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Post Office Box 12194
Analytical and Chemical Sciences
Research Triangle Park, NC 27709-2194

AUTHOR(s):

Bernardo Date: 05-31-95

Date:

Date:

APPROVED BY:

Principal Investigator: E Pellizzari Date: 6/2/95

QA Officer: D J Smith Date: 6/12/95

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ANALYSIS OF AIR PARTICULATE FOR LEAD, ARSENIC, CADMIUM, AND CHROMIUM

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this protocol is to provide guidelines for the analysis of Teflon filter samples for toxic metals, either by graphite furnace atomic absorption spectrometry (GFAAS) or by hydride generation atomic fluorescence spectrometry (HGAF). These Teflon filters were used in personal air monitoring samplers to collect particulate in air.

1.2 The selected toxic metals pertained to this analysis are given in Table 1, along with the analytical techniques that will be used.

2.0 SUMMARY OF THE METHOD

This method involves the extraction of analytes from Teflon filter samples using 50% ultra-pure nitric acid, and subsequent analysis by atomic absorption/fluorescence spectrometry. The analytes are identified by the presence of absorption or fluorescence signal at their specific wavelengths and quantitation of analytes is carried out using standard calibration curves. Recovery of analytes 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 both field blanks and method blanks as described in Sections 8.1 and 8.2.

3.2 Memory effects are a common source of contamination in HGAF and in GFAAS. Flushing of sample delivery system and 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 clean lab facility are described in NHX/SOP-300-006, "Standard Operating Procedures for the ACS Inorganic Class 100/10,000 Clean Lab Facility."

3.4 Contaminants in reagents, solvents, labware and other components of sample processing apparatus can also cause interferences which may lead to increased analyte and/or background signals. Atomic absorption/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 8.2.

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 NHX/SOP-300-007, "Standard Operating Procedure for Cleaning Labware in the ACS Inorganic Class 100/10,000 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

5.1.3 Apparatus

Hot plates

Variable speed horizontal shaker

Automatic pipettors, fixed and variable volume

Refrigerator

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

5.2 Graphite Furnace Atomic Absorption Spectrometer (GFAAS)

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 The graphite furnace must have a background correction system, preferably a Zeeman background correction system, in order to accurately measure the background absorption exactly at the same wavelength as the analytical absorbance. Temperature programming should be available for the graphite furnace and the temperatures should be able to be set independently for all three steps (Drying, Charring and Atomization). Pyrolytically coated graphite

tubes with pyrolytically coated L'vov platforms must be used for sample analysis to provide STPF conditions for the atomization of analyte(s).

5.2.3 An autosampler is required to deliver reproducible sample volumes into the furnace in a consistent manner (position of sample deposition on the platform).

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 absorbances, subtracting background, 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.

5.3 Hydride Generation Atomic Fluorescence Spectrometer (HGAF)

5.3.1 The spectrometer must have the capability of automation to reduce personnel errors to a minimum. Lamp current setting and PMT gain setting should be able to be controlled individually. This is necessary to optimize these parameters during method development and also to provide consistency of these parameters during the analysis from sample to sample.

5.3.2 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.

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

5.3.4 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)

Triton-X (reagent grade)

Sodium borohydride (ACS reagent grade)

Sodium hydroxide (ACS reagent grade)

Potassium iodide (ACS reagent grade)

Ascorbic acid (ACS reagent grade)

Hydrogen peroxide (ACS reagent)

Palladium Nitrate (ACS reagent grade)

Magnesium nitrate (ACS reagent grade)

Ammonium phosphate ($NH_4H_2PO_4$)

Deionized water, 18 mS quality

6.2 Stock Standard Solutions

Individual stock standard solutions of analytes are prepared from NIST traceable standards. A suitable diluent may be used in preparing the stock solutions in order to stabilize the analyte(s). In most cases 1000 mg/L atomic absorption standards (NIST

traceable) are used as stock standard solutions. The stock standard solutions are stored in appropriate containers under conditions required by the analyte(s).

6.3 Working Standard Solutions

Working standard solutions are prepared by diluting appropriate stock standard solutions using a diluent appropriate to stabilize the analyte(s). Working standard solutions are stored in appropriate 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(s) 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 aerosol samples, and are given in Table 2.

7.0 SAMPLE STORAGE

All samples are placed in appropriate airtight plastic containers and 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 Field Blanks

Field blanks are unspiked filters taken to the field along with the other samples except that they are not used for sample collection. These field blanks help in monitoring contamination, if any, from handling and transportation.

