Designation: E32 – 21

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.

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 desired, but the analytical results obtained on such samples shall not be a basis for compliance 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 (Annex A1). 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.

1.3 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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]

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

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

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

3. Terminology

3.1 For definitions of terms used in these practices, refer to Terminology E135.

4. Significance and Use

4.1 These practices for the sampling of ferroalloys and steel additives are intended for use with test methods used to demonstrate compliance with composition 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.

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 2.00 mm (No. 10) sieve and be retained on the 850 µm (No. 20) sieve.

[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.

Current edition approved May 1, 2021. Published May 2021. Originally approved in 1939. Last previous edition approved in 2015 as E32 – 15. DOI: 10.1520/E0032-21.

[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.

5.1.3 *Riffles*—Riffles are usually preferable to the use of hand methods for dividing samples. If available, riffles should be of the enclosed type to reduce dust losses. Riffle openings should be three (3) times greater than the largest size of the material to be divided. This can be accomplished through the use of fixed chute riffles or adjustable finger riffles.

5.1.3.1 For fixed chute riffles, the following are example sizes that may be used. Riffles with openings of 12.7 mm, 25.4 mm, 50.8 mm, and 76.2 mm (½ in., 1 in., 2 in., and 3 in.) should be available: the 12.7 mm (½ in.) riffle to be used for samples containing particles up to 3.2 mm (⅛ in.) in size, the 25.4 mm (1 in.) riffle for samples containing particles up to 9.6 mm (⅜ in.), the 50.8 mm (2 in.) for samples containing particles up to 19.1 mm (¾ in.), and the 76.2 mm (3 in.) for samples containing particles up to 50.8 mm (2 in.) in size.

5.1.3.2 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 1). 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 1—*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 °C 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.

5.1.4.1 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 or more 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. Regardless, 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 180 µm (No. 80) sieve. If the sample is to be analyzed by more than one laboratory, it shall be mixed, coned, and quartered upon glazed paper (Note 2). The sample or samples thus prepared shall be thoroughly mixed, dried for 1 h at 105 °C 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 2—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

random pigs or pieces at regular intervals during the loading or unloading. Surface sampling of piles of the material will not give a representative sample. When piles of the material must be sampled, the pieces shall be selected according to some fixed plan which assures the obtaining of pieces comprising the gross sample from uniformly distributed points throughout, a condition requiring the moving of all or many of the pieces in the pile. For lots of 45 Mg (50 tons) or larger, 1 pig or piece shall be taken for each 9 Mg (10 tons), and for small lots the number of pieces shall be proportionately increased to 10 pieces for a 9 Mg (10 ton) lot, or 5 pieces for a 0.9 Mg (1 ton) lot. The various pigs thus collected shall be broken approximately in half by any convenient means, and one of the halves of each pig shall be reserved. From the fractured surface of each of these half pigs, an approximately equal portion shall be taken by any suitable means (as by spalling with a heavy hammer), care being taken by the sampler to see that these spalls are not all from the outer edges of the pigs but at least some are obtained from the central portion, and that none contains portions of the outer surface which may be contaminated with sand or other foreign material. The spallings from each half pig as collected shall be placed in separate envelopes and weighed to the nearest 1 g. Each portion so selected shall be of approximately the same mass.

7.1.2.1 The portions shall then be combined to form the sample and alternately crushed (preferably in a hardened-alloy steel mortar) and sieved until it passes a 3.35 mm (No. 6) sieve. Between 280 g and 420 g (10 oz and 15 oz) shall then be separated from the crushed sample by riffling and this portion shall be pulverized to pass a 180 µm (No. 80) sieve. The pulverizing of over-sizes is best done with the hardened steel mortar and pestle, while sieving frequently to keep the size close to 180 µm and prevent loss of dust. The pulverized sample shall be thoroughly mixed upon glazed paper, divided if necessary, labeled, and dried prior to analysis, as directed in 7.1.1.

