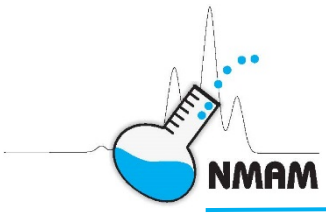

BACKUP DATA REPORT
NIOSH Method No. 7302

Title: Elements by ICP using microwave digestion

Analyte: 33 elements

Author/developer: Lee C. Stone, James B. Perkins, David M. Rogers
Date: April 17, 2003

NOTE: Report written under contract CDC-200-2001-08000

DATA CHEM LABORATORIES, INC.

ELEMENTS BY ICP USING MICROWAVE DIGESTION

NMAM 7302, ISSUE 1

BACKUP DATA REPORT

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FINAL
APRIL 17, 2003

CONTRACTS CDC-200-2001-08000

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ELEMENTS by ICP using MICROWAVE DIGESTION: Backup Data Report

INTRODUCTION

This procedure is applicable to industrial hygiene air monitoring both in the work place and in the environment in general. This method incorporates the speed of Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) multi-element analysis along with the simplicity of microwave single acid digestion. For all of the elements studied the method is viable for these elements as metals, nitrates, and most of their oxides.

REAGENTS AND STANDARDS

Presented in Tables 1 and 2 are the lists of reagents and standards used for this method and its evaluation. All of the standards were prepared in 20% nitric acid by the vendors.

TABLE 1. LIST OF CHEMICALS

Chemical	Vendor	CAS#	Purity	Lot#
Nitric Acid	J. T. Baker	7697-37-2	70%	9598-34
Water	DataChem Laboratories	7732-18-5	ASTM Type II [1]	--

TABLE 2. LIST OF STANDARDS

Analyte	Primary ID#	Primary Conc.	Vendor	Lot#
Al	IP-PS-02-012	1000 µg/mL	CPI	2AF233
As	IP-PS-02-012	500 µg/mL	CPI	2AF233
Ba	IP-PS-02-012	200 µg/mL	CPI	2AF233
Be	IP-PS-02-012	200 µg/mL	CPI	2AF233
Ca	IP-PS-02-012	1000 µg/mL	CPI	2AF233
Co	IP-PS-02-012	200 µg/mL	CPI	2AF233
Cr	IP-PS-02-012	200 µg/mL	CPI	2AF233
Cu	IP-PS-02-012	200 µg/mL	CPI	2AF233
Fe	IP-PS-02-012	500 µg/mL	CPI	2AF233
Li	IP-PS-02-012	200 µg/mL	CPI	2AF233
Mg	IP-PS-02-012	500 µg/mL	CPI	2AF233
Mn	IP-PS-02-012	200 µg/mL	CPI	2AF233
Mo	IP-PS-02-012	200 µg/mL	CPI	2AF233
Na	IP-PS-02-012	500 µg/mL	CPI	2AF233
Ni	IP-PS-02-012	500 µg/mL	CPI	2AF233
Pb	IP-PS-02-012	500 µg/mL	CPI	2AF233
Se	IP-PS-02-012	1000 µg/mL	CPI	2AF233
Sr	IP-PS-02-012	200 µg/mL	CPI	2AF233
Ti	IP-PS-02-012	200 µg/mL	CPI	2AF233
V	IP-PS-02-012	200 µg/mL	CPI	2AF233
Zn	IP-PS-02-012	200 µg/mL	CPI	2AF233
Zr	IP-PS-02-012	200 µg/mL	CPI	2AF233
B	IP-PS-02-026	10000 µg/mL	EM Science	B1065147
K	IP-PS-02-068	10000 µg/mL	EM Science	B2025030
P	IP-PS-02-035	10000 µg/mL	EM Science	B2025035
Si	IP-PS-02-026	10000 µg/mL	EM Science	B1125060
Sn	IP-PS-02-038	10000 µg/mL	EM Science	B0065078
Te	IP-PS-02-040	1000 µg/mL	EM Science	B1125041
Tl	IP-PS-02-046	10000 µg/mL	EM Science	A8075015
Ag	IP-PS-02-058	10000 µg/mL	EM Science	B2015115
Cd	IP-PS-02-028	10000 µg/mL	EM Science	B1025044
Sb	IP-PS-02-084	1000 µg/mL	EM Science	B1045121
Pt	IP-PS-02-123	10000 µg/mL	EM Science	B0125146

