



Standard Practices for Sampling Aluminum and Aluminum Alloys for Spectrochemical Analysis¹

This standard is issued under the fixed designation E 716; 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 practices describe the sampling of aluminum and aluminum-base alloys to obtain a chill-cast disk suitable for quantitative optical emission spectrochemical analysis. The disk in the region to be excited is representative of the melt or product and gives a repeatability of results which approaches that of the reference materials used.

1.2 These practices describe procedures for representative sampling of molten metal, from fabricated or cast products which can be melted, and from other forms which cannot be melted.

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.* Specific precautionary statements are given in 5.1 and 6.2.

2. Referenced Documents

2.1 ASTM Standards:

E 101 Test Method for Spectrographic Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique²

E 227 Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique²

E 401 Practice for Bonding Thin Spectrochemical Samples and Standards to a Greater Mass of Material³

E 607 Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique, Nitrogen Atmosphere³

E 1251 Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Argon Atmosphere, Point-to-Plane, Unipolar Self-Initiating Capacitor Discharge³

3. Summary of Practices

3.1 Molten metal representative of the furnace melt is poured into a specified mold to produce a chill-cast disk. The disk is machined to a specified depth that represents the average composition and produces an acceptable surface for excitation.

3.2 Fabricated, cast, or wrought products are remelted and cast into molds, briquetted and remelted, bonded to more massive material, or excited directly without remelting.

3.3 Special practices are included for the sampling and analysis of aluminum-silicon alloys, containing greater than 14 % silicon.

4. Significance and Use

4.1 These practices, used in conjunction with the following appropriate quantitative optical emission spectrochemical methods, Test Methods E 101, E 227, E 607, and E 1251, are suitable for use in manufacturing control, material or product acceptance, and research and development.

5. Apparatus

5.1 *Ladle*, capable of holding a minimum of 250 g (8.8 oz) of molten metal, with a handle of sufficient length to reach into a furnace, trough, or crucible. The ladle should be lightly coated with a tightly adhering ladle wash that will not contaminate the sample (Note 1).

NOTE 1—**Caution:** Traces of moisture in the coating may cause dangerous spattering.

NOTE 2—A suitable ladle wash may be prepared as follows: Mix 255 g (9 oz) of fine whiting (CaCO_3) with 3.8 L (1 gal) of water and boil for 20 min. Add 127 g (4.5 oz) of sodium silicate solution (40 to 42°Bé) and boil for 30 min. Stir well before using.⁴

5.2 *Sample Molds*, capable of producing homogenous chill-cast disks having smooth surfaces, free of surface pockets and porosity. These castings should have a spectrochemical response similar to the reference materials used in preparing the analytical curves and must have a repeatability from excitation-to-excitation of no more than 2 % relative on major elements.

¹ These practices are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.04 on Aluminum and Magnesium.

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² *Annual Book of ASTM Standards*, Vol 03.05.

³ *Annual Book of ASTM Standards*, Vol 03.06.

⁴ Other proprietary ladle washes such as Dycote, available from Foseco, Inc., P. O. Box 8728, Cleveland, OH 44135; and Zirconite, available from Titanium Alloy Mfg. Co., 111 Broadway, New York, NY 10006, have been found suitable for this purpose.

They must be representative of the melt in the region excited. Several types of molds have been found acceptable:

5.2.1 *Type A*, book mold, is shown in Fig. 1. The advantage of this mold is simplicity and low cost. This mold produces a vertically cast disk with the sprue on its edge. The mold dimensions are such as to produce a disk approximately 64 mm (2.5 in.) in diameter by 6 to 8 mm (0.24 to 0.32 in.) in thickness. A circular central recess 15 to 25 mm (0.6 to 1.0 in.) in diameter on one side of the disk facilitates machining of that side in preparation for excitation. It also promotes more uniform freezing of the raised peripheral area. The mold material should be steel or cast iron and should weigh approximately 2 to 3 kg (5 to 7 lb).

