GUIDELINES ON METHOD OF SAMPLING AND ANALYSIS FOR AIRBORNE LEAD

FACTORIES AND MACHINERY (LEAD) REGULATIONS, 1984
(REGULATION 15)

DEPARTMENT OF OCCUPATIONAL SAFETY AND HEALTH
MINISTRY OF HUMAN RESOURCES
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PREFACE

The purpose of these guidelines is to supplement the requirement of regulation 15 of the Factories and Machinery (Lead) Regulations 1984, specifying a method of monitoring and analysis of accuracy, to a confidence level of 95%, within a margin of plus or minus 20%, for airborne concentrations of lead equal to or greater than seventy five micrograms per cubic metre (75 µgm/ m^3) of air. An employer or an occupier who has an industrial process within his place of work which uses or handles inorganic lead, and in which there exists potential that any employee may be exposed to airborne lead during work, is advised to refer to these guidelines in complying with the employee exposure monitoring requirements as stipulated in Part III of the above Regulations. An employer or an occupier may use any other method of monitoring and analysis, but the method used must be proven to be on a par with or better than the method recommended in these guidelines.

The Department of Occupational Safety and Health also administratively screens:

a) person who wish to conduct exposure monitoring for airborne lead; and

b) laboratories which wish to conduct analysis of airborne lead monitoring samples;

in compliance with the above Regulations and approves those found to satisfy the requirements of the Department. These approved persons and approved laboratories are expected to be familiar with the method of monitoring and analysis recommended in these guidelines, as appropriate to their specific area of activity, and to use the method for compliance purposes.

Director General
Department of Occupational Safety and Health
Malaysia

April 1997

GUIDELINES ON METHOD OF SAMPLING FOR AIRBORNE LEAD

FACTORIES AND MACHINERY (LEAD) REGULATIONS, 1984
(REGULATION 15)

1. GENERAL INFORMATION

1.1. Worker Breathing Zone

This is defined as a hemisphere of 300 mm radius extending in front of the face and measured from a line bisecting the ears.

1.2. Personal Samples

In order to estimate worker exposure, samples must be taken in the worker's breathing zone and be obtained under typical working conditions, preferably over the full shift. Sampling procedures should aim at minimal interference with the activities of the worker.

2. PRINCIPLE

A measured volume of air is drawn through a membrane filter mounted on a filter holder, and lead contaminant collected will be sent to the analytical laboratory for lead analysis.

2.1 Apparatus

2.1.1. Sampling System

The apparatus needed for personal sampling are a sampling pump, a three-piece cassette and cassette holder, a pump's calibrator, a silicon tubing and cellulose ester membrane filter. The essential features of all sampling systems are the filter (on which the sample is collected) and the pump for pulling the air through it. An airflow indicating device may be provided. The pump unit must be capable of maintaining a smooth flow of the specified rate throughout the sampling period. The permitted error limits of flow may be achieved either by using a flow stabilised pump or by sufficiently frequent adjustment of the flowrate.

2.1.2. Personal Sampling For Lead In Air

The personal sampler for lead in air consist of a three-piece cassette of 37 mm diameter (see fig.1) with the male section removed during sampling and a pump unit capable of maintaining a smooth flow rate of 2.0 :t. 0.1 litre/min. throughout the sampling period.

Membrane filters made of cellulose ester with 0.8 micrometre pore size or equivalent must be used. The size of the filter should be 37 mm in diameter.

2.1.3. Ancillary Equipment

Cellulose bands for filter holders
Bubble-meter to calibrate pump flow rate
Stop watch
Thermometer to measure air temperature
Tweezers for handling filters