SAMPLE PREPARATION AND INSTRUMENT CONDITIONS

Sample Preparation

Calibrated personal sampling pumps equipped with MCE filters are used to monitor work place exposure conditions for airborne inorganic contaminants. The pumps are set at a flow rate of between 1 and 4 L/min. For a total sample volume see Table 3. Sample loading limits can be estimated from TWA values (see Table 4), instrument sensitivity, and sampling flow rate [2]. A filter loading of approximately 2 mg of dust should not be exceeded. Once collected the sample cassette filter holders are removed from the personal sampling pumps and submitted for ICP-AES analysis.

For this backup study, blank MCE filters were spiked with the elements of interest and transferred to clean PTFE digestion vessels. To each vessel was added 10 mL of 1:1 nitric acid and ASTM type II water. The vessels and their contents were then weighed and the weights recorded. The associated method blanks and spiked QC filters were also prepared in this same manner. The digestion vessels were then placed into a programmable microwave oven (CEM MDS-2100) and digested using the following conditions:

Stage	1	2	3	4	5
POWER	80%	80%	0%	0%	0%
PRESSURE	50	75	0	0	0
RUN TIME	10:00	20:00	5:00	0	0
TIME	5:00	15:00	0	0	0
TEMPERATURE	150 °C	150 °C	0 °C	0 °C	0 °C
FAN SPEED	100%	100%	100%	100%	100%
NUMBER OF VESSELS:	12				

After digestion was complete the vessels were allowed to cool and then they were re-weighed and the weights recorded. The lids were then removed and the contents of the vessels

were rinsed into 25 mL volumetric flasks with ASTM type II water and diluted to the mark. The samples were then submitted for ICP-AES analysis.

NOTE: The purpose for weighing the vessels before and after digestion was to determine if there had been any sample loss. If there had been significant loss the sample in question was discarded and a new sample prepared and digested. In the case of real world samples if the loss was greater than one half gram but less than three grams a note would be made in the lab records. If greater than three grams a note would be made in the analytical report. If a loss occurs there is the possibility that one or more of the analytes may experience some loss. Where the digestion ovens are working perfectly weighing of the samples is not necessary.

TABLE 3. PROPERTIES AND SAMPLING VOLUMES

	Properties [3]		Air Volume, L @ OSHA PEL [4]	
Element (Symbol)	Atomic 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)	137.3	727	5(2)	200(2)
Boron (B)(1)	10.81	2300	5	2000
Beryllium (Be)	9.01	1278	1250	2000
Calcium (Ca)(1)	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) (1)	39.10	63	5	2000
Lithium (Li)(1)	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
Sodium (Na)(1)	22.99	98	13	2000
Nickel (Ni)	58.71	1453	5	1000
Phosphorus (P)	30.97	44	25	2000
Lead (Pb)	207.19	328	50	2000
Platinum (Pt)	195.09	1769	1250	2000
Antimony (Sb)	121.76	630	10(2)	2000(2)
Selenium (Se)	78.96	217	13	2000
Strontium (Sr)(1)	87.62	769	5	2000
Tin (Sn)	118.69	232	20(2)	2000(2)
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
Yttrium (Y)	88.91	1495	5	1000
Zinc (Zn)	65.37	419	5	200
Zirconium (Zr)	91.22	1852	5	200

(1) No PEL, REL, STEL data found [5][6].
(2) Air Volumes Estimated from TWA and LOQs (see Tables 4 and 7) [2].

