



Designation: E 117 - 64 (Reapproved 1989)

Standard Method for Spectrographic Analysis of Pig Lead by the Point-to-Plane Technique¹

This standard is issued under the fixed designation E 117; 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 This method covers the spectrographic analysis of pig lead for the following elements in the ranges indicated:

Element	Concentration Range, %
Bismuth	0.0005 to 0.3
Tellurium	0.01 to 0.1
Copper	0.0005 to 0.08
Arsenic	0.001 to 0.03
Antimony	0.001 to 0.03
Zinc ⁴	0.001 to 0.02
Silver	0.0005 to 0.02
Nickel	0.001 to 0.01
Tin	0.001 to 0.01
Cadmium	0.0003 to 0.01
Iron ⁴	approximately 0.0005

⁴ Determined only semiquantitatively because of segregation.

1.2 This method is designed for testing specimens in the form of chill-cast disks or miscellaneous lead samples on which a flat surface area of 25 mm (1 in.) in diameter can be prepared. Other forms may be analyzed, provided they are sufficiently massive (about 50 g) to prevent overheating during excitation. For irregularly shaped samples or chipped samples, specimens are slowly heated to the melting point under palm oil and cast in disk form.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- E 37 Methods for Chemical Analysis of Pig Lead²
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²
- E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis³
- E 116 Practice for Photographic Photometry in Spectrochemical Analysis³
- E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes³

¹ This method is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry of Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.05 on Zn, Sn, Pb, Cd, Be, and Other Metals.

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² Annual Book of ASTM Standards, Vol 03.05.

³ Annual Book of ASTM Standards, Vol 03.06.

E 135 Terminology Relating to Analytical Atomic Spectroscopy³

3. Terminology

3.1 For definitions of terms used in this method, refer to Terminology E 135.

4. Summary of Method

4.1 The sample is excited by a controlled spark or triggered capacitor discharge using the point-to-plane technique. The spectra are photographed on a calibrated emulsion and the intensity ratios of selected pairs of analytical lines and internal standard lead lines are determined photometrically. Concentration of the elements is read from analytical curves relating log intensity ratio to concentration.

5. Significance and Use

5.1 This method is designed for impurity testing of lead from primary sources (that is, ore-based material). Usually, recycled lead from secondary sources is outside the range of elements described in the scope of this method. Also, the samples must be of significant size (at least 50 g) to be adequately analyzed by this procedure. Users of this method should be cautioned that specimens that are remelted and recast for analysis may not be representative of the original sample.

6. Apparatus

6.1 Sample Preparation Equipment:

6.1.1 *Sample Mold*, suitable for forming disks 70 mm (2¾ in.) in diameter by 13 mm (½ in.) thick.

6.1.2 Lathe.

6.2 *Electrode Cutter*, for shaping electrodes to the configuration described in 7.2.

6.3 *Excitation Source*—A high-voltage controlled spark or condensed arc discharge source that will provide the parameters listed in 11.1, or equivalent.

6.4 *Spectrograph*—A grating spectrograph having a reciprocal linear dispersion of approximately 7 Å/mm, or a prism instrument with sufficient dispersion and resolving power to separate the analytical lines in the spectral region 2150 to 4350 Å. The spectrograph shall be illuminated by an image of the source focused on the grating or collimator. Suitable filters shall be placed in the light path to control the intensity.

6.5 *Photographic Processing Equipment*, providing developing, fixing, washing, and drying operations and conforming to the requirements of Practice E 115.

6.6 *Microphotometer*, having a precision of at least $\pm 1.0\%$ for transmittances between 5 and 90 %.

6.7 *Calculating Equipment*—A calculating board with