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STANDARD OPERATING PROCEDURE

# SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION

**MET-7742**

Revision: 3

February 19, 2010

Approved By: _____ Supervisor — 1/29/10 Date

_____ QA Manager — 1/29/10 Date

_____ Laboratory Manager — 1/29/10 Date

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: JDB Date: 1/20/11
Initials: _____ Date: _____
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**Standard Operating Procedure**
for
**SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION**
SOP: MET-7742
Revision 3
Date: January 28, 2010
Effective Date: February 19, 2010

## Procedure Change Form

| SOP Section Number | Date Procedure Change Implemented | Description of Procedure Change | Supervisor Approval and Notification of Other Analysts |
|---|---|---|---|
| 7.1.3 | 2/1/10 | 0.2% Na $BH_4$ is used. 4g $NaBH_4$ plus 1g NaOH dissolved in 2000ml of Reagent Water | JDB/AC |
| 11.3.1 | 2/1/10 | 20ml HCl is added, instead of 25ml | JDB/AC |
| 11.5 | 2/1/10 | 0.2%, not 0.5% $NaBH_4$ | JDB/AC |
| 11.6.2 | 2/1/10 | Analysis Order is ICV, ~~CRA~~ JB 2/4/10, ICB, CRA, CCV, CCB | JDB/AC |
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# SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION

## 1. SCOPE AND APPLICATION

1.1. This Standard Operating Procedure (SOP) describes the procedure used for the analysis of Selenium by borohydride reduction atomic absorption using EPA Method 7742 or Standard Method 3114B. This procedure describes the analysis procedures used to determine the analyte concentration and reporting limits listed. The sample preparation procedures are described in sample preparation SOPs MET-3010A, MET-3050B, and MET-TDIG.

1.2. This procedure is used to determine Selenium in water, soil, and tissue matrices. The procedure may be applied to other miscellaneous sample matrices providing that the analyst demonstrates the ability of the procedure to give data of acceptable quality in that matrix. The Method Reporting Limits (MRLs) are presented in Table 1. Method Detection Limits (MDLs) that have been achieved are also given.

1.3. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD)* may supersede the requirements defined in this SOP.

## 2. METHOD SUMMARY

2.1. Samples are prepared according to the nitric acid digestion procedure described in Method 3010 for aqueous and extract samples, the nitric/peroxide/hydrochloric acid digestion procedure described in Method 3050 for sediments, soils, and sludges, and the nitric/closed vessel digestion for tissues. Excess peroxide is removed by evaporating samples to near-dryness at the end of the digestion followed by dilution to volume and degassing the samples upon addition of urea. The selenium is converted to the +4 oxidation state during digestion in HCl. Selenium is then converted to its volatile hydride using hydrogen produced from the reaction of the acidified sample with sodium borohydride in a continuous-flow hydride generator.

2.2. The volatile hydrides are swept into, and decompose in, a heated quartz absorption cell located in the optical path of an atomic absorption spectrophotometer. The resulting absorption of the lamp radiation is proportional to the selenium concentration..

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## 3. DEFINITIONS

3.1. Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc...) The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.2. Independent Calibration Verification (ICV) - Initial calibration verification standards that are analyzed after initial calibration with newly prepared standards but prior to sample analysis, in order to verify the validity of the standards used in calibration. The ICV standards are prepared from a materials obtained from a source different from that used to prepare calibration standards.

3.3. Matrix Spike (MS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis.

3.4. Duplicate Sample (DUP) A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.5. Standard Curve - A standard curve is a calibration curve that plots concentrations of a known analyte standard versus the instrument response to the analyte. A linear regression calibration model is used. The appropriate criteria for assessing the validity of the calibration curve must be followed prior to quantitation of target analytes in actual sample analyses.

3.6. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.7. Continuing Calibration Verification Standard (CCV) - A mid-level standard analyzed at specified intervals. Used to verify that the initial calibration curve is still valid for quantitative purposes.

3.8. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into subsequent sample analyses.

