



Standard Practice for Sampling Magnesium and Magnesium Alloys for Spectrochemical Analysis¹

This standard is issued under the fixed designation B953; 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 This practice describes the sampling of magnesium and magnesium-base alloys to obtain a chill-cast sample suitable for quantitative atomic emission spectrochemical analysis. The disk in the region to be excited is representative of the melt and gives a repeatability of results that approach that of the reference materials used.

1.2 This practice describes the procedure for representative sampling of molten metal.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 5.1.

2. Referenced Documents

2.1 *ASTM Standards:*²

B954 Test Method for Analysis of Magnesium and Magnesium Alloys by Atomic Emission Spectrometry

B881 Terminology Relating to Aluminum- and Magnesium-Alloy Products

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

E1257 Guide for Evaluating Grinding Materials Used for Surface Preparation in Spectrochemical Analysis

3. Summary of Practice

3.1 Molten metal representative of the furnace melt is poured directly into a specified mold (described in 5.2.1) to

¹ These practices are under the jurisdiction of ASTM Committee B07 on Light Metals and Alloys and are the direct responsibility of Subcommittee B07.04 on Magnesium Alloy Cast and Wrought Products.

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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.

produce a chill-cast disk. The disk is machined to a specific depth that represents the average melt composition and produces an acceptable surface for excitation.

3.2 Fabricated, cast, or wrought products may be excited directly without remelting; however, accuracy of results may suffer in highly alloyed samples due to the potential for elemental segregation.

4. Significance and Use

4.1 This practice, used in conjunction with the following quantitative atomic emission spectrochemical test method, B954, is suitable for use in manufacturing control, material or product acceptance, certification, and research and development.

5. Apparatus

5.1 *Ladle*, capable of holding a minimum of 400 g (7.7 oz) of molten metal, with a handle of sufficient length to reach into a furnace, trough, or crucible sufficiently deep to obtain a sample representative of the melt being cast. The ladle may be lightly coated with a tightly adhering ladle wash that will not contaminate the sample. Boron nitride can be applied at elevated temperatures to form a tenacious coating. **Warning**—Trace moisture in the coating or on the tool may cause dangerous spattering. Preheat all sampling tools and molds prior to use.

5.2 *Sample Molds*, capable of producing homogeneous 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 alloying elements. They must be representative of the melt in the region excited. Several types of molds have been found acceptable:

5.2.1 *Type A, open cavity mold*, is shown in Fig. 1. The advantage of this mold is its simple design and ability to produce a sample disk that can be excited around the entire annular area. Mold dimensions can be modified to produce a disk size ranging from approximately 44 to 64 mm (1.75 to 2.5 in.) in diameter by 10 to 15 mm (0.4 to 0.6 in.) in thickness. A circular central recess 10 to 20 mm (0.4 to 0.8 in.) in diameter

