



Standard Practices for Sampling and Sample Preparation of Aluminum and Aluminum Alloys for Determination of Chemical Composition by Spectrochemical Analysis¹

This standard is issued under the fixed designation E716; 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 procedures for producing a chill cast disk sample from molten aluminum during the production process, and from molten metal produced by melting pieces cut from products.

1.2 These practices describe a procedure for obtaining qualitative results by direct analysis of product using spark atomic emission spectroscopy.

1.3 These practices describe procedures for preparation of samples and products prior to analysis.

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

1.5 *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 6.1 and 7.2.

2. Referenced Documents

2.1 ASTM Standards:²

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E401 Practice for Bonding Thin Spectrochemical Samples and Standards to a Greater Mass of Material³

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.

Current edition approved Sept. 1, 2010. Published December 2010. Originally approved in 1980. Last previous edition approved in 2002 as E716 – 94 (2002). DOI: 10.1520/E0716-10.

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³ Withdrawn.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

E607 Test Method for Atomic Emission Spectrometric Analysis Aluminum Alloys by the Point to Plane Technique Nitrogen Atmosphere

E1251 Test Method for Analysis of Aluminum and Aluminum Alloys by Atomic Emission Spectrometry

3. Terminology

3.1 For definitions of terms used in this practice, refer to Terminology **E135**.

4. Summary of Practices

4.1 Molten metal representative of the furnace melt is poured or drawn by vacuum 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 analysis by spark atomic emission spectroscopy.

4.2 Pieces of solid aluminum fabricated, cast, or wrought products are remelted and cast into molds or briquetted then remelted and cast into molds.

4.3 Product can be qualitatively analyzed directly without remelting after suitable surface preparation. Product with insufficient mass for direct analysis may be bonded to more massive material prior to analysis.

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

5. Significance and Use

5.1 The practice for taking a sample of molten metal during production and producing a chill cast disk, used in conjunction with the following appropriate quantitative spark atomic emission spectrochemical methods, Test Methods **E607** and **E1251**, is suitable for use in manufacturing control or certifying, or both, that the entire lot of alloy sampled meets established composition limits.

5.2 The practice for melting a piece of a product to produce a chill cast disk analyzed in conjunction with the following appropriate quantitative spark atomic emission spectrochemical methods, Test Methods **E607** and **E1251**, is suitable, if a

representative sample is taken, for determining if the piece sampled meets Aluminum Association composition limits.

5.3 The practice for direct analysis of product is suitable for determining an approximate composition of the piece analyzed

6. Apparatus

6.1 *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 serve in part to prevent contamination of the sample and also prevent contact of molten aluminum with metal oxides, that is, rust. (**Warning**—Traces of moisture in the coating may cause dangerous spattering.)

NOTE 1—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 °Bé to 42 °Bé) and boil for 30 min. Stir well before using.

NOTE 2—Molten aluminum in contact with rust may initiate a thermite reaction.

6.2 *Sample Molds* shall be capable of producing homogeneous chill-cast disks having smooth surfaces, free of surface pockets and porosity. These chill cast disks should have a spectrochemical response similar to the reference materials used in preparing the analytical curves and should at least have a spark to spark repeatability of no more than 2 % relative on major elements. They must be representative of the melt in the region excited. Several types of molds have been found acceptable:

6.2.1 *Type B*,⁴ center-pour mold, is shown in Fig. 1. 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 of one side. The mold dimensions are such as to produce a disk approximately 50 mm to 64 mm (1.97 in. to 2.5 in.) in diameter by 6 mm to 13 mm (0.24 in. to 0.50 in.) in thickness. A circular central recess 10 mm to 20 mm (0.4 in. 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°, 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 kg to 4.5 kg (8 lb to 10 lb).

6.2.2 *Vacuum Mold*⁵ is shown in Fig. 2. 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 is typically a rubber syringe bulb connected to the mold body.

6.2.3 *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 sufficient for the intended use of the results. Furthermore such samples should have a sizeable area that represents the bulk composition of the entire sample and have a spectrochemical response similar to the reference materials used for preparing the analytical curve.

6.3 *Lathe or Milling Machine*, capable of machining a smooth flat surface and capable of repeating the selected depth of cut to within ± 0.013 mm (± 0.005 in.).

