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ANALYSIS OF SOIL FOR ARSENIC

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1.0 SCOPE AND APPLICATION

The purpose of this protocol is to provide guidelines for the analysis of soil samples for arsenic (As) by hydride generation atomic fluorescence spectrometry (HGAF). This protocol describes the methodology and all other analytical aspects involved in the analysis of soil samples for arsenic by hydride generation atomic fluorescence spectrometry.

2.0 SUMMARY OF THE METHOD

This method involves the extraction of the analyte from soil samples using a 50% ultra-pure nitric acid, and subsequent analysis by hydride generation atomic fluorescence spectrometry. The analyte is identified by the presence of fluorescence signal at its specific wavelength. Quantitation of the analyte is carried out using a standard calibration curve. Recovery of the analyte is monitored using samples that are spiked with standards prior to the extraction process.

3.0 INTERFERENCES

3.1 In most cases contamination occurs through reagents, sample handling and sample collection materials. These sources of contamination are monitored by the analysis of method blanks as described in Section 8.1.

3.2 Memory effects are a common source of contamination in HGAF. A proper cleaning cycle must be employed to ensure the complete removal of any traces of analytes from the sample (atomizer) cell and sample transport system.

3.3 Once samples are brought to the analytical lab, all sample handling is performed in a clean laboratory environment under HEPA filtered air. All sample preparation steps must be carried out in the Class 100 clean lab environment to eliminate contamination that may result from the environment. If samples need to be carried out from the Class 100 clean lab at any stage of the sample processing, they should be tightly capped before taken out from the clean lab and never be opened to outside environment. All sample analyses must be performed in

the Class 10,000 clean lab to eliminate any contamination that may occur during analysis. Proper procedures for the use of ACS Inorganic Class 100/10,000 clean lab facility are described in NHX/SOP-300-006, "Standard Operating Procedures for the ACS Clean Lab Facility."

3.4 Contaminants in reagents, solvents, labware (glassware and plasticware) and other components of sample processing apparatus can also cause interferences which may lead to increased analyte and/or background signals. Atomic fluorescence reagents and apparatus are checked routinely to make sure they are free from interferences under the conditions of the analysis by method blanks as described in Section 8.1.

4.0 SAFETY

Since the toxicity of the chemicals used in this method is not clearly defined, they should be treated as potential health hazards at all times. All laboratory personnel are advised to take full precautions (wearing gloves, eye protection etc.) when handling these chemicals and personal exposure to these chemicals should be minimized. Safety issues in the ACS clean lab facility are discussed in the NHX/SOP-300-002, "Standard Operating Procedures for Personnel Safety in the ACS Inorganic Clean Lab Facility" in detail.

5.0 EQUIPMENT

5.1 Laboratory Equipment

5.1.1 All labware (glassware and plasticware) must be thoroughly cleaned by washing with detergent and water followed by rinsing with deionized water. Once the washing is completed, all labware must be soaked in 50% HNO_3 for not less than 24 hours, or heated in 50% HNO_3 for not less than 2 hours, rinsed with deionized water and dried in HEPA filtered air. All cleaned labware must be stored in an airtight plastic bag if they are not used immediately. Cleaning of labware in the ACS Inorganic Clean Lab Facility is

described in ACS/SOP-174-002, "Standard Operating Procedures for Cleaning Labware in the ACS Inorganic Class 1000/10,000 Clean Lab Facility."

5.1.2 Labware

Beakers, Teflon and glass

Volumetric flasks, polypropylene and glass

Pipettes, glass

Extraction tubes, polypropylene

Storage bottles, plastic

Filter paper, Whatman

Sieves, stainless steel or brass or plastic (250 Fm pore size or larger)

5.1.3 Apparatus

Hot plates

Variable speed horizontal shaker

Automatic pipettors, fixed and variable volume

Refrigerator

Centrifuge

5.1.4 Analytical balance with 0.01 mg to 205 g weighing range. Capable of 0.01 mg readability.

5.2 Hydride Generation Atomic Fluorescence Spectrometer (HGAF)

5.2.1 The spectrometer must have the capability of automation to reduce personnel errors to a minimum. Wavelength selection, lamp current setting, slit width setting and PMT gain setting should be able to be controlled through the spectrometer operational software. This is necessary to provide consistency of these parameters during the analysis from sample to sample.

