



Designation: B982 – 14

# Standard Specification for Sampling and Sample Preparation of Lead and Lead Alloys for Optical Emission Spectrometric or ICP Analysis<sup>1</sup>

This standard is issued under the fixed designation B982; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This specification covers the sampling of lead and lead alloys to obtain a sample suitable for quantitative optical emission spectrochemical analysis. Included are procedures for sample preparation, obtaining representative samples from molten metal, from fabricated, or cast products that can be melted, and from other forms that cannot be melted.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

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 establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 The following documents of the issue in effect on the date of material purchase form a part of this specification to the extent referenced herein.

2.2 *ASTM Standards:*<sup>2</sup>

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E37 Test Methods for Chemical Analysis of Pig Lead

E88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee B02 on Nonferrous Metals and Alloys and is the direct responsibility of Subcommittee B02.02 on Refined Lead, Tin, Antimony, and Their Alloys.

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

## 3. Significance and Use

3.1 This specification, used in conjunction with an appropriate quantitative optical emission spectrochemical method, is suitable for use in manufacturing control, material or product acceptance, and development and research.

## 4. Ordering Information

4.1 Orders for refined lead under this specification shall include the following information:

4.1.1 ASTM designation and year of issue,

4.1.2 Quantity (weight),

4.1.3 Name of material (for example, pure lead),

4.1.4 Size and shape (see Section 7),

4.1.5 Grade, and

4.1.6 Certification or test report if specified (Section 13).

## 5. Materials and Manufacture

5.1 Lead shall be supplied in commercial standard forms or shapes requested by the purchaser in the following grades:

5.1.1 Low bismuth low silver pure lead,

5.1.2 Refined pure lead,

5.1.3 Pure lead, and

5.1.4 Chemical copper lead.

5.1.5 Calcium alloys.

5.1.6 Antimony alloys.

5.2 The grade of lead listed in 5.1.1 – 5.1.6 shall be produced by any smelting and refining process from ore or recycled materials to meet the chemical requirements of this specification.

## 6. Composition

6.1 This specification shall apply to lead alloys that contain greater than 80 % Pb and meet the requirements of 5.2 for material manufacture.

## 7. Sizes and Shapes

7.1 Pigs shall weigh up to a nominal 110 lb (50 kg).

7.2 Blocks or hogs shall be square or oblong and weigh up to 2530 lb (1150 kg).

## 8. Appearance

8.1 The lead shall be reasonably free from surface corrosion, surface cavities, excessive condensation or water, and adhering foreign material.

## 9. Lot

9.1 All lead of the same type produced and cast at one time shall constitute a lot for chemical analysis. Each pig or block of the lot shall bear a single identifying number that can be related to the manufacturing lot.

## 10. Sampling for Chemical Analysis

10.1 The sample for chemical analysis shall be selected by one of the following methods:

10.1.1 Test samples taken from the lot during casting applies to all materials.

10.1.2 Test samples taken from the final solidified cast product, for all materials except 5.1.5, calcium alloys.

10.2 *Sampling for Lot Analysis*—The supplier may obtain samples from the lot of molten metal during casting. A sample should be taken at the beginning, middle and end of casting. All or part of these samples may be cast into shapes suitable for use in spectrographic analytical methods. The composite element average for the three grab samples will represent the lot.

### 10.3 *Sampling of Cast Product:*

10.3.1 If the lead is in the form of standard pigs (Fig. 1), the sample for chemical analysis shall be taken in accordance with 10.3.3.1, 10.3.3.2, or 10.3.3.3.

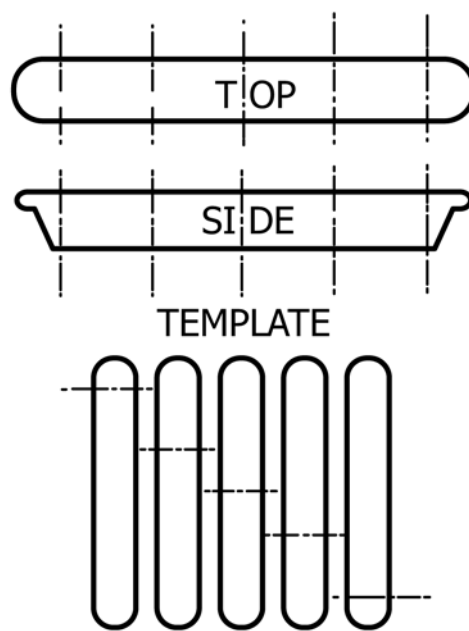
10.3.1.1 If the pigs differ in shape from those shown in Fig. 1 or the product is cast into blocks or hogs, the supplier and the purchaser shall agree mutually as to the method to be followed in sampling such shapes. The method of sampling in 10.3 does not apply to alloys that exhibit segregation of alloy agents during casting. This means the calcium aluminum alloys are not to be sampled according to this procedure. The preferred method of determining lot chemistry for calcium aluminum lots is supplier sampling of quick frozen wafers during casting.

