

METHOD 4A

DETERMINATION OF LEAD COLLECTED ON PARTICULATE FILTERS BY MICROWAVE EXTRACTION AND ANALYSIS BY ATOMIC ABSORPTION SPECTROMETRY

REF: Reg. 11-1-301
11-1-302
11-1-303

1. PRINCIPLE

- 1.1 This method is applicable to the determination of particulate lead collected on 8 inch by 10 inch or other sized filter media using high volume, Size Selection Inlet (SSI) and other particulate air samplers.
- 1.2 One quarter or the whole collected filter sample is extracted with nitric acid in a closed vessel using pressure controlled microwave heating to extract the lead.
- 1.3 The acid extracts are then diluted with distilled water, filtered and the solutions analyzed for lead by flame atomic absorption spectroscopy.
- 1.4 This method may be applicable to other types of samples containing nitric acid soluble metals.

2. APPARATUS

2.1 CEM Model MDS-2000 Microwave Sample Preparation System, CEM Corp., Matthews, North Carolina.

2.1.1 Two sets of 12 Advanced Composite Digestion Vessels and ancillary supplies are used in conjunction with the Microwave Digestion System (MDS).

CAUTION: Never install an MDS-2000 system inside a laboratory fume hood. Acid and chemical fumes may attack electrical components resulting in damage and malfunctioning of the door safety interlocks.

2.1.2a Program for filters with low organic content

STAGE 1

Power	85%
PSI	0040
Time	35:00 Min.
TAP*	30:00 Min.
Temp.	140° C

Fan Power 100%

* TAP = Time at set parameters of pressure or temperature whichever is achieved first.

2.1.2b Program for filters with high organic content

	<u>STAGE 1</u>	<u>STAGE 2</u>
Power	85%	85%
PSI	0040	0065
Time	35:00 Min.	25:00 Min.
TAP*	30:00 Min.	30:00 Min.
Temp.	140° C	165° C
Fan Power	100%	100%

* TAP = Time at set parameters of pressure or temperature whichever is achieved first.

2.2 Atomic Absorption Spectrophotometer

2.2.1 Instrument Operating Parameters

2.2.1a Program for lead analysis

Lamp current	5 ma
Slit Width	1.0 nm
Slit Height	normal
Wavelength	217.0 nm
Flame	Air-Acetylene
Replicates	2
Measurement Time	1.0 sec
Delay Time	0 sec
Background Correction	ON

2.3 Lead Hollow Cathode Lamp.

2.4 Automatic Dispensing Pipettes, Brinkman Adjustable Dispensers, 5-25 ml and 10-50 ml size or equivalent.

2.5 Conical Centrifuge Tubes, 50 ml with screw caps, VWR # 21008-725.

2.6 Folded Filter Paper, Whatman # 2V 12.5 cm, VWR # 28420-024.

2.7 Funnel, Polypropylene, 65 mm top diameter.

2.8 Test Tube Racks, Polypropylene, VWR # 60985-288

2.9 Volumetric Flask, 100 ml

2.10 Paper cutter, 9 in x 10 in

3. REAGENTS

- 3.1 Lead Atomic Absorption Standard, 1000 ppm.VWR # VW4213-1 or equivalent.
- 3.2 Concentrated Nitric Acid (69 to 70%)
- 3.3 Acetylene
- 3.4 Compressed Air

CAUTION : Concentrated nitric acid is corrosive and the fumes are toxic. Handle samples containing the acid in a well ventilated fume hood . Wear rubber gloves and eye protection.

4. ANALYTICAL PROCEDURE

- 4.1 Wear rubber gloves when handling Particulate Filters. Cut the filters into quarters using a paper cutter.
- 4.2 Roll a quarter filter sheet of an 8 x 10 inch filter along the short edge with the exposed surface of the filter in the inside of the roll or the full circle of a 3.5 inch filter. Insert the rolled filter into an Advanced Composite Digestion Vessel liner with the white unexposed border of the filter at the bottom of the digestion vessel. This area of the vessel is poorly extracted during the microwaving process.
- 4.3 Fill the microwave turntable with 12 Advanced Composite Digestion Vessel liners containing the quarter filters. Place the turntable in a fume hood and add 12 ml of concentrated nitric acid from an automatic dispenser to each vessel.
- 4.4 Assemble the Advanced Composite Vessels according to procedures in the instrument manual. Check each part to make sure that they are tight and leakproof, especially the gray vent fitting that holds the rupture membrane, before microwaving each batch of 12 samples.

CAUTION: Be sure all of the parts of the digestion vessels are dry before microwaving the samples. Moisture on the outer surfaces of the vessel liners will cause hot spots that can damage the liners.

