
**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

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.

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 of 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 www.iso.org/iso/foreword.html.

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

finishing

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](#).

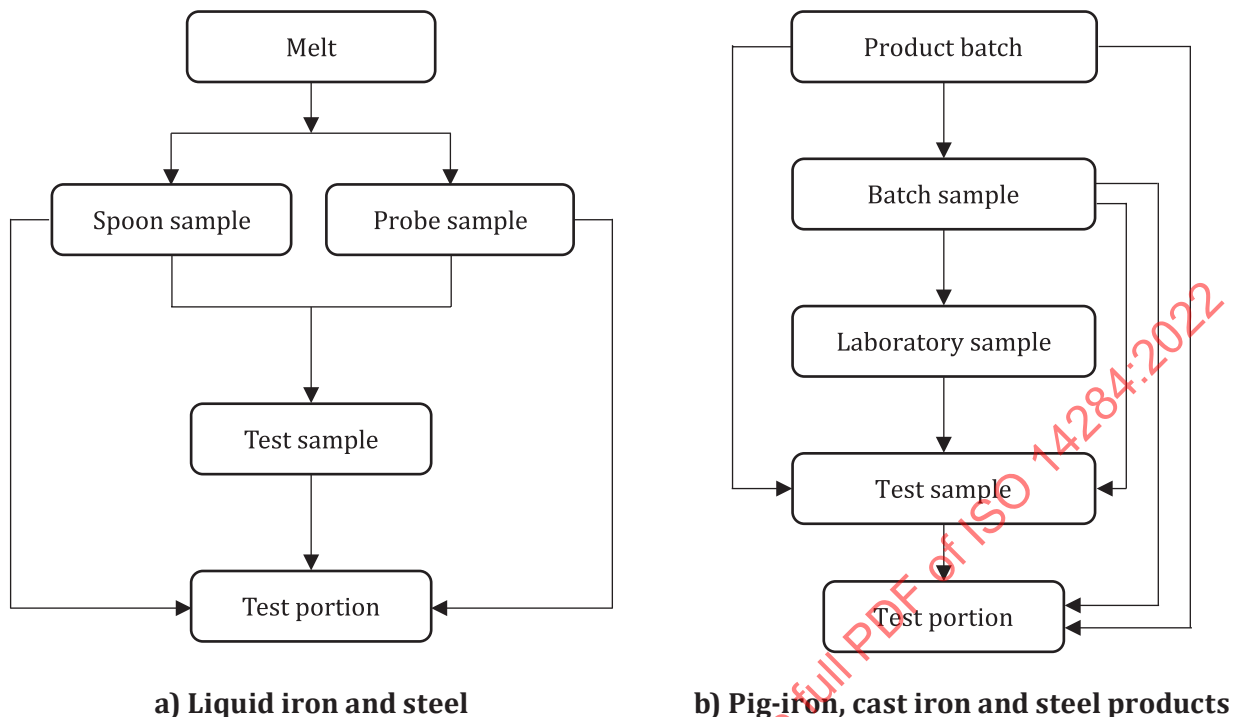


Figure 1 — Sequence of sampling and sample preparation

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 finishing 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.

5.2 Spoon sampling

5.2.1 Methods

For sampling from a melt, immerse a preheated steel spoon or a ceramic fiber spoon into the melt and fill with liquid iron. Withdraw the spoon and remove any slag by skimming the surface of the liquid iron in the spoon.

For sampling from a stream, introduce a preheated steel spoon or a ceramic fiber spoon into the stream from the ladle and fill it with liquid iron.

Pour the liquid iron from the spoon without delay into a metal mould to chill the iron as rapidly as possible. Remove the sample from the mould and break off any riser.

The liquid iron shall be poured into a cold mould to ensure adequate chilling. If necessary, the mould should be air-cooled before use. The mould shall be free from moisture.

A disc-shaped sample, commonly described as a coin sample, may be obtained using a two-piece steel/ copper/ copper-chromium mould. The mould is constructed in two pieces that are clamped together while in use: one piece is a flat chill plate, the other is a block with the mould cavity.

NOTE 1 Typically, a sample having 35 mm to 40 mm in diameter with a thickness varying from 5 mm to 6 mm can be used.

NOTE 2 In the case of cast irons, samples with thicknesses greater than 6 mm will probably not have a complete white cast iron microstructure.

The edge of the mould cavity may be tapered, to facilitate removal of the sample from the mould.

NOTE 3 For example, from 38 mm to 32 mm.

NOTE 4 A coin sample can be vertically or horizontally cast in the mould.

A coin sample with one or more attached pins may be obtained using a combination-type mould. The pins are broken off from the disc and used, if required, as test portions for analysis by a thermal method. An example of a combination-type mould for use with liquid iron intended for the production of cast iron is shown in [Figure 2](#).

A thin slab-shaped sample with a rounded end may be obtained using a cast iron or steel split-mould. The two halves of the mould are bevelled at the top to give a feeder head and are clamped together while in use. This type of mould can be preferred for use with liquid iron having high carbon content.

NOTE 5 Typically, the dimensions of the sample are 70 mm x 35 mm with a thickness of 4 mm.



