

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

7300

MW: Table 1

CAS: Table 2

RTECS: Table 2

METHOD: 7300, Issue 3

EVALUATION: PARTIAL

Issue 1: 15 August 1990

Issue 3: 15 March 2003

OSHA: Table 2

PROPERTIES: Table 1

NIOSH: Table 2

ACGIH: Table 2

| ELEMENTS: |  | aluminum*  | calcium   | lanthanum   | nickel     | strontium | tungsten*  |
|-----------|--|------------|-----------|-------------|------------|-----------|------------|
|           |  | antimony*  | chromium* | lithium*    | potassium  | tellurium | vanadium*  |
|           |  | arsenic    | cobalt*   | magnesium   | phosphorus | tin       | yttrium    |
|           |  | barium     | copper    | manganese*  | selenium   | thallium  | zinc       |
|           |  | beryllium* | iron      | molybdenum* | silver     | titanium  | zirconium* |
|           |  | cadmium    | lead*     |             |            |           |            |

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

| SAMPLING                                               |                                                                                                     | MEASUREMENT                              |                                                                                                 |
|--------------------------------------------------------|-----------------------------------------------------------------------------------------------------|------------------------------------------|-------------------------------------------------------------------------------------------------|
| <b>SAMPLER:</b>                                        | FILTER<br>(0.8- $\mu$ m, cellulose ester membrane, or<br>5.0- $\mu$ m, polyvinyl chloride membrane) | <b>TECHNIQUE:</b>                        | INDUCTIVELY COUPLED ARGON<br>PLASMA, ATOMIC EMISSION<br>SPECTROSCOPY (ICP-AES)                  |
| <b>FLOWRATE:</b>                                       | 1 to 4 L/min                                                                                        | <b>ANALYTE:</b>                          | elements above                                                                                  |
| <b>VOL-MIN:</b>                                        | Table 1                                                                                             | <b>ASHING</b>                            |                                                                                                 |
| <b>-MAX:</b>                                           | Table 1                                                                                             | <b>REAGENTS:</b>                         | conc. HNO <sub>3</sub> / conc. HClO <sub>4</sub> (4:1), 5 mL;<br>2mL increments added as needed |
| <b>SHIPMENT:</b>                                       | routine                                                                                             | <b>CONDITIONS:</b>                       | room temperature, 30 min; 150 °C to near<br>dryness                                             |
| <b>SAMPLE</b>                                          |                                                                                                     | <b>FINAL</b>                             |                                                                                                 |
| <b>STABILITY:</b>                                      | stable                                                                                              | <b>SOLUTION:</b>                         | 4% HNO <sub>3</sub> , 1% HClO <sub>4</sub> , 25 mL                                              |
| <b>BLANKS:</b>                                         | 2 to 10 field blanks per set                                                                        | <b>WAVELENGTH:</b>                       | depends upon element; Table 3                                                                   |
| <b>ACCURACY</b>                                        |                                                                                                     | <b>BACKGROUND</b>                        |                                                                                                 |
|                                                        |                                                                                                     | <b>CORRECTION:</b>                       | spectral wavelength shift                                                                       |
| <b>RANGE STUDIED:</b>                                  | not determined                                                                                      | <b>CALIBRATION:</b>                      | elements in 4% HNO <sub>3</sub> , 1% HClO <sub>4</sub>                                          |
| <b>BIAS:</b>                                           | not determined                                                                                      | <b>RANGE:</b>                            | varies with element [1]                                                                         |
| <b>OVERALL PRECISION (<math>\hat{S}_{r,r}</math>):</b> | not determined                                                                                      | <b>ESTIMATED LOD:</b>                    | Tables 3 and 4                                                                                  |
| <b>ACCURACY:</b>                                       | not determined                                                                                      | <b>PRECISION (<math>\hat{S}</math>):</b> | Tables 3 and 4                                                                                  |

**APPLICABILITY:** The working range of this method is 0.005 to 2.0 mg/m<sup>3</sup> 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 furnace 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 (HClO<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. HClO<sub>4</sub>.
4. Calibration stock solutions, 1000 µg/mL. Commercially available, or prepared per instrument manufacturer's recommendation (see step 12).
5. Dilution acid, 4% HNO<sub>3</sub>, 1% HClO<sub>4</sub>. 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 °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 µg/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, Tl, 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<sub>s</sub> (µg/mL), and the average media blank, C<sub>b</sub> (µg/mL), from the instrument.
18. Using the solution volumes of sample, V<sub>s</sub> (mL), and media blank, V<sub>b</sub> (mL), calculate the concentration, C (mg/m<sup>3</sup>), of each element in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, \text{mg} / \text{m}^3$$

NOTE: µg/L ≡ mg/m<sup>3</sup>

**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.

**Issue 3**

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 Laboratories, Salt Lake City, UT.