3.9. Method of standard additions (MSA) - The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique attempts to compensate for a sample constituent that enhances or

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depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.

## 4. INTERFERENCES

4.1. Very high (>1000 mg/L) concentrations of cobalt, copper, iron, mercury, and, nickel can cause analytical interferences through precipitation as reduced metals and associated blockage of transfer lines and fittings.

4.2. Traces of peroxides left following the sample work-up can result in analytical interferences. Peroxides must be removed by evaporating each sample to near-dryness followed by reacting each sample with urea and allowing sufficient time for degassing before analysis.

4.3. Even after acid digestion, flame gases and organic compounds may remain in the sample. Flame gases and organic compounds can absorb at the analytical wavelengths and background correction should be used.

## 5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

## 6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Aqueous samples are typically collected in plastic containers. Aqueous samples are preserved with nitric acid (pH<2), then refrigerated at 4 ± 2°C from receipt until analysis.

6.2. Non-aqueous samples may be collected in plastic or glass jars. Non-aqueous samples are refrigerated at 4 ± 2°C from receipt until analysis. Non-aqueous samples should be analyzed as soon as possible following sampling.

6.3. Tissue samples are typically collected in plastic or glass jars. Prepared samples are stored frozen at < 10ºC until preparation. Tissue samples can be held up to one year.

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## 7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination.

7.1.2. Concentrated Hydrochloric Acid (HCl)

7.1.3. 4% Sodium Borohydride ($NaBH_4$). 20g $NaBH_4$ plus 2g of sodium hydroxide dissolved in 500mL of reagent water.

7.1.4. Urea ($H_2NCONH_2$)

7.1.5. Reagent and grade

7.2. Standards

7.2.1. Stock standard solutions may be purchased from a number of vendors. All standards purchased from vendors must be traceable to NIST or A2LA certified reference materials. Purchased standards are typically prepared at a concentration of 1000 ppm, and are prepared in 500 mL plastic bottles. The vendor-assigned expiration date is used.

7.2.2. A 1000 ppb intermediate stock standard is prepared by pipetting 0.100 mL of 1000 ppm stock standard, plus 1.0 mL of concentrated $HNO_3$, into a 100 mL volumetric and diluting to volume with reagent water.

7.2.3. The ICV is prepared at 10 ppb as described above. The 1000 ppb intermediate stock used for the ICV is prepared from a source independent of the original 1000 ppm purchased standard.

## 8. APPARATUS AND EQUIPMENT

8.1. Perkin Elmer AAnylst 200 - Atomic Absorption Spectrophotometer.

8.1.1. FIAS -100 Vapor Generator

8.1.2. Quartz Absorbance Cell

8.1.3. Selenium Hollow Cathode Lamp

8.2. Digestion Hot-Block capable of maintaining 50°C

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8.3. 50 mL Centrifuge Tubes

8.4. Hot Plates capable of maintaining 50°C

8.5. 50 mL Volumetric Flasks

## 9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, description of maintenance, date, and name. The log should contain a reference to return to analytical control.

9.2. Typical preventive maintenance measures include, but are not limited to, the following items:

- Cleaning the nebulizer and burner head
- Cleaning the gas liquid separator
- Inspection of the hollow cathode and deuterium lamp conditions

## 10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in Methods 7742 and 3114B, is also the responsibility of the department supervisor/manager.

## 11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water samples are first prepared by EPA Method 3010A (CAS SOP; MET-3010A), soil samples by EPA Method 3050B (CAS SOP; MET-3050) with modifications noted below, and tissue samples by closed vessel digestion (CAS SOP; MET-TDIG).

**Note:** The 3050B soil digestion is modified as follows: After the final peroxide addition (i.e. before the final reduction stage) add 5.0mL of concentrated

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hydrochloric acid and reduce the digestate volume to less than 5.0mL, but not to dryness. After cooling, dilute the digestate to 100mL with reagent water.