5.2.2 *Type B*, center-pour mold, is shown in Fig. 2. The advantage of this mold is that the sample obtained may be excited around the entire annular area. This mold produces a horizontally cast disk with the sprue over the center on the back side. The mold dimensions are such as to produce a disk approximately 64 mm (2.5 in.) in diameter by 6 to 13 mm (0.24 to 0.50 in.) in thickness. A circular central recess 10 to 20 mm (0.4 to 0.8 in.) in diameter on one side of the disk facilitates machining of that side in preparation for excitation. It also promotes more uniform freezing of the raised peripheral area, but the corresponding raised portion of the mold must not be so large as to restrict the throat for the sprue. A slight taper, 1 to 2 deg, on the hinged portion of the mold facilitates opening when a disk has been cast. The mold material should be steel or cast iron and should weigh approximately 3.5 to 4.5 kg (8 to 10 lb).

NOTE 3—Prepare the surface of the mold cavity to minimize the formation of gas pockets on the surface of the castings and to resist rusting of the mold cavity surface. To do this, blast the inner surface with a sharp grit that cuts rather than peens. The resulting finely roughened face is essential for obtaining a smooth and uniform surface on the cast disk. Next, degrease the mold, place in a cold furnace, and raise the temperature to 400°C (752°F). At this temperature and throughout the remainder of the heating cycle, introduce steam into the furnace. Raise the temperature to 540°C (1004°F) and maintain for 4 h. The resulting black oxide coating is tenacious and of a dull black appearance.

5.2.2.1 *Special Type B Mold*, which produces a disk 6 mm (0.24 in.) thick, is required for undiluted aluminum-silicon alloys containing greater than 14 % silicon.

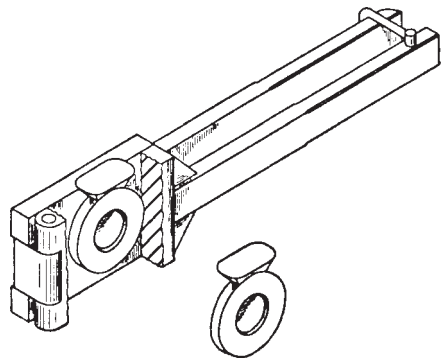


FIG. 1 Type A Mold and Sample

5.2.3 *Vacuum Mold*⁵ is shown in Fig. 4. This mold produces disks that are 38 mm (1.5 in.) in diameter and 13 mm (0.5 in.) thick and weigh approximately 40 g (1.4 oz). The mold consists of a solid copper base and a porous bronze wall in the form of a composite mold insert which is located in a steel mold body. A graphite coated cast iron tip is attached to the mold body by a spring clamp assembly. The vacuum source can be either a small battery-operated vacuum pump or a rubber syringe connected to the mold body.

5.2.4 *Other Types of Molds*—Other molds of different types, materials, and dimensions may be substituted provided that the uniformity of the samples so obtained is comparable to the uniformity of samples obtained from Type A or B molds, and furthermore that such samples have a spectrochemical response similar to the reference materials used for preparing the analytical curve.

5.3 *Lathe*, capable of machining a smooth flat surface and having automatic cross feed. A milling machine may also be used.

5.4 *Tool Bits*—Either alloy steel or cemented carbide is recommended. The best shape of the tool varies with the type and speed of the lathe, but in general, soft metals require less top and side rake than steel. For example, for pure aluminum, a top rake of 0° and a side rake of 0 to 6° should prove satisfactory. Also a side clearance of about 6° and a front clearance of 15° should be satisfactory for all aluminum disk samples. The nose of the tool should be rounded. A tool bit design that has been found satisfactory for most aluminum alloys is shown in Fig. 5.

5.5 *Portable Electric Melting Furnace*,⁶ equipped with a graphite crucible with a minimum capacity of 200 g (7.1 oz) of molten aluminum, and capable of maintaining temperatures for melting aluminum alloys.

6. Materials

6.1 *Graphite Rods*—6.15 by 300-mm (0.242 by 12-in.) spectroscopic electrodes are satisfactory.

6.2 *Phosphorus*, red, amorphous.

NOTE 4—**Caution:** Provide adequate ventilation when phosphorus is added to molten metal.