TABLE 1. PROPERTIES AND SAMPLING VOLUMES

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

TABLE 2. EXPOSURE LIMITS, CAS #, RTECS

| Element<br>(Symbol) | CAS #      | RTECS     | Exposure Limits, mg/m <sup>3</sup> (Ca = carcinogen) |                                                             |                                                     |
|---------------------|------------|-----------|------------------------------------------------------|-------------------------------------------------------------|-----------------------------------------------------|
|                     |            |           | OSHA                                                 | NIOSH                                                       | ACGIH                                               |
| Silver (Ag)         | 7440-22-4  | VW3500000 | 0.01 (dust, fume, metal)                             | 0.01 (metal, soluble)                                       | 0.1 (metal)<br>0.01 (soluble)                       |
| Aluminum (Al)       | 7429-90-5  | BD0330000 | 15 (total dust)<br>5 (respirable)                    | 10 (total dust)<br>5 (respirable fume)<br>2 (salts, alkyls) | 10 (dust)<br>5 (powders, fume)<br>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<br>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)<br>0.1 (fume)                        | 1 (dust)<br>0.1 (fume)                                      | 1 (dust, mists)<br>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<br>5 (respirable)                 | 10 (fume) as oxide                                          | 10 (fume) as oxide                                  |
| Manganese (Mn)      | 7439-96-5  | OO9275000 | C 5                                                  | 1; STEL 3                                                   | 5 (dust)<br>1; STEL 3 (fume)                        |
| Molybdenum (Mo)     | 7439-98-7  | QA4680000 | 5 (soluble)<br>15 (total insoluble)                  | 5 (soluble)<br>10 (insoluble)                               | 5 (soluble)<br>10 (insoluble)                       |
| Nickel (Ni)         | 7440-02-0  | QR5950000 | 1                                                    | 0.015, Ca                                                   | 0.1 (soluble)<br>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 (Tl)       | 7440-28-0  | XG3425000 | 0.1 (skin) (soluble)                                 | 0.1 (skin) (soluble)                                        | 0.1 (skin)                                          |
| Vanadium (V)        | 7440-62-2  | YW2400000 | --                                                   | C 0.05                                                      | --                                                  |
| Tungsten            | 7440-33-7  | --        | 5                                                    | 5<br>10 (STEL)                                              | 5<br>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)**

| Element<br>(a) | wavelength<br>nm | Est. LOD<br>µg/<br>Filter | LOD<br>ng/mL | Certified<br>3x LOD<br>(b) | % Recovery<br>(c) | Percent<br>RSD<br>(N=25) | Certified<br>10x LOD<br>(b) | %<br>Recovery<br>(c) | Percent<br>RSD<br>(N=25) |
|----------------|------------------|---------------------------|--------------|----------------------------|-------------------|--------------------------|-----------------------------|----------------------|--------------------------|
| 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                     |
| Mo             | 202              | 0.020                     | 0.8          | 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                     |
| <b>Sb</b>      | 206              | 0.192                     | 7.7          | 3.08                       | 90.2              | 11.4                     | 12.9                        | <b>41.3</b>          | 32.58                    |
| Se             | 196              | 0.135                     | 5.4          | 2.3                        | 87.6              | 11.6                     | 9.64                        | 84.9                 | 4.78                     |
| <b>Sn</b>      | 189              | 0.040                     | 1.6          | 0.77                       | 90.2              | 18.0                     | 3.21                        | <b>49</b>            | 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                     |
| Tl             | 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                     |
| <b>W</b>       | 207              | 0.075                     | 3.0          | 1.54                       | <b>72.2</b>       | 10.1                     | 6.40                        | <b>57.6</b>          | 14.72                    |
| Y              | 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                     |
| <b>Zr</b>      | 339              | 0.022                     | 0.9          | 0.31                       | 88.0              | 19.4                     | 1.29                        | <b>25</b>            | 57.87                    |

