

# APEX ENVIRONMENTAL CONSULTANTS, INC.

Quality • Integrity • Professionalism

PHONE: (913) 338-APEX (913) 338-2739

FAX: (913) 338-2741 E-MAIL: apex@4apex.com

May 15, 2006

Mr. Aaron Zack Sunlight Saunas, Inc. 14119 Marshall Drive Lenexa, Kansas 66215

Tel: (913) 754-0831 Fax: (775) 514-4314

RE: Air Sampling Armana Sauna

Sunlight Saunas, Inc. 14119 Marshall Drive Lenexa, Kansas 66215

APEX Project No. 60125I

Dear Mr. Zack:

In response to your request and authorization, APEX Environmental Consultants, Inc. conducted air sampling in an Armana Sauna on May 2<sup>nd</sup> and 3<sup>rd</sup>, 2006. The purpose of the sampling effort was to determine if heater contaminates were being liberated inside the sauna during operation. It is our understanding that this work was initiated due to one of your competitors making claims that your saunas are unsafe due to the liberation of various contaminates from the heater system.

Per your request, air sampling for carbon, silicon, and sulfur dioxide was performed inside an operating Armana sauna. The samples were collected over a 420 to 480 minute sampling period with the sauna adjusted to 150 degrees Fahrenheit. Background air samples were also collected to determine normal background levels for these contaminates outside the sauna. The Armana sauna is equipped with a far infrared Solocarbon heater.

Based on laboratory results, no silicon was detected in the sauna air sample at an instrument detection limit of 1.0 microgram/sample. At this detection limit, we can say that the air concentration in the operating sauna was at least less than 0.0005 mg/m<sup>3</sup>. This airborne concentration is less than 30,000 times what the Occupational Safety and Health Administration (OSHA) considers a safe exposure limit for silicon. The OSHA permissible exposure limit (PEL) for silicon is 15 mg/m<sup>3</sup> (average total concentration).



No sulfur dioxide was detected in the sauna air sample at an instrument detection limit of 1.3 micrograms/sample. At this detection limit, we can say that the air concentration in the operating sauna was at least less than 0.69 parts per billion. This airborne concentration is less than 7000 times what the Occupational Safety and Health Administration (OSHA) considers a safe exposure limit for sulfur dioxide. The OSHA permissible exposure limit (PEL) for sulfur dioxide is 5000 parts per billion.

Carbon was detected in the operating sauna at 0.06 milligrams/sample. Based on the volume of air sampled, we can say the air concentration in the operating sauna was 0.07 mg/m³. However, the background air sample collected outside the sauna showed an even higher airborne concentration of carbon at 0.08 mg/m³. This is likely the result of carbon being a common outdoor pollutant, the largest source being internal combustion engines (automobile). Based on these sample results, the Solocarbon heater does not appear to be contributing to any type of carbon contamination inside the sauna. The OSHA permissible exposure limit (PEL) for carbon black is 3.5 mg/m³ (average total concentration). The measured concentration is less than 50 times what the Occupational Safety and Health Administration (OSHA) considers a safe exposure limit for carbon.

As you can see from the sampling data, there should be absolutely no concerns regarding exposure to the subject contaminates during the operation of your saunas. If you have questions about the information contained in this report or require additional services, we would be happy to assist you in any way we can.

Respectfully submitted,

APEX Environmental Consultants, Inc.

David Nold, M.S., CIH, REHS

Industrial Hygienist

# APPENDIX A LABORATORY ANALYSIS REPORTS



# APEX Environmental Consultants, Inc. OC - SWI - 205

14955 W. 101st Terrace • Lenexa, Kansas 66215 • (913) 338-2739 • FAX (913) 338-2741

# Sample Chain of Custody

Sample Date: May 3, 2006

Page: 1 of 1

Project Name: Sunlight Saunas, Lenexa, KS

Contact: David Nold

Project #: 601251

	Notes Notes	12488	(7.4.K)	0000	1370	17497	1.00							
)	Total Volume (		840 Liters	1920 Liters	1920 Liters	720 Liters	720 Liters							
	Flow Rate	2 L/min x·420 min	2 L/min x 420 min	4 L/min x 480 min	4 L/min x 480 min	1.5 L/min x 480 min	1.5 L/min x 480 min							
	Sample Type	Carbon Black / NIOSH 5000	Carbon Black / NIOSH 5000	Silicon / NIOSH 7300	Silicon / NIOSH 7300	Sulfur Dioxide / OSHA ID 200	Sulfur Dioxide / OSHA ID 200							
	Location Description	Inside Armana Sauna at 150° F		Inside Armana Sauna at 150° F	Background sample	Inside Armana Sauna at 150° F	Background sample							
		CB-01		Si-01			S0 <sub>2</sub> -02							

structions:	
nal II	
litio	
ğ	

- Standard Turnaround Please
   E-mail results to: dnold@4apex.0

Date 5-4-06 Received by JAMM? Relinquished By:

TS(0) 00/5/5:18

Date:

Date:

Please Fax results to Attn. Dave at (913) 338-2741
 Send involute to: Accts. Payable, APEX Environmental Consultants

Relinquished By:

İ
ž.
d b
ive
rece.
1
_
Dat
7
ı

Received by:



Dave Nold Submitted To:

> Apex Environmental 14955 W. 101st Terrace

Lenexa, KS 66215

Reference Data:

Carbon Black

Client Sample No.:

CB-01 through CB-02

P.O. No.:

Not Available

Sample Location:

Sunlight Saunas, Lenexa, KS,

Project Number:

60125I

Sample Type:

Filter

Method Reference: DCL Set ID No.:

NIOSH 5000

06-W-2057

DCL Sample ID No.:

06-12488 through 06-12489

Sample Receipt Date:

5/5/2006

Analysis Date:

5/8/2006

The samples were analyzed in accordance with NIOSH method 5000.

Sample condition was acceptable upon receipt except where noted.

The results are provided in the enclosed data table. Results relate only to the items tested and are not blank corrected unless indicated in the data table.

This report shall not be reproduced except in full, without the written approval of the laboratory.

Rob Nieman

Analyst

CINCINNATI OFFICE 4388 GLENDALE-MILFORD ROAD CINCINNATI, OHIO 45242-3706 513 733-5336, FAX 513 733-5347 WEST COAST OFFICE 11 SANTA YORMA COURT NOVATO, CALIFORNIA 94945 800 280-8071, FAX 415 893-9469

# Results Carbon Black

Client #	DCL #	Sample Volume (L)	mg/sample	mg/m <sup>3</sup>
CB-01	06-12488	840	0.06	0.07
CB-02	06-12489	840	0.07	0.08
EQL			0.05	

ND indicates not detected at or above the estimated quantitation limit (EQL).

