Designation: E 255 – 02

Standard Practice for Sampling Copper and Copper Alloys for the Determination of Chemical Composition[1]

This standard is issued under the fixed designation E 255; 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 copper (except electrolytic cathode) and copper alloys in either cast or wrought form for the determination of composition.

1.2 Cast products may be in the form of cake, billet, wire bar, ingot, ingot bar, or casting.

1.3 Wrought products may be in the form of flat, pipe, tube, rod, bar, shape, or forging.

1.4 This practice is not intended to supersede or replace existing specification requirements for the sampling of a particular material.

1.5 The values stated in inch-pound units are to be regarded as the standard. The values in parentheses are given for information only.

1.6 *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.* A specific precautionary statement appears in Appendix X4.

2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *casting, n*—a general term for a metal object produced at or near-finished shape by pouring or otherwise introducing molten metal into a mold and allowing it to solidify.

2.1.2 *wrought, adj*—a general term referring to a metal object produced by either hot-working or cold-working operations such as forming, bending, cupping, drawing, and hot pressing.

2.1.3 *lot, n—as used in this practice,* the unit to be sampled, in pounds or pieces.

2.1.4 *gross sample, n*—the total amount of all test pieces selected to represent the lot.

2.1.5 *test piece, n*—an individual product selected at one time from a lot.

2.1.6 *test sample, n*—a composite of material taken by approximate proportional weight from each of the test pieces and from which the test portion shall be taken.

2.1.7 *test portion, n*—the quantity of material taken from the test sample which is subjected to an analytical procedure.

NOTE 1—Appendix X1 describes the refinery shapes. Appendix X2 describes the fabricators' forms.

3. Significance and Use

3.1 This practice is intended primarily for the sampling of copper and copper alloys for compliance with compositional specification requirements.

3.2 The selection of correct test pieces and the preparation of a representative sample from such test pieces are necessary prerequisites to every analysis. The analytical results will be of little value unless the sample represents the average composition of the material from which it was prepared.

4. Selection of Test Pieces

4.1 *Casting*:

4.1.1 *Finished Product or Shipment Sample*:

4.1.1.1 A shipping lot shall consist of all castings of the same configuration and size necessary to comply with the requirements of a single purchase order.

4.1.1.2 Castings are frequently produced in advance of orders and manufacturer's or foundry lots may intermingle in stock.

4.1.1.3 Since the size and configuration of castings vary, the number of test pieces to be selected shall be subject to agreement between the manufacturer or supplier and the purchaser.

4.1.2 *Manufacturer's or Foundry Sample*:

4.1.2.1 For routine sampling and at the option of the manufacturer, a lot shall consist of the following:

(a) (*a*) All of the metal poured from a single furnace or crucible melt, or

(b) (*b*) All of the metal poured from two or more furnaces into a single ladle, or

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(c) (*c*) All of the metal poured from a continuous melting furnace between charges, or

(d) (*d*) All of the metal poured from an individual melting furnace, or group of melting furnaces, having a uniform melting stock, operating during the course of one-half shift, not to exceed 5 h.

4.1.2.2 The sample taken for lot analysis shall be obtained during the pouring of the liquid metal into the mold, or molds, in such a manner as to be representative of the lot and able to be drilled or used in solid form.

4.1.2.3 Plant sampling practices should be developed and implemented which will give homogeneous samples representative of the cast or heat, and free of porosity. Analytical results are frequently obtained by an optical emission technique and, depending upon the metallurgical history of the sample, results may vary. Therefore, it is advisable to cool or quench the sample in a reproducible manner.

4.1.2.4 When foundry lot traceability is specified in the purchase order, additional samples shall be taken, identified, and set aside when so requested by the purchaser.

4.2 *Cast Product*:

4.2.1 For routine sampling and at the option of the manufacturer, a lot shall consist of all of the metal poured from a single furnace melt or all of the metal poured from a continuous melting furnace during a single casting cycle.

