ELEMENTS ON WIPES

METHOD: 9102, Issue 1
EVALUATION: PARTIAL
Issue 1: 15 March 2003

OSHA: N/A
NIOSH: N/A
ACGIH: N/A

PROPERTIES: Table 1

ELEMENTS:
- Arsenic
- Iron
- Selenium
- Zinc
- Barium
- Lanthanum
- Silver
- Zirconium
- Beryllium
- Lead
- Strontium
- Cobalt
- Molybdenum
- Thallium
- Copper
- Nickel
- Vanadium
- Chromium
- Phosphorus
- Yttrium

SAMPLER: WIPE
FLOW RATE: N/A
VOL-MIN: N/A
VOL-MAX: N/A
SHIPMENT: Routine
SAMPLE STABILITY: Stable
BLANKS: 2 to 10 field blanks per set

TECHNIQUE: INDUCTIVELY COUPLED ARGON PLASMA, ATOMIC EMISSION SPECTROSCOPY (ICP-AES)

ANALYTE: Elements above
ASHING:
REAGENTS:
- Conc. HNO₃, 20 mL; and conc. HClO₄, 1 mL
CONDITIONS:
- Room temperature, 30 min; 150 °C to near dryness
FINAL SOLUTION:
- 4% HNO₃, 1% HClO₄, 10 mL
WAVELENGTH:
Table 2
BACKGROUND CORRECTION:
Spectral wavelength shift
CALIBRATION:
- Elements in 4% HNO₃, 1% HClO₄
RANGE:
Table 2
ESTIMATED LOD:
Table 2
PRECISION (\(\bar{r}\);):
Table 2

APPLICABILITY: This method is a simultaneous elemental analysis, not compound specific. Verify that the types of compounds in the samples are soluble with the ashing procedure selected. Because this method deals with wipes samples it is important to keep in mind that the values obtained are qualitative.

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.

OTHER METHODS: This method supplements NMAM Method 7300 [1]. Additional element methods, whether single element or multi-element, may be found in NMAM. Alternate instrumentation includes flame atomic absorption spectroscopy for certain elements and graphite furnace AAS for increased sensitivity.
REAGENTS:

1. Nitric acid (HNO₃), conc., ultra pure.*
2. Perchloric acid (HClO₄), conc., ultra pure.*
3. Calibration stock solutions, 1000 µg/mL commercially available, or prepared per instrument manufacturer’s recommendation (see step 20).
4. Dilution acid, 4:1 HNO₃:HClO₄. Mix 4 volumes conc. HNO₃ with 1 volume conc. HClO₄.
5. Argon.
6. Distilled, de-ionized water.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: Wash ‘n Dri or ASTM equivalent[2] [pre-packaged moist disposable towelette (wipe)].
2. Inductively coupled plasma-atomic emission spectrometer, equipped as specified by the manufacturer for analysis of elements of interest.
3. Regulator, two-stage for argon.
4. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with watchglass covers.**
5. Volumetric flasks, 10- and 100-mL.**
6. Assorted volumetric pipettes as needed.**
7. Hotplate, surface temperature 150 °C.
8. Polystyrene centrifuge tubes, 50-mL and 15-mL.

** Clean all glassware with conc. nitric acid and rinse thoroughly in de-ionized water.

SPECIAL PRECAUTIONS: Nitric and perchloric acids are strong oxidizers and extremely corrosive. Perform all perchloric acid digestions in a perchloric acid hood. When working with acids use gloves and avoid inhalation or contact with skin or clothing.

SAMPLING:[3]

1. Wearing a clean pair of powder-less, plastic gloves, place the template over the area to be sampled and secure it. If the area to be sampled is in a confined area and a template cannot be used, measure the sampling area, and delineate the area to be sampled with masking tape.
2. Remove a wipe from its package, and unfold it.
3. Wipe the surface to be sampled using fingertips held together and applying firm pressure. Use an overlapping ‘S’ pattern to cover the entire surface with horizontal strokes.
4. Fold the exposed side of the wipe in, and wipe the same area using vertical ‘S’-strokes.
5. Fold the wipe again to reveal an unexposed surface, and wipe the surface a third time as described in step 3.
6. Fold the wipe, exposed side in, and place it into a clean hard-walled sample container (e.g., 50-mL centrifuge tube). Seal securely and label the sample container.
   NOTE: Compositing of wipe samples is not recommended, because (a) they cause sample preparation and analytical difficulties, and (b) site-specific analytical information is lost.
7. Clean the template in preparation for the next wipe sample using water or a wipe. A disposable template may also be used.
8. Remove gloves and discard. Clean gloves should be worn for each new sample.
9. Field blanks: 10% of samples, at least three per batch. Remove unexposed wipes from packaging and place into sample containers. The field blanks should be collected at the beginning, middle, and end of sampling.

