

ELEMENTS ON WIPES

9102

MW: Table 1

CAS: Table 1

RTECS: Table 1

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 Barium Beryllium Cadmium Cobalt Copper Chromium	Iron Lanthanum Lead Manganese Molybdenum Nickel Phosphorus	Selenium Silver Strontium Tellurium Thallium Vanadium Yttrium	Zinc Zirconium
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SAMPLING	MEASUREMENT
<p>SAMPLER: WIPE</p> <p>FLOW RATE: N/A</p> <p>VOL-MIN: N/A -MAX:</p> <p>SHIPMENT: Routine</p> <p>SAMPLE STABILITY: Stable</p> <p>BLANKS: 2 to 10 field blanks per set</p>	<p>TECHNIQUE: INDUCTIVELY COUPLED ARGON PLASMA, ATOMIC EMISSION SPECTROSCOPY (ICP-AES)</p> <p>ANALYTE: Elements above</p> <p>ASHING REAGENTS: Conc. HNO₃, 20 mL; and conc. HClO₄, 1 mL</p> <p>CONDITIONS: Room temperature, 30 min; 150 °C to near dryness</p> <p>FINAL SOLUTION: 4% HNO₃, 1% HClO₄, 10 mL</p> <p>WAVELENGTH: Table 2</p> <p>BACKGROUND CORRECTION: Spectral wavelength shift</p> <p>CALIBRATION: Elements in 4% HNO₃, 1% HClO₄</p> <p>RANGE: Table 2</p> <p>ESTIMATED LOD: Table 2</p> <p>PRECISION (S_r): Table 2</p>
ACCURACY	
<p>RANGE STUDIED: Not Studied</p> <p>BIAS: Not determined</p> <p>OVERALL PRECISION (S_{rT}): Not determined</p> <p>ACCURACY: Not determined</p>	

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 .
9. Hard-walled sample container.

** 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, Tl, 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.
24. Analyze standards and samples.
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, C_s (µg/mL), and the average media blank, C_b (µg/mL), from the instrument.
26. Using the solution volumes of sample, V_s (mL), and media blank, V_b (mL), calculate the concentration, C (µg/cm²), of each element on the surface area sampled, S (cm²):

$$C = \frac{C_s V_s - C_b V_b}{S}, \mu\text{g} / \text{cm}^2.$$

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:

- [1] NIOSH [1994]. Elements by ICP, Method 7300 in: Eller PM, Cassinelli ME, eds. NIOSH Manual of Analytical Methods, 4th ed., Cincinnati, OH: U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety & Health. DHHS (NIOSH) Publication No. 94-113.
- [2] ASTM [2002]. Annual Book of ASTM Standards, Standard Specification for Wipe Sampling Materials for Lead in Surface Dust. West Conshohocken, PA: ASTM International, E 1792-01.
- [3] ASTM [2002]. Annual Book of ASTM Standards, Standard Practice for Field Collection of Settled Dust Samples Using Wipe Sampling Methods for Lead Determination by Atomic Spectrometry Techniques. West Conshohocken, PA: ASTM International, E 1728-99.

METHOD WRITTEN BY:

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TABLE 1. PROPERTIES

Element (Symbol)	Atomic Weight	MP (° C)	CAS #	RTECS
Silver (Ag)	107.87	961	7440-22-4	VW3675000
Arsenic (As)	74.92	817	7440-38-2	CG0525000
Barium (Ba)	137.33	725	7440-39-3	CQ8370000
Beryllium (Be)	9.01	1287	7440-41-7	DS1750000
Cadmium (Cd)	112.41	321	7440-43-9	EU9800000
Cobalt (Co)	58.93	1493	7440-48-4	GF8750000
Chromium (Cr)	52.00	1900	7440-47-3	GB4200000
Copper (Cu)	63.54	1083	7440-50-8	GL5325000
Iron (Fe)	55.85	1535	7439-89-6	NO4565500
Lanthanum (La)	138.91	920	7439-91-0	
Manganese (Mn)	54.94	1244	7439-96-5	OO9275000
Molybdenum (Mo)	95.94	2622	7439-98-7	QA4680000
Nickel (Ni)	58.69	1555	7440-02-0	QR5950000
Phosphorus (P)	30.97	44	7723-14-0	TH3495000
Lead (Pb)	207.2	328	7439-92-1	OF7525000
Selenium (Se)	78.96	217	7782-49-2	VS7700000
Strontium (Sr)	87.62	769	7440-24-6	WK7849000
Tellurium (Te)	127.60	450	13494-80-9	WY2625000
Thallium (Tl)	204.38	304	7440-28-0	XG3425000
Vanadium (V)	50.94	1917	7440-62-2	YW1355000
Yttrium (Y)	88.91	1509	7440-65-5	ZG2980000
Zinc (Zn)	65.39	420	7440-66-6	ZG8600000
Zirconium (Zr)	91.22	1857	7440-67-7	ZH7070000

TABLE 2. WAVELENGTH, LIMITS OF DETECTION (LOD), RANGE & PRECISION

Element (Symbol)	Wavelength (nm)	Instrumental LOD (µg/wipe)	Range (µg/wipe)	Precision* (S_r)
Silver (Ag)	328	0.035	0.118-25.0	0.0701
Arsenic (As)	189	0.078	0.261-105.	0.0549
Barium (Ba)	455	0.010	0.0333-10.0	0.0370
Beryllium (Be)	313	0.010	0.0333-10.0	0.0267
Cadmium (Cd)	226	0.010	0.0333-41.7	0.0273
Cobalt (Co)	228	0.010	0.0333-10.0	0.0301
Chromium (Cr)	267	0.010	0.0333-10.0	0.0287
Copper (Cu)	324	0.042	0.141-114.	0.0317
Iron (Fe)	259	0.074	0.246-50.0	0.0516
Lanthanum (La)	408	0.018	0.0593-25.0	0.0279
Manganese (Mn)	257	0.010	0.0333-638.	0.0333
Molybdenum (Mo)	202	0.010	0.0333-10.0	0.0441
Nickel (Ni)	231	0.010	0.0340-20.6	0.0629
Phosphorus (P)	178	0.043	0.142-860.	0.128
Lead (Pb)	168	0.042	0.141-1160	0.0267
Selenium (Se)	196	0.10	0.338-75.0	0.0364
Strontium (Sr)	407	0.010	0.0333-10.0	0.0368
Tellurium (Te)	214	0.047	0.156-50.0	0.0359
Thallium (Tl)	190	0.041	0.136-50.0	0.0256
Vanadium (V)	292	0.010	0.0333-25.0	0.0284
Yttrium (Y)	371	0.010	0.0333-10.0	0.0280
Zinc (Zn)	213	0.77	2.56-350.	0.101
Zirconium (Zr)	339	0.026	0.0877-10.0	0.344

* Values were obtained with a Spectro ICP-AES; LOD may vary with instrument and should be independently verified.