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Copper and copper alloys — Selection and preparation of samples for chemical analysis —

Part 1 :

Sampling of cast unwrought products

Cuivre et alliages de cuivre — Sélection et préparation des échantillons pour l'analyse chimique —

Partie 1 : Échantillonnage des formes brutes de coulée

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1811-1 was prepared by Technical Committee ISO/TC 26, *Copper and copper alloys.*

Together with ISO 1811-2 and 1811-3, it cancels and replaces ISO Recommendation R 1811 : 1971, of which it constitutes a technical revision.

ISO 1811 consists of the following parts, under the general title *Copper and copper alloys — Selection and preparation of samples for chemical analysis :*

- *Part 1 : Sampling of cast unwrought products*
- *Part 2 : Sampling of wrought products and castings*
- *Part 3 : Sampling of cathodes*

Annex A forms an integral part of this part of ISO 1811.

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Copper and copper alloys — Selection and preparation of samples for chemical analysis —

Part 1 :
Sampling of cast unwrought products

1 Scope

This part of ISO 1811 specifies methods for the selection and preparation of samples for chemical analysis of copper and copper alloys in the form of cast unwrought products, except cathodes.[1] [2]

The methods specified in this part of ISO 1811 apply to copper and copper alloys in the form of cast unwrought products (refinery shapes), such as cakes, billets, wire bars and ingots, and are intended to determine compliance with the requirements for chemical composition.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 1811. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 1811 are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 197-1 : 1983, *Copper and copper alloys — Terms and definitions — Part 1 : Materials.*

ISO 197-2 : 1983, *Copper and copper alloys — Terms and definitions — Part 2 : Unwrought products.*

ISO 1811-2 : 1988, *Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 2 : Sampling of wrought products and castings.*

3 Definitions

For the purpose of this part of ISO 1811, the definitions for copper and copper alloys in ISO 197-1 and for cast unwrought products in ISO 197-2 apply.

In addition, the following definitions apply.

3.1 lot of cast unwrought products (except cathodes)[2] : Unless otherwise agreed between producer and purchaser, for the purpose of sampling, a lot of cast unwrought products shall be defined as material of a particular form, such as cakes, billets, wire bars or ingots, that is produced from a single melting furnace charge or from a 24 h cycle of a continuous melting furnace.

Materials of different forms, or of different sizes, and/or cast from different furnaces, shall be considered as separate lots.

3.2 sampling unit : An individual cast unwrought product selected from a lot.

3.3 gross sample : The total amount of all sampling units selected from a lot.

3.4 test sample : A sample prepared from the gross sample and from which the test portions will be taken.

3.5 test portion : The quantity of material taken from the test sample and on which the analysis is actually carried out.

4 Selection of sampling units

Unless otherwise agreed between producer and purchaser, the following number of sampling units shall be selected from a single lot.

4.1 Single furnace charges

Three sampling units, taken at the beginning, the middle and the end of the casting period, respectively, shall represent one lot of cast unwrought products from a single furnace charge.

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1) Sampling theory is explained in annex A of this part of ISO 1811.

2) Methods for the sampling of copper cathodes are specified in ISO 1811-3 (to be published).

In cases in which several furnace charges are mixed in a ladle prior to pouring, one sampling unit shall be taken from the combined melts prior to pouring the castings.

4.2 Continuous melting furnace

One sampling unit taken during each three-hour casting period shall represent one lot of cast unwrought products from a continuous melting furnace.

5 Sample preparation

5.1 General procedure

5.1.1 Sampling units taken from cast unwrought products not subject to significant segregation shall be sampled by drilling completely through each test piece. The location and number of holes shall be such as to ensure that the sample taken is representative. Cast unwrought products subject to significant segregation shall have the whole cross-section of each sampling unit sampled by milling. Sampling may also be carried out by sawing or clipping.

5.1.2 Before sampling, the sampling units shall be clean and free from scale, dirt, oil, grease and other contaminants. If necessary, the sampling units may be cleaned in ethyl ether or acetone, rinsed in ethanol, and dried. Scale and dirt may be removed by suitable mechanical or chemical treatment. If chemical methods of cleaning are used, such operations shall not alter the metal surface in any way.

5.1.3 The saw, drill bit, cutter or other tool used for sampling shall be thoroughly cleaned prior to use. The speed of sampling shall be so regulated that excessive heating and consequent oxidation of the sample is avoided. Lubricants shall not be used. Carbide-tipped tools are recommended; steel tools if used shall be made of magnetic material to assist in the subsequent removal of extraneous iron.

5.1.4 For alloys with magnetic phases, carbide-tipped tools shall be used; magnetic cleaning shall not be used. Drillings, millings, sawings or clippings of alloys with no magnetic phases shall be treated carefully with a strong magnet to remove any particles of iron introduced during sampling. The drillings, millings, sawings or clippings shall be carefully examined, and any foreign material introduced in taking the sample shall be removed.

5.1.5 In certain cases a complete cross-section from a sampling unit may be required for spectrometric examination. Each cross-section shall be taken and properly marked before using the remainder of the sampling unit for other sampling operations.

5.1.6 The gross sample shall be prepared by thoroughly mixing equal masses of drillings, millings, sawings or clippings taken from each sampling unit. The chips obtained by milling, drilling, etc., shall be uniformly small in size.

5.1.7 The gross sample shall weigh at least four times the amount required for the total analysis, and shall be divided into four test samples of equal mass, each of which shall be placed in a container and sealed: one for the producer, one for the purchaser, one for reference purposes, if necessary, and one as reserve.

