	104	Degrees C
Oven ID	F	NONE
ID number of the thermometer	227-694	NONE
Date and Time Samples in Desiccator	02/06/15 0655	NONE
Date and Time Samples out of Desicc	02/06/15 0805 cml	NONE
Batch Comment	na	NONE

Ok Cancel

Attachment 2.

Example Gravimetric Calculation Benchsheet



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Title: Total and Amenable Cyanide by SM 4500-CN⁻ B, 4500-CN⁻ C, 4500-CN⁻ E, 4500-CN⁻ G, SW 9012A, and SW 9012B, Weak Acid Dissociable Cyanide by 4500-CN⁻ I

Approvals (Signature/Date):

Sierra Hohulin 09/30/21
Sierra Hohulin — Date
Department Specialist

Reed Pottruff 10/1/2021
Reed Pottruff — Date
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Maria Fayard 10/1/21
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Quality Assurance Manager

Scott Hall 10/1/21
Scott Hall — Date
Technical Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 Analytes, Matrices, and Reporting Limits

1.1.1 This procedure is for the determination of:

Analyte	CAS Number	Reporting Limit Water (mg/L)	Reporting Limit Soil (mg/kg)
Total Cyanide	57-12-5	0.01	0.50
Amenable Cyanide	STL00015	0.01	0.50
Weak Acid Dissociable Cyanide	STL00195	0.01	0.50
Free Cyanide	STL00131	0.01	0.50

1.1.2 This procedure is applicable to drinking, ground, surface and saline waters, solids and wastes.

1.1.3 This procedure detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine total cyanide, amenable cyanide and weak acid dissociable cyanide as described in Standard Methods 4500-CN^- B, 4500-CN^- C, 4500-CN^- E, 4500-CN^- G and 4500-CN^- I and total and amenable cyanide by SW-846 Methods 9012A and 9012B.

1.1.4 Cyanide amenable to chlorination and weak acid dissociable cyanide appear to be identical, however, in some industrial effluents the cyanide amenable to chlorination yields negative values and the measure of weak acid dissociable is a better alternative.

1.1.5 This procedure describes the reduced volume version of the methods and uses the same reagents and molar ratio to meet the quality control and performance requirement stated in the method.

1.2 Dynamic Range

The approximate working range extends from 0.01 mg/L to 0.4 mg/L for water samples and 0.5 mg/kg to 20 mg/kg for soil samples. Samples with higher concentrations are analyzed at a dilution. Current method detection limits are maintained in the Eurofins TestAmerica LIMS (TALS).

2.0 Summary of Method

2.1 Total Cyanide

Cyanide, as hydrocyanic acid (HCN) is released from samples by means of reflux-distillation under acidic condition and absorbed in a scrubber containing sodium

hydroxide (NaOH) solution. The cyanide concentration in the scrubber solution is determined using an automated analyzer. The cyanide is converted to cyanogen chloride by reactions with Chloramine-T that subsequently reacts with pyridine and barbituric acid to give a red-colored complex. The color intensity which is proportionate to the cyanide concentration is measured at 570 nm. The concentration of NaOH must be the same in the standards, the scrubber solutions and any dilution of the original scrubber solution.

2.2 Amenable Cyanide

Cyanide amenable to chlorination is determined by using two sample aliquots. The first aliquot is distilled for total cyanide and the second aliquot is chlorinated under an alkaline condition prior to distillation and is used to determine cyanide not amenable to chlorination. Cyanide amenable to chlorination is the difference in these two values.

2.3 Weak Acid Dissociable Cyanide (WAD)

Weak acid dissociable cyanide is determined by distillation of the sample to which has been added acetate buffer and zinc acetate solution and sufficient acetic acid to obtain a pH of approximately 4.5 - 6 as determined by methyl red indicator. The cyanide concentration collected under these conditions is determined as described in Section 10.4.

3.0 Definitions

3.1 Cyanide: The term “cyanide” refers to all of the CN groups in cyanide compounds that can be determined as the cyanide ion, CN^- by various chemical methods. These compounds include both simple and complex cyanides.

3.2 Total Cyanide: All cyanides including nondissociable cyanides and cyanide bound in complexes that are readily dissociable or of intermediate stability

3.3 Cyanide Amenable to Chlorination: Free cyanide and complex cyanides that are potentially dissociable, wholly or in large degree.

3.4 Weak Acid Dissociable Cyanide: Cyanide that is available in a slightly acidified solution, including free cyanide.

3.5 Free Cyanide: Cyanide that is not complexed in the sample. Free cyanide is determined as weak acid dissociable cyanide.

4.0 Interferences

4.1 Oxidizing agents such as chlorine will destroy cyanide. Ascorbic acid is used to remove chlorine interferences.

4.2 Some unidentified organic compounds may oxidize or form decomposition products during chlorination, giving higher results for cyanide after chlorination than before chlorination; this gives a negative value for cyanide amenable to chlorination. Examples include samples from petroleum refineries, the steel

industry, and pulp from paper processing. The weak acid dissociable method should be used for these samples. See Section 9.7.

4.3 Samples that contain sulfide compounds may produce hydrogen sulfide during the distillation and interfere with color development. This is treated by adding cadmium chloride (Standard Methods) or bismuth nitrate (SW-846 methods) to the sample prior to distillation, which removes sulfur by precipitation as cadmium sulfide or bismuth sulfide.

NOTE: Requirements for sample and standard processing are different for Standard Methods or SW-846 Methods. See Section 9.4.2.

4.4 Chlorine added to the sample for amenable cyanide must be completely destroyed before distillation. Otherwise, it may distill over and destroy the non-amenable cyanide.

4.5 Nitrate and/or nitrite may react with organic compounds during distillation to form cyanide. Sulfamic acid is added to remove the nitrate and/or nitrite interference.

4.6 Samples containing surfactants may foam excessively during distillation.

4.7 High carbonate concentrations may react violently when sulfuric acid is added to the samples during distillation.

4.8 Aldehydes, glucose, and other sugars may convert cyanide to cyanohydrin during distillation. If the client has indicated the possible presence of aldehydes, add 3.5% ethylenediamine as described in Section 9.4.3.

4.9 Amino acids may distill with the cyanide and interfere with the analysis.

4.10 Fatty acids may interfere by forming soaps in the absorption solution.

4.11 Thiocyanate greater than 10 mg/L may interfere.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, latex or nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

Potassium cyanide and sodium cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of HCN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, and dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Potassium Hydroxide	Corrosive Poison Reactive	2 mg/m^3 - ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting and diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness and swelling. Greater exposures cause severe burns with possible blindness.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Hydrochloric Acid	Corrosive Poison	5 ppm - ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat and upper respiratory tract and in severe cases, pulmonary edema, circulatory failure and death. Can cause redness, pain and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric acid	Corrosive Poison Irritant, Carcinogen	1 mg/m^3 TWA	Inhalation symptoms may include irritation of the nose and throat, and labored breathing. Swallowing can cause severe burns of the mouth, throat, and stomach, leading to death. Can cause sore throat, vomiting, and diarrhea. Skin contact can cause redness, pain, and severe burn. Eye contact can cause blurred vision, redness, pain and severe tissue burns.
Calcium hypochlorite	Strong oxidizer	None listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.
Glacial acetic acid	Corrosive Poison Flammable Irritant	10 ppm TWA	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Swallowing can cause severe injury leading to death. Skin contact may include redness, pain, and skin burns. Eye contact may cause severe eye damage followed by loss of sight.
Sulfamic acid	Corrosive Irritant	None listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.
Chloramine-T	Irritant	None listed	May cause irritation to the mucous membranes and upper respiratory tract, skin and eyes.
Barbituric acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as a potential health hazard; do not ingest.
Bismuth nitrate	Oxidizer	None	May cause irritation to the respiratory tract, skin and eyes.
Silver Nitrate	Corrosive Poison Oxidizer	0.01 mg/m^3 (TWA) for silver metal dust and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns, and eye damage.
Ethylenediamine	Corrosive Flammable Irritant	TWA 10 ppm (25 mg/m^3)	Inhalation symptoms may include irritation nose, respiratory system; sensitization dermatitis; asthma; liver, kidney damage
Cadmium Chloride	Acutely Toxic Environmental Hazard	TWA 0.2 mg/m^3	Ingestion causes gastroenteric distress, pain, and prostration. Sensory disturbances, liver injury, and convulsions have been observed in severe intoxications.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5 Build-up of pressure in the distillation apparatus will cause the hot, acidic solution to spray out of the thistle tube. In case vacuum is lost, the condensers must be opened to prevent build-up of pressure. If the solution overflows onto the heating block, turn it off.

5.6 All distillations are to be performed with adequate ventilation.

5.7 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.8 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit. For cyanide amenable to chlorination, the chlorination step will also be performed in a fume hood.

5.9 All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica Denver associate. The situation must be reported immediately to a laboratory supervisor and the Health and Safety Officer.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Flow Solution 3700 Automated Chemistry Analyzer (OI Analytical)

- OI Analytical 3180 Autosampler
- IPC High Precision Multichannel Pump
- Injection module
- Colorimeter with 570 nm filter and 10 mm flow cell
- FlowView Software

6.1.2 Distillation Apparatus consisting of an Environmental Express 12 well HotBlock, 50 mL distillation tubes, 25 mL collection traps, and associated connectors and tubes. See Attachment 2.

This apparatus does not require the use of cold finger condensers.

6.1.3 Vacuum pump.

6.1.4 Recirculating chiller for cold fingers.

6.2 Supplies

6.2.1 Disposable auto-sampler vials or culture tubes for samples.

6.2.2 Syringe filters with 0.45 µm filter.

6.2.3 Pipettes, various sizes.

6.2.4 Volumetric flasks, class A, various sizes.

6.2.5 Volumetric pipettes, class A, various sizes.

6.2.6 Miscellaneous laboratory apparatus (e.g., magnetic stirrer) and glassware.

6.2.7 pH test strips.

6.2.8 Lead acetate test paper.

6.2.9 Potassium iodide-starch test paper.

6.2.10 Boiling chips.

6.3 Computer Software and Hardware

Please refer to the master list of documents, software, and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

NOTE: TALS IDs for standards and reagents are given in parentheses.

7.1 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.

7.2 One of the calibration standards (ICAL or ICV) must be obtained from a calibration laboratory accredited to ISO/IEC 17025 (ISO Guide 34) by a recognized Accreditation Body. The other may be obtained from other acceptable sources.

7.3 Cyanide Calibration Stock Standard, 1,000 mg/L (CN CAL STD)

7.3.1 This standard is purchased commercially.

7.3.1.1 All intermediate and working standard concentrations are adjusted from the nominal concentrations shown below to the exact concentrations based on the certified concentration from the vendor.

7.4 Intermediate Standard I, 10 mg/L (CN 10ppm)

7.4.1 Obtain a clean 100 mL class A volumetric flask.

7.4.2 Add approximately 50 mL of the 1% (0.25 N) sodium hydroxide (1% NaOH) to the flask.

7.4.3 Pipette 1.0 mL of the 1,000 mg/L calibration stock standard (Section 7.3) into the flask.

7.4.4 Dilute to volume with 1% (0.25 N) sodium hydroxide (1% NaOH). Prepare every 7 days from the standardized stock.

7.5 Calibration Standards

7.5.1 Working Standard, 1.0 mg/L (CN CAL 1 ppm)

7.5.1.1 Pipette 10.0 mL of Intermediate Standard I (Section 7.4) into a 100 mL volumetric flask.

7.5.1.2 Dilute to volume with 1% (0.25 N) sodium hydroxide and mix. Prepare daily.

7.5.2 Initial Calibration Standards

Dilute the 1.0 mg/L or 0.1 mg/L cyanide working standard with 1% (0.25 N) sodium hydroxide as follows:

Standard Level	Intermediate	Vol of Std Used (mL)	Final Volume (mL)	Concentration (mg/L)
1	1 mg/L	0	50	0.00 (Blank)
2	1 mg/L	0.5	50	0.01
3	1 mg/L	1.0	50	0.02
4	1 mg/L	2.5	50	0.05
5	1 mg/L	5.0	50	0.10
6	1 mg/L	10.0	50	0.20
7	1 mg/L	10.0	25	0.40

Calculate the exact concentration for each calibration curve standard using Equations 2 and 3 if stock concentration is different from 1.0 mg = 1.0 mL. When using the TALS Reagent Module, the concentration of the calibration standards will be automatically calculated. Prepare daily.

7.6 Continuing Calibration Verification Standard (CCV), 0.2 mg/L:

The Level 6 calibration standard described in Section 7.5.2 is used as the working CCV standard.

7.7 Second-Source Standards

7.7.1 Initial Calibration Verification (ICV) Stock Standard, 1,000 mg/L (CN ICV Std)

The second-source standard is obtained from a different source than the calibration standards. This standard is available commercially. Manufacturer's expiration date is used.

7.7.2 Intermediate ICV (second-source) Standard, 10 mg/L (CN ICV Int)

Pipette 1.0 mL of the 1,000 mg/L ICV stock (Section 7.7.1) into a 100 mL volumetric flask. Dilute to volume with 1% (0.25 N) sodium hydroxide. Prepare every 7 days.

7.7.3 Working ICV (second-source) Standard, 0.10 mg/L (CN ICV Daily)

Spike 1 mL of the 10 mg/L intermediate ICV standard (Section 7.7.2) into a 100 mL volumetric flask and fill to the mark with 1% (0.25 N) NaOH. Prepare daily.

7.8 Pyridine-Barbituric Acid Solution, per OI Manual (CN PYR/BARB_)

7.8.1 This standard is purchased commercially.

7.8.2 *Alternatively*, the Pyridine-Barbituric acid solution can be made as described here.

7.8.3 In a hood, place 15 g barbituric ($C_4H_4N_2O_3$) acid (Barbitiric) in a 250 mL volumetric flask and add about 100 mL reagent water, rinsing down the sides of the flask.

7.8.4 Place on a magnetic stirrer and add a stir bar.

7.8.5 Add 75 mL pyridine (Pyridine) while mixing.

7.8.6 Carefully add 15 mL concentrated hydrochloric acid (HCL) while mixing.

7.8.7 Add 150 mL of reagent water and stir until the barbituric acid is dissolved.

7.8.8 Dilute to volume with reagent water.

7.8.9 Store in an amber bottle.

7.8.10 Expires 6 months from preparation.

7.9 Phosphate Buffer Solution 1 M (CN BUFFER)

7.9.1 Dissolve 138 g sodium dihydrogen phosphate monohydrate ($NaH_2PO_4 \bullet H_2O$) (Sodium Phosphate) in reagent water and dilute to 1,000 mL.

7.9.2 Add 4 mL of Brij-35 to the solution and mix gently.

NOTE: Actual volume varies depending upon equipment operation. It may be necessary to add additional Brij-35 for smooth operation of the equipment.

7.9.3 Store at room temperature.

7.9.4 Filter the solution through a glass fiber filter.

7.9.5 This solution expires 3 months from preparation.

7.10 Chloramine-T, per OI Manual (CN CHLOR-T)

7.10.1 Dissolve 2.0 g Chloramine-T in reagent water and dilute to 250 mL.

7.10.2 Prepare fresh daily.

7.11 Sodium Hydroxide, 10 N (10N_NaOH)

Purchased ready to use.

7.12 Sodium Hydroxide, 2% wt/wt (0.5 N) (2% NaOH_)

7.12.1 Place 80 g NaOH in a plastic 4 liter container.

7.12.2 Add 2,000 mL of reagent water to the container.

7.12.3 Add a magnetic stir bar and stir until the NaOH is dissolved.

7.12.4 Add 2,000 mL of reagent water to the flask. Stir to mix.

7.13 Sodium Hydroxide Dilution Solution, 1% wt/wt (0.25 N) (1% NaOH_)

7.13.1 Add 25 mL of 10 N NaOH to a 1 liter volumetric flask.

7.13.2 Bring to volume with reagent water, and mix well.

7.14 Sulfuric acid, concentrated, reagent grade (H2SO4)

7.15 Sulfuric acid, 0.02 N (0.02 H2SO4)

7.15.1 If commercial solution is not available, this solution can be prepared as follows. In a 2,000 mL volumetric flask, carefully add 1 mL concentrated sulfuric acid to approximately 1,900 mL reagent water.

7.15.2 Dilute to final volume of 2,000 mL with reagent water and mix.

7.16 Calcium hypochlorite solution, 0.35 M, $Ca(OCl)_2$

7.16.1 Combine 5 g of calcium hypochlorite and 100 mL of reagent water. Shake well before using. Replace monthly.

7.16.2 Alternatively, fragrance free commercial liquid bleach (e.g., Clorox® Bleach) can be purchased and used in place of the calcium hypochlorite solution. Replace 1 month after opening.

7.17 Magnesium Chloride solution, 2.5 M (CN Mag Chl)

This reagent is purchased through an approved vendor.

7.18 Glacial acetic acid, reagent grade (Acetic acid).

7.19 Acetate buffer (WAD Acetate)

7.19.1 This buffer is purchased commercially.

7.19.2 *Alternatively,* dissolve 410 g sodium acetate trihydrate (SODIUM ACETATE) in approximately 450 mL reagent water.

7.19.3 Adjust the pH to 4.5 with glacial acetic acid and dilute to final volume of 500 mL with reagent water.

7.19.4 The solution expires 1 year from preparation.

7.20 Zinc Acetate solution (Zinc Buffer)

7.20.1 Dissolve 100 g zinc acetate dihydrate in a 1 liter volumetric flask filled with approximately 500 mL reagent water.

7.20.2 Dilute to final volume of 1,000 mL with deionized water.

7.20.3 Store in a 1 L plastic container.

7.20.4 The solution expires 1 year from preparation.

7.21 Methyl Red Indicator solution (Methyl Red)

7.21.1 Dissolve 0.1 g methyl red in 100 mL reagent water.

7.21.2 Expires 1 year from preparation.

7.22 Acetic acid, 10% (10% Acetic Acid)

7.22.1 In a vent hood, carefully add 100 mL glacial acetic acid to about 500 mL reagent water, mix, and dilute to 1,000 mL.

7.22.2 The solution expires 1 year from preparation.

7.23 Sulfamic Acid (NH_2SO_3H), 10% wt/wt (CN SULFAMIC)

7.23.1 Dissolve 100 g sulfamic acid in 1,000 mL of deionized water. Mix well.

7.23.2 Store in a repipetter container.

7.23.3 Expires 1 year from preparation.

7.24 Ascorbic acid crystals (Ascorbic Acid)

Used to remove chlorine interference.

7.25 Cadmium Chloride powder, ($CdCl_2$), (CAD CHL CN)

Used to remove sulfide interference.

7.26 3.5% Ethylenediamine (EDTA) solution

Used to remove client-identified aldehyde interferences

7.27 Bismuth Nitrate ($Bi(NO)_3 \bullet 5H_2O$), 0.062 M, (CN_BiN3O9)

7.27.1 Obtain a clean, dry 250 mL volumetric flask.

7.27.2 Add approximately 75 mL of reagent water to the flask.

7.27.3 Add 7.5 g bismuth nitrate to the flask and stir to dissolve.

7.27.4 Slowly add 60 mL of glacial acetic acid, swirling frequently.

7.27.5 Stir until completely dissolved.

7.27.6 Bring to volume with reagent water.

7.28 Brij-35 Start-Up Solution

Concentrated Brij-35 is a buffer solution obtained from the equipment vendor. The start-up solution is prepared by diluting 1 mL of the Brij-35 concentrate to 500 mL with reagent water.

7.29 Reagent Water

Water with a resistivity of 1 Megohm-cm or greater. The Eurofins TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

7.30 Teflon boiling chips for use as solid matrix for LCS and MB. Record lot number in TALS in prep batch information.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water[1]	HDPE, Glass	500 mL	Cool, ≤ 6 °C	48 hours **	40 CFR Part 136.3
Water[1,2]	HDPE, Glass	500 mL	NaOH, pH > 10; Cool, ≤ 6 °C;	14 days	40 CFR Part 136.3
Water[2]	HDPE, Glass	500 mL	NaOH, pH > 12; Cool, ≤ 6 °C	14 days	SW-846
Solid	HDPE, Glass	5 g	Cool, ≤ 6 °C	14 days	SW-846

[1] Add 1.2 g of ascorbic acid per liter of sample if residual chlorine is present.

[2] Preservation to pH > 10 for NPDES compliance samples (Method 335.4, the SM 4500-CN^- methods); pH >12 for Methods 9012A and 9012B.

**NOTE: If the client sample arrives unpreserved the sample needs to be checked for interferences (Section 9.4). If chlorine is found, the sample can be treated prior to preservation, but sulfide treatment must be conducted on a sample at a stable pH >12 and during the distillation process (see WI-DV-0022 Cyanide Preservation for USEPA Method 335.4 and SOP DV-WC-0081 Total Cyanide by EPA Method 335.4 and Amenable Cyanide by 335.1). If there are no interferences preserve with NaOH (pH noted above) and follow the 14 day holding time. Document the preservation using an observation Nonconformance Memo (see SOP DV-QA-0031 Non-Conformance and Corrective Action System).

Quality Control

8.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply.

8.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver Policy DV-QA-003P Quality Control Program.

8.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver Policy DV-QA-024P QA/QC Requirements for Federal Programs. This procedure meets all criteria for DoD QSM unless otherwise stated. Any deviation or exceptions from DoD QSM requirements must have prior approval in the project requirements.

8.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

8.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. This is in addition to the corrective actions described in the following sections.

8.2 Sample QC: The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	<1/2 Reporting Limit or < 10% of sample concentration
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Total Cyanide: 90-110% LCS/LCSD RPD: ≤ 10% Statistical Limits[3] for WAD
Matrix Spike (MS)[2]	1 in 10 or fewer samples	Total Cyanide: 90-110% LCS/LCSD RPD: ≤ 10% Statistical Limits[3] for WAD
MS Duplicate (MSD)[2]	1 in 10 or fewer samples	Total Cyanide: 90-110% MS/MSD RPD: ≤ 10% Statistical Limits[3] for WAD
High Distilled Standard (HLCS) (0.35 mg/L)	1 in 20 or fewer samples	WAD: 75-120%
Low Distilled Standard (LLCS) (0.10 mg/L)	1 in 20 or fewer samples	Total Cyanide: 90-110%[4] WAD:75-120%

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client.
[3] Statistical control limits are updated annually and are stored in TALS.
[4] For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs.

8.3 Method Blank

8.3.1 Preparation

8.3.1.1 Water Samples:

Add 50 mL of 2% (0.5 N) NaOH into a distillation flask immediately prior to distillation.

8.3.1.2 Solid Samples:

Weigh 1.0 g of Teflon chips into a distillation flask and add 50 mL 2% (0.5 N) NaOH.

8.3.2 Acceptance

Acceptance Criteria: Concentrations in the method blank must be less than one-half the reporting limit or less than 10% of the sample concentration.

Corrective Action: The corrective action for method blank failures is redistillation and reanalysis of all samples in the batch. If there is insufficient sample for reanalysis, a Nonconformance Memo (NCM) must be prepared and the client contacted by the laboratory Project Manager.

8.4 LCS / LCSD (Second Source)

8.4.1 Preparation

8.4.1.1 Water Samples

8.4.1.1.1 Measure 50 mL of 2% NaOH in a graduated cylinder and transfer to the distillation flask.

8.4.1.1.2 Spike 0.5 mL of the 10 mg/L second source intermediate standard (Section 7.7.2) into the flask. Swirl to mix.

8.4.1.2 Soil Samples

8.4.1.2.1 Weigh 1.0 g of Teflon chips into a distillation flask.

8.4.1.2.2 Measure 50 mL of 2% NaOH in a graduated cylinder and transfer to the distillation flask.

8.4.1.2.3 Spike with 0.5 mL of the 10.0 mg/L second source intermediate standard (Section 7.7.2) into the flask. Swirl to mix.

8.4.2 Acceptance

Acceptance Criteria: LCS recoveries for Total Cyanide are 90-110%. Recoveries for Weak Acid Dissociable Cyanide (WAD) are compared to the historical limits stored in TALS. See also Section 8.2.

For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs.

The LCS for Cyanide Amenable to Chlorination should be 0% which demonstrates that the chlorination process has worked effectively. (This is not reported.)

If a LCSD is also analyzed the RPD must be within ± 10% for Total Cyanide and within historical limits stored in TALS not to exceed ± 20% for WAD. See Also Section 8.2.