5.2.2 Vapor generator must have the capability to generate hydride species in both continuous mode and flow injection mode. It should have an active pumping system to deliver the sample, blank and reagent to the mixing chamber with high reproducibility. The presence of an efficient separation technique to separate gaseous species from the solution is also important and finally there

should be an effective transport system to transport the gaseous species to the atomizer cell.

5.2.3 An autosampler is required to deliver reproducible volumes of sample and reagents into the reaction chamber or mixing vessel in a consistent manner (addition of reagents in proper order).

5.2.4 The data system must be capable of data acquisition, storage and processing including producing an output. Data acquisition software must allow the use of different integration periods and also be capable of acquiring data under both peak height and peak area modes. Data processing software must be capable of analyzing the raw data, calculating net fluorescence, calculating statistics (mean, standard deviation, relative standard deviation), constructing calibration curves and calculating analyte concentrations. It is also recommended that the data system be able to transfer its data from the instrument to peripheral computers for further processing.

6.0 REAGENTS AND STANDARDS

6.1 Reagents

Hydrochloric acid ("Trace Metal" grade or better)
Nitric acid ("Optima" grade or better)
Sodium tetrahydroborate (ACS reagent grade)
Sodium hydroxide (ACS reagent grade)
Potassium iodide (ACS reagent grade)
Ascorbic acid (ACS reagent grade)
Methanol (HPLC grade)

6.2 Stock Standard Solutions

Commercially available, spectrometric grade, certified arsenic reference solution (NIST traceable) at 1000 mg/L is used as the stock solution.

6.3 Working Standard Solutions

Working standard solutions are prepared by diluting the stock standard solution using 2% HCl to stabilize the analyte. Working standard solutions are stored in acid washed polypropylene containers and prepared freshly when necessary.

6.4 Calibration Standards

A series of calibration standards are prepared on a daily basis by dilution of working standard solutions with deionized water. A suitable matrix component may also be added to calibration standards before they are made up to their final volumes, to stabilize the analyte and also to match the matrix to that of prepared samples. At least five different concentration standards must be used to define the linear portion of the calibration curve, and a calibration blank should also be prepared and included in the calibration. Calibration blank should contain all the components in the calibration standards except the analyte. Calibration standards should cover the full range of analyte concentrations expected to be analyzed by the instrument. The concentrations of calibration standards are based on the concentrations found in typical dust samples, and are given in Table 1.

7.0 SAMPLE STORAGE

All samples are placed in appropriate airtight plastic containers and are stored in a refrigerator prior to sample preparation.

All prepared samples are stored in plastic containers with air tight screw caps, in a refrigerator.

8.0 QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

8.1 Method Blanks

Method blanks are extracting solvent carried throughout the sample preparation procedure along with each batch of samples analyzed. Analysis of these will provide information on contamination that result from sample extraction and preparation steps.

8.2 Method Controls

Method controls are extracting solvents spiked with an NIST certified calibration standard at levels described in Section 6.4 and treated the same as method blanks. Analysis of these will provide information on recoveries of the target analyte from sample extraction and preparation steps.

8.3 Performance Evaluation Samples

Once acceptable performance of the method is demonstrated for the analyte by method controls, a performance evaluation sample is analyzed to further evaluate the overall performance of the method. The performance evaluation sample must contain the target analyte with the level certified by an approved material certification agency, in a matrix almost identical to the sample matrix.

Performance of the method may also be evaluated by the analysis of a simulated sample provided by NIST.

8.4 Custody and Sample Tracking Procedures

8.4.1 Sample Tracking Procedures

Each and every sample will be identified by a unique bar code. These bar codes are clearly placed on all samples and will be inspected at various stages, from the transportation to the field, to their final analysis.

Detailed information regarding sample transportation to the monitoring sites (sampling sites), and to sample analysis site (analytical lab), sample weighing, sample extraction and sample analysis will be recorded on RTI log books.