4.2.2 Unless otherwise agreed between the manufacturer and the purchaser, sampling of a single lot shall be as follows:

4.2.2.1 *Single Furnace Charge*—The number of samples required depends on the size of the melting furnace and homogeneity of the melt. A small well-stirred, alloying furnace such as one inductively heated and of less than 50 000 lb (22 680 kg) shall require but one sample taken midway in the pour. A large mechanically stirred furnace shall require a minimum of three samples taken, one each at the beginning, middle, and end of the casting period.

4.2.2.2 *Continuous Melting Furnace*—A minimum of one sample shall be taken for each 3 h of the casting cycle.

4.3 *Wrought Products*:

4.3.1 *Finished Product or Shipment Sample*:

4.3.1.1 The lot size, gross sample size, and selection of test pieces shall be as follows:

(a) (*a*) *Lot Size*—An inspection lot shall be 10 000 lb or less of the same mill form, alloy, temper, and nominal dimensions, subject to inspection at one time; or it shall be the product of one cast bar from a single melt charge, whose weight shall not exceed 20 000 lb, which was continuously processed and subject to inspection at one time.

(b) (*b*) *Gross Sample*—The gross sample shall be four or more pieces selected to be representative of the lot. Should the lot consist of four pieces or less, the entire lot shall constitute the gross sample.

(c) (*c*) *Test Piece*—Each test piece shall be selected so as to be representative of the lot.

4.3.1.2 When possible, test pieces shall be selected in a manner that will represent correctly the material furnished but also avoid needless destruction of finished product (such as when samples representative of the material are available from other sources).

4.3.2 *Manufacturer's or Foundry Sample*:

4.3.2.1 For routine sampling, the manufacturer shall have the option of taking samples during the course of manufacture. Samples may be taken at the time castings are poured or from the semifinished product.

4.3.2.2 When samples are taken at the time castings are poured, at least one sample shall be taken for each group of castings poured from the source of molten metal.

4.3.2.3 When samples are taken from the semifinished product, a sample shall be taken to represent each 10 000 lb, or fraction thereof, except that not more than one sample shall be required per piece. Only one sample need be taken from the semifinished product of one cast bar from a single melt charge continuously processed.

5. Sampling

5.1 *General Considerations*:

5.1.1 The saw, drill bit, cutter, or other tool used shall be thoroughly cleaned prior to use. The speed of sampling shall be so regulated that excessive heating and consequent oxidation is avoided. Carbide-tipped tools are recommended. Steel tools, when used, must be magnetizable to assist in the removal of extraneous iron. Only carbide-tipped or other wear-resistant tools shall be used to sample metal which contains a magnetic phase.

5.1.1.1 In the rare instance where tool lubricant is necessary to obtain a satisfactory sample, the lubricant must be one that will not react with the metal. The lubricant must be completely removed immediately after the sampling operation by washing with a solvent which also does not react with the metal.

5.1.2 The test pieces shall be clean and free from scale, dirt, oil, grease, and other extraneous contaminants before sampling.

5.1.3 Test pieces taken from product not subject to significant segregation shall be sampled by drilling, milling, or sawing each test piece. Sampling may also be carried out by clipping in the case of thin or small pieces.

5.1.4 Test pieces taken from product subject to significant segregation shall be sampled by drilling or sawing completely through each test piece, or by milling the entire cross section of each test piece.

5.2 *Finished Product or Shipment Sampling*:

5.2.1 *Castings*:

5.2.1.1 Different parts of a casting may vary in composition. Therefore, a sample from a single casting must be taken with care if it is to be representative of that casting. To obtain a sample representative of a lot of the finished product a number of test pieces should be sampled individually. In any case, the sample should be taken so as to be representative and large enough to suffice for all of the required determinations.

5.2.1.2 Where possible, depending on size and configuration, the casting shall be sampled by drilling five holes equally spaced around or along the casting. The drilling shall be done dry and the drill size shall be the largest practical, but not less than ¼ in. (6.4 mm). Care shall be exercised that no dirt, scale, or other foreign material is included with the drillings.