TABLE 4. EXPOSURE LIMITS, CAS #, RTECS [5][6]

Exposure Limits, mg/m^3 (Ca=carcinogen; C=ceiling limit; *not adopted due to adverse effects at this level

Element (Symbol)	CAS #	RTECS	OSHA	NIOSH	ACGIH
Silver (Ag)	7440-22-4	VW3500000	0.01 (soluble, metal)	0.01 (soluble, metal)	0.1 (metal) 0.01 (soluble)
Aluminum (Al)	7429-90-5	BD0330000	15 (total dust) 5 (respirable)	10 (total dust) 5 (respirable, fume) 2 (salt, alkyls)	10 (dust) 5 (powder, fume) 2 (salt, alkyls)
Arsenic (As)	7440-38-2	CG0525000	0.010 (inorganic)	C 0.002, Ca	0.01, Ca
Barium (Ba)	7440-39-3		0.5 [6]		
Boron (B)	7440-42-8				
Beryllium (Be)	7440-41-7	DS1750000	0.002, C 0.005	Not to exceed 0.0005, Ca	0.002, Ca
Calcium (Ca)	7440-70-2		Varies	Varies	varies
Cadmium (Cd)	7440-43-9	EU9800000	0.2, C 0.6 (dust) 0.1, C 0.3 (fume)	lowest feasible conc., Ca	0.01 (total), Ca 0.002 (respirable), Ca
Cobalt (Co)	7440-48-4	GF8750000	0.1	0.05 (dust, fume)	0.05 (dust, fume)
Chromium (II) (Cr)	22541-79-3	GB6260000	0.5	0.5	Not given
Chromium (III) (Cr)	16065-83-1	GB6261000	0.5	0.5	0.5
Chromium (VI) (Cr)	18540-29-9	GB6262000	C 0.1	0.001 (dust)	0.05 (soluble) 0.05 (insoluble), Ca
Copper (Cu)	7440-50-8	GL5325000	1 (dust, mists) 0.1 (fume)	1 (dust, mists) 0.1 (fume)	1 (dust, mists) 0.2 (fume)
Iron (Fe)	1309-37-1	NO7400000	10 (fume) as oxide	5 (dust, fume) as oxide	5 (fume) as oxide
Potassium (K)	7440-09-7				
Lithium (Li)	7439-93-2				
Magnesium (Mg)	1309-48-4	OM3850000	15 (dust) as oxide	10 (fume) as oxide*	10 (fume) as oxide
Manganese (Mn)	7439-96-5	OO9275000	C 5	1; STEL 3	0.2
Molybdenum (Mo)	7439-98-7	QA4680000	5 (soluble) 15 (total insoluble)	5 (soluble)* 10 (insoluble)*	5 (soluble) 10 (insoluble)
Sodium (Na)	7440-23-5				
Nickel (Ni)	7440-02-0	QR5950000	1	0.015, Ca	1.5 (metal) (soluble) 0.2 (insoluble), Ca
Phosphorus (P)	7723-14-0	TH3500000	0.1	0.1	
Lead (Pb)	7439-92-1	OF7525000	0.05	<0.1	0.15
Platinum (Pt)	7440-06-4	TP2160000	0.002	1 (metal)	1 (metal)
Antimony (Sb)	7440-36-0	CC4025000	0.5	0.5	
Selenium (Se)	7782-49-2	VS7700000	0.2	0.2	0.2
Strontium (Sr)	7440-24-6				
Tin (Sn)	7440-31-5	XP7320000	2	2	
Tellurium (Te)	13494-80-9	WY2625000	0.1	0.1	0.1
Titanium (Ti)	7440-32-6	XR1700000	As TiO_2, 15	lowest feasible, Ca	10
TiO_2	13463-67-7	XR2275000	as TiO_2, 5 (respirable)		
Thallium (Tl)	7440-28-0	XG3425000	0.1 (skin) (soluble)	0.1 (skin) (soluble)	0.1 (skin)
Vanadium (V)	7440-62-2	YW240000	C 0.5 (respirable) as V_2O_5	C 0.05	0.05 (respir.) as V_2O_5
V_2O_5	1314-62-1	YW1355000	C 0.1 (fume) as V_2O_5		
Yttrium (Y)	7440-65-5	ZG2980000	1	1	1
Zinc (Zn)	1314-13-2	ZH4810000	5 (ZnO fume) 15 (ZnO dust) 5 (ZnO respirable)	5; STEL 10 (ZnO fume) 5; C 15 (ZnO dust)	5; STEL 10 (ZnO fume) 10 (ZnO dust)
Zirconium (Zr)	7440-67-7	ZH7070000	5	5, STEL 10	5, STEL 10