5.2.2 Maintenance of equipment

Moulds should be remachined if the internal surfaces become worn. This avoids the need for additional machining of the sample during surface preparation.

5.3.1 General

Probe sampling is influenced by such factors as the angle and depth of immersion of the sampler in the melt, and immersion time may vary depending on the temperature of the liquid iron. These factors should be determined for the particular iron-making practice and thereafter strictly controlled to maintain the quality of the test sample.

5.3.2 Methods

For sampling from a melt, immerse a suitable immersion probe sampler into the melt at an angle as near as possible to the vertical plane.

When sampling from the runner of a blast furnace, select the position of immersion to give a sufficient depth of liquid metal for the use of a probe sampler. A depth of approximately 200 mm is adequate for most types of sampling probe.

For sampling from a stream of liquid iron, introduce a suitable suction probe sampler into the metal flow from a ladle, at an angle of approximately 45° to the vertical plane, at a position as near as possible to the nozzle of the vessel.

Withdraw the probe sampler from the melt after a predetermined interval of time, break it apart and allow the sample to cool in air.

5.4 Preparation of a test sample

5.4.1 Preliminary preparation

Remove any surface oxidation from a sample taken from the melt that can contaminate the test sample during subsequent preparation.

5.4.2 Test sample for a chemical method

For white microstructure cast iron, break the sample into small pieces and crush the pieces using a percussion mortar or a vibratory grinding mill to obtain a sufficient amount of test sample of particle size preferably less than about 150 µm.

For grey microstructure cast iron, obtain chips by drilling the sample at a low speed as described in [8.3.1](#).

5.4.3 Test sample for a thermal method

Break the pin of a disc-shaped sample into pieces of a suitable mass for use as test portions, or use the lugs of a probe sample.

For oxygen determination, the test portion shall be taken from the bulk of the disk sample.

Alternatively, crush the pin or lugs in a percussion mortar to obtain a sufficient amount of test sample of particle size approximately 1 mm to 2 mm.

Avoid the production of fine material during crushing. In the case of a slab-shaped sample, break the sample into small pieces and crush the pieces in a similar manner.

5.4.4 Test sample for a physical method

A white cast iron microstructure obtained by chill-casting shall be used for analysis by physical methods.

In the case of a disc-shaped sample, remove any lugs or pins, as necessary, and then grind the surface of the sample to obtain a surface free of contamination or oxidation that is representative of the sample. The amount of material to be removed in this way shall be determined for the chemical composition of the particular cast iron and the conditions of sampling. The thickness of the layer to be removed normally lies between 0,5 mm and 1 mm (see [Clause A.6](#)).

In the case of a slab-shaped sample, break the slab into two pieces to obtain a sample of suitable size for analysis.

Prepare the sample by grinding avoiding over-heating of its surface.

Particular care shall be taken when preparing the surface of thin samples. A chuck shall be specially designed to hold the sample securely during grinding operations.

6 Liquid iron for cast iron production

6.1 General

The following methods are applicable to the sampling of liquid iron from cupola furnaces and electric furnaces, from holding furnaces in duplex processes, and from ladles and treatment vessels.

Liquid iron intended for the production of iron castings can be subject to inhomogeneity, and particular care shall be taken in the design of strategies and methods of sampling to meet the requirements of the particular production process. For example, liquid iron in holding furnaces tends to stratify and sampling shall ensure that the analysis is representative of the melt as a whole.

In batch processes, two or more samples shall be taken from melting furnaces, preferably when approximately one-third and two-thirds of the melt has been discharged, and an average analysis determined. In continuous processes, samples shall be taken at regular intervals of time.

Methods of sampling are normally designed to chill the liquid metal of a sample cast from a spoon as rapidly as possible to produce a white cast iron metallurgical microstructure (graphite free). A white cast iron microstructure obtained by chill-casting shall be used for analysis by physical methods.

Non-chilled samples may also be used for wet chemistry determinations. In this case, samples may be specially cast from a spoon, or a test sample may be selected from a test bar or keel block intended for mechanical testing. Test bars or blocks are cast from the same metal as that used to produce the casting or castings.

By agreement with the customer, when large castings or large numbers of castings are produced, two or more samples should be taken.

Special considerations apply to the sampling and sample preparation of liquid iron for the determination of oxygen and nitrogen (see [6.5](#)).

6.2 Spoon sampling

6.2.1 General

Sampling should take place before any additions of inoculating substances have been made to the melt.

Alternatively, sufficient time should be allowed to elapse for the immediate effect of the additions to have faded and the melt should be thoroughly stirred before sampling. Failure to allow adequate standing time before taking the sample will seriously impair the representativeness of the sampling.

Spheroidal graphite cast iron is particularly difficult to sample due to the possibility of contamination by dross during the production process. In this case, a suitable sample may be obtained by filtering the melt using a ceramic disc.

In order to obtain a sample that is representative of the chemical composition of the cast product, the sample shall be taken after the addition of inoculating substances, except in case of automatic inoculation in stream.

6.2.2 Methods

A graphite spoon or a steel spoon lined with a layer of a refractory such as ganister or ceramic fiber spoon are suitable for use in accordance with one of the following methods:

- a) Remove any slag from the surface of the melt by skimming and then immerse a preheated spoon into the melt and fill with liquid iron.

b) Introduce a preheated spoon into the stream during pouring and fill with liquid iron.