5.1.8 In the event of the oxygen content of Cu-OFE (and Cu-HCP if necessary) being determined, an additional group of test samples shall be taken from the selected sampling units. Each test sample shall consist of a single piece of adequate size, cut from the sampling unit by a method agreeable to both producer and purchaser at a mutually agreeable location in the sampling unit. Each test sample shall be cut into three test portions of approximately equal mass: one for the producer, one for the purchaser, and one for reference purposes, if necessary. Each party shall determine oxygen on his test portion using an agreed procedure.

5.2 Horizontally cast unwrought products

5.2.1 Five holes shall be drilled through from the set surface at points equally spaced between the ends of the sampling unit. For billets, ingots and wire bars, these holes shall be along the middle line, and for cakes on a diagonal line between opposite corners.

5.2.2 Alternatively, any horizontally cast unwrought product may be milled at similar points through a section from side to centre or through an entire cross-section of the sampling unit.

5.3 Vertically cast unwrought products

5.3.1 Five holes shall be drilled at points equally spaced between the ends of the sampling unit. For billets and wire bars these holes shall be along the middle line, and for cakes on a diagonal line between opposite corners.

5.3.2 Alternatively, any vertically cast unwrought product may be milled at similar points through a section from side to centre or through an entire cross-section of the sampling unit.

5.4 Sampling units of mass greater than 450 kg

Sampling of individual sampling units of mass greater than 450 kg shall be by agreement between the producer and the purchaser.

Annex A
(normative)

Sampling theory

A.1 Principles

The 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 is useful to outline the fundamental theory of correct metallurgical sampling.

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 liquidus.

Castings, however, even solidified 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.

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.

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:

- 25 % of the volume for shapes of rectangular horizontal section;
- 12,5 % of the volume for shapes of square horizontal section;
- a segment of any width for shapes of circular horizontal section

A somewhat simplified but still expensive compromise in sampling a cast metal shape is based on removing samples from two parallel diagonal planes. For a rectangular shape, the two diagonal planes are those which connect the centreline of the bottom with a parallel edge of the top and an edge of the bottom with the parallel centreline of the top. For a cylindrical shape, the two parallel planes extend between the top diameter and the point at which a parallel tangent meets the bottom circumference, and between the bottom diameter and the point at which a parallel tangent meets the top circumference. The samples might be produced by sawing, or by milling the exposed areas. It is recognized that, although a theoretically correct sample would result, the cost of this method is too high for practical consideration. It is presented only to indicate that simplification of the sampling geometry may result in a deterioration of sampling accuracy, particularly in the case of heavily segregated castings.

A.2 Application

A.2.1 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 t furnace charge of electrolytically refined cathodes than to high lead or tin alloy castings from a small furnace charge. Guidelines for sampling frequencies are given in clause 4 of this part of ISO 1811 and in clause 4 of ISO 1811-2.

A.2.2 Sampling

A.2.2.1 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 subsequently poured from the ladle 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 in practice.

Molten metal can also be sampled using evacuated glass tubes several millimetres in diameter and 10 to 12 cm long. This procedure has been used for sampling phosphorized copper and tin-bronzes. If the tubing used is too large or too long, central porosity will occur; in extreme cases the sample will become hollow. With careful technique and the diameter limited to about 4 mm, sound metal pins can be produced which show good correlation between the analysis of the pin and the subsequently cast shape.

A.2.2.2 Sampling of cast shapes

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

To ensure that the different parts of the cast shape are represented in the correct proportions, the pattern according to which drilled or sawn samples are taken shall be designed with due consideration to the geometry of the sampled shape. With the possible exception of very large castings, sampling units shall be drilled or sawn completely through in order to avoid over- or under-representation of the centre portion.

If steel tools are used, these shall be made of magnetic material to assist in the subsequent removal of any ferrous particles. Carbide-tipped tools are preferred, since fine iron particles may become embedded in large copper particles and may not be removed subsequently with a magnet. Carbide-tipped tools shall be used for alloys with magnetic phases.

Drilling or other methods of sampling shall be performed at a speed and force which does not cause oxidation of the sample. To prevent sample contamination, the use of cooling or lubricating substances shall be avoided.

The fragments obtained by any of the sampling methods shall be uniformly small in size. The proportion of very small or powdery fragments shall be kept to a minimum.

A.2.3 Sample preparation

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

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.

Analysis may be performed separately on the two fractions, treating each in the same way as a test portion, and the results calculated on the basis of proportional weights, or a single test portion may be weighed out, using the correct proportions of the coarse and fine fractions, and analysed directly.

In most cases it is necessary to reduce the size of the gross sample to obtain test 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 gross sample shall be mixed thoroughly (for example by repeated rolling) before it is split.

If it is necessary to dry the test samples, drying shall be accomplished at a temperature at which no significant oxidation can occur.

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

A.3 Resampling

In the event of disputes not resolved by the first round of sampling or by the use of referee services, the following procedure may be considered.

Either party may require that the material be resampled in the presence of representatives of both parties.

The sampling procedures shall be identical to those specified in clauses 4 and 5 of this part of ISO 1811 and in clauses 4 and 5 of ISO 1811-2, except that each of the two parties shall select the prescribed number of sampling units from a lot, thereby doubling the sampling frequency. The thoroughly mixed test sample shall be divided into four equal portions: one for each party, one for reference purposes and one as reserve.

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

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Descriptors : copper, copper alloys, copper products, unwrought products, chemical analysis, sampling, specimen preparation.

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