7. Preparation of Samples

7.1 Molten Metal:

7.1.1 Chill-Cast Disk by Molds A or B:

7.1.1.1 When a furnace or crucible of molten metal is to be sampled, the temperature must be well above the point at which any solid phase could be present. Using the ladle or a separate skimming tool, coated with a dry, tightly adhering mold wash (Note 2) and free of any remaining previous metal, push any dross away from the sampling area. Next, dip the ladle sideways into the clear area well below the surface and stir momentarily. Then turn the ladle upright, and quickly withdraw. Two things are thus accomplished, namely, heating

⁵ A portable Vacuum Sampler, available from Aluminum Company of America, Alcoa Center, PA 15069, has been found suitable for this purpose.

⁶ A Jelrus Handy-Melt furnace and graphite crucible, available from Cole-Parmer Instrument Co., 7425 North Oak Park Ave., Chicago, IL 60648, has been found suitable for this purpose.

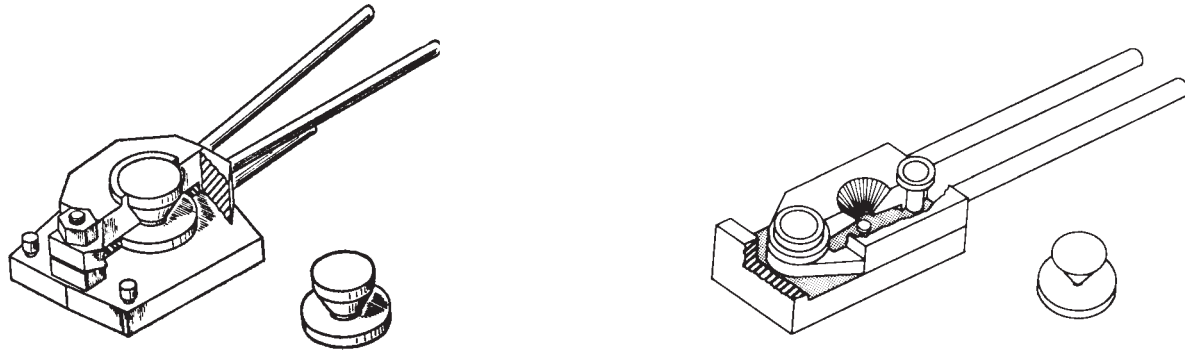
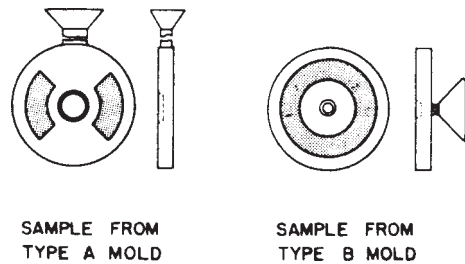


FIG. 2 Type B Molds and Samples



SAMPLE FROM
TYPE A MOLD

SAMPLE FROM
TYPE B MOLD

NOTE 1—Shaded areas acceptable for analysis.

FIG. 3 Samples from Type A and B Molds

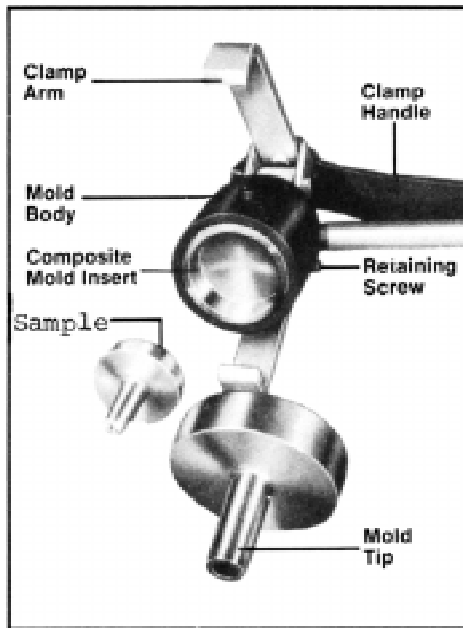


FIG. 4 Mold for Vacuum Cast Samples

the ladle prevents metal freezing on the wall and obtaining metal well beneath the surface minimizes the danger of inclusion of small particles of oxide.