8.2 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 results from sample extraction and preparation steps.

8.3 Method Controls

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

8.4 Laboratory Controls

Laboratory controls are blank filters spiked with NIST certified calibration standards at levels described in Section 6.4 and treated the same as field samples.

Laboratory control samples will be processed and analyzed prior to processing any samples. These are used to demonstrate the performance of the method before analyzing field samples.

Laboratory controls are designed to represent field samples. These samples undergo all extraction and sample preparation steps that field samples undergo. The recovery of all the analytes will be monitored as well.

8.5 Performance Evaluation Samples

Once acceptable performance of the method is demonstrated for the analytes by laboratory controls, a performance evaluation sample is analyzed to further evaluate the overall performance of the method. The performance evaluation sample must contain all the target analytes with their levels certified by an approved material certification agency, in a

matrix almost identical to the sample matrix. An example of this type of material is NIST SRM 1648, a certified urban particulate.

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

8.6 Custody and Sample Tracking Procedures

8.6.1 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.

Split samples will be identified with the use of an additional character ("A" for arsenic analysis, and "M" for other metals).

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.

8.6.2 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

Samples are placed in separate 35 mL polypropylene test tubes and 10.0 mL of 50% HNO_3 is added to each test tube. Tubes are sealed with airtight plastic screw caps and are placed on a horizontal shaker for 30 minutes. The extract is decanted into a 50 mL Teflon beaker and the extraction is repeated twice with a fresh portion of acid each time (total extract volume = 30 mL). All three extracts are combined in a 50 mL beaker. A volume of 15.0 mL of the extract is pipetted into a clean 50 mL Teflon beaker and 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 12.5 mL of 50% HCl is added. The solution is warmed and filtered if necessary using a Whatman filter paper into a 25 mL volumetric flask and diluted with deionized water. The final solution is transferred to a clean plastic storage bottle and is stored in a refrigerator until analysis for arsenic by HGAF.

The remainder of the extract (15 mL) is evaporated down to about 5 mL on a hot plate and 0.250 mL of H_2O_2 is added. The solution is placed back on the hot plate and evaporated to about 0.5 mL. The beaker is removed from the hot plate, allowed to cool down to room temperature. The solution is filtered if necessary using a Whatman filter paper into a 5 mL volumetric flask and diluted with deionized water. The final solution is transferred to a clean plastic storage bottle and is stored in a refrigerator until analysis for Pb, Cd, and Cr by GFAAS.

10.0 CALIBRATION AND STANDARDIZATION

An acceptable performance of the instrument must be demonstrated and documented as described in Section 10.1.1 of this document and in Section 2.2 of NHX/SOP-171-005, "Standard Operating Procedure for the Calibration of Perkin Elmer (PE) Model 5100 ZL Atomic Absorption Spectrometer: Graphite Furnace", and in Section 2.3 of NHX/SOP-300-001, "Standard Operating Procedure 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 GFAAS Analysis

Instrument parameters are set up for the analyte of interest as given in Table 3 and a specified reference solution is analyzed. Operation of the instrument is described in detail in the "Standard Operating Procedure for the Calibration of Perkin Elmer (PE) Model 5100 ZL Atomic Absorption Spectrometer: Graphite Furnace" (NHX/SOP-171-005). The characteristic mass (m_o) of the analyte is calculated and compared to the manufacturer specified value. If the characteristic mass value is within ±20% of the manufacturer specified value, proceed with the initial calibration (starting from Section 10.1.3). If the characteristic mass for the analyte is more than ±20% of the specified, necessary steps (Section 10.2.5) will be taken to bring the m_o to an acceptable level.

10.1.2 HGAF Analysis

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". If the performance test meets the acceptance criteria, defined in the SOP, proceed with the initial calibration (starting from Section 10.1.3).

If the performance test does not meet the acceptance criteria necessary steps (Section 10.2.6) will be taken to bring the performance of the instrument to an acceptable level.

10.1.3 Calibration blank and calibration standards are analyzed by the GFAAS (for Pb, Cd & Cr) and by HGAF system (for As) 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.

10.1.4 Absorbance or fluorescence is measured for each calibration standard and a least square linear regression calibration curve is constructed as follows for each analyte by the instrument software.

$$y = a + bx$$

where:

y = absorbance or fluorescence

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 during the 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 10.1.3.