6.4 *Tool Bits*—Diamond tipped, alloy steel, or cemented carbide bits are recommended. The best shape of the lathe tool varies with the type and speed of the lathe. A tool bit design that has been found satisfactory for most aluminum alloys is shown in Fig. 3.

6.5 *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.

7. Materials

7.1 *Graphite Rods*, of suitable diameter for stirring the molten aluminum.

7.2 *Phosphorus*, red, amorphous, or other suitable source of phosphorus for grain refining of high silicon alloys during the remelt procedure. (**Warning**—Provide adequate ventilation when phosphorus is added to molten metal.)

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.

⁴ Type B molds, available from Machine and Welding Incorporated, 713 Fortune Crescent, Kingston, ON Canada K7P 2T4, have been found suitable for this purpose.

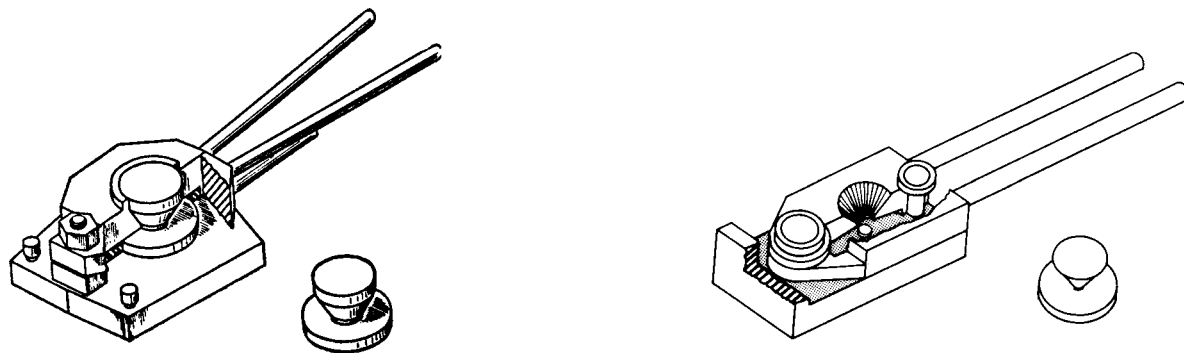


FIG. 1 Type B Mold

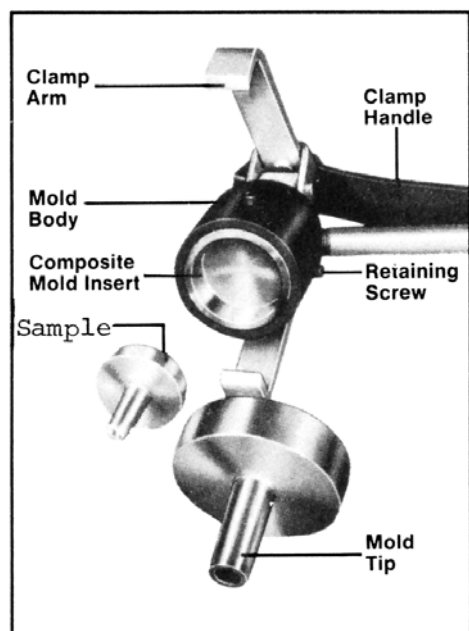


FIG. 2 Mold for Vacuum Cast Samples

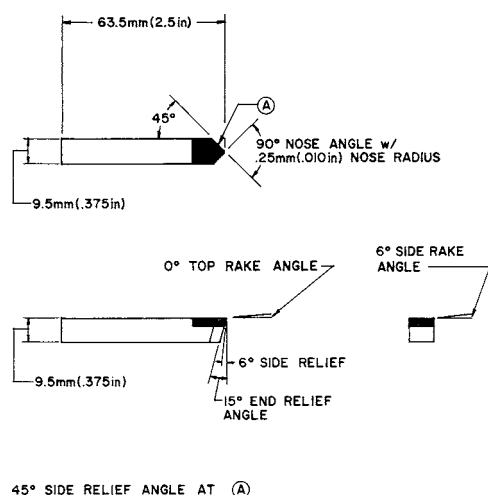


FIG. 3 Tool Bit

8. Preparation of Samples

8.1 Molten Metal:

8.1.1 Chill-Cast Disk by Molds A or B:

8.1.1.1 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 1) and free of any remaining previous metal, push as much dross as possible 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 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.

8.1.1.2 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 without jarring. The surface of the disk must be free of any shrinkage, inclusions, cracks, or roughness.