11.2. Digestate Preparation for hydride analysis

11.2.1. Waters – 25 mL of the water digestate is aliquoted to a 50 mL centrifuge tube and 20 mL of concentrated HCl is added. The centrifuge cap is loosely placed on the tube and the sample is heated in a digestion hot-block at 50°C for 30 minutes. After cooling the sample is diluted to 50 mL as needed resulting in a two fold dilution of the original digestate.

11.2.2. Soil/Sediment - 25 mL of the soil/sediment digestate is aliquoted to a 50 mL centrifuge tube. 0.5 g of Urea is added followed by 20 mL of concentrated HCl. The centrifuge cap is loosely placed on the tube and the sample is heated in a digestion hot-block at 50°C for 30 minutes. After cooling the sample is diluted to 50 mL as needed resulting in a two fold dilution of the original digestate.

11.2.3. Tissues – 8.0 mL of the tissue digestate is aliquoted to a 50 mL centrifuge tube. 0.4 g of Urea is added followed by 16 mL of concentrated HCl. The centrifuge cap is loosely placed on the tube and the sample is heated in a digestion hot-block at 50°C for 30 minutes. After cooling the sample is diluted to 40 mL as needed resulting in a five fold dilution of the original digestate.

11.3. Standard Preparation

11.3.1. Working standards are prepared in 100 mL volumetric flasks by aliquoting 0, 0.025, 0.05, 0.25, 0.375, and 0.5 mL of 1000 ug/ L intermediate standard into 50 mL centrifuge tube containing approximately 15 mL of reagent water. When analyzing tissues, an additional low point using 0.01 mL of the 1000ug/L intermediate is prepared. The appropriate amounts of concentrated HCl and/or $HNO_3$ are added in order to replicate the matrix of the initial 3010A, 3050B, or tissue digests. An additional 25 mL of concentrated HCl is added and the beakers are covered with a watchglass and heated on a hotplate at 50°C of 30 minutes. After cooling the standards are quantitatively transfered to 50 mL volumetric flask and diluted to volume with reagent water.

11.3.2. An Independent Calibration Verification (ICV) standard is prepared by aliquoting 0.375 mL of a 1000 ug/L intermediate standard prepared from a different source than the intermediate used for the calibration standards. This standard is then processed as described above.

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11.4. Method of Standard Additions (MSA):

11.4.1. For Soil and Tissue digestions the single point MSA technique is utilized by default. The matrix of these digests has been shown to produce a low bias of varying degrees when analyzed using the Perkin Elmer hydride system. This low bias is corrected for using the MSA procedure described in section 9.10.1 of EPA method 7000B Revision 2. After the sample digestate is prepared as described above in section 11.1 of this SOP, two 10 mL aliquots are placed in separate autosampler tubes. To the MSA aliquot 0.05 mL of 1000 ppb standard is added. After the two sample aliquots are analyzed the solution concentration ($C_X$) can be calculated using the following equation:

$$C_x = \frac{S_B * V_S * C_S}{(S_A - S_B)\, V_x}$$

Where:

$S_B$ = analytical signal of parent sample
$S_A$ = analytical signal of MSA sample
$V_S$ = volume of standard added to the MSA sample
$V_X$ = volume of sample aliquots
$C_S$ = concentration of spike solution

Alternately, $C_X$ can be calculated with solution concentrations using the following equation:

$$C_x = \frac{C_P * V_S * C_S}{(C_M - C_P)\, V_x}$$

Where:

$C_P$ = measured concentration of the parent sample
$C_M$ = measured concentration of the MSA sample
$V_S$ = volume of standard added to the MSA sample
$V_X$ = volume of sample aliquots
$C_S$ = concentration of spike solution

Once $C_X$ is determined the final sample concentration is calculated by the appropriate reporting software based on the initial sample aliquot and subsequent digestion and instrumental dilutions, and total solids if applicable.

**Note:** Since the change of volume from spiking the MSA aliquot is only 0.5%, which is insignificant relative to the accuracy and precision of the final result, this dilution is ignored in the calculations above.

