Designation: E37 – 05 (Reapproved 2011)

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

This standard is issued under the fixed designation E37; 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	21-30
Copper, Bismuth, Silver, and Zinc by the Atomic Absorption Method	10-20

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.* Specific precautionary statements are given in the individual test methods.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B29 Specification for Refined Lead
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry
E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]
E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E135.

4. Significance and Use

4.1 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.

5. Apparatus, Reagents, and Photometric Practice

5.1 Apparatus and reagents required for each determination are listed in separate sections of each test method. The apparatus, standard solutions, and reagents conform to the requirements prescribed in Practices E50. Photometers shall conform to the requirements prescribed in Practice E60.

6. Safety Hazards

6.1 For precautions to be observed in the use of certain reagents in these test methods, refer to Practices E50.

7. Sampling

7.1 For procedures for sampling the material, refer to Specification B29.

---

[1] These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.05 on Cu, Pb, Zn, Cd, Sn, Be, their Alloys, and Related Metals.

Current edition approved Feb. 1, 2011. Published March 2011. Originally approved in 1942. Last previous edition approved in 2005 as E37 – 05. DOI: 10.1520/E0037-05R11.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

8. Rounding Calculated Values

8.1 Calculated values shall be rounded to the desired number of places as directed in Practice E29.

9. Interlaboratory Studies

9.1 These test methods have been evaluated in accordance with Practice E173, unless otherwise noted in the precision section.

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

10. Scope

10.1 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 %.

11. Summary of Test Method

11.1 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 324.7, Bi 223.0, Ag 328.0, and Zn 213.8 nm

12. Concentration Range

12.1 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:

12.1.1 Prepare a dilute standard solution as directed in Section 16. Refer to 16.1 for suggested initial concentrations.

12.1.2 Prepare the instrument for use as directed in 18.1. 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 14.1.1 and 14.1.2, respectively.

12.1.3 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 12.1.5.

12.1.4 If the minimum response is not achieved, prepare another dilute standard solution to provide a higher concentration range, and repeat 12.1.2 and 12.1.3. If the calibration curve does not meet the linearity criterion, prepare another dilute standard solution to provide a lower concentration range, and repeat 12.1.2 and 12.1.3. 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.

12.1.5 Perform the stability test as directed in 14.1.3. 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.

13. Interferences

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

14. Apparatus

14.1 *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 14.1.1-14.1.3.

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

14.1.1 *Minimum Response*— Calculate the difference between the readings of the two highest of five equally spaced (16.2) 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.

14.1.2 *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 14.1.1 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 12.1.4.

14.1.3 *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 12.1.5.

$$V_C = \frac{100}{\bar{C}} \sqrt{\frac{\sum \left(C - \bar{C}\right)^2}{n - 1}} \tag{1}$$

$$V_o = \frac{100}{\bar{C}} \sqrt{\frac{\sum \left(O - \bar{O}\right)^2}{n - 1}} \tag{2}$$

where:

V_C = percent variability of the highest calibration readings,

$\bar{C}$ = average absorbance value for the highest calibration solution,

$\sum (C - \bar{X})^2$ = 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,

$\sum (O - \bar{O})^2$ = 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.

15. Reagents

15.1 *Bismuth, Standard Solution* (1 mL = 1 mg Bi)—Transfer 1 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.

15.2 *Copper, Standard Solution* (1 mL = 1 mg Cu)—Proceed as directed in 15.1, but substitute 1 g of copper (purity: 99.9 % min) for the bismuth.

15.3 *Silver, Standard Solution* (1 mL = 1 mg Ag)—Proceed as directed in 15.1 but substitute 1 g of silver (purity: 99.9 % min) for the bismuth.

15.4 *Zinc, Standard Solution* (1 mL = 0.1 mg Zn)—Proceed as directed in 15.1 but substitute 0.1 g of zinc (purity: 99.9 % min) for the bismuth.

16. Calibration

16.1 *Dilute Standard Solution*—Using pipets, transfer to 500-mL volumetric flasks the following volumes of each standard solution: bismuth, 20 mL; copper, 10 mL; silver, 5 mL; and zinc, 10 mL. Dilute to volume and mix. Adjust the concentration of a dilute standard solution if the proper range is not obtained when the 5, 10, 15, 20, and 25-mL portions are diluted to 100 mL and tested.

16.2 *Calibration Solutions*—Prepare five calibration solutions for each element to be determined. Using pipets, transfer 5, 10, 15, 20, and 25-mL portions of the appropriate dilute standard solution to 100-mL volumetric flasks. Add sufficient volumes of HCl and $HClO_4$ to each flask to yield final acid concentrations equal to that of the corresponding test solution, dilute to volume, and mix. Do not use solutions that have stood more than 24 h.

17. Procedure

17.1 *Test Solution:*

17.1.1 Transfer a 10 g sample, weighed to the nearest 10 mg, to a 300-mL Erlenmeyer flask (Note 3). Add 3 mL of HNO_3 and 15 mL of $HClO_4$, and heat until dissolution is complete. Evaporate to strong fumes of perchloric acid and cool.

NOTE 3—Due to the limited solubility of silver chloride, the silver concentration in the sample solution should be less than 1 mg/100 mL. If the expected silver concentration is higher than 0.01 %, choose a sample weight that limits the silver concentration to less than 1 mg/100 mL.

17.1.2 Add 50 mL of water and, while swirling, heat to boiling. Add 25 mL of HCl. If less than a 10-g sample is used, add 20 mL HCl plus 0.5 mL for each gram of sample used. Heat again to boiling and cool to room temperature.

17.1.3 Transfer the solution and precipitate to a 100-mL volumetric flask, dilute to volume with water, and mix thoroughly. Allow the precipitated lead chloride to settle. Use the supernatant solution, or dilute an appropriate aliquot of the supernatant solution to provide a concentration of the element being measured which lies within the concentration range determined in Section 12.

17.2 *Reagent Blank Solution*—Prepare a reagent blank by adding 3 mL of HNO_3 and 15 mL of $HClO_4$ to a 300-mL Erlenmeyer flask and proceed as directed in 17.1.

18. Measurement

18.1 *Instrument Adjustment*—Optimize the response of the instrument as directed in 18.1.1-18.1.4.

18.1.1 Set the instrument parameters approximately at the values obtained in 14.1, and light the burner.

18.1.2 Adjust the instrument to the approximate wavelength for the element to be determined, permit the instrument to reach thermal equilibrium, and complete the wavelength adjustment to obtain maximum absorption while aspirating the highest calibration solution.

18.1.3 Optimize fuel, air, and burner adjustments while aspirating the highest calibration solution.

18.1.4 Aspirate water long enough to establish that the absorbance reading is stable and then set the initial reading (approximately zero absorbance or 100 % transmittance).

18.2 *Photometry:*

18.2.1 Aspirate the test solution and note, but do not record the reading.

NOTE 4—Avoid transferring particles of precipitated lead chloride that may clog the aspirator during the measurements of the test solution.

18.2.2 Aspirate water until the initial reading is again obtained. Aspirate the calibration solutions and test solution in order of increasing instrument response, starting with the reagent blank. When a stable response is obtained for each solution, record the reading.

18.2.3 Proceed as directed in 18.2.2 at least twice more.