



Designation: E716 – 16 (Reapproved 2021)^{ε1}

Standard Practices for Sampling and Sample Preparation of Aluminum and Aluminum Alloys for Determination of Chemical Composition by Spark Atomic Emission Spectrometry¹

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.

^{ε1} NOTE—An editorial change was made to 8.3 in January 2022.

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 spectrometry.

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 concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 6.1 and 7.2.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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. Referenced Documents

2.1 *ASTM Standards:*²

B985 Practice for Sampling Aluminum Ingots, Billets, Castings and Finished or Semi-Finished Wrought Aluminum Products for Compositional Analysis

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 Material (Withdrawn 1995)³

E607 Test Method for Atomic Emission Spectrometric Analysis Aluminum Alloys by the Point to Plane Technique Nitrogen Atmosphere (Withdrawn 2011)³

E1251 Test Method for Analysis of Aluminum and Aluminum Alloys by Spark 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 spectrometry.

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

*A Summary of Changes section appears at the end of this standard

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 (CaCO₃) 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 Mold*⁴ center-pour mold, is shown in Fig. 1. 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). A special Type B mold is recommended for hypereutectic aluminum-silicon alloys. It produces the thinner samples 13 mm (0.24 in.) thick.

NOTE 3—About sample molds: Previously two relatively simple types of massive iron or steel sample molds were considered suitable, Type A and Type B. Type A molds produced vertical chill cast samples with the sprue and riser on the edge of the sample, as opposed to the Type B which produces a horizontal chill cast sample with the sprue and riser on the back of the sample. The Type A sampler was later found to not produce a repeatable sparking surface, even in the restricted sparking areas. The Type A mold was removed from the list of recommended conventional molds. Because many people are familiar with the terms “Type A” and “Type B” molds, reference to “Type B” mold remains in the text of this standard even though reference to the “Type A” no longer appears.

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

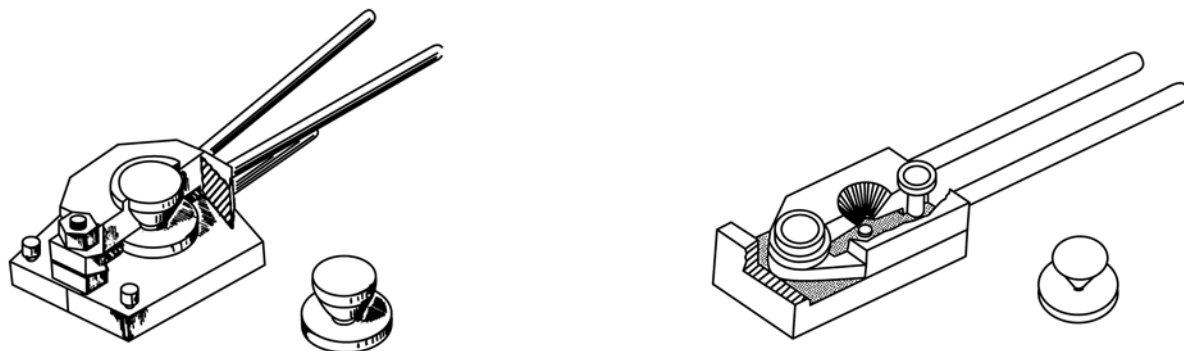


FIG. 1 Type B Mold

6.2.2 *Scissor Mold*⁵ is shown in Fig. 2. This mold produces disks that are 60 mm (2.4 in.) in diameter and 13 mm (0.5 in.) thick and weigh approximately 100 g (3.5 oz). The mold consists of two halves weighing about 3 kg (6.6 lbs). The halves are connected by a pivot bolt which allows the halves to function as scissors. When the upper half with the sprue hole is moved to cover the sample cavity in the lower half, molten metal is poured into the riser cup, through the sprue hole into the sample cavity. After the metal has frozen, the user holds the steel spring heat dissipater surrounding one handle and strikes the other handle on the ground causing the upper half to pivot away and shear off the riser at the sprue. The sample and the sprue can then be easily removed.

6.2.3 *Vacuum Mold* is shown in Fig. 3. 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.

NOTE 4—This sampler is made by Alcoa and is recommended in previous issues of this standard. This device is no longer commercially available from Alcoa, but the description remains in this standard because it is still used within the aluminum industry.

6.2.4 *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 sufficient for the intended use of the results. Furthermore such samples should 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, or 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. 4.