SAMPLE PREPARATION:

10. Transfer the samples and blanks to clean beakers.
11. Add 20 mL concentrated nitric acid and 1 mL concentrated perchloric acid. Cover beaker with a watchglass. Let stand 30 min at room temperature. Start a reagent blank at this step.
12. Allow samples to heat for approximately 8 hours @150 °C.
13. Remove watchglass and rinse into the beaker with de-ionized water.
14. Continue to heat on hotplate (120 °C).
   NOTE: Some species will not be completely solubilized by this procedure. Alternative solubilization
   techniques for most of these elements can be found elsewhere.
15. Add additional amounts of HNO₃, if necessary, until the solution is clear and the wipe media is
   completely destroyed.
16. Take the sample to near dryness (ca. 0.5 mL).
17. Dissolve the residue in 0.5 mL dilution acid.
18. Transfer the solutions quantitatively to 15-mL centrifuge tubes or 10 mL volumetric flasks.
19. Dilute to 10 mL with de-ionized water.

CALIBRATION AND QUALITY CONTROL:

20. Calibrate the spectrometer according to the manufacturers recommendations.
   NOTE: Typically, an acid blank and 10 µg/mL multi-element working standards are used. The
   following multi-element combinations are chemically and spectrally compatible in
   4% HNO₃/1% HClO₄:
   a. Al, As, Ba, Be, Cd, Ca, Cr, Co, Cu, In, Fe, La;
   b. Pb, Li, Mg, Mn, Ni, P, K, Sc, Se, Ag, Sr, Ti, V, Y, Zn;
   c. Sb, Mo, Te Sn, Ti, W, Zr;
   d. Pt
21. Analyze a standard for every six samples.
22. Check recoveries with at least two spiked media blanks per ten samples.

MEASUREMENT:

23. Set spectrometer to conditions specified by manufacturer.
   NOTE: If the values for the samples are above the range of the standards, dilute the solutions with
   dilution acid, re-analyze and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

25. Obtain the solution concentrations for the sample, Cₛ (µg/mL), and the average media blank, Cᵇ
    (µg/mL), from the instrument.
26. Using the solution volumes of sample, Vₛ (mL), and media blank, Vᵇ (mL), calculate the concentration,
    C (µg/cm²), of each element on the surface area sampled, S (cm²):

    \[ C = \frac{Cₛ Vₛ - Cᵇ Vᵇ}{S}, \mu g / cm². \]

EVALUATION OF METHOD:

This method was evaluated using wipe media spiked with aqueous standards (fortified samples) and wipe
media spiked with SRM 2711 (Montana Soil). Recoveries for the twenty-three elements listed on page 9201-1
were within the range of 75-115% for the fortified samples. Ten of the elements found in SRM 2711 were
recoverable within the same range. These are: silver, arsenic, cadmium, cobalt, copper, manganese, nickel,
phosphorus, lead, and zinc.
Because this method is a simultaneous elemental analysis, it is not compound specific. The data provided is for those compounds compatible with the acids used in the digestion. Verification of the solubility of specific compounds of interest should be completed and the sample matrix carefully examined. For example, if silicates are present total elemental values will not be measured without the use of hydrofluoric acid in the digestion process.

REFERENCES:


METHOD WRITTEN BY:

Ronnee N. Andrews, Mark Millson, and Donald D. Dollberg, NIOSH/DART

TABLE 1. PROPERTIES

<table>
<thead>
<tr>
<th>Element (Symbol)</th>
<th>Atomic Weight</th>
<th>MP (° C)</th>
<th>CAS #</th>
<th>RTECS</th>
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<tbody>
<tr>
<td>Silver (Ag)</td>
<td>107.87</td>
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<td>Manganese (Mn)</td>
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NIOSH Manual of Analytical Methods (NMAM), Fourth Edition
<table>
<thead>
<tr>
<th>Element (Symbol)</th>
<th>Wavelength (nm)</th>
<th>Instrumental LOD (µg/wipe)</th>
<th>Range (µg/wipe)</th>
<th>Precision* (S_r)</th>
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</thead>
<tbody>
<tr>
<td>Silver (Ag)</td>
<td>328</td>
<td>0.035</td>
<td>0.118-25.0</td>
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<td>Arsenic (As)</td>
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<td>Beryllium (Be)</td>
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<td>Cadmium (Cd)</td>
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<td>Cobalt (Co)</td>
<td>228</td>
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<td>Chromium (Cr)</td>
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<td>Copper (Cu)</td>
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<td>Iron (Fe)</td>
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<td>Lanthanum (La)</td>
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<td>Manganese (Mn)</td>
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<td>0.0333</td>
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<td>Nickel (Ni)</td>
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<td>Phosphorus (P)</td>
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<td>0.128</td>
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<tr>
<td>Lead (Pb)</td>
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<td>0.042</td>
<td>0.141-1160</td>
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<td>0.026</td>
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<td>0.344</td>
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* Values were obtained with a Spectro ICP-AES; LOD may vary with instrument and should be independently verified.