6.2.3 Chilled sample

Pour the liquid iron from the spoon without delay into a split mould made from copper to obtain a sample in the form of a small, flat plate, 5 mm to 6 mm in thickness. Remove the sample from the mould as soon as it is solid to avoid overheating the mould and the risk of breakage of the sample, then break off any riser.

The sample, commonly described as a coin sample, can be circular, rectangular or square in shape.

NOTE 1 The related typical sizes are respectively 35 mm to 40 mm diameter, 50 mm × 27 mm and 50 mm × 50 mm.

NOTE 2 Generally, disc samples are vertically cast and rectangular and square samples are horizontally cast.

The mould is constructed in two pieces that are clamped together while in use: one piece is a flat chill plate, the other is a block with the mould cavity. The edge of the mould cavity may be tapered to facilitate removal of the sample from the mould.

A coin sample with one or more attached pins may be obtained using a combination-type mould. The pins are broken off from the disc and used, if required, as test portions for analysis by a thermal method.

NOTE 3 An example of a combination-type vertical mould of this type, commonly described as a book mould and made from copper or internally water-cooled copper is shown in [Figure 2](#). The sample obtained is a disc, 35 mm to 40 mm in diameter and 5 mm to 6 mm thick, with three 4 mm-diameter pins.

The temperature of the liquid iron in the spoon shall be as high as possible and compatible with the mould material. The mould shall be cold to ensure adequate chilling for the production of a test sample with a white cast iron microstructure. If necessary, the mould should be air-cooled before use. The mould shall be free from moisture.

In the case of process where samples are required to be taken at frequent intervals, several moulds shall be provided to ensure that a cold mould is available for use.

Thermal stress due to overheating of the mould can cause breakage of the coin sample and shall be avoided.

6.2.4 Non-chilled sample

Pour the liquid iron from the spoon without delay into a sand mould to obtain a cylindrical block-shaped sample.

NOTE 1 Typically, the cylindrical block shaped samples are 50 mm in diameter and 40 mm to 50 mm long.

Alternatively, a test sample may be selected from a test bar or keel block intended for mechanical testing. Test bars or blocks are cast either from liquid iron taken from a ladle using a spoon or, if a small hand-ladle is used for pouring, directly from the ladle itself. Bars may be vertically or horizontally cast in a sand mould.

NOTE 2 Typically, the bars are 30 mm in diameter and 150 mm long.

Samples shall be allowed to cool completely before removal from the mould.

Non-chilled samples shall be used for wet chemistry determinations only.

6.2.5 Maintenance of equipment

Keep spoons and metal moulds clean and dry. After use, remove any slag and skull and clean the surfaces of the mould with a wire brush.

Moulds should be remachined if the internal surfaces become worn. This avoids the need for additional machining of the sample during surface preparation.

6.3 Probe sampling

Probe sampling is used only to a limited extent in the manufacture of cast iron products. Sampling probes, if required, shall be designed to provide samples from the melt of a quality and metallurgical microstructure required by the method of analysis.

6.4 Preparation of a test sample

6.4.1 Preliminary preparation

Prepare the sample in accordance with one of the procedures described in [6.4.2](#), [6.4.3](#) or [6.4.4](#), depending upon the method selected for analysis.

6.4.2 Test sample for chemical methods

6.4.2.1 General

Machining to obtain chips should be carried out by drilling or turning at a low speed (100 r/min to 150 r/min) using a tungsten-carbide tipped tool, adjusting the speed and feed to produce chips of uniform size while avoiding the production of fine particles. Care shall be taken to avoid overheating both the sample and the tool.

In the case of grey cast irons, chips shall be as solid and compact as possible, with a mass of approximately 10 mg (100 chips per gram) in order to prevent crumbling and loss of graphite. Chips should not be cleaned with a solvent or treated magnetically because of the risk of altering the distribution of metal and graphite. A tool with a diameter of 10 mm is suitable for obtaining chips by drilling.

The size range of chips intended for the determination of total carbon should be 1 mm to 2 mm.

When machining is not practicable, the sample may be broken into pieces that are then crushed using a percussion mortar or vibratory grinding mill to obtain a sufficient amount of test sample, of particle size less than 150 µm. This method should only be used in cases where it is shown that comminution does not lead to contamination of the sample.

6.4.2.2 Methods

For a chilled sample, drill the sample, if practicable, discarding chips obtained from the surface.

For a non-chilled sample that shall only be used for wet chemistry determinations, in the case of a cylindrical block, drill a hole transversely at a position one-third along the length of the block. Then, drill another hole from the opposite side. Discard chips from one-third of the radial depth in both directions. Continue drilling through the centre of the block to obtain the test sample.

In the case of a test bar, use one of the following methods:

- a) Grind two flats on opposite sides of the bar and drill from one side to the other at a position one-third along the bar.
- b) Turn the test bar using a lathe with a maximum cut of 0,25 mm; do not use a cutting fluid or coolant. Use a radial cut from edge to centre, or face turn a cross-section of the bar; do not confine turning to the surface of the bar. Discard chips obtained from the surface of the bar.

