

8.1.04

AOAC Official Method 974.02 Lead in Paint

Atomic Absorption Spectrophotometric Method First Action 1974 Final Action 1976

A. Reagents and Apparatus

(a) *Lead standard solutions.*—(1) *Stock solution.*—1 mg Pb²⁺/mL 1% (v/v) HNO₃. Dissolve 159.9 mg Pb(NO₃)₂ in 1% HNO₃ and dilute to 100 mL with 1% HNO₃. (2) *Intermediate solution.*—300 g/mL dilute HNO₃. Dilute 15 mL stock solution to 50 mL with 0.5 mL 1% HNO₃ and H₂O. (3) *Working solutions.*—To each of seven 100 mL volumetric flasks containing 1 mL HNO₃, add 0, 1, 2, 3, 4, 5, and 6 mL intermediate solution, respectively, and dilute to volume with H₂O to yield solutions with 0, 3, 6, 9, 12, 15, and 18 g Pb²⁺/mL.

(b) *Atomic absorption spectrophotometer.*—With Pb hollow cathode lamp and 4 in. single slot or 3 slot Belling burner head, capable of detecting 0.5 g Pb²⁺/mL (Perkin-Elmer, 549 Albany St, Boston, MA 02118-2512, USA; www.perkinelmer.com; Model 4100 or equivalent). Operating conditions: 283.3 nm, 0.7 nm band width slit, time constant (0.25-1 s, if recorder used), air-C₂H₂ flame (gas flows adjusted according to directions of manufacturer).

(c) *Heater for digestion.*—Drill 8.5 cm³ Al cube to hold 16 test tubes, 16 150 mm. Place on hot plate capable of maintaining medium at 160–170°C (Model PC300; Nova Analytics Corp., 6E Gill St, Woburn, MA 01801, USA; www.novaanalytic.com; or equivalent). Sand bath may be used instead of Al block.

(d) *Boiling chips.*—Unglazed boiling chips, 1.5 mm diameter, Pb-free.

(e) *Aluminum dish.*—63 mm diameter.

B. Determination of Solids

Thoroughly mix test materials manually for 10 min or mechanically for 5 min. Accurately weigh 0.3–0.4 g into preweighed Al dish. Add 3–5 mL hexane or petroleum ether to oil-based paints or H₂O to latex paints and swirl to disperse. Warm on hot plate, in an exhaust hood, swirling to allow solvent to evaporate and film to form. Heat film in oven 4 h at 105°C, cool, and

weigh. (*Caution:* Hexane and petroleum ether are extremely flammable.)

$$\text{Solids, \%} = \frac{\text{g dried test sample}}{\text{g original test sample}} \times 100$$

C. Determination of Lead

Introduce ca 0.6 g (0.3 mL) thoroughly mixed test portion to bottom of 16 150 mm tared test tube, using a syringe, and weigh test portion accurately. Add 5 0.2 mL concentrated HNO₃ and 2 boiling chips to each, including blanks. Place tubes in heated block or bath at 90–100°C so that liquid surface is slightly above heated surface. (Use exhaust hood.) After initial fuming has subsided, increase temperature until vapors are condensing within 1–2 cm of tube's mouth (bath temperature, ca 165°C) and maintain at this temperature for 3 h. Cool to 50–60°C and transfer tube contents including chips and any precipitate to 25 mL volumetric flask. Rinse tube with four 4 mL portions H₂O, transferring as much residue as possible. Dilute to volume with H₂O and let settle 0.5–1 h. Floating residue may be removed with a disposable pipet.

Aspirate solutions and standards into AA spectrophotometer, avoiding introduction of precipitate. If *A* of test sample is greater than highest standard, dilute test sample with H₂O and re-aspirate. Determine g Pb²⁺/mL from standard curve.

$$\text{Pb in paint solids, \%} = \frac{(\text{g Pb}^{2+} / \text{mL}) F \cdot 10^2}{\text{g test portion} \cdot \% \text{ solids in test sample}}$$

$$F (\text{dilution factor}) = \frac{1}{(1/25)(b/c)(d/e)\dots}$$

where 25 = volume original test portion digest, *b* = aliquot of original 25 mL diluted to *c* mL; *d* = aliquot of *c* (mL) diluted to *e* mL; etc. When starting analysis with dry paint film, assume % solids in test sample = 100.

Reference: *JAOAC* 57, 614(1974).

CAS-7439-92-1 (lead)