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

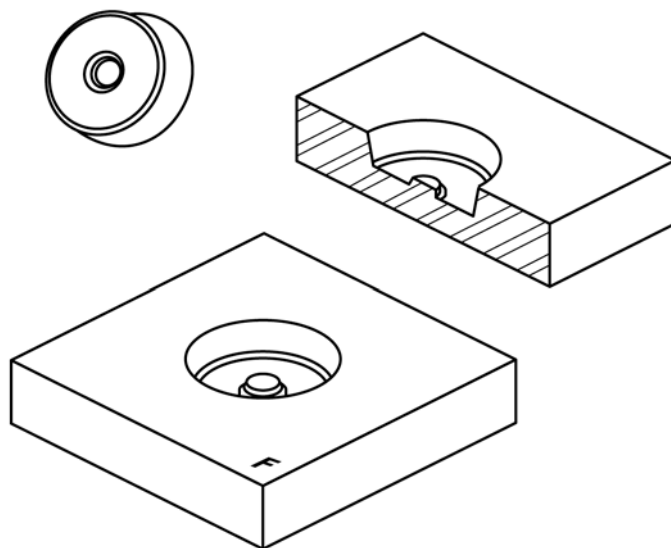


FIG. 1 Type A Mold – Open Cavity Mold and Sample

on one side of the disk (analytical side) facilitates machining of that side in preparation for excitation. It also promotes a more uniform freezing of the raised peripheral area. The mold material should be mild steel and should weigh approximately 2 to 3 kg (4.5 to 6.5 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 backside. The mold dimensions are such as to produce a disk approximately 64 mm (2.5 in.) in diameter by 13 mm (0.5 in.) in thickness. A circular central recess 10 to 13 mm (0.4 to 0.5 in.) in diameter on one side of the disk (analytical side) 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 mild steel and should weigh approximately 3.5 to 4.5 kg (8 to 10 lb).

5.2.3 *Type U, U-Block mold*, is shown in Fig. 3. This side gated block mold produces a cylindrical block sample of approximately 40 mm (1.6 in.) in diameter by 60 mm (2.4 in.) high. The mold is placed on top of a massive 150 mm (5.9 in.) diameter by 50 mm (2.0 in.) thick copper chill plate with a knurled working surface. The mold material is mild steel. The downsprue directionally choked side gate promotes a controlled fill. The strong directional solidification provided by the

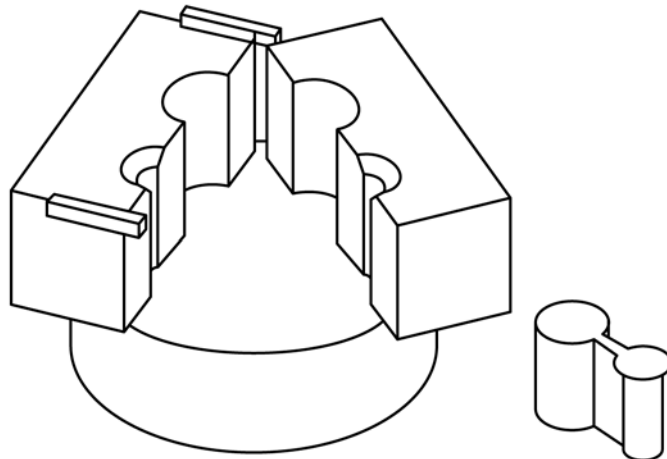


FIG. 3 Type U Mold – U-Block Mold and Sample

chill plate produces a spectrochemically homogenous sample for at least the first third and usually up to two thirds of the length of the U-block sample from chill the cast face (Note 1).

NOTE 1—To improve the performance and durability of the molds described above, 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

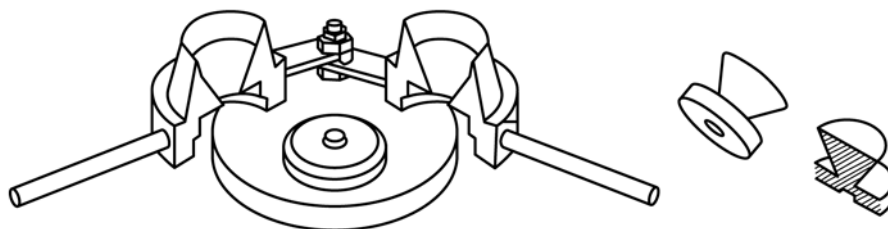


FIG. 2 Type B Mold – Center Pour Mold and Sample

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.

5.2.4 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 the Type A, B, or U 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 on the reference materials and samples. A milling machine may also be used.

5.4 Tool Bits, either alloy steel, carbide-tipped, or carbide insert tool bits are recommended. The best shaped of tool varies with the type and speed of the lathe, but in general, soft metals require less top and side rake than steel.