5.2.1.3 When limited by size or configuration, or both, the castings shall be sampled by milling the entire cross-section,

by sawing through the cross section at several points, or by drilling entirely through the casting at several points.

5.2.2 *Cast Products*—Horizontally or vertically cast products shall be sampled by drilling a minimum of five holes at points equally spaced between the test piece ends. For billet, wire bar, and ingot these holes shall be along the middle line and for cake on a diagonal line between opposite corners. Alternatively, the cross section of the product may be milled at similar points. Sampling test pieces of mass greater than 1000 lb (454 kg) shall be subject to agreement between the manufacturer and the purchaser.

5.2.3 *Wrought Products*—Flat product, rod, bar, shape, tube, or forging shall be sampled by drilling, milling, or sawing the entire cross section at a minimum of three points along the length of the test piece. Thin material may be folded or stacked for sampling or, alternatively, it may be clipped.

5.3 *Manufacturer's or Foundry Lot*:

5.3.1 The sampling procedure used is dependent on the nature of the particular operation and, therefore, shall be at the discretion of the particular operation.

5.3.2 When a complete cross section is required for spectrochemical analysis, it must be properly identified prior to using the remainder of the test piece for other test sampling.

NOTE 2—Appendix X3 addresses principles of sampling theory. Appendix X4 addresses application of sampling theory.

6. Sampling Preparation

6.1 *Finished Product or Shipment Lot*:

6.1.1 For metal that does not contain a magnetic phase, drillings, millings, sawings, or clippings shall be carefully subjected to a strong magnet to remove any iron particles introduced during sampling.

6.1.2 For metal that contains a magnetic phase, a wear-resistant tool, such as carbide tipped, must be used and magnetic cleaning must not be applied.

6.1.3 The test sample shall be prepared by thoroughly mixing equal masses of drillings, millings, sawings, or clippings which shall have first been determined to be of uniform size.

6.1.4 The test sample shall weigh at least four times that required for the total analysis, and shall be divided into four equal portions. Each portion shall be placed in an identified container and sealed; one portion each shall be reserved for the manufacturer and the purchaser; one portion shall constitute the reserve; and, when necessary, one portion shall be used for any umpire work.

6.1.4.1 Material to be stored over a long period, which oxidizes readily, or which alters in composition under varying atmospheric conditions should be kept under a protective gas, such as nitrogen, in an airtight container of suitable size and composition. This same storage should be used when contamination by paper or cardboard fibers is a concern.

6.2 *Manufacturer's or Foundry Sample*—The preparation of the manufacturer's or foundry sample shall be at the discretion of the reporting laboratory.

7. Preparation of Test Portion

7.1 Preparation of the test portion for analysis varies with the particular method used and shall be the responsibility of the reporting laboratory.

8. Resampling

8.1 In case of dissatisfaction with the sample prepared from the finished product, either party may require the material to be resampled.

9. Keywords

9.1 copper; copper alloys; sampling

APPENDIXES

(Nonmandatory Information)

X1. DEFINITIONS OF REFINERY SHAPES

X1.1 *billet*—cast shape used for piercing and extrusion into tubular products or for extrusion into rods, bars, and shapes; circular in cross section, usually 3 to 16 in. (76 to 406 mm) in diameter, normally ranging in weight from 100 to 4200 lb (45 to 1905 kg).

X1.2 *cake*—cast shape used for rolling into plate, sheet, strip, or shape; rectangular in cross section and of various sizes, normally ranging in weight from 140 to 62 000 lb (63 to 28 200 kg).

X1.3 *cathode*—unmelted, electrodeposited, and somewhat flat plate normally used for melting. The customary size is about 3 ft (0.914 m) square, about ½ to 1 in. (12.7 to 25.4 mm) thick, weighing up to about 360 lb (163 kg), and may have hanging loops attached. Cathodes may also be cut to smaller dimensions.