Corrective Action: If the LCS fails, redistill and reanalyze all samples in the batch. If reanalysis is not possible, a Nonconformance Memo must be prepared and the client contacted by the laboratory Project Manager. See the Eurofins TestAmerica Denver Policy DV-QA-003P Quality Control Program for additional guidance.

8.5 MS/MSD

8.5.1 Preparation

8.5.1.1 Water Samples

Measure 50 mL of sample into a class A graduated cylinder. Spike the aliquot with 0.5 mL of the 10.0 mg/L second source intermediate standard (Section 7.7.2) and bring to 50 mL with sample. The matrix spike and matrix spike duplicate are prepared in the same manner. Both the matrix spike and matrix spike duplicate are taken through the distillation and analysis process.

8.5.1.2 Soil Samples

Weigh 1.0 g of sample into a distillation flask. Spike the sample aliquot with 0.5 mL of the 10.0 mg/L second source intermediate standard (Section 7.7.2), then add 2% (0.5 N) NaOH for a total volume of 50 mL. The matrix spike and matrix spike duplicate are prepared in the same manner. Both the matrix spike and matrix spike duplicate are taken through the distillation and analysis process.

8.5.2 Acceptance

Acceptance Criteria: MS/MSD recoveries are compared to the limits stored in TALS. The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. See also Section 8.2. For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and

batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check the calculations and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an

over-range measurement (e.g., the E-flag qualifier).

- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of target analytes. Any samples that have target analytes at such low levels do not provide useful precision data via duplicate analyses. If a sample duplicate is performed for DoD QSM, see SOP DV-QA-024P QA/QC Requirements for Federal Programs for specific criteria.

8.6 High and Low Distilled Standards (HLCS & LLCS), 0.4 mg/L and 0.10 mg/L

Standards are distilled to monitor the efficiency of the distillation process and verify the linearity of the curve.

8.6.1 Preparation

8.6.1.1 Water Samples

In each of two graduated cylinders, add 25 mL of 2% NaOH (see Section 7.12).

8.6.1.1.1 For the HLCS, add 1.75 mL of the 10 mg/L cyanide standard (CN 10ppm) and bring to 50 mL with reagent water.

8.6.1.1.2 For the LLCS, add 0.5 mL of the 10 mg/L cyanide standard (CN 10ppm) and bring to 50 mL with reagent water.

8.6.1.2 Soil Samples

Weigh 1 g of Teflon chips into a distillation flask for each standard.

8.6.1.2.1 For the HLCS, add 25 mL of 2% NaOH to the distillation flask and spike 2.0 mL of the 10 mg/L cyanide standard to the flask and add 25 mL of 2% NaOH for a total volume of 50 mL.

8.6.1.2.2 For the LLCS add 25 mL of 2% NaOH to the distillation flask and spike 0.5 mL of the 10 mg/L cyanide standard to the flask and add 25 mL of 2% NaOH for a total volume of 50 mL.

8.6.2 Acceptance

Acceptance Criteria: Recoveries for the HLCS and LLCS standards must be ± 10% of the true value. For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs.

Corrective Action: Distilled standard failure results in re-distillation and reanalysis of all associated samples. One possible exception is the situation in which recoveries are greater than 110% and cyanide was not detected in the samples. In that case, a Nonconformance Memo should be prepared and the failure noted in the report together with the sample results without taking other corrective action. For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs.

8.7 Cyanide QC

8.7.1 The amenable cyanide method requires three different procedures.

8.7.2 The first is the total cyanide. The QC for total cyanide is performed as described in Sections 8.3 -8.6.

8.7.3 The second is the Non-Amenable Cyanide. The QC required is as follows:

8.7.3.1 A treated Method Blank. The Method Blank is prepared the same as described in Section 8.3. It goes through the treatment process as described in Section 9.5.

8.7.3.2 A treated LCS (see Section 9.5). The treated LCS is prepared the same as described in Section 8.4. It goes through the treatment process as described in Section 9.5. The treated LCS should have a 0% recovery to show that the chlorination process is working correctly.

8.7.3.3 An LCS/LCSD that is spiked post-treatment. The LCS/LCSD are treated method blanks that are than spiked after the chlorination treatment but just prior to distillation. The post-treated LCS/LCSD are to show that the distillation procedure is working correctly and there is no excess chlorine.

8.7.3.4 A sample duplicate is performed for demonstration of batch precision.

8.7.4 The third is the Amenable Cyanide. The Amenable Cyanide is a calculation method using the total cyanide and non-amenable cyanide results.

8.8 Instrument QC

8.8.1 Initial Calibration Verification (ICV)/ Initial Calibration Blank (ICB)

Immediately after the initial calibration, the calibration is verified using a second-source ICV standard and an initial calibration blank ICB (1% NaOH).

Acceptance Criteria: The measured result for the ICV must be within 10% of the expected value.

The ICB must be less than the reporting limit.

For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs.

Corrective Action: If these criteria are not met, check the accuracy of the standards and recalibrate.

8.8.2 Continuing Calibration Verification (CCV) / Continuing Calibration Blank (CCB)

A blank CCB (1% NaOH) and standard check CCV (see preparation in Section 7.6) are required after every 10 or fewer samples and at the end of the run.

Acceptance Criteria: The standard check (CCV) must be within 10% of the expected value.

Blanks must be less than the reporting limit.

For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs.

Corrective Action: If either continuing calibration check fails, all samples since the last successful calibration check must be reanalyzed.

9.0 Procedure

9.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. The NCM shall be filed in the project file and addressed in the case narrative.

9.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

9.3 Record identification for all pipettes, balances, and other equipment used in the batch record.

9.4 Sample Preparation

9.4.1 The following sections describe separate preparation procedures for the different forms of cyanide.

Section 9.5: Cyanide Amenable To Chlorination
Section 9.6: Total Cyanide
Section 9.7: Weak Acid Dissociable Cyanide

9.4.2 Check aqueous samples for sulfide prior to distillation using lead acetate paper.

9.4.2.1 Moisten the paper with 2 or 3 drops of acetate buffer, and then place 1 drop of sample on the paper. A dark color indicates a positive test for sulfide (see WI-DV-0022 Cycanide Preservation for USEPA Method 335.4). Record the result as "positive" or "negative" in the TALS prep batch.

9.4.2.1.1 If the test for sulfide is positive, Method 9012A and Method 9012B require that the samples be treated with bismuth nitrate rather than cadmium chloride and processed in a separate batch. The standards must be treated in the same manner as the samples, including addition of bismuth nitrate and distillation. See Section 9.6.10.

9.4.2.1.2 If the samples test positive using lead acetate paper and are to be analyzed using Standard Methods,

treat 75 mL of the stabilized sample (pH > 12) with cadmium chloride.

9.4.2.2 Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper.

9.4.2.3 Filter the solution. From the filtrate, measure the sample aliquot to be used for analysis.

9.4.2.4 The filters are contaminated with cadmium. Use the designated disposal container for these filters.

9.4.3 If the client has specifically indicated that aldehydes may be present, 2 mL of 3.5% ethylenediamine is added per 100 mL of sample upon receipt.

9.5 Cyanide Amenable To Chlorination Sample Preparation

9.5.1 Two sample aliquots are required for the determination of cyanide amenable to chlorination. The first aliquot is distilled for total cyanide (see Section 9.6). The second aliquot is chlorinated under an alkaline condition prior to distillation and is used to determine cyanide not amenable to chlorination.

NOTE: If the results for Total Cyanide are less than the reporting limit, the cyanide amenable to chlorination is not determined and reported as ND. The non-amenable cyanide is then equal to the total cyanide result. The method blank reported in this case is the Total Cyanide method blank.

NOTE: The chlorination process must be performed in a fume hood.

9.5.2 Measure the sample aliquots to be chlorinated (including a method blank, LCS and sample duplicate) into 100 mL beakers covered with a large plastic tub. Keep beaker covered with wrapped watch glass. Alternative means of protecting samples from light may be used.

9.5.2.1 For water samples, use 50 mL of sample.

9.5.2.2 For soil samples, use 1.0 g of sample and add 50 mL 2% (0.5 N) NaOH.

9.5.2.3 Clearly label the samples with the proper identification and "chlorinated" as appropriate.

9.5.3 Check the pH of samples with pH test strips. Record the results on the bench sheet.

9.5.4 Adjust the pH of the samples in the beakers to between 11 and 12 with the 10 N sodium hydroxide solution.

9.5.5 Test the samples with KI-Starch paper and add bleach solution drop-wise to each sample while mixing (use a magnetic stirrer) until a positive test is obtained. A positive test is indicated by a blue or black color on the paper.

9.5.6 Maintain the excess chlorine level in the sample for 1 hour while keeping the pH of the samples between 11 and 12 with constant mixing (use a magnetic stirrer). Add bleach solution and sodium hydroxide as necessary. Document the pH in Worksheet tab.

9.5.7 After 1 hour, add 0.1 g portions of ascorbic acid crystals until a negative test is obtained with KI-Starch paper.

9.5.8 Add an additional 0.1 g of ascorbic acid crystals to the sample to ensure an excess of the reagent

9.5.9 Transfer the contents of the beakers into distillation flasks quantitatively, rinsing with reagent water.

9.5.10 Proceed to Section 9.6.4 for the distillation process.

9.6 Total Cyanide Sample Preparation

9.6.1 Check the pH of aqueous samples with pH test strips. Record the results on the TALS bench sheet. If the sample pH is $\leq$ 12 for Methods 9012A or 9012B or $\leq$ 10 for SM 4500 methods, document the improper preservation with a NCM.

9.6.2 Check aqueous samples for oxidizing agents such as chlorine.

9.6.2.1 Place one drop of sample on a strip of potassium iodide (KI)-starch test paper. A blue color indicates the need for treatment.

9.6.2.2 Record the result as “positive” or “negative” on the bench sheet.

9.6.2.3 If a positive test is obtained, add a few crystals of ascorbic acid at a time until a drop of sample produces no color on the indicator paper.

9.6.2.4 Add an additional 0.1 g of ascorbic acid in excess.

9.6.3 Measure sample aliquots into the distillation flasks as follows:

9.6.3.1 For water samples use 50 mL of sample.

9.6.3.2 For solid samples use 1.0 g of sample and add 50 mL 2% (0.5 N) NaOH.

9.6.3.3 Prepare the batch QC samples as described in Section 8 (MB, LCS, MS/MSD, LLCS, HLCS)

9.6.4 Place 25 mL 2% sodium hydroxide into the absorption tubes.

9.6.5 Turn on the vacuum pump and chiller. Also be sure that the slot hood is operating.

9.6.6 Assemble the distillation apparatus. All distillations are to be performed under the slot hood.

NOTE: Batch QC samples must be rotated through all distillation glassware and positions. Do **not** use specific glassware or distillation positions for the MB and LCS/LCSD.

9.6.7 Turn on the vacuum pump and ensure the chiller is on. Also be sure that the slot hood is operating.

9.6.8 Adjust the vacuum to provide a flow rate of approximately 2-3 bubbles per second (i.e., this is approximately 1/8-1/4 inch of foam in the scrubber) in the distillation flask.

9.6.9 Verify that there are no leaks in the system by observing the flow into the absorber tube. The flow rate may not remain constant during the distillation; readjust as necessary.

9.6.10 If the samples are logged for Method 9012A or 9012B and the test for sulfide is positive, all standards (minimum of five standards and blank), QC and samples must be processed in a separate batch.

9.6.10.1 Add 5 mL of 0.062 M bismuth nitrate solution (see Section 7.277) through the thistle tube to every standard, sample and QC sample in the analytical batch.

9.6.10.2 Samples designated for analysis using DoD QSM where the project specifically requires MSA are analyzed using the method of standard addition utilizing a 2-point spike and calculating the sample concentration using the MSA data. Consult your Supervisor, a Technical Specialist, or the QA Manager before proceeding.

9.6.11 Add 2 mL of 10% sulfamic acid solution (Section 7.23) through the thistle tube. Allow to mix for 3 minutes.

9.6.12 Slowly and carefully, add 2.5 mL concentrated sulfuric acid through the thistle tube. Rinse the tube with a little reagent water and allow to mix for 3 minutes.

9.6.13 Add 2 mL of magnesium chloride solution (Section 7.17) and mix. If excessive foaming is observed, add additional magnesium chloride.

9.6.14 Turn on the heating mantles and heat the samples to boiling. While distilling the samples, carefully watch to make sure that vacuum is maintained on all of the stills. Adjust the flow as necessary.

9.6.15 Allow samples to reflux for 1.5 hours by initiating the timer on the heating mantle.

9.6.16 After 1.5 hours of refluxing, allow the samples to cool for 15 - 30 minutes while air is flowing.

9.6.17 *While the vacuum is still on*, remove the absorption tube from the distillation apparatus. Rinse the inside and outside of the bubbler into the tube with reagent water.

Note: It is important to keep the vacuum on to prevent the distillate from being trapped inside the bubbler.

9.6.18 Remove the flasks and dispose of the contents as directed in Section 13. Residue not removed by this method must be scrubbed out.

9.6.19 Dilute the sodium hydroxide in the absorption tube to 50 mL with Elga water using a class A graduated cylinder and store in labeled plastic vials.

9.6.20 Place each batch of distillates in a box and store the samples at 4 °C in the sample refrigerator until they are analyzed.

9.6.21 At the end of the day, turn off the vacuum.

9.6.22 Proceed to Section 9.8 for colorimetric analysis of the distillates.

9.7 Weak Acid Dissociable Cyanide in Water – Sample Preparation (SM 4500-CN I)

9.7.1 Measure the pH of water samples with a pH test strip. Record the results on the bench sheet. If the sample pH is less than 12, document the improper preservation with a NCM.

9.7.2 Measure 50 mL or an aliquot diluted to 50 mL into a distillation flask for each sample plus matrix spike and matrix spike duplicate. (See Section 8.5).

9.7.3 Prepare the batch QC samples as described in Section 0 (MB, LCS, LLCS, and HLCS)

9.7.4 Record the sample volume on the bench sheet.

9.7.5 Place 25 mL 2% (0.5 N) sodium hydroxide into the absorption tubes.

9.7.6 Turn on the vacuum pump and chiller. Also be sure that the slot hood is operating.

9.7.7 Assemble the distillation apparatus. All distillations are to be performed under the slot hood.

9.7.8 Turn on the vacuum pump and chiller. Also be sure that the slot hood is operating.

9.7.9 Adjust the vacuum to provide a flow rate of about 4 bubbles per second in the distillation flask, verifying that there are no leaks in the system by

observing the flow into the absorber tube. The flow rate may not remain constant during the distillation; readjust as necessary.

9.7.10 Add through the air inlet tube: 2 mL acetate buffer (Section 7.19), 2 mL zinc acetate solution (Section 7.200), and 2 or 3 drops of methyl red indicator (Section 7.21). Rinse tube with about 2 mL reagent water and allow to mix.

9.7.11 If the solution is not pink, add 10% acetic acid drop-wise until a pink color persists, rinsing in with reagent water.

9.7.12 Turn on the controller and heat the samples to boiling. While distilling the samples, carefully watch to make sure that vacuum is maintained on all of the stills. Adjust the flow as necessary.

9.7.13 Allow samples to reflux for 1.5 hours. Then allow the samples to cool for 15 minutes while air is flowing. Record start and end times for the distillation in the batch record.

9.7.14 *While the vacuum is still on*, remove the absorption tube from the distillation apparatus. Rinse the inside and outside of the bubbler into the tube with reagent water.

Note: It is important to keep the vacuum on to prevent the distillate from being trapped inside the bubbler.

9.7.15 Dilute the sodium hydroxide in the absorption tube to 50 mL with Elga water using a class A graduated cylinder and store in labeled plastic vials.

9.7.16 Place each batch of distillates in a box and store the labeled distillates at 4 °C until they are analyzed.

9.7.17 Raise the cold-finger condensers and rinse into the distillation flasks with de-ionized water. Dispose of the contents as directed in Section 13. Residue not removed by this method must be scrubbed out.

9.7.18 Rinse absorber tubes with reagent water.

9.7.19 At the end of the day, turn off the vacuum.

9.7.20 Proceed to Section 9.8 for colorimetric analysis of the distillates.

9.8 Instrument Set-Up

9.8.1 Verify that the 570 nm filter is installed.

9.8.2 Instrument Stabilization

9.8.2.1 Connect the reagent pump tubes to a reagent bottle containing the Brij Solution.

9.8.2.2 Start the pump, allowing the start-up solution to flow through the entire system.

9.8.2.3 Make sure that the flow cell of the detector is purged of all bubbles and the flow is stable and free from surging.

9.8.2.4 Once a stable flow is achieved, connect the reagent pump tubes to their respective reagent bottles, as shown in the schematic in Attachment 3.

9.8.2.5 Allow the reagents to flow through the entire system, then, once again, verify that the flow cell of the detector is purged of all bubbles.

9.9 Initial Calibration

9.9.1 Calibration is performed daily or each time the instrument is set up using the standards shown in Section 7.5 and the external standard method.

NOTE: The use of internal standards is not applicable for this spectrophotometric method.

9.9.2 A minimum of five standards and a blank are required for the calibration. The high standard in Section 7.5 may be dropped if needed and sample dilutions performed appropriately.

NOTE: If sulfide was detected during the sample preparation step and the samples are logged for 9012A or 9012B, then bismuth nitrate must be used to precipitate the sulfide. The method of standard additions spike must be prepared, and all calibration standards must be treated and distilled in the same manner as the samples, including the addition of bismuth nitrate. A minimum of five standards and a blank shall be distilled. Use the same calibration levels as shown in the table in Section 7.5.2.

9.9.3 The calibration function is calculated by least-squares linear regression. See Section 10.2.

Acceptance Criteria: The correlation coefficient, r, must be $\geq$ 0.995 ($r^2 \geq 0.99$) and the absolute value of the intercept must be lower than one-half the response for the reporting limit.

Corrective Action: If the correlation coefficient is < 0.995 or the absolute value of the intercept is too large, locate and correct the problem and re-calibrate the instrument.

9.9.4 The Relative Error (%RE) must be calculated for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level. See section 10.0 for example calculations. The %RE for the standard at the lowest level must not exceed ±20%. The %RE for the mid-point standard must not exceed ±10%. This calculation can be done using the corporate form CA-Q-WI-013 *Calibration Regression Calc. Spreadsheet* found on OASIS. This form is also located at \\tafs\Lab2\Denver\Admin\QA\Edit\FORMS\Wet Chemistry. If these criteria are not met, check the accuracy of the standards and recalibrate.

9.9.5 Assess the peak height of the synchronization (sync) standard.

Acceptance Criteria: The peak height of the sync standard should be ± 10% of pervious sync.

Corrective Action: If the peak height is < 62,000, the flow cell of the instrument must be cleaned (consult manufacturer's instructions), and then the instrument must be recalibrated

9.9.6 Initial Calibration Checks

Immediately after the initial calibration, the calibration is verified using a second-source, initial calibration verification (ICV, see preparation in Section 7.7.3) standard and an initial calibration blank (ICB, 1% NaOH).

Acceptance Criteria: The measured result for the ICV must be within 10% of the expected value, and the ICB must be less than the reporting limit.

Corrective Action: If these criteria are not met, check the accuracy of the standards and recalibrate.

9.9.7 Continuing Calibration Checks

A standard check (CCV; see preparation in Section 7.66) and a blank (CCB made up of 1% NaOH) and are required after every 10 or fewer samples and at the end of the run.

Acceptance Criteria: The measured result for the CCV must be within 10% of the expected value, and the CCB must be less than the reporting limit.

Corrective Action: If either or both the CCV and CCB fail, all samples since the last successful calibration check must be reanalyzed.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	≥0.995 correlation coefficient and %RE < ±20% (Lowest Level) %RE < ±10% (Mid-point Level)
Initial Cal. Verification (ICV)	Immediately after the calibration	± 10% of the expected value
Initial Cal. Blank (ICB)	Immediately after the calibration	Less than the reporting limit
Continuing Cal. Verif. (CCV)	Prior to / after every 10 injections	± 10% of the expected value
Continuing Cal. Blank (CCB)	Prior to / after every 10 injections	Less than the reporting limit

9.10 Sample Analysis

9.10.1 Following instrument set up and calibration, the sample distillates are analyzed in exactly the same manner as the calibration standards. The routine run sequence is as follows:

Sync
Carryover
Baseline
Cal 0.00 ppm
Cal 0.02 ppm
Cal 0.05 ppm
Cal 0.10 ppm
Cal 0.20 ppm
Cal 0.40 ppm
Blank
Baseline
Second-source ICV
ICB
Baseline
High concentration distilled standard (HLCS)
Low concentration distilled standard (LLCS)
LCS
LCSD
Method blank
5 samples (may include MS/SD)
Blank
Baseline
CCV
CCB
Baseline
10 samples (may include MS/SD)
Blank

Baseline
CCV
CCB
Baseline
Additional cycles of 10 samples with CCV/CCB
Blank
Baseline
Closing CCV
Closing CCB
Baseline

9.11 Instrument Shut-Down

9.11.1 Disconnect the reagent lines and put all of them into reagent water, except the buffer line.

9.11.2 The buffer line is put into the brij water.

9.11.3 Rinse all tubes for at least 10 minutes

9.11.4 Turn off instrument and pump.

9.11.5 Loosen tubing and cassettes.

9.11.6 Empty Waste.

9.12 Troubleshooting / Maintenance

9.12.1 Ensure vacuum system is free of leaks by *lightly* applying stopcock grease to all ground glass joints. DO NOT over-grease.

9.12.2 Ensure chiller is on and functioning.

9.12.3 Ensure glassware is free of etching and chips.

9.12.4 Ensure standard solutions are at pH ≥12.

9.12.5 Verify instrument reagent flows are proper and that pump tubing is in good condition.

10.0 Calculations / Data Reduction

10.1 Detailed calibration equations can be found in the Eurofins TestAmerica Policy, CA-Q-P-003 Calibration Curves and the Selection of Calibration Points and under the public folder, *Arizona Calibration Training*.

10.2 Total Cyanide

10.2.1 A linear calibration model is used to relate the cyanide concentration to the absorbance as follows:

$$y = mx + b \qquad \textit{Equation 4}$$

Where:

y	=	Absorbance of cyanide standard at 570 nm.
x	=	Cyanide concentration of standard, mg/L.
m	=	Slope of the fitted straight line.
b	=	y-intercept of the fitted straight line.

10.2.2 The cyanide concentration in an unknown aqueous sample extract is calculated by solving the calibration equation (Equation 1) for concentration (x) and using the measured absorbance of the sample, as follows:

$$x = \frac{y - b}{m} \qquad \textit{Equation 5}$$

Where:

x	=	cyanide concentration in sample (mg/L)
y	=	absorbance of the distillate at 570 nm
m	=	slope of the calibration line
b	=	y-intercept of the calibration line

10.2.3 If an aqueous sample was diluted, use Equation 5 to calculate a final result.

$$C_s = x \times DF \qquad \textit{Equation 6}$$

Where:

C_s	=	Cyanide concentration in the original sample (mg/L).
x	=	Cyanide concentration in sample distillate (mg/L).
DF	=	Dilution factor, if applicable.

10.2.4 The cyanide concentration in an unknown solid sample extract is calculated using the following equation:

$$C_s = \frac{x \times V_t}{W \times D} \times DF \qquad \textit{Equation 7}$$

Where:

C_s	=	Cyanide concentration in the original sample (mg/L)
x	=	Extract analyte concentration, mg/L
V_t	=	Volume of distillate, L (nominal 0.050 L)
W	=	Weight of sample, kg (nominal 0.001 kg)
D	=	(100 - % moisture in sample)/100, for a dry-weight basis or 1 for a wet-weight basis
DF	=	Dilution factor, if applicable.

NOTE: All routine calculations for total cyanide are performed by the instrument data system and TALS, provided dilutions and other information have been correctly entered.

10.3 Cyanide Amenable to Chlorination:

Amenable Cyanide = Total CN Result – Treated Result *Equation 8*

10.3.1 The "Total CN Result" is the cyanide concentration for the sample aliquot that was distilled without treatment with chlorine. The treated result is the sample portion that was chlorinated and then distilled. The treated result is reported as Nonamenable Cyanide (the cyanide remaining after chlorination) and is calculated in the same manner as Total Cyanide. See Section 10.2 for detailed descriptions of the calculations.

10.3.2 If the chlorinated aliquot shows more cyanide than the unchlorinated aliquot, a corrective action and/or a discussion in the final report is required. Iron-cyanides can cause this to occur. Weak acid dissociable cyanide would be a better method for these types of samples.