- (a) Bold values are qualitative only because of low recovery.  
(b) Values are certified by Inorganic Ventures INC. at 3x and 10x the approximate instrumental LOD  
(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 µm)**

| Element<br>(c) | wavelength<br>nm | Est. LOD<br>µg per<br>filter | LOD<br>ng/mL | Certified<br>3x LOD<br>(b) | %<br>Recovery<br>(a) | Percent<br>RSD<br>(N=25) | Certified <sup>17</sup><br>10x LOD<br>(b) | %<br>Recovery<br>(a) | Percent<br>RSD<br>(N=25) |
|----------------|------------------|------------------------------|--------------|----------------------------|----------------------|--------------------------|-------------------------------------------|----------------------|--------------------------|
| Ag             | 328              | 0.042                        | 1.7          | 0.78                       | 104.2                | 8.20                     | 3.18                                      | 81.8                 | 18.9                     |
| Al             | 167              | 0.115                        | 4.6          | 1.56                       | 77.4                 | 115.24                   | 6.40                                      | 92.9                 | 20.9                     |
| As             | 189              | 0.140                        | 5.6          | 3.10                       | 100.7                | 5.13                     | 12.70                                     | 96.9                 | 3.2                      |
| Ba             | 455              | 0.005                        | 0.2          | 0.31                       | 102.4                | 3.89                     | 1.270                                     | 99.8                 | 2.0                      |
| Be             | 313              | 0.005                        | 0.2          | 0.31                       | 106.8                | 3.53                     | 1.270                                     | 102.8                | 2.1                      |
| <b>Ca</b>      | 317              | 0.908                        | 36.3         | 15.6                       | <b>68.1</b>          | 12.66                    | 64.00                                     | 96.8                 | 5.3                      |
| Cd             | 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                        | 0.8          | 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                      |
| <b>La</b>      | 408              | 0.048                        | 1.9          | 0.78                       | <b>45.5</b>          | 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                      |
| <b>Mg</b>      | 279              | 0.098                        | 3.9          | 1.56                       | <b>54.8</b>          | 20.59                    | 6.40                                      | <b>64.5</b>          | 5.7                      |
| Mn             | 257              | 0.005                        | 0.2          | 0.31                       | 101.9                | 4.18                     | 1.27                                      | 99.3                 | 2.4                      |
| Mo             | 202              | 0.020                        | 0.8          | 0.31                       | 106.6                | 5.82                     | 1.27                                      | 98.1                 | 3.8                      |
| Ni             | 231              | 0.020                        | 0.8          | 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                      |
| <b>Sb</b>      | 206              | 0.192                        | 7.7          | 3.10                       | <b>64.6</b>          | 22.54                    | 12.70                                     | <b>38.1</b>          | 30.5                     |
| Se             | 196              | 0.135                        | 5.4          | 2.30                       | 83.1                 | 26.23                    | 9.50                                      | 76.0                 | 17.2                     |
| <b>Sn</b>      | 189              | 0.040                        | 1.6          | 0.78                       | 85.7                 | 27.29                    | 3.18                                      | <b>52.0</b>          | 29.4                     |
| <b>Sr</b>      | 407              | 0.005                        | 0.2          | 0.31                       | <b>71.8</b>          | 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                      |
| Tl             | 190              | 0.092                        | 3.7          | 1.56                       | 110.3                | 4.04                     | 6.40                                      | 101.9                | 2.0                      |
| V              | 292              | 0.028                        | 1.1          | 0.78                       | 108.3                | 3.94                     | 3.18                                      | 102.5                | 2.6                      |
| <b>W</b>       | 207              | 0.075                        | 3.0          | 1.56                       | <b>74.9</b>          | 15.79                    | 6.40                                      | <b>44.7</b>          | 19.6                     |
| Y              | 371              | 0.012                        | 0.5          | 0.31                       | 101.5                | 3.63                     | 1.27                                      | 101.4                | 2.5                      |
| Zn             | 213              | 0.310                        | 12.4         | 4.70                       | 91.0                 | 68.69                    | 19.1                                      | 101.0                | 9.6                      |
| <b>Zr</b>      | 339              | 0.022                        | 0.9          | 0.31                       | <b>70.7</b>          | 54.20                    | 1.27                                      | <b>40.4</b>          | 42.1                     |

- (a) Values reported were obtained with a Spectro Analytical Instruments EOP ICP; performance may vary with instrument and should be independently verified.
- (b) Values are certified by Inorganic Ventures INC. at 3x and 10x the approximate instrumental LOD [12].
- (c) Bold values are qualitative only because of low recovery. Other digestion techniques may be more appropriate for these elements and their compounds.