Rob Nieman Analyst

Reviewer



Submitted To: Dave Nold

> Apex Environmental 14955 W. 101<sup>st</sup> Terrace Lenexa, KS 66215

Reference Data:

Silicon

Client Sample No.:

Si-01 through Si-02

P.O. No.:

Not Available

Sample Location:

Sunlight Saunas, Lenexa, KS

Project Number:

601251

Sample Type:

Filter NIOSH 7300

Method Reference:

DCL Set ID No.:

06-S-2057

DCL Sample ID No.:

06-12490 through 06-12491

Sample Receipt Date: Preparation Date:

5/5/2006 05/10/06

Analysis Date:

05/10/06

The samples were prepared and analyzed in accordance with NIOSH method 7300 using a Perkin Elmer 3000XL ICP.

The sample condition upon receipt was acceptable except where noted.

The results are in the enclosed data table. Results relate only to the items tested and are not blank corrected unless indicated in the data table.

This report shall not be reproduced except in full, without the written approval of the laboratory.

Chris Bauques

Analyst

# Results Silicon

Client #	DCL #	DCL # Sample Volume (L)		mg/m³	
Si-01	06-12490	1920	ND	<0.0005	
Si-02	06-12491	1920	ND	<0.0005	
	Prep Blank		ND		
% Recovery	LCS 1		102.		
% Recovery	LCS 2		96.		
RPL			1.		

ND = not detected at or above the reporting limit (RPL). LCS = laboratory control sample.

Chris Baugues Analyst

Reviewe



Submitted To: Dave Nold

> Apex Environmental 8600 W. 110th St. Overland Park, KS

66210

Reference Data:

Sulfur Dioxide

Sample Location:

Sunlight Saunas, Lenexa, KS, Project Number:

601251

Sample Type:

IABC tube

Client Sample No.:

S02-01 through S02-02

Not Available

Method Reference:

OSHA 200

Sample Set ID#:

06-L-2057

DATACHEM Lab No.:

06-12492 through 06-12493

Date Received:

05/05/06

Preparation Date:

05/09/06

Analysis Date:

05/10/06

Sample condition was acceptable upon receipt except where noted.

Analysis was completed on a Dionex IC2000 Ion Chromatograph equipped with a Dionex AS18-AG18 column and a conductance detector. The eluent used was 35mM Potassium Hydroxide at a flow rate of 1.0 mL per minute.

Compound identification is based upon retention time matching only. Any compound with a similar retention time will interfere.

The results are provided in the enclosed data table. Results relate only to the items tested and are not blank corrected.

This report shall not be reproduced except in full, without the written approval of the laboratory.

Jeff Ogle Analyst

Test Report Page 2 of 2

Sample Set ID: 06-L-2057

# Data Table Sulfur Dioxide

Client ID	ient ID Lab ID		µg/sample	dqq
SO2-01	06-12492	720.	ND	<.69
SO2-02	06-12493	720.	ND	<.69
	EQL		1.3	

ND indicates analyte was not detected at or above the estimate quantitation limit (EQL).

Jeff Ogle

Analyst

viewer

# APPENDIX B PHOTO LOG

**PHOTO DATE:** May 2, 2006 **APEX Proj. No.:** 60125I

PROJECT NAME: Air Sampling
PROJECT LOCATION: Sunlight Saunas, Lenexa, Kansas



Photo No. 1

View of showroom at Sunlight Saunas, Lenexa, Kansas.

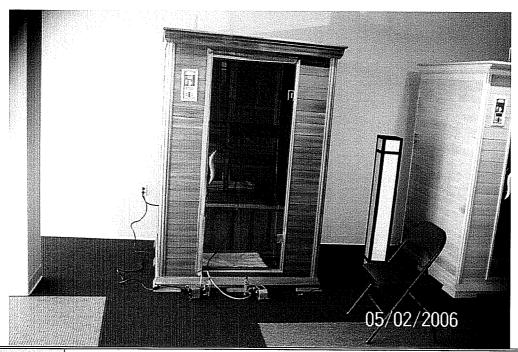


Photo No. 2

View of Armana sauna. Note doors were sealed with masking tape during testing.

# APPENDIX C NIOSH ANALYTICAL METHODS

С

MW: 12.01

CAS: 1333-86-4

RTECS: FF5800000

METHOD: 5000, Issue 2

**EVALUATION: FULL** 

Issue 1: 15 May 1989 Issue 2: 15 August 1994

**OSHA:** 3.5 mg/m<sup>3</sup>

5.5 mg/m

PROPERTIES: solid; may contain polynuclear aromatic

hydrocarbons (PAH)

NIOSH: 3.5 mg/m³ (in presence of PAHs: carcinogen/PAHs to 0.1 mg/m³, cyclohexane extractable fraction)

ACGIH: 3.5 mg/m<sup>3</sup>

SYNONYMS: acetylene black; amorphous carbon; furnace black; lamp black

	SAMPLING			MEASUREMENT
SAMPLER:	FILTER (tared 5-µm PVC me	mbrane)	TECHNIQUE:	GRAVIMETRIC (FILTER WEIGHT)
FLOW RATE:	1 to 2 L/min	,	ANALYTE:	airborne particulate material
VOL-MIN: -MAX:	30 L @ 3.5 mg/m <sup>3</sup> 570 L		BALANCE:	0.001 mg sensitivity; use same balance before and after sample collection
SHIPMENT:	routine		CALIBRATION:	National Institute of Standards and Technology, Class S-1.1 weights or ASTM Class 1 weights
BLANKS:	indefinitely  LANKS: 2 to 10 field blanks per set		RANGE:	0.1 to 2 mg per sample
	p	J. 301	ESTIMATED LOD:	0.03 mg per sample
			PRECISION (Š <sub>r</sub> ):	0.025 @ 3.5 mg/m <sup>3</sup> [1, 2]
	ACCURACY			
RANGE STUDIE		mg/m³ _ samples)		
BIAS:	0.01%	•		
OVERALL PREC	CISION (Ŝ <sub>rt</sub> ): 0.056 [1]			
ACCURACY:	± 11.0	9%		
****				

**APPLICABILITY:** The working range is 0.5 to 10 mg/m<sup>3</sup> for a 200-L air sample. This method is not applicable for the determination of "cyclohexane-solubles" [3]. This analysis is simple but the method is nonspecific. Information on any o ther particulate materials present should be assessed. The method may be extended to higher air concentrations (e.g., nuisanc e dust levels) by collecting a smaller sample volume [4].

**INTERFERENCES:** The presence of any other particulate material in the air being sampled will be a positive interference since this is a gravimetric method.

**OTHER METHODS:** This is Method S262 [5] in a revised format. It is similar, except for collecting device, to the method described in the carbon black criteria document [3].

# **EQUIPMENT:**

- 1. Sampler: 37-mm, 5-µm pore size PVC filter and stainless steel support screen in 37-mm, cassette filter holder (preferably, conductive).
- 2. Personal sampling pump, 1 to 2 L/min, with flexible connecting tubing.
- 3. Microbalance capable of weighing to 0.001 mg.
- 4. Static neutralizer; e.g. Po-210; replace nine months after production date.
- 5. Forceps (preferably nylon).
- 6. Environmental chamber or room for balance (e.g.  $20 \pm 1$  °C and  $50 \pm 5\%$  RH).