When a batch of samples is submitted to the analytical lab for analysis, the analyst is responsible for recording sample information along with the analysis information in the sample submission form. An example for this type of record is given in Figure 1.

Method blanks and method control samples will be linked to sample batches by a date code. Sample preparation and analysis dates will be recorded in laboratory notebooks.

8.4.2 Custody

Custody procedures will be designed to track the sample flow within the study. Each sample is accompanied by a custody document which provides the information on that sample. An example is given in Figure 2.

9.0 SAMPLE PREPARATION

9.1 Processing and Weighing

The sieving unit (the sieve and the collector) is washed with deionized water and methanol in that order and air dried prior to use and between samples. The soil sample is passed through the sieve and mixed with a Teflon spatula to facilitate uniform mixing. Lumps of soil are forced through the screen, but stones, roots, grass and other debris are discarded. If the sample is too wet, it is evenly distributed on a plastic plate and air dried prior to sieving. About 1 g of representative sample of the sieved soil is obtained for analysis by spreading the soil on the collector plate and taking small portions of sample randomly. The sample is taken into a preweighed, 35 mL polypropylene extraction tube with an airtight plastic screw cap and the weight of the "sample + tube" is recorded in laboratory notebook. The remainder of the sieved sample is transferred to an air tight plastic bag. All samples are labeled appropriately and stored in a refrigerator until analysis.

9.2 Sample Extraction

Ten mL of 50% HNO_3 is added to all samples in extraction tubes. Tubes are sealed with an airtight plastic screw caps and are placed on a horizontal shaker for 30 minutes. The extract is decanted into a 125 mL Teflon beaker and the extraction is repeated twice with a fresh portion of acid each time (total extract = 30 mL). All three extracts are combined in a 125 mL beaker. The extract is transferred to a polypropylene test tube, centrifuged and 20 mL of clear solution is transferred to a 125 mL Teflon beaker. The extract is evaporated down to about 0.5 mL on a hot plate. The beaker is removed from the hot plate, allowed to cool down, and 25 mL of 50% HCl is added. The solution is warmed and filtered if necessary using a Whatman filter paper into a 50 mL polyproylene volumetric flask and

dilute with deionized water. The final solution is transferred to a clean plastic storage bottle and is stored in a refrigerator until analysis.

10.0 CALIBRATION AND STANDARDIZATION

An acceptable performance of the instrument must be demonstrated and documented as described in Section 2.3 of NHX/SOP-300-001, "Standard Operation Procedures for the Operation of PS Analytical Hydride Generation Atomic Fluorescence Spectrometer (HGAF)," prior to any sample analysis and the demonstrated acceptable level must be maintained throughout the analysis. A new calibration curve is constructed at the beginning of each day of analysis. Additional calibration checks are performed periodically, when necessary.

10.1 Initial Calibration

10.1.1 An instrument performance test is performed prior to the analysis of any samples as described in Section 2.3 of the NHX/SOP-300-001, "Standard Operating Procedure for the Operation of PS Analytical Hydride Generation Atomic Fluorescence Spectrometer (HGAF)". If the performance test meets the acceptance criteria, defined in the SOP, proceed with the initial calibration (starting from Section 10.1.2).

If the performance test does not meet the acceptance criteria necessary steps (Section 10.2.5) will be taken to bring the performance of the instrument to an acceptable level.

10.1.2 Calibration blank and calibration standards are analyzed for arsenic starting from the lowest concentration to the highest. Each solution (blank and standards) is analyzed at least in duplicate in order to perform statistical evaluation of the analysis. For each calibration standard and blank, the mean, the standard deviation (SD) and, the percent relative standard deviation (%RSD) is calculated. If the RSD is greater than 10% for any standard, further aliquots of that solution will be analyzed or necessary steps will be taken to improve the performance of the instrument.

10.1.3 The fluorescence intensity is measured for each calibration standard and a least square linear regression calibration curve is constructed as follows for the analyte by the instrument software.

$$y = a + bx$$

where:

y = net fluorescence intensity

a = y intercept

b = slope

x = analyte concentration (ng/mL)

10.2 Initial Calibration Check

At the beginning of each day of analysis, a new calibration curve is constructed. The performance of the initial calibration must be verified prior to any sample analysis as follows.

10.2.1 Linearity of the calibration curve must be verified both visually and mathematically. The correlation coefficient (r) must be greater than 0.99.

10.2.2 Concentration of the analyte in the calibration standards calculated using the measured signal and the calibration curve must be ±20% of the nominal concentration for lowest calibration standard and ±10% for all other calibration standards.

10.2.3 Analyze a predetermined QC check and compare the new results with the previous results obtained from Section 10.1.2.

10.2.4 Calculate the difference between the two values and verify that it has not changed by more than 10% from the initial calibration. If it has changed more than the above limits, adjustments must be made to restore system sensitivity (Section 10.2.7) and recalibration is required.

10.2.5 Analyze a NIST certified standard to ensure that the analytical accuracy and precision remains within acceptable limits. If the results differ more than 10% from the certified value or from one of the value limits, a second aliquot will be analyzed or the NIST standard will be re-prepared and re-analyzed. Questionable situations will be reviewed with the laboratory manager or facility supervisor and the QA officer, and a final decision taken at that time.

10.2.6 Control Charts

Daily performance test results will be plotted against time (in days) for the HGAF instrument. The sensitivity in (Pk.Ht. per ng mL^{-1}) will be used as the performance parameter. This plot will be used as a control chart to continuously monitor the performance of the instrument over time. The operator of the instrument is responsible for maintaining the chart. The chart must be presented to the laboratory manager or to the task leader for his/her review at least once a month. The control chart and data must be stored along with the sample analysis data.

10.2.7 Remedial Actions

Possible remedial action are described in the "Standard Operating Procedure for the Maintenance of PS Analytical Hydride Generation Atomic Fluorescence Spectrometer" (NHX/SOP-300-005) and in the "Standard Operating Procedure for the Operation of the PS Analytical Hydride Generation Atomic Fluorescence Spectrometer" (NHX/SOP-300-001).

11.0 ANALYSIS PROCEDURE

11.1 Analysis Conditions

The instrumental parameters for the analyte are given in Table 2. Each sample is analyzed similar to standards as described in Section 10.1.2.

11.2 Analysis Batches

Sample preparation and analysis are carried out in batches of samples. A batch is defined as a fixed number of samples that can be handled conveniently and efficiently in the laboratory. A batch includes 30 samples, 2 method blanks, and 1 method control. The order of analysis is:

QC check standard

Calibration blank

NIST reference standard

Method blank

Method control

Samples

Periodic QC checks

Prior to any sample analysis, a set of calibration standards will be analyzed and a calibration curve constructed. A pre-determined calibration standard will be selected as the QC check standard and analyzed periodically along with the calibration blank immediately after the calibration, every 10 samples and at the end of the analysis. All QC check standards must be within 10% of their nominal value or initial value for the data to be accepted. Samples for which the QC checks differ by more than 10% of their nominal and initial values will be re-analyzed.

Method blanks will be analyzed with each batch of samples and subtracted from method controls/samples when they are found to be significant. The analyst will make this determination.

A method control will be run with each batch of samples and the recovery of analyte(s) will be monitored. If the recoveries are outside the range 90 to 100% from the certified value or from one of the value limits, a second aliquot will be analyzed. Questionable results will be reviewed with the laboratory manager or facility supervisor and the QA officer and a final decision is made at that time.

11.3 Data Acquisition

Data acquisition is carried out by the instrument software. The fluorescence signal given out by the excited atoms is measured during data acquisition. Once the data acquisition is completed, the concentration of the analyte in the sample is calculated based on the standard calibration curve.

11.4 Identification of the Analyte

Analyte is identified by the presence of fluorescence signal, at wavelength specific to the analyte. The analyte is present in the sample, if the concentration of the analyte is greater than its method detection limit (MDL) (Section 12.0).

12.0 METHOD PERFORMANCE

12.1 Method Detection Limit (MDL)

Method detection limit (MDL) is defined as 3 times the standard deviation of method blanks. Method detection limit is calculated for the analyte using the following equation;

$$MDL = 3 \times SD_{bl}$$

MDL = Method detection limit, ng/mL or ppb.

