



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.

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 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

[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](#)

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 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.

[and Standards to a Greater Mass of Material \(Withdrawn 1995\)³](#)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.

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.

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

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 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.

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

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.

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

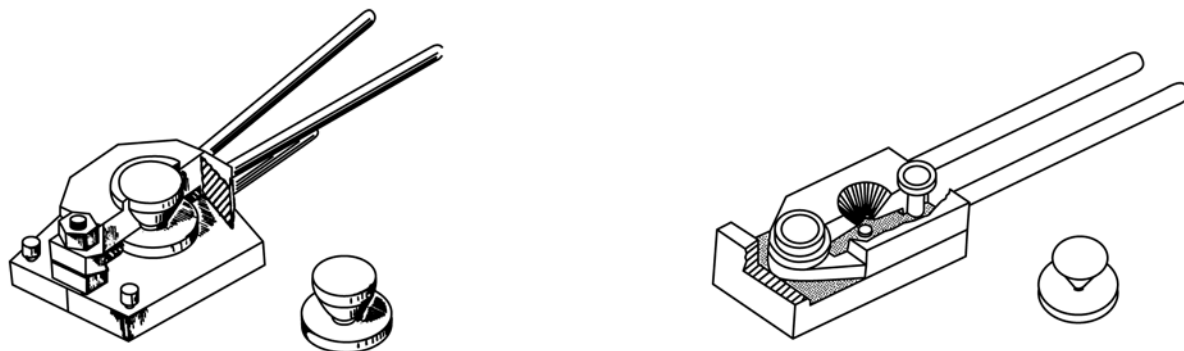


FIG. 1 Type B Mold



FIG. 2 Scissor Mold

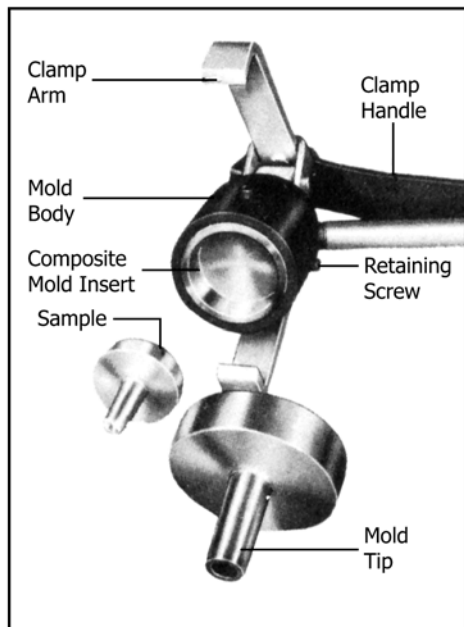


FIG. 3 Mold for Vacuum Cast Samples

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.

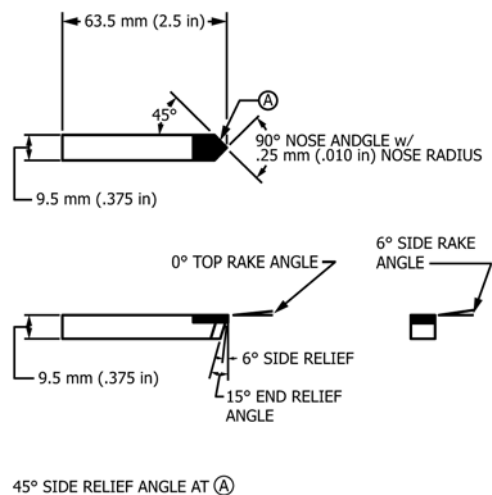


FIG. 4 Tool Bit

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 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

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

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 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.

8.1.5 *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 qualitative and not quantitative. Segregation of elements during solidification and metallurgical differences between the product and the reference materials used for calibration may cause biased results. Direct analysis of wrought or cast aluminum products shall 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. Aluminum is not as thermally conductive as pure copper. Nonetheless, a thick, pure copper disk may be kept on hand to act as a heat sink by

placing it behind the sheet of aluminum (away from the analytical electrode and thus, the spark), provided that the aluminum sheet is thick enough to not burn through and excite the copper during the sparking.

8.3 Hyper-Eutectic Aluminum-Silicon Alloys—A special Type B mold is recommended here. The 13 mm (0.24 in.) thick sample freezes more quickly than the thicker samples. This assists the phosphorus addition in producing fine grain primary silicon (**Note 9**).

NOTE 9—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. Phosphorus is a means of refining the excess primary silicon particles (that excess of silicon which is not in the Al-Si eutectic). Phosphorus additions are usually made as copper-phosphorus, or other strictly metallic additions. Elemental phosphorus and many suitable phosphorus salts are becoming increasingly difficult to obtain. Although the beneficial effects of added phosphorus can start to fade after the first addition, many foundries make this addition early just to get the correct spectrometric analysis, and then add more refining material as needed when starting to pour the heat. If phosphorus is to be added to the small, unrefined molten sample, it must be of a compound which will not contaminate the analysis of the other elements in the alloy, or the analysis of that element must be ignored.

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 the phosphorus refiner (if the melt is not already refined) and stir briskly with a graphite rod. Skim the melt, 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. 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, obtain a representative sample following Practice **B985**, and 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 9**.)

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 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**. 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 9**.)

8.3.2.3 Prepare the diluted sample for analysis by removing the sprue, and machining 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:

$$\text{Dilution ratio} = \frac{M_1 + M_2}{M_2} \quad (1)$$

where:

M_1 = mass of 99.99 % aluminum, and
 M_2 = mass of original material to be diluted.

10. Keywords

10.1 aluminum; aluminum alloys; atomic emission spectrometry; chemical composition; sample preparation; sampling; spark atomic emission spectrometry; specimen preparation; spectrometric analysis; spectrometry

SUMMARY OF CHANGES

Committee E01 has identified the location of selected changes to this standard since the last issue (E716 – 10) that may impact the use of this standard. (Approved June 1, 2016.)

- (1) Section **2.1**: Added Practice **B985**.
- (2) Footnote 4: Added the name Danton to complete the name “Machine and Welding.” This is the full and correct name. (Similar molds are available from other sources.)
- (3) Section **6.5**: Changed description of the furnace and crucibles to products that any lab can find in a lab supply catalog.
- (4) Footnote 6: Removed reference to the Jelrus “portable” electric furnace, which is no longer available. User can buy any furnace and crucibles.
- (5) **Note 3**: Added explanation of continued reference to the Type B sample mold in light of the no longer recommended Type A mold.

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