**SPECIAL PRECAUTIONS:** Carbon black containing polynuclear aromatic hydrocarbons (cyclohexane extractable materials) in excess of 0.1% (w/w) should be treated as a suspect carcinogen [3].

# PREPARATION OF FILTERS BEFORE SAMPLING:

- 1. Equilibrate the filters in an environmentally controlled weighing area or chamber for at least 2 h. NOTE: An environmentally controlled chamber is desirable, but not required.
- 2. Number the backup pads with a ballpoint pen and place them, numbered side down, in filter cassette bottom sections.
- 3. Weigh the filters in an environmentally controlled area or chamber. Record the filter tare weights,  $W_1$ , (mg).
  - a. Zero the balance before each weighing.
  - b. Handle the filter with forceps. Pass the filter over an antistatic radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
- 4. Assemble the filters in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette. Place a cellulose shrink band around the filter cassette, allow to dry, and mark with the same number as the backup pad.

# SAMPLING:

- Calibrate each personal sampling pump with a representative sampler in line.
- 6. Sample at 1 to 2 L/min for a total sample volume of 30 to 570 L. Do not exceed a filter loading of approximately 2 mg total dust. Take two to four replicate samples for each batch of field samples for quality assurance on the sampling procedures.

# **SAMPLE PREPARATION:**

- 7. Wipe dust from the external surface of the filter cassette with a moist paper towel to minimize contamination. Discard the paper towel.
- 8. Remove the top and bottom plugs from the filter cassette. Equilibrate for at least 2 h in the balance room.
- Remove the cassette band, pry open the cassette, and remove the filter gently to avoid loss of dust.
  - NOTE: If the filter adheres to underside of cassette top, gently lift using the dull side of scalpel blade. Take care not to tear the filter.

# **CALIBRATION AND QUALITY CONTROL:**

- Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Calibrate the balance with National Institute of Standards and Technology Class S-1.1 or ASTM Class 1 weights.
- 11. The set of replicate samples should be exposed to the same dust environment, either in a laboratory dust chamber [6] or in the field [7]. The quality control samples must be taken with the same equipment, procedures and personnel used in the routine field samples. Calculate precision from these replicates and record  $\bar{S}_r$  on control charts. Take corrective action when the precision is out of control [6].

# **MEASUREMENT:**

12. Weigh each filter, including field blanks. Record the post-sampling weight, W <sub>2</sub> (mg). Record anything remarkable about a filter (e.g., overload, leakage, wet, torn, etc.).

# **CALCULATIONS:**

13. Calculate the concentration, C (mg/m <sup>3</sup>), of carbon black in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) - (B_2 - B_1)}{V} \cdot 10^3, \text{ mg/m}^3.$$

where:  $W_1$  = tare weight of filter before sampling (mg)

W<sub>2</sub> = post-sampling weight of sample-containing filter (mg)

 $B_1$  = tare weight of blank filter (mg)

B<sub>2</sub> = post-sampling weight of blank filter (mg)

# **EVALUATION OF METHOD:**

Method S262 [5] was issued on January 30, 1976, and validated over the range 1.9 to 7.7 mg/m  $^3$  for a 200-L sample and over the range 7.8 to 28 mg/m  $^3$  for a 100-L sample using Vulcan XC72 (0.03- $\mu$ m particle size; Cabot Corp.) in a Wright Dust Feeder [1]. Overall precision,  $\hat{S}_{rT}$ , was 0.056. Collection efficiency was between 99 and 100%.

# **REFERENCES:**

- [1] Documentation of the NIOSH Validation Tests, S262 and S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
- [2] Unpublished data from Non-textile Cotton Study, NIOSH/DRDS/EIB.
- [3] NIOSH Criteria for a Recommended Standard ... Occupational Exposure to Carbon Black, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-204, 80-88 (1978).
- [4] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

- [5] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S262, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [6] Bowman, J.D., D.L. Bartley, G.M. Breuer, L.J. Doemeny, D.J. Murdock. Accuracy Criteria Recommendation for the Certification of Gravimetric Coal Mine Dust Personal Samplers. U.S. Department of Health and Human Services, NTIS Pub. No. 85-222446 (1984).
- [7] Breslin, J.A., S.J. Page, R.A. Jankowski. Precision of Personal Sampling of Respirable Dust in Coal Mines, U.S. Bureau of Mines Reports of Investigations #8740 (1983).

# **METHOD REVISED BY:**

Frank Hearl, P.E., NIOSH/DRDS; S262 and S349 originally validated under NIOSH Contract CDC-99-74-45.

SO,

MW: 64.06

CAS: 7446-09-5

RTECS: WS4550000

METHOD: 6004, Issue 2

**EVALUATION: PARTIAL** 

Issue 1: 15 May 1989 Issue 2: 15 August 1994

OSHA: NIOSH: ACGIH:

2 ppm; STEL 5 ppm; Group I Pesticide

2 ppm; STEL 5 ppm (1 ppm = 2.62 mg/m<sup>3</sup> @ NTP) PROPERTIES:

gas; vapor density 2.26 (air = 1);

BP -10 °C; MP -72.7 °C;

nonflammable

SYNONYMS: none

SAMPLING

FILTER + TREATED FILTER

(cellulose + Na<sub>2</sub>CO<sub>3</sub>; preceded by

0.8-µm cellulose ester membrane)

FLOW RATE:

SAMPLER:

0.5 to 1.5 L/min

VOL-MIN: -MAX:

4 L @ 5 ppm

200 L

SHIPMENT: routine

SAMPLE

STABILITY:

not determined

FIELD BLANKS: 2 to 10 field blanks per set

MEASUREMENT

**TECHNIQUE:** 

ION CHROMATOGRAPHY

ANALYTE:

sulfite and sulfate ions

**EXTRACTION:** 

10 mL 1.75 m $\underline{\mathrm{M}}$  NaHCO $_{\mathrm{3}}$ /2.0 m $\underline{\mathrm{M}}$  Na $_{\mathrm{2}}$ CO $_{\mathrm{3}}$ 

INJECTION LOOP VOLUME: 50 µL

ELUENT:

1.75 mM NaHCO<sub>3</sub>/2.0 mM Na<sub>2</sub>CO<sub>3</sub>,

2 to 3 mL/min

COLUMNS:

ion Pac AS4A separator, ion Pac AG4A

guard; micromembrane suppressor [2]

CONDUCTIVITY SETTING: 10 µS full scale

**CALIBRATION:** 

standard solutions of SO  $_3^{2-}$  and SO $_4^{2-}$ 

in eluent

RANGE:

11 to 200 μg SO<sub>2</sub> per sample

ESTIMATED LOD:

3 µg SO₂ per sample [2]

PRECISION (S,):

0.042 [2]

RANGE STUDIED:

not studied

ACCURACY

BIAS:

not determined

OVERALL PRECISION ( $\hat{S}_{rT}$ ): not determined

ACCURACY:

not determined

APPLICABILITY: The working range is 0.2 to 8 ppm (0.5 to 20 mg/m <sup>3</sup>) for a 100-L air sample. The method is applicable to STEL samples.  $SO_2$  is collected on the back (treated) filter. Sulfuric acid, sulfate salts, and sulfite salts are collected on the front filter and may be quantitated as total particulate sulfate.