- 4.5 Install a pressure/temperature control cover on the vessel located in the number 12 position of the turntable and incline and rotate each filled vessel so that the quarter filter is completely wetted with digestion acid.
- 4.6 Put the filled turntable in the microwave oven with the number 12 position in front of the door.

- 4.7 Turn the handle of the two way valve of the pressure sensing system, located on the left side of the instrument, counterclockwise to the horizontal (open) position.
- 4.8 Attach the microwave transparent valve to the pressure sensing line with its' valve open. (The orange colored tear-drop shaped top is removed) Flush the line with distilled water using the water filled syringe. Collect the water in a beaker and discard.
- 4.9 Return the handle of the two way valve of the pressure sensing system to the vertical (neutral) position.
- 4.10 Connect the microwave transparent valve to the pressure sensing fitting on the temperature/pressure control cover.
- 4.11 Carefully pull the fiberoptic temperature sensing probe from its storage area and insert the free end into the top of the thermowell until the free end reaches the bottom of the thermowell.

CAUTION: Handle the fiberoptic probe very carefully. Do not force the probe or place any strain or sharp bends on the fiberoptic probe. The probe is flexible but is subject to breakage.
- 4.12 Turn the slotted retaining ring to the open position. The ring is located on the top of the vertical standoff at the center of the turntable. Insert the pressure sensing line and the fiberoptic probe in the standoff and close the slotted retaining ring.
- 4.13 With the microwave door open, press F4 to rotate the turntable. Make sure that the fiber optic probe does not become entangled with the pressure sensing tube or pull out of the vessel thermowell. Make sure that there is no loose pressure sensing tubing to interfere with the microwave deflector blades.
- 4.14 Close the microwave door and start the microwave program by following the steps as outlined in the operation manual.
- 4.15 Prepare the second set of digestion vessels while the first set is processing.

CAUTION: On short sample runs when less than 12 filter samples are processed, always monitor the run with a filter sample in the No. 12 position where the temperature/pressure control cover can monitor the sample. Do not monitor a vessel containing only nitric acid as the vessels containing the filters will become over-pressurized and overheated.
- 4.16 When the microwave completes the digestion program on the set of samples, let the contents of the oven cool for about 10 minutes.

- 4.17 Open the slotted retaining ring and release the pressure sensing line and fiberoptic probe.
- 4.18 Push the fiberoptic probe into the open port located in the cavity of the oven for safe storage.
- 4.19 Reattach the orange colored tear-drop top to the microwave transparent valve and close until finger tight.
- 4.20 Disconnect the pressure sensing line leaving the closed microwave transparent valve attached to the digestion vessel.
- 4.21 Carefully remove the hot turntable and put it in the hood for cooling.
- 4.22 When the vessels are cooled to room temperature, carefully open the vessels and add 38 ml of distilled water to the contents of the vessels. Mix the diluted acid extract by manually shaking the closed vessels at least 10 times.
- 4.23 Filter each sample through a dry Whatman No. 2V folded filter paper into a 50 ml conical centrifuge tube with screw cap which is used to store the samples for analysis.
- 4.24 Analyze each extract for lead using the atomic absorption spectrophotometer.

5. STANDARD CURVE

- 5.1 Working Standard Lead Solution. Prepare the working standard by diluting 10 ml of the 1000 ppm lead atomic absorption standard to 100 ml with 10% nitric acid in a 100 ml volumetric flask. This solution contains 100 µg Pb/ml.
- 5.2 Prepare 6 individual calibration standards in 100 ml volumetric flasks in the range of 0, 0.5, 1, 2, 3, 5 ppm Pb by diluting the working standard lead solution.
- 5.3 Set the atomic absorption spectrophotometer parameters to obtain maximum sensitivity at a wave length of 217.0 nm.
- 5.4 Aspirate each standard to obtain the lead standard calibration curve.
- 5.5 Aspirate each sample and blank together with a standard periodically to insure that instrument response has not changed during the analysis of the samples.

6. CALCULATIONS

6.1 Total $\mu\text{g Pb} = \text{ppm Pb} \times 50$

6.2 $\mu\text{g Pb}/\text{M}^3 = \frac{\text{ppm Pb} \times 200}{\text{Air volume M}^3}$

where 200 = 50 ml sample extract x 4 quarter sheets of 8 x 10 inch filter media.

7. REFERENCE

- 7.1 Operation Manual, Microwave Sample Preparation System MDS-2000, CEM Corp., Matthews, North Carolina, 1994.
- 7.2 AIHL Method No. 54, Analysis for Lead Content of Atmospheric Particulate Matter Collected on High-Volume Glass Fiber Filters, AIHL, Berkeley Ca. 1974
- 7.3 Analytical Methods for Flame Spectroscopy, Varian Corp. Publication No. 85-100009-00 1979