THREE PIECE CASSETTE HOLDER

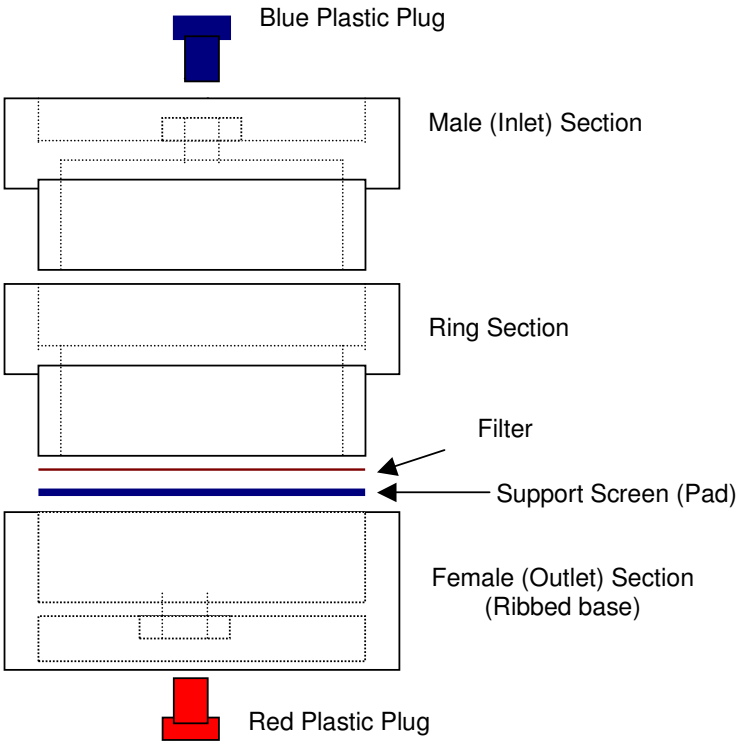

FIGURE 1

2.1.4 Calibration of pump flow rate

Before calibration, make sure the pump is fully charged and in good order. Set up the apparatus according to the arrangement as shown in Fig.2. The bubble meter uses the principle of cylindrical air displacement with nearly 'frictionless' piston. The interior surface is wetted with detergent solution to make it 'frictionless'. A soap-film bubble is placed and suction is provided by the pump to be calibrated. The bubble will be drawn up the cylinder. The column displacement per unit time can be determined by measuring the time required f or the bubble to pass between two scale marking which enclose a known volume.

By adjusting the flow rate of the pump, the required flowrate as derived from the bubble meter can be found. Mark the position of the float on the rotameter incorporated on the pump. The temperature of the room and the barometric pressure must be recorded during the calibration. After sampling, the pump flowrate has to be rechecked to calibrated flowrate. If the error is more then 10%, the sample must be rejected.

2.2 Sampling

Personal samples are taken using a fully charged battery operated portable sampling pump fitted with an air flow smoothing device.

Approximately fifteen minutes before sampling is to begin connect the pump to the sampler (with filter). Let the pump run of fifteen minutes and adjust the float on the rotameter (by adjusting the flow rate) to the marked position i.e the calibrated flow rate. Then replace the sampler with another sampler containing a clean filter. Readjust the float to its correct position. Experience may show that this warm-up period is unnecessary for some types of stabilised flow pumps.

Place the sampling equipment assembly on the selection person. The wearer of the equipment should be provided with a suitable belt or harness to with the pump can be conveniently fixed, unless the pump is small enough to fit into the pocket. Attach the sampler or sampling head to the operator, preferably on his lapel, not more than 30 cm away from the nose-mouth region.

When ready to commence sampling, turn on the pump and record the following information:

a. Starting time
b. Filter number or cassette number
c. Pump identification number
d. Pump start time and date
e. Flow rate
f. Worker's name
g. Type of operation
h. Ventilation control and atmospheric conditions, where relevant
i. Temperature (every one hour); and
j. Ending time

CALIBRATION ASSEMBLY FOR TOTAL DUST SAMPLING

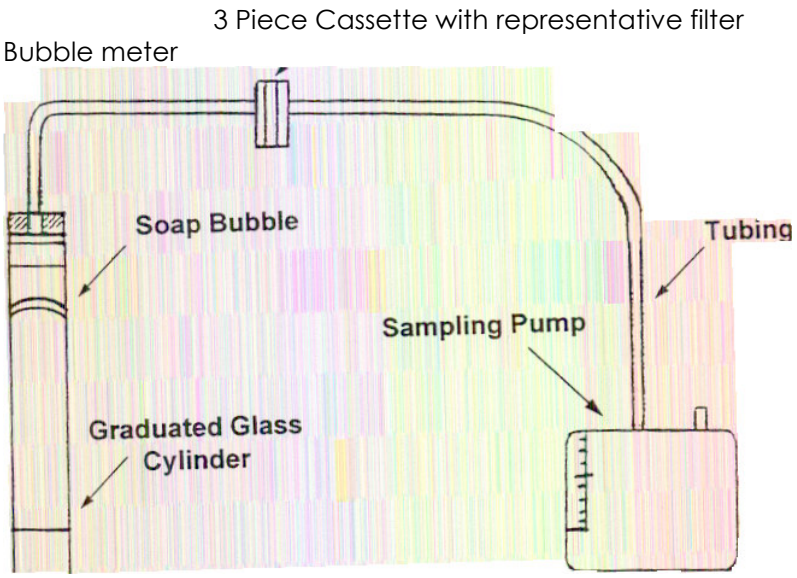

FIGURE 2

If a pump unit without stabilized flow is used, check, record and readjust the flow rate as necessary at the end of each hour. At the end of the sampling period note the time switch off the pump and remove the cassette.