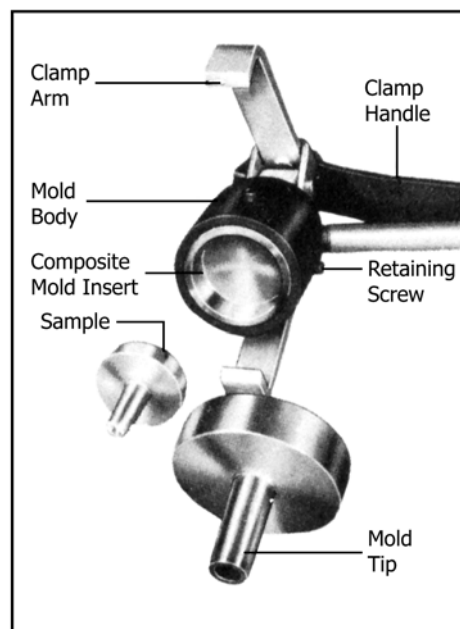


FIG. 3 Mold for Vacuum Cast Samples

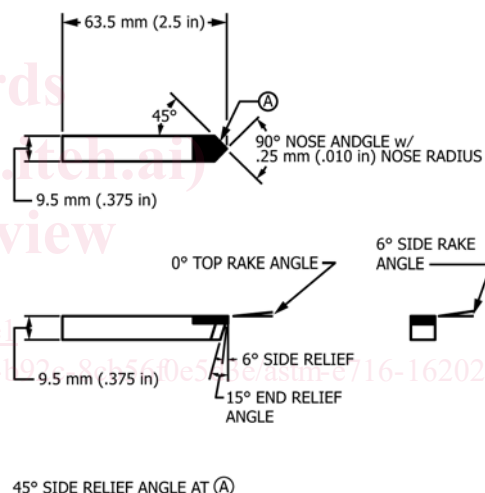


FIG. 4 Tool Bit

6.5 *An Electric Melting Furnace*, using a clay or graphite crucible with a minimum capacity of 100 g (3.5 oz) of molten aluminum and capable of maintaining temperatures for melting aluminum alloys.

7. Materials

7.1 *Graphite Rods*,⁶ for stirring the molten aluminum.

7.2 *A source of phosphorus*, for grain refining of high silicon alloys before spectrometric analysis. Grain refining of the primary silicon is important for an accurate analysis of silicon.

NOTE 5—Previous versions of these standard practices specified the addition of red phosphorus to the sample ladle of molten hypereutectic Al-Si aluminum alloy. The requirement of the addition of red phosphorus

⁵ A scissor mold available from Herschal Products, 3778 Timberlake Dr., Richfield, OH 44286 has been found suitable for this purpose.



FIG. 2 Scissor Mold

⁶ Graphite stirring rods are available from Budget Casting Supply LLC, 20811 Upper Hillview Dr., Sonora, CA 95370

was based on the assumption that the larger quantity of molten aluminum alloy (which was sampled by the ladle) had not previously been grain refined with phosphorus. Red phosphorus is no longer available without a special license. The recommended replacement grain refining additive is a copper-8 % phosphorus alloy. The entire molten bath should be refined before the sample ladle removes the smaller amount of molten metal for the sample. If the sample must be taken prior to grain refinement of the main bath, either a small amount of copper-8 % phosphorus alloy should be added to the ladle, with the expectation that the copper concentration of the spectrometric analysis will be wrong, or a phosphorus chemical compound without an interfering element should be added. This other compound will be hazardous and must be handled carefully by an experienced chemist. A suitable compound is phosphorus penta-chloride (PCl₅). In either case, the phosphorus recovery after the alloying addition will be low, in a range of 15 % to 40 %.

8. Preparation of Samples

8.1 Molten Metal:

8.1.1 When 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.

8.1.2 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.

8.1.3 *Chill Cast Disk Using Vacuum Mold*—Skim the dross from the molten metal as in 8.1.1, 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 skin 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.

8.1.4 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 6—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.

8.1.4.1 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} -mm (63- μ m.) 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.

8.1.5 *Other Accepted Molds*—If molds other than Type B, the scissor mold, 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, the Scissor 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 as described in Practice B985 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 briquette before melting and proceeding as in 8.1. Details of briquette size and formation are not critical to the success of preparing a melt. The largest briquette 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 – 8.1.4 for preheating each particular sampler type.

NOTE 7—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 8—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