SLOVENSKI STANDARD
oSIST prEN ISO 14284:2022

01-januar-2022

Jeklo in železo - Vzorčenje in priprava vzorcev za ugotavljanje kemijske sestave (ISO/DIS 14284:2021)

Steel and iron - Sampling and preparation of samples for the determination of chemical composition (ISO/DIS 14284:2021)

Eisen un Stahl-Entnahme und Vorbereitung von Proben für die Bestimmung der chemischen Zusammensetzung (ISO/DIS 14284:2021)

Fontes et aciers - Prélèvement et préparation des échantillons pour la détermination de la composition chimique (ISO/DIS 14284:2021)

Ta slovenski standard je istoveten z: **prEN ISO 14284**

ICS:

77.080.01	Železne kovine na splošno	Ferrous metals in general

oSIST prEN ISO 14284:2022 **en,fr,de**

DRAFT INTERNATIONAL STANDARD

ISO/DIS 14284

ISO/TC **17**/SC **1**

Secretariat: **JISC**

Voting begins on:
2021-11-09

Voting terminates on:
2022-02-01

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

Fontes et aciers — Prélèvement et préparation des échantillons pour la détermination de la composition chimique

ICS: 77.080.01

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 14284:2021(E)

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Published in Switzerland

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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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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

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

— The figures have been modified and some new figures have been added.

— Some definitions have been modified and some definitions have been added to Clause 3.

— Some terms have been modified.

— Some paragraphs and sentences have been modified.

— Some new paragraphs and sentences have been added.

— Some new sampling probes have been added.

— The units have been changed to SI.

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

The following document is referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9147:1987, *Pig-irons — Definition and classification*

3 Terms and definitions

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

ISO and IEC maintain terminological 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 of analysis
method for the determination of chemical composition in which the sample is submitted to chemical reactions

3.2
physical method of analysis
method for the determination of chemical composition in which the determination is carried out without submitting the sample to chemical reactions, for example an optical emission spectrometric (OES) method or an X-ray fluorescence spectrometric (XRF) method

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

3.4
melt
liquid metal from which a sample is taken

3.5
spoon sampling
method in which a sample 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 obtained from the *spoon sampling* (3.5) method

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

3.8
immersion sampling
method of *probe sampling* (3.6) in which the probe is immersed in the *melt* (3.3); the sample chamber in the probe fills by ferrostatic pressure or gravity

3.9
suction sampling
method of *probe sampling* (3.7) in which the probe is immersed in the *melt* (3.4); 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; the sample chamber in the probe fills by the force of metal flow

3.11
probe sample
sample obtained from a *probe sampling* (3.7) method

3.12
cast product
steel or cast iron product which has not been subjected to deformation, for example, an ingot, a semi-finished product obtained by continuous casting, a casting

3.13
wrought product
general term for products obtained by hot and/or cold plastic deformation processes such as extruding, forging, hot rolling, cold rolling or drawing, either exclusively or in combinationExamples of wrought products are 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
sample 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), or part of a *laboratory sample* (3.15) taken from a batch sample, or part of a sample taken from the *melt* (3.4), brought to the appropriate condition required for analysisThe test sample may also be the batch sample itself or a sample taken from the melt

Note 1 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 taken from the *melt* (3.4), submitted to analysis. In some cases, the test portion may be selected from the *batch sample* (3.14) itself

Note 1 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), *grinding* (3.21) or pulverization

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

3.23
consignment
quantity of metal delivered at one time

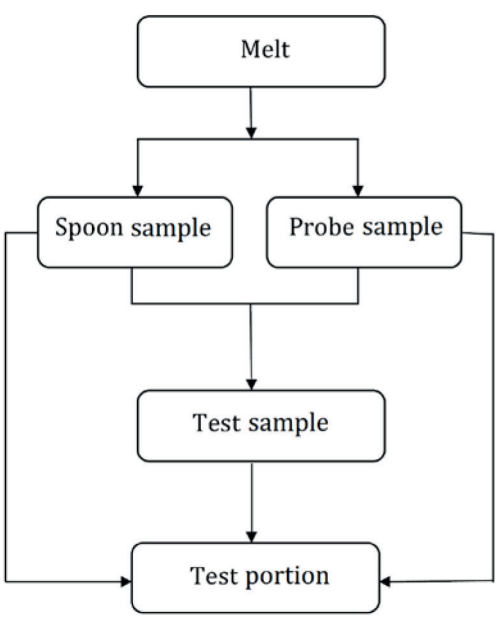
3.24
increment
quantity of metal obtained by sampling at one time from a *consignment* (3.23)

4 Requirements for sampling and sample preparation

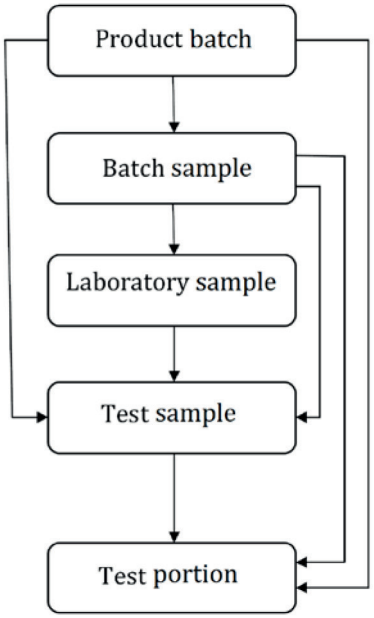
4.1 General

This clause covers the general requirements for the sampling, the sample and the sample preparation of liquid iron and steel. Special requirements apply to each category of liquid and solid metal and these are considered 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. Special considerations apply 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 samples for analysis, may be 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 porosity, 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 is consistent from sample to sample.

Analysis by some physical methods may 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 samples for analysis to be taken for re-analysis.

Samples for analysis 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 are essentially determined to ensure:

— 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 sample will be determined by the dimensions of the sample chamber. The dimensions for samples for analysis given in this document should be regarded as indicative only.

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 sample.

NOTE Sampling probes for use with liquid iron and steel may be obtained from a number of suppliers. The main features of several types of probe 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, turning or punching. 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 shall not be 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 which does not leave any deposit.

Chips shall be thoroughly mixed before weighing the test portion. For most purposes, it is advisable to mix the chips 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 which 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 comminution 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.