Instrument Conditions and Calibration

The ICP spectrometer should be setup and calibrated according to the manufacturers recommendations. Typically acid blank and multi-element working standards are used for calibration. These two point curves cover the linear working range of the analytes of interest. Listed in Table 5 are the concentrations of the standards that make up the top end of the curves set up for this work.

The following multi-element combinations are chemically compatible in 20% HNO_3.

a. Al, As, Ba, Be, Ca, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Na, Ni, Pb, Se, Sr, Ti, V, Y, Zn, Zr;

b. B, K, P, Si, Sn, Te, Tl;

c. Ag, Cd, Sb;

d. Pt.

Continuing Calibration Verification Standard (CCV) or standards that contain analytes of interest are analyzed after every ten analyses (minimum frequency), and recoveries are checked with media blanks and spikes, every twenty samples.

TABLE 5. Calibration Standard Concentrations

Analyte	Primary ID#	Primary Conc.	Volume of Primary	Final Volume	Final Concentration
Al	IP-PS-02-012	1000 µg/mL	20 mL	2000 mL	10 µg/mL
As	IP-PS-02-012	500 µg/mL	20 mL	2000 mL	5 µg/mL
Ba	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Be	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Ca	IP-PS-02-012	1000 µg/mL	20 mL	2000 mL	10 µg/mL
Co	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Cr	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Cu	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Fe	IP-PS-02-012	500 µg/mL	20 mL	2000 mL	5 µg/mL
Li	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Mg	IP-PS-02-012	500 µg/mL	20 mL	2000 mL	5 µg/mL
Mn	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Mo	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Na	IP-PS-02-012	500 µg/mL	20 mL	2000 mL	5 µg/mL
Ni	IP-PS-02-012	500 µg/mL	20 mL	2000 mL	5 µg/mL
Pb	IP-PS-02-012	500 µg/mL	20 mL	2000 mL	5 µg/mL
Se	IP-PS-02-012	1000 µg/mL	20 mL	2000 mL	10 µg/mL
Sr	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Ti	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
V	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Zn	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
Zr	IP-PS-02-012	200 µg/mL	20 mL	2000 mL	2 µg/mL
B	IP-PS-02-026	10000 µg/mL	1 mL	1000 mL	10 µg/mL
K	IP-PS-02-068	10000 µg/mL	5 mL	1000 mL	50 µg/mL
P	IP-PS-02-035	10000 µg/mL	2 mL	1000 mL	20 µg/mL
Si	IP-PS-02-026	10000 µg/mL	5 mL	1000 mL	50 µg/mL
Sn	IP-PS-02-038	10000 µg/mL	1 mL	1000 mL	10 µg/mL
Te	IP-PS-02-040	1000 µg/mL	10 mL	1000 mL	10 µg/mL
Tl	IP-PS-02-046	10000 µg/mL	2 mL	1000 mL	20 µg/mL
Ag	IP-PS-02-058	10000 µg/mL	0.2 mL	1000 mL	2 µg/mL
Cd	IP-PS-02-028	10000 µg/mL	0.2 mL	1000 mL	2 µg/mL
Sb	IP-PS-02-084	1000 µg/mL	10 mL	1000 mL	10 µg/mL
Pt	IP-PS-02-123	10000 µg/mL	2 mL	2000 mL	10 µg/mL

LIMITS OF DETECTION

The limits of detection (LOD) and quantitation (LOQ) are determined according to the protocol given in the Environmental Protection Agency's 40 CFR [7] in the which seven or more duplicates within the range of 1#TC/MDL#5 are analyzed and calculated (see Table 6).