10.4 Weak Acid Dissociable Cyanide

The concentration of cyanide measured after the procedure in Section 9.7 is the weak acid dissociable cyanide. See Section 10.2 for detailed descriptions of the calculations.

10.5 Accuracy

ICV, CCV, LCS, HLCS, LLCS:

$$\%\ \mathrm{Recovery} = \frac{observed\ concentration}{known\ concentration} \times 100\% \qquad \textit{Equation 9}$$

MS/MSD

$$\%\ \mathrm{Recovery} = \frac{(spiked\ sample\ conc.) - (unspiked\ sample\ conc.)}{spike\ concentration} \times 100\% \qquad \textit{Equation 10}$$

10.6 Precision (RPD)

$$\%\ \mathrm{RPD} = \frac{|\ orig.\ sample\ value - dup.\ sample\ value\ |}{[(orig.\ sample\ value + dup.\ sample\ value)/2]} \times 100\% \qquad \textit{Equation 11}$$

10.7 Relative Error (%RE)

$$\%Relative\ Error = \frac{x'_i - x_i}{x_i} \times 100 \qquad \textit{Equation 12}$$

Where:

x_i = True value for the calibration standard
x'_i = Measured concentration of the calibration standard

This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level.

10.8 Data Upload

Refer to Work Instruction WI-DV-0068 Cyanide Uploads to TALS for upload instructions into TALS.

10.9 Reporting

10.9.1 Reporting units are mg/L for water samples and mg/kg for solids samples.

10.9.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

10.9.3 Solid samples are reported on a dry-weight basis unless otherwise requested by the client. Reporting limits are adjusted for both sample size and percent solids.

10.9.4 All associated data are entered or uploaded into TALS as required. For soil samples, the total cyanide concentration is calculated by the LIMS using equation 7 (Section 10.2.4). If the non-amenable cyanide data are uploaded, the soil concentration is also calculated by TALS. If the non-amenable cyanide data are manually entered, the analyst must first calculate the soil concentration from the instrument result using equation 7 (Section 10.2.4). In either case, the amenable cyanide result is calculated by TALS using equation 8 (Section 10.3). If dilutions are performed, the amenable cyanide result in TALS does not reflect an adjusted RL due to the dilution.

Note: Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

10.10 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 Data Review for more detail on the review process.

11.0 Method Performance

11.1 Method Detection Limit Study (MDL)

11.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Eurofins TestAmerica Denver's Policy No. CA-Q-S-006 Detection and Quantitation Limits. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements indicate a greater frequency.

11.1.2 The current MDL value is maintained in TALS.

11.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of capability (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

11.2.1 Four aliquots of the QC check sample or LCS (independent source from the calibration) and a method blank are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample (or LCS) should be equivalent to a mid-level calibration.

11.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

11.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. The method blank must be less than ½ the RL. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

11.2.4 For Amenable Cyanide, a purchased QC or PT sample is used in place of LCS aliquots.

11.2.5 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

11.2.6 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024 Training.

11.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024 Training.

12.0 Pollution Control

12.1 In general, the quantity of chemicals purchased by Eurofins TestAmerica Denver is based on expected usage during its shelf life. The volume of reagents and standards prepared for this procedure reflects anticipated usage.

12.2 Source reduction is achieved through the use of midi-distillation followed by an automated colorimetric determination.

12.3 The volume of hazardous waste is minimized through proper segregation and management of the various waste streams generated by this procedure.

13.0 Waste Management

13.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Environmental Health and Safety Manual, *Waste Management and Pollution Prevention,* and SOP DV-HS-001P Waste Management Plan.

13.2 The following waste streams have been identified for this method:

13.2.1 Cyanide standardization waste – Aqueous Alkaline (E)

13.2.2 Distilled sample – Aqueous Acidic (F)

13.2.3 Distillate – Aqueous Alkaline (E)

13.2.4 OI Analytical/cyanide process waste – Aqueous Alkaline, contains pyridine (E)

13.2.5 Contents of sampler cups – Aqueous Alkaline (E)

13.2.6 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

14.0 References / Cross-References

14.1 Standard Methods for the Examination of Water and Wastewater, On-line Edition.

14.1.1 4500-CN^- A-1999. Introduction

14.1.2 4500-CN^- B-1999. Preliminary Treatment of Samples

14.1.3 4500-CN^- C-1999. Total Cyanide after Distillation

14.1.4 4500-CN^- E-1999. Colorimetric Method

14.1.5 4500-CN^- G-1999. Cyanides Amenable to Chlorination after Distillation

14.1.6 4500-CN^- I-1999. Weak Acid Dissociable Cyanide

14.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

14.2.1 Method 9012A, Total and Amenable Cyanide (Automated Colorimeteric, with Off-Line Distillation), Revision 1, December 1996.

14.2.2 Method 9012B, Total and Amenable Cyanide (Automated Colorimeteric, with Off-Line Distillation), Revision 2, August 2002.

14.3 OI Manual (available on instrument computer)

14.4 Department of Defense Quality Systems Manual for Environmental Laboratories, DoD QSM Version 5.0, July 2013.

15.0 Method Modifications:

Item	Method[1]	Modification
1	SM 4500-CN⁻ SW 9012A SW 9012B	This SOP substitutes bleach for calcium hypochlorite solution.
2	SM 4500-CN⁻	There are differences among the referenced methods concerning the sodium hydroxide concentration in working standards: Standard Method 4500-CN E states in Section 4a that working standards are made using a solution containing 1.6 grams per liter of water, which is equal to 0.04 N. Methods 9012A and 9012B state in Section 7.4.1 that calibration standards are prepared using 50 mL of 1.25 N sodium hydroxide and diluting to 250 mL, which produces a 0.25 N sodium hydroxide concentration. This procedure uses 0.25 N NaOH to ensure stability of standards. This is the same concentration used in EPA Method 335.4 and SW-846 Method 9012.
3	SM 4500-CN⁻ SW 9012A SW 9012B	The stock standard is verified if no certificate of analysis is available. Monthly verifications are sufficient to monitor the concentration due to the rate of use of the standard.
4	SM 4500-CN⁻ SW 9012A SW 9012B	The reflux time for Cyanide Amenable to Chlorination and Total Cyanide has been changed to 1.5 hours versus 1.0 hours to accommodate the reflux time for samples requiring distillation under the Clean Water Act (EPA Method 335.4).
5	SM 4500-CN⁻	When the sulfide test is positive for analysis by SM 4500-CN⁻, the samples will be treated with cadmium chloride rather than the lead (IV) carbonate listed in SM 4500-CN⁻ B or lead(II)carbonate listed in SM 4500-CN⁻ C. This change reduces environmental pollution and provides a standardized sulfide treatment across all cyanide methods.
6	SM 4500-CN⁻ SW 9012A SW 9012B	SM 4500-CN⁻ B-1999 states that use of ascorbic acid be used for preservation when residual chlorine is suspected. 40 CFR Part 136.3, Table II states use of reducing agent with none specified. This laboratory uses the ascorbic acid rather than sodium arsenite as sodium arsenite is a hazardous material and the regulation is not explicit regarding which reducing agent to use.
7	SW 9012A SW 9012B	Methods 9012A and 9012B state the amenable cyanide test must be performed under amber light. In this procedure the beakers and watch glasses are wrapped with foil or kept in the dark by alternate means as described in SM 4500-CN⁻.

Item	Method[1]	Modification
8	SW 9012A SW 9012B	Calibration is verified with an independently prepared check standard (ICV) with every analytical run and a CCV is run after every 10 samples, instead of for every 15 samples. Acceptance criteria used is ± 10% rather than ± 15%.
9	SW 9012A SW 9012B	Methods 9012A and 9012B state the use of a 15 mm flow cell. The equipment used in this laboratory utilizes a 10 mm flow cell, which is that specified in SM 4500-CN^-.
10	SW 9012A SW 9012B	Methods 9012A and 9012B utilize the method of standard additions if MS/MSD recoveries exceed acceptance limits. Because MS/MSD results may not have a direct bearing on other samples in the batch, matrix effect is assumed if MS/MSD recoveries exceed acceptance limits when LCS recoveries are acceptable.
11	SM 4500-CN^- SW 9012A SW 9012B	Chloramine-T solution is prepared by adding 2.0 g Chloramine-T to deionized water and diluting to 250 mL. Methods 9012A/B use a proportion of 1.0 g to 250 mL and SM 4500 CN uses 1 g to 100 mL. This laboratory has determined that the more concentrated solution, consistent with the SM 4500 CN provides more consistent coloration of samples.

[1] SM 4500-CN^- refers to approved version 1999 or 2011.

16.0 Attachments

Attachment 1: Example Cyanide Preparation Bench Sheet

Attachment 2: Cyanide Microblock Distillation Apparatus

Attachment 3: Alpkem Manifold Schematic

17.0 Revision History

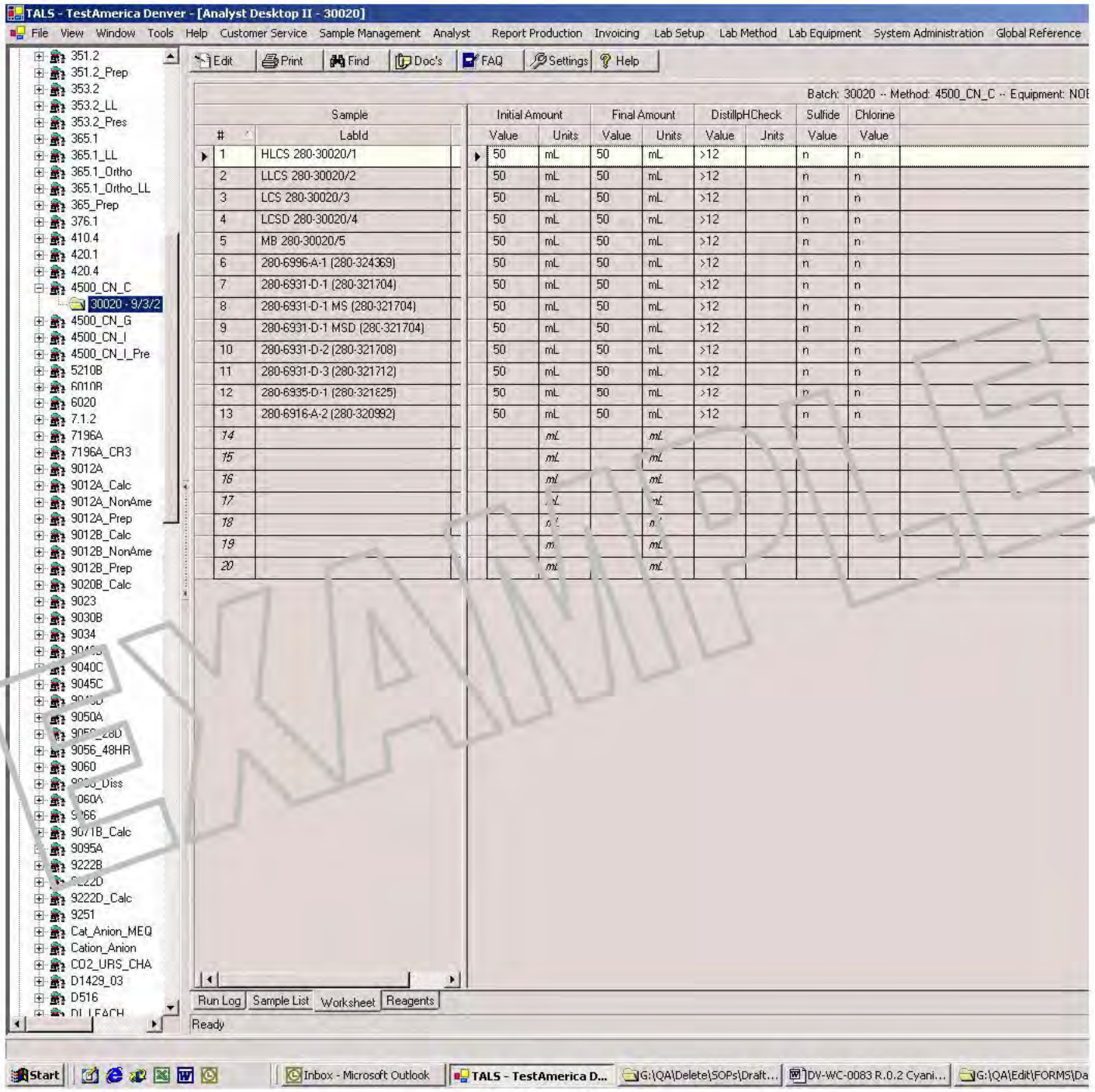
This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files and available upon request.

- Revision 13, dated 1 October 2021
  - Added sections 9.9.4 and 10.7 about Relative Error.

- Revision 12, dated 11 June 2021
  - Annual Review and Method Review
  - Updated copyright information
  - Changed TestAmerica to Eurofins TestAmerica throughout
  - Removed QSM versions and instead referenced SOP DV-QA-024P QA/QC Requirements for Federal Programs for information about DoD QSMs.
  - Minor formatting and language corrections throughout
  - Added Ethylenediamine and cadmium chloride to hazards in Section 5.4
  - Added information for OI Analyzer in Section 6.1.1
  - Removed distillation apparatus consisting of Environmental Express 10 well distiller in section 6.1.2.
  - Updated Section 16 with new attachment 2
  - Replaced Attachment 2 with the correct image of microblock glassware

Attachment 1.

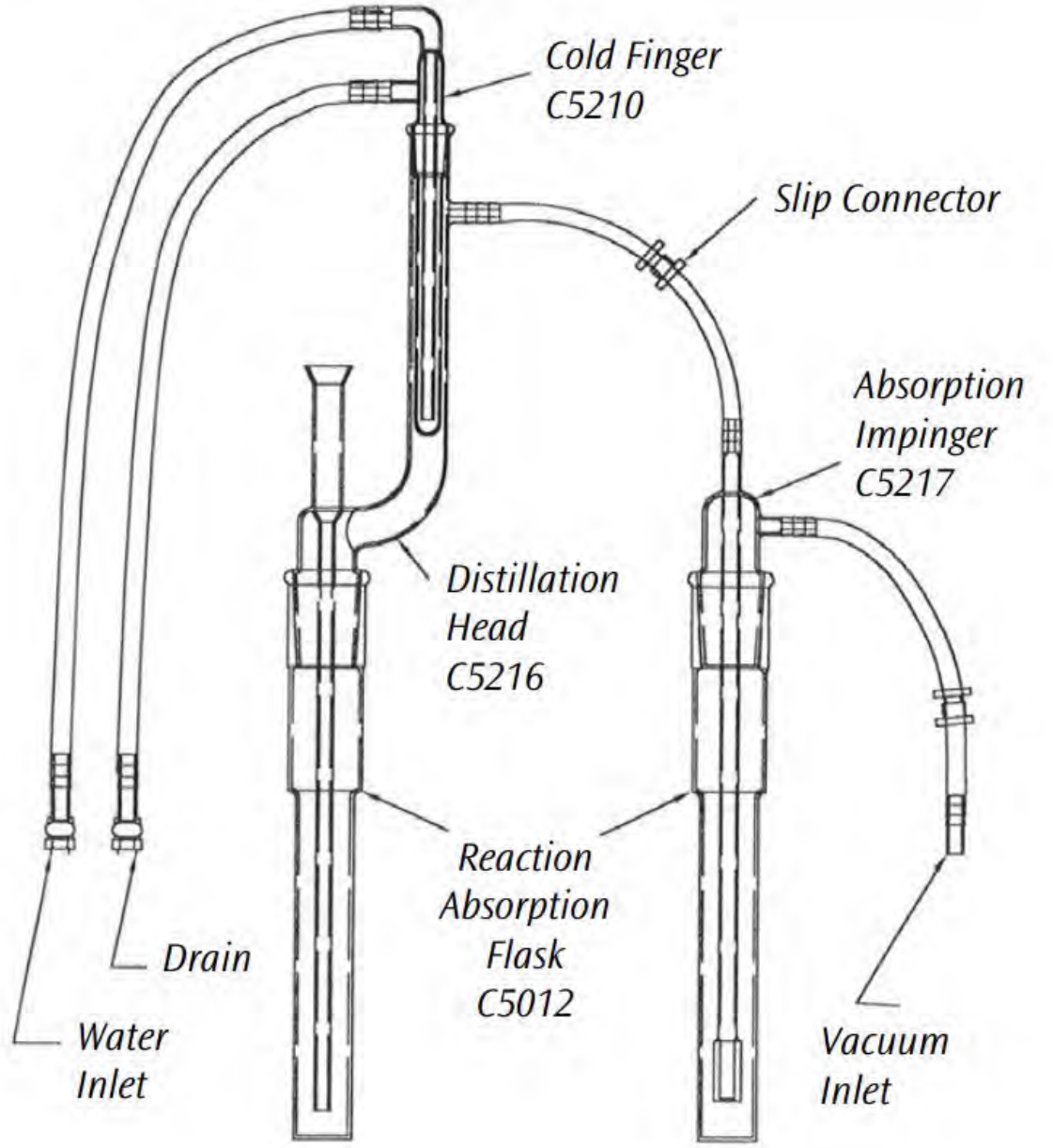
Example Cyanide Preparation Bench Sheet



Attachment 2.

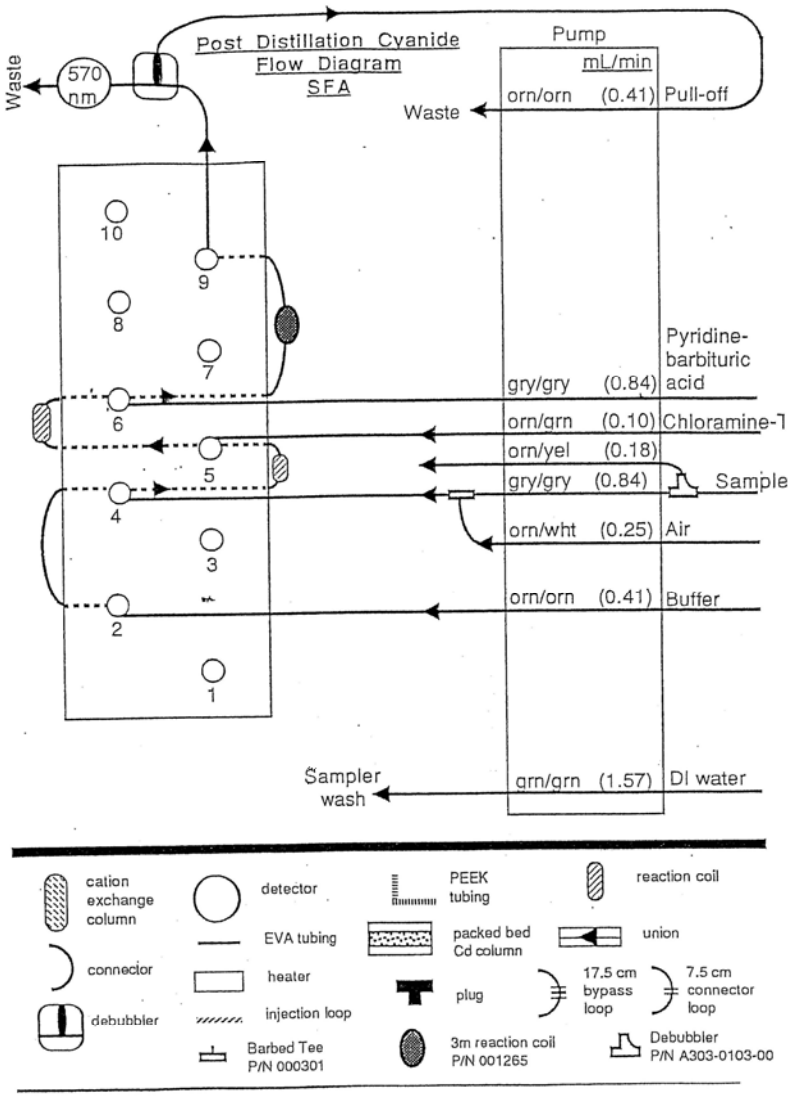
Cyanide Microblock Distillation Apparatus

Figure 2 - Cyanide Glassware



Attachment 3.

Alpkem Manifold Schematic



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Title: Acid-Soluble and Acid-Insoluble Sulfides: Distillation and Titration [SW 9030B / SW9034]

Approvals (Signature/Date):

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Health & Safety Manager / Coordinator

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Scott Hall Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:** _____

1.0 Scope and Application

1.1 This SOP covers both the sample pretreatment procedure designed to remove interfering substances prior to analysis and the determination of the concentration of acid-soluble sulfide. It is based on SW-846 Methods 9030B and 9034.

1.2 This method does not measure acid-insoluble sulfides.

1.3 This method is applicable to the measurement of total acid-soluble sulfides in drinking water, surface and saline waters, domestic and industrial wastes and solid waste materials.

1.4 The reporting limit for water samples is 4 mg/L and for soil samples is 10 mg/kg. Samples that contain more than 50 ppm sulfide should be diluted.

2.0 Summary of Method

2.1 The sulfide method is a two part method involving the distillation of the sample using a micro-distillation apparatus followed by titration of the collected scrubber solution.

2.2 The separation of sulfide from the sample matrix is accomplished by the addition of sulfuric acid to the sample in a closed system. The sample is then heated (for total sulfide) to 70 °C. The H_2S which is formed is carried by a nitrogen stream into a zinc acetate/formaldehyde gas scrubbing bottle. Under these conditions it is precipitated out as ZnS.

2.3 The titration is accomplished by the addition of excess iodine to a sample which has been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate.

3.0 Definitions

Please refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual and SOP DV-QA-003P *Quality Control Program* for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Aqueous samples should be taken with a minimum of aeration to avoid the oxidation or volatilization of sulfide.

4.2 Reduced sulfur compounds, such as sulfite and bisulfite decompose in acid to form SO_2. The SO_2, if carried over into the scrubbing solution, may yield false positives. The addition of formaldehyde in the scrubber solution removes this interference. This method shows no sensitivity to sulfite or bisulfite at concentrations up to 10 ppm.

4.3 Many metals (e.g., Hg, Cd, Cu) form insoluble sulfides and give low recoveries.

4.4 The iodometric method suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds, both solid and dissolved. These compounds are common in some sample types, such as refinery waste and boiler feed water.

4.5 High levels of color and turbidity may interfere.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.3.3 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3.4 Hydrogen sulfide (H_2S) gas is generated by the addition of sulfuric acid. Inhalation of H_2S gas can cause headache, dizziness, nausea, and unconsciousness and potentially death.

5.3.5 Acid should be added dropwise until the sample reactivity is observed, particularly for soil samples and discolored water samples. If a strong reaction is observed, increase stirring rate and continue to add slowly.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Formaldehyde	Toxic	0.75 ppm (TWA)	Inhalation may result in spasm, inflammation, and edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. Extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Iodine	Poison Corrosive Oxidizer	0.1 ppm-Ceiling	Vapors severely irritate and can burn the mucous membranes and respiratory tract. Liquid contact may cause blistering burns, irritation, and pain. Vapors may be severely irritating to the skin. Vapors are severely irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Sulfide	Corrosive	10 ppm (TWA) 15 ppm (STEL)	Will form hydrogen sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
Sodium Hydroxide	Corrosive	2 mg/m^3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and with greater exposures, it can cause burns that may result in permanent impairment of vision, even blindness.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Nitrogen gas.

6.2 Boiling Tubes

6.3 Dropping funnels, 50 mL

6.4 Scrubber bottle, 50 mL

6.5 Tygon tubing

6.6 Glass tubing

6.7 Smart-Dist capable of maintaining 70 °C ± 5 °C

6.8 5 mL autopipettor

6.9 Burette, 25 mL, Class A.

6.10 Bubbler Vessel, 50 mL

6.11 Assorted analytical glassware, as needed.

6.12 pH strips

6.13 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 All reagents listed herein shall be ACS Reagent Grade, unless specified otherwise.

7.2 Reagent Water**:** Water that is free of the substances that interfere with the analytical method. Water with a resistivity of 1 megohm-cm or greater. The Eurofins TestAmerica Denver house deionized water meets this requirement with a minimum resistivity of 10 megohm-cm.

