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Standard Test Method for Spectrographic Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique¹

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1. Scope

1.1 This test method covers the spectrographic analysis of aluminum and aluminum alloys for the following elements in the ranges indicated:

Element	Concentration Range, %
Silicon	0.02 to 14.0
Copper	0.001 to 10.0
Magnesium	0.001 to 10.0
Zinc	0.03 to 8.0
Nickel	0.03 to 3.0
Iron	0.02 to 2.0
Manganese	0.005 to 2.0
Lead	0.03 to 0.7
Bismuth	0.03 to 0.7
Chromium	0.01 to 0.5
Titanium	0.01 to 0.5
Tin	0.01 to 0.5
Beryllium	0.0002 to 0.5
Calcium	0.0005 to 0.1
Sodium	0.0005 to 0.05

1.2 This test method is applicable primarily to the analysis of chill-cast disks, but it may be extended, with proper precautions, to the analysis of samples in other forms on which a flat surface for sparking can be machined. Standards and samples should be of similar composition and metallurgical state; when they are not, corrections must be applied to compensate for these effects.

1.3 *This standard does not purport to address all of the safety problems, 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 ASTM Standards:

- E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis²
- E 116 Practice for Photographic Photometry in Spectrochemical Analysis²
- E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes²
- E 135 Terminology Relating to Analytical Atomic Spectroscopy²
- E 409 Practice for Description and Performance of the Microphotometer²

¹ This test 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 E01.04 on Aluminum and Magnesium.

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² Annual Book of ASTM Standards, Vol 03.06.

E 716 Practices for Sampling Aluminum and Aluminum Alloys for Spectrochemical Analysis²

3. Terminology

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

4. Summary of Test Method

4.1 A flat sample surface is excited by a controlled spark 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 lines are determined photometrically. Concentrations of the elements are read from analytical curves relating log intensity ratio to concentration.³

5. Apparatus

5.1 Sample Preparation Equipment:

5.1.1 *Sample Molds*—Refer to Practices E 716.

5.1.2 *Lathe*—Refer to Practices E 716.

5.2 *Electrode Cutter*, for shaping electrodes to the configuration described in 6.1.

5.3 *Excitation Source*, providing a controlled spark with the parameters described in Section 10.

5.4 *Excitation Stand*, a Petrey stand to accommodate a flat disk electrode in opposition to a graphite counter electrode. The lower support has a spring clip for holding the graphite rod vertically. The upper electrode supports the sample. The top surface of this support is in a horizontal or slightly inclined position, so arranged that an extension of the plane of this surface passes through the top of the condensing lens and the center of the spark column is on the optical axis.

5.5 *Spectrograph*—Almost any of the commercial instruments offered for metallurgical work may be applied to this method. Of the prism instruments, the large quartz Littrow or Wadsworth types are preferred, although the medium Cornu type is acceptable. Grating spectrographs having a 1.5-m focal length for 960 grooves/mm grating, a 2-m focal length for a 600 grooves/mm grating, or a 3-m focal length for a 600 grooves/mm grating are satisfactory.

5.6 *Microphotometer*, conforming to the requirements of Practice E 409.

³ This test method is based largely on the following material:

Churchill, J. R., *Selected Topics in Modern Instrumental Analysis*, edited by David F. Boltz, Prentice-Hall, Inc., Chapter 5, 1952, pp. 160-235.

Churchill, J. R., "Techniques of Quantitative Spectrographic Analysis," *Industrial and Engineering Chemistry*, Analytical Edition, IECIA Vol 16 1944, pp. 653-670.

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

5.8 *Calculating Equipment* shall be provided for converting microphotometer readings to percentage concentrations. Two types that have been used satisfactorily are the Dunn-Lowry calculator³ and the multiple determination calculating rule.³

6. Materials

6.1 *Counter Electrode*, high-purity graphite rod, 6.15 mm in diameter, capable of being machined to conform to Type C-5a as given in Practice E 130. Remachine the rods before each sparking.

6.2 *Iron Rod*, for emulsion calibration.

6.3 *Photographic Emulsion*, suitable for the spectral range covered.⁴ Plate or film may be used.

6.4 *Photographic Processing Solutions*, as given in Practices E 115.