Where:
TC = test concentration
MDL = the method detection limit or LOD

The wavelength at which an analyte is measured is generally the most sensitive available. Where there are spectral interferences from other elements in a sample, it may be necessary to use an alternate wavelength. With a fixed wavelength instrument this is not always possible. In this study two different ICP'S were used. Most of the data for this study was generated using a fixed channel Fisons ARL Accuris ICP-AES. For those analytes were there were possible interference problems on the Fisons, a Perkin Elmer Optima 3000 DV ICP-AES was employed (see Table 6).

The duplicates for this study were prepared following the procedure in the sample preparation section. They were processed through the sample digestion procedure along with filter blanks and quality control (QC) samples.

From the LODs the LOQs for each of the analyts are calculated using the formula:

LOQ= 3.3X LOD.

TABLE 6. WAVELENGTHS AND DETECTON LIMITS

Element[(a)]	Wavelength (nm)[8]	LOD (µg/sample)
Ag	328.1	0.1
Al	308.2	1
As	193.8	1
Ba	493.4	0.06
B	249.7	0.5
Be	313.0	0.009
Ca	315.9	2
Cd	228.8	0.1
Co	228.6	0.3
Cr[(b)]	267.7	0.4
Cu	324.8	0.07
Fe	259.9	2
K	766.5	2
Li	670.8	0.03
Mg	279.1	0.5
Mn	257.6	0.02
Mo	202.0	0.2
Na	589.0	4
Ni	231.6	0.2
P	214.9	2
Pb	220.4	0.6
Pt[(b)]	265.9	8
Sb	206.8	0.4
Se	196.1	3
Sn[(b)]	189.9	0.8
Sr	421.6	0.02
Te[(b)]	214.3	2
Ti	337.3	0.2
Tl	190.9	0.9
V	292.4	0.1
Y[(b)]	371.0	0.02
Zn[(b)]	213.9	0.1
Zr	339.2	0.06

(a) Unless otherwise specified all reported values were obtained with a Fisons ARL Accuris ICP-AES; performance may vary with instrument and should be independently verified.

(b) Values reported were obtained with a Perkin Elmer (PE) Optima 3000 DV ICP-AES. Sample concentration was based on Fisons ICP-AES LOD data.

PRECISION ACCURACY AND QUANTITATION STUDY

To determine the precision and accuracy of this method, six concentrations ranging from 1X LOQ to 300X LOQ for each element were studied. For each level, six blank filters were spiked with certified standards and digested following the procedure outlined in the section on sample preparation. After digestion the samples were analyzed, the raw data collected, and precision and accuracy calculated (see Tables 7 and 8).

The acceptable NIOSH recovery range is from 75% to 125%. The percent recoveries at the 1X LOQ level, range from 65.6% to 127% compared to 92.4% to 112% for the 3X LOQ level. Thus the range of quantitation is in general 3X LOD and above.