7.3 Reagent sand, such as Ottawa sand for use as blank solid matrix.

7.4 Sulfuric acid, concentrated. (H_2SO_4).

7.5 Hydrochloric acid, concentrated (HCl).

7.6 Sodium Sulfide nonahydrate, $Na_2S \bullet 9H_2O$, crystals (SFD ICV STK or SFD CAL STK)

7.7 Sulfide Standard Solution (Na_2S), approximately 1,000 mg/L (SFD CAL INT or SFD ICV INT)

Select larger pieces of sodium sulfide nonahydrate (SFD CAL STK) rather than small pieces that are more likely to have been oxidized. Spray the crystals with reagent water to remove the cloudy film covering it. Dry with a Kimwipe® as quickly as possible to limit the amount of crystal dissolved then weigh approximately 4 g of the crystals. Record the exact weight of sodium sulfide used. Dissolve the crystals in reagent water and add to a 500 mL volumetric flask to which 2 mL of 50% sodium hydroxide has been added. Dilute to volume with reagent water. The target concentration should be 1,100 – 1,200 mg/L. Store the solution in a glass amber bottle and keep refrigerated when not in use. Replace the standard when the daily standardization concentration is reduced to 800 mg/L.

7.8 Zinc Acetate Dihydrate, (ZINC ACETATE)

Zinc acetate dihydrate, 99.9%, solid, purchased directly from vendor. .

7.9 Zinc Acetate Solution, 0.5 M (znac)

Dissolve 110 g of zinc acetate dihydrate (Section 7.8) in 200 mL of reagent water. Add 1 mL of concentrated HCl and dilute to 1 liter.

7.10 Formaldehyde, 37% (formalin)

Obtain solution from a commercial vendor.

7.11 Hydrochloric Acid, 6 N (1:1) (HCL Sol)

Very carefully and slowly, and with constant mixing, add 250 mL of concentrated hydrochloric acid (HCl) to 250 mL of reagent water and mix. Allow to cool before use.

7.12 Starch Indicator (Starch Ind)

7.12.1 The starch indicator solution is purchased commercially.

7.12.2 Alternatively, to prepare the indicator solution in the lab, dissolve 2 g of laboratory-grade soluble starch and 2.0 g of salicylic acid, as a preservative, in 100 mL of hot reagent water. Mix, cool, and store in a labeled poly bottle.

7.13 Sodium Thiosulfate, 0.0250 N (Na thio)

This solution is purchased as a standardized 0.0250 N solution.

7.14 Iodine Solution, 0.0250 N (Iod)

This solution is purchased as a standardized 0.0250 N solution.

7.15 Sodium Hydroxide (NaOH), 50%

7.15.1 This solution is usually purchased from commercial sources.

7.15.2 If prepared in the lab, very carefully and slowly, with constant stirring, add 250 g of NaOH to 200 mL of reagent water in a beaker.

CAUTION: This solution gets very hot. Add the NaOH to the water slowly to allow the heat to dissipate.

7.15.3 Allow to cool, then transfer the solution to a 500 mL volumetric flask and dilute to volume with reagent water.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	HDPE	250 mL	NaOH/Zn Acetate pH>9 Cool, ≤ 6 °C	7 Days	SW-846
Soil	Glass	4 oz. jar	Cool, ≤ 6 °C	7 Days	SW-846

NOTE: Samples should be collected with a minimum of aeration. **The sample bottle should be filled completely, excluding all head space, and sealed.** Analysis should commence as soon as possible, and samples should be kept in a cool, dark place until analysis begins.

8.1 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the Eurofins TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply.

8.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver Policy DV-QA-003P *Quality Control Program*.

8.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver Policy DV-QA-024P *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM

unless otherwise stated. Any deviation or exceptions from QSM requirements must have prior approval in the project requirements.

8.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via method comments in TALS and in the Quality Assurance Summaries (QAS) available in the public folders.

8.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 *Non-Conformance and Corrective Action System*. This is in addition to the corrective actions described in the following sections.

8.2 Sample QC - The following quality control samples are prepared with each batch of samples.

8.2.1 Method Blank

A minimum of one method blank must be included in each QC batch of 20 or fewer field samples. The method blank consists of reagent water for batches of aqueous samples or Ottawa sand for batches of solid samples. Prepare and analyze the blank in the same manner as samples.

Acceptance criteria: The result for the method blank must be less than one-half the reporting limit for the analyte of interest or less than 10% of the lowest analyte concentration found in the associated samples, whichever is higher. In the latter case, an NCM must be generated explaining the analyte detection in the blank

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed after first checking all reagents and glassware for sources of contamination, checking the condition and cleanliness of the burette delivering the titrant, and correcting any problems found.

If the analyte was <u>not</u> detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

If there is insufficient sample for reanalysis, an NCM must be prepared and the client contacted by the laboratory Project Manager.

8.2.2 Laboratory Control Sample (LCS)

A minimum of one LCS must be included in each QC batch of 20 or fewer field samples. The LCS is prepared by spiking 50 mL of reagent water or 10 g of Ottawa sand with 1 mL of the Sulfide Standard Solution (Section 7.7). The nominal spike concentration for the water LCS is 20 mg/L and for the solid LCS is 100 mg/Kg. The LCS is analyzed in the same manner as the sample distillates. A duplicate LCS (LCSD) may be prepared and analyzed to provide a measure of analytical precision if required by the client or project and in cases where there is insufficient sample to prepare an MS and MSD.

Acceptance criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean and must be no wider than the limits specified in the reference methods.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed. If reanalysis is not possible, an NCM must be prepared and the client contacted by the laboratory Project Manager.

8.2.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair is required with each batch of 20 or fewer samples for total sulfide. The MS and MSD are prepared by adding 1 mL of the Sulfide Standard Solution to the sample aliquot required (Section 7.7). If insufficient sample volume is available for the preparation of an MS/MSD or a sample duplicate, a duplicate LCS (or sample duplicate) must be analyzed.

Acceptance Criteria: The recovery results for the MS and MSD must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean. The relative percent difference (RPD) between the MS and MSD, or between the sample and sample duplicate, must be less than

the established RPD limit, which is set at 3 standard deviations above the historical mean.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).

- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 *Non-Conformance and Corrective Action system*. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Record verification of volumetric pipettes and the repipettors used in this procedure in the designated spreadsheets.

10.4 Sample Preparation

10.4.1 Prepare the boiling tube by adding 47 mL of reagent water to a 50 mL bottle. Add 2 mL of zinc acetate solution and 1 mL of 37% formaldehyde (formalin).

10.4.2 Prepare reaction vessels, as follows:

10.4.2.1 Solids and Sludges

10.4.2.1.1 Weigh 10 g of soil or sludge into a boiling flask. Add 50 mL of reagent water and stopper immediately. The sample weight can be reduced to 5 g if the matrix absorbs water or does not stir efficiently.

10.4.2.2 Liquids

10.4.2.2.1 Check the pH of the sample with wide-range pH paper and record on the benchsheet. Shake the sample container to suspend any solids and then quickly transfer 50 mL of sample with a graduated cylinder into a boiling flask.

10.4.3 Assemble the apparatus as shown in Attachment 1. Connect the addition funnel to the reaction flask. Connect the nitrogen stream to the flask as shown in Attachment 1. Be sure all fittings and joints are air-tight.

10.4.4 Adjust the nitrogen flow to about 5 bubbles per second in the scrubber bottle.

10.4.5 Add sulfide standard solution by pipette to the LCS and MS/MSD (if MS/MSD are analyzed). Keep the tip of the pipette beneath the water surface and seal the vessel up tightly after addition. Spiking is normally done with 1 mL of sulfide standard solution (Section 7.7).

NOTE: Be sure the location of the MB and LCS are rotated amongst all distillation positions over time to demonstrate cleanliness of all glassware and proper functioning of hotplates. Make a note in either the Batch Comments or in the Worksheet comments regarding the positions used in each batch.

10.4.6 Once the gas flow has stabilized, purge the system for 15 minutes.

10.4.7 Add 5mL of concentrated sulfuric acid into the top of the addition funnel. Open the addition funnel so that the concentrated sulfuric acid drips slowly into the sample at a rate of approximately 5mL/min. Some samples may react violently with the acid, in which case the addition rate of acid may be decreased.

10.4.8 Allow the reaction to proceed for 90 minutes, checking the temperature frequently. Record the start and end times of the distillation in the batch record.

10.4.9 Once the distillation is complete, use low level pH paper to check the pH of the spent solution in the reaction vessel. If the pH is not less than or equal to 1, reprepare the sample using more acid.

10.4.10 If the pH is less than or equal to 1, close the scrubber bottles and proceed to the analysis outlined in Section 10.6 for total sulfide determination by titration by Method 9034.

10.5 Calibration

10.5.1 Standardization of Sulfide Standard Solution

10.5.1.1 Pipette 20.0 mL of iodine solution (Section 7.14) into a 250 mL Erlenmeyer flask.

10.5.1.2 Add 2 mL of 6 N hydrochloric acid (Section 7.11).

10.5.1.3 Pipette 5.0 mL of the Sulfide Standard Solution (Section 7.7) into the flask, making sure that the delivery tip of the pipette is below the surface of the solution. Dilute to approximately 100 mL with reagent water.

10.5.1.4 Titrate with 0.0250 N sodium thiosulfate solution (Section 7.13) to a pale yellow straw color.

10.5.1.5 Add ~1 mL of starch indicator solution (Section 7.12) and swirl until a homogenous blue color develops.

10.5.1.6 Continue the titration until the blue color disappears. Record the volume of the sodium thiosulfate titrant used in the Sulfide by Titration Bench sheet (Attachment 3).

10.5.1.7 Calculate the sulfide concentration using the Sulfide by Titration Bench sheet (Attachment 3).

10.5.1.8 Perform the standardization titrations in duplicate. Use the average of the two results as the final sulfide concentration of the Sulfide Standard Solution.

10.6 Sample Analysis

10.6.1 Determination of Total Sulfide (Acid-Soluble Sulfides)

10.6.1.1 Add 0.4 mL of 6 N HCl (Section 7.11) to the distilled sample in the Erlenmeyer flask and swirl.

10.6.1.2 Using a calibrated pipette, dispense a known amount of 0.025 N iodine solution (Section 7.14) into the flask. The amount added

needs to be in excess of the amount needed to oxidize the sulfide. This is typically 1 mL for "ND" samples and 5 mL for samples with sulfide in the range of the LCS samples. Add enough iodine solution to turn the liquid in the flask a deep amber color. Record the volume of iodine used.

NOTE: There must be enough iodine to oxidize the entire amount of sulfide present as zinc sulfide precipitate in the scrubber solution. Use about 1.5 mL of the 0.0250 N iodine solution for every mg of sulfide estimated to be in the sample. The amount of iodine necessary can be estimated by the amount of precipitate present in the scrubber solution. After addition of the iodine, the solution should be a deep amber color. If, in the analyst's opinion, the amount of iodine needed to oxidize the sulfide present in the sample will take more than 25 mL, a smaller sample aliquot may be taken for analysis. This volume must be measured and recorded.

10.6.1.3 Add a stir bar and place the flask on a magnetic stirrer.

10.6.1.4 Titrate the solution with 0.0250 N sodium thiosulfate solution (Section 7.13) to a pale yellow color.

10.6.1.5 Add sufficient starch indicator solution (Section 7.12) to achieve a deep and homogeneous blue color. Typically this volume is approximately 1 mL.

10.6.1.6 Continue the titration until the blue color just disappears. Allow the sample to equilibrate to ensure the endpoint is not missed.

10.6.1.7 Record the volume of sodium thiosulfate solution used in TALS worksheet.

10.7 Troubleshooting and Maintenance (Distillation)

10.7.1 Ensure the Nitrogen inlet tube is below the sample surface and is not clogged.

10.7.2 Check the ground glass joints for light grease and not excessive grease.

10.7.3 Check the tubing for cracks or signs of frailty or excessive wear.

10.7.4 Ensure the inlet and outlet tubes for the nitrogen are not chipped.

11.0 Calculations / Data Reduction

11.1 Data for the standardization of sulfide standard solution are entered into the designated spreadsheet. Data for titration of samples and QC are entered directly into TALS.

11.2 All calculations are performed in the sulfide titration benchsheet or in TALS. For manual verification use the following calculations.

11.3 One mL of 0.0250 N iodine solution reacts with 0.4 mg of sulfide present in the titration flask. Use the following equation to calculate sulfide concentration:

$$S = \frac{[(A \times B) - (C \times D)] \times 16000}{V_s}$$ *Equation 1*

Where:

S = Concentration of sulfide in sample (mg/kg or mg/L)
A = Volume of iodine solution added (mL)
B = Normality of iodine solution
C = Volume of sodium thiosulfate solution (mL)
D = Normality of sodium thiosulfate solution
V_s = Volume (mL) or weight (g) of original sample aliquot

11.4 If the normality of the iodine solution is exactly the same as that of the sodium thiosulfate solution, i.e., 0.0250 N, the following calculation may be used instead of the equation in section 11.3:

$$S = \frac{(A - C) \times 400}{V_s}$$ *Equation 2*

Where:

S = Concentration of sulfide in sample (mg/kg or mg/L)
A = Volume of iodine solution added (mL)
C = Volume of sodium thiosulfate solution (mL)
V_s = Volume (mL) or weight (g) of original sample aliquot

11.5 LCS Percent Recovery

Use the following equation to calculate the percent recovery of sulfide in the LCS:

$$\text{LCS \%Recovery} = \frac{\text{Measured Value}}{\text{True Value}} \times 100\%$$ *Equation 3*

11.6 MS and MSD Percent Recovery

Use the following equation to calculate the percent recovery of the added sulfide in the MS and MSD samples:

$$\text{MS \%Recovery} = \frac{SSR - SR}{SA}$$ *Equation 4*

Where:

SSR = Spiked sample result (concentration of sulfide in the MS or MSD).
SR = Sample result (concentration of sulfide in the parent sample).

SA = Concentration of added spike (concentration of sulfide in the sample in addition to any native sulfide as the result of adding the spiking solution).

11.7 Relative Percent Difference (RPD)

Use the following equation to calculate the relative percent difference between two analytical results (e.g., MS and MSD, LCS and LCSD, sample and sample duplicate).

$$RPD = \frac{\left|(R_1 - R_2)\right|}{1/2\,(R_1 + R_2)} \times 100\% \qquad \textit{Equation 5}$$

Where:

R_1 = Result for first sample (MS, LCS, or sample).
R_2 = Result for duplicate sample (MSD, LCSD, or sample duplicate).

11.8 All data undergo first and second level review using a checklist. See SOP DV-QA-0020 *Data Review* for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

There is no true MDL for this method. For the purposes of method detection the smallest increment of titrated volume is calculated and used as the MDL. Whenever a new burette is used the volume is rechecked and put through the calculation. The current MDL value is maintained in TALS.

12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.2 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

12.2.3 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.4 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024 *Training*.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024 *Training*.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P *Waste Management Plan*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.2 Scrubber waste – Aqueous Acidic (Reactivity) - Waste Stream F

14.2.3 Contents of reaction vessel – Aqueous Acidic (Reactivity) - Waste Stream F

14.2.4 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

15.1.1 Method 9030B: Acid-Soluble and Acid-Insoluble Sulfides: Distillation, Revision 2, December 1996.

15.1.2 Method 9034, Titrimetric Procedure for Acid-Soluble and Acid Insoluble Sulfides, Revision 0, December, 1996.

16.0 Method Modifications

Item	Method	Modification
1	SW 9030B	In this SOP the distillation apparatus uses only one scrubber.
2	SW 9030B	For total sulfide, 5 mL of concentrated sulfuric acid is added to the system without first titrating the sample. After the 90 minute reaction period, the spent sample solution pH is measured. If the pH is not ≤ 1.0, the sample is re-distilled using additional sulfuric acid.
3	SW 9034	Rinse solution of standardized 0.0250 N iodine, 1 mL of 6 N HCl, and reagent water is not used in the above procedure. As samples are preserved with 1% sodium hydroxide and zinc acetate, and the total sulfides are preserved with formaldehyde and zinc acetate, rinsing the bottles with reagent water introduces very minimal loss of sulfide due to oxidation.
4	SW 9034	The sodium thiosulfate and iodine solutions are purchased standardized from a vendor and therefore are not standardized in the laboratory.
5	SW 9034	The Method 9034 procedure is to add the iodine solution into a 500 mL flask, bring to 100 mL with reagent water, add acid, and pipette the scrubber solutions from method 9030 under the iodine solution. The scrubber solutions are approximately 200 mL. Due to the large volume of scrubber solution, the laboratory transfers the scrubber solutions into a flask and then adds the acid and iodine solution.
6	SW 9030B	Soil samples are refrigerated upon receipt and are not preserved with zinc acetate. The laboratory uses the 7 day holding time stated in SW-846. Since sample composition and moisture content vary so widely, it is difficult to preserve the sample in the field as prescribed. It is recommended that the sample be taken with no headspace.
7	SW 9030B	Aqueous samples are measured volumetrically in graduated cylinders, not by weight as indicated in the source method. Generally the liquid samples received by the lab are water rather than non-aqueous wastes.

17.0 Attachments

Attachment 1: Distillation Apparatus
Attachment 2: Example Sample Preparation Benchsheet - TALS
Attachment 3: Example Titration Benchsheet – Excel Spreadsheet
Attachment 4: Example Titration Benchsheet – TALS

18.0 Revision History

This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files and available upon request.

- Revision 5, dated 27 August 2021
  - Annual review and method review
  - Updated copyright information
  - Changed TestAmerica to Eurofins TestAmerica throughout
  - Switched sections 10.4.5 and 10.4.6 so the system is purged after the addition of the sulfide standard solution.
  - Updated formatting and language throughout

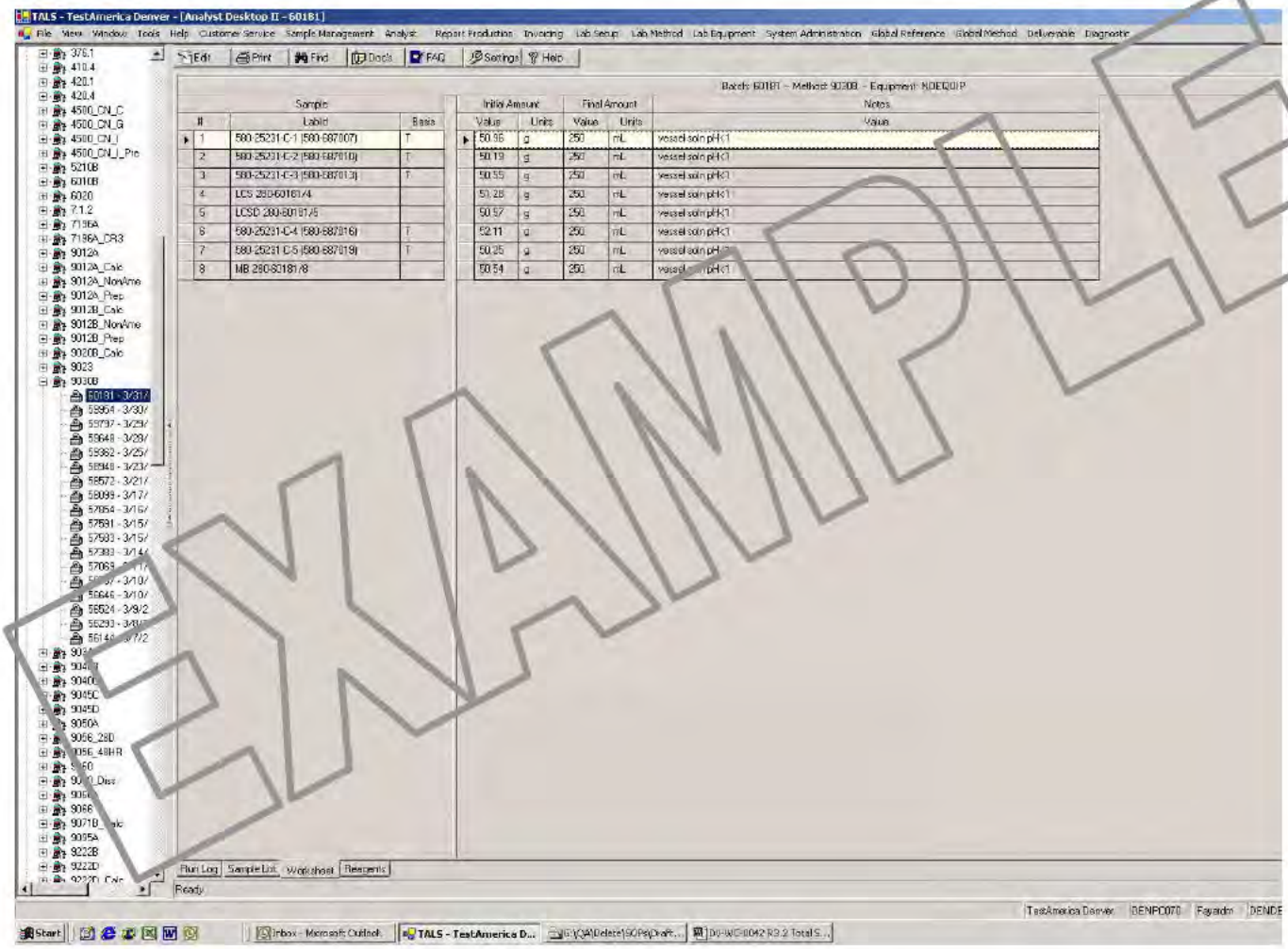
- Revision 4, dated 02 April 2020
  - Annual review
  - Updated copyright information

Attachment 1.

Distillation Apparatus

Attachment 2.

Example Sample Preparation Benchsheet - TALS



Attachment 3.
Example Titration Benchsheet – Excel Worksheet

Laboratory Bench Sheet

TestAmerica-Denver

Sulfide by Titration

Analyst:		SOP Information:	
Date:		Number:	
Titration Solutions		Calibration Information	
Solution 1:	Iodine	Source/Ver-Lot#:	
TALS ID		Prep Date:	
Normality:	0.025	Made By:	
Solution 2:	sodium thiosulfate	Concentration:	#DIV/0!
TALS ID		Expiration Date:	
Normality:	0.025		
	Starch Indicator		
TALS ID			

	CAL Volume	Buret Start	Buret End	ml Iodine	Final mL	Conc mg/L
CAL					0.00	#DIV/0!
CAL					0.00	#DIV/0!

R:\QA\Edit\FORMS\Wet Chemistry\Sulfide\Sulfide by Titration_Master.xls
Revision 6 - 1/31/2017

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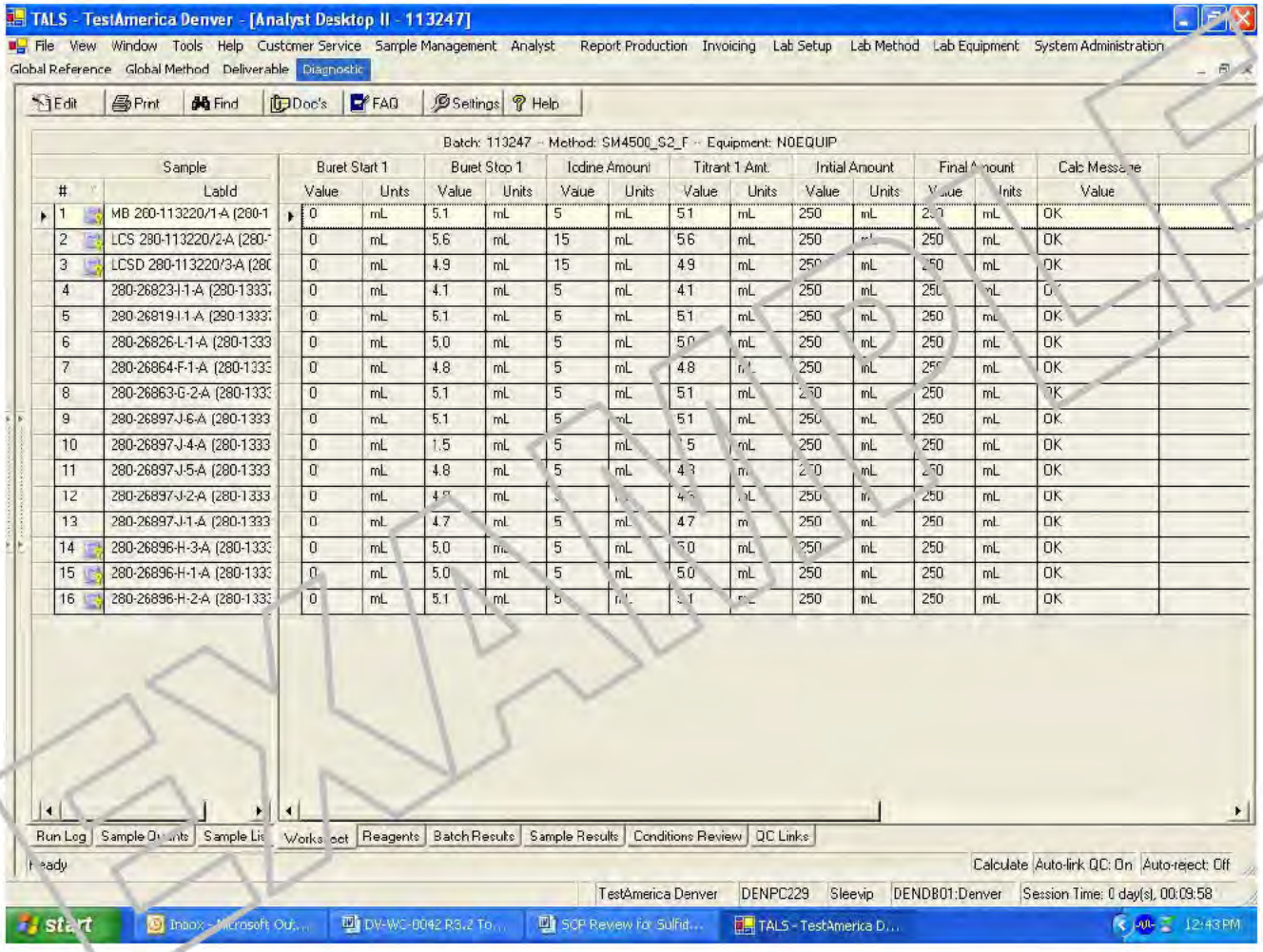
Attachment 4.

