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Steel and iron — Sampling and preparation of samples for the determination of chemical composition

Aciers et fontes — Prélèvement et préparation des échantillons pour la détermination de la composition chimique

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 459/SC 2, *Methods of chemical analysis for iron and steel,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 14284:1996), which has been technically revised. The main changes are as follows:

— figures updated;

— Clause 3 updated;

— text updated;

— new sampling probes added;

— units changed to SI units.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Steel and iron — Sampling and preparation of samples for the determination of chemical composition

1 Scope

This document specifies methods for sampling and sample preparation for the determination of the chemical composition of pig irons, cast irons and steels.

Methods are specified for both liquid and solid metal.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
chemical method
method for the determination of chemical composition in which the *test sample* (3.16) or the *test portion* (3.17) is submitted to chemical reactions

3.2
physical method of analysis
physical method
method for the determination of chemical composition in which the determination is carried out without submitting the *test sample* (3.16) to chemical reactions

EXAMPLE Optical emission spectrometric (OES) method or X-ray fluorescence spectrometric (XRF) method.

3.3
thermal method of analysis
thermal method
method for the determination of chemical composition in which the *test sample* (3.16) is submitted to a process of heating, combustion or fusion

3.4
melt
liquid metal from which a *sample* (3.25) is taken

3.5
spoon sampling
method in which a *sample* (3.25) is taken from the *melt* (3.4), or during the pouring of the melt, using a long-handled spoon and poured into a small mould

3.6
spoon sample
sample (3.25) obtained from *spoon sampling* (3.5)

3.7
probe sampling
method in which a *sample* (3.25) is taken from the *melt* (3.4) using a commercially available sampling probe that is immersed in the melt

3.8
probe sample
sample (3.25) obtained from *probe sampling* (3.7)

3.9
suction sampling
method of *probe sampling* (3.7) in which the probe is immersed in the *melt* (3.4) and the sample chamber in the probe fills by aspiration

3.10
stream sampling
method of *probe sampling* (3.7) in which the probe is inserted into a stream of liquid metal and the sample chamber in the probe fills by the force of the metal flow

3.11
immersion sampling
method of *probe sampling* (3.7) in which the probe is immersed in the *melt* (3.4) and the sample chamber in the probe fills by ferrostatic pressure or gravity

3.12
cast product
steel or cast iron product that has not been subjected to deformation

EXAMPLE An ingot, a semi-finished product obtained by continuous casting, a casting.

3.13
wrought product
product obtained by hot and/or cold plastic deformation processes such as extruding, forging, hot rolling, cold rolling or drawing, either exclusively or in combination

EXAMPLE Rods, bars, wires, tubes, profiles, sheets, strips, forgings.

3.14
batch sample
sufficient amount of cast iron, pig iron or steel selected from a product batch for the purpose of obtaining one or more *laboratory samples* (3.15)

3.15
laboratory sample
part of a *sample* (3.25) that is processed so that it can be sent to the laboratory for the purpose of obtaining one or more *test samples* (3.16)

3.16
test sample
part of a *batch sample* (3.14), part of a *laboratory sample* (3.15) taken from a batch sample or part of a *sample* (3.25) taken from the *melt* (3.4) and brought to the appropriate condition required for analysis

Note 1 to entry: The test sample can also be the batch sample itself or a sample taken from the melt.

Note 2 to entry: The categories of test samples are the following:

— sample in the form of a solid block;

— sample obtained by remelting;

— sample in the form of chips obtained by machining;

— sample in the form of fragments obtained by *crushing* (3.19);

— sample in the form of powder obtained by *comminution* (3.18).

3.17
test portion
part of a *test sample* (3.16), or part of a *sample* (3.25) taken from the *melt* (3.4), submitted to analysis

Note 1 to entry: In some cases, the test portion may be selected from the *batch sample* (3.14) itself.

Note 2 to entry: Specific types of test portions in the form of solid blocks are the following:

— small disc, commonly described as a slug, obtained by punching;

— small appendage, commonly described as a lug;

— small-diameter rod, commonly described as a pin, obtained by cutting.