SD_{bl} = Standard deviation of method blanks, ng/mL or ppb.

12.2 Method Quantitation Limit (MQL)

Method quantitation limit (MQL) is defined as 10 times the standard deviation of method blanks. Method quantitation limit is calculated for the analyte using the following equation;

$$MQL = 10 \times SD_{bl}$$

MQL = Method quantitation limit, ng/mL or ppb.

SD_{bl} = Standard deviation of the method blank, ng/mL or ppb.

13.0 DATA MANAGEMENT

13.1 Processing of Data Output

Instrument software is capable of processing the raw data (fluorescence intensities) and generating printed output. The output contains information on sample I.D., analyte tested, fluorescence, concentration of the analyte in the sample, and statistical data. This type of output helps in visual examination of data for any unusual behavior and/or any inadequate method performance.

13.2 Data Storage

All raw data acquired during the analysis are stored on the hard drive of the computer dedicated to the instrument, along with the processed data. At the end of each day of analysis all data are transferred to floppy disks. These floppy disks are labelled and stored in a central storage area along with the printed outputs.

13.3 Data Transmission

Once reviewed and accepted by the laboratory manager, raw and processed data generated by the instrument are converted to ASCII format and handed over to the database manager for further processing. Example of an ASCII data file generated by HGAF instrument software is given in Table 3.

14.0 CALCULATIONS

The concentration of the analyte in the sample is calculated during the analysis by the instrument software against the reference calibration curve (Section 10.1.3) using the measured fluorescence as follows:

$$C_x = \frac{(y - a)}{b}$$

where:

C_x	=	Uncorrected concentration of the analyte in ng/mL or parts-per-billion (ppb).
y	=	The measured fluorescence for the sample.
a	=	y intercept of the reference calibration curve in fluorescence.
b	=	Slope of the reference calibration curve in fluorescence intensity per ng mL^{-1}.

Samples with concentrations below MDL/MQL will be expressed appropriately, whereas over-range samples will be diluted and re-analyzed.

Once the data are transmitted to the central database, calculations may be performed to express the final results in any of the following formats.

14.1 The following format may be used to calculate the final results in ng of analyte per gram of soil sampled.

$$C_{x2} = (C_x - C_{MB}) \times V_x \times DF \times \frac{1}{WT}$$

where:

C_{x2}	=	Corrected concentration of the analyte in ng/g of particulates.
C_x	=	Uncorrected concentration of the analyte in ng/mL or ppb.
V_x	=	Final volume of the sample in mL.
DF	=	Dilution factor.
WT	=	Mass of the soil sample in g.
C_{MB}	=	The average concentration of the analyte in method blanks in ng/mL or ppb.

TABLE 1. SUGGESTED CALIBRATION SOLUTIONS

	Concentration of the Analyte [ng/mL]						
Analyte	#1	#2	#3	#4	#5	#6	#7
As	0.0	0.05	0.1	0.3	0.5	0.7	1.0

TABLE 2. OPERATING PARAMETERS FOR THE INSTRUMENT

Hydride generation atomic fluorescence spectrometer [HGAF]	
Experimental parameters:	
Element	As
Source	BDHCL
Power-current (mA)	Iry - 27.5 Boost - 35
Sample volume (mL)	10 - 12
Measurement mode	Peak Ht.
Sensitivity	Range - 100 Fine - 10
PMT setting[a]	5.5
Analysis cycle:	
Delay (sec)	10
Rise (sec)	25
Analysis (min)	0.5
Memory (sec)	40
Reductant	1.3% $NaBH_4$ in 0.1 M NaOH
Blank	25% HCl in 1% KI and 0.05% Ascorbic acid
Carrier gas flow (L/min)	0.3 (Ar)

[a]Depends on the lamp intensity.