2.3 End of Sampling

Measure the pump flow rate at the end of sampling before switching the pump off-a later check may give false results due to the temporary recovery of a discharged battery. Switch off the pump and record the time. Remove the cassette and immediately place it in a dust free box with the contaminated side facing upwards, ensuring that it cannot be inadvertently used again.

3. CARE OF SAMPLING EQUIPMENT

3.1 Filter Holder

Before sampling, check that the filter holder is clean and dry. Ensure that the filter is properly place in the filter holder. Check properly that the filter holder halves are held together finger tight-too loose will cause a leak, whilst too tight may cause damage to the membrane filter.

3.2 Connecting Tubes

The ends of the connecting tubes will become stretched after repeated use and may leak or become detached during use. They must be examined before each use and renewed if too loose, or if leakage is suspected.

3.3 Filter

After sampling the sample must be ensured that it is tamper free. The sample must be discarded if there are evidence suggesting it has been tampered with e.g finger print mark on the filter, excessive loose dust in the filter holder, etc.

3.4 Packing of the Samples

Disconnect the filter after sampling. Cap the inlet and outlet of the filter holder with plugs. Label the sample .Record pertinent sampling data including times of beginning and end of sampling, initial and final air temperatures, relative humidity and atmospheric pressure or elevation above sea level. Record the type of personal sampling pump used and location of sampler.

3.5 Transportation of Samples to the Laboratory

Ship the samples to the laboratory as soon as possible in a suitable container designed to prevent damage in transit. Ship bulk material to the laboratory in a glass container with a PTFE-lined cap. Never store, transport or mail the bulk sample in the same container as the samples from the same lot for use as media blanks.

4. PRECISION AND ACCURACY

In this method the term 'accuracy' is used to describe the difference between a measured concentration and the true concentration of the sample. Accuracy has two components: precision and bias. Precision is a measure of variability of method about its own mean and is usually expressed as a standard deviation (absolute value) or coeffient of variation (CV% of standard deviation relative to the mean). Bias is the difference between the average result and the true value from the determination. The accuracy of this method is $\pm$ 17.6 %

5. REFERENCES

i) NIOSH Manual of Analytical Methods (NMAM)
U.S Department Of Health , Education And Welfare
National Institute of Occupational Safety and Health
Fourth Edition, 1994
U.S.A;

ii) Occupational Exposure Sampling Strategy Manual
U.S Department of Health, Education and Welfare
National Institute of Occupational Safety and Health, 1997
U.S.A ; and

iii) Approved Method of Monitoring and Analysis for Mineral Dust
Department of Occupational Safety and Health,
Malaysia.

GUIDELINES ON METHOD OF ANALYSIS FOR AIRBORE LEAD

NIOSH Manual of Analytical Method (NMAM)
Fourth Edition, 1994

LEAD by Flame AAS 7082

Pb MW: 207.19 (Pb) 223.19 (Pbc)	CAS: 7439-92-1 (Pb) 1317-36-8 (Pbc)	ATECS: OF7525000 (Pb) OG1750000 (Pbc)
METHOD: 7082, Issue 2	EVALUATION : FULL	Is-sue 1: 15 February 1984 Issue 2: 15 August 1984

OSHA : 0.05 mg/m3
NIOSH: < 0.1 mg/m3: blood Pb < 60 ug/100 g,
ACGIH: 0.15 mg/m3 ,

PROPERTIES: soft metal:
d 11.3 glcmJ : MP 327.5 °C
valences -2.-4 in salts

SYNONYMS: elemental lead and lead compounds except alkyl lead

SAMPLING

SAMPLER: FIL TER
(0.8-um cellulose ester membrane)

FLOW RATE : 1 to 4 Umin
VOL- MIN. 200 L @ 0.05 mg/mJ
MAX: 1500 L
SHIPMENT: routine
SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.13 to 0.14 mg/mJ[1],
0.15 to 1.7 mg/mJ (fume) [2]
BIAS: -3.1%
OVERALL PRECISION (S): 0.072 [1],
0.068 (fume) [2]
ACCURACY: = 17.5%

MEASUREMENT

TECHNIQUE: ATOMIC ABSORPTION
SPECTROPHOTOMETER FLAME

ANAL YTE: lead
ASHING: conc. HNOJ, 5ml -30% H2O2 -
1 ml, 140 °C
FINAL SOLUTION: 10% HNOJ, 10 mi
FLAME: air-acetylene, oxidizing .