INTERFERENCES: Sulfur trioxide gas, if present in dry atmospheres, may give a positive interference for SO  $_2$ .

OTHER METHODS: This revises P&CAM 268 [3]. P&CAM 146 [4], P&CAM 163 [5], and S308 [6] use 0.3 NH<sub>2</sub>O<sub>2</sub> for sampling, followed by titration with NaOH or barium perchlorate. P&CAM 160 [7] uses tetrachloromercurate solution and visible spectrophotometry. P&CAM 204 [8] uses a solid sorbent (molecular sieve 5A), thermal desorption, and mass spectrometry.

#### **REAGENTS:**

- Water, deionized, filtered, specific conductance ≤10 µS/cm.
- Fixative solution. Dissolve 25 g Na <sub>2</sub>CO<sub>3</sub> in deionized water. Add 20 mL glycerol and dilute with deionized water to 1 L.
- Eluent: 1.75 m M NaHCO<sub>3</sub>/2.0 m Na<sub>2</sub>CO<sub>3</sub>.
   Dissolve 0.588 g NaHCO<sub>3</sub> and 0.848 g Na<sub>2</sub>CO<sub>3</sub> in 4 L filtered deionized water.
- Calibration stock solutions,
   1 mg/mL (as the anion). Prepare in duplicate.
  - a. Sulfite: dissolve 0.1575 g
     Na<sub>2</sub>SO<sub>3</sub> in water. Add 2 mL glycerol.
     Dilute to 100 mL. Prepare fresh daily.
  - b. Sulfate: dissolve 0.1479 g
     Na<sub>2</sub>SO<sub>4</sub> in deionized water. Dilute to 100 mL. Stable several weeks.
  - \* See SPECIAL PRECAUTIONS.

#### **EQUIPMENT:**

- Sampler: two 37-mm diameter cassette filter holders (connected in series by a M-M Luer adapter, e.g., Millipore XX1102503, or a short piece of plastic tubing) containing:
  - a. (Front cassette) cellulose ester membrane filter, 0.8-μm pore size, supported by a backup pad.
  - b. (Back cassette) cellulose filter (Whatman 40 or equivalent) which has been saturated with fixative solution and dried 20 to 30 min at 100 °C, supported by a porous plastic support pad.
- 2. Personal sampling pump, 0.5 to 1.5 L/min, with flexible connecting tubing.
- Vials, glass, 20-mL, screw-cap, such as scintillation vials.\*\*
- Ion chromatograph, HPIC-AS4A anion separator and HPIC-AG4A guard, anion micromembrane suppressor, conductivity detector, and strip chart recorder. (Optional: integrator.)
- 5. Syringes, 10-mL, polyethylene, with luer tip.\*\*
- 6. Filters, in-line, luer-tip holder with membrane filter, 13- or 25-mm, 0.45-μm pore size.
- Micropipets, 50- to 1000-μL, with disposable tips.\*\*
- 8. Volumetric flasks, 50- and 100-mL.\*\*
- 9. Pipet, 10-mL.\*\*
- 10. Polyethylene bottles, 250-mL.\*\*
  - \*\* Clean by rinsing thoroughly with deionized water.

SPECIAL PRECAUTIONS: None.

# SAMPLING:

- 1. Calibrate each personal sampling pump with a representative sampler in line.
- 2. Remove end caps of sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
- 3. Sample at an accurately known flow rate between 0.5 and 1.5 L/min for a total sample size of 40 to 200 L. Do not exceed a total particulate loading of 2 mg on the front filter.
- 4. Seal the sampler and pack securely for shipment.

NOTE: If determination of sulfuric acid is required, transfer the front (membrane) filter to a clean vial within 4 h to avoid low recovery of sulfate. Handle the filter with tweezers to avoid contamination.

# **SAMPLE PREPARATION:**

5. Put the two filters from the sampler into separate, clean vials. Discard the backup pads. Add 10.0 mL eluent to each vial and let stand, with occasional vigorous shaking, for 30 min.

NOTE: The SO<sub>2</sub> collected on the treated (back) filter is present as sulfite, which oxidizes in air slowly (over several weeks) to sulfate. The contributions of sulfite and sulfate found on the back filter must be summed, with appropriate stoichiometric factors applied, to give the SO

6. Pour each sample into a syringe fitted with an in-line filter.

# CALIBRATION AND QUALITY CONTROL:

- 7. Calibrate daily with at least six working standards.
  - a. Add known aliquots of sulfate calibration stock solution to eluent in 50-mL volumetric flasks and dilute to the mark to produce solutions containing 1 to 20  $\mu$ g/mL SO  $^{2}_{4}$ .
  - b. Prepare sulfite standards in the same manner over the same range.
  - c. Store working standards in tightly-capped polyethylene bottles. Prepare fresh working
  - d. Analyze working standards with samples and blanks (steps 8 through 10). Prepare a calibration graph for each anion [peak height (mm or µS) vs. µg sulfite or sulfate].

# **MEASUREMENT:**

- 8. Set ion chromatograph to conditions given on page 6004-1, according to manufacturer's instructions.
- 9. Inject sample aliquot. For manual operation, inject 2 mL of sample from syringe to ensure complete
  - NOTE: All samples, eluents, and water flowing through the ion chromatograph must be filtered to avoid plugging system valves or columns.
- 10. Measure peak heights of sulfite and sulfate peaks.
  - NOTE: If peak height exceeds linear calibration range, dilute with eluent, reanalyze, and apply the appropriate dilution factor in calculations.

# **CALCULATIONS:**

- 11. Determine the mass,  $\mu g$ , of sulfate equivalent found on the front (W  $_{f}$ ) and back (W  $_{b}$ ) filters and in the corresponding average media blanks (B  $_{\rm f}$  and B  $_{\rm b}$ ).
  - NOTE: The sulfate equivalent is the sum of the sulfate peak, µg, and 1.200 times the sulfite peak,  $\mu$ g, on the chromatogram (1.200 = MW SO  $_4^2$ /MW SO $_3^2$ ):  $\mu$ g<sub>sulfate equivalent</sub> =  $\mu$ g<sub>sulfate</sub> +
- 12. Calculate the concentration, C <sub>SO2</sub>, of sulfur dioxide, applying the factor 0.667 (MW SO <sub>2</sub>/MW SO<sup>2</sup><sub>4</sub>):

$$C_{SO_2} = \frac{(W_b - B_b)}{V} \cdot 0.667, mg/m^3.$$

13. Calculate the concentration, C  $_{\rm SO4}$ , of particulate sulfate (including sulfuric acid) in the air volume

$$C_{SO_4} = \frac{(W_f - B_f)}{V}, mg/m^3.$$

# **EVALUATION OF METHOD:**

The sampler was adapted from that of Pate, et al. [9]. In experiments in which SO  $_2$  was generated by permeation tube and collected in impingers containing H  $_2$ O $_2$ , untreated 0.8-µm cellulose ester membrane filters were shown to allow complete passage of SO  $_2$  [10]. In subsequent sampling of an atmosphere containing ca. 10 ppm SO  $_2$  at 1 L/min for 30 min, two treated filters were placed in series following a cellulose ester membrane filter. Recoveries were: 0.667 mg SO  $_2$  from the first treated filter, 0.02 mg SO $_2$  from the second treated filter, and less than 0.003 mg SO  $_2$  in the backup impinger containing 0.3 Network H $_2$ O extraction, 98.5% using hot H $_2$ O extraction, and 82.5% using 0.01 Mel HCl for extraction.