TABLE 3. EXAMPLE OF AN ASCII DATA FILE GENERATED BY THE HGAF SOFTWARE

No.	Tag	Sample	Ref	El	Concentration	Runs	SD	Output		Time	Date
2	Std	1	0.000	As	0.000 ppb	2	0.034	O/P	0.034	17:15	20Dec94
Posn=5	Runs:1=0.000	2=0.068									
3	Std	2	0.050	As	0.050 ppb	2	9.723	O/P	14.56	17:22	20Dec94
Posn=6	Runs:1=4.843	2=24.29									
4	Std	3	0.100	As	0.100 ppb	2	0.125	O/P	10.20	17:27	20Dec94
Posn=7	Runs:1=10.33	2=10.08									
5	Std	4	0.300	As	0.300 ppb	2	0.558	O/P	35.90	17:31	20Dec94
Posn=8	Runs:1=36.46	2=35.34									
6	Std	5	0.700	As	0.700 ppb	2	0.334	O/P	86.67	17:36	20Dec94
Posn=9	Runs:1=87.00	2=86.33									
7	Std	6	1.000	As	1.000 ppb	2	60.34	O/P	60.34	17:40	20Dec94
Posn=10	Runs:1=120.6	2=0.000									
8	Blank	BLANK		As	0.022 ppb	1	0.000	ppb	2.563	18:38	20Dec94
Posn=5	Runs:1=2.563										
Least Sq To 1.000ppb RSF=1.000 Slope=139.8 CorrCoeff=0.999 YIntercept=-0.57											
9	AutoQC	0.300		As	0.289 ppb	1	0.000	ppb	39.88	18:41	20Dec94
Posn=8	Runs:1=39.88										
11	112294	RB1		As	0.012 ppb	1	0.000	ppb	1.126	18:45	20Dec94
Posn=11	Runs:1=1.126										
12	112294	RB2		As	0.011 ppb	1	0.000	ppb	1.097	18:47	20Dec94
Posn=12	Runs:1=1.097										
13	112294	LWW-BLK1		As	0.244 ppb	1	0.000	ppb	33.59	18:50	20Dec94
Posn=13	Runs:1=33.59										
14	112294	LWW-BLK2		As	0.253 ppb	1	0.000	ppb	34.91	18:52	20Dec94
Posn=14	Runs:1=34.91										
15	112294	LWW-BLK3		As	0.251 ppb	1	0.000	ppb	34.58	18:54	20Dec94
Posn=15	Runs:1=34.58										
16	112294	LWW-003		As	0.008 ppb	1	0.000	ppb	0.546	18:56	20Dec94
Posn=16	Runs:1=0.546										
19	112294	LWW-005		As	0.194 ppb	1	0.000	ppb	26.67	19:08	20Dec94
Posn=17	Runs:1=26.67										
20	112294	LWW-007		As	0.400 ppb	1	0.000	ppb	55.42	19:10	20Dec94
Posn=18	Runs:1=55.42										
22	112294	LWW-009		As	0.093 ppb	1	0.000	ppb	12.56	19:15	20Dec94
Posn=19	Runs:1=12.56										
23	Blank	BLANK		As	0.041 ppb	1	0.000	ppb	5.185	19:18	20Dec94
Posn=4	Runs:1=5.185										
24	AutoQC	0.300		As	0.300 ppb	1	0.000	ppb	41.45	19:20	20Dec94
Pos=7	Run:1=41.45										

ACS - SAMPLE SUBMISSION FORM

Project: _____ Number of Samples: _____

Date: _____ Analyst: _____

Sample bar codes for all the samples submitted			

Analyte	Standard # or sample #	Concentration	Comments

Figure 1. Batch sample submission form (example).

NATIONAL HUMAN EXPOSURE ASSESSMENT SURVEY

SAMPLE COLLECTION AND CUSTODY RECORD

RTI/EOHSI CONSORTIUM

SAMPLE CODE: ***PARTICIPANT I.D.:*** ***COUNTY I.D.:***

SAMPLE TYPE:	House Dust
TO BE ANALYZED FOR:	As

COLLECTION COMMENTS: _____

_____

_____

_____

PROCESSING COMMENTS: _____

_____

_____

_____

CUSTODY RECORD

--- CUSTODY OF ---				
ORG.	*INITIALS*	*ID*	*DATE*	***OPERATION PERFORMED***
RTI				Sample Collected
RTI				Sample Shipped to RTI

Research Triangle Institute, Analytical and Chemical Sciences, P.O. Box 12194, RTP, NC 27709

Figure 2. Example of a printed custody record.