WAVELENGTH: 283.3 nm

BACKGROUND
CORRECTION: 02 or H2 lamp or

CALIBRATION: Pbl in 10% HNOJ
RANGE: 10 to 200 ug per sample [4]
PRECISION (S): 0.03 [1]

APPLICABILITY: The working range is 0.05 to > 1 mg/m3 for a 200-L air sample The method is applicable to elemental lead. including Pb fume. and all other aerosols containing lead. This is an elemental analysis not compound specific. Aliquots of samples can be analyzed separately for additional elements.

INTERFERENCES: Use O2 or H2 continuum or Zeeman background correction to control flame or molecular absorption. High concentrations of calcium, sulfate, carbonate, phosonate, iodide, fluoride, or acetate can be corrected-

OTHER METHODS: This method combines and replaces P&CAM 173[3] and S341 [4,5] for lead. Method 7300 (ICP-AES) and 7105 (AAS/GF) are alternate analytical methods. Method 7505 is specific for lead sulfide. The following have not been revised- the dithizone method, which appears in P&CAM 102 [5] and the lead criteria document [6]; and P&CAM 191 (ASV) [7].

REAGENTS:

1. Nitric acid, conc.* .
2Nitric acid, 10% (v/v). Add 100 mi conc. HNOJ 500 mL water; dilute to 1 L..
3. Hydrogen peroxide. 30% H2O2 (w/w) reagent grade. .
4. Calibration stock solution. 1000 ug/mL Pb Commercial standard or dissolve 1.00 g Pb metal in minimum volume of (1-1)HCIand dilute bottle. Stable > one year. 1 L with 1% (v/v) HCI. Store in a polyethylene
5. Air. compressed, filtered.
6. Acetylene
7. Distilled or deionized water.

See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: Cellulose ester filter. 0.8 – *um*pore size. 37- mm diameter in cassette filter holder
2. Personal sampling pump. 1 to 4 *Umin.* with flexible connecting tubing.
3. Atomic Absorption Spectrophotometer with an air-acetylene burner head and background. correction.
4. Lead hollow cathode lamp or electrode dischargeless lamp.
5. Regulators. two-stage, for air and acetylene.
6. Beakers. Phillips. 125-mL or Griffin, 50- mL with watch glass covers.
7. Volumetric flasks. 10- and 100 -mL. **
8. Assorted volumetric pipets as needed.
9. Hotplate. surface temperature 140 °C.
10. Bottles. polyethylene. 100- mL.

Clean all glassware with conc. nitric acid and rinse thoroughly with distilled or deionized water before use.

SPECIAL PRECAUTIONS: Conc. nitric acid is an irritant and may bum skin. Perform all acid digestions in a fume hood. Hydrogen peroxide is a strong oxidizing agent, a strong irritant, and corrosive to the skin. Wear gloves and eye protection.

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 up to 8 h for a total sample size of 200 to 1500 L for TWA measurements. Do not exceed a filter loading of ea. 2 mg total dust.

SAMPLE PREPARATION:

NOTE 1: The following sample preparation gave quantitative recovery (see EVALUATION OF METHOD) [4]. Steps 4 through 9 of Method 7300 or other quantitative ashing techniques may be substituted, especially if several metals are to be determined on a single filter.

NOTE 2: The Appendix gives a microwave digestion procedure which may be necessary for complete recovery of lead from some matrices, especially epoxy-based paint.

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.

4. Add 3 mL. conc. HNOJ, and 1 mL 30% H2O2 and cover with a watch glass. Start reagent blanks at this step.
NOTE: If PbO2 is not present in the sample, the 30% H2O2 need not be added [2,4].