For a non-machinable sample, break pieces from the sample or cut a 3 mm slice or disc from the cross-section near the bottom of a test bar. Crush these pieces using a percussion mortar or vibratory grinding mill to obtain a sufficient mass of test sample of particle size less than 150 µm.

6.4.3 Test sample for thermal methods

In the case of a chilled sample, remove the pin from the sample and break or cut the pin into pieces for use as test portions.

Alternatively, crush the pin in a percussion mortar to provide a test sample of particle size approximately 1 mm to 2 mm. Avoid the production of fine material during crushing.

NOTE Oxygen cannot be determined on pieces obtained by crushing.

6.4.4 Test sample for physical methods

In the case of a chilled sample, remove any pins and then use a fixed-head grinder to obtain a surface free of contamination or oxidation that is representative of the sample. The amount of material to be removed in this way shall be determined for the chemical composition of the particular cast iron and the conditions of sampling; the thickness of the layer to be removed is normally at least 1 mm.

Air-cooling is recommended during grinding in order to avoid overheating the sample. When the microstructure of the sample is suspected not to be full chill along its whole thickness, excessive grinding can lead to errors in analysis. Chilled samples shall be examined regularly in routine practice to ensure the suitability of the metallurgical microstructure of the prepared sample for the method of analysis.

Overheating of the sample during surface preparation shall be avoided. This can result in surface crazing that will affect the accuracy of the analysis.

Care shall be taken when preparing the surface of a thin coin-sample. A chuck shall be specially designed to hold the sample securely during grinding operations.

NOTE A fixed-head grinder is preferable to a swing grinder for surface preparation. The swing grinder cannot give a flat surface to the test sample.

6.5 Sampling and sample preparation for the determination of oxygen and nitrogen

6.5.1 General

The determination of oxygen and nitrogen is needed only to a limited extent in the production of castings. Sampling and sample preparation methods shall avoid contamination of the sample by oxygen and nitrogen (see 7.5).

6.5.2 Method

For the determination of oxygen and nitrogen, pins broken from a chill-cast sample are normally suitable. Such samples can be obtained from the melt using a spoon and casting the liquid iron, as described in 6.2, into a combination-type book mould to obtain pin-shaped samples of diameter 4 mm. For this purpose, modify the construction of the mould shown in Figure 2 by enlarging the three pin-shaped cavities to produce pins of the required diameter.

6.5.3 Preparation of the test portion

Remove all traces of surface oxidation from the pin by turning using a lathe and a tungsten-carbide-tipped tool. Use a separate parting tool to cut the pin transversely in order to obtain a test portion of a suitable mass for analysis. Avoid overheating the pin during the preparation of test portions.

Ensure that there is no delay between the preparation of the test portion and analysis.

7 Liquid steel for steel production

7.1 General

The following methods are applicable to the sampling of liquid steel from furnaces, ladles and other vessels, and from tundishes and moulds during the melting, secondary treatment and casting of the steel.

Special considerations apply to sampling and sample preparation of liquid steel for the determination of oxygen (see 7.5), nitrogen (see 7.5) and hydrogen (see 7.6).

7.2 Spoon sampling

7.2.1 Methods

For sampling from a melt, lower the spoon through the slag into the melt and fill it with liquid steel. The spoon shall first be immersed in the slag layer to coat it with slag so as to reduce chilling and prevent adhesion of the metal to the spoon. Withdraw the spoon and remove any slag by skimming the surface of the liquid steel in the spoon.

For sampling from a stream, introduce the spoon into the stream from the ladle and fill it with liquid steel. Then withdraw the spoon.

Care shall be taken when introducing the spoon into the stream because of the force of the liquid metal emerging from the nozzle; it can be necessary to reduce the rate of metal flow during sampling.

If necessary, add a measured quantity of deoxidant to the liquid steel in the spoon. When the liquid steel is quiescent (after an interval of up to 10 s), pour without interruption into a one-piece steel mould designed to produce a tapered cylindrical sample. The sample should be approximately 25 mm to 40 mm in diameter at the top and 20 mm to 35 mm in diameter at the base, and 40 mm to 75 mm long.

Remove the sample from the mould and cool in a manner that is designed to prevent cracking. Cool the samples sufficiently slowly to ensure ease of machining.

For sampling stainless steel, a refractory ring placed on a cast iron plate may be used as a mould; the ring should have a wall thickness of 10 mm to 12 mm. The sample is removed from the mould by breaking off the refractory.

A pin type sample may be taken by use of quartz tube with dropper from the spoon sample. It may be taken from the mould directly, if possible.

NOTE Aluminium wire is frequently used as deoxidant in spoon sampling, provided that aluminium does not cause interference in the method of analysis and that the determination of the aluminium content of the melt is not required. The amount of aluminium added is usually between 0,1 % (mass fraction) and 0,2 % (mass fraction). Other deoxidants, such as titanium or zirconium, can be used with similar restrictions.

7.2.2 Maintenance of equipment

Keep spoons and metal moulds clean and dry. After use, remove any slag and skull and clean the surfaces of the mould with a wire brush or any other appropriate means.