A study on filter impregnating solutions compared NaHCO  $_3$  and KOH. The chromatograms of samples from the KOH-treated filters had noticeably flattened and broadened peak shapes as well as retention times reduced by approximately 10% when compared to the chromatograms of H  $_2$ SO $_4$  spiked on filters impregnated with NaHCO  $_3$ [1].

# **REFERENCES:**

- [1] Williamson, G.Y. NIOSH/MRSB Sequence 7452-B (unpublished, July 6, 1992).
- [2] Williamson, G.Y. NIOSH/MRSB Sequence 7452-C (unpublished, July 22, 1992).
- [3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 5, P&CAM 268, U.S. Department of Health and Human Services, Publ. (NIOSH) 79-141 (1979).
- [4] Ibid., 2nd ed., Vol. 1, P&CAM 146, Publ. (NIOSH) 77-157-A (1977).
- [5] Ibid., 2nd ed., Vol. 1, P&CAM 163, Publ. (NIOSH) 77-157-A (1977).
- [6] Ibid., 2nd ed., Vol. 4, S308, Publ. (NIOSH) 78-175 (1978).
- [7] Ibid., 2nd ed., Vol. 1, P&CAM 160, Publ. (NIOSH) 77-157-A (1977).
- [8] Ibid., 2nd ed., Vol. 1, P&CAM 204, Publ. (NIOSH) 77-157-A (1977).
- [9] Pate, J.B., J.P. Lodge, Jr., and M.P. Neary. The Use of Impregnated Filters to Collect Traces of Gases in the Atmosphere, <u>Anal. Chim. Acta, 28</u>:341 (1963).
- [10] Grote, A.A. (NIOSH, unpublished results, 1973).
- [11] Eller, P.M. and M.A. Kraus. Methods for the Determination of Oxidized Sulfur and Nitrogen Species in Air, Internal Report, (unpublished, July 29, 1977).

# **METHOD REVISED BY:**

Peter M. Eller, Ph.D., and Mary Ellen Cassinelli, NIOSH/DPSE.

# 7300

# **ELEMENTS by ICP** (Nitric/Perchloric Acid Ashing)

MW: Table 1

CAS: Table 2

RTECS: Table 2

METHOD: 7300, Issue 3 **EVALUATION: PARTIAL** Issue 1: 15 August 1990

lanthanum

magnesium

lithium\*

Issue 3: 15 March 2003

OSHA: Table 2 PROPERTIES: Table 1

NIOSH: Table 2 ACGIH: Table 2

**ELEMENTS:** aluminum\*

calcium antimony\* chromium\* arsenic cobalt\* barium copper beryllium\*

iron lead\*

**ACCURACY** 

manganese\* molybdenum\*

silver

phosphorus selenium

potassium

nickel

tellurium tin thallium titanium

strontium

vanadium\* yittrium zinc zirconium\*

tungsten\*

cadmium \*Some compounds of these elements require special sample treatment.

> **SAMPLING MEASUREMENT**

SAMPLER: FILTER **TECHNIQUE:** INDUCTIVELY COUPLED ARGON

(0.8-µm, cellulose ester membrane, or PLASMA, ATOMIC EMISSION 5.0-µm, polyvinyl chloride membrane) SPECTROSCOPY (ICP-AES)

**BACKGROUND** 

FLOWRATE: 1 to 4 L/min ANALYTE: elements above

VOL-MIN: Table 1 **ASHING** -MAX: Table 1 REAGENTS:

conc. HNO<sub>3</sub>/ conc. HClO<sub>4</sub> (4:1), 5 mL; 2mL increments added as needed SHIPM ENT: routine

**CONDITIONS:** room temperature, 30 min; 150 °C to near

SAMPLE dryness STABILITY: stable

SOLUTION: 4% HNO<sub>3</sub>, 1% HCIO<sub>4</sub>, 25 mL **BLANKS:** 2 to 10 field blanks per set

**WAVELENGTH:** depends upon element; Table 3

**CORRECTION:** spectral wavelength shift

**CALIBRATION:** elements in 4% HNO<sub>3</sub>, 1% HClO<sub>4</sub> **RANGE STUDIED:** not determined

RANGE: varies with element [1] BIAS: not determined

ESTIMATED LOD: Tables 3 and 4 OVERALL PRECISION (\$,,): not determined

PRECISION (S): Tables 3 and 4 ACCURACY: not determined

APPLICABILITY: The working range of this method is 0.005 to 2.0 mg/m3 for each element in a 500-L air sample. This is simultaneous elemental analysis, not compound specific. Verify that the types of compounds in the samples are soluble with the ashing procedure selected.

INTERFERENCES: Spectral interferences are the primary interferences encountered in ICP-AES analysis. These are minimized by judicious wavelength selection, interelement correction factors and background correction [1-4].

OTHER METHODS: This issue updates issues 1 and 2 of Method 7300, which replaced P&CAM 351 [3] for trace elements. Flame atomic absorption spectroscopy (e.g., Methods 70XX) is an alternate analytical technique for many of these elements. Graphite fumace AAS (e.g., 7102 for Be, 7105 for Pb) is more sensitive.

# **REAGENTS:**

- 1. Nitric acid (HNO<sub>3</sub>), conc., ultra pure.
- 2. Perchloric acid (HCIO<sub>4</sub>), conc., ultra pure.\*
- 3. Ashing acid: 4:1 (v/v) HNO<sub>3</sub>:HClO<sub>4</sub>. Mix 4 volumes conc. HNO<sub>3</sub> with 1 volume conc.
- 4. Calibration stock solutions, 1000  $\mu g/mL$ . Commercially available, or prepared per instrument manufacturer's recommendation (see step 12).
- 5. Dilution acid, 4%  $\mathrm{HNO_{3}}$ , 1%  $\mathrm{HClO_{4}}$ . Add 50 mL ashing acid to 600 mL water; dilute to 1 L.
- 6. Argon.
- 7. Distilled, deionized water.
  - See SPECIAL PRECAUTIONS.