3.18
comminution
operation of reducing particle size by *crushing* (3.19) or *grinding* (3.21)

3.19
crushing
mechanical reduction of the particle size of a material by fracturing large pieces into multiple smaller pieces

3.20
linishing
method of preparing a *sample* (3.25) of metal for a *physical method of analysis* (3.2) in which the surface of the *test sample* (3.16) is abraded using a rotating disc or a continuous belt coated with an abrasive material

3.21
grinding
method of preparing a *sample* (3.25) of metal for a *physical method of analysis* (3.2) in which the surface of the *test sample* (3.16) is abraded using an abrasive wheel

3.22
milling
method of preparing chips or the surface of a *sample* (3.25) for a *physical method of analysis* (3.2) in which the surface of the *sample* (3.25) is machined using a rotating, multi-edged cutting tool

3.23
consignment
quantity of metal delivered at one time

3.24
increment
quantity of metal obtained by sampling at one time from a *consignment* (3.23)

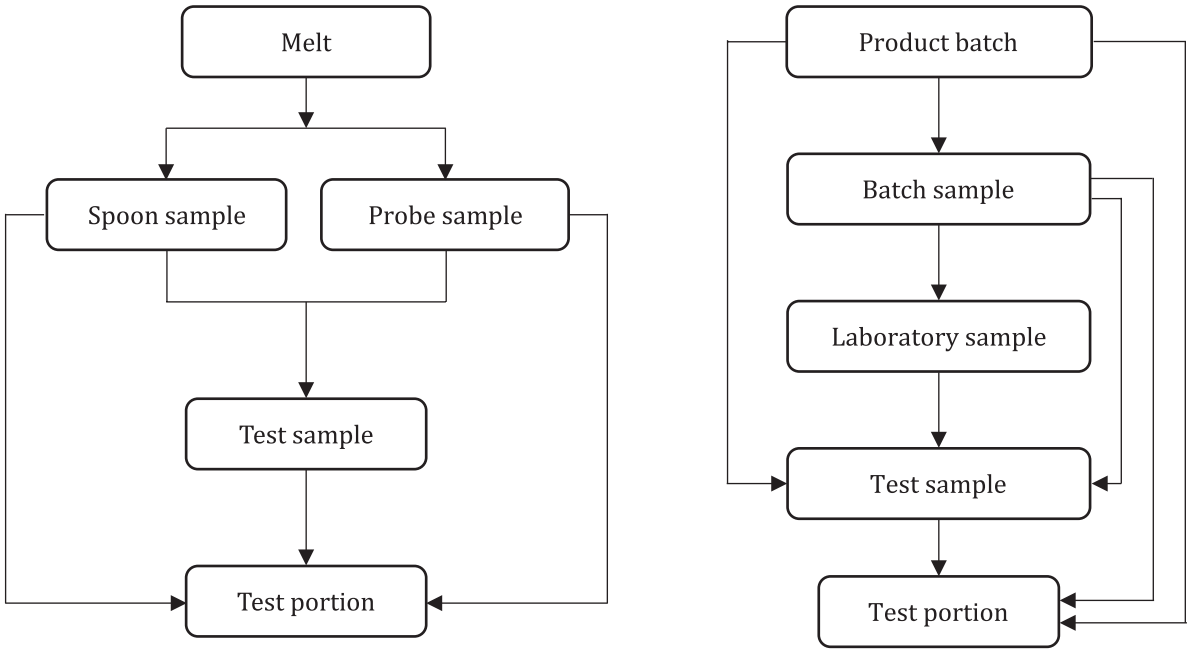
3.25
sample
portion of material selected from a larger quantity of material

4 Requirements for sampling and sample preparation

4.1 General

This clause covers the general requirements for the sampling, the sample and the sample preparation of liquid iron and steel. Specific requirements applying to each category of liquid and solid metal are given in the relevant subclauses.

The sequence of sampling and sample preparation of liquid iron and steel, pig-iron, cast iron and steel products is shown in Figure 1. For requirements applying to pig irons, see Clause 8.



a) Liquid iron and steel **b) Pig-iron, cast iron and steel products**

Figure 1 — Sequence of sampling and sample preparation

4.2 Sample

4.2.1 Quality

Sampling practices shall be designed to provide a test sample that is representative of the chemical composition of the melt or the batch sample.

The test sample shall be sufficiently homogeneous with respect to chemical composition such that inhomogeneity does not appreciably contribute to the uncertainty of the results of the analysis. However, in the case of a sample taken from a melt, some variability in analysis, both within and between test samples, is unavoidable. This variability will form an inherent part of the accuracy of the analysis.

