ASTM
INTERNATIONAL

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Standard Test Methods for
Chemical Analysis of Pig Lead[1]

This standard is issued under the fixed designation E 37; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the chemical analysis of pig lead having chemical compositions within the following limits:

Element	Concentration Range, %			
Antimony	0.001	to	0.02
Arsenic	0.0005	to	0.02
Bismuth	0.002	to	0.2
Copper	0.001	to	0.1
Iron	0.0005	to	0.005
Lead	99.5	to	99.99
Silver	0.001	to	0.03
Tin	0.001	to	0.02
Zinc	0.001	to	0.005

1.2 The test methods appear in the following order:

	Sections
Antimony by the Rhodamine-B Photometric Method	19-28
Copper, Bismuth, Silver, and Zinc by the Atomic Absorption Method	8-18

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For precautions to be observed in the use of certain reagents, refer to Practices E 50. Specific hazard statements are given in the individual test methods.

2. Referenced Documents

2.1 *ASTM Standards:*
B 29 Specification for Pig Lead[2]
E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications[3]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[4]
E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals[4]
E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[4]

3. Significance and Use

3.1 These test methods for the chemical analysis of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use these methods will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

4. Apparatus, Reagents, and Photometric Practice

4.1 Apparatus and reagents required for each determination are listed in separate sections of each test method. The apparatus, standard solutions, and certain other reagents used in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E 50, except that photometers shall conform to the requirements prescribed in Practice E 60.

5. Sampling

5.1 For procedures for sampling the material, refer to Specification B 29.

6. Rounding Calculated Values

6.1 Calculated values shall be rounded to the desired number of places as directed in 3.4 to 3.6 of Practice E 29.

7. Interlaboratory Studies

7.1 These test methods have been evaluated in accordance with Practice E 173, unless otherwise noted in the precision section.

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COPPER, BISMUTH, SILVER, AND ZINC BY THE ATOMIC ABSORPTION METHOD

8. Scope

8.1 This test method covers the determination of bismuth in concentrations from 0.002 to 0.2 %, copper from 0.001 to 0.1 %, silver from 0.001 to 0.03 %, and zinc from 0.001 to 0.005 %.

9. Summary of Test Method

9.1 The sample is dissolved in a nitric-perchloric acid mixture, the solution is fumed, and hydrochloric acid is added to precipitate lead chloride. The hydrochloric-perchloric acid solution is aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. The absorption of the resonance line energy from the spectrum of each element is measured and compared with that of calibration solutions of the same element. The lines used were Cu 3247, Bi 2230, Ag 3280, and Zn 2138 Å.

10. Concentration Range

10.1 The concentration range for each element must be determined experimentally because the optimum range will depend upon the individual instrument. Determine the appropriate concentration range of each element as follows:

10.1.1 Prepare a dilute standard solution as directed in Section 14. Refer to 14.1 for suggested initial concentrations.

10.1.2 Prepare the instrument for use as directed in 16.1. Measure the instrument response while aspirating water, the calibration solution with the lowest concentration, and the two with the highest concentrations. Determine the minimum response and the curve linearity as directed in 12.1.1 and 12.1.2, respectively.

10.1.3 If the instrument meets or surpasses the minimum response and curve linearity criteria, the initial concentration range may be considered suitable for use. In this case proceed as directed in 10.1.5.

10.1.4 If the minimum response is not achieved, prepare another dilute standard solution to provide a higher concentration range, and repeat 10.1.2 and 10.1.3. If the calibration curve does not meet the linearity criterion, prepare another dilute standard solution to provide a lower concentration range, and repeat 10.1.2 and 10.1.3. If a concentration range cannot be found for which both criteria can be met, do not use this method until the performance of the apparatus has been improved.

10.1.5 Perform the stability test as directed in 12.1.3. If either of the minimum stability requirements is not met, do not use this method until the repeatability of the readings has been suitably improved.

11. Interferences

11.1 Elements ordinarily present do not interfere if their concentrations are under the maximum limits shown in 1.1.

12. Apparatus

12.1 *Atomic Absorption Spectrophotometer*—Use hollow-cathode lamps, operated in accordance with manufacturers' recommendations as sources for the following lines: Cu 324.7, Bi 223.0, Ag 328.0, and Zn 213.8 nm. Aspirate the solutions into an air-acetylene flame of a premix burner. Determine that the atomic absorption spectrophotometer is satisfactory for use in this method by proceeding as directed in 12.1.1-12.1.3.

NOTE 1—Optimum settings for the operating parameters of the atomic absorption spectrophotometer vary from instrument to instrument.

12.1.1 *Minimum Response*— Calculate the difference between the readings of the two highest of five equally spaced (14.2) calibration solutions. This difference must be at least 40 scale units.

NOTE 2—The scale unit is defined as the smallest numerical interval that is estimated in taking each reading on the instrument. If the scale is non-linear, the largest unit defined in this manner is used.

12.1.2 *Curve Linearity*— Calculate the difference between the scale readings obtained with water and the lowest of the five equally spaced calibration solutions. If necessary, convert this difference and the difference calculated in 12.1.1 to absorbance. Divide the difference for the highest interval by that for the lowest interval. If this ratio is not 0.70 or greater, proceed as directed in 10.1.4.

12.1.3 *Minimum Stability*—If the variability of the readings of the highest calibration solution and of water is not less than 1.8 % and 1.4 %, respectively, as calculated below, proceed as directed in 10.1.5.

$$V_C = \frac{100}{\bar{C}} \sqrt{\frac{\Sigma(C - \bar{C})^2}{n - 1}} \qquad (1)$$

$$V_o = \frac{100}{\bar{C}} \sqrt{\frac{\Sigma(O - \bar{O})^2}{n - 1}}$$

where:

V_C	=	percent variability of the highest calibration readings,
$\bar{C}$	=	average absorbance value for the highest calibration solution,
$\Sigma(C - \bar{X})^2$	=	sum of the squares of the n differences between the absorbance readings of the highest calibration solution and their average,
V_O	=	percent variability of the readings on water relative to $\bar{C}$,
$\bar{O}$	=	average absorbance value of water,
$\Sigma(O - \bar{O})^2$	=	sum of the squares of the n difference between the absorbance readings of water and their average, and
n	=	number of determinations, three or more.

13. Reagents

13.1 *Bismuth, Standard Solution* (1 mL = 1.00 mg Bi)— Transfer 1.00 g of bismuth (purity: 99.9 % min) to a 400-mL beaker and dissolve in 50 mL of HNO_3 (1 + 1), heating gently if necessary. When dissolution is complete, cool, transfer to a 1-L volumetric flask, add 100 mL of HNO_3 (1 + 1), dilute to volume, and mix. Store in a polyethylene bottle.

13.2 *Copper, Standard Solution* (1 mL = 1.00 mg Cu)— Proceed as directed in 13.1, but substitute 1.00 g of copper (purity: 99.9 % min) for the bismuth.