10.3.2 *Sampling Pig Lead*—A portion representative of the total shipment shall be selected at random for the final sample. For lots containing at least 100 000 lb (45 400 kg) of pig lead, one pig shall be taken from every 20 000 lb (9080 kg). For smaller lots, a total of five pigs shall be taken.

10.3.3 *Sample Preparation*—Each pig shall be cleaned thoroughly to rid the surface of dirt or adhering foreign material prior to sampling by one of the following methods: sawing, drilling, or melting.

10.3.3.1 *Sawing*—The pigs selected shall be sawed completely through as illustrated in Fig. 1. The sawings from the pigs shall be mixed thoroughly and quartered, and the samples for analysis taken from the mixed material. The sawings must be free of extraneous material introduced from the saw blade. All sawings shall be treated with a strong magnet in order to remove iron introduced by sawing.

10.3.3.2 *Drilling*—The pigs shall be drilled at least halfway through from two opposite sides as illustrated in Fig. 2. A drill



Pigs sampled in sets of five according to template as shown above.

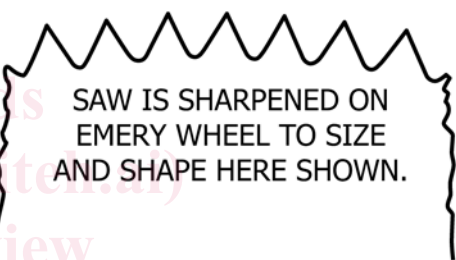


FIG. 1 Method of Sampling Lead by Sawing

of about 1/2 in. (12.7 mm) in diameter shall be used. In drilling, the holes shall be spaced along a diagonal line from one corner of the pig to the other. Holes may be made in a single pig or in each of several pigs placed as illustrated in Fig. 2. The drillings shall be clipped into pieces not over 1/2 in. (12.7 mm) in length, mixed thoroughly, and treated with a strong magnet to remove iron introduced by drilling.

10.3.3.3 *Melting*—Whole pigs, portions of pigs produced by sawing, drillings, or sawings shall be melted in a clean vessel. The melting temperature must not exceed 685°F (363°C) to prevent excessive drossing. The lead must be stirred immediately prior to sampling. The molten lead shall be cast into shapes suitable for use in spectrographic analysis, cast into thin sample bars not to exceed 3/8 in. (9.5 mm) thick for sawing, or granulated by pouring into distilled water and drying the material thoroughly. For sample bars, saw cuts shall be made halfway across the bar from each side and staggered so that they are about 1/2 in. (12.7 mm) apart. The sawings so produced are treated in accordance with 10.3.3.1.

### 10.3.4 *Sample Size:*

10.3.4.1 For spectrographic analysis, three samples shall be prepared of a size and shape satisfactory for use by the laboratory at which the analysis is to be made.