TABLE 7. PRECISION AND ACCURACY

Element[a]	1X LOQ (µg/sample)	% Recovery (N=6)	% RSD	3X LOQ (µg/sample)	% Recovery (N=6)	% RSD	10X LOQ (µg/sample)	% Recovery (N=6)	% RSD
Ag	0.495	84.6	10.3	1.50	95.5	1.01	4.95	101	1.07
Al	2.48	74.5	40.5	7.50	92.7	0.981	24.8	82.9	3.87
As	2.48	100	10.7	7.50	101	2.22	24.8	104	2.12
Ba	0.248	111	8.59	0.752	104	3.09	2.48	102	0.664
B	1.24	127	6.19	3.75	112	2.96	12.4	100	0.618
Be	0.025	89.2	7.15	0.076	95.8	2.36	0.251	99.6	0.995
Ca	7.43	134	2.94	22.5	107	2.87	74.3	110	0.701
Cd	0.495	94.6	7.30	1.50	98.8	3.46	4.95	105	0.746
Co	1.24	94.9	8.71	3.75	99.7	1.72	12.4	103	0.403
Cr[b]	1.24	109	7.97	3.75	103	7.87	12.4	82.4	1.13
Cu	0.248	85.8	12.9	0.752	98.8	3.47	2.48	102	2.10
Fe	4.95	123	10.4	15.0	112	2.43	49.5	90.1	2.97
K	4.95	86.8	23.0	15.0	98.3	5.70	49.5	98.6	2.86
Li	0.248	80.4	4.78	0.752	92.4	2.98	2.48	93.7	1.05
Mg	2.48	81.9	12.9	7.50	89.3	3.52	24.8	101	1.65
Mn	0.248	72.1	4.78	0.752	86.2	2.38	2.48	104	0.555
Mo	0.743	99.8	7.59	2.25	96.8	5.41	7.43	111	0.874
Na	12.4	110	5.72	37.5	100	0.823	124	98.7	1.94
Ni	0.743	72.6	16.8	2.25	98.3	5.21	7.43	87.9	5.93
P	4.95	96.8	6.62	15.0	100	5.67	49.5	100	0.974
Pb	2.48	112	7.66	7.50	98.9	3.94	24.8	98.7	1.00
Pt[b]	25.0	99.2	1.43	75.0	98.3	0.282	250	99.1	2.63
Sb	2.48	105	5.08	7.50	94.4	3.21	24.8	103	1.01
Se	12.4	98.9	4.80	37.5	104	3.21	124	88.1	3.83
Sn[b]	12.4	110	4.62	37.5	105	5.04	124	103	2.51
Sr	1.24	101	1.48	3.75	92.6	2.36	12.4	101	0.393
Te[b]	4.95	114	4.83	15.0	90.1	21.8	49.5	106	1.87
Ti	0.495	101	8.18	1.50	101	1.70	4.95	99.1	0.625
Tl	2.48	93.6	7.52	7.5	103	4.14	24.8	104	1.85
V	0.248	65.6	22.0	0.752	93.7	4.74	2.48	100	1.92
Y[b]	0.124	111	5.28	0.376	107	4.44	1.24	88.3	2.23
Zn[b]	0.495	105	17.5	1.50	106	13.1	4.95	86.0	1.41
Zr	0.248	74.1	19.3	0.750	93.1	5.35	2.48	98.6	1.95

(a) Unless otherwise specified all reported values were obtained with a Fisons ARL Accuris ICP-AES; performance may vary with instrument and should be independently verified.
(b) Values reported were obtained with a Perkin Elmer Optima 3000 DV ICP-AES. Sample concentration was based on Fisons ICP-AES LOD data.