Moulds should be remachined if the internal surfaces become worn. This avoids the need for additional machining of the sample during surface preparation.

7.3 Probe sampling

7.3.1 General

The main features of the different types of commercially available sampling probes for use with liquid steel are described in [Annex A](#).

Probe sampling is influenced by such factors as the angle and depth of immersion of the sampler in the melt, and the time of immersion in the melt. These factors shall be determined for the particular conditions of composition and temperature of the steel concerned and thereafter strictly controlled to maintain the quality required for the analysis.

Precautions shall be taken to ensure that the operation of probe sampling does not contaminate the sample of liquid steel, particularly when sampling for the determination of elements present in low contents (for example: ULC steel samples). The selection of materials used for construction of the sampling probe, the design of the capping and entrance system, and the method of deoxidation shall be such as to minimize the risk of contamination (other than from the deoxidant itself).

7.3.2 Methods

For sampling from deep melts, such as melting furnaces and ladles, quickly immerse a suitable probe sampler through the slag layer into the melt as near as possible to the centre of the melt, at an angle as near as possible to 90°.

For sampling from shallow melts, such as in tundishes, and from the tops of ingot moulds and from continuous casting moulds, introduce the entry tube of a suitable suction sampling probe through the slag or covering powder into the melt. Create a partial vacuum in the sampler for 2 s to 4 s (depending on sampler shape and size) to fill the mould.

Some tundishes can contain a sufficient depth of liquid metal to permit the use of an immersion sampling probe.

For sampling from a stream, introduce a suitable stream sampling probe into the metal flow from a ladle, at an angle of 45°, at a position as near as possible to the nozzle of the ladle.

In case of determination of nitrogen, suction sampling probe should be used.

Care shall be taken when inserting the probe sampler into the stream; it can be necessary to reduce the rate of metal flow during sampling.

Withdraw the probe sampler from the melt after a predetermined interval of time and break it apart. Allow the probe sample to cool in air to a dull red colour, and then quench in water in a manner that does not cause cracking.

In some cases, probe samples are transported to the laboratory while still hot.

7.4 Preparation of a test sample

7.4.1 Preliminary preparation

Remove any surface oxidation from a sample taken from the melt that can contaminate the test sample during subsequent preparation.

7.4.2 Test sample for chemical methods

In the case of a spoon sample, drill the cylindrical sample at a point one-third from the bottom through to the centre of the sample, discarding the chips obtained from the surface layer of the sample.

Alternatively, remove the bottom third of the cylindrical sample using a cutting-off machine and mill across the whole of the exposed face of the remainder. 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.

In the case of a probe sample, obtain chips from the disc section of the sample by drilling or milling as described in [10.4.2](#).

7.4.3 Test sample for thermal methods

In the case of a probe sample with attached lugs, break off one of the lugs to obtain a test portion.

In the case of a dual-thickness probe sample, punch a slug from the thin section of the disc to obtain a test portion. Heat treatment under an adequate atmosphere or environment (to ensure that the chemical composition is not changed) may be performed to soften the probe sample for an easier punching if the Rockwell hardness of the sample exceeds approximately 25 HRC, provided that the product has been submitted to the same heat treatment.

In the case of a disc-and-pin or pin probe sample, cut off a test portion from the pin of a suitable mass for analysis.

In the case of a cylindrical sample, obtain chips by drilling or milling the sample.

Particular care shall be taken in the case of samples for the determination of carbon in low-carbon steels, to prevent contamination during the preparation of the test portion. Use tweezers for all manipulations.

7.4.4 Test sample for physical methods

In the case of a cylindrical sample, cut off the base of the sample, using an abrasive cutting-off disc or tool to obtain the test sample, usually 20 mm to 30 mm thick. The surface shall be milled, ground or lished before analysis.

In the case of a probe sample, remove any lugs or pins, as necessary, and then mill, grind or lish the surface of the disc to expose a surface which is representative of the sample. The amount of material to be removed in this way shall be determined for the chemical composition of the particular steel and the conditions of sampling; the thickness of the layer to be removed normally lies between 1 mm and 2 mm (see [Clause A.6](#)). In the case of a dual-thickness probe sample, prepare the thick section of the disc.

For ULC steels, the thickness of the layer to be removed shall normally be at least 2 mm.

In the case of samples of leaded steels, equipment used for surface preparation shall be placed in an enclosure and fitted with dust-extraction equipment.

CAUTION — Swarf arising from machining and the surface preparation of leaded steels and dust from dust extraction filter systems shall be collected and disposed of safely. It is presupposed that it is done in accordance with applicable regulations for waste materials containing lead.

7.5 Sampling and sample preparation for the determination of nitrogen and oxygen

7.5.1 Methods of sampling

Methods of sampling liquid steel for the determination of nitrogen and oxygen are based upon the use of commercially available sampling probes. The main features of the different types of probes are described in [Annex A](#). Methods of use shall be designed to ensure that sampling operations do not influence the equilibrium between carbon and oxygen in the melt. Avoid contamination of the sample and remove all surface oxidation at each stage in sample preparation.