5.5 Metallographic Polisher/Grinder, A metallographic polisher/grinder may also be used to prepare the sample surface provided care has been taken in selecting a non-contaminating abrasive compound. Metallographic grade wet/dry silicon carbide discs of 120 grit or higher will produce a good sample surface with essentially no silicon carryover to the sample. This must be verified by making a comparison between freshly prepared surfaces on a polisher/grinder and that of a lathe or milling machine. Reference Guide [E1257](#) for a description of contamination issues with various abrasive compounds.

6. Preparation of Samples

6.1 Molten Metal:

6.1.1 Chill-Cast Disk by Molds A, B, or U:

6.1.1.1 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, optionally coated with a dry, tightly adhering mold wash (described in [5.1](#)) 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 the ladle prevents metal freezing on the wall and obtaining metal well beneath the surface minimizes the risk of inclusion of small particles of oxide.

6.1.1.2 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 that allows the escape of air from the mold. Do not dump the metal into the mold. Avoid overfilling the sprue, if applicable, 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.

6.1.1.3 For samples obtained from the Type A mold, using a lathe or milling machine remove a 2- to 3-mm (0.1- to 0.15-in.) layer from the mold-chilled surface of the sample to eliminate the oxidized and contaminated area. For samples obtained from the Type B mold, cut off the sprue from the back of the sample and using a lathe or milling machine remove a 2- to 3-mm (0.1-

to 0.15-in.) layer from the mold-chilled surface of the sample to eliminate the oxidized and contaminated area. For samples obtained from the Type U mold, cut off the side gate from the sample and using a lathe or milling machine remove a 5-mm (0.2-in.) layer from the mold-chilled surface of the sample to eliminate the oxidized and contaminated area. This cut depth will be deep enough to eliminate contamination pick up from the mold surface while remaining within the most rapidly crystallized region of the disk. The final cut on each of the sample types should be a fine cut of approximately 0.2 mm (0.01 in.) in depth. 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 or a 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 Types A, B, and U disks may be excited around the entire annular area. For these disk 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 be avoided. The shaded zone in [Fig. 4](#) illustrates the approximate area suitable for excitation.

6.1.2 Other Accepted Molds—If molds other than Types A, B or U are used, the same instructions given in [6.1](#) 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.

6.2 Fabricated and Cast Products:

6.2.1 Direct Excitation Without Casting a Sample—When the sample preparation procedures described in [6.1](#) cannot be followed, for example, where the samples are sheet, plate, extrusions, or cast parts, usually only approximate analyses can be made. For best results the sample must be (1) sufficiently massive to prevent undue heating, (2) allow machining to provide a clean, flat surface which creates a seal between the specimen and the spark stand, and (3) reference materials of a similar metallurgical condition (spectrochemical response) and chemical composition must be available. On sheet and plate samples, machine off approximately 0.8 mm (0.03 in.) or one fourth of the sample thickness, whichever is the smaller. On other products, machine a flat surface approximately 1.5 mm (0.06 in.) below the original surface. Choose the depth, location, and number of areas to be analyzed to provide a representative analysis of the product. Thin, flat material may

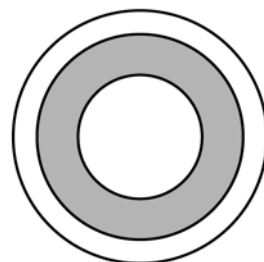


FIG. 4 Disk Excitation Area is Shaded

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. Keywords

7.1 alloys; analysis; magnesium; sampling; spectrochemical

SUMMARY OF CHANGES

Committee B07 has identified the location of selected changes to this standard since the last issue (B953–07) that may impact the use of this standard. (Approved May 1, 2013.)

(1) Added reference to Terminology B881, “Terminology Relating to Aluminum and Magnesium Alloy Products” and Terminology E135, “Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials.”

(2) Deleted withdrawn Practice E401, “Standard Practice for Bonding Thin Spectrochemical Samples and Standards to a Greater Mass of Material (Withdrawn 1995).”

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