The test sample shall be free from surface coatings, and from moisture, dirt or other forms of contamination.

As far as possible, the test sample should be free from voids, cracks and porosities, and from fins, laps or other surface imperfections.

Particular care shall be taken when selecting and preparing the test sample, where a sample taken from a melt is expected to be heterogeneous or contaminated in any way. If such inconsistencies are found in the samples, they shall be rejected.

A sample taken from a melt shall be cooled in such a manner that its chemical composition and metallurgical microstructure are consistent from sample to sample.

Analysis by some physical methods can be influenced by the metallurgical microstructure of the sample, particularly in the case of cast irons (even with white microstructure) and steels in the as-cast and wrought conditions.

4.2.2 Size

The dimensions of a laboratory sample in the form of a solid block shall be sufficient to permit additional test samples to be taken for re-analysis.

Test samples shall have a sufficient mass to allow any further analysis. Generally, a mass of 100 g will be sufficient for a sample in the form of chips or powder.

The shape and dimensions of the samples shall be determined to ensure the following:

— their homogeneity;

— their acceptability as representative with respect to the composition of the melt;

— a microstructure adapted to the techniques of analysis of solid samples.

In the case of optical emission and X-ray fluorescence spectrometric methods, the shape and size of the test sample will be determined by the dimensions of the sample chamber.

4.2.3 Identification

A test sample shall be assigned a unique identification in order to trace back the melt from which it was taken and, if necessary, the processing conditions of the melt or the location of the laboratory sample or the test sample in the batch sample.

A test sample of pig iron shall be assigned a unique identification in order to trace back the consignment or part of a consignment and the increment from which it was taken.

Labelling or some equivalent method of marking shall be used to ensure that the assigned identification remains associated with the test sample.

The identification, status and condition of the sample shall be recorded to ensure that confusion cannot arise as to the identity of the item to which analysis and records refer.

4.2.4 Sample conservation

Adequate storage facilities shall be provided to separate and protect the test sample. During and after preparation, the test sample shall be stored in such a way as to prevent contamination or chemical change.

It is permitted to conserve the laboratory sample in the form of a solid block, and a test sample may then be prepared when required.

The test sample, or the laboratory sample in the form of a solid block, shall be kept for a sufficient period of time in the laboratory for audits and/or retests purposes.

4.2.5 Sample for arbitration

In the case of samples intended for arbitration, the test samples shall be prepared jointly by the supplier and purchaser, or by their representatives. The records shall be kept of the methods used for preparing the test samples.

Containers with test samples intended for arbitration shall be sealed by both parties or by their representatives. Unless otherwise agreed, these containers shall be kept by the representatives of each party responsible for the preparation of samples.

4.3 Sampling

4.3.1 Sample from a melt

Melts are sampled at various stages of the manufacturing process for the purposes of monitoring and controlling the process. Samples may be taken during the casting of the melt to verify chemical composition in accordance with the specification of the cast product. In the case of liquid metal intended for the production of a casting, the test sample may be selected from test bars or blocks specially cast from the same metal as that of the casting for purposes of mechanical testing, in accordance with the product standard.

Sampling practices for melts shall be designed to provide samples during a particular manufacturing process in accordance with requirements related to the quality of the sample (see 4.2.1). The sample obtained from a melt is usually in the form of a small ingot, a cylindrical or rectangular block, a chill-cast disc, pins or a combination of a disc with one or more attached pins. In some cases, small lugs are attached to a disc.

NOTE Sampling probes for use with liquid iron and steel can be obtained from a number of suppliers. The main features of several types of probes are given in Annexes A and B.

4.3.2 Sample from a product

The laboratory sample or the test sample may be selected from the batch sample at the location indicated in the product specification for the selection of material for mechanical testing, when available.

In the case of an iron casting, the test sample may be selected from a bar or block cast onto the casting.

In the case of a forging, the test sample may be selected from the initial starting material from which the forging has been made, or from prolongations of the forging or from additional forgings.

In the absence of requirements given in the product standard, or of a specification when ordering the product, the test sample may be selected from the sample for mechanical testing or from the test piece, or directly from the batch sample.

The laboratory sample or the test sample may be obtained from the batch sample by machining or any other appropriate means. Special considerations apply in the case of sampling for the determination of certain elements.