Example Titration Benchsheet - TALS



APPENDIX C

LABORATORY CERTIFICATIONS

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

EUROFINS TESTAMERICA DENVER
4955 Yarrow Street
Arvada, CO 80002
Maria Fayard Phone: 303-736-0166
www.testamericainc.com

ENVIRONMENTAL

Valid To: October 31, 2023 Certificate Number: 2907.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with the 2009 and 2016 TNI Environmental Testing Laboratory Standard, the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.4 of the DoD/DOE Quality Systems Manual for Environmental Laboratories), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, O_2), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, Total Organic Carbon, Total Organic Halide

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Metals			
Aluminum	EPA 200.7	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Antimony	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Arsenic	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Barium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Beryllium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Bismuth	----------	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Boron	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Cadmium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Calcium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Chromium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Cobalt	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Copper	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Iron	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Lead	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Lithium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Magnesium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Manganese	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Mercury	EPA 245.1	EPA 7470A	EPA 7471A/7471B
Molybdenum	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Nickel	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Potassium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Selenium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Silica	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Silicon	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Silver	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Sodium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Strontium	EPA 200.7	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Sulfur	----------	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Thallium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Thorium	----------	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Tin	EPA 200.7	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Titanium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Tungsten	----------	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Uranium	EPA 200.8	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Vanadium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Zinc	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Zirconium	----------	EPA 6010B/6010C/6010D	----------
Nutrients			
Nitrate (as N)	EPA 300.0 By calculation	EPA 300.0 EPA 9056/9056A By Calculation/Nitrate by Calc	EPA 9056/9056A By Calculation/Nitrate by Calc

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Nitrate-Nitrite (as N)	EPA 300.0 EPA 353.2	EPA 300.0 EPA 353.2 EPA 9056/9056A	EPA 9056/9056A
Nitrite (as N)	EPA 300.0 EPA 353.2 SM 4500-NO_2 B	EPA 300.0 EPA 353.2 EPA 9056/9056A SM 4500-NO_2 B	EPA 353.2 EPA 9056/9056A
Orthophosphate (as P)	EPA 300.0	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Total Phosphorus	EPA 365.1	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Demands			
Total Organic Carbon	----------	EPA 9060/9060A	EPA 9060/9060A
Total Organic Halides	----------	EPA 9020B	----------
Wet Chemistry			
Alkalinity (Total Bicarbonate, Carbonate, and Hydroxide Alkalinity)	SM 2320B	SM 2320B	SM 2320B
Ammonia	EPA 350.1	EPA 350.1	----------
Biological Oxygen Demand	SM 5210B	SM 5210B	----------
Bromide	EPA 300.0	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Chloride	EPA 300.0 SM 4500-CL E	EPA 300.0 EPA 9056/9056A SM 4500-CL E	EPA 9056/9056A
Chemical Oxygen Demand	EPA 410.4	EPA 410.4	----------
Conductivity	----------	EPA 9050/9050A	EPA 9050/9050A
Cyanide	----------	EPA 9012A/9012B	EPA 9012A/9012B
Ferrous Iron	SM 3500Fe B, D	SM 3500Fe B, D	----------
Fluoride	EPA 300.0	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Flashpoint	----------	EPA 1010A	----------
Hexavalent Chromium	----------	EPA 7196A	EPA 7196A
Hardness, Total	SM 2340C	SM 2340C	----------
pH	SM 4500 H+B	EPA 9040B/9040C	EPA 9045C/9045D
Oil and Grease (HEM and SGT-HEM)	----------	EPA 1664A/1664B	----------
Percent Moisture	----------	----------	ASTM D2216
Perchlorate	----------	EPA 6860	EPA 6860
Phenols	----------	EPA 9066	----------
Solids, Total	----------	----------	SM 2540B
Solids, Total Suspended	SM 2540D	SM 2540D	SM 2540D
Solids, Total Dissolved	SM 2540C	SM 2540C	SM 2540C
Sulfate	EPA 300.0 SM 4500-SO4 E	EPA 300.0 EPA 9056/9056A SM 4500-SO4 E	EPA 9056/9056A
Sulfide, Total	SM 4500S2 D	EPA 9034/SM 4500S2 D	EPA 9034
Sulfide	----------	EPA 9030B	EPA 9030B

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Total Kjeldahl Nitrogen	EPA 351.2	EPA 351.2	EPA 351.2
Purgeable Organics (Volatiles)			
1,1,1,2-Tetrachloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,1-Trichloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,2,2-Tetrachloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,2-Trichloro-1,2,2-trifluoroethane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,2-Trichloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1-Dichloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1-Dichloropropene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2 Dibromoethane (EDB)	EPA 624/624.1	EPA 8260B/8260C/8260D EPA 8011	EPA 8260B/8260C/8260D EPA 8011
1,2,3-Trichlorobenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2,3-Trichloropropane	EPA 624/624.1	EPA 8260B/8260C/8260D EPA 8011	EPA 8260B/8260C/8260D EPA 8011
1,2,3-Trimethylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2,4-Trichlorobenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2,4-Trimethylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dibromo-3-chloropropane (DBCP)	EPA 624/624.1	EPA 8260B/8260C/8260D EPA 8011	EPA 8260B/8260C/8260D EPA 8011
1,2-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dichloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dichloropropane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Xylene (o-Xylene)	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/OK DEQ GRO
1,3,5-Trichlorobenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3,5-Trimethylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3-Dichloropropane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,4-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,4-Dioxane	EPA 624/624.1	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM
1-Chlorohexane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2,2-Dichloropropane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Butanone [Methyl Ethyl Ketone (MEK)]	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Chloro-1,3-butadiene (Chloroprene)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Chloroethyl Vinyl Ether	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Chlorotoluene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Hexanone	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Nitropropane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Pentanone	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
4-Chlorotoluene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
4-Isopropyltoluene (p-Cymene)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
4-Methyl-2-pentanone (MIBK)	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acetone	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acetonitrile	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acetylene	----------	RSK-175	----------
Acetylene Ethane	----------	RSK-175	----------
Acrolein	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acrylonitrile	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Allyl Chloride (3-Chloro-1-propene)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Benzene	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/OK DEQ GRO
Bromobenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromochloromethane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromodichloromethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromoform	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromomethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Butadiene	----------	EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D SIM
Carbon Disulfide	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Carbon Tetrachloride	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chlorobenzene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chloroform	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chloromethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
cis-1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
cis-1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
cis-1,4-Dichloro-2-butene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Cyclohexane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Cyclohexanone	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dibromochloromethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dibromomethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dichlorodifluoromethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dichlorofluoromethane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Diethyl Ether (Ethyl Ether)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Di-isopropylether (Isopropyl ether)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethane	----------	RSK-175	----------
Ethanol	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethyl Acetate	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethyl Benzene	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/OK DEQ GRO
Ethyl Methacrylate	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethyl Tert-Butyl Ether	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethylene (Ethene)	----------	RSK-175	----------
Gas Range Organics (GRO)	----------	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO/NWTPH-Gx	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO/NWTPH-Gx

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Hexachlorobutadiene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Hexane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Iodomethane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Isobutyl alcohol (2-Methyl-1-propanol)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Isopropyl Alcohol	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Isopropylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
m+p-Xylene	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/ K DEQ GRO
Methacrylonitrile	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methane	----------	RSK-175	----------
Methyl Acetate	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methyl Cyclohexane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methyl Methacrylate	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methyl Tert-Butyl Ether (MtBE)	EPA 624/624.1	EPA 8260B/8260C/8260D OK DEQ GRO	EPA 8260B/8260C/8260D OK DEQ GRO
Methylene Chloride	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Naphthalene	EPA 624/624.1	EPA 8260B/8260C/8260D OK DEQ GRO	EPA 8260B/8260C/8260D OK DEQ GRO
n-Butyl Alcohol (n-Butanol)	----------	EPA 8260B/8260C/8260D EPA 8015B/8015C	EPA 8260B/8260C/8260D EPA 8015B/8015C
n-Butylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
n-Propylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Pentachloroethane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Propionitrile	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
sec-Butylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Styrene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
tert-Butyl Alcohol (2-Methyl-2-propanol)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
tert-Butylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Tetrachloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Tetrahydrofuran	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Toluene	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/OK DEQ GRO
Total Petroleum Hydrocarbons (TPH)	EPA 1664A/1664B	EPA 1664A/1664B	----------
trans-1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
trans-1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
trans-1,4-Dichloro-2-butene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Trichloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Trichlorofluoromethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Vinyl Acetate	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Vinyl Chloride	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Xylenes, Total	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/OK DEQ GRO
Extractable Organics (Semivolatiles)			
1,1-Biphenyl	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
1,2,4,5-Tetrachlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,2,4-Trichlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,2-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,2-Diphenylhydrazine (Azobenzene)	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,3,5-Trinitrobenzene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,3-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,3-Dinitrobenzene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,4-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,4-Dinitrobenzene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,4-Dioxane	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,4-Naphthoquinone	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1-Chloronaphthalene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1-Methylnaphthalene	----------	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
1-Naphthylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,2-oxybis(1-chloropropane) [bis (2-Chloroisopropyl) Ether]	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,3,4,6-Tetrachlorophenol	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4,5-Trichlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4,6-Tribromophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4,6-Trichlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4-Dichlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4-Dimethylphenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4-Dinitrophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4-Dinitrotoluene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,6-Dichlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,6-Dinitrotoluene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Acetylaminofluorene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Chloronaphthalene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Chlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-methyl-4,6-Dinitrophenol (Dinoseb)	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Methylnaphthalene	----------	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
2-Methylphenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Naphthylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Nitroaniline	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Nitrophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Picoline	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-sec-butyl-4,6-Dinitrophenol	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
3,3'-Dichlorobenzidine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
3,3-Dimethylbenzidine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
3+4-Methylphenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
3-Methylcholanthrene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
3-Nitroaniline	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4,6-Dinitro-2-methylphenol	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
4-Aminobiphenyl	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-Bromophenyl phenyl ether	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-chloro-3-Methylphenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-Chloroanilene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-Chlorophenyl phenyl ether	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-Nitroaniline	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-Nitrophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
5-nitro-o-Toluidine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
7,12-Dimethylbenz(a)anthracene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Acenaphthene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Acenaphthylene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Acetophenone	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Alachlor	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
alpha-, alpha-Dimethylphenethylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Aniline	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Anthracene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Aramite	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Atrazine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Azobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Benzaldehyde	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Benzidine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Benzo(a)anthracene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Benzo(a)pyrene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Benzo(b)fluoranthene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Benzo(ghi)perylene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Benzo(k)fluoranthene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Benzoic Acid	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Benzyl Alcohol	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
bis (2-Chloroethoxy) Methane	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
bis (2-Chloroethyl) Ether	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
bis (2-Ethylhexyl) Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
butyl Benzyl Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Caprolactam	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Carbazole	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Chlorobenzilate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Chrysene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Cresols	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Diallate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Dibenzo (a,h) anthracene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Dibenzofuran	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Diesel Range Organics (DRO)	----------	EPA 8015B/8015C/8015D AK102/8015D/OK DEQ DRO/NWTPH-Dx	EPA 8015B/8015C/8015D AK102/8015D/OK DEQ DRO/NWTPH-Dx
Diethyl Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Dimethoate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Dimethyl Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
di-n-butyl Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
di-n-octyl Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
woDiphenylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Disulfoton	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Ethyl Methanesulfonate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Famphur	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Fluorene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Fluoroanthene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Hexachlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Hexachlorobutadiene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Hexachlorocyclopentadiene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Hexachloroethane	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Hexachlorophene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Hexachloropropene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Indeno (1,2,3-cd) pyrene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Isodrin	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Isophorone	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Isosafrole	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Methapyrilene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Methyl Methane Sulfonate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Motor Oil (Residual Range Organics)	----------	EPA 8015B/8015C/8015D AK103/OK DEQ RRO	EPA 8015B/ 8015C/8015D AK103/ OK DEQ RRO
Naphthalene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Nitrobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Nitroquinoline-1-oxide (4-Nitroquinoline-1-oxide)	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosodiethylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosodimethylamine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosodi-n-butylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosodi-n-propylamine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosodiphenylamine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosomethylethylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosomorpholine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
N-Nitrosopiperidine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosopyrrolidine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
o,o,o-triethyl Phosphorothioate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
o-Toluidine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Parathion, ethyl	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Parathion, methyl	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
p-Dimethylaminoazobenzene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pentachlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pentachloroethane	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pentachloronitobenzene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pentachlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8321A/8321B	EPA 8270C/8270D/8270E EPA 8321A/8321B
Phenacetin	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Phenanthrene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Phenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Phorate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
p-Phenylene Diamine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pronamide	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pyrene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Pyridine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Safrole	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Sulfotepp	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Thionazin	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Tributyl phosphate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pesticides/Herbicides/PCBs			
2,4,5-T	----------	EPA 8151A	EPA 8151A
2,4,5-TP	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-D	----------	EPA 8151A	EPA 8151A
2,4-DB	----------	EPA 8321A/8321B	EPA 8321A/8321B
4,4'-DDD	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDE	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDT	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Aldrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
alpha-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
alpha-Chlordane (cis-Chlordane)	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Atrazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos ethyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos methyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
beta-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Bolstar	----------	EPA 8141A/8141B	EPA 8141A/8141B
Chlordane (technical)	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Chloropyrifos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Coumaphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dalapon	----------	EPA 8151A EPA 8321A/8321B	EPA 8151A EPA 8321A/8321B

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
delta-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Demeton, total	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton-O	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton-S	----------	EPA 8141A/8141B	EPA 8141A/8141B
Diazinon	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dicamba	----------	EPA 8151A EPA 8321A/8321B	EPA 8151A EPA 8321A/8321B
Dichloroprop	----------	EPA 8151A EPA 8321A/8321B	EPA 8151A EPA 8321A/8321B
Dichlorovos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dieldrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Dimethoate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dinoseb (2-methyl-4,6-Dinitrophenol)	----------	EPA 8151A EPA 8321A/8321B	EPA 8321A/8321B
Disulfoton	----------	EPA 8141A/8141B	EPA 8141A/8141B
Endonsulfan sulfate	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan I	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan II	EPA 608/608.3	EPA 8081A /8081B	EPA 8081A/8081B
Endrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin aldehyde	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin ketone	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
EPN	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethoprop	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethyl Parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Famphur	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fensulfothion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fenthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
gamma-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
gamma-Chlordane (trans-Chlordane)	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor epoxide	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Hexachlorobenzene	----------	EPA 8081A/8081B	EPA 8081A/8081B
Malathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
MCPA	----------	EPA 8151A EPA 8321A/8321B	EPA 8151A EPA 8321A/8321B
MCPP	----------	EPA 8151A EPA 8321A/8321B	EPA 8151A EPA 8321A/8321B
Merphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Methoxychlor	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Methyl parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Mevinphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Naled	----------	EPA 8141A/8141B	EPA 8141A/8141B
o,o,o-Triethylphos Phorothioate	----------	EPA 8141A/8141B	EPA 8141A/8141B
PCB-1016 (Arochlor)	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1221	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1232	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
PCB-1242	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1248	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1254	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1260	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1262	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1268	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
Pentachlorophenol	----------	EPA 8151A	EPA 8151A
Phorate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Phosmet	----------	EPA 8141A/8141B	EPA 8141A/8141B
Picrolam	----------	EPA 8151A	EPA 8151A
Propazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ronnel	----------	EPA 8141A/8141B	EPA 8141A/8141B
Simazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Stirophos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Sulfotepp	----------	EPA 8141A/8141B	EPA 8141A/8141B
Thionazin	----------	EPA 8141A/8141B	EPA 8141A/8141B
Tokuthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Total PCBs	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
Toxaphene	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Trichloronate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Explosives			
1,3,5-Trinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8330B EPA 8321A/8321B	EPA 8330B EPA 8321A/8321B
2,4-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,4-Diamino-6-nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,6-Diamino-4-nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-amino-4,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-amino-2,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Nitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitroglycerin	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitroguanidine	----------	EPA 8321A/8321B	EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Picric acid	----------	EPA 8330A/8330B	EPA 8330A/8330B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
DNX (Hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
MNX (Hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
TNX (hexahydro-1,3,5-trinitroso-1,3,5-triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Triaminotrinitrobenzene (TATB)	----------	EPA 8330B EPA 8321A/8321B	EPA 8330B EPA 8321A/8321B
Explosives LC/MS/MS			
1,3,5-Trinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Amino-4,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Amino-2,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
DNX (hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
MNX (hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitroglycerin	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitroguanidine	----------	EPA 8321A/8321B	EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8321A/8321B	EPA 8321A/8321B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8321A/8321B	EPA 8321A/8321B
TNX (hexahydro-1,3,5-trinitroso-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Tris(o-cresyl)phosphate	----------	EPA 8321A/8321B	EPA 8321A/8321B
Triaminotrinitrobenzene (TATB)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Chemical Warfare Agents			
Thiodiglycol (2,2'-Thiodiethanol)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Hazardous Waste Characteristics			
Conductivity	SM 2510B	EPA 9050A	EPA 9050A
Corrosivity	SM 4500 H+B	EPA 9040B/9040C	EPA 9045C/9045D
Paint filter liquids test	----------	EPA 9095A	EPA 9095A
Synthetic Precipitation Leaching Procedure (SPLP)	----------	EPA 1312	EPA 1312
Toxicity Characteristic Leaching Procedure	----------	EPA 1311	EPA 1311
California Waste Extraction Test	----------	CA WET	CA WET
Turbidity	EPA 180.1	----------	----------
Organic Prep Methods			
Continuous liquid-liquid extraction	----------	EPA 3520C	----------
Microwave extraction	----------	----------	EPA 3546
Separatory funnel liquid-liquid extraction	----------	EPA 3510C	----------
Solid phase extraction	----------	EPA 3535A	----------
Soxhlet extraction	----------	----------	EPA 3540C
Ultrasonic extraction	----------	----------	EPA 3550B/3550C
Volatiles purge and trap	----------	EPA 5030B	EPA 5030A EPA 5035/5035A
Waste dilution	----------	EPA 3580A	EPA 3580A
Organic Cleanup Procedures			
Florisil Cleanup	----------	EPA 3620B	EPA 3620B
Florisil Cleanup	----------	EPA 3620C	EPA 3620C
Sulfur Cleanup	----------	EPA 3660A/EPA 3660B	EPA 3660A/EPA 3660B
Sulfuric Acid/Permanganate Cleanup	----------	EPA 3665A	EPA 3665A
Metals Digestion			
Acid Digestion for Total Metals	----------	EPA 3010A	----------
Acid Digestion for Total Metals	----------	EPA 3020A	----------
Acid Digestion of Sediments, Sludges and Soils	----------	----------	EPA 3050B
Acid Digestion Total Recoverable or Dissolved Metals	----------	EPA 3005A	----------

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

WYOMING STORAGE TANK PROGRAM

Parameter/Analyte	**Method(s)**
Metals	
Cadmium	EPA 6010C/6010D
Chromium	EPA 6010C/6010D
Lead	EPA 6010C/6010D
Wet Chemistry	
Hexavalent chromium	EPA 7196A
Pureable Organics (Volatiles)	
tert-Amyl Methyl Ether	EPA 8260B/8260C
Benzene	EPA 8260B/8260C
tert-Butyl alcohol (2-Methyl-2-propanol)	EPA 8260B/8260C
1,2-Dichloroethane	EPA 8260B/8260C
Di-isopropylether	EPA 8260B/8260C
Ethyl benzene	EPA 8260B/8260C
Ethyl tert-butyl ether	EPA 8260B/8260C
Gas Range Organics (GRO)	EPA 8015B/8015C/8015D
Methyl tert-butyl ether (MTBE)	EPA 8260B/8260C
Naphthalene	EPA 8260B/8260C
Toluene	EPA 8260B/8260C
Xylenes, total	EPA 8260B/8260C
1,2-Xylene	EPA 8260B/8260C
m+p-Xylene	EPA 8260B/8260C
Extractable Organics (Semivolatiles)	
Diesel Range Organics (DRO)	EPA 8015B/8015C/8015D (WY: C10-C32)
Organic Prep Methods	
Volatiles Purge and Trap	EPA 5030B (water) /5030A (solids)

Accredited Laboratory

A2LA has accredited

EUROFINS TESTAMERICA DENVER

Arvada, CO

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2009 and 2016 TNI Environmental Testing Laboratory Standard, the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.4 of the DoD/DOE Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

Presented this 1st day of December 2021.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 2907.01
Valid to October 31, 2023

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

CERTIFICATE OF ACCREDITATION

ANSI National Accreditation Board

11617 Coldwater Road, Fort Wayne, IN 46845 USA

This is to certify that

TestAmerica Savannah

5102 LaRoche Avenue
Savannah, GA 31404

has been assessed by ANAB and meets the requirements of international standard

ISO/IEC 17025:2017

and the

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V5.3)

while demonstrating technical competence in the field of

TESTING

Refer to the accompanying Scope of Accreditation for information regarding the types of activities to which this accreditation applies

L2463
Certificate Number

ANAB Approval

Certificate Valid Through: 09/22/2022
Version No. 003 Issued: 08/19/2019

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

ANSI National Accreditation Board

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017 AND U.S. DEPARTMENT OF DEFENSE (DOD) QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES (DOD QSM V5.3)

TestAmerica Savannah
5102 LaRoche Avenue
Savannah, GA 31404
Kim Chamberlain
912-354-7858

TESTING

Valid to: **September 22, 2022** Certificate Number: **L2463**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
General Chemistry	EPA 1664A	Oil and Grease
General Chemistry	EPA 1664A	Total Petroleum Hydrocarbons
General Chemistry	EPA 1664B	Oil and Grease
General Chemistry	EPA 1664B	Total Petroleum Hydrocarbons
ICP	EPA 6010C	Aluminum
ICP	EPA 6010C	Antimony
ICP	EPA 6010C	Arsenic
ICP	EPA 6010C	Barium
ICP	EPA 6010C	Beryllium
ICP	EPA 6010C	Boron
ICP	EPA 6010C	Cadmium
ICP	EPA 6010C	Calcium
ICP	EPA 6010C	Chromium
ICP	EPA 6010C	Cobalt
ICP	EPA 6010C	Copper
ICP	EPA 6010C	Iron

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP	EPA 6010C	Lead
ICP	EPA 6010C	Magnesium
ICP	EPA 6010C	Manganese
ICP	EPA 6010C	Molybdenum
ICP	EPA 6010C	Nickel
ICP	EPA 6010C	Potassium
ICP	EPA 6010C	Selenium
ICP	EPA 6010C	Silica
ICP	EPA 6010C	Silicon
ICP	EPA 6010C	Silver
ICP	EPA 6010C	Sodium
ICP	EPA 6010C	Strontium
ICP	EPA 6010C	Thallium
ICP	EPA 6010C	Tin
ICP	EPA 6010C	Titanium
ICP	EPA 6010C	Vanadium
ICP	EPA 6010C	Zinc
ICP	EPA 6010D	Aluminum
ICP	EPA 6010D	Antimony
ICP	EPA 6010D	Arsenic
ICP	EPA 6010D	Barium
ICP	EPA 6010D	Beryllium
ICP	EPA 6010D	Boron
ICP	EPA 6010D	Cadmium
ICP	EPA 6010D	Calcium
ICP	EPA 6010D	Chromium
ICP	EPA 6010D	Cobalt
ICP	EPA 6010D	Copper
ICP	EPA 6010D	Iron
ICP	EPA 6010D	Lead