TABLE 8. PRECISION AND ACCURACY

Element[a]	30X LOQ (µg/sample)	% Recovery (N=6)	% RSD	100X LOQ (µg/sample)	% Recovery (N=6)	% RSD	300X LOQ (µg/sample)	% Recovery (N=6)	% RSD
Ag	15.0	99.9	1.01	50.0	99.1	0.593	150	99.0	0.497
Al	75.0	102	0.295	250	101	0.474	750	98.7	0.462
As	75.0	101	0.754	250	104	1.16	750	107	0.340
Ba	7.52	100	0.297	25.0	102	0.839	75.2	101	0.438
B	37.5	101	0.813	125	99.7	0.964	375	99.5	0.454
Be	0.760	96.9	0.448	2.53	101	1.24	7.60	103	0.714
Ca	225	100	0.395	749	103	1.08	2250	104	0.620
Cd	15.0	100	1.01	50.0	103	1.43	150	104	0.701
Co	37.5	99.9	0.455	125	102	1.04	375	103	0.566
Cr[b]	37.5	99.7	1.43	125	96.6	1.59	375	94.2	3.36
Cu	7.52	103	0.518	25.0	103	0.628	75.2	101	0.371
Fe	150	102	0.331	500	104	1.35	1500	103	0.263
K	150	101	0.631	500	100	0.438	1500	98.8	0.472
Li	7.52	100	0.528	25.0	99.8	0.744	75.2	95.1	0.749
Mg	75.0	99.7	0.296	250	100	0.674	750	98.2	0.309
Mn	7.52	101	0.313	25.0	104	0.974	75.2	103	0.389
Mo	22.5	105	0.461	75.0	109	1.06	225	110	0.373
Na	375	99.7	0.766	1250	101	0.561	3750	97.7	0.457
Ni	22.5	103	0.315	75.0	104	1.12	225	104	0.592
P	150	99.3	0.609	500	101	1.83	1500	104	0.315
Pb	75.0	93.7	0.283	250	95.1	1.02	750	95.7	0.570
Pt[b]	750	94.1	0.985	3330	93.6	1.22	10000	95.7	1.49
Sb	75.0	101	0.446	250	102	0.913	750	103	0.255
Se	375	102	0.483	1250	104	1.07	3750	106	0.270
Sn[b]	375	101	1.04	1250	96.6	1.81	3750	90.3	3.23
Sr	37.5	97.1	0.430	125	99.5	0.689	375	97.5	0.553
Te[b]	150	107	0.914	500	105	1.44	1500	103	0.614
Ti	15.0	96.7	0.249	50.0	99.0	0.888	150	98.8	0.575
Tl	75.0	97.6	0.395	250	99.4	1.31	750	99.3	0.352
V	7.52	101	0.700	25.0	103	0.966	75.2	103	0.341
Y[b]	3.76	105	1.23	12.5	103	1.60	37.6	102	3.33
Zn[b]	15.0	101	1.66	50.0	98.9	1.09	150	97.4	3.42
Zr	7.50	95.3	1.20	25.0	95.9	0.233	75.0	95.4	0.971

(a) Unless otherwise specified all reported values were obtained with a Fisons ARL Accuris ICP-AES; performance may vary with instrument and should be independently verified.
(b) Values reported were obtained with a Perkin Elmer Optima 3000 DV ICP-AES. Sample concentration was based on Fisons ICP-AES LOD data.

Media Background

Media blank results for MCE filters showed no background greater than the LOQ of any of the elements. Thus, subtraction of the media blank from the spiked filter results was not found to be necessary (see Table 9). The LOQs listed in Table 9 are calculated from the LODs of the Fisons ICP-AES (see section on Limits of Detection and Quantitation Study) and approximate the 1X LOQs found in Tables 7 and 8.

TABLE 9. MEDIA BACKGROUND RESULTS

Element[a]	Average (N=6) (µg/sample)	LOQ (µg/sample)
Ag	-0.0838	0.4
Al	-0.7439	4
As	-0.1216	3
Ba	0.3529	0.2
B	0.0325	2
Be	-0.0025	0.03
Ca	2.4224	6
Cd	-0.0505	0.4
Co	-0.2234	1
Cr[b]	0.7468	0.8
Cu	0.0149	0.2
Fe	1.2912	7
K	-0.8592	8
Li	-0.0514	0.1
Mg	-0.4986	2
Mn	-0.0831	0.07
Mo	-0.0912	0.7
Na	0.4418	10
Ni	-0.2431	0.5
P	-0.1945	7
Pb	0.3096	2
Pt[b]	-0.0215	25
Sb	0.0840	1
Se	-0.1967	10
Sn[b]	0.0652	3
Sr	-0.0171	0.06
Te[b]	0.0228	1
Ti	0.0242	0.7
Tl	-0.6471	3
V	-0.0827	0.4
Y[b]	-0.0019	0.007
Zn[b]	0.1618	0.4
Zr	0.0147	0.2

(a) Values and LOQs reported were obtained with a Fisons ARL Accuris ICP-AES; performance may vary with instrument and should be independently verified.
(b) Values and LOQs reported were obtained with a Perkin Elmer Optima 3000 DV ICP-AES.

SUMMARY

This method is for the analysis of metal and nonmetal dust collected on MCE filters in the work place and environment in general. This method increases the applicability of NMAM 7300 [9] for trace elements and the simultaneous elemental analysis. Using a microwave digestion approach simplifies and expedites the analysis. The elimination of perchloric acid [10] in the sample digestion helps to improve the safety of the method.

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