4.4 Preparation of a sample

4.4.1 Preliminary preparation of a sample

If any part of the sample is liable to be non representative in chemical composition, for example due to oxidation, it may be agreed, following an investigation to establish the nature and extent of any change in composition, to remove from the sample those parts that have changed. After this operation, the sample shall be protected in order to avoid any change in composition.

If necessary, the surface of the metal shall be laid completely bare at the location of machining, by any suitable means, to remove any coating that has been applied during manufacture. If necessary, the surface of the metal shall be degreased by means of a suitable solvent. Care shall be taken to ensure that the manner of degreasing does not affect the accuracy of the analysis.

4.4.2 Test sample in the form of chips

The test sample shall consist of chips of a regular size and shape. These may be obtained by methods such as drilling, milling or turning. The chips shall not be taken from a part of the sample that has been affected by the heat of a cutting tool.

The tools, machines and containers used during preparation of the sample shall be cleaned beforehand to prevent any contamination of the test sample.

Machining shall be carried out in such a way that the chips are not subject to overheating, as indicated by a change in the colour (blueing or blackening) of the chips. Unavoidable coloration of chips obtained from some types of alloy steels, for example manganese and austenitic steels, may be minimized by selection of appropriate tools and cutting speeds.

Depending on the technique of analysis, heat treatment under an adequate atmosphere or environment (to ensure that the chemical composition is not changed) may be performed to soften the sample for machining, provided that the product has been submitted to the same heat treatment. For some cases such as carbon or oxygen determination, heat treatment is not allowed.

The use of coolants during machining is only permitted in exceptional cases; after which the chips shall be cleaned by means of a suitable solvent that does not leave any deposit.

Chips shall be thoroughly mixed before weighing the test portion. For most purposes, the chips should be mixed by rolling the container on a level surface and/or gently tumbling the container.

4.4.3 Test sample in the form of fragments

Where drilling of the sample to obtain chips is impracticable, it shall be cut or broken into pieces. These pieces shall then be crushed using a percussion mortar or a vibratory grinding mill, also known as a disc mill or ring mill, to obtain a test sample in the form of small fragments, the whole of which passes through a sieve of a specified aperture size.

In some applications for the determination of carbon using a thermal method of analysis, the sample is crushed in a percussion mortar to obtain a test sample in the form of fragments with a particle size range of approximately 1 mm to 2 mm.

Equipment used for comminution shall be constructed from material that does not alter the sample composition. Suitable tests may be necessary to show that the use of such equipment does not affect the composition of the test sample in any way.

Comminution shall not be used for the preparation of samples of graphite-bearing cast irons.

The sieving operation shall be performed taking all precautions necessary to avoid contamination or loss of material. When sieving hard materials, care shall be taken to avoid damaging the fabric of the sieve.

The test sample shall be homogenized before weighing the test portion. Small fragments may be homogenized by stirring.

CAUTION — Finely-divided metals of particle size less than approximately 150 µm can present a fire risk. Ensure that there is adequate ventilation during comminution.

4.4.4 Test sample in the form of a solid block

4.4.4.1 Selection of the test sample

The test sample shall be obtained by cutting, from the batch sample or laboratory sample, a piece of size and shape suitable for the method of analysis. Samples shall be cut by sawing, abrasive cutting, shearing or punching.

In the absence of any indication in the product standard, analysis by a physical method shall be carried out on that part of the sample corresponding to a transverse section of the product, provided that the material has sufficient thickness.

4.4.4.2 Surface preparation of the test sample

The test sample shall be prepared to expose a surface suitable for the method of analysis. Preparation of a surface for analysis shall not be carried out on any part of a sample that has been thermally affected. The equipment used for sample preparation shall be designed to minimize overheating the sample and, where appropriate, shall incorporate systems of cooling.

The main types of equipment used for surface preparation are as follows:

a) A milling machine capable of removing a preselected depth of metal in a reproducible manner, for use with samples that are within a hardness range suitable for milling. The equipment shall be able to be used, if required, with a sample taken from a melt where the sample is still hot.

b) A grinding machine with a fixed, rotating or oscillating head capable of removing a preselected depth of metal in a reproducible manner.

c) A flat-bed linishing machine with abrasive grinding discs, or a machine with continuous abrasive belts, able to be used to prepare the surface of the test sample to varying grades of finish.

d) A machine for blasting with sand, grit, or metal shot, able to be used in special applications to clean the surface of the test sample.