#### EQUIPMENT:

- 1. Sampler: cellulose ester membrane filter, 0.8-µm pore size; or polyvinyl chloride membrane, 5.0-μm pore size; 37-mm diameter, in cassette filter holder.
- 2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
- 3. Inductively coupled plasma-atomic emission spectrometer, equipped as specified by the manufacturer for analysis of elements of interest.
- 4. Regulator, two-stage, for argon.
- 5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with watchglass covers.\*\*
- 6. Volumetric flasks, 10-, 25-,100-mL., and 1-L\*\*
- 7. Assorted volumetric pipets as needed.\*\*
- 8. Hotplate, surface temperature 150 °C.
  - \*\* Clean all glassware with conc. nitric acid and rinse thoroughly in distilled water before use.

SPECIAL PRECAUTIONS: All perchloric acid digestions are required to be done in a perchloric acid hood. When working with concentrated acids, wear protective clothing and gloves.

# SAMPLING:

- 1. Calibrate each personal sampling pump with a representative sampler in line.
- 2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample size of 200 to 2000 L (see Table 1) for TWA measurements. Do not exceed a filter loading of approximately 2 mg total dust.

# SAMPLE PREPARATION:

- 3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
- 4. Add 5 mL ashing acid. Cover with a watchglass. Let stand 30 min at room temperature. NOTE: Start a reagent blank at this step.
- 5. Heat on hotplate (120 °C) until ca. 0.5 mL remains.
  - NOTE 1: Recovery of lead from some paint matrices may require other digestion techniques. See Method 7082 (Lead by Flame AAS) for an alternative hotplate digestion procedure or Method 7302 for a microwave digestion procedure.
  - NOTE 2: Some species of Al, Be, Co, Cr, Li, Mn, Mo, V, and Zr will not be completely solubilized by this procedure. Alternative solubilization techniques for most of these elements can be found elsewhere [5-10]. For example, aqua regia may be needed for Mn [6,12].
- 6. Add 2 mL ashing acid and repeat step 5. Repeat this step until the solution is clear.
- 7. Remove watchglass and rinse into the beaker with distilled water.
- 8. Increase the temperature to 150  $^{\circ}$ C and take the sample to near dryness (ca. 0.5 mL).
- 9. Dissolve the residue in 2 to 3 mL dilution acid.
- 10. Transfer the solutions quantitatively to 25-mL volumetric flasks.
- 11. Dilute to volume with dilution acid.
  - NOTE: If more sensitivity is required, the final sample volume may be held to 10 mL.

# CALIBRATION AND QUALITY CONTROL:

12. Calibrate the spectrometer according to the manufacturers recommendations.

NOTE: Typically, an acid blank and  $1.0~\mu\text{g/mL}$  multielement working standards are used. The following multielement combinations are chemically compatible in 4% HNO<sub>3</sub>/1% HClO<sub>4</sub>:

- a. Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, La, In, Na
- b. Ag, K, Li, Mg, Mn, Ni, P, Pb, Se, Sr, Ti, V, Y, Zn, Sc
- c. Mo, Sb, Sn, Te, Ti, W, Zr
- d. Acid blank
- 13. Analyze a standard for every ten samples.
- 14. Check recoveries with at least two spiked blank filters per ten samples.

# **MEASUREMENT:**

- 15. Set spectrometer to conditions specified by manufacturer.
- 16. Analyze standards and samples.

NOTE: If the values for the samples are above the range of the standards, dilute the solutions with dilution acid, reanalyze and apply the appropriate dilution factor in the calculations.

# **CALCULATIONS:**

- 17. Obtain the solution concentrations for the sample,  $C_s$  (µg/mL), and the average media blank,  $C_b$  (µg/mL),
- 18. Using the solution volumes of sample,  $V_s$  (mL), and media blank,  $V_b$  (mL), calculate the concentration, C (mg/m³), of each element in the air volume sampled, V (L):

$$C = \frac{CsVs - CbVb}{V}, mg/m^3$$

NOTE:  $\mu g/L = mg/m^3$ 

# **EVALUATION OF METHOD:**

# Issues 1 and 2

Method, 7300 was originally evaluated in 1981 [2,3]. The precision and recovery data were determined at 2.5 and 1000 µg of each element per sample on spiked filters. The measurements used for the method evaluation in Issues 1 and 2 were determined with a Jarrell-Ash Model 1160 inductively Coupled Plasma Spectrometer operated according to manufacturer's instructions.

In this update of NIOSH Method 7300, the precision and recovery data were determined at approximately 3x and 10x the instrumental detection limits on commercially prepared spiked filters [12] using 25.0 mL as the final sample volume. Tables 3 and 4 list the precision and recovery data, instrumental detection limits, and analytical wavelengths for mixed cellulose ester (MCE) and polyvinyl chloride (PVC) filters. PVC Filters which can be used for total dust measurements and then digested for metals measurements were tested and found to give good results. The values in Tables 3 and 4 were determined with a Spectro Analytical Instruments Model End On Plasma (EOP)(axial) operated according to manufacturer's instructions.

# REFERENCES:

- [1] Millson M, Andrews R [2002]. Backup data report, Method 7300, unpublished report, NIOSH/DART.
- [2] Hull RD [1981]. Multielement Analysis of Industrial Hygiene Samples, NIOSH Internal Report, presented at the American Industrial Hygiene Conference, Portland, Oregon.
- [3] NIOSH [1982]. NIOSH Manual of Analytical Methods, 2nd ed., V. 7, P&CAM 351 (Elements by ICP), U.S. Department of Health and Human Services, Publ. (NIOSH) 82-100.
- [4] NIOSH [1994]. Elements by ICP: Method 7300, Issue 2. In: Eller PM, Cassinelli ME, eds., NIOSH Manual of Analytical Methods, 4<sup>th</sup> ed. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 94-113.
- [5] NIOSH [1994]. Lead by FAAS: Method 7082. In: Eller PM, Cassinelli ME, eds., NIOSH Manual of Analytical Methods, 4<sup>th</sup> ed. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 94-113.
- [6] NIOSH [1977]. NIOSH Manual of Analytical Methods, 2nd ed., V. 2, S5 (Manganese), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B.
- [7] NIOSH [1994]. Tungsten, soluble/insoluble: Method 7074. In: Eller PM, Cassinelli ME, eds., NIOSH Manual of Analytical Methods, 4<sup>th</sup> ed. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 94-113.
- [8] NIOSH [1979]. NIOSH Manual of Analytical Methods, 2nd ed., V. 5, P&CAM 173 (Metals by Atomic Absorption), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141.
- [9] NIOSH [1977]. NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S183 (Tin), S185 (Zirconium), and S376 (Molybdenum), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C.
- [10] ISO [2001]. Workplace air Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry - Part 2: Sample preparation. International Organization for Standardization. ISO 15202-2:2001(E).
- [11] ASTM [1985]. 1985 Annual Book of ASTM Standards, Vol. 11.01; Standard Specification for Reagent Water; ASTM, Philadelphia, PA, D1193-77 (1985).
- [12] Certification Inorganic Ventures for spikes.

