Designation:E716–94(Reapproved2002) Designation: E716 – 10

Standard Practices for Sampling Aluminum and Aluminum Alloys for Spectrochemical AnalysisSampling and Sample Preparation of Aluminum and Aluminum Alloys for Determination of Chemical Composition by Spectrochemical Analysis[1]

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

1.1These 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.

1.2These 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

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 5.1 6.1 and 6.27.2.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E101Test Method for Spectrographic Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique

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

E227Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique

E401 Practice for Bonding Thin Spectrochemical Samples and Standards to a Greater Mass of Material[3]

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. Summary of Practices

3.1Molten 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.

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

[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 Oct. 10, 2002.Sept. 1, 2010. Published December 2002.2010. Originally approved in 1980. Last previous edition approved in 19942002 as E716 – 94 (2002). DOI: 10.1520/E0716-94R02.10.1520/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.

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

4.1These practices, used in conjunction with the following appropriate quantitative optical emission spectrochemical methods, Test Methods E101, E227,

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 and E1251, are suitable for use in manufacturing control, material or product acceptance, and research and development.

5., 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

5.1

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 not contaminate the sample (Note 1). , 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.)

NOTE1—**Caution:**Traces of moisture in the coating may cause dangerous spattering. 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.

NOTE2—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.

5.2 *2—Molten aluminum in contact with rust may initiate a thermite reaction.*

6.2 *Sample Molds*, shall be capable of producing homogenous chill-cast disks having smooth surfaces, free of surface pockets and porosity. These castings chill cast disks should have a spectrochemical response similar to the reference materials used in preparing the analytical curves and must should at least have a repeatability from excitation-to-excitation 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:

5.2.16.2.1 *Type AType B*, book [4] center-pour 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).

[4] A portable Vacuum Sampler, available from Aluminum Company of America, Alcoa Center, PA 15069, 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.

~~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.~~

~~5.2.3*Vacuum Mold*~~. 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. 4~~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 ~~can be either~~ is typically a ~~small battery-operated vacuum pump or a~~ rubber syringe bulb connected to the mold body.

~~5.2.4~~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 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~~—*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*~~—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~~

~~[5] 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.~~

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

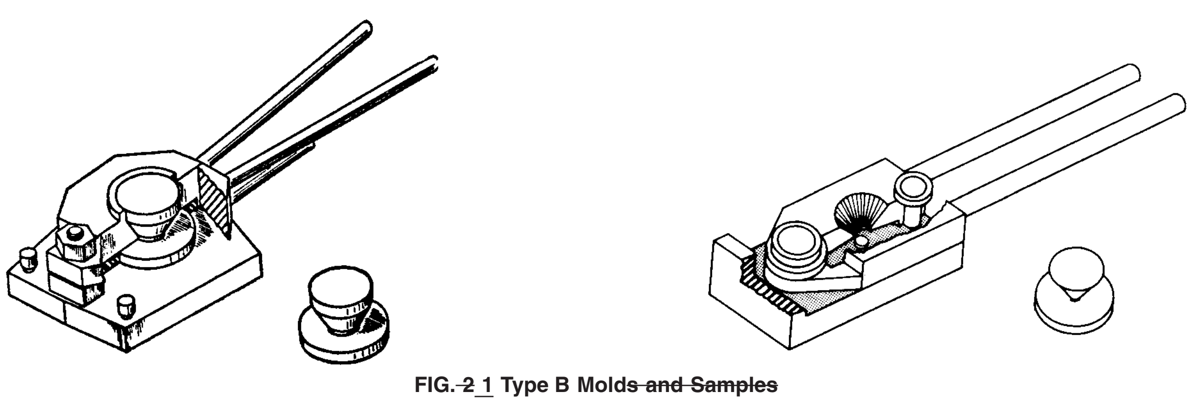

FIG. ~~2~~ 1 Type B Molds ~~and Samples~~

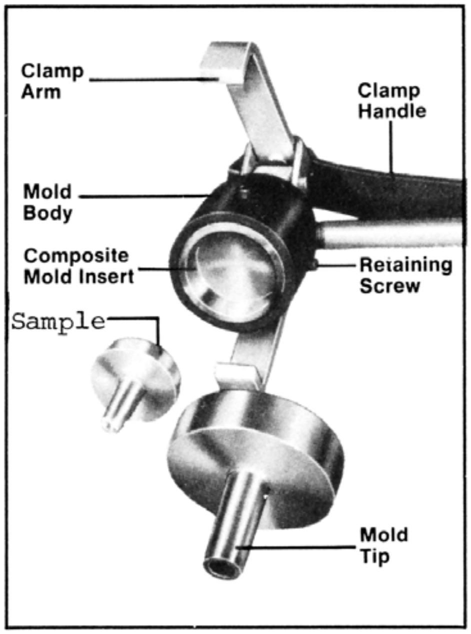

FIG. ~~4~~ 2 Mold for Vacuum Cast Samples

~~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~~—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.

~~5.5~~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,~~aluminum and capable of maintaining temperatures for melting aluminum alloys.

~~6.~~7. Materials

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

~~6.2~~

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

7.2 *Phosphorus*~~, red, amorphous.~~

[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.

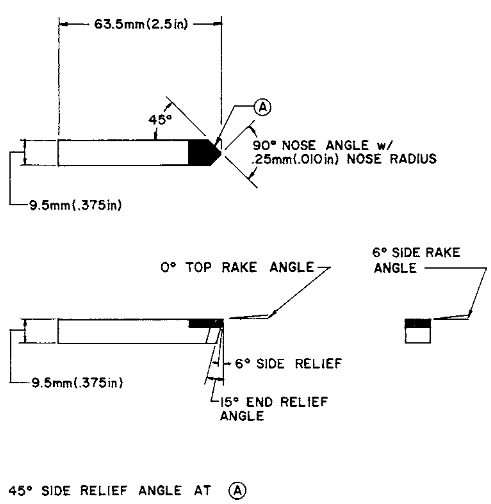

FIG. ~~5~~ 3 Tool Bit