For the preparation of ultra-low carbon (ULC) steel samples, a milling machine is recommended.

After preparation, the surface of the test sample shall be flat and free from imperfections that affect the accuracy of the analysis.

Cutting and surface preparation may be performed either manually or automatically. In the case of samples taken from melts, commercially available systems, which perform each stage of preparation automatically, may be used. Systems for the automatic preparation of surfaces of dual-thickness probe samples [see A.2.3 c)], and for the punching of slugs forming test portions, may incorporate facilities for the sand-blasting of the sample.

NOTE In order to soften the sample before punching, a heat treatment under an adequate atmosphere or environment (to ensure that the chemical composition is not changed) can be performed.

The abrasive materials used in the final stage of preparing the test sample shall be selected so as to avoid contaminating the surface with elements that are to be determined. The grit size of the abrasive shall be in accordance with the grade of surface finish required for the method of analysis.

In the case of optical emission spectrometric methods, an abrasive with a grade of 60 grit to 120 grit is normally suitable. In the case of X-ray fluorescence spectrometric methods, it shall be ensured that the method selected for surface preparation produces a grade of surface finish that is reproducible from sample to sample. In addition, there should be no smearing of the surface.

The effect of abrasive materials depends on the analytical method. When using optical emission spectrometric methods, the action of pre-sparking will normally clean the surface of the test sample by volatilizing any grinding contaminants. However, particular care shall be taken to avoid surface contamination when using a new abrasive disc.

When using X-ray fluorescence spectrometric methods, all the phases of surface preparation shall be examined for potential surface contamination effects.

The test sample shall be examined visually after preparation to establish that the surface is free from particulate matter or imperfections; the sample shall be resurfaced or discarded if imperfections are present. The test sample shall be dry and care shall be taken to protect the prepared surface from contamination.

4.4.5 Preparation of a test sample by remelting

A sample in the form of small pieces or chips, or a part of the batch sample itself, may be remelted in an atmosphere of argon using commercially available melting equipment. The sample is converted into a disc. Some types of remelting equipment incorporate facilities for the centrifugal casting of the disc.

NOTE Typically, the sample is 30 mm to 40 mm in diameter and 4 mm thick, which is suitable for analysis by a physical method.

Partial losses of some elements can occur during the remelting process. It is essential to ensure that any selective volatilization or segregation of elements, or any other change in composition, which occurs is quantitatively known and does not significantly influence the results of the analysis. Suitable tests shall be carried out to show that any change in composition is both small in magnitude and reproducible.

The equipment used and the method adopted for remelting shall be designed to prevent or minimize a change in composition and to ensure that any change is reproducible. A deoxidant, for example 0,1 % (mass fraction) zirconium, should be used during remelting.

Not all ferrous metals may be remelted in this manner.

This method shall not be used for sample preparation for the determination of an element that is subjected to a significant and non-reproducible change in composition when remelted.

4.5 Safety precautions

4.5.1 Personal protection

Personal protective equipment shall be provided to minimize the risk of injury during sampling and sample preparation operations. Provisions shall include protective clothing, hand protection and face visors resistant to splashes, for use during the sampling of liquid metal, and respiratory protection for use where necessary.

4.5.2 Machinery

The use of machinery for sampling and sample preparation shall be in accordance with appropriate national standards or procedures adopted by the manufacturer.

4.5.3 Hazardous materials

It is presupposed that procedures are in accordance with applicable legal requirements with regard to the use of solvents for the cleaning and drying of samples and test portions.

5 Liquid iron for steelmaking and pig-iron production

5.1 General

The following methods are applicable to the sampling of liquid blast-furnace iron intended for steelmaking or for the casting of pig-iron. The liquid iron is normally sampled from the blast furnace runner while the melt is poured into torpedo ladles or from transfer vessels or during secondary treatment processes in the ladle or during the casting of the melt into an ingot mould.

The chemical composition of cast iron can fluctuate during run-out from the blast furnace. Two or more samples shall be taken from the melt at timed intervals and an average composition determined.

When physical methods for analysis are used, the method of sampling shall be designed to chill the liquid metal in a manner that ensures that the metallurgical microstructure of the sample is suitable for the requirements of the method of analysis selected.