8. Sampling Ferrosilicon, Standard Ferromanganese, Silicomanganese, Ferrophosphorus, and 12 % to 15 % Zirconium Alloy

8.1 Alloys in this group are shipped in both lump and crushed form, in bulk as well as in containers. Carload lots are generally shipped in bulk, except the finely crushed sizes which are usually shipped in containers. Different procedures are required for sampling the lump and the crushed alloy, and the work of sampling is most conveniently done while loading or unloading.

8.2 *Lump Alloy (Above 50.8 mm (2 in.) In Size)*—In sampling bulk shipments, lumps of average size shall be set aside for the sample at regular intervals in the ratio of one lump from approximately each 270 kg (600 lb). The sample shall be accumulated throughout the loading or unloading operation so that all parts of the shipment will be equally represented. If the alloy is in containers, every fifth container shall be dumped, and one representative lump shall be taken from each 55 kg (120 lb) of alloy which is equivalent to one lump per 270 kg (600 lb) for the lot. The sample shall also include a representative amount of edge metal, small lumps, and any fines that may be present. From each of the lumps in the sample, there shall be broken three small pieces each about 19 mm (¾ in.) in size, one from each of two opposite surfaces (top and bottom, if present) and one from the center, the three pieces constituting a partial vertical cross-section of the lump. Alternatively, a single piece constituting an entire vertical cross section of the lump may be taken.

8.2.1 The pieces, together with a representative portion of any fines present, shall be combined and crushed to pass a 6.4 mm (¼ in.) sieve. Not less than 9 kg (20 lb) shall be separated from the crushed sample by riffling and at least a quarter portion of this shall be rolled to pass a 2.00 mm (No. 10) sieve. A 170 g to 220 g (6 oz to 8 oz) portion obtained by riffling (a larger amount when more than one sample is required) of the 2.00 mm sample shall then be pulverized to pass a 150 µm (No. 100) sieve. The pulverizing is best done with the hardened alloy-steel mortar and pestle, or a ring pulverizer, while sieving frequently to keep the size close to 150 µm and prevent loss of dust. The pulverized sample shall be poured upon glazed paper, mixed thoroughly, and divided, if necessary (Note 2) by quartering, dried for 1 h at 105 °C to 110 °C, and then preserved in a well-stoppered bottle or bottles.

8.3 *Crushed Alloy (Below 50.8 mm (2 in.) In Size)*—One container out of every five in the shipment shall be opened and the contents dumped. A sample representative of both lumps and fines shall be taken from each of the dumped containers to give a combined sample of approximately 0.5 % of the mass of the lot or shipment, this sample being composed of equal amounts of the samples taken from all containers dumped. If in bulk, a fixed portion of representative material shall be taken with a shovel or scoop at regular intervals during the loading or unloading to accumulate a sample of about 0.5 % of the mass of the lot.

8.3.1 The 0.5 % sample shall be crushed to pass a 25.4 mm (1 in.) sieve, mixed, and divided twice if its mass is between 90 kg and 135 kg (200 lb and 300 lb) or three times if it weighs more than 135 kg (300 lb). The portion reserved shall be crushed to pass a 6.4 mm (¼ in.) sieve. Preparation of the sample shall then be completed as described for 6.4 mm (¼ in.) material in 8.2.1.

9. Sampling High-Carbon Ferrochromium, Medium-Carbon Ferromanganese, Low-Carbon Ferromanganese, Silicon Metal, Calcium-Silicon, and 35 % to 40 % Zirconium Alloy

9.1 These alloys are shipped in both lump and crush form, bulk, or in containers.

9.2 *Lump Alloy (Above 50 mm (2 in.) In Size)*—One out of every five containers shall be dumped. Pieces 13 mm to 19 mm (½ in. to ¾ in.) in size shall be broken from the lumps or a single piece constituting an entire vertical cross section of the lump shall be taken. A fair proportion of any fines that may be present shall be included. The gross sample shall contain approximately one piece for each 115 kg (250 lb) of alloy. Bulk material shall be sampled as directed in 8.2. The accumulated sample shall be mixed and reduced in size as directed in 8.2.