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP	EPA 6010D	Magnesium
ICP	EPA 6010D	Manganese
ICP	EPA 6010D	Molybdenum
ICP	EPA 6010D	Nickel
ICP	EPA 6010D	Potassium
ICP	EPA 6010D	Selenium
ICP	EPA 6010D	Silica
ICP	EPA 6010D	Silicon
ICP	EPA 6010D	Silver
ICP	EPA 6010D	Sodium
ICP	EPA 6010D	Strontium
ICP	EPA 6010D	Thallium
ICP	EPA 6010D	Tin
ICP	EPA 6010D	Titanium
ICP	EPA 6010D	Vanadium
ICP	EPA 6010D	Zinc
ICP/MS	EPA 6020A	Aluminum
ICP/MS	EPA 6020A	Antimony
ICP/MS	EPA 6020A	Arsenic
ICP/MS	EPA 6020A	Barium
ICP/MS	EPA 6020A	Beryllium
ICP/MS	EPA 6020A	Boron
ICP/MS	EPA 6020A	Cadmium
ICP/MS	EPA 6020A	Calcium
ICP/MS	EPA 6020A	Chromium
ICP/MS	EPA 6020A	Cobalt
ICP/MS	EPA 6020A	Copper
ICP/MS	EPA 6020A	Iron
ICP/MS	EPA 6020A	Lead
ICP/MS	EPA 6020A	Magnesium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020A	Manganese
ICP/MS	EPA 6020A	Mercury
ICP/MS	EPA 6020A	Molybdenum
ICP/MS	EPA 6020A	Nickel
ICP/MS	EPA 6020A	Potassium
ICP/MS	EPA 6020A	Selenium
ICP/MS	EPA 6020A	Silver
ICP/MS	EPA 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 6020A	Thallium
ICP/MS	EPA 6020A	Tin
ICP/MS	EPA 6020A	Titanium
ICP/MS	EPA 6020A	Vanadium
ICP/MS	EPA 6020A	Zinc
ICP/MS	EPA 6020B	Aluminum
ICP/MS	EPA 6020B	Antimony
ICP/MS	EPA 6020B	Arsenic
ICP/MS	EPA 6020B	Barium
ICP/MS	EPA 6020B	Beryllium
ICP/MS	EPA 6020B	Boron
ICP/MS	EPA 6020B	Cadmium
ICP/MS	EPA 6020B	Calcium
ICP/MS	EPA 6020B	Chromium
ICP/MS	EPA 6020B	Cobalt
ICP/MS	EPA 6020B	Copper
ICP/MS	EPA 6020B	Iron
ICP/MS	EPA 6020B	Lead
ICP/MS	EPA 6020B	Magnesium
ICP/MS	EPA 6020B	Manganese
ICP/MS	EPA 6020B	Mercury

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020B	Molybdenum
ICP/MS	EPA 6020B	Nickel
ICP/MS	EPA 6020B	Potassium
ICP/MS	EPA 6020B	Selenium
ICP/MS	EPA 6020B	Silver
ICP/MS	EPA 6020B	Sodium
ICP/MS	EPA 6020B	Strontium
ICP/MS	EPA 6020B	Thallium
ICP/MS	EPA 6020B	Tin
ICP/MS	EPA 6020B	Titanium
ICP/MS	EPA 6020B	Vanadium
ICP/MS	EPA 6020B	Zinc
Colorimetry	EPA 7196A	Chromium 3+
Colorimetry	EPA 7196A	Chromium 6+
CVAA	EPA 7470A	Mercury
GC/ECD	EPA 8011	1,2,3-Trichloropropane
GC/ECD	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/FID	EPA 8015C	#2 Diesel Fuel
GC/FID	EPA 8015C	Diesel Range Organics
GC/FID	EPA 8015C	Gasoline Range Organics
GC/FID	EPA 8015C	Kerosene
GC/FID	EPA 8015C	Mineral Spirits
GC/FID	EPA 8015C	Motor Oil
GC/FID	EPA 8015C	Oil Range Organics
GC/FID	EPA 8015C-DAI	2,2'-Oxybisethanol
GC/FID	EPA 8015C-DAI	2-Butoxyethanol
GC/FID	EPA 8015C-DAI	2-Propoxy ethanol
GC/FID	EPA 8015C-DAI	Cellosolve acetate
GC/FID	EPA 8015C-DAI	Di-propylene glycol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/FID	EPA 8015C-DAI	Di-propylene glycol methyl ether
GC/FID	EPA 8015C-DAI	Ethanol
GC/FID	EPA 8015C-DAI	Ethyl acetate
GC/FID	EPA 8015C-DAI	Ethylene glycol
GC/FID	EPA 8015C-DAI	Isoamyl acetate
GC/FID	EPA 8015C-DAI	Isobutanol
GC/FID	EPA 8015C-DAI	Isobutyl acetate
GC/FID	EPA 8015C-DAI	Isopropanol
GC/FID	EPA 8015C-DAI	Isopropyl acetate
GC/FID	EPA 8015C-DAI	Methanol
GC/FID	EPA 8015C-DAI	Methyl acetate
GC/FID	EPA 8015C-DAI	n-Butanol
GC/FID	EPA 8015C-DAI	n-Butyl acetate
GC/FID	EPA 8015C-DAI	n-Heptanol
GC/FID	EPA 8015C-DAI	n-Propanol
GC/FID	EPA 8015C-DAI	n-Propyl acetate
GC/FID	EPA 8015C-DAI	Phenol
GC/FID	EPA 8015C-DAI	Propylene glycol
GC/FID	EPA 8015C-DAI	sec-Butanol
GC/FID	EPA 8015C-DAI	sec-Butyl acetate
GC/FID	EPA 8015C-DAI	Tert-amyl alcohol
GC/FID	EPA 8015C-DAI	tert-Butyl alcohol
GC/FID	EPA 8015C-DAI	Tetraethylene glycol
GC/FID	EPA 8015C-DAI	Triethylene glycol
GC/ECD	EPA 8081B	2,4' DDE
GC/ECD	EPA 8081B	2,4'-DDD
GC/ECD	EPA 8081B	2,4'-DDT
GC/ECD	EPA 8081B	4,4' DDE
GC/ECD	EPA 8081B	4,4'-DDD
GC/ECD	EPA 8081B	4,4'-DDT

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC
GC/ECD	EPA 8081B	beta-BHC
GC/ECD	EPA 8081B	Chlordane (alpha)
GC/ECD	EPA 8081B	Chlordane (gamma)
GC/ECD	EPA 8081B	Chlordane (technical)
GC/ECD	EPA 8081B	Chlorobenzilate
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I (alpha)
GC/ECD	EPA 8081B	Endosulfan II (beta)
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin ketone
GC/ECD	EPA 8081B	gamma-BHC
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Isodrin
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Toxaphene
GC/ECD	EPA 8082A	PCB-1016
GC/ECD	EPA 8082A	PCB-1221
GC/ECD	EPA 8082A	PCB-1232
GC/ECD	EPA 8082A	PCB-1242
GC/ECD	EPA 8082A	PCB-1248
GC/ECD	EPA 8082A	PCB-1254
GC/ECD	EPA 8082A	PCB-1260
GC/ECD	EPA 8082A	PCB-1262

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	PCB-1268
GC/ECD	EPA 8082A	PCBs, Total
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4,6-Trichlorophenol
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	2,6-Dichlorophenol
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	DCPA (Dacthal)
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Picloram
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,1-Trichloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethene
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane
GC/MS	EPA 8260B	1,2-Dichloroethene, Total
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,3-Dichloropropene, Total
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1,4-Dioxane
GC/MS	EPA 8260B	1-Chlorohexane
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone
GC/MS	EPA 8260B	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone
GC/MS	EPA 8260B	3-Chloro-1-propene
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Isopropyltoluene
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acetonitrile
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	BTEX, Total
GC/MS	EPA 8260B	Carbon disulfide
GC/MS	EPA 8260B	Carbon tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	Chloroprene
GC/MS	EPA 8260B	cis-1,2-Dichloroethene
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane
GC/MS	EPA 8260B	Diethyl ether
GC/MS	EPA 8260B	Ethanol
GC/MS	EPA 8260B	Ethyl benzene
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Furan
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Hexane
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isobutanol
GC/MS	EPA 8260B	Isopropyl ether
GC/MS	EPA 8260B	Isopropylbenzene
GC/MS	EPA 8260B	m & p-Xylene
GC/MS	EPA 8260B	Methacrylonitrile
GC/MS	EPA 8260B	Methyl acetate
GC/MS	EPA 8260B	Methyl cyclohexane
GC/MS	EPA 8260B	Methyl isobutyl ketone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260B	Methylene chloride
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Heptane
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	o-Xylene
GC/MS	EPA 8260B	Pentachloroethane
GC/MS	EPA 8260B	Propionitrile
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene
GC/MS	EPA 8260B	Tetrahydrofuran
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260B	Trichloroethene
GC/MS	EPA 8260B	Trichlorofluoromethane
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl chloride
GC/MS	EPA 8260B	Xylenes, total
GC/MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260C	1,1,2-Trichloroethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	1,1-Dichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethene
GC/MS	EPA 8260C	1,1-Dichloropropene
GC/MS	EPA 8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260C	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260C	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260C	1,2-Dichloroethane
GC/MS	EPA 8260C	1,2-Dichloroethene, Total
GC/MS	EPA 8260C	1,2-Dichloropropane
GC/MS	EPA 8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260C	1,3-Dichloropropane
GC/MS	EPA 8260C	1,3-Dichloropropene, Total
GC/MS	EPA 8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260C	1,4-Dioxane
GC/MS	EPA 8260C	1-Chlorohexane
GC/MS	EPA 8260C	2,2-Dichloropropane
GC/MS	EPA 8260C	2-Butanone
GC/MS	EPA 8260C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260C	2-Chlorotoluene
GC/MS	EPA 8260C	2-Hexanone
GC/MS	EPA 8260C	3-Chloro-1-propene
GC/MS	EPA 8260C	4-Chlorotoluene
GC/MS	EPA 8260C	4-Isopropyltoluene
GC/MS	EPA 8260C	Acetone
GC/MS	EPA 8260C	Acetonitrile

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Acrolein
GC/MS	EPA 8260C	Acrylonitrile
GC/MS	EPA 8260C	Benzene
GC/MS	EPA 8260C	Bromobenzene
GC/MS	EPA 8260C	Bromochloromethane
GC/MS	EPA 8260C	Bromodichloromethane
GC/MS	EPA 8260C	Bromoform
GC/MS	EPA 8260C	Bromomethane
GC/MS	EPA 8260C	BTEX, Total
GC/MS	EPA 8260C	Carbon disulfide
GC/MS	EPA 8260C	Carbon tetrachloride
GC/MS	EPA 8260C	Chlorobenzene
GC/MS	EPA 8260C	Chloroethane
GC/MS	EPA 8260C	Chloroform
GC/MS	EPA 8260C	Chloromethane
GC/MS	EPA 8260C	Chloroprene
GC/MS	EPA 8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260C	Cyclohexane
GC/MS	EPA 8260C	Dibromochloromethane
GC/MS	EPA 8260C	Dibromomethane
GC/MS	EPA 8260C	Dichlorodifluoromethane
GC/MS	EPA 8260C	Diethyl ether
GC/MS	EPA 8260C	Ethanol
GC/MS	EPA 8260C	Ethyl benzene
GC/MS	EPA 8260C	Ethyl methacrylate
GC/MS	EPA 8260C	Furan
GC/MS	EPA 8260C	Hexachlorobutadiene
GC/MS	EPA 8260C	Hexane
GC/MS	EPA 8260C	Iodomethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Isobutanol
GC/MS	EPA 8260C	Isopropyl ether
GC/MS	EPA 8260C	Isopropylbenzene
GC/MS	EPA 8260C	m & p-Xylene
GC/MS	EPA 8260C	Methacrylonitrile
GC/MS	EPA 8260C	Methyl acetate
GC/MS	EPA 8260C	Methyl cyclohexane
GC/MS	EPA 8260C	Methyl isobutyl ketone
GC/MS	EPA 8260C	Methyl methacrylate
GC/MS	EPA 8260C	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260C	Methylene chloride
GC/MS	EPA 8260C	Naphthalene
GC/MS	EPA 8260C	n-Butylbenzene
GC/MS	EPA 8260C	n-Heptane
GC/MS	EPA 8260C	n-Propylbenzene
GC/MS	EPA 8260C	o-Xylene
GC/MS	EPA 8260C	Pentachloroethane
GC/MS	EPA 8260C	Propionitrile
GC/MS	EPA 8260C	sec-Butylbenzene
GC/MS	EPA 8260C	Styrene
GC/MS	EPA 8260C	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260C	tert-Butylbenzene
GC/MS	EPA 8260C	Tetrachloroethene
GC/MS	EPA 8260C	Tetrahydrofuran
GC/MS	EPA 8260C	Toluene
GC/MS	EPA 8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260C	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260C	Trichloroethene
GC/MS	EPA 8260C	Trichlorofluoromethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Vinyl acetate
GC/MS	EPA 8260C	Vinyl chloride
GC/MS	EPA 8260C	Xylenes, total
GC/MS	EPA 8260D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,1-Trichloroethane
GC/MS	EPA 8260D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260D	1,1,2-Trichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethene
GC/MS	EPA 8260D	1,1-Dichloropropene
GC/MS	EPA 8260D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260D	1,2,3-Trichloropropane
GC/MS	EPA 8260D	1,2,4-Trichlorobenzene
GC/MS	EPA 8260D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260D	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260D	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260D	1,2-Dichlorobenzene
GC/MS	EPA 8260D	1,2-Dichloroethane
GC/MS	EPA 8260D	1,2-Dichloroethene, Total
GC/MS	EPA 8260D	1,2-Dichloropropane
GC/MS	EPA 8260D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260D	1,3-Dichlorobenzene
GC/MS	EPA 8260D	1,3-Dichloropropane
GC/MS	EPA 8260D	1,3-Dichloropropene, Total
GC/MS	EPA 8260D	1,4-Dichlorobenzene
GC/MS	EPA 8260D	1,4-Dioxane
GC/MS	EPA 8260D	1-Chlorohexane
GC/MS	EPA 8260D	2,2-Dichloropropane
GC/MS	EPA 8260D	2-Butanone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	2-Chloroethyl vinyl ether
GC/MS	EPA 8260D	2-Chlorotoluene
GC/MS	EPA 8260D	2-Hexanone
GC/MS	EPA 8260D	3-Chloro-1-propene
GC/MS	EPA 8260D	4-Chlorotoluene
GC/MS	EPA 8260D	4-Isopropyltoluene
GC/MS	EPA 8260D	Acetone
GC/MS	EPA 8260D	Acetonitrile
GC/MS	EPA 8260D	Acrolein
GC/MS	EPA 8260D	Acrylonitrile
GC/MS	EPA 8260D	Benzene
GC/MS	EPA 8260D	Bromobenzene
GC/MS	EPA 8260D	Bromochloromethane
GC/MS	EPA 8260D	Bromodichloromethane
GC/MS	EPA 8260D	Bromoform
GC/MS	EPA 8260D	Bromomethane
GC/MS	EPA 8260D	BTEX, Total
GC/MS	EPA 8260D	Carbon disulfide
GC/MS	EPA 8260D	Carbon tetrachloride
GC/MS	EPA 8260D	Chlorobenzene
GC/MS	EPA 8260D	Chloroethane
GC/MS	EPA 8260D	Chloroform
GC/MS	EPA 8260D	Chloromethane
GC/MS	EPA 8260D	Chloroprene
GC/MS	EPA 8260D	cis-1,2-Dichloroethene
GC/MS	EPA 8260D	cis-1,3-Dichloropropene
GC/MS	EPA 8260D	Cyclohexane
GC/MS	EPA 8260D	Dibromochloromethane
GC/MS	EPA 8260D	Dibromomethane
GC/MS	EPA 8260D	Dichlorodifluoromethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Diethyl ether
GC/MS	EPA 8260D	Ethanol
GC/MS	EPA 8260D	Ethyl benzene
GC/MS	EPA 8260D	Ethyl methacrylate
GC/MS	EPA 8260D	Furan
GC/MS	EPA 8260D	Hexachlorobutadiene
GC/MS	EPA 8260D	Hexane
GC/MS	EPA 8260D	Iodomethane
GC/MS	EPA 8260D	Isobutanol
GC/MS	EPA 8260D	Isopropyl ether
GC/MS	EPA 8260D	Isopropylbenzene
GC/MS	EPA 8260D	m & p-Xylene
GC/MS	EPA 8260D	Methacrylonitrile
GC/MS	EPA 8260D	Methyl acetate
GC/MS	EPA 8260D	Methyl cyclohexane
GC/MS	EPA 8260D	Methyl isobutyl ketone
GC/MS	EPA 8260D	Methyl methacrylate
GC/MS	EPA 8260D	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260D	Methylene chloride
GC/MS	EPA 8260D	Naphthalene
GC/MS	EPA 8260D	n-Butylbenzene
GC/MS	EPA 8260D	n-Heptane
GC/MS	EPA 8260D	n-Propylbenzene
GC/MS	EPA 8260D	o-Xylene
GC/MS	EPA 8260D	Pentachloroethane
GC/MS	EPA 8260D	Propionitrile
GC/MS	EPA 8260D	sec-Butylbenzene
GC/MS	EPA 8260D	Styrene
GC/MS	EPA 8260D	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260D	tert-Butylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Tetrachloroethene
GC/MS	EPA 8260D	Tetrahydrofuran
GC/MS	EPA 8260D	Toluene
GC/MS	EPA 8260D	trans-1,2-Dichloroethene
GC/MS	EPA 8260D	trans-1,3-Dichloropropene
GC/MS	EPA 8260D	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260D	Trichloroethene
GC/MS	EPA 8260D	Trichlorofluoromethane
GC/MS	EPA 8260D	Vinyl acetate
GC/MS	EPA 8260D	Vinyl chloride
GC/MS	EPA 8260D	Xylenes, total
GC/MS	EPA 8270D	1,1-Biphenyl
GC/MS	EPA 8270D	1,2,3-Trichlorobenzene
GC/MS	EPA 8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270D	1,2-Diphenylhydrazine
GC/MS	EPA 8270D	1,3,5-Trichlorobenzene
GC/MS	EPA 8270D	1,3,5-Trinitrobenzene
GC/MS	EPA 8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dioxane
GC/MS	EPA 8270D	1,4-Naphthoquinone
GC/MS	EPA 8270D	1-Methylnaphthalene
GC/MS	EPA 8270D	1-Naphthylamine
GC/MS	EPA 8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270D	2,3,6-Trichlorophenol
GC/MS	EPA 8270D	2,3-Dimethylphenol
GC/MS	EPA 8270D	2,3-Xylenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	2,4 & 2,5-Dimethylphenol
GC/MS	EPA 8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270D	2,4-Dichlorophenol
GC/MS	EPA 8270D	2,4-Dimethylphenol
GC/MS	EPA 8270D	2,4-Dinitrophenol
GC/MS	EPA 8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270D	2,5-Dimethylphenol
GC/MS	EPA 8270D	2,6-Dichlorophenol
GC/MS	EPA 8270D	2,6-Dimethylphenol
GC/MS	EPA 8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270D	2-Acetylaminofluorene
GC/MS	EPA 8270D	2-Chloronaphthalene
GC/MS	EPA 8270D	2-Chlorophenol
GC/MS	EPA 8270D	2-Methyl-4,6-Dinitrophenol
GC/MS	EPA 8270D	2-Methylnaphthalene
GC/MS	EPA 8270D	2-Methylphenol
GC/MS	EPA 8270D	2-Naphthylamine
GC/MS	EPA 8270D	2-Nitroaniline
GC/MS	EPA 8270D	2-Nitrophenol
GC/MS	EPA 8270D	2-Picoline
GC/MS	EPA 8270D	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270D	2-Toluidine (o-Toluidine)
GC/MS	EPA 8270D	3 & 4-Methylphenol
GC/MS	EPA 8270D	3,3-Dichlorobenzidine
GC/MS	EPA 8270D	3,3'-Dimethylbenzidine
GC/MS	EPA 8270D	3,4-Dimethylphenol
GC/MS	EPA 8270D	3,4-Xylenol
GC/MS	EPA 8270D	3-Methylcholanthrene
GC/MS	EPA 8270D	3-Nitroaniline

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	4-Aminobiphenyl
GC/MS	EPA 8270D	4-Bromophenylphenyl ether
GC/MS	EPA 8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270D	4-Chloroaniline
GC/MS	EPA 8270D	4-Chlorophenol
GC/MS	EPA 8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270D	4-Nitroaniline
GC/MS	EPA 8270D	4-Nitrophenol
GC/MS	EPA 8270D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270D	7,12-Dimethylbenz (a) anthracene
GC/MS	EPA 8270D	Acenaphthene
GC/MS	EPA 8270D	Acenaphthylene
GC/MS	EPA 8270D	Acetophenone
GC/MS	EPA 8270D	alpha-, alpha-Dimethylphenethlylamine
GC/MS	EPA 8270D	alpha-Pinene
GC/MS	EPA 8270D	Aniline
GC/MS	EPA 8270D	Anthracene
GC/MS	EPA 8270D	Aramite, Total
GC/MS	EPA 8270D	Atrazine
GC/MS	EPA 8270D	Benzaldehyde
GC/MS	EPA 8270D	Benzidine
GC/MS	EPA 8270D	Benzo (a) anthracene
GC/MS	EPA 8270D	Benzo (a) pyrene
GC/MS	EPA 8270D	Benzo (b) fluoranthene
GC/MS	EPA 8270D	Benzo (ghi) perylene
GC/MS	EPA 8270D	Benzo (k) fluoranthene
GC/MS	EPA 8270D	Benzoic acid
GC/MS	EPA 8270D	Benzyl alcohol
GC/MS	EPA 8270D	Bis (2-chloroethoxy) methane
GC/MS	EPA 8270D	Bis (2-chloroethyl) ether