Small appendages to probe samples, such as a pin of diameter less than about 5 mm or a lug, are generally not suitable for preparation as a test portion that is free from surface oxidation. A slug obtained by punching from a dual-thickness probe sample may be satisfactory. For some applications, it can be preferable to obtain a sample of larger mass by using a sampling probe filled by gravity.

Make sure that the probes are dry before sampling. Keep them under dry conditions until used.

In case of taking sample by use of quartz tube with dropper, see [7.2.1](#).

7.5.2 Preparation of the test portion

Remove oxidation products from the surface of the probe sample by abrasion in a manner that does not cause overheating.

Cut a slice from the disc of a probe sample. Then cut a cube-shaped test portion from this slice of a mass suitable for analysis.

Place the test portion in a stainless steel holding block or some other device to hold it firm and abrade each surface using a fine-cut file. Use tweezers for all manipulations.

Immerse the test portion in acetone or ethyl alcohol and dry in air or by exposure to a rough vacuum. Analyse immediately; there shall be no delay between the preparation of the test portion and analysis.

7.6 Sampling and sample preparation for the determination of hydrogen

7.6.1 General

Methods of sampling liquid steel for the determination of hydrogen are based on the use of commercial sampling probes. The main features of the different types of probes are described in [Annex B](#). Methods of use shall be designed to minimize and to control the rapid diffusion of hydrogen from the probe sample which occurs during sampling, storage of the sample, and preparation of the test portion. Losses by diffusion can be large at ambient temperatures, especially from small-diameter samples.

The probe sample shall be free from cracks and surface porosities and from moisture, particularly entrapped water. The condition of the test portion can strongly influence the analysis. If a suction sampling probe is used, the method of operation shall be designed to avoid the risk of introduction of moisture into the sample.

The choice of a method of sampling is dependent on the temperature of the melt, the method of analysis and the required accuracy. These relationships shall be investigated to establish a suitable method for a particular steelmaking practice that will give samples of the quality required. Procedural detail shall be rigidly adhered to, in order to obtain consistency in the quality of analysis.

Maintain the probe sample and the test portion at a temperature that is as low as possible at all stages following sampling, and during storage and sample preparation. The sample shall be stored in a refrigerant; either liquefied nitrogen or a mixture of acetone and solid carbon dioxide in the form of slurry, freezer or cold box with solid carbon dioxide is suitable.

Such storage shall be down for ferritic steels. Diffusion of hydrogen in austenitic steels is slow but for any particular material, in the absence of experimental evidence, the suggested refrigerant storage shall be used.

The probe sample and the test portion shall be kept cold when cutting the sample and during the preparation of the test portion. Cooling may be carried out by immersion in a refrigerant. Any moisture present on the surface of the test portion after cooling shall be removed. The test portion shall be immersed in acetone and then dried by exposure for a few seconds to a rough vacuum.

Samples that have been inadequately cooled or stored shall be discarded.

Preparation of the surface of the test portion by abrasion shall be kept to a minimum, consistent with the need to remove all oxidation products and surface imperfections. The test portion shall be analysed immediately after preparation.

7.6.2 Methods of sampling

A range of commercially-available sampling probes, designed to provide pin-shaped or pencil-shaped samples of varying diameter (see [Annex B](#)) is available. Use the selected sampling probe in accordance with the manufacturer's instructions.

The probe sample shall be quenched in cold water and the water shall be stirred vigorously and continuously during quenching. There shall be no delay; quenching shall take place within an interval not greater than 10 s after taking the sample. The silica sheath used as the sample mould shall be removed quickly to allow rapid cooling.

When the sample is sufficiently cooled, immerse the probe sample in a refrigerant for storage and transport it to the laboratory.

If the probe is designed to capture diffusible hydrogen, it shall be quenched in order to be cooled sufficiently for handling purposes.

In case of taking sample by use of quartz tube with dropper, see [7.2.1](#).

7.6.3 Preparation of the test portion

Cut a test portion of a suitable mass for analysis from the central sections of the probe sample. Cutting shall be carried out in such a manner as to minimize heating the probe sample. Use a copious flow of cooling fluid during cutting or cool the sample at frequent intervals, or use a combination of both methods of cooling.

Prepare the surface of the test portion by filing, grit-blasting or light grinding. If filing is used, abrade the surface by hand with a fine-cut file. If grit-blasting is used, a grit-blasting machine shall be reserved exclusively for this purpose to avoid contamination of the test portion from the grit. If grinding is used, cool the test portion at frequent intervals.

Degrease the test portion by immersion in acetone, dry by exposure to a rough vacuum and analyse immediately. Alternatively, the test portion may be degreased by immersion in 2-propanol (isopropyl alcohol) and then dried with diethyl ether.

8 Pig-irons

8.1 General

The following methods are applicable to the sampling of iron from the blast furnace, which has been cast into blocks of a simple shape, known as pigs, usually in the form of a double trapeze or some other similar shape. The various types of pig-irons are classified in ISO 9147.

Special care shall be taken to ensure the collection of a representative sample for pig-irons.

8.2 Increment sampling

8.2.1 Number of increments

The number of pigs taken as incremental samples shall be representative of the batch or consignment. In the case of a consignment supplied in bulk, if there is no other agreement between the supplier and purchaser, the minimum number of pigs to be taken from a consignment shall be in accordance with [Table 1](#) (see ISO 9147).

