

FORMULA: Pb

LEAD

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

METHOD: 7105

ISSUED: 8/15/90

OSHA: 0.05 mg/m³

NIOSH: <0.1 mg/m³ [1]

ACGIH: 0.15 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 #7439-92-1 (Pb); CAS #1317-36-8 (PbO).

SAMPLING	MEASUREMENT
SAMPLER: FILTER (0.8-µm cellulose ester membrane)	! TECHNIQUE: ATOMIC ABSORPTION, GRAPHITE FURNACE !
FLOW RATE: 1 to 4 L/min	! ANALYTE: lead !
VOL-MIN: 1 L @ 0.05 mg/m ³ -MAX: 1500 L	! ASHING: conc. HNO ₃ , 3 mL; 30% H ₂ O ₂ , 1 mL; 140 °C !
SHIPMENT: routine	! FINAL SOLUTION: 10 mL 5% HNO ₃ !
SAMPLE STABILITY: stable	! WAVELENGTH: 283.3 nm !
FIELD BLANKS: 10% of samples	! GRAPHITE TUBE: pyrolytic coated !
	! INJECTION: 20 µL + 10 µL matrix modifier, ! DRY: 110 °C, 70 sec; ! CHAR: 800 °C, 30 sec; ! ATOMIZE: 1800 °C, 5 sec. !
	! BACKGROUND CORRECTION: D ₂ , H ₂ or Zeeman !
	! CALIBRATION: Pb ⁺⁺ in 5% HNO ₃ !
	! RANGE: 0.05 to 1.0 µg per sample [2] !
	! ESTIMATED LOD: 0.01 µg per sample [2] !
	! PRECISION (s _p): not determined !

APPLICABILITY: The working range is 0.0002 to >1 mg/m³ for a 200-L air sample. If high concentrations are expected, the samples should be analyzed by flame AAS. 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 sample may be analyzed separately for additional elements.

INTERFERENCES: Use D₂ or H₂ continuum or Zeeman background correction to control molecular absorption. High concentrations of calcium, sulfate, carbonate, phosphate, iodide, fluoride, or acetate can be offset by an additional sample treatment step.

OTHER METHODS: This revises and replaces P&CAM 214 (3). Method 7300 (ICP-AES) is an alternate analytical method. Method 7505 is specific for lead sulfide. Method 7082 is a flame AA method.

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16. Perform a matrix spike of a sample occasionally to check for matrix interferences. If an adequate recovery is not obtained (85 to 115%), an alternate method of analysis should be used, such as flame AA or ICP.

MEASUREMENT:

17. Set spectrophotometer as specified by the manufacturer and to conditions on page 7105-1.

NOTE: An alternate wavelength is 217.0 nm [6]. Analyses at 217.0 nm have slightly greater sensitivity, but poorer signal-to-noise ratio compared to 283.3 nm. Also, nonatomic absorption is significantly greater at 217.0 nm, making the use of D₂, H₂ continuum, or Zeeman background correction mandatory at that wavelength.

18. Add matrix modifier to samples and standards in proper ratio of 2 to 1 (sample or standard to matrix modifier).
19. Analyze standards, samples, and blanks. Record absorbance readings.
- NOTE: If the absorbance value for the sample is above the linear range of the standards, dilute with 5% HNO₃, reanalyze, and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

20. 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.
21. Using the solution volumes (mL) of the sample, V_s, and media blanks, V_b, calculate the concentration, C (mg/m³), of lead in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Method S341 [5] 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 [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 using flame AAS gave the results [3]:

Species	Digestion Method	Analytical Recovery, %
Pb metal	HNO ₃ only	92 ± 4
Pb metal	HNO ₃ + H ₂ O 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 ₂ O ₂	100 ± 1
Pb in paint*	HNO ₃ only	95 ± 6
Pb in paint*	HNO ₃ + H ₂ O ₂	95 ± 6

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

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 µm [3]. Mean collection efficiency for 24 sampling runs at flow rates between 0.15 and 4.0 L/min was 97 ± 2%. Overall precision, s_p, was 0.072 for lead nitrate aerosol [2,5] and 0.068 for Pb fume [3].

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