



Designation: **E634 – 18** E634 – 23

Standard Practice for Sampling of Zinc and Zinc Alloys for Analysis by Spark Atomic Emission Spectrometry¹

This standard is issued under the fixed designation E634; 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 covers the sampling of zinc and zinc alloys to obtain a sample suitable for quantitative spark atomic emission spectrochemical analysis. Included are procedures for obtaining representative samples from molten metal, from fabricated or cast products that can be melted, and from other forms that cannot be melted.

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

1.3 *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.*

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*² [s.iteh.ai/catalog/standards/sist/da728257-7b42-42c4-abff-9637088122fb/astm-e634-23](https://standards.iteh.ai/catalog/standards/sist/da728257-7b42-42c4-abff-9637088122fb/astm-e634-23)
[E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials](#)

3. Terminology

3.1 *Definitions*—For definitions of terms used in this standard, refer to Terminology [E135](#).

4. Summary of Practice

4.1 Molten metal representative of the furnace melt is poured into a mold to produce a chill-cast sample. The sample, which must represent the average composition, is machined to a specified shape to produce an acceptable surface for excitation.

4.2 Fabricated or cast products are remelted and cast into molds or are excited directly without remelting.

¹ This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.05 on Cu, Pb, Zn, Cd, Sn, Be, Precious Metals, their Alloys, and Related Metals.

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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.

5. Significance and Use

5.1 This practice, used in conjunction with an appropriate quantitative spark atomic emission spectrochemical method, is suitable for use in manufacturing control, material or product acceptance, and development and research.

6. Apparatus

6.1 *Ladle*, of steel, designed to hold sufficient molten metal to completely fill the sample mold, with a handle of sufficient length to reach into a furnace, trough, pot, or crucible.

NOTE 1—Pure zinc metal (Special High Grade) is sampled using a ceramic or graphite ladle, as the solubility of iron in Special High Grade Zinc is sufficient to cause a measurable contamination.

6.2 *Sample Molds*, designed to produce homogeneous chill-cast specimens having smooth surfaces, free from surface pockets and pores. The specimens shall be representative (in the region to be excited) of the product metal. The samples shall have a spectrochemical response similar to the standards like the reference materials used in preparing the analytical calibration curves. This is ensured by casting standards reference materials and specimens in the same manner. Also, the specimens shall have a repeatability of measurement for major elements from excitation to excitation between excitations with a relative error of no more than not exceeding 2 %. Several types of molds have been found acceptable.

6.2.1 *Type A, Pin Mold (Fig. 1)*—This mold produces two diagonally cast pins with sprues at the top of the specimens. The mold dimensions are such as to will produce pins approximately 100 mm (3.9 in.) in length by 11 mm (0.4 in.) in diameter. The mold is made of steel or cast iron and weighs approximately 4.5 to 5.5 kg (10 to 12 lb). Pin specimens have been found to be very homogeneous. If properly prepared, these specimens provide very reliable results with only one burn. However, pin specimens must be reshaped for each additional burn.

6.2.2 *Type B, Book Mold (Fig. 2)*—This mold produces a vertically cast disk with a sprue on the edge of the specimen. The mold dimensions are such as to will produce a disk of approximately 64 mm (2.5 in.) 64 mm (2.5 in.) in diameter by 6 to 8 mm (0.2 to 0.3 in.) 6 mm to 8 mm (0.2 in. to 0.3 in.) in thickness. A circular central recess, 15 to 25 mm (0.6 to 1.0 in.) 15 mm to 25 mm (0.6 in. to 1.0 in.) in diameter, on one side of the specimen facilitates machining of that side in preparation for excitation. It also promotes more uniform freezing of the raised peripheral area. The mold is made of steel or cast iron and weighs approximately 2 to 3 kg (4 to 7 lb). 2 kg to 3 kg (4 lb to 7 lb). This mold works well for high purity zinc grades, but with alloys may cause segregation due to solidification phenomena. Specimens should be excited only in the areas indicated in Fig. 3, and it may be

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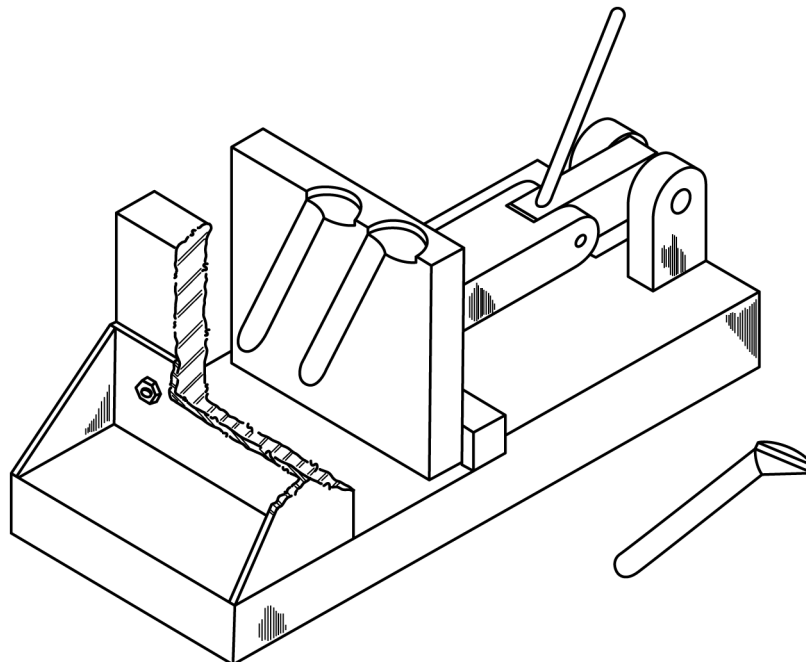


FIG. 1 Type A Mold and Specimen

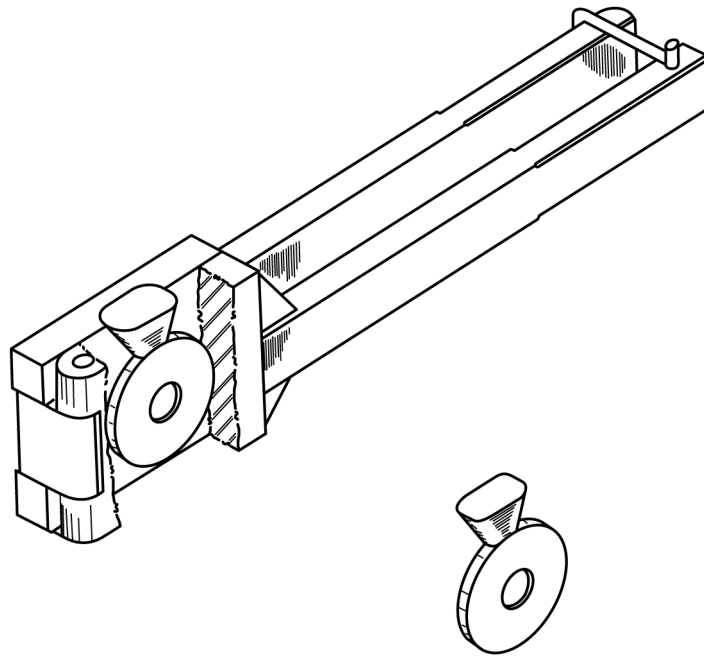
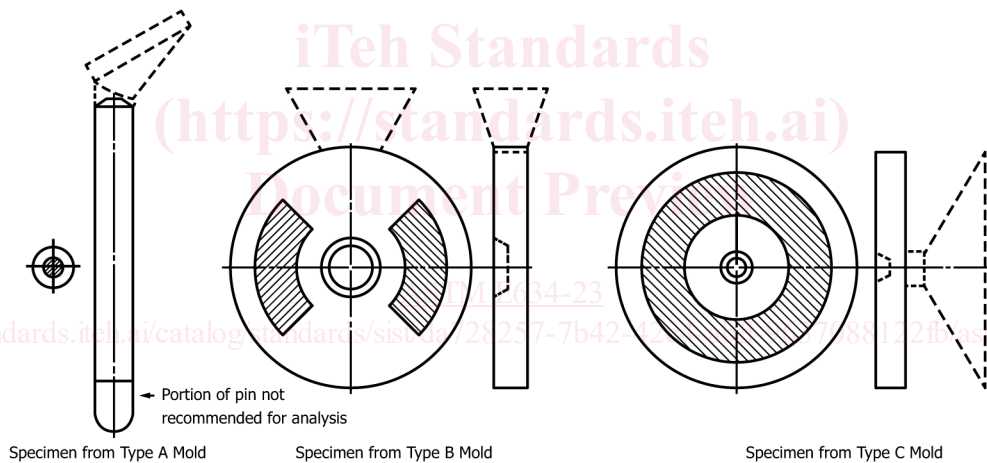


FIG. 2 Type B Mold and Specimen



NOTE 1—Shaded areas are acceptable for analysis.

FIG. 3 Specimens from Type A, B, and C Molds

necessary to make several burns and report an average. The user is strongly cautioned to thoroughly investigate specimen homogeneity for each alloy system to be analyzed.

