FORMULA: Pb

LEAD

METHOD: 7082
ISSUED: 2/15/84

M.W.: 207.19 (Pb); 223.19 (PbO)

OSHA: 0.05 mg/m³
NIOSH: 0.05 mg/m³ [1]
ACGIH: 0.15 mg/m³; STEL 0.45 mg/m³

PROPERTIES: soft metal;
d 11.3 g/cm³; MP 327.5 °C;
valences +2, +4 in salts

SYNONYMS: vary depending upon the chemical form (elemental lead and lead compounds except alkyl lead); CAS #1317-36-8 (PbO); CAS #7439-92-1 (Pb).

SAMPLING

SAMPLER: FILTER
(0.8-µm cellulose ester membrane)

FLOW RATE: 1 to 4 L/min

VOL-MIN: 200 L @ 0.05 mg/m³
-MAX: 1200 L

SHIPMENT: routine

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.13 to 0.4 mg/m³ [2];
0.15 to 1.7 mg/m³ (fume) [3]

BIAS: not significant [2]

OVERALL PRECISION (s_r): 0.072 [2];
0.068 (fume) [3]

MEASUREMENT

!TECHNIQUE: ATOMIC ABSORPTION, FLAME

!ANALYTE: lead

!ASHING: conc. HNO_3, 6 mL; 140 °C

!FINAL SOLUTION: 10% HNO_3, 10 mL

!FLAME: air-acetylene, oxidizing

!WAVELENGTH: 283.3 nm

!BACKGROUND CORRECTION: D_2 or H_2 lamp

!CALIBRATION: Pb^{++} in 10% HNO_3

!RANGE: 10 to 200 µg per sample [3,8]

!ESTIMATED LOD: 2.6 µg per sample [9]

!PRECISION (s_r): 0.03 [2]

APPLICABILITY: The working range is 0.025 to 0.5 mg/m³ for a 400-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 the samples can be analyzed separately for additional elements.

INTERFERENCES: Use D_2 or H_2 continuum background correction to control flame or molecular absorption. High concentrations of calcium, sulfate, carbonate, phosphate, iodide, fluoride, or acetate can be corrected.

OTHER METHODS: This method combines and replaces P&CAM 173 [8] and S341 [7,9] for lead. Method 7300 (ICP-AES) is an alternate analytical method.Method 7505 is specific for lead sulfide. The following have not been revised: the dithizone method, which appears in P&CAM 102 [*] and the lead criteria document [1]; P&CAM 191 (ASV) [5]; and P&CAM 214 (graphite furnace-AAS) [6].

CALIBRATION AND QUALITY CONTROL:

11. Prepare a series of working standards covering the range 1 to 20 µg Pb/mL (1 to 200 µg Pb per sample) by adding aliquots of calibration stock solution to 100-mL volumetric flasks. Dilute to volume with 10% HNO_3. Store the working standards in polyethylene bottles and prepare fresh weekly.
12. Analyze the working standards together with the blanks and samples (steps 17 and 18).
13. Prepare a calibration graph of absorbance vs. solution concentration (µg/mL).
14. Aspirate a standard for every 10 samples to check for instrument drift.
15. Check recoveries with at least one spiked media blank per 10 samples.
16. Use method of additions occasionally to check for interferences.

MEASUREMENT:

17. Set spectrophotometer as specified by the manufacturer and to conditions on page 7082-1.
    NOTE: An alternate wavelength is 217.0 nm [10]. 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 D_2 or H_2 continuum background correction mandatory at that wavelength.
18. Aspirate standards, samples, and blanks. Record absorbance readings.
    NOTE: If the absorbance values for the samples are above the linear range of the standards, dilute with 10% HNO_3, reanalyze, and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

19. Using the measured absorbances, calculate the corresponding concentrations (µg/mL) of lead in the sample, C_s, and average media blank, C_b, from the calibration graph.
20. Using the solution volumes (mL) of the sample, V_s, and media blanks, V_b, calculate the concentration, C (mg/m^3), of lead in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, \ mg/m^3.$$

EVALUATION OF METHOD:

Method S241 [7] was issued on October 24, 1975, and validated over the range 0.13 to 0.4 mg/m^3 for a 180-L air sample, using generated atmospheres of lead nitrate [2]. Recovery in the range 18 to 72 µg Pb per sample was 98%, and collection efficiency of 0.8-µm mixed cellulose ester filters (Millipore Type AA) was 100% for the aerosols. Subsequent studies on analytical recovery of 200 µg Pb per sample gave the results [3,9]:

Species	Digestion Method	Analytical Recovery, %
Pb metal	HNO_3 only	92 ± 4
Pb metal	HNO_3 + H_2O_2	103 ± 3
PbO	HNO_3 only	93 ± 4
PbS	HNO_3 only	93 ± 5
PbO_2	HNO_3 only	82 ± 3
PbO_2	HNO_3 + H_2O_2	100 ± 1
Pb in paint*	HNO_3 only	95 ± 6
Pb in paint*	HNO_3 + H_2O_2	95 ± 6

*Standard Reference Material #1579, U.S. National Bureau of Standards.