X1.4 *ingot and ingot bar*—cast shape used for remelting (not fabrication). Ingots normally range in weight from 20 to 35 lb (9 to 16 kg) and ingot bars from 50 to 70 lb (23 to 323 kg). Both are usually notched to facilitate breaking into smaller pieces.

X1.5 *wire bar*—a refinery shape used for rolling into rod or flat product for subsequent processing into wire, strip, or shape. Approximately 3.5 to 5 in. (89 to 127 mm) square in cross section, usually 54 in. (1.56 m) in length and ranging in weight

from 200 to 420 lb (91 to 191 kg). Usually tapered at both ends.

X2. DEFINITIONS OF FABRICATORS' PRODUCTS

X2.1 *flat product*—a rectangular or square solid section of relatively great length in proportion to thickness. Included in the designation" flat product," depending on the width and thickness, are plate, sheet, strip, and bar. Also included is the product known as "flat wire."

X2.2 *pipe*—a tube conforming to the particular dimensions commercially known as "standard pipe sizes."

X2.3 *rod*—a solid section, round, hexagonal, or octagonal in straight lengths. Round rod for further processing into wire (known as" hot-rolled rod," "wire-rod," "redraw wire," or "drawing stock") is furnished coiled.

X2.4 *shape*—a solid section, other than flat product, rod or wire, furnished in straight lengths; shapes are usually made by extrusion but may also be fabricated by drawing.

X2.5 *tube*—an unidirectionally elongated hollow product of uniform round or other cross section having a continuous periphery.

X2.6 *wire*—a solid section, including rectangular flat wire but excluding other flat products, furnished in coils or on spools, reels, or bucks.

X3. PRINCIPLES OF SAMPLING THEORY

X3.1 Some theoretical aspects of sampling cast metal shapes and other metallurgical materials for chemical analysis are described in this Annex. While it is recognized that cost, time, and other practical considerations may necessitate substantial deviations, it was deemed necessary to outline the theories and foundations of correct metallurgical sampling.

X3.1.1 Molten alloys will be homogeneous only when all components are completely dissolved and perfectly distributed by mixing, and when the temperature throughout the molten mass is above that of the liquids.

X3.1.2 Casting, however, solidified even from such a homogeneous melt, will in most cases be heterogeneous. The degree of heterogeneity of the solid phase will depend mainly on the composition and rate of cooling of the melt. For example, a cylindrical casting of high-lead alloy may contain almost pure lead in its central core which solidified last. Conversely, the cooled surface areas of copper-tin alloy castings often contain tin concentrations far above the average for the whole casting. Due to the almost ever-present problem of segregation, sampling of cast metal shapes shall be adapted to the particular conditions of each separate case.

X3.1.3 Continuously cast shapes may be sampled by removing material of uniform thickness from a complete cross section. Such samples can be conveniently obtained by sawing, turning, or milling. Although such samples provide correct representation of the cast at the time of sampling, they would, of course, not be valid if the composition of the melt changed during the casting operation.

X3.1.4 A hypothetically correct but prohibitively expensive method of sampling horizontally and vertically cast metal shapes would be to cut complete segments from the top to the bottom of the casting. The minimum sample volume required for geometrically correct representation of all diffusion layers would be as follows:

X3.1.4.1 Twenty-five percent of the volume for shapes of rectangular top view;

X3.1.4.2 Twelve-and-one-half percent of the volume for shapes of square top view;

X3.1.4.3 A segment of any width for shapes of circular top view.

X4. APPLICATION OF SAMPLING THEORY

X4.1 *Sample Selection*—The number of cast shapes selected from a lot will be dependent on a number of considerations which do not lend themselves to generalization and must be determined separately for each individual case. Clearly, different considerations apply to small refined copper ingots derived from a 300-ton furnace charge of electrolytically refined cathodes, than apply to high lead or tin alloy castings from a small furnace charge.