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Bis (2-chloroisopropyl) ether
GC/MS	EPA 8270D	Bis (2-ethylhexyl) phthalate
GC/MS	EPA 8270D	Butyl benzyl phthalate
GC/MS	EPA 8270D	Caprolactam
GC/MS	EPA 8270D	Carbazole
GC/MS	EPA 8270D	Chrysene
GC/MS	EPA 8270D	Cresols
GC/MS	EPA 8270D	Di(2-ethylhexyl)adipate
GC/MS	EPA 8270D	Diallate
GC/MS	EPA 8270D	Dibenz(a,h) anthracene
GC/MS	EPA 8270D	Dibenzofuran
GC/MS	EPA 8270D	Diethyl phthalate
GC/MS	EPA 8270D	Dimethoate
GC/MS	EPA 8270D	Dimethyl phthalate
GC/MS	EPA 8270D	Di-n-butyl phthalate
GC/MS	EPA 8270D	Di-n-octyl phthalate
GC/MS	EPA 8270D	Diphenyl ether
GC/MS	EPA 8270D	Disulfoton
GC/MS	EPA 8270D	Ethyl methane sulfonate
GC/MS	EPA 8270D	Famphur
GC/MS	EPA 8270D	Fluoranthene
GC/MS	EPA 8270D	Fluorene
GC/MS	EPA 8270D	Hexachlorobenzene
GC/MS	EPA 8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270D	Hexachloroethane
GC/MS	EPA 8270D	Hexachlorophene
GC/MS	EPA 8270D	Hexachloropropene
GC/MS	EPA 8270D	Hexachlrobutadiene
GC/MS	EPA 8270D	Indeno (1,2,3-cd) pyrene
GC/MS	EPA 8270D	Isophorone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Isosafrole
GC/MS	EPA 8270D	Methapyrilene
GC/MS	EPA 8270D	Methyl methane sulfonate
GC/MS	EPA 8270D	Methylbenzoate
GC/MS	EPA 8270D	Naphthalene
GC/MS	EPA 8270D	Nitrobenzene
GC/MS	EPA 8270D	N-Nitrosodiethylamine
GC/MS	EPA 8270D	N-Nitrosodimethylamine
GC/MS	EPA 8270D	N-Nitroso-di-n-butylamine
GC/MS	EPA 8270D	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270D	N-Nitrosomethylethylamine
GC/MS	EPA 8270D	N-Nitrosomorpholine
GC/MS	EPA 8270D	N-Nitrosopiperidine
GC/MS	EPA 8270D	N-Nitrosopyrrolidine
GC/MS	EPA 8270D	o,o',o"-Triethylphosphorothioate
GC/MS	EPA 8270D	Parathion ethyl
GC/MS	EPA 8270D	Parathion methyl
GC/MS	EPA 8270D	p-Dimethylaminoazobenzene
GC/MS	EPA 8270D	Pentachlorobenzene
GC/MS	EPA 8270D	Pentachlorophenol
GC/MS	EPA 8270D	Pentachlronitrobenzene
GC/MS	EPA 8270D	Phenacetin
GC/MS	EPA 8270D	Phenanthrene
GC/MS	EPA 8270D	Phenol
GC/MS	EPA 8270D	Phenyl ether
GC/MS	EPA 8270D	Phorate
GC/MS	EPA 8270D	p-Phenylene diamine
GC/MS	EPA 8270D	Pronamide
GC/MS	EPA 8270D	Pyrene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Pyridine
GC/MS	EPA 8270D	Safrole, Total
GC/MS	EPA 8270D	Sulfotepp
GC/MS	EPA 8270D	Thionazin
GC/MS	EPA 8270E	1,1-Biphenyl
GC/MS	EPA 8270E	1,2,3-Trichlorobenzene
GC/MS	EPA 8270E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270E	1,2,4-Trichlorobenzene
GC/MS	EPA 8270E	1,2-Dichlorobenzene
GC/MS	EPA 8270E	1,2-Diphenylhydrazine
GC/MS	EPA 8270E	1,3,5-Trichlorobenzene
GC/MS	EPA 8270E	1,3,5-Trinitrobenzene
GC/MS	EPA 8270E	1,3-Dichlorobenzene
GC/MS	EPA 8270E	1,3-Dinitrobenzene
GC/MS	EPA 8270E	1,4-Dichlorobenzene
GC/MS	EPA 8270E	1,4-Dioxane
GC/MS	EPA 8270E	1,4-Naphthoquinone
GC/MS	EPA 8270E	1-Methylnaphthalene
GC/MS	EPA 8270E	1-Naphthylamine
GC/MS	EPA 8270E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270E	2,3,6-Trichlorophenol
GC/MS	EPA 8270E	2,3-Dimethylphenol
GC/MS	EPA 8270E	2,3-Xylenol
GC/MS	EPA 8270E	2,4 & 2,5-Dimethylphenol
GC/MS	EPA 8270E	2,4,5-Trichlorophenol
GC/MS	EPA 8270E	2,4,6-Trichlorophenol
GC/MS	EPA 8270E	2,4-Dichlorophenol
GC/MS	EPA 8270E	2,4-Dimethylphenol
GC/MS	EPA 8270E	2,4-Dinitrophenol
GC/MS	EPA 8270E	2,4-Dinitrotoluene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	2,5-Dimethylphenol
GC/MS	EPA 8270E	2,6-Dichlorophenol
GC/MS	EPA 8270E	2,6-Dimethylphenol
GC/MS	EPA 8270E	2,6-Dinitrotoluene
GC/MS	EPA 8270E	2-Acetylaminofluorene
GC/MS	EPA 8270E	2-Chloronaphthalene
GC/MS	EPA 8270E	2-Chlorophenol
GC/MS	EPA 8270E	2-Methyl-4,6-Dinitrophenol
GC/MS	EPA 8270E	2-Methylnaphthalene
GC/MS	EPA 8270E	2-Methylphenol
GC/MS	EPA 8270E	2-Naphthylamine
GC/MS	EPA 8270E	2-Nitroaniline
GC/MS	EPA 8270E	2-Nitrophenol
GC/MS	EPA 8270E	2-Picoline
GC/MS	EPA 8270E	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270E	2-Toluidine (o-Toluidine)
GC/MS	EPA 8270E	3 & 4-Methylphenol
GC/MS	EPA 8270E	3,3-Dichlorobenzidine
GC/MS	EPA 8270E	3,3'-Dimethylbenzidine
GC/MS	EPA 8270E	3,4-Dimethylphenol
GC/MS	EPA 8270E	3,4-Xylenol
GC/MS	EPA 8270E	3-Methylcholanthrene
GC/MS	EPA 8270E	3-Nitroaniline
GC/MS	EPA 8270E	4-Aminobiphenyl
GC/MS	EPA 8270E	4-Bromophenylphenyl ether
GC/MS	EPA 8270E	4-Chloro-3-methylphenol
GC/MS	EPA 8270E	4-Chloroaniline
GC/MS	EPA 8270E	4-Chlorophenol
GC/MS	EPA 8270E	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270E	4-Nitroaniline

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	4-Nitrophenol
GC/MS	EPA 8270E	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270E	7,12-Dimethylbenz (a) anthracene
GC/MS	EPA 8270E	Acenaphthene
GC/MS	EPA 8270E	Acenaphthylene
GC/MS	EPA 8270E	Acetophenone
GC/MS	EPA 8270E	alpha-, alpha-Dimethylphenethlylamine
GC/MS	EPA 8270E	alpha-Pinene
GC/MS	EPA 8270E	Aniline
GC/MS	EPA 8270E	Anthracene
GC/MS	EPA 8270E	Aramite, Total
GC/MS	EPA 8270E	Atrazine
GC/MS	EPA 8270E	Benzaldehyde
GC/MS	EPA 8270E	Benzidine
GC/MS	EPA 8270E	Benzo (a) anthracene
GC/MS	EPA 8270E	Benzo (a) pyrene
GC/MS	EPA 8270E	Benzo (b) fluoranthene
GC/MS	EPA 8270E	Benzo (ghi) perylene
GC/MS	EPA 8270E	Benzo (k) fluoranthene
GC/MS	EPA 8270E	Benzoic acid
GC/MS	EPA 8270E	Benzyl alcohol
GC/MS	EPA 8270E	Bis (2-chloroethoxy) methane
GC/MS	EPA 8270E	Bis (2-chloroethyl) ether
GC/MS	EPA 8270E	Bis (2-chloroisopropyl) ether
GC/MS	EPA 8270E	Bis (2-ethylhexyl) phthalate
GC/MS	EPA 8270E	Butyl benzyl phthalate
GC/MS	EPA 8270E	Caprolactam
GC/MS	EPA 8270E	Carbazole
GC/MS	EPA 8270E	Chrysene
GC/MS	EPA 8270E	Cresols

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Di(2-ethylhexyl)adipate
GC/MS	EPA 8270E	Diallate
GC/MS	EPA 8270E	Dibenz(a,h) anthracene
GC/MS	EPA 8270E	Dibenzofuran
GC/MS	EPA 8270E	Diethyl phthalate
GC/MS	EPA 8270E	Dimethoate
GC/MS	EPA 8270E	Dimethyl phthalate
GC/MS	EPA 8270E	Di-n-butyl phthalate
GC/MS	EPA 8270E	Di-n-octyl phthalate
GC/MS	EPA 8270E	Diphenyl ether
GC/MS	EPA 8270E	Disulfoton
GC/MS	EPA 8270E	Ethyl methane sulfonate
GC/MS	EPA 8270E	Famphur
GC/MS	EPA 8270E	Fluoranthene
GC/MS	EPA 8270E	Fluorene
GC/MS	EPA 8270E	Hexachlorobenzene
GC/MS	EPA 8270E	Hexachlorocyclopentadiene
GC/MS	EPA 8270E	Hexachloroethane
GC/MS	EPA 8270E	Hexachlorophene
GC/MS	EPA 8270E	Hexachloropropene
GC/MS	EPA 8270E	Hexachlrobutadiene
GC/MS	EPA 8270E	Indeno (1,2,3-cd) pyrene
GC/MS	EPA 8270E	Isophorone
GC/MS	EPA 8270E	Isosafrole
GC/MS	EPA 8270E	Methapyrilene
GC/MS	EPA 8270E	Methyl methane sulfonate
GC/MS	EPA 8270E	Methylbenzoate
GC/MS	EPA 8270E	Naphthalene
GC/MS	EPA 8270E	Nitrobenzene
GC/MS	EPA 8270E	N-Nitrosodiethylamine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	N-Nitrosodimethylamine
GC/MS	EPA 8270E	N-Nitroso-di-n-butylamine
GC/MS	EPA 8270E	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270E	N-Nitrosodiphenylamine
GC/MS	EPA 8270E	N-Nitrosomethylethylamine
GC/MS	EPA 8270E	N-Nitrosomorpholine
GC/MS	EPA 8270E	N-Nitrosopiperidine
GC/MS	EPA 8270E	N-Nitrosopyrrolidine
GC/MS	EPA 8270E	o,o',o"-Triethylphosphorothioate
GC/MS	EPA 8270E	Parathion ethyl
GC/MS	EPA 8270E	Parathion methyl
GC/MS	EPA 8270E	p-Dimethylaminoazobenzene
GC/MS	EPA 8270E	Pentachlorobenzene
GC/MS	EPA 8270E	Pentachlorophenol
GC/MS	EPA 8270E	Pentachlronitrobenzene
GC/MS	EPA 8270E	Phenacetin
GC/MS	EPA 8270E	Phenanthrene
GC/MS	EPA 8270E	Phenol
GC/MS	EPA 8270E	Phenyl ether
GC/MS	EPA 8270E	Phorate
GC/MS	EPA 8270E	p-Phenylene diamine
GC/MS	EPA 8270E	Pronamide
GC/MS	EPA 8270E	Pyrene
GC/MS	EPA 8270E	Pyridine
GC/MS	EPA 8270E	Safrole, Total
GC/MS	EPA 8270E	Sulfotepp
GC/MS	EPA 8270E	Thionazin
General Chemistry	EPA 9012B	Cyanide
General Chemistry	EPA 9013 EPA 9012B	Cyanide amenable to chlorination

Non-Potable Water		
Technology	**Method**	**Analyte**
General Chemistry	EPA 9020B	Total organic halides
General Chemistry	EPA 9030B EPA 9034	Sulfide
General Chemistry	EPA 9038	Sulfate
General Chemistry	EPA 9040C	pH
General Chemistry	EPA 9050A	Specific conductance
IC	EPA 9056A	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate (as N)
IC	EPA 9056A	Nitrate (as NO3)
IC	EPA 9056A	Nitrate-nitrite (as N)
IC	EPA 9056A	Nitrate-nitrite (as NO3-NO2)
IC	EPA 9056A	Nitrite (as N)
IC	EPA 9056A	Nitrite (as NO2)
IC	EPA 9056A	Sulfate
General Chemistry	EPA 9060A	Dissolved carbon
General Chemistry	EPA 9060A	Dissolved inorganic carbon
General Chemistry	EPA 9060A	Dissolved organic carbon
General Chemistry	EPA 9060A	Total carbon
General Chemistry	EPA 9060A	Total inorganic carbon
General Chemistry	EPA 9060A	Total organic carbon
General Chemistry	EPA 9065A	Phenols
General Chemistry	EPA 9251	Chloride
GC/FID/TCD	RSK-175	Ethane (FID)
GC/FID/TCD	RSK-175	Ethene (FID)
GC/FID/TCD	RSK-175	Methane (FID)
GC/FID/TCD	RSK-175	Methane (TCD)
Preparation	**Method**	**Type**
Organic Extraction	EPA 3520C	Continuous Liquid-Liquid Extraction

Non-Potable Water		
Technology	**Method**	**Analyte**
TLCP Preparation	EPA 1311	Toxicity Characteristics Leaching Procedure
SPLP Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Purge & Trap	EPA 5030B	Purge & Trap for Aqueous volatile
Acid Digestion	EPA 3005A	Metals Prep
Acid Digestion (Aqueous samples)	EPA 3010A	Acid Digestion for Metals (Aqueous samples)
Distillation	EPA 9030B	Sulfide

Drinking Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 504.1	1,2,3-Trichloropropane
GC/ECD	EPA 504.1	1,2-Dibromo-3-chloropropane (DBCP)
GC/ECD	EPA 504.1	1,2-Dibromoethane (EDB)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
General Chemistry	EPA 1030	Ignitability
ICP	EPA 6010C	Aluminum
ICP	EPA 6010C	Antimony
ICP	EPA 6010C	Arsenic
ICP	EPA 6010C	Barium
ICP	EPA 6010C	Beryllium
ICP	EPA 6010C	Boron
ICP	EPA 6010C	Cadmium
ICP	EPA 6010C	Calcium
ICP	EPA 6010C	Chromium
ICP	EPA 6010C	Cobalt

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	EPA 6010C	Copper
ICP	EPA 6010C	Iron
ICP	EPA 6010C	Lead
ICP	EPA 6010C	Magnesium
ICP	EPA 6010C	Manganese
ICP	EPA 6010C	Molybdenum
ICP	EPA 6010C	Nickel
ICP	EPA 6010C	Potassium
ICP	EPA 6010C	Selenium
ICP	EPA 6010C	Silver
ICP	EPA 6010C	Sodium
ICP	EPA 6010C	Strontium
ICP	EPA 6010C	Thallium
ICP	EPA 6010C	Tin
ICP	EPA 6010C	Titanium
ICP	EPA 6010C	Vanadium
ICP	EPA 6010C	Zinc
ICP	EPA 6010D	Aluminum
ICP	EPA 6010D	Antimony
ICP	EPA 6010D	Arsenic
ICP	EPA 6010D	Barium
ICP	EPA 6010D	Beryllium
ICP	EPA 6010D	Boron
ICP	EPA 6010D	Cadmium
ICP	EPA 6010D	Calcium
ICP	EPA 6010D	Chromium
ICP	EPA 6010D	Cobalt
ICP	EPA 6010D	Copper
ICP	EPA 6010D	Iron
ICP	EPA 6010D	Lead

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	EPA 6010D	Magnesium
ICP	EPA 6010D	Manganese
ICP	EPA 6010D	Molybdenum
ICP	EPA 6010D	Nickel
ICP	EPA 6010D	Potassium
ICP	EPA 6010D	Selenium
ICP	EPA 6010D	Silver
ICP	EPA 6010D	Sodium
ICP	EPA 6010D	Strontium
ICP	EPA 6010D	Thallium
ICP	EPA 6010D	Tin
ICP	EPA 6010D	Titanium
ICP	EPA 6010D	Vanadium
ICP	EPA 6010D	Zinc
ICP/MS	EPA 6020A	Aluminum
ICP/MS	EPA 6020A	Antimony
ICP/MS	EPA 6020A	Arsenic
ICP/MS	EPA 6020A	Barium
ICP/MS	EPA 6020A	Beryllium
ICP/MS	EPA 6020A	Boron
ICP/MS	EPA 6020A	Cadmium
ICP/MS	EPA 6020A	Calcium
ICP/MS	EPA 6020A	Chromium
ICP/MS	EPA 6020A	Cobalt
ICP/MS	EPA 6020A	Copper
ICP/MS	EPA 6020A	Iron
ICP/MS	EPA 6020A	Lead
ICP/MS	EPA 6020A	Magnesium
ICP/MS	EPA 6020A	Manganese
ICP/MS	EPA 6020A	Mercury

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020A	Molybdenum
ICP/MS	EPA 6020A	Nickel
ICP/MS	EPA 6020A	Potassium
ICP/MS	EPA 6020A	Selenium
ICP/MS	EPA 6020A	Silver
ICP/MS	EPA 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 6020A	Thallium
ICP/MS	EPA 6020A	Tin
ICP/MS	EPA 6020A	Titanium
ICP/MS	EPA 6020A	Vanadium
ICP/MS	EPA 6020A	Zinc
ICP/MS	EPA 6020B	Aluminum
ICP/MS	EPA 6020B	Antimony
ICP/MS	EPA 6020B	Arsenic
ICP/MS	EPA 6020B	Barium
ICP/MS	EPA 6020B	Beryllium
ICP/MS	EPA 6020B	Boron
ICP/MS	EPA 6020B	Cadmium
ICP/MS	EPA 6020B	Calcium
ICP/MS	EPA 6020B	Chromium
ICP/MS	EPA 6020B	Cobalt
ICP/MS	EPA 6020B	Copper
ICP/MS	EPA 6020B	Iron
ICP/MS	EPA 6020B	Lead
ICP/MS	EPA 6020B	Magnesium
ICP/MS	EPA 6020B	Manganese
ICP/MS	EPA 6020B	Mercury
ICP/MS	EPA 6020B	Molybdenum
ICP/MS	EPA 6020B	Nickel

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP/MS	EPA 6020B	Potassium
ICP/MS	EPA 6020B	Selenium
ICP/MS	EPA 6020B	Silver
ICP/MS	EPA 6020B	Sodium
ICP/MS	EPA 6020B	Strontium
ICP/MS	EPA 6020B	Thallium
ICP/MS	EPA 6020B	Tin
ICP/MS	EPA 6020B	Titanium
ICP/MS	EPA 6020B	Vanadium
ICP/MS	EPA 6020B	Zinc
CVAA	EPA 7471B	Mercury
GC/FID	EPA 8015C	#2 Diesel Fuel
GC/FID	EPA 8015C	Diesel Range Organics
GC/FID	EPA 8015C	Gasoline Range Organics
GC/FID	EPA 8015C	Kerosene
GC/FID	EPA 8015C	Mineral Spirits
GC/FID	EPA 8015C	Motor Oil
GC/FID	EPA 8015C	Oil Range Organics
GC/FID	EPA 8015C	2,2'-Oxybisethanol
GC/FID	EPA 8015C	2-Butoxyethanol
GC/FID	EPA 8015C	2-Propoxy ethanol
GC/FID	EPA 8015C	Cellosolve acetate
GC/FID	EPA 8015C	Di-propylene glycol
GC/FID	EPA 8015C	Di-propylene glycol methyl ether
GC/FID	EPA 8015C	Ethanol
GC/FID	EPA 8015C	Ethanol
GC/FID	EPA 8015C	Ethyl acetate
GC/FID	EPA 8015C	Ethylene glycol
GC/FID	EPA 8015C	Isoamyl acetate
GC/FID	EPA 8015C	Isobutanol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/FID	EPA 8015C	Isobutyl acetate
GC/FID	EPA 8015C	Isopropanol
GC/FID	EPA 8015C	Isopropyl acetate
GC/FID	EPA 8015C	Methanol
GC/FID	EPA 8015C	Methyl acetate
GC/FID	EPA 8015C	n-Butanol
GC/FID	EPA 8015C	n-Butyl acetate
GC/FID	EPA 8015C	n-Heptanol
GC/FID	EPA 8015C	n-Propanol
GC/FID	EPA 8015C	n-Propyl acetate
GC/FID	EPA 8015C	Phenol
GC/FID	EPA 8015C	Propylene glycol
GC/FID	EPA 8015C	sec-Butanol
GC/FID	EPA 8015C	sec-Butyl acetate
GC/FID	EPA 8015C	Tert-amyl alcohol
GC/FID	EPA 8015C	tert-Butyl alcohol
GC/FID	EPA 8015C	Tetraethylene glycol
GC/FID	EPA 8015C	Triethylene glycol
GC/ECD	EPA 8081B	2,4' DDE
GC/ECD	EPA 8081B	2,4'-DDD
GC/ECD	EPA 8081B	2,4'-DDT
GC/ECD	EPA 8081B	4,4' DDE
GC/ECD	EPA 8081B	4,4'-DDD
GC/ECD	EPA 8081B	4,4'-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC
GC/ECD	EPA 8081B	beta-BHC
GC/ECD	EPA 8081B	Chlordane (alpha)
GC/ECD	EPA 8081B	Chlordane (gamma)
GC/ECD	EPA 8081B	Chlordane (technical)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	Chlorobenzilate
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I (alpha)
GC/ECD	EPA 8081B	Endosulfan II (beta)
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin ketone
GC/ECD	EPA 8081B	gamma-BHC
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Isodrin
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Toxaphene
GC/ECD	EPA 8082A	PCB-1016
GC/ECD	EPA 8082A	PCB-1221
GC/ECD	EPA 8082A	PCB-1232
GC/ECD	EPA 8082A	PCB-1242
GC/ECD	EPA 8082A	PCB-1248
GC/ECD	EPA 8082A	PCB-1254
GC/ECD	EPA 8082A	PCB-1260
GC/ECD	EPA 8082A	PCB-1262
GC/ECD	EPA 8082A	PCB-1268
GC/ECD	EPA 8082A	PCBs, Total
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4,6-Trichlorophenol
GC/ECD	EPA 8151A	2,4-D

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	2,6-Dichlorophenol
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	DCPA (Dacthal)
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Picloram
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,1-Trichloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethene
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane
GC/MS	EPA 8260B	1,2-Dichloroethene, Total
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,2-Xylene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	1,3 & 1,4-Xylene
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,3-Dichloropropene, Total
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1,4-Dioxane
GC/MS	EPA 8260B	1-Chlorohexane
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone
GC/MS	EPA 8260B	3-Chloro-1-propene
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Isopropyltoluene
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acetonitrile
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	BTEX, Total
GC/MS	EPA 8260B	Carbon disulfide
GC/MS	EPA 8260B	Carbon tetrachloride
GC/MS	EPA 8260B	Chlorobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	Chloroprene
GC/MS	EPA 8260B	cis-1,2-Dichloroethene
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane
GC/MS	EPA 8260B	Diethyl ether
GC/MS	EPA 8260B	Ethanol
GC/MS	EPA 8260B	Ethyl benzene
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Furan
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Hexane
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isobutanol
GC/MS	EPA 8260B	Isopropyl ether
GC/MS	EPA 8260B	Isopropylbenzene
GC/MS	EPA 8260B	Methacrylonitrile
GC/MS	EPA 8260B	Methyl acetate
GC/MS	EPA 8260B	Methyl cyclohexane
GC/MS	EPA 8260B	Methyl isobutyl ketone
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260B	Methylene chloride
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	n-Heptane
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	Pentachloroethane
GC/MS	EPA 8260B	Propionitrile
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene
GC/MS	EPA 8260B	Tetrahydrofuran
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260B	Trichloroethene
GC/MS	EPA 8260B	Trichlorofluoromethane
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl chloride
GC/MS	EPA 8260B	Xylenes, total
GC/MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethane
GC/MS	EPA 8260C	1,1-Dichloroethene
GC/MS	EPA 8260C	1,1-Dichloropropene
GC/MS	EPA 8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260C	1,2,4-Trichlorobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260C	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260C	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260C	1,2-Dichloroethane
GC/MS	EPA 8260C	1,2-Dichloroethene, Total
GC/MS	EPA 8260C	1,2-Dichloropropane
GC/MS	EPA 8260C	1,2-Xylene
GC/MS	EPA 8260C	1,3 & 1,4-Xylene
GC/MS	EPA 8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260C	1,3-Dichloropropane
GC/MS	EPA 8260C	1,3-Dichloropropene, Total
GC/MS	EPA 8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260C	1,4-Dioxane
GC/MS	EPA 8260C	1-Chlorohexane
GC/MS	EPA 8260C	2,2-Dichloropropane
GC/MS	EPA 8260C	2-Butanone
GC/MS	EPA 8260C	2-Chlorotoluene
GC/MS	EPA 8260C	2-Hexanone
GC/MS	EPA 8260C	3-Chloro-1-propene
GC/MS	EPA 8260C	4-Chlorotoluene
GC/MS	EPA 8260C	4-Isopropyltoluene
GC/MS	EPA 8260C	Acetone
GC/MS	EPA 8260C	Acetonitrile
GC/MS	EPA 8260C	Acrolein
GC/MS	EPA 8260C	Acrylonitrile
GC/MS	EPA 8260C	Benzene
GC/MS	EPA 8260C	Bromobenzene
GC/MS	EPA 8260C	Bromochloromethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Bromodichloromethane
GC/MS	EPA 8260C	Bromoform
GC/MS	EPA 8260C	Bromomethane
GC/MS	EPA 8260C	BTEX, Total
GC/MS	EPA 8260C	Carbon disulfide
GC/MS	EPA 8260C	Carbon tetrachloride
GC/MS	EPA 8260C	Chlorobenzene
GC/MS	EPA 8260C	Chloroethane
GC/MS	EPA 8260C	Chloroform
GC/MS	EPA 8260C	Chloromethane
GC/MS	EPA 8260C	Chloroprene
GC/MS	EPA 8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260C	Cyclohexane
GC/MS	EPA 8260C	Dibromochloromethane
GC/MS	EPA 8260C	Dibromomethane
GC/MS	EPA 8260C	Dichlorodifluoromethane
GC/MS	EPA 8260C	Diethyl ether
GC/MS	EPA 8260C	Ethanol
GC/MS	EPA 8260C	Ethyl benzene
GC/MS	EPA 8260C	Ethyl methacrylate
GC/MS	EPA 8260C	Furan
GC/MS	EPA 8260C	Hexachlorobutadiene
GC/MS	EPA 8260C	Hexane
GC/MS	EPA 8260C	Iodomethane
GC/MS	EPA 8260C	Isobutanol
GC/MS	EPA 8260C	Isopropyl ether
GC/MS	EPA 8260C	Isopropylbenzene
GC/MS	EPA 8260C	Methacrylonitrile
GC/MS	EPA 8260C	Methyl acetate