# **METHOD REVISED BY:**

Mark Millson and Ronnee Andrews, NIOSH/DART.

Method originally written by Mark Millson, NIOSH/DART, and R. DeLon Hull, Ph.D., NIOSH/DSHEFS, James B. Perkins, David L. Wheeler, and Keith Nicholson, DataChem Labortories, Salt Lake City, UT.

TABLE 1. PROPERTIES AND SAMPLING VOLUMES

Element	Proper	<u>ties</u>				
Element	Atomic		Air Volum	Air Volume, L @ OSHA PEL		
(Symbol)	Weight	MP, °C	MIN	MAX		
Silver (Ag)	107.87	961	250	2000		
Aluminum (AI)	26.98	660	5			
Arsenic (As)	74.92	817	5 5	100		
Barium (Ba)	137.34	710	50	2000		
Beryllium (Be)	9.01	1278	1250	2000		
Calcium (Ca)	40.08	842	5	2000		
Cadmium (Cd)	112.40	321	13	200		
Cobalt (Co)	58.93	1495	25	2000		
Chromium (Cr)	52.00	1890		2000		
Copper (Cu)	63.54	1083	5	1000		
Iron (Fe)	55.85	1535	5	1000		
Potassium (K)	39.10	63.65	5	100		
Lanthanum	138.91	920	5	1000		
Lithium (Li)	6.94	179	5	1000		
Magnesium (Mg)	24.31	651	100	2000		
Manganese (Mn)	54.94	1244	5	67		
Molybdenum (Mo)	95.94		5	200		
Nickel (Ni)	58.71	651	5	67		
Phosphorus (P)	30.97	1453	5	1000		
Lead (Pb)	207.19	44	25	2000		
Antimony (Sb)	121.75	328	50	2000		
Selenium (Se)	78.96	630.5	50	2000		
Tin (Sn)	118.69	217	13	2000		
Strontium (Sr)		231.9	5	1000		
Tellurium (Te)	87.62	769	10	1000		
Titanium (Ti)	127.60	450	25	2000		
Thallium (TI)	47.90	1675	5	100		
Vanadium (V)	204.37	304	25	2000		
Tungsten (W)	50.94	1890	5	2000		
Yttrium (Y)	183.85	3410	5	1000		
Zinc (Zn)	88.91	1495	5	1000		
Zirconium (Zr)	65.37	419	5	200		
-1100Hulli (Zr)	91.22	1852	5	200		

TABLE 2. EXPOSURE LIMITS, CAS #, RTECS

Element (Symbol)	CAS#	RTECS	Expos OSHA	Exposure Limits, mg/m³ (Ca = carcinogen) OSHA NIOSH ACGIH					
Silver (Ag)	(Ag) 7440-22-4 VW35000		0.01 (dust, fume, metal)	0.01 (metal, soluble)	0.1 (metal) 0.01 (soluble)				
Aluminum (Al)	7429-90-5	BD0330000	15 (total dust) 5 (respirable)	10 (total dust) 5 (respirable fume) 2 (salts, alkyls)	10 (dust) 5 (powders, fume) 2 (salts, alkyls)				
Arsenic (As)	7440-38-2	CG0525000	varies	C 0.002, Ca	0.01, Ca				
Barium (Ba)	7440-39-3	CQ8370000	0.5	0.5	0.5				
Beryllium (Be)	7440-41-7	DS1750000	0.002, C 0.005	0.0005, Ca	0.002, Ca				
Calcium (Ca)	7440-70-2	-	varies	varies	varies				
Cadmium (Cd)	7440-43-9	EU9800000	0.005	lowest feasible, Ca	0.01 (total), Ca 0.002 (respir.), Ca				
Cobalt (Co)	7440-48-4	GF8750000	0.1	0.05 (dust, fume)	0.02 (dust, fume)				
Chromium (Cr)	7440-47-3	GB4200000	0.5	0.5	0.5				
Copper (Cu)	7440-50-8	GL5325000	1 (dust, mists) 0.1 (fume)	1 (dust) 0.1 (fume)	1 (dust, mists) 0.2 (fume)				
Iron (Fe)	7439-89-6	NO4565500	10 (dust, fume)	5 (dust, fume)	5 (fume)				
Potassium (K)	7440-09-7	TS6460000		****					
Lanthanum	7439-91-0		-	_					
Lithium (Li)	7439-93-2				***				
Magnesium (Mg)	7439-95-4	OM2100000	15 (dust) as oxide 5 (respirable)	10 (fume) as oxide	10 (fume) as oxide				
Manganese (Mn)	7439-96-5	OO9275000	C 5	1; STEL 3	5 (dust) 1; STEL 3 (fume)				
Molybdenum (Mo)	7439-98-7	QA4680000	5 (soluble) 15 (total insoluble)	5 (soluble) 10 (insoluble)	5 (soluble) 10 (insoluble)				
Nickel (Ni)	7440-02-0	QR5950000	1	0.015, Ca	0.1 (soluble) 1 (insoluble, metal)				
Phosphorus (P)	7723-14-0	TH3500000	0.1	0.1	0.1				
Lead (Pb)	7439-92-1	OF7525000	0.05	0.05	0.05				
Antimony (Sb)	7440-36-0	CC4025000	0.5	0.5	0.5				
Selenium (Se)	7782-49-2	VS7700000	0.2	0.2	0.2				
Tin (Sn)	7440-31-5	XP7320000	2	2	2				
Strontium (Sr)	7440-24-6		-	****					
Tellurium (Te)	13494-80-9	WY2625000	0.1	0.1	0.1				
Titanium (Ti)	7440-32-6	XR1700000							
Thallium (Ti)	7440-28-0	XG3425000	0.1 (skin) (soluble)	0.1 (skin) (soluble)	0.1 (skin)				
Vanadium (V)	7440-62-2	YW240000		C 0.05					
Tungsten	7440-33-7	_	5	5 10 (STEL)	5 10 (STEL)				
Yttrium (Y)	7440-65-5	ZG2980000	1	N/A	1				
Zinc (Zn)	7440-66-6	ZG8600000							
Zirconium (Zr)	7440-67-7	ZH7070000	5	5, STEL 10	5, STEL 10				

TABLE 3. MEASUREMENT PROCEDURES AND DATA [1]. Mixed Cellulose Ester Filters (0.45 μm)