7.1.1.2 Unless the mold is already hot, cast a preliminary disk into the clean mold in order to preheat it and discard this disk. Remove excess metal from the ladle, dip into the molten metal as before, and fill the mold with an even rate of pour which allows the escape of air from the mold. Do not dump the metal into the mold. Avoid overfilling the sprue, otherwise the mold may be difficult to open. Allow the metal to freeze quietly

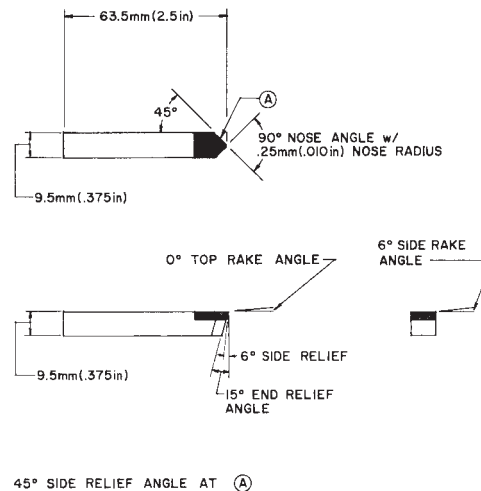


FIG. 5 Tool Bit

without jarring. The surface of the disk must be free of any shrinkage, inclusions, cracks, or roughness. Cut off the sprue and machine the raised peripheral area surrounding the recess to a depth of 14 to 22 % of the original thickness. This depth is important because it corresponds to the composition on the phase diagram that best represents the average composition of the whole disk and therefore the actual composition of the melt. Any other depth may result in a different analysis and therefore cannot be accepted as valid. It is advisable to determine the most appropriate machining depth for the particular disk thickness used. The machined surface must be smooth and free of scuffs, pits, or inclusions. The ideal surface is neither polished nor visibly grooved but should be a surface showing very fine tool marks. More specifically, the ideal surface may be defined as approximately a 1.6×10^{-3} -mm (63- μ in.) standard machine finish. A surface much finer or much coarser may result in an apparent analytical difference. Furthermore, it is important that both sample and reference material have the same machine finish. Note that Type A disks may be excited only in certain areas (7:30 to 10:30 o'clock and 1:30 to 4:30 o'clock positions) while Type B disks may be excited around the entire annular area. For both types, the outer 5 mm (0.2 in.) to the edge and the inner region up to approximately 12-mm (0.48-in.) radius should not be excited. Fig. 3 illustrates both types of samples and the areas suitable for exciting are shaded.

7.1.2 *Chill Cast Disk Using Vacuum Mold*—Skim the dross

from the molten metal as in 7.1.1.1, using a skimming tool. Preheat the cast iron mold tip and attach it to the mold body using the clamp arm assembly. Insert the mold tip into the molten metal and immediately apply the vacuum to draw the metal into the mold cavity. Remove the mold tip from the metal, detach the mold tip from the mold body, and remove the disk. The disks are prepared for analysis by machining a smooth surface on the side opposite the sprue at a depth of 2.0 mm (0.08 in.) below the original surface. Analysis can be made 360° around the disk in the annular area adjacent to the edge, avoiding the center area.

7.1.3 Other Accepted Molds—If molds other than Types A and B or the vacuum mold are used, the same instructions given in 7.1 would apply. In addition, since a mold of different dimensions may result in a different freezing pattern, each new type of mold must be evaluated in order to ascertain the proper depth of machining to represent the true composition of the melt.

7.2 Fabricated and Cast Products:

7.2.1 Chill-Cast Disk by Molds A, B, or the Vacuum Mold—When the metal to be analyzed is in wrought or cast form and a destructive test is applicable, remelt a representative portion of the metal at a temperature well above the liquidus line of the alloy. A clay-graphite or other inert crucible may be used and placed in a convenient laboratory electric furnace. Then cast the melt in one of the molds as described in 7.1. If the sample is in the form of turnings, thin sheet, or other finely divided material, remove grease or any coatings with a suitable solvent and press into a briquet before melting and proceeding as in 7.1. Details of briquet size and formation are not critical to the success of preparing a melt. The largest briquet that can be successfully formed and that will fit into the remelt crucible will obviously speed up the remelt process. Carry out the melting and casting operation as rapidly as possible, and use as large melt as practical to minimize losses of volatile elements.