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11.5. Instrument Setup

A Perkin Elmer AA200 is used in conjunction with the FIAS-100 hydride generator, and a burner head mounted quartz cell. The sample and carrier (10% HCl) flows are set at 4.2 mL/min and the borohydride (0.5% Sodium Borohydride) flow at 2.1 mL/min. The instrument is optimized to wavelength of 196.0 nm, with a slit width 1.0. The burner is ignited and the system is allowed to warm up for 10 minutes prior to starting the analysis.

**CAUTION: The hydride of selenium is very toxic. Precautions must be taken to avoid inhaling the gas.**

11.6. Calibration and Analysis

11.6.1. The sampling tube is placed into the calibration blank, allowed to come to equilibrium, and then analyzed. The remaining calibration standards are analyzed similarly in ascending order. After the calibration curve is complete the r value is calculated. If the r value is not $\geq$ 0.995 then the calibration is rejected and must be re-analyzed.

11.6.2. Immediately following the calibration the ICV solution is analyzed followed in order by an Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), Continuing Calibration Blank (CCB), and the MRL check standard (i.e. the low calibration standard). ICV and CCV recoveries must fall within ± 10% of their true value, and ICB and CCB results must be less than 3x MDL.

11.6.3. Once the above calibration and QC check standards have been successfully run the samples are ready to be analyzed. After a maximum of 10 samples have been analyzed an additional set of CCV and CCB standard must be analyzed using the criteria described above. If one or both of these standards are out of control the problem must be corrected, the instrument recalibrated, the initial QC check standards analyzed, and the samples following the last compliant CCV/CCB check re-analyzed.

11.7. List any matrix modifiers or reducing agents used in the analysis on the associated raw data

## 12. QUALITY CONTROL

12.1. Initial Precision and Recovery Validation

The precision of the procedure must be validated before sample analysis, or whenever significant changes to the procedures have been made. To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed.

12.2. Method Detection Limits

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12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike seven blank matrix (water or soil) samples at a level near, or below the MRL. Follow the analysis procedures in Section 11 to analyze the samples.

12.2.2. Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to the CAS *SOP Performing Method Detection Limit Studies and Establishing Limits of Detection and Limits of Quantification* . The MDL study must be verified annually.

12.3. Limits of Quantification - LOQ

12.3.1. Method 7000B requires the laboratory establish a LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard prepared at the LOQ concentration levels or use of the LOQs as the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries must be within **30%** of the true values to verify the data reporting limit. Refer to the CAS *SOP Performing Method Detection Limit Studies and Establishing Limits of Detection and Limits of Quantification.*

12.4. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). For example projects managed under the DoD ELAP must follow requirements defined in the DoD *Quality Systems Manual for Environmental Laboratories.* General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management (ADM-DOD).* General QC Samples and their default control criteria are:

12.4.1. Method Blank

12.4.1.1. A method blank is extracted and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the MRL for standard applications, or ½ the MRL for DoD projects, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis. Reanalysis may only be done once.

12.4.2. Lab Control Sample (LCS)

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12.4.2.1. The laboratory control sample for water samples is composed of analyte-free water into which Selenium is spike. The laboratory control sample for soils and tissues consists of an appropriate reference material (e.g. ERA DO65-540 for soils, NRCC TORT-3 for tissues). The LCS is designed to monitor the accuracy of the procedure. The concentration of the spike in the LCS matrix should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

12.4.3. A lab control sample (LCS) must be prepared and analyzed with every batch of 20 (or fewer) samples. Calculate the LCS recovery as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

The acceptance criterion is **80-120%.** Acceptance limits for other reference materials is specific to the material used. Also, other project-specific limits may be required. If the LCS fails acceptance criteria, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-extraction and reanalysis.

12.4.4. Matrix Spike

12.4.5. A matrix spike (MS) must be prepared and analyzed with every batch of 20 (or fewer) samples. The MS is prepared by adding a known volume of the matrix spike solution to the sample and determining the spiked sample concentration. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

12.4.6. The acceptance limits for the MS is **75-125%** for all matrices. Also, other project-specific limits may be required. If the MS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-preparation and reanalysis.

12.4.7. A Duplicate sample is prepared and analyzed one per batch, or per 20 samples. Calculate Relative Percent Difference (RPD) as:

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$$RPD = \frac{|R1 - R2|}{(R1 + R2) / 2} \times 100$$

Where R1= result for the sample
R2= result for the sample duplicate

The RPD criterion is **20%**. If outside the limit, redigest the sample batch. Determine if the sample is non-homogenous and redigest if it is homogenous.

12.5. Recovery test (post-digestion spike)

12.5.1. The recovery test must be done on all water samples within a digestion batch were that batch's MS fails.

12.5.2. The same sample from which the MS/MSD aliquots were prepared (assuming the MS/MSD recoveries are unacceptable) should also be spiked with a post digestion spike. Otherwise another sample from the same preparation should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within **80% to 120%** of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed.

12.6. Prior to preparation of samples, blanks should be analyzed to determine possible interferences from sample handling steps, reagents, or glassware. If the blanks show contamination, the source of the contamination should be isolated and minimized.

## 13. DATA REDUCTION AND REPORTING

13.1. The concentration of the analyte(s) in the sample digest (Cex) is calculated using the calibration curve. The concentration of analytes in the original samples is computed using the following equations:

Aqueous Samples:

$$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

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Where Cex = Concentration in digestate in µg/mL
Vf = Final volume of extract in mL
D = Dilution factor
Vs = Volume of sample digested, liters

Nonaqueous Samples:

$$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$$

Where Cex = Concentration in digestate in µg/mL
Vf = Final volume of extract in mL
D = Dilution factor
W = Weight of sample digested in grams. The wet or dry weight may be used, depending upon the specific client requirements.

13.2. Sample concentrations are reported when all QC criteria for the analysis has been met. Reported results not meeting QC criteria must be qualified with a standard CAS footnote.

13.3. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details.

13.4. Reporting

13.4.1. Reports are generated in the MARRS or Harold reporting softwares by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). The compiled data is also used to create EDDs.

13.4.2. As an alternative, reports are generated using Excel© templates located in R:\ICP\FORMS. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The results are then transferred, by hand or electronically, to the templates.

## 14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Nonconformity and Corrective Action* for correct procedures for identifying and documenting such data. Procedures

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for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

## 15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP *Performing Method Detection Limit Studies and Establishing Limits of Detection andQuantification* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

## 16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

## 17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

## 18. TRAINING OUTLINE

18.1. Review literature (see references section). Review the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.2. The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of approximately two weeks. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

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18.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file.

## 19. CHANGES SINCE THE LAST REVISION

19.1. Sec 1.3 is new.
19.2. Updated references to include method 7000B.
19.3. Sec 3.9 Added definitions for MSA.
19.4. Remove old section 7.2.3.
19.5. Sec 11.2 is new
19.6. Sec 11.3.1 Updated range on procedure for standard prep.
19.7. Sec 11.3.2 Changed ICV amt from 1 ml to 0.375 ml.
19.8. Sec 11.4 MSA is new
19.9. Sec 12.2.2 Updated MDL reference and requirement to verify annually.
19.10. Sec 12.3 on LOQ is new.
19.11. Sec 12.4 is new.
19.12. Sec 12.4.1 Rewritten to include DoD criteria.
19.13. Sec 12.4.3 and 12.4.6 Updated recovery limits to follow 7000B
19.14. Sec 12.4.7 Updated RPD limit to follow 7000B.
19.15. Sec 12.5 is new.

## 20. REFERENCES

20.1. EPA Method 7742, Revision 0, 1994

20.2. EPA Method 7000B, Revision 2, 1998

20.3. *Standard Methods* 3114B, 20th Edition

### TABLE 1

### SELENIUM MRLs, and MDLs

| Analyte | Method Reporting Limit | Method Detection Limit |
|---|---|---|
| Water | 1.0 ug/L | 0.2 ug/L |
| Soil | 0.1 mg/Kg (dry) | 0.03 mg/Kg (dry) |
| Tissue | 0.1 mg/Kg (dry) | 0.05 mg/Kg (dry) |