8.1.2 *Chill Cast Disk Using Vacuum Mold*—Skim the dross from the molten metal as in 8.1.1.1, using a skimming tool. Attach the cast iron mold tip to the mold body using the clamp arm assembly. Squeeze the rubber syringe bulb while immersing the mold into the metal to prevent oxide skim from entering the mold tip. Wait about five seconds to allow time for preheating the sampler. Release the rubber syringe bulb to apply vacuum that will draw the metal into the sampler. Remove the mold tip from the metal, detach the mold tip from the mold body, and remove the disk. The surface of the disk must be free of any shrinkage, inclusions, cracks, or roughness.

8.1.3 Machine the disk to appropriate depth for the particular sampler dimensions. Typically a depth of between 14 % and 22 % of the original thickness 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. It is advisable to determine the most appropriate machining depth for the particular disk thickness used and to target and tightly control that specific depth. Machining to different depths may result in a different analysis and therefore cannot be accepted as valid.

NOTE 3—Aluminum samples shall not be prepared by sanding or grinding. Sanding or grinding tends to smear the relatively soft aluminum phase over the harder constituent phases or cause hard grains to be torn from the sample and may cause biased results for spark atomic emission spectrometry.

8.1.3.1 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} –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. Analysis can be made 360° around the disk in the annular area adjacent to the edge, avoiding the center area.

8.1.4 *Other Accepted Molds*—If molds other than Type B or the vacuum mold are used, the same instructions given in 8.1 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.

8.2 Remelting and Casting a Sample from Fabricated and Cast Products:

8.2.1 *Chill-Cast Disk by Type B Mold, 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 a portion of the melt in one of the

molds as described in 8.1. 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 8.1. 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. Follow the procedures in 8.1.1-8.1.3 for preheating each particular sampler type.

NOTE 4—Analysis of samples can be used to determine compliance with composition analysis for the piece sampled if a representative sample is obtained. Direct analysis of samples obtained from fabricated and cast aluminum product shall not be used for determining compliance with composition specifications. Cast lot composition should be determined using samples taken during pouring of castings or ingots.

NOTE 5—Remelting is not satisfactory for the determination of volatile elements such as sodium, calcium, lithium, strontium, and some magnesium may also be lost if the melt is overheated or kept molten for an excessive time.

8.2.2 Direct Analysis of Wrought or Cast Products—Pieces of wrought or cast aluminum product can be analyzed directly on the surface when the sample preparation procedures described in 8.1 cannot be followed, for example, when there is insufficient sample for remelting and casting a disk or where melting would cause loss of a volatile constituent, or where it is otherwise impractical. The results should be considered qualitative and not quantitative. Segregation of elements during solidification and metalurgical differences between the product and the reference materials used for calibration may cause biased results. Direct analysis of wrought or cast aluminum products should not be used to determine compliance with composition requirements.

8.2.2.1 The sample must be sufficiently massive to prevent undue heating during analysis, and it must have a sufficiently flat surface for excitation. Further, 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 E401, 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. In all cases the prepared area must be large and flat enough to form a good seal with the spectrometer spark stand table.

8.3 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.

8.3.1 Analysis Without Dilution:

8.3.1.1 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. A 400-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.

8.3.1.2 Cast Products—When the metal to be analyzed is in cast form, remelt the metal and prepare a disk sample as in 8.3.1.1. Carry out the melting and casting operation as rapidly as possible. (See Note 6.)

8.3.2 Analysis With Dilution:

8.3.2.1 Molten Metal—Sample the molten metal as in 8.1 or 8.3.1.1, 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 6.2. For Type 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.

8.3.2.2 Cast Products—Metal which is in cast form, should be remelted and a disk sample prepared as in 8.3.2.1. Complete the melting and casting operation as rapidly as possible. (See Note 6.)

8.3.2.3 Prepare the diluted sample for analysis by removing the sprue, and machining Type B disks to a depth of 14 % to 22 % of the original thickness. It is advisable to determine the most appropriate machining depth for the particular disk thickness used and to target and tightly control that specific depth. Machining to different depths may result in a different analysis and therefore cannot be accepted as valid. Vacuum cast samples 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.

9. Calculations for Analysis with Dilution

9.1 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 % aluminum, and

W_2 = weight of original material to be diluted.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the ASTM website (www.astm.org/COPYRIGHT/).