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260C	Methyl cyclohexane
GC/MS	EPA 8260C	Methyl isobutyl ketone
GC/MS	EPA 8260C	Methyl methacrylate
GC/MS	EPA 8260C	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260C	Methylene chloride
GC/MS	EPA 8260C	Naphthalene
GC/MS	EPA 8260C	n-Butylbenzene
GC/MS	EPA 8260C	n-Heptane
GC/MS	EPA 8260C	n-Propylbenzene
GC/MS	EPA 8260C	Pentachloroethane
GC/MS	EPA 8260C	Propionitrile
GC/MS	EPA 8260C	sec-Butylbenzene
GC/MS	EPA 8260C	Styrene
GC/MS	EPA 8260C	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260C	tert-Butylbenzene
GC/MS	EPA 8260C	Tetrachloroethene
GC/MS	EPA 8260C	Tetrahydrofuran
GC/MS	EPA 8260C	Toluene
GC/MS	EPA 8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260C	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260C	Trichloroethene
GC/MS	EPA 8260C	Trichlorofluoromethane
GC/MS	EPA 8260C	Vinyl acetate
GC/MS	EPA 8260C	Vinyl chloride
GC/MS	EPA 8260C	Xylenes, total
GC/MS	EPA 8260D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,1-Trichloroethane
GC/MS	EPA 8260D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	1,1,2-Trichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethene
GC/MS	EPA 8260D	1,1-Dichloropropene
GC/MS	EPA 8260D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260D	1,2,3-Trichloropropane
GC/MS	EPA 8260D	1,2,4-Trichlorobenzene
GC/MS	EPA 8260D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260D	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260D	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260D	1,2-Dichlorobenzene
GC/MS	EPA 8260D	1,2-Dichloroethane
GC/MS	EPA 8260D	1,2-Dichloroethene, Total
GC/MS	EPA 8260D	1,2-Dichloropropane
GC/MS	EPA 8260D	1,2-Xylene
GC/MS	EPA 8260D	1,3 & 1,4-Xylene
GC/MS	EPA 8260D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260D	1,3-Dichlorobenzene
GC/MS	EPA 8260D	1,3-Dichloropropane
GC/MS	EPA 8260D	1,3-Dichloropropene, Total
GC/MS	EPA 8260D	1,4-Dichlorobenzene
GC/MS	EPA 8260D	1,4-Dioxane
GC/MS	EPA 8260D	1-Chlorohexane
GC/MS	EPA 8260D	2,2-Dichloropropane
GC/MS	EPA 8260D	2-Butanone
GC/MS	EPA 8260D	2-Chlorotoluene
GC/MS	EPA 8260D	2-Hexanone
GC/MS	EPA 8260D	3-Chloro-1-propene
GC/MS	EPA 8260D	4-Chlorotoluene
GC/MS	EPA 8260D	4-Isopropyltoluene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Acetone
GC/MS	EPA 8260D	Acetonitrile
GC/MS	EPA 8260D	Acrolein
GC/MS	EPA 8260D	Acrylonitrile
GC/MS	EPA 8260D	Benzene
GC/MS	EPA 8260D	Bromobenzene
GC/MS	EPA 8260D	Bromochloromethane
GC/MS	EPA 8260D	Bromodichloromethane
GC/MS	EPA 8260D	Bromoform
GC/MS	EPA 8260D	Bromomethane
GC/MS	EPA 8260D	BTEX, Total
GC/MS	EPA 8260D	Carbon disulfide
GC/MS	EPA 8260D	Carbon tetrachloride
GC/MS	EPA 8260D	Chlorobenzene
GC/MS	EPA 8260D	Chloroethane
GC/MS	EPA 8260D	Chloroform
GC/MS	EPA 8260D	Chloromethane
GC/MS	EPA 8260D	Chloroprene
GC/MS	EPA 8260D	cis-1,2-Dichloroethene
GC/MS	EPA 8260D	cis-1,3-Dichloropropene
GC/MS	EPA 8260D	Cyclohexane
GC/MS	EPA 8260D	Dibromochloromethane
GC/MS	EPA 8260D	Dibromomethane
GC/MS	EPA 8260D	Dichlorodifluoromethane
GC/MS	EPA 8260D	Diethyl ether
GC/MS	EPA 8260D	Ethanol
GC/MS	EPA 8260D	Ethyl benzene
GC/MS	EPA 8260D	Ethyl methacrylate
GC/MS	EPA 8260D	Furan
GC/MS	EPA 8260D	Hexachlorobutadiene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Hexane
GC/MS	EPA 8260D	Iodomethane
GC/MS	EPA 8260D	Isobutanol
GC/MS	EPA 8260D	Isopropyl ether
GC/MS	EPA 8260D	Isopropylbenzene
GC/MS	EPA 8260D	Methacrylonitrile
GC/MS	EPA 8260D	Methyl acetate
GC/MS	EPA 8260D	Methyl cyclohexane
GC/MS	EPA 8260D	Methyl isobutyl ketone
GC/MS	EPA 8260D	Methyl methacrylate
GC/MS	EPA 8260D	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260D	Methylene chloride
GC/MS	EPA 8260D	Naphthalene
GC/MS	EPA 8260D	n-Butylbenzene
GC/MS	EPA 8260D	n-Heptane
GC/MS	EPA 8260D	n-Propylbenzene
GC/MS	EPA 8260D	Pentachloroethane
GC/MS	EPA 8260D	Propionitrile
GC/MS	EPA 8260D	sec-Butylbenzene
GC/MS	EPA 8260D	Styrene
GC/MS	EPA 8260D	Tert-butyl alcohol (TBA)
GC/MS	EPA 8260D	tert-Butylbenzene
GC/MS	EPA 8260D	Tetrachloroethene
GC/MS	EPA 8260D	Tetrahydrofuran
GC/MS	EPA 8260D	Toluene
GC/MS	EPA 8260D	trans-1,2-Dichloroethene
GC/MS	EPA 8260D	trans-1,3-Dichloropropene
GC/MS	EPA 8260D	trans-1,4-dichloro-2-butene
GC/MS	EPA 8260D	Trichloroethene
GC/MS	EPA 8260D	Trichlorofluoromethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Vinyl acetate
GC/MS	EPA 8260D	Vinyl chloride
GC/MS	EPA 8260D	Xylenes, total
GC/MS	EPA 8270D	1,1-Biphenyl
GC/MS	EPA 8270D	1,2,3-Trichlorobenzene
GC/MS	EPA 8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270D	1,2-Diphenylhydrazine
GC/MS	EPA 8270D	1,3,5-Trichlorobenzene
GC/MS	EPA 8270D	1,3,5-Trinitrobenzene
GC/MS	EPA 8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dioxane
GC/MS	EPA 8270D	1,4-Naphthoquinone
GC/MS	EPA 8270D	1-Methylnaphthalene
GC/MS	EPA 8270D	1-Naphthylamine
GC/MS	EPA 8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270D	2,3,6-Trichlorophenol
GC/MS	EPA 8270D	2,3-Dimethylphenol
GC/MS	EPA 8270D	2,3-Xylenol
GC/MS	EPA 8270D	2,4 & 2,5-Dimethylphenol
GC/MS	EPA 8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270D	2,4-Dichlorophenol
GC/MS	EPA 8270D	2,4-Dimethylphenol
GC/MS	EPA 8270D	2,4-Dinitrophenol
GC/MS	EPA 8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270D	2,5-Dimethylphenol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	2,6-Dichlorophenol
GC/MS	EPA 8270D	2,6-Dimethylphenol
GC/MS	EPA 8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270D	2-Acetylaminofluorene
GC/MS	EPA 8270D	2-Chloronaphthalene
GC/MS	EPA 8270D	2-Chlorophenol
GC/MS	EPA 8270D	2-Methyl-4,6-Dinitrophenol
GC/MS	EPA 8270D	2-Methylnaphthalene
GC/MS	EPA 8270D	2-Methylphenol
GC/MS	EPA 8270D	2-Naphthylamine
GC/MS	EPA 8270D	2-Nitroaniline
GC/MS	EPA 8270D	2-Nitrophenol
GC/MS	EPA 8270D	2-Picoline
GC/MS	EPA 8270D	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270D	2-Toluidine (o-Toluidine)
GC/MS	EPA 8270D	3 & 4-Methylphenol
GC/MS	EPA 8270D	3,3-Dichlorobenzidine
GC/MS	EPA 8270D	3,3'-Dimethylbenzidine
GC/MS	EPA 8270D	3,4-Dimethylphenol
GC/MS	EPA 8270D	3,4-Xylenol
GC/MS	EPA 8270D	3-Methylcholanthrene
GC/MS	EPA 8270D	3-Nitroaniline
GC/MS	EPA 8270D	4-Aminobiphenyl
GC/MS	EPA 8270D	4-Bromophenylphenyl ether
GC/MS	EPA 8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270D	4-Chloroaniline
GC/MS	EPA 8270D	4-Chlorophenol
GC/MS	EPA 8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270D	4-Nitroaniline
GC/MS	EPA 8270D	4-Nitrophenol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270D	7,12-Dimethylbenz (a) anthracene
GC/MS	EPA 8270D	Acenaphthene
GC/MS	EPA 8270D	Acenaphthylene
GC/MS	EPA 8270D	Acetophenone
GC/MS	EPA 8270D	alpha-, alpha-Dimethylphenethylamine
GC/MS	EPA 8270D	alpha-Pinene
GC/MS	EPA 8270D	Aniline
GC/MS	EPA 8270D	Anthracene
GC/MS	EPA 8270D	Aramite, Total
GC/MS	EPA 8270D	Atrazine
GC/MS	EPA 8270D	Benzaldehyde
GC/MS	EPA 8270D	Benzidine
GC/MS	EPA 8270D	Benzo (a) anthracene
GC/MS	EPA 8270D	Benzo (a) pyrene
GC/MS	EPA 8270D	Benzo (b) fluoranthene
GC/MS	EPA 8270D	Benzo (ghi) perylene
GC/MS	EPA 8270D	Benzo (k) fluoranthene
GC/MS	EPA 8270D	Benzoic acid
GC/MS	EPA 8270D	Benzyl alcohol
GC/MS	EPA 8270D	Bis (2-chloroethoxy) methane
GC/MS	EPA 8270D	Bis (2-chloroethyl) ether
GC/MS	EPA 8270D	Bis (2-chloroisopropyl) ether
GC/MS	EPA 8270D	Bis (2-ethylhexyl) phthalate
GC/MS	EPA 8270D	Butyl benzyl phthalate
GC/MS	EPA 8270D	Caprolactam
GC/MS	EPA 8270D	Carbazole
GC/MS	EPA 8270D	Chrysene
GC/MS	EPA 8270D	Cresols
GC/MS	EPA 8270D	Di(2-ethylhexyl)adipate

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	Diallate
GC/MS	EPA 8270D	Dibenz(a,h) anthracene
GC/MS	EPA 8270D	Dibenzofuran
GC/MS	EPA 8270D	Diethyl phthalate
GC/MS	EPA 8270D	Dimethoate
GC/MS	EPA 8270D	Dimethyl phthalate
GC/MS	EPA 8270D	Di-n-butyl phthalate
GC/MS	EPA 8270D	Di-n-octyl phthalate
GC/MS	EPA 8270D	Diphenyl ether
GC/MS	EPA 8270D	Disulfoton
GC/MS	EPA 8270D	Ethyl methane sulfonate
GC/MS	EPA 8270D	Famphur
GC/MS	EPA 8270D	Fluoranthene
GC/MS	EPA 8270D	Fluorene
GC/MS	EPA 8270D	Hexachlorobenzene
GC/MS	EPA 8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270D	Hexachloroethane
GC/MS	EPA 8270D	Hexachlorophene
GC/MS	EPA 8270D	Hexachloropropene
GC/MS	EPA 8270D	Hexachlrobutadiene
GC/MS	EPA 8270D	Indeno (1,2,3-cd) pyrene
GC/MS	EPA 8270D	Isophorone
GC/MS	EPA 8270D	Isosafrole
GC/MS	EPA 8270D	Methapyrilene
GC/MS	EPA 8270D	Methyl methane sulfonate
GC/MS	EPA 8270D	Methylbenzoate
GC/MS	EPA 8270D	Naphthalene
GC/MS	EPA 8270D	Nitrobenzene
GC/MS	EPA 8270D	N-Nitrosodiethylamine
GC/MS	EPA 8270D	N-Nitrosodimethylamine

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D	N-Nitroso-di-n-butylamine
GC/MS	EPA 8270D	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270D	N-Nitrosomethylethylamine
GC/MS	EPA 8270D	N-Nitrosomorpholine
GC/MS	EPA 8270D	N-Nitrosopiperidine
GC/MS	EPA 8270D	N-Nitrosopyrrolidine
GC/MS	EPA 8270D	o,o',o"-Triethylphosphorothioate
GC/MS	EPA 8270D	Parathion ethyl
GC/MS	EPA 8270D	Parathion methyl
GC/MS	EPA 8270D	p-Dimethylaminoazobenzene
GC/MS	EPA 8270D	Pentachlorobenzene
GC/MS	EPA 8270D	Pentachlorophenol
GC/MS	EPA 8270D	Pentachlronitrobenzene
GC/MS	EPA 8270D	Phenacetin
GC/MS	EPA 8270D	Phenanthrene
GC/MS	EPA 8270D	Phenol
GC/MS	EPA 8270D	Phenyl ether
GC/MS	EPA 8270D	Phorate
GC/MS	EPA 8270D	p-Phenylene diamine
GC/MS	EPA 8270D	Pronamide
GC/MS	EPA 8270D	Pyrene
GC/MS	EPA 8270D	Pyridine
GC/MS	EPA 8270D	Safrole, Total
GC/MS	EPA 8270D	Sulfotepp
GC/MS	EPA 8270D	Thionazin
GC/MS	EPA 8270E	1,1-Biphenyl
GC/MS	EPA 8270E	1,2,3-Trichlorobenzene
GC/MS	EPA 8270E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270E	1,2,4-Trichlorobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	1,2-Dichlorobenzene
GC/MS	EPA 8270E	1,2-Diphenylhydrazine
GC/MS	EPA 8270E	1,3,5-Trichlorobenzene
GC/MS	EPA 8270E	1,3,5-Trinitrobenzene
GC/MS	EPA 8270E	1,3-Dichlorobenzene
GC/MS	EPA 8270E	1,3-Dinitrobenzene
GC/MS	EPA 8270E	1,4-Dichlorobenzene
GC/MS	EPA 8270E	1,4-Dioxane
GC/MS	EPA 8270E	1,4-Naphthoquinone
GC/MS	EPA 8270E	1-Methylnaphthalene
GC/MS	EPA 8270E	1-Naphthylamine
GC/MS	EPA 8270E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270E	2,3,6-Trichlorophenol
GC/MS	EPA 8270E	2,3-Dimethylphenol
GC/MS	EPA 8270E	2,3-Xylenol
GC/MS	EPA 8270E	2,4 & 2,5-Dimethylphenol
GC/MS	EPA 8270E	2,4,5-Trichlorophenol
GC/MS	EPA 8270E	2,4,6-Trichlorophenol
GC/MS	EPA 8270E	2,4-Dichlorophenol
GC/MS	EPA 8270E	2,4-Dimethylphenol
GC/MS	EPA 8270E	2,4-Dinitrophenol
GC/MS	EPA 8270E	2,4-Dinitrotoluene
GC/MS	EPA 8270E	2,5-Dimethylphenol
GC/MS	EPA 8270E	2,6-Dichlorophenol
GC/MS	EPA 8270E	2,6-Dimethylphenol
GC/MS	EPA 8270E	2,6-Dinitrotoluene
GC/MS	EPA 8270E	2-Acetylaminofluorene
GC/MS	EPA 8270E	2-Chloronaphthalene
GC/MS	EPA 8270E	2-Chlorophenol
GC/MS	EPA 8270E	2-Methyl-4,6-Dinitrophenol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	2-Methylnaphthalene
GC/MS	EPA 8270E	2-Methylphenol
GC/MS	EPA 8270E	2-Naphthylamine
GC/MS	EPA 8270E	2-Nitroaniline
GC/MS	EPA 8270E	2-Nitrophenol
GC/MS	EPA 8270E	2-Picoline
GC/MS	EPA 8270E	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270E	2-Toluidine (o-Toluidine)
GC/MS	EPA 8270E	3 & 4-Methylphenol
GC/MS	EPA 8270E	3,3-Dichlorobenzidine
GC/MS	EPA 8270E	3,3'-Dimethylbenzidine
GC/MS	EPA 8270E	3,4-Dimethylphenol
GC/MS	EPA 8270E	3,4-Xylenol
GC/MS	EPA 8270E	3-Methylcholanthrene
GC/MS	EPA 8270E	3-Nitroaniline
GC/MS	EPA 8270E	4-Aminobiphenyl
GC/MS	EPA 8270E	4-Bromophenylphenyl ether
GC/MS	EPA 8270E	4-Chloro-3-methylphenol
GC/MS	EPA 8270E	4-Chloroaniline
GC/MS	EPA 8270E	4-Chlorophenol
GC/MS	EPA 8270E	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270E	4-Nitroaniline
GC/MS	EPA 8270E	4-Nitrophenol
GC/MS	EPA 8270E	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270E	7,12-Dimethylbenz (a) anthracene
GC/MS	EPA 8270E	Acenaphthene
GC/MS	EPA 8270E	Acenaphthylene
GC/MS	EPA 8270E	Acetophenone
GC/MS	EPA 8270E	alpha-, alpha-Dimethylphenethylamine
GC/MS	EPA 8270E	alpha-Pinene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Aniline
GC/MS	EPA 8270E	Anthracene
GC/MS	EPA 8270E	Aramite, Total
GC/MS	EPA 8270E	Atrazine
GC/MS	EPA 8270E	Benzaldehyde
GC/MS	EPA 8270E	Benzidine
GC/MS	EPA 8270E	Benzo (a) anthracene
GC/MS	EPA 8270E	Benzo (a) pyrene
GC/MS	EPA 8270E	Benzo (b) fluoranthene
GC/MS	EPA 8270E	Benzo (ghi) perylene
GC/MS	EPA 8270E	Benzo (k) fluoranthene
GC/MS	EPA 8270E	Benzoic acid
GC/MS	EPA 8270E	Benzyl alcohol
GC/MS	EPA 8270E	Bis (2-chloroethoxy) methane
GC/MS	EPA 8270E	Bis (2-chloroethyl) ether
GC/MS	EPA 8270E	Bis (2-chloroisopropyl) ether
GC/MS	EPA 8270E	Bis (2-ethylhexyl) phthalate
GC/MS	EPA 8270E	Butyl benzyl phthalate
GC/MS	EPA 8270E	Caprolactam
GC/MS	EPA 8270E	Carbazole
GC/MS	EPA 8270E	Chrysene
GC/MS	EPA 8270E	Cresols
GC/MS	EPA 8270E	Di(2-ethylhexyl)adipate
GC/MS	EPA 8270E	Diallate
GC/MS	EPA 8270E	Dibenz(a,h) anthracene
GC/MS	EPA 8270E	Dibenzofuran
GC/MS	EPA 8270E	Diethyl phthalate
GC/MS	EPA 8270E	Dimethoate
GC/MS	EPA 8270E	Dimethyl phthalate
GC/MS	EPA 8270E	Di-n-butyl phthalate

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Di-n-octyl phthalate
GC/MS	EPA 8270E	Diphenyl ether
GC/MS	EPA 8270E	Disulfoton
GC/MS	EPA 8270E	Ethyl methane sulfonate
GC/MS	EPA 8270E	Famphur
GC/MS	EPA 8270E	Fluoranthene
GC/MS	EPA 8270E	Fluorene
GC/MS	EPA 8270E	Hexachlorobenzene
GC/MS	EPA 8270E	Hexachlorocyclopentadiene
GC/MS	EPA 8270E	Hexachloroethane
GC/MS	EPA 8270E	Hexachlorophene
GC/MS	EPA 8270E	Hexachloropropene
GC/MS	EPA 8270E	Hexachlrobutadiene
GC/MS	EPA 8270E	Indeno (1,2,3-cd) pyrene
GC/MS	EPA 8270E	Isophorone
GC/MS	EPA 8270E	Isosafrole
GC/MS	EPA 8270E	Methapyrilene
GC/MS	EPA 8270E	Methyl methane sulfonate
GC/MS	EPA 8270E	Methylbenzoate
GC/MS	EPA 8270E	Naphthalene
GC/MS	EPA 8270E	Nitrobenzene
GC/MS	EPA 8270E	N-Nitrosodiethylamine
GC/MS	EPA 8270E	N-Nitrosodimethylamine
GC/MS	EPA 8270E	N-Nitroso-di-n-butylamine
GC/MS	EPA 8270E	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270E	N-Nitrosodiphenylamine
GC/MS	EPA 8270E	N-Nitrosomethylethylamine
GC/MS	EPA 8270E	N-Nitrosomorpholine
GC/MS	EPA 8270E	N-Nitrosopiperidine
GC/MS	EPA 8270E	N-Nitrosopyrrolidine

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	o,o',o"-Triethylphosphorothioate
GC/MS	EPA 8270E	Parathion ethyl
GC/MS	EPA 8270E	Parathion methyl
GC/MS	EPA 8270E	p-Dimethylaminoazobenzene
GC/MS	EPA 8270E	Pentachlorobenzene
GC/MS	EPA 8270E	Pentachlorophenol
GC/MS	EPA 8270E	Pentachlronitrobenzene
GC/MS	EPA 8270E	Phenacetin
GC/MS	EPA 8270E	Phenanthrene
GC/MS	EPA 8270E	Phenol
GC/MS	EPA 8270E	Phenyl ether
GC/MS	EPA 8270E	Phorate
GC/MS	EPA 8270E	p-Phenylene diamine
GC/MS	EPA 8270E	Pronamide
GC/MS	EPA 8270E	Pyrene
GC/MS	EPA 8270E	Pyridine
GC/MS	EPA 8270E	Safrole, Total
GC/MS	EPA 8270E	Sulfotepp
GC/MS	EPA 8270E	Thionazin
General Chemistry	EPA 9012B	Cyanide
General Chemistry	EPA 9013 EPA 9012B	Cyanide amenable to chlorination
General Chemistry	EPA 9030B EPA 9034	Sulfide
General Chemistry	EPA 9038	Sulfate
General Chemistry	EPA 9045D	pH
General Chemistry	EPA 9050A	Specific conductance
IC	EPA 9056A	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate (as N)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
IC	EPA 9056A	Nitrate (as NO3)
IC	EPA 9056A	Nitrate-nitrite (as N)
IC	EPA 9056A	Nitrate-nitrite (as NO3-NO2)
IC	EPA 9056A	Nitrite (as N)
IC	EPA 9056A	Nitrite (as NO2)
IC	EPA 9056A	Sulfate
General Chemistry	EPA 9065A	Phenols
General Chemistry	EPA 9071B	Oil and Grease
General Chemistry	EPA 9071B	Total Petroleum Hydrocarbons
General Chemistry	EPA 9095B	Free Liquid
General Chemistry	EPA 9251	Chloride
Preparation	**Method**	**Type**
Organic preparation	EPA 3546	Microwave Extraction
TCLP preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
SPLP Preparation	EPA 1312	Synthetic Precipitation Leaching procedure
Purge & Trap	EPA 5035A	Volatiles Prep
Acid Digestion	EPA 3050B	Metals Prep
Preparation	EPA 5050	Bomb Prep
Distillation	EPA 9030B	Sulfide

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2463.

Vice President