Table 1 — Minimum number of pigs to be taken as incremental samples from a consignment

Mass of consignment t	Minimum number of pigs
≤10	9
>10 to ≤20	11
>20 to ≤40	12
>40 to ≤80	14
>80 to ≤160	16
>160 to ≤300	18
>300 to ≤600	21
>600	24

8.2.2 Methods

During unloading or loading operations, or any other displacement of a consignment, obtain pigs as incremental samples at intervals of time or mass that are approximately equal.

In the case of a consignment supplied in wagons or trucks, the points of sampling shall be disposed in a definite order. For example, from five positions, that is at the centre of the wagon, and at one-sixth of the distance from the corners of the wagons along the two diagonals.

In the case of a stockpile, throw a rope having a certain number of knots onto the pile and take the pigs touched by these knots. Repeat the operation until a sufficient number of pigs is obtained.

Where it is not possible to gain access to the whole of the surface of the stockpile, or where access to the stockpile is unsafe, the locations of sampling shall be disposed in a definite order on the surface of the stockpile.

Alternatively, use a mechanical shovel to obtain a number of subsamples from positions in the stockpile selected at random. Then select one pig at random from each subsample.

8.2.3 Consignment of mixed pig-irons

A consignment of pig-iron may contain a number of different batches of pig-iron, which may be from different sources. If pigs of different shapes and sizes can be distinguished in the consignment, a visual assessment shall be made of the proportions of each type of pig-iron present.

Incremental samples shall then be taken of each type of pig-iron in the consignment to constitute separate subsamples of pigs in order to obtain a weighted average analysis for the consignment.

8.3 Preparation of a test sample

8.3.1 General

If the pigs taken as incremental samples contain remanent magnetism as a result of handling with magnetic grabs, they shall be demagnetized by means of a demagnetizing coil to prevent separation of coarse and fine particles during drilling.

Machining of the sample to obtain chips should be carried out by drilling at a low speed (100 r/min to 150 r/min) using a freshly sharpened tool, adjusting the speed and feed to produce chips of uniform size with a minimum amount of fine particles. A drill with a diameter of 12 mm to 14 mm is suitable for obtaining chips. The drill shall be resharpened at frequent intervals and care shall be taken to avoid overheating both the sample and the tool.

For some types of iron, for example oxygen-blown iron, it can be necessary to use a tungsten-carbide tipped drill.

Chips shall be as solid and compact as possible in order to prevent crumbling and loss of graphite. The particle size range of chips intended for the determination of carbon shall be approximately 1 mm to 2 mm.

Milling shall not be employed owing to the high proportion of fines produced.

The prepared sample shall not be cleaned with a solvent or treated magnetically because of the risk of altering the distribution of metal and graphite.

8.3.2 Test sample for chemical methods

The method of preparation of each of the incremental samples shall be in accordance with one of the following methods:

- For machinable iron, clean one face of the pig by grinding at a position midway along the length and width to expose an area of metallic surface of at least 50 mm in diameter. Drill a hole through the cross-section of the pig; cease drilling at a point approximately 5 mm from the opposite face. If necessary, drill another hole parallel to the first hole, see [Figure 3](#).
- For non-machinable iron, break the pig at a position midway along its length. Break off pieces from the fractured face, crush these pieces to a particle size of approximately 5 mm and then grind using a vibratory grinding mill to a particle size of less than 150 µm.

Mix equal quantities of the material obtained from each pig. From this mixture, obtain a sample of sufficient mass for analysis by coning and quartering.

Alternatively, analyse the material obtained from each pig separately to obtain an average value for the consignment.

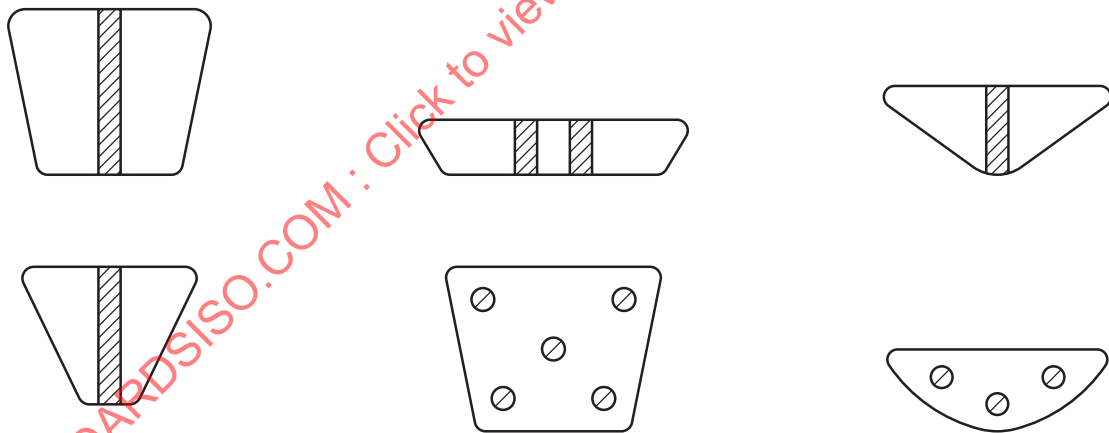


Figure 3 — Sampling positions for pig irons

8.3.3 Test sample for thermal methods

8.3.3.1 General

The method of preparation for each of the incremental samples shall be in accordance with either [8.3.3.2](#) or [8.3.3.3](#), depending upon the condition of the pigs and the type of sample required for analysis.