E	wavelength				% Recovery		Certified	%	Percent
Element	nm	μg/	ng/m L	3x LOD	(c)	RSD	10x LOD	Recovery	RSD
(a)		Filter		(b)		(N=25)	(b)	(c)	(N=25)
۸	200	0.040	17	0.77	400.0	0.64	2.04	00.0	4.50
Ag	328	0.042	1.7	0.77	102.9	2.64	3.21	98.3	1.53
Al	167	0.115	4.6	1.54	105.4	11.5	6.40	101.5	1.98
As	189	0.140	5.6	3.08	94.9	2.28	12.9	93.9	1.30
Ba	455	0.005	0.2	0.31	101.8	1.72	1.29	97.7	0.69
Be	313	0.005	0.2	0.31	100.0	1.44	1.29	98.4	0.75
Ca	317	0.908	36.3	15.4	98.7	6.65	64.0	100.2	1.30
Cd	226	0.0075	0.3	0.31	99.8	1.99	1.29	97.5	0.88
Co	228	0.012	0.5	0.31	100.8	1.97	1.29	98.4	0.90
Cr	267	0.020	0.8	0.31	93.4	16.3	1.29	101.2	2.79
Cu	324	0.068	2.7	1.54	102.8	1.47	6.40	100.6	0.92
Fe	259	0.095	3.8	1.54	103.3	5.46	6.40	98.0	0.95
K	766	1.73	69.3	23.0	90.8	1.51	96.4	97.6	0.80
La	408	0.048	1.9	0.77	102.8	2.23	3.21	100.1	0.92
Li	670	0.010	0.4	0.31	110.0	1.91	1.29	97.7	0.81
Mg	279	0.098	3.9	1.54	101.1	8.35	6.40	98.0	1.53
Mn	257	0.005	0.2	0.31	101.0	1.77	1.29	94.7	0.73
Мо	202	0.020	8.0	0.31	105.3	2.47	1.29	98.6	1.09
Ni	231	0.020	0.8	0.31	109.6	3.54	1.29	101.2	1.38
P	178	0.092	3.7	1.54	84.4	6.19	6.40	82.5	4.75
Pb	168	0.062	2.5	1.54	109.4	2.41	6.40	101.7	0.88
Sb	206	0.192	7.7	3.08	90.2	11.4	12.9	41.3	32.58
Se	196	0.135	5.4	2.3	87.6	11.6	9.64	84.9	4.78
Sn	189	0.040	1.6	0.77	90.2	18.0	3.21	49	21.79
Sr	407	0.005	0.2	0.31	101.0	1.55	1.29	97.3	0.65
Te	214	0.078	3.1	1.54	102.0	2.67	6.40	97.4	1.24
Ti	334	0.050	2.0	0.77	98.4	2.04	3.21	93.4	1.08
TI	190	0.092	3.7	1.54	100.9	2.48	6.40	99.1	0.80
V	292	0.028	1.1	0.77	103.2	1.92	3.21	98.3	0.84
w	207	0.075	3.0	1.54	72.2	10.1	6.40	57.6	14.72
Υ	371	0.012	0.5	0.31	100.5	1.80	1.29	97.4	0.75
Zn	213	0.310	12.4	4.60	102.2	1.87	19.3	95.3	0.90
Zr	339	0.022	0.9	0.31	88.0	19.4	1.29	25	57.87
				•			1		

<sup>(</sup>a) Bold values are qualitative only because of low recovery.

<sup>(</sup>b) Values are certified by Inorganic Ventures INC. at 3x and 10x the approximate instrumental LOD

<sup>(</sup>c) Values reported were obtained with a Spectro Analytical Instruments EOP ICP; performance may vary with instrument and should be independently verified.

TABLE 4. MEASUREMENT PROCEDURES AND DATA [1]. Polyvinyl Chloride Filter (5.0  $\mu m$ )

Element	wavelength		LOD	Certified	%		Certified <sup>17</sup>	%	Percent
(c)	nm	µg per	ng/m L	3x LOD	Recovery	RSD	10x LOD	Recovery	RSD
(0)		filter		(b)	(a)	(N=25)	(b)	(a)	(N=25)
Ag	328	0.042	, ,						
Al	167		1.7	0.78	104.2	8.20	3.18		18.9
As	189	0.115	4.6	1.56	77.4	115.24	6.40	92.9	20.9
Ba		0.140	5.6	3.10	100.7	5.13	12.70	96.9	3.2
Ве	455	0.005	0.2	0.31	102.4	3.89	1.270	99.8	2.0
	313	0.005	0.2	0.31	106.8	3.53	1.270	102.8	2.1
<b>Ca</b> Cd	317	0.908	36.3	15.6	68.1	12.66	64.00	96.8	5.3
	226	0.0075	0.3	0.31	105.2	5.57	1.27	101.9	2.8
Co	228	0.012	0.5	0.31	109.3	4.67	1.27	102.8	2.8
Cr	267	0.020	8.0	0.31	109.4	5.31	1.27	103.4	4.1
Cu	324	0.068	2.7	1.56	104.9	5.18	6.40	101.8	2.4
Fe	259	0.095	3.8	1.56	88.7	46.82	6.40	99.1	9.7
K	766	1.73	69.3	23.4	96.4	4.70	95.00	99.2	2.2
La	408	0.048	1.9	0.78	45.5	4.19	3.18	98.8	2.6
Li	670	0.010	0.4	0.31	107.7	4.80	1.27	110.4	2.7
Mg	279	0.098	3.9	1.56	54.8	20.59	6.40	64.5	5.7
Mn	257	0.005	0.2	0.31	101.9	4.18	1.27	99.3	2.4
Мо	202	0.020	8.0	0.31	106.6	5.82	1.27	98.1	3.8
Ni	231	0.020	8.0	0.31	111.0	5.89	1.27	103.6	3.2
P	178	0.092	3.7	1.56	101.9	17.82	6.40	86.5	10.4
Pb	168	0.062	2.5	1.56	109.6	6.12	6.40	103.2	2.9
Sb	206	0.192	7.7	3.10	64.6	22.54	12.70	38.1	30.5
Se	196	0.135	5.4	2.30	83.1	26.23	9.50	76.0	17.2
Sn	189	0.040	1.6	0.78	85.7	27.29	3.18	52.0	29.4
Sr	407	0.005	0.2	0.31	71.8	4.09	1.27	81.2	2.7
Te	214	0.078	3.1	1.56	109.6	7.49	6.40	97.3	3.8
Ti	334	0.050	2.0	0.78	101.0	9.46	3.18	92.4	5.5
ΤI	190	0.092	3.7	1.56	110.3	4.04	6.40	92.4 101.9	
V	292	0.028	1.1	0.78	108.3	3.94	3.18	101.9	2.0
W	207	0.075	3.0	1.56	74.9	15.79	6.40		2.6
Υ	371	0.012	0.5	0.31	101.5	3.63	1.27	44.7	19.6
Zn	213	0.310	12.4	4.70	91.0	68.69		101.4	2.5
Zr	339	0.022	0.9	0.31	70.7		19.1	101.0	9.6
<b></b>		0.022	0.0	0.51	10.1	54.20	1.27	40.4	42.1

<sup>(</sup>a) Values reported were obtained with a Spectro Analytical Instruments EOP ICP; performance may vary with instrument and should be independently verified.

<sup>(</sup>b) Values are certified by Inorganic Ventures INC. at 3x and 10x the approximate instrumental LOD [12].

<sup>(</sup>c) Bold values are qualitative only because of low recovery. Other digestion techniques may be more appropriate for these elements and their compounds.