7. Standards

7.1 *Secondary Standards*—Reference materials suitable for use in this test method are available commercially.⁵

7.2 *Tertiary Standards*—Standards of local origin, usually referred to as tertiary standards, are required when appropriate secondary standards are not available. These include chill-cast disks, sections of extrusions, sheet samples, and castings standardized by other appropriate, usually independent, methods. Such tertiary standards are often required when the analytical sample differs metallurgically from available secondary standards.

8. Preparation of Samples

8.1 *Chill-Cast Disks*:

8.1.1 *Molten Metal*—Refer to Practices E 716.

8.1.2 *Fabricated and Cast Products*—Refer to Practices E 716.

9. Electrode System

9.1 Insert a freshly cut counter electrode in the Petrey stand and adjust to an analytical gap of 3.0 mm. Place the sample, machined side down on the Petrey stand so that the spark will impinge midway between the edge of the sample and the periphery of the central recess. In the case of a sample having a peripheral sprue, such as Type A in Fig. 1, orient the sample so that the spark falls within the 2 to 4 o'clock or the 8 to 10 o'clock sectors with the sprue location assumed as the 12 o'clock position. If the sample has been previously sparked, position it so that the two sparked areas do not touch. Clamp the sample firmly in place so that good electrical contact with the Petrey plate is established.

⁴ Eastman Kodak Spectrum Analysis No. 1, 103-F, and I-N have been found satisfactory for wavelength ranges 2200 to 4000 Å, 2200 to 6000 Å, and 2200 to 8000 Å, respectively.

⁵ *Report of Available Standard Samples, Reference Samples, and High-Purity Materials for Spectrochemical Analysis, ASTM DS 2, ASTM, 1916 Race St., Philadelphia, PA 19103, 1964.*

10. Excitation and Exposure

10.1 Produce and record the spectra in accordance with the following conditions:

10.1.1 *Electrical Parameters*—See Table 1.

NOTE 3—Other excitation units can be used, provided it can be shown that equivalent precision and accuracy are obtained. Other units may include rectified or nonrectified high-voltage spark units controlled by air-interrupted or motor-driven synchronous auxiliary gaps.

10.1.2 *Exposure Conditions*:

Spectral region, Å	2300 to 5900
Slit width, μm (Note 4):	
Prism spectrograph	30 to 60
Grating spectrograph	25 to 80
Preburn, s	5 to 10
Exposure, s	10 to 30
	(Note 5)
Filters	(Note 6)

NOTE 4—On prism instruments, the maximum slit width is limited by interference effects, and the width chosen should give a smooth, single-maximum contour on microphotometric scanning. On grating instruments, the main factor limiting slit width is line-to-background ratio. A slit width of 80 μm is satisfactory for most determinations, but slit widths as small as 25 μm may be used under this method when higher line-to-background ratios are desired.

NOTE 5—A minimum exposure of 10 s is required to obtain representative radiation. Exposure time, filter transmittances, and optical adjustments affecting line density shall be selected to give suitable line transmittances.

NOTE 6—Neutral filters, grating apertures, collimator apertures, and external lenses of various focal lengths may be used for the purpose of adjusting optical speed. However, the effects of these variables on the sampling of the radiation and on the uniformity of spectrum lines must be taken into account by the use of standard samples. Fixed or removable filters may be used to obtain more favorable intensity ratios. A removable filter may be used to provide a dual concentration range, thus reducing the number of different lines used.

10.1.3 *Emulsion Calibration Exposure*—Make the exposure for emulsion calibration by exciting pure iron or plain carbon steel relatively free from alloying elements following the conditions described in 10.1.1 and 10.1.2 and in accordance with Practice E 116.

11. Photographic Processing

11.1 Process the emulsion in accordance with Practices E 115.

12. Photometry

12.1 Select analytical lines to be measured from those given in Table 2 (Note 7). A1 3059.93 Å is used as the internal standard line for all determinations of concentrations greater than 0.05 %.

NOTE 7—In selecting an analytical line, consider the interference by other elements, the concentration index, proximity to the internal standard wavelength (A1 3059.93 Å) and the transmittance of the line image under the conditions of test. Select a line from Table 2 which has a concentration index near the concentration expected in the sample, and for which no interferences are indicated for the alloy being analyzed. If the line is found to have too high a concentration index, that is, if the intensity ratio is too low in the spectrum of the sample, select a line of lower concentration index from the table. Similarly, if the concentration index proves too low, select a line of higher concentrations index. The usable ranges of intensity ratio and transmittance shall be governed by the concentration, as follows: