Designation: E32 – 86(Reapproved 2006)ε1

Standard Practices for Sampling Ferroalloys and Steel Additives for Determination of Chemical Composition[1]

This standard is issued under the fixed designation E32; 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.

ε[1] NOTE—Updated Section 2, Referenced Documents in December 2006.

1. Scope

1.1 These practices include procedures for the sampling of the various ferroalloys and steel additives, either before or after shipment from the plants of the manufacturers. They are designed to give results representative of each lot that will be comparable with the manufacturer's guaranteed analysis for the same lot. For check analysis, the purchaser may use any sampling procedure he desires, but the analytical results obtained on such samples shall not be a basis for complaint or rejection, unless the procedure followed is of an accuracy equivalent to that prescribed in these methods.

1.2 In sampling ferroalloys and steel additives, serious errors often occur from contamination of the samples by iron from the sampling appliances. Therefore, special precautions should be observed to avoid this source of error. Metallic iron may be removed with a magnet from nonmagnetic alloys; its estimation in other alloys requires special analytical procedures (Note 1). To avoid this error, parts of crushers and pulverizing equipment contacting the samples shall be of steel or other material showing a high resistance to abrasion of the type involved.

NOTE 1—Metallic iron in ferrochromium and ferrosilicon may be determined as follows: Transfer 5 g of the sample of alloy to a 150-mL beaker, add 25 mL of HNO_3 (1 + 3), cover, boil 5 min, filter into a 250-mL beaker, and wash with hot water. Add NH_4OH in slight excess, heat to boiling, filter, and wash with hot water. Dissolve the precipitate on the paper with a minimum quantity of hot HCl (1 + 2), wash the filter with hot water, and titrate the iron by a standard procedure such as that described in Test Method E354.

1.3 The values stated in SI units are to be regarded as the standard. The inch-pound values in parenthesis are given 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E354 Test Methods for Chemical Analysis of High-Temperature, Electrical, Magnetic, and Other Similar Iron, Nickel, and Cobalt Alloys

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

3. Terminology

3.1 *Definitions*—Except as defined as follows, for definitions of terms used in this guide, refer to Terminology E135.

4. Significance and Use

4.1 These practices for the sampling of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use these methods will be trained samplers capable of performing common sampling procedures skillfully and safely.

5. Apparatus for Preparing Samples

5.1 The following equipment is required for the preparation of analytical samples of ferroalloys:

5.1.1 *Crusher*—A strongly built jaw crusher capable of rapidly crushing 100-mm (4-in.) lumps to sizes 6.4 mm (¼ in.) and smaller shall be used. The crushing plates of this machine shall be made of a hard and abrasion-resistant steel, such as manganese steel or a properly hardened alloy or hypereutectoid carbon steel.

[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 E01.01 on Iron, Steel, and Ferroalloys.

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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.

5.1.2 *Roll Crusher*—A roll crusher, the rolls of which are fitted with tires of hardened and tempered chromium steel to avoid iron contamination of the sample, shall be used to reduce the 6.4-mm (¼-in.) pieces to a particle size that will pass the No. 10 (2.00-mm) sieve and be retained on the No. 20 (850-µm) sieve.

5.1.3 *Riffles*—Riffles, also designated as Jones dividers, are usually preferable to the use of hand methods for dividing samples. Riffles with openings of 12.7, 25.4, 50.8, and 76.2 mm (½, 1, 2, and 3 in.) should be available; the ½-in. riffle to be used for samples containing particles up to 3.2 mm (⅛ in.) in size, the 1-in. riffle for samples containing particles up to 9.6 mm (⅜ in.), the 2-in. for samples containing particles up to 19.1 mm (¾ in.), and the 3-in. for samples containing particles up to 50.8 mm (2 in.) in size. Riffles should be of the enclosed type to reduce dust losses. The use of multiple riffles is not approved.

5.1.4 *Mortar and Pestle*—The mortar and pestle shall both be made of properly hardened alloy steel of a kind and grade designed to resist severe abrasive forces (Note 2). Suitable dimensions of the mortar are 79.4 mm (3⅛ in.) in outside height, 76.2 mm (3 in.) in outside diameter, 39.7 mm (1⁹⁄₁₆ in.) in inside diameter, and 60.3 mm (2⅜ in.) in inside depth, the bottom 12.7 mm (½ in.) of which shall be rounded. The pestle shall be 152 mm (6 in.) in length, 38.1 mm (1½ in.) in diameter, and rounded at the bottom. The upper part of the pestle should be slightly softer than the remainder in order to decrease the tendency to shatter. Both the mortar and pestle, after hardening, shall be polished with abrasive paper to remove all scale. The narrow clearance between the pestle and the sides of the mortar reduces the dust loss.

NOTE 2—*For example:* steel mortars and pestles of the following composition, after proper hardening and tempering treatments, have been found satisfactory:

Carbon, %	0.60
Manganese, %	0.25
Phosphorus, %	0.02
Sulfur, %	0.02
Silicon, %	0.25
Chromium, %	1.25
Tungsten, %	2.20
Vanadium, %	0.10

After machining annealed steel of this grade to the usual form and dimensions, each part is heated to between 760 and 800°C, quenched in a light, mineral quenching oil and tempered at once. The pestle may be treated by quenching the lower portion only, the upper portion being permitted to air cool, and then tempering the quenched portion.

NOTE 3—Mechanically operated pulverizing equipment such as a ring pulverizer may be substituted for the mortar and pestle, provided suitable tests show that the use of such equipment does not affect the composition of a sample of any material obtained by these methods.

5.1.5 *Sieves*—The sieves shall conform to Specification E11.

6. Unit Quantities for Sampling and Analysis

6.1 Each shipment, except as otherwise agreed upon by the purchaser and the manufacturer, shall constitute a unit for sampling and analysis. It is recommended that shipments of any alloy exceeding 450 Mg (500 tons) be divided into smaller lots for sampling according to some plan best adapted to the material and conditions, such as each cast, each carload, each ladleful, or each binful.

6.2 *Division of Samples*—In these methods the term "divide" is used to indicate a division of a sample into two approximately equal parts of similar composition as in riffling.

7. Sampling Spiegeleisen and 15 % Ferrosilicon

7.1 Spiegeleisen is generally cast in pigs and shipped in bulk. Since this alloy is very hard and somewhat tough, sampling is most accurately and easily accomplished during the tapping of the metal from the furnace or during the pig-casting operation by taking small spoonfuls and pouring the metal quickly into a test mold designed to solidify the metal quickly and give a clean test pig that is easily broken. Sampling of the metal in the solid state is difficult, and is best done during the loading or unloading, except when the material is loaded from bins or unloaded by dumping. The procedure, therefore, may be varied to suit the conditions but shall always conform to the following requirements:

7.1.1 *Sampling at Furnace*—The purchaser may arrange with the manufacturer to have the sampling done at the furnace. If so, each shipment or each cast may constitute a unit sample for analyzing. The sample shall be obtained by collecting portions with a spoon from the runner as the metal flows from the furnace, unless the metal is treated in the runner or ladle to change its composition, in which event the portions shall be taken as the metal flows from the ladle to the pig casting machine. In any case, at least two spoonfuls of metal shall be taken from each ladle, one spoonful while the first third of a ladleful is flowing into or from the ladle and the second while the last third is flowing. Each spoonful shall be taken in a manner to avoid collecting dirt or slag, and the clean metal shall be immediately poured into a clean shallow mold to form a thin chill casting from which small pieces approximately equal in size may be readily broken. When the spiegeleisen is cast in sand beds, the molten metal being run from the furnace directly to the casting floor, the samples shall be taken by dipping skimmed molten metal from the runner trough and pouring it into a small quartered cast-iron button mold. A sample shall be taken in this manner to represent the metal being cast in each pig bed. From the test castings thus obtained to represent a shipment, approximately equal portions shall be taken and combined to form the sample which shall have a gross mass of not less than 200 g. The sample shall then be alternately crushed in a mortar and sieved until it all passes through a No. 80 (180-µm) sieve. If the sample is to be analyzed by more than one laboratory, it shall be mixed, coned, and quartered upon glazed paper (Note 4). The sample or samples thus prepared shall be thoroughly mixed, dried for 1 h at 105 to 110°C, and preserved for analysis in well-stoppered bottles properly labeled for full identification, including the name of the material, the manufacturer, the date, the cast or lot number, etc.

NOTE 4—Finished samples are frequently divided into four portions: one for the purchaser, one for the manufacturer, one for an umpire if necessary, and one held in reserve.

7.1.2 *Sampling Solid Forms*—When the metal is in the solid state, a gross sample shall first be collected by selecting