5. Heat on 140 °C hotplate until volume is reduced to about 0.5 mL.

6. Repeat two more times using 2 mL conc. HNOJ and 1 mL 30% H2O2 each time.

7. Heat on 140 °C hotplate until ca. 0.5 mL liquid remains.

8. When sample is dry, rinse the watch glass and walls of the beaker with 3 to 5 mL 10% HNOJ. Allow the solution to evaporate to dryness.

9. Cool each beaker and dissolve the residues in 1 mL conc. HNOJ.

10. Transfer the solution quantitatively to a 10-mL volumetric flask and dilute to volume with distilled water.

NOTE: If the concentration ~ of any of the following is expected to exceed the lead concentration (M) by 10 -fold or more, add 1 mL 1M Na2EDTA to each flask before dilution to volume: CO , PO -, 1 -, F -, CH3COO -.If Ca2- or 5O2 are present in 10-fold or greater excess, make all standards and samples 1% (w/w) in La2- [3].

CALIBRATION AND QUALITY CONTROL:

11. Prepare a series of working standards covering the range 0.25 to 20 *ug/mL* Pb (2.5 to 200 *ug* Pb per sample).

a. Add aliquots of calibration stock solution to 100-mL volumetric flasks. Dilute to volume with 10% HNO3. Store the working standards in polyethylene bottles and prepare fresh weekly.
b. Analyze the working standards together with the blanks and samples (steps 14 and 15). c. Prepare a calibration graph of absorbance vs. solution concentration *(ug/mL)*.

12. Aspirate a standard for every 10 samples to check for instrument drift.

13. Check recoveries with at least one spiked media blank per 10 samples. Use method of standard additions occasionally to check interferences.

MEASUREMENT.

14. Set spectrophotometer as specified by the manufacturer and to conditions on page 7082-1.

NOTE: An alternate wavelength is 217.0 nm [8]. Analyses at 217.0 nm have slightly greater sensitivity, but poorer signal-to-noise ratio compared to 283.3 nm. Also non-atomic absorption is significantly greater at 217.0 nm, making the use of D2 or H2 continuum or Zeeman background correction mandatory at the wavelength.

15. Aspirate standards, samples and blanks. Record absorbance readings. 6

NOTE: If the absorbance values for the samples are above the linear range of the standards dilute with 10% HNO3, reanalyze and apply the appropriate dilution factor in the calculations.

CALCULATIONS.

16. Using the measured absorbances, calculate the corresponding concentrations *(ug/mL)* of lead in the sample, C and average media blank C9from the calibration graph.

17. Using the solution volumes (mL) of the sample, V s' and media blanks V 9' calculate the concentration, C *(mg/m3)* of lead in the air volume sampled V (L):

$$C = \frac{CsVs - CgV9}{V} \; mg/m3$$

EVALUATION OF METHOD:

Method S341 [9] was issued on October 24, 1975 and validated over the range 0.13 to 0.4 *mg/m3* for a 180 L air sample, using generated atmospheres of lead nitrate [1]. Recovery in the range 18 to 72 *ug Pb per sample was 98% and collection efficiency of 0.8 urn mixed cellulose filters (Millipore type* M) was 100% for the aerosols. Subsequent studies on analytical recovery of 200 *ug* Pb per sample gave the following results [2,4].

Species	**Diqestion Method**	**Analytical Recovery, %**
Pb metal	HNO3 only	92 = 4
Pb metal	HNO3 -H2O2	103 = 3
PbO	HNO3 only	93 = 4
PbS	HNO3 only	93 = 5
PbO2	HNO3 only	82 = 3
PbO2	HNO3- H2O2	100 = 1
Pb in paint*	HNO3 only	95 = 6
Pb in paint*	HNO3- H2O2	95 = 6

*Standard Reference Material # 1579, U.S. National Institute of Standards and Technology.

Additional collection efficiency studies were also done using Gelman GN-4 filters for the collection of Pb fume, which had geometric mean diameter of 0.1 *urn* [2]. Mean collection efficiency for 24 sampling runs at flow rates between 0.15 and 4.0 L min was > 97 = 2%. Overall precision, S was 0.072 for lead nitrate aerosol [1.9] and 0.068 for Pb fume [2,4].