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 10.2.8) and recalibration is required.

10.2.5 Analyze a NIST certified standard after the calibration 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 both GFAAS and HGAF instruments. The characteristic mass (in pg per 0.0044 absorbance) and the sensitivity (in Pk.Ht. per ng mL^{-1}) will be used as performance parameters for GFAAS and HGAF respectively. These will be used as control charts to continuously monitor the performance of the instruments over time. The operator of the instrument is responsible for maintaining these charts. The charts must be presented to the laboratory manager or to the task leader for his/her review at least once a month. All control charts and data must be stored along with the sample analysis data.

10.2.7 Remedial Actions for GFAAS

Possible remedial actions are described in detail in the "Standard Operating Procedure for Maintenance of Perkin Elmer (PE) Model ZL5100 PC Atomic Absorption Spectrometer: Graphite Furnace" (NHX/SOP-171-006) and in the "Standard Operation Procedure for the Calibration of the Perkin Elmer (PE) Model 5100 ZL Atomic Absorption Spectrometer: Graphite Furnace" (NHX/SOP-171-005).

10.2.8 Remedial Actions for HGAF

Possible remedial actions 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

Instrumental conditions are different for each individual analyte. The instrumental parameters for each individual analyte are given in Table 3. Each sample is analyzed for Pb, Cd and Cr similar to standards as described in Section 10.1.3 by GFAAS, whereas for arsenic, samples are analyzed by HGAF similar to standards as described in Section 10.1.3.

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 15 samples, 2 method blanks, and 1 method control. Field blanks are also included in the batch but, as they are unknown to the analyst they will be treated as samples. 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 values or initial values 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 decrease in source intensity, due to the absorption of source energy by analyte atoms, is measured in GFAAS, whereas the fluorescence signal given out by the excited atoms is measured in HGAF during data acquisition. Once the data acquisition is completed, the concentration of analyte in the sample is calculated based on the standard calibration curve for that analyte.

11.4 Identification of Analytes

Analytes are identified by the presence of absorption/fluorescence, at wavelengths specific to the analytes. An analyte is present in the sample, if the concentration of the analyte is greater than the method detection limit (MDL) of that analyte (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 the method blank. Method detection limits are calculated for each 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.

Method detection limits may also be calculated based upon the mass of sample collected or the volume of air sampled.

12.2 Method Quantitation Limit (MQL)

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

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

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

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

Method quantitation limits may also be calculated based upon the mass of sample collected or the volume of air sampled.

13.0 DATA MANAGEMENT

13.1 Processing of Data Output

Instrument software is capable of processing the raw data (incident, transmittance and fluorescence intensities) and generating printed output. The output contains information on sample I.D., analyte tested, absorbance or 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. Examples of ASCII data files generated by GFAAS and HGAF instrument software are given in the following sections.

13.3.1 An example of ASCII data files generated by GFAAS instrument software is given in Table 4.

13.3.2 An example for a typical ASCII data file generated by the HGAF instrument software is given in Table 5.

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 10.1.4) using the measured absorbance or 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 absorbance or fluorescence for the sample.

a = y intercept of the reference calibration curve in absorbance or fluorescence.

b = Slope of the reference calibration curve in absorbance per ng mL^{-1} or fluorescence per ng mL^{-1}.

The corrected concentration of the analyte is calculated by subtracting the average concentration of the analyte in method blanks from the C_x.

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 µg of analyte per m^3 of air or µg of analyte per gram of particulate or any other formats.

TABLE 1. TARGET ANALYTES AND TECHNIQUES USED

Analyte	Technique
Pb	GFAAS
As	HGAF
Cd	GFAAS
Cr	GFAAS

GFAAS = Graphite furnace atomic absorption spectrometry

HGAF = Hydride generation atomic fluorescence spectrometry.

