Designation: E 32 – 86 (Reapproved 1996)ε1

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

This standard is issued under the fixed designation E 32; 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.

ε1 NOTE—Keywords were added in June 1996.

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 Methods E 38.

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 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[2]

E 38 Methods for Chemical Analysis of Nickel-Chromium and Nickel-Chromium-Iron Alloys[3]

3. Significance and Use

3.1 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.

4. Apparatus for Preparing Samples

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

4.1.1 *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.

4.1.2 *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.

4.1.3 *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

[1] These practices are under the jurisdiction of ASTM Committee E-1 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 April 25, 1986. Published August 1986. Originally published as E 32 – 39 T replacing former A 103. Last previous edition E 32 – 86.

[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] Discontinued; see *1989 Annual Book of ASTM Standards*, Vol 03.05.

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.

4.1.4 *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.

4.1.5 *Sieves*—The sieves shall conform to Specification E 11.

5. Unit Quantities for Sampling and Analysis

5.1 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.

5.2 *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.

6. Sampling Spiegeleisen and 15 % Ferrosilicon

6.1 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:

6.1.1 *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.

6.1.2 *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.

6.1.2.1 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 No. 6 (3.35-mm) sieve. Between 280 and 420 g (10 and 15 oz) shall then be separated from the crushed sample by riffling and this portion shall be pulverized to pass a No. 80 (180-µm) 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, in accordance with 6.1.1.

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

7.1 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.

7.2 *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.

7.2.1 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 No. 10 (2.00-mm) sieve. A 170- to 220-g 6- 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 No. 100 (150-µm) 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 4) by quartering, dried for 1 h at 105 to 110°C, and then preserved in a well-stoppered bottle or bottles.

7.3 *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.

7.3.1 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 and 135 kg (200 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 7.2.

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

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

8.2 *Lump Alloy* (above 50 mm (2 in.) in size)—One out of every five containers shall be dumped. Pieces 13 to 19 mm (½ 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 in accordance with 7.2. The accumulated sample shall be mixed and reduced in size in accordance with 7.2.

8.3 *Crushed Alloy* (below 50 mm (2 in.) in size)—This shall be as described in 7.3, except that a 2 % representative sample shall be taken from each container opened to give an 0.5 % gross sample. For lots of 9 Mg (10 tons) or more, the 0.5 % sample shall be crushed to pass a 25.00-mm (1-in.) sieve, mixed, and divided in half twice. For lots of less than 9 Mg (10 tons), dividing the sample at this stage shall be omitted. The portion retained shall be crushed to pass a 25.4-mm (1-in.) sieve (if above this size) in a heavy crusher provided with smooth plates of manganese steel, and passed through a riffle to obtain a sample of about 23 kg (50 lb). This portion shall be crushed to pass a 6.4-mm (¼-in.) sieve, divided twice, and the quarter portion reserved shall be crushed to pass a No. 10 (2.00-mm) sieve. Between 170 and 220 g (6 and 8 oz) shall then be separated from the crushed sample by riffling, and this portion shall be prepared for analysis in accordance with 7.2.

8.3.1 For lots larger than 9 mg (10 tons), a somewhat smaller percentage of the lump shall be crushed for the sample,

while for smaller lots the percentage shall be increased somewhat to provide a suitable amount of sample for mixing and riffling to size.

9. Sampling Low-Carbon Ferrochromium

9.1 Low-carbon ferrochromium is shipped in both crushed and lump form, in bulk and in containers. The alloy usually contains about 70 % chromium, and has a carbon content ranging from 0.06 to 2.0 %, according to the maximum specified. The combination of hardness and toughness characteristic of this material, particularly of the lower carbon grades, makes it the most difficult of any of the ferroalloys to sample properly. In view of the great importance of the accurate determination of the carbon content, the utmost care shall be taken to avoid contamination of the sample with fragments of steel from the tools used in preparing the sample. Bucking boards shall not be used.

9.2 When the alloy is in lump form, a piece or pieces representing a full cross section of the original cast shall be taken from points distributed throughout the lot, to give a gross sample amounting to about 0.5 % of the mass of the lot. The cross section pieces should be as nearly uniform in size as possible.

9.3 When the alloy is in crushed form in containers, one container out of each five shall be emptied and sufficient representative material taken from each to give a gross sample of about 0.5 % of the mass of the lot. For shipments in bulk, representative portions shall be selected with a shovel at regular intervals during the unloading operation to accumulate a 0.5 % sample.