REFERENCES:

[1] Documentation of the NIOSH Validation Tests. U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
[2] Heavy Metal Aerosols: Collection and Dissolution Efficiencies, Final Report of NIOSH Contract 210-79-0058, W.F. Gutknecht, M.H. Ranade, P.M. Gronse, A. Damie and D. O'Neal. Research Triangle Institute: available as Order No. PB 83-106740 from NTIS, Springfield. VA*22161 (1981).
[3] NIOSH Manual of Analytical Methods, 2nd. ed., V.1. P&CAM 102, U.S. Department of Health, Education, and Welfare. Publ. (NIOSH) 77-157-A (1979).
[4] NIOSH Manual of Analytical Methods, 2nd. ed., V. 7. S341 (revised 3/25/81), U.S. Department of Health and Human Services, Publ. (NIOSH) 82-100 (1982).
[5] NIOSH Manual of Analytical Methods, 2nd. ed., V. 1. P&CAM 102, U.S. Department of Health, Education and Welfare. Publ. (NIOSH) 77-157-A (1977).
[6] Criteria for a Recommended Standard...Occupational Exposure to inorganic Lead (Revised Criteria), U.S. Department of Health, Education and Welfare. Publ. (NIOSH) 78-158 (1978).
[7) NIOSH Manual of Analytical Methods. 2nd. ed.. P&CAM 191.
[8] Analytical Methods for Atomic Absorption Spectrophotometry , Perkin-Elmer (1976).
[9] NIOSH Manual of Analytical Methods, 2nd ed.. V.#. S341, U.S. Department of Health, Education and Welfare, Publ. (NIOSH) 77-157-C (1977).
[10] DataChern Laboratories in-house procedure for microwave sample digestion.
[11] Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. 3rd Ed: U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC. SW-846 (1986).
[12] Kingston.:H.M. and LB. Jassie. "Safety Guidelines for Microwave Systems in the Analytical Laboratory". Introduction to Microwave Acid Decomposition: Theory and Practice: Kingston H.M. and Jassie, L3..Eds.: ACS Professional Reference Book Series: American Chemical Society: Washington, DC. (1988)
[13] 1985 Annual Book of ASTM Standards. Vol. 11.01: "Standard Specification for Reagent Water: ASTM, Philadelphia. PA. D1193 -77 (1985).
[14] Introduction to Microwave Sample Preparation: Theory and Practice: Kingston. H.M. and Jassie. L.B., Eds.: ACS Professional Reference Book series: American Chemical Society: Washington DC (1988).
[15] Kingston. H.M. EPA IAG #DW1-393254-01-0 January 1- March 31, 1988. Quarterly Report.
[16] Binstock. D.A. Yeager. W.M. Grohse. P .M. and Gaskill. A. Validation of a Method for Determining Elements in Solid Waste by Microwave Digestion, Research Triangle Institute Technical Report Draft. RTI Project Number 321 U-3579-24, prepared for the Office of Solid Waste. U.S. Environmental Protection Agency, Washington, DC 20460 (November, 1989).

METHOD REVISED BY:

Mark Millson, NIOSH/DPSE and R. Delon Hull, Ph.D., NIOSH/DBBS: S341 originally validated under NIOSH Contract CDC-94-74-45: additional studies under NIOSH Contract 210-79-0058.

James B. Perkins. David L. Wheeler and Keith Nicholson, Ph.D., DataChem Laboratories, Salt Lake City, UT. prepared the microwave digestion in the Appendix.

APPENDIX -MICROWAVE DIGESTION FOR LEAD IN PAINT CHIPS (ANP OTHER MATRICES) ,

This procedure is an alternative to the procedure presented in the Sample Preparation section of this method. It provides a rapid, complete acid digestion prior to analysis by flame atomic absorption (FAA), heated graphite furnace atomic absorption (HGFAA), and inductively coupled plasma spectroscopy (ICP) [10].

Apparatus and Material [11-16]

1. Microwave apparatus requirements
    a. The microwave unit provides programmable power with a minimum of 574 Wand can be programmed to within = 10 W of the required power .
    b. The microwave unit cavity is corrosion resistant as well as ventilated. All electronics are protected against corrosion for safe operation.
    c. The system requires Teflon PFA digestion vessels (120-mL capacity) capable of withstanding pressures up to 7.5 = 0.7 atm (110 = 10 psi) and capable of controlled pressure relief at pressures exceeding 7.5 = 0.7 atm (110 = 10 psi).
    d. A rotating turntable is employed to ensure homogeneous distribution of microwave radiation within the unit. The speed of the turntable should be a minimum of 3 rpm.
    e. A safety concern relates to the use of sealed containers without pressure relief valves in the unit. Temperature is the important variable controlling the reaction. Pressure is needed to attain ele- vated temperatures but must be safely contained [12].
    f. Polymeric volumetric ware in plastic (Teflon or polyethylene), 50- or 100-mL capacity. g. Disposable polypropylene filter funnel.
    h. Analytical balance. 300-g capacity, and minimum = 0.001 g.