NOTE 5—Remelting is not satisfactory for the determination of sodium, calcium, and lithium, and some magnesium may also be lost if the melt is overheated or held for an excessive time.

7.2.2 Direct Excitation Without Casting a Sample—When the sample preparation procedures described in 7.1 cannot be followed, for example, where melting would cause loss of a volatile constituent, or where it is otherwise impractical, usually only approximate analyses can be made. (1) The sample must be sufficiently massive to prevent undue heating, (2) it must have a sufficiently flat surface for excitation, and (3) reference materials having a similar spectrochemical response must be available. On sheet and plate samples, machine-off approximately 0.8 mm (0.032 in.) or one fourth of the sample thickness, whichever is the smaller. On other products, machine a flat surface at least 1.3 mm (0.052 in.) below the original surface. Choose the depth, location, and number of areas to be analyzed to provide a representative analysis of the product. In accordance with Practice E 401, thin flat material may also be bonded by means of a heat and electrically conducting epoxy-type adhesive to a more massive section to provide a heat sink.

7.3 Hyper-Eutectic Aluminum-Silicon Alloys (Note 6)

NOTE 6—These procedures are required only for the accurate determination of silicon at levels greater than 14 %. Other elements of interest may be determined satisfactorily without either the addition of phosphorus or dilution with high-purity aluminum.

7.3.1 Analysis Without Dilution:

7.3.1.1 Molten Metal—Heat the metal to be sampled to 760°C (1400°F). Preheat the sampling ladle. Add red phosphorus in a ratio of 1 g (0.036 oz) per 400 g (14.1 oz) of metal to a ladle of the melt. Stir briskly with a graphite rod, skim, and make a preliminary casting, using the special Type B mold producing a 6-mm (0.24-in.) thick sample. Discard the first disk, and make a second disk for analysis. Remove the sprue, and machine the sample to a depth of 1.1 mm (0.044 in.) below the original surface. Using a carbide-tipped tool which has been used less than 30 times, continue to machine to a depth of 1.2 mm (0.048 in.) below the original surface. A400-r/min spindle speed and a 0.68-in. (17-mm) radius/min crossfeed are recommended for the final cut. If equivalent analytical performance can be shown, the use of other materials, such as a diamond-tipped bit, may also be used. Reference materials and samples shall be machined under identical conditions.

7.3.1.2 Cast Products—When the metal to be analyzed is in cast form, remelt the metal and prepare a disk sample as in 7.3.1.1. Carry out the melting and casting operation as rapidly as possible.

7.3.2 Analysis With Dilution:

7.3.2.1 Molten Metal—Sample the molten metal as in 7.1 or 7.3.1.1, omitting the red phosphorus. Weigh this original sample to 0.01 g, and remelt with a similar amount of 99.99 % aluminum in a laboratory electric furnace. Stir thoroughly with a graphite rod, and cast a new sample, using any of the molds described in 5.2. For Types A and B molds, preheat the mold on a hot plate at 177°C, (350°F) and cast a sample for analysis. Make vacuum-cast samples by inserting the mold tip into the molten metal and applying vacuum to draw the metal into the mold cavity.

7.3.2.2 Cast Products—When the metal to be analyzed is in cast form, remelt the original metal and prepare the sample as in 7.3.2.1.

7.3.2.3 Prepare the diluted sample for analysis by removing the sprue, and machining Type A or Type B disks to a depth of 14 to 22 % of the original thickness. Those vacuum cast should be machined to a depth of 2.0 mm (0.08 in.) below the original surface. Analyze the diluted sample, using appropriate reference materials with a similar composition and metallurgical structure. Dilutions with pure aluminum can be made with ratios other than 1:1 in order to match the diluted composition with existing reference materials. Volatile elements such as sodium and calcium can be lost on remelting and should be determined on the original sample.

8. Calculations for Analysis with Dilution

8.1 Calculate the composition of the original sample by multiplying the composition of the diluted sample by the dilution ratio. The dilution ratio is computed as follows:

$$\text{Dilution ratio} = \frac{W_1 + W_2}{W_2} \quad (1)$$

where:

W_1 = weight of 99.99 % Al, and

W_2 = weight of original material to be diluted.

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