9.4 The 0.5 % sample shall be crushed to pass a 25.4-mm (1-in.) sieve (if above this size) in a heavy crusher provided with smooth plates of manganese steel, and riffled twice. The resulting quarter shall be crushed to pass a 12.7-mm (½-in.) sieve and riffled once. The sample shall be further crushed to pass a 6.4-mm (¼-in.) sieve and riffled three times. The resulting eighth portion of the sample shall be reduced to pass a No. 6 (3.35-mm) sieve by pounding in a hardened alloy-steel mortar, and riffled to a weight of 170 to 220 g (6 to 8 oz). This amount shall be pulverized to pass a No. 30 (600-µm) sieve in a hardened alloy-steel mortar, while sieving frequently in order to keep the sample as near to this size as possible, until the entire sample has passed the sieve. The pulverized sample shall be mixed thoroughly upon glazed paper, divided if necessary (Note 4) by quartering, dried for 1 h at 105 to 110°C, and preserved in a well-stoppered bottle or bottles.

10. Sampling Ferrovanadium, Ferromolybdenum, Ferrotungsten, Ferrocolumbium, Ferrotitanium, Ferrozirconium, and Ferroboron

10.1 These alloys are shipped in containers and are all high-priced materials. Therefore, it is important that the sampling be thoroughly representative, irrespective of the amount of material involved.

10.2 *Shipments 9 Mg (10 tons) or Under in Mass*—All the containers of a shipment shall be emptied to form a cone shaped pile. The pile shall be sampled by shoveling, the mass of the gross sample being adjusted to the size of the lumps of the alloy. For lots of more than 3.6 Mg (4 tons), one shovelful out of every four shall be reserved for the sample. If the lot weighs less than 3.6 Mg (4 tons), one shovelful out of three or out of two, or shovelfuls otherwise adjusted so as to obtain a gross sample larger than the amounts specified below, shall be taken. The gross sample thus collected shall be coned and again divided by shoveling. This procedure shall be repeated, if necessary, until the weight of the gross sample is reduced to 0.9 Mg (1 ton) for 64-mm (2½-in.) material, 115 kg (250 lb) for 25-mm (1-in.) pieces, or 46 kg (100 lb) for alloy crushed to 6.4-mm (¼-in.) size. In the case of 6.4 mm (¼-in.) material, the sample shall then be mixed and riffled once to 23 kg (50 lb), but larger samples shall be crushed and divided as follows:

10.2.1 *Coarse Material, 64 mm (2—½* in.) Maximum—The 0.9-Mg (1-ton) sample shall be crushed in a heavy crusher provided with smooth plates of manganese steel to pass through a 25.4-mm (1-in.) sieve, mixed thoroughly by coning at least three times, and riffled to 115 kg (250 lb).

10.2.2 *One-Inch Material*—The 115 kg (250-lb) sample shall be crushed in a heavy crusher provided with plates of manganese steel to pass a 64-mm (¼-in.) sieve. After having been mixed thoroughly by coning at least three times, it shall be riffled to about 23 kg (50 lb).

10.2.3 *One-Fourth-Inch Material*—The 23-kg (50-lb) sample of 6.4-mm (¼-in.) material obtained in mixing and reduction of gross samples of 64 or 25-mm (2½ or 1-in.) material or in splitting the gross sample of 6.4-mm (¼-in.) material shall be further crushed in laboratory rolls to pass a No. 10 (2.00-mm) sieve, again mixed thoroughly by coning, and riffled to 4.5 or 7.0 kg (10 or 15 lb). This sample shall be crushed to pass a No. 20 (850-µm) sieve, mixed thoroughly by coning, and divided with a riffle to 500 g (1 lb). The 500-g sample shall be mixed thoroughly by coning and divided by riffling into four portions of about 125 g each. Three of these portions shall be held in reserve, and one portion shall be pulverized in the hardened alloy-steel mortar to pass a No. 100 (150-µm) sieve. The pulverized sample shall be dried for 1 h at 105 to 110°C, poured upon glazed paper, mixed thoroughly, divided, if necessary, (Note 4) by quartering and then preserved in a well-stoppered bottle or bottles.

10.3 *Shipments Over 9 Mg (10 tons) in Mass*—When the shipment exceeds 9 Mg (10 tons), it shall be divided as nearly as possible into lots of 9 Mg (10 tons) each or fraction thereof, and each lot shall be treated separately as described in 10.2. The resulting 500-g (1-lb) samples taken shall be combined and mixed thoroughly by coning at least three times. This sample shall then be divided by riffling to 500 g (or 1 lb), weight which shall be further divided and pulverized as in accordance with 10.2.3.

11. Keywords

11.1 chemical composition; ferroalloys; sampling; steel additives