6.2.3 *Type C, Center Pour Mold* (Fig. 4)—This mold produces a horizontally cast disk with a sprue over the center on the back side. The mold dimensions are such as to will produce a disk approximately 64 mm (2.5 in.) in diameter by 8 mm (0.3 in.) in thickness. A circular central recess, 10 to 20 mm (0.4 to 0.8 in.) 10 mm to 20 mm (0.4 in. to 0.8 in.) in diameter, on one side of the specimen 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 shall not be so large as to be sized to not restrict the throat for the sprue. A slight taper, 1 to 2 deg, 1 deg to 2 deg, on the hinged portion of the mold facilitates opening when a specimen has been cast. The mold is made of steel or cast iron and weighs approximately 3.5 to 4.5 kg (8 to 10 lb). The advantage of this mold is that the specimen obtained may be excited 3.5 kg to 4.5 kg (8 lb to 10 lb). This mold permits excitation around the entire annular area. However, as with like the vertically cast disk specimens, although not as severe, segregation can be a problem. Therefore, it may be necessary to make several burns and report an average.

6.2.4 *Other Molds*—Molds of different types, materials, and dimensions may be used provided that the uniformity of the

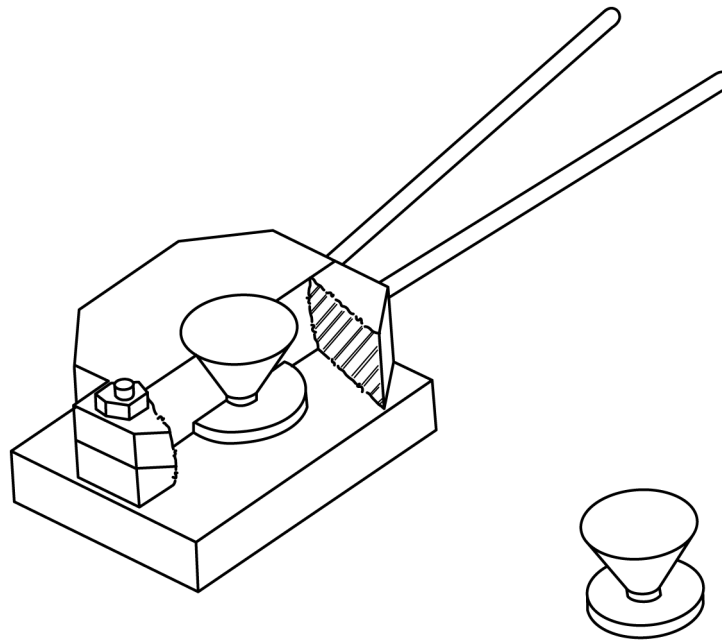


FIG. 4 Type C Mold and Specimen

specimens obtained is comparable to the uniformity of specimens obtained from Type A, B, or C molds. Further, the specimens shall have a spectrochemical response similar to the standards like the reference materials used for preparing the analytical calibration curves.

6.3 *Lathe or Milling Machine*, capable of machining a smooth flat surface.

NOTE 2—When using pin specimens, reproducibility of the exact pin shape is very important. To maintain the integrity of the pin shapes it may be necessary to equip a micrometer cross-fed lathe with a special pin-shaping tool.

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6.4 *Cutting Tools*—Either alloy steel or cemented carbide is recommended.

7. Sampling

7.1 *Molten Metal*—When molten metal is sampled, the temperature shall be well above the point at which any solid phase can be present. Using a clean ladle, push any dross away from the sampling area. Then dip the ladle sideways into the clear area, well below the surface, and stir momentarily. Turn the ladle upright and quickly withdraw. Heating the ladle prevents metal freezing, while obtaining metal well beneath the surface minimizes the danger of ~~inclusion of oxide~~ oxide inclusions. Unless the mold is already hot, cast and discard a preliminary specimen. Remove any excess metal from the ladle, dip it into the molten metal as before, ~~metal~~, and fill the mold using an even rate of pour so as to allow for the escape of air. Do not ~~dump~~ rapidly pour the metal into the mold. Avoid overfilling the sprue or the mold may be difficult to open. Allow the metal to freeze without jarring. The cast surfaces of the specimen, upon removal from the mold, shall be free from shrinkage, inclusions, cracks, or roughness.

NOTE 3—A change in the temperature profile of the mold, caused by a significant change in the sampling rate, may result in different chill-cast characteristics and poor repeatability. Therefore, it is recommended that the sampling schedule be considered when an analysis program is being developed. Also, experience has shown that pin specimens are much less sensitive than disk samples to temperature profile changes in the mold.

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