X4.2 *Sampling:*

X4.2.1 *Sampling of Molten Metal*—Sampling of molten metal is subject to numerous pitfalls. If it is necessary to obtain a molten sample using a ladle, it should be remembered that if the molten material and the ladle are not at the same temperature, segregation will occur as part of the material freezes in the ladle. The metal poured thereafter will have a composition different from the skull which remains in the ladle. The alternative possibilities of diverting the molten stream at intervals, or part of the stream all of the time, are techniques difficult to accomplish for practical sampling. Molten metals can also be sampled using evacuated glass tubes several millimetres in diameter and 100 to 120 mm long, or copper tubing approximately 9.53 mm in diameter and 400 to 450 mm

long attached to a spring loaded vacuum pump. Both procedures have been used for sampling. If either tubing used is too large or too long, center porosity will occur; in extreme cases the sample will become hollow. With careful technique, sound metal pins can be produced which show good correlation between the analysis of the pin and the subsequently cast shape.

X4.2.2 *Sampling of Cast Shapes*:

X4.2.2.1 Samples from cast shapes can be obtained by various methods, such as drilling, turning, sawing, or milling. Extraneous contamination shall be removed from the area to be sampled.

X4.2.2.2 To avoid unequal representation of parts, the drilling pattern or sawing pattern must be designed with due consideration to the geometry of the sampled shape.

X4.2.2.3 With the possible exception of very large castings, test pieces should be drilled or sawed completely through in order to avoid over, or under, representation of the center portion.

X4.2.2.4 If steel tools are used, these should be magnetically attractable to assist in the subsequent removal of any ferrous particles. Carbide-tipped tools shall be used for alloys in which there are magnetic phases.

X4.2.2.5 Drilling or other methods of sampling shall be performed at a speed and force which does not cause oxidation of the sample. Any discoloration of the chips can be assumed to be the result of oxidation.

NOTE X4.1—**Caution:** To prevent sample contamination, avoid the use of cooling or lubricating substances.

X4.2.2.6 The fragments obtained by any of the sampling methods shall be uniformly small in size. The proportion of fines to coarse fractions should be less than 5 %.

X4.3 *Sample Preparation:*

X4.3.1 If sieving is necessary to obtain a representative laboratory sample, the total quantity of the gross sample (drillings, sawings, etc.) should pass through a specified sieve size. Oversize sample material should be further processed by grinding or by other suitable methods to ensure its passage through a sieve.

X4.3.2 For certain analyses it may be desirable to further separate coarse and fine particles on a second sieve of smaller aperture. In such cases the coarse and fine fractions shall be weighed and kept separate.

X4.3.3 Analysis may be performed separately on the two portions and the results calculated on the basis of proportional weights, or a single sample may be weighed in proportion to the coarse and fine fractions and analyzed directly.

X4.3.4 In most cases it is necessary to reduce the size of the gross sample to obtain the laboratory samples. The size reduction may be accomplished by the quadrisectioning ("coning and quartering") method, or by the use of mechanical splitters or dividers. In the latter cases the sample shall be mixed thoroughly (for example by repeated rolling) before it is split.

X4.3.5 If it necessary to dry the laboratory samples, drying shall be accomplished under an inert atmosphere or vacuum and at a temperature at which no significant oxidation can occur.

X4.3.6 Except for alloys in which there are magnetic phases, samples shall be treated with a strong magnet to remove any particles of iron introduced during sampling or sample preparation. Alloys in which there are known to be magnetic phases should be sampled using carbide-tipped tools, and shall not be magnetically cleaned.

X4.4 *Resampling:*

X4.4.1 In case of disputes not resolved by the first round of sampling and by the use of referee services, consider the following procedure:

X4.4.1.1 Either party may require that the material be resampled in the presence of representative of both parties. Both parties shall each select the prescribed number of test pieces from the lot, thereby doubling the sampling frequency. The thoroughly mixed laboratory sample shall be divided into four equal portions.

X4.4.1.2 Each portion shall be placed in a container and sealed. If analysis by the two parties does not resolve the dispute, the third laboratory sample portion shall be submitted to a referee agreeable to both parties.