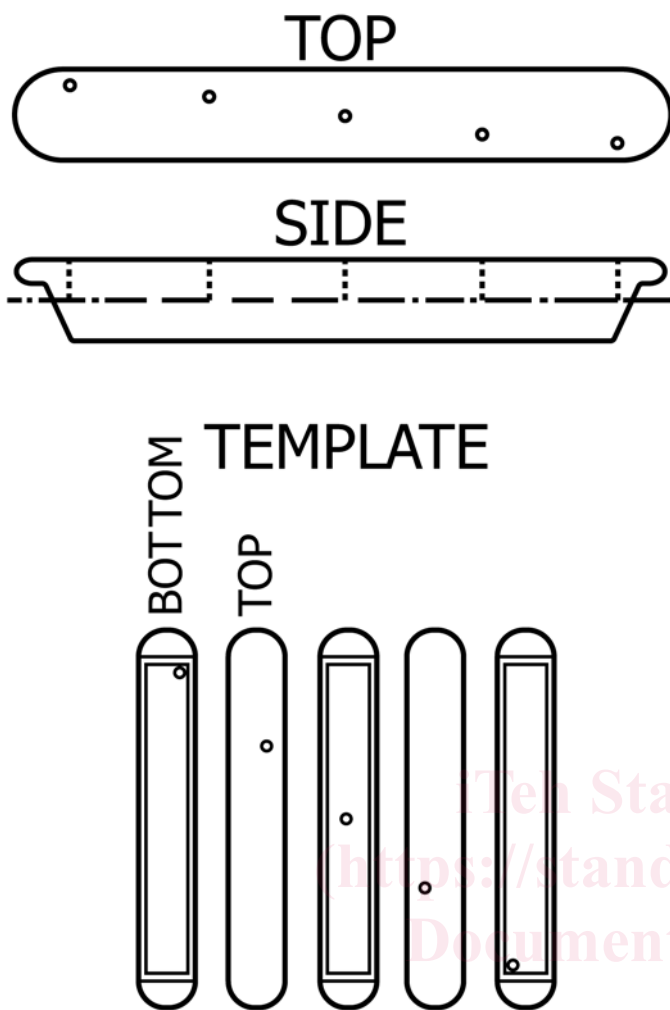


FIG. 2 Method of Sampling Lead by Drilling

casting standards and specimens in the same manner. Also, the specimens shall have a repeatability of measurement for major elements from excitation-to-excitation with a relative error of no more than 2 %. Several types of molds have been found acceptable.

11.2.1 *Type A, Mass Chill Wafer Mold (Fig. 3)*—This mold produces one diagonally cast wafer without a sprues on the top of the specimen. The mold dimensions are such as to produce wafers approximately 2.5 in. (64 mm) in diameter by 0.5 in. (13 mm) thickness. The mold excluding the handle must be made of mild steel with a thickness in all dimensions of at least 0.5 in. This ensures that samples will cool quickly. The mold is made of steel or cast iron and weigh approximately 1.5 to 2.5 lb (0.45 to 1.1 kg). Wafer specimens have been found to be very homogeneous. If properly prepared, these specimens provide very reliable results with only one burn. However, all wafer specimens must be lathed according to 11.3 before analysis or drilling.

11.2.2 *Other Molds*—Molds of different types, materials, and dimensions may be used provided that the uniformity of the specimens obtained is comparable to the uniformity of specimens obtained from Type A molds. Further, the specimens shall have a spectrochemical response similar to the standards used for preparing the analytical curves.

11.3 *Lathe or Milling Machine*—Lathe should be capable of machining a smooth flat surface, 0.25 in. of material must be removed from the surface.

## 12. Sampling

12.1 *Molten Metal*—When molten metal is sampled, the temperature shall be well above the point at which any solid phase can be present. Using a clean ladle, push any dross away from the sampling area. Then dip the ladle sideways into the clear area, well below the surface, and stir momentarily. Turn the ladle upright and quickly withdraw. Heating the ladle prevents metal freezing, while obtaining metal well beneath the surface minimizes the danger of inclusion of oxide. Unless the mold is already hot, cast and discard a preliminary specimen.

12.2 *Separate Sampling Ladles and Molds*—Separate sampling molds and ladles are required where cross contamination can occur between alloys.

12.3 *Pouring of Samples*—Remove any excess metal from the ladle, dip it into the molten metal as before, and fill the mold (Section 11). Avoid overfilling the mold. Allow the metal to freeze without jarring. The sample should be visibly solid within 5 s. The cast surfaces of the specimen, upon removal from the mold, shall be free from shrinkage, inclusions, cracks, or roughness.

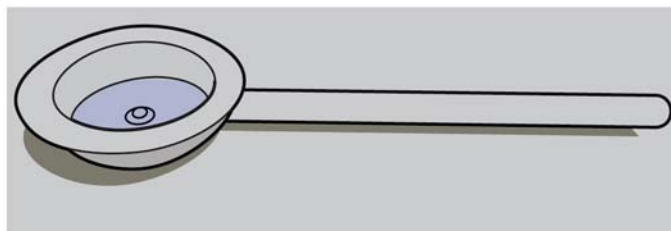


FIG. 3 Wafer Mold

10.3.4.2 For wet chemical analysis, each prepared sample (sawings, drillings, or granules) shall weigh at least 600 g.<sup>3</sup>

10.3.5 Aspects of sampling and sample preparation not specifically covered in this specification shall be carried out in accordance with Practice E88.

## 11. Apparatus

11.1 *Ladle*, of steel, designed to hold sufficient molten metal to completely fill the sample mold, with a handle of sufficient length to reach into a furnace, trough, pot, or crucible.

NOTE 1—Pure lead metal (Special High Grade) is sampled using a ceramic or graphite ladle, as the solubility of iron in Special High Grade Lead is sufficient to cause a measurable contamination.

11.2 *Sample Molds*, designed to produce homogeneous chillcast specimens having smooth surfaces, free from surface pockets and pores. The specimens shall be representative (in the region to be excited) of the product metal. The samples shall have a spectrochemical response similar to the standards used in preparing the analytical curves. This is ensured by

<sup>3</sup> Fox, G. J., "Determination of As, Sb, and Te in Lead and Lead Alloys Using Hydride Generation Atomic Absorption Spectrometry," *Atomic Spectroscopy*, Vol 11, No. 1, January 1990, p. 13.