Reagents

1. Nitric acid, concentrated, spectroscopy grade.
2. Reagent Water. Reagent water shall be interference free. All references to water in the method refer to reagent water that meets the ASTM Type 2 standard.

Procedure

1. Calibration of Microwave Equipment
   Calibrate microwave equipment in accordance with manufacturer's instructions. If calibration instructions are not available. see EPA Method 3051 [11].

2. All digestion vessels are volumetric ware must be carefully acid washed and rinsed with reagent water. All digestion vessels should be cleaned by bleaching with hot (1:1) nitric acid for a minimum of fifteen minutes, rinsed with reagent water, and dried in a clean environment.

3. Sample Digestion

a. Tare the Teflon PFA digestion vessel.
b. Weigh out 0.1 g paint chip sample to the nearest 0.001 g into the tared Teflon PFA sample vessel. With large paint chip samples, measure out a 2 cm2 piece, weigh to the nearest 0.001 g, and quantitatively transfer it to the vessel.

c. Add 5.0 = 0.1 mL concentrated nitric acid to the sample vessel in a fume hood. If a vigorous reaction occurs, allow the reaction to stop before capping the vessel. Cap the vessel and torque the cap to 12 ft-lb (16 N-m) according to the manufacturer's direction. The sample vessel may be connected to an overflow vessel using Teflon PFA connecting tubes. Place the vessels in the microwave carrousel. Connect the overflow vessels to the center well of the unit.

d. Place the vessels evenly distributed in the turntable of the microwave unit using groups of two, six or 12 sample vessels. Any vessels containing 5 mL of nitric acid for reagent blank purposes are counted as sample vessels. When fewer than the recommended number of samples are to be digested, i.e. three samples plus one blank, the remaining vessels should be filled with 5 mL of nitric acid to achieve the full complement of vessels. This provides an energy balance since the microwave power absorbed is proportional to the total mass in the cavity [14]. Irradiate each group of samples to achieve a temperature of 180 °C in five minutes at a pressure of 50 psi. Continue to irradiate to achieve a temperature of 180 °C at 100 psi after 25 minutes. Continue digestion for five minutes. A sample digestion program for 12 samples is presented in Table 1.

Table 1

Program Variables for Paint Chips Sample Digestion with Nitric Acid

Stage	(1)	(2)	(3)
Power	90%	90%	0%
Pressure, psi	50	100	0
Run Time, min.	10:00	20:00	05:00
Time @ P. min	05:00	15:00	00:00
Temperature	180 °C	180 °C	0 °C
Fan Speed	100%	100%	100%
Number of Vessels:	12		
Liquid Volume per Vessel:	5 mL		
Sample Weight:	0.1 g		

If the analyst wishes to digest other than two, six, or 12 samples at a time, use different values of power as long as they result in the same time and temperature conditions.

e. At the end of the microwave program, allow the vessels to cool for a minimum of five minutes before removing them from the microwave unit. If a loss of sample is detected (e.g. material in overflow collection vessel, liquid outside liner), determine the reason for the loss (e.g. loss of vessel seal integrity , use of a digestion time longer than 30 minutes, too large a sample, or improper heating conditions). Once the source of the loss has been corrected, prepare a new sample beginning at Section 2. If sufficient material is available for reanalysis, dilute remaining digestate and note that some sample loss may have occured.

f. Uncap and vent each vessel in a fume hood. Add 20 mL reagent water, then reseal vessels and shake to mix thoroughly. Transfer the sample to an acid-cleaned polyethylene bottle. If the digested sample contains particulates which may clog nebuilizers or interfere with injection of the sample into the instrument, allow the sample to settle or filter it:

Settling: Allow the sample to stand until the supernatant is clear (usually, overnight is sufficient. If it does not clear, filter the sample.

Filtering: The filtering apparatus must be thoroughly precleaned and rinsed with dilute nitric acid. Filter the sample through quantitative filter paper into a second acid-cleaned container .

The digestate is now ready for analysis for elements of interest using the appropriate method.

4. Calculations: Report the concentrations based on the actual weight of the original sample.