TABLE 2. SUGGESTED CALIBRATION SOLUTIONS

	Concentration of Analytes [ng/mL]						
Analyte	#1	#2	#3	#4	#5	#6	#7
Pb	0.0	1.0	4.0	8.0	10.0	15.0	20.0
As	0.0	0.05	0.10	0.30	0.50	0.70	1.0
Cd	0.0	0.2	0.5	1.0	1.5	2.0	3.0
Cr	0.0	1.0	2.0	5.0	8.0	10.0	15.0

TABLE 3. OPERATING PARAMETERS FOR INSTRUMENTS

A. Graphite furnace atomic absorption spectrometer (GFAAS)			
Instrument	Perkin-Elmer 5100 ZL		
Experimental parameters:	Pb	Cd	Cr
Source	HCL	HCL	HCL
Power-current (mA)[a]	5	4	12
Graphite tube & platform	Pyrolytically coated		
Sample size (µL)	20	20	20
Wavelength (nm)	217.0	228.8	357.9
Slit width (nm)	0.7	0.7	0.7
Furnace conditions:			
Step 1, EC [Ar flow, mL/min]	110 [250]	100 [250]	100 [250]
Step 2	130 [250]	130 [250]	130 [250]
Step 3	800 [250]	500 [250]	1000 [250]
Step 4	1500 [0]	1600 [0]	2200 [0]
Step 5	20 [250]	2400 [250]	2500 [2500]
Step 6	2400 [250]		
Matrix Modifiers	$Mg(NO_3)_2$ $NH_4H_2PO_4$	$Mg(NO_3)_2$ $NH_4H_2PO_4$	$Mg(NO_3)_2$
B. 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[b]	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] Manufacturer recommended, depends on the manufacturer.
[b] Depends on the lamp intensity.

TABLE 4. EXAMPLE OF AN ASCII DATA FILE GENERATED BY GFAAS SOFTWARE

"Sample_ID"	"EL"	"Sam Date"	"RSD"	"Mean_SA"	"Dilu"	"Abs.1"	"Abs.2"	"Abs.3"
"5% HNO3"	"Cd"	"11/21/94"	47.06			-0.0007657	-0.0002675	-0.0007156
"0.2 ppb Cd"	"Cd"	"11/21/94"	2.490			0.01514289	0.01587524	0.01530341
"0.5 ppb Cd"	"Cd"	"11/21/94"	0.820	0.18800000		0.01454766	0.01457441	0.01435705
"1.0 ppb Cd"	"Cd"	"11/21/94"	0.950	0.37900000		0.02892365	0.02932159	0.02945870
"2.0 ppb Cd"	"Cd"	"11/21/94"	1.020	1.48100000		0.04255393	0.04323946	0.04257400
"4.0 ppb Cd"	"Cd"	"11/21/94"	0.700	3.63000000		0.07603774	0.07666307	0.07700082
"10.0 ppb Cd"	"Cd"	"11/21/94"	0.340	10.3260000		0.18947070	0.18983520	0.18863804
"QC 2ppb CHECK"	"Cd"	"11/21/94"	0.950	2.14300000		0.04331972	0.04257065	0.04283149
"QC BLK CHECK"	"Cd"	"11/21/94"	6.340	-0.1680000		-0.0004001	-0.0000022	-0.0001961
"RB1111-1"	"Cd"	"11/21/94"	102.7	-0.0300000	0.0000	0.00288367	0.00175673	0.00250245
"AA9950"	"Cd"	"11/21/94"	10.14	-0.1740000	1.0000	0.00006799	-0.0005673	-0.0003934
"AA9951"	"Cd"	"11/21/94"	25.04	-0.0630000	1.0000	0.00164637	0.00152933	0.00209113
"AA7001"	"Cd"	"11/21/94"	33.04	-0.1040000	1.0000	0.00073345	0.00053281	0.00172997
"AA7002"	"Cd"	"11/21/94"	9.230	-0.0490000	1.0000	0.00199081	0.00211788	0.00195737
"AA7003"	"Cd"	"11/21/94"	7.570	0.30100000	1.0000	0.00807361	0.00888621	0.00869560
"AA7004"	"Cd"	"11/21/94"	1.620	2.00200000	1.0000	0.04069131	0.03958109	0.04055086
"AA7005"	"Cd"	"11/21/94"	24.00	-0.1190000	1.0000	0.00055622	0.00027532	0.00130863
"AA7006"	"Cd"	"11/21/94"	10.50	0.35200000	1.0000	0.00870228	0.00990279	0.00988607
"QC 2ppb CHECK"	"Cd"	"11/21/94"	1.610	2.05000000		0.04172461	0.04051407	0.04131330
"QC BLK CHECK"	"Cd"	"11/21/94"	8.010	-0.2250000		-0.0012696	-0.0009184	-0.0015906

TABLE 5. 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:	Air Particulate
TO BE ANALYZED FOR:	Pb, As, Cd, Cr

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.