8.3.3.2 Sample in the form of chips or fragments

For machinable iron, at the centre of each pig, and on opposite faces, drill a hole 12 mm to 14 mm in diameter. Remove scale and any other impurities around the holes on both sides of the pig. Then drill

another hole coaxial with the first hole, 20 mm to 24 mm in diameter, in such a manner that large chips, of size approximately 1 mm to 2 mm, are obtained.

For non-machinable iron, obtain small pieces from the pig as described in 8.3.2 b), and then crush these pieces using a percussion mortar to a particle size of between approximately 1 mm and 2 mm.

Mix equal quantities of the material obtained from each pig. From this mixture, obtain a test sample by coning and quartering.

Alternatively, analyse the material obtained from each pig separately to obtain an average value for the consignment.

8.3.3.3 Sample in the form of a solid block

Cut a slice, approximately 3 mm thick, from the complete cross-section of the pig at a position midway along its length and clean the edges by grinding. From this slice, cut pieces at positions corresponding to those shown in Figure 3 to obtain test portions of a mass suitable for analysis.

Alternatively, saw or break the pig at a position mid-way along its length. Using a trepanning tool, drill either three or five holes at positions corresponding to those shown in Figure 3 to obtain pins of diameter approximately 3 mm. Break the pins into pieces to obtain test portions of a mass suitable for analysis.

Analyse a representative number of the test portions to obtain an average value for each pig.

8.3.4 Test sample for physical methods

Prepare the sample in a suitable form by remelting small pieces of the sample (see 4.4.5).

9 Cast iron products

9.1 General

The location and method of taking a laboratory sample or a test sample from a cast iron product shall be agreed between the supplier and purchaser according to one of the methods described in 9.2.2, 9.2.3 or 9.2.4.

Special care shall be taken to ensure that a representative test sample is collected from cast iron products. There can be differences in chemical composition, particularly in the contents of carbon, sulfur, phosphorus, manganese and magnesium, between the selected sample and that of the casting or castings as a whole. Segregated elements can concentrate towards the upper surfaces of a casting and under cores; these regions shall be avoided when selecting a laboratory sample or a test sample. Sectional dimensions and areas of differential heating or cooling require special attention. Careful consideration is necessary in the design of sampling strategies for lamellar graphite cast iron with high phosphorus content, and malleable and spheroidal graphite cast irons. Particular attention shall be taken when sampling grey cast irons to ensure that the test sample is representative of the chemical composition of the product, especially where segregation is suspected.

9.2 Sampling and sample preparation

9.2.1 General

Sampling and sample preparation shall be in accordance with the grade of cast iron and type of casting, and the method selected for analysis.

The batch sample or laboratory sample shall be cleaned by scratch-brushing, grinding or shot-blasting, to remove adhering particles of sand and to expose areas of metallic surface. It shall be ensured that both the inner and outer surfaces of hollow castings are clean.

9.2.2 Test sample for chemical methods

9.2.2.1 General

Machining of the sample to obtain chips should be carried out by drilling or turning at a low speed (100 r/min to 150 r/min) using a tungsten-carbide tipped tool, adjusting the speed and feed to produce chips of uniform size with a minimum amount of fine particles. Care shall be taken to avoid overheating both the sample and the tool. There is a risk of fracture of the tool when using a carbide-tipped drill, in which case the chips shall be discarded.

Milling shall not be employed owing to the high proportion of fines produced.

Chips shall be as solid and compact as possible, with a mass of approximately 10 mg (100 chips per gram), in order to prevent crumbling of graphite. Chips shall not be cleaned with a solvent or treated magnetically because of the risk of altering the distribution of metal and graphite. A tool with a diameter of 10 mm is suitable for obtaining chips by drilling.

The size range of chips intended for the determination of carbon or nitrogen should be approximately 1 mm to 2 mm.

When machining is not practicable, the sample may be broken into pieces that are then crushed using a percussion mortar or a vibratory grinding mill to obtain a sufficient mass of test sample, of particle size less than 150 μm . This method should only be used in cases where it is shown that comminution does not lead to contamination of the sample.

9.2.2.2 Methods

The method of sampling and sample preparation shall be in accordance with the type of cast iron as follows:

- a) For grey cast irons, obtain chips from the central section of a casting that is from an area representing approximately one-third of a complete section of the casting. Do not use chips obtained from the as-cast surface for analysis. Where possible and depending upon the shape of the casting, obtain the chips by drilling the casting at several positions. Mix chips obtained in this way to constitute the test sample.

For a casting of large section, it might be impracticable to drill through the casting. In such a case, the aim should be to drill half-way through the section of the casting.

In the case of a hollow casting such as a pipe, drill completely through the wall of the pipe at each end and in the middle, with the axes of the three drill holes at 120° to each other.

In the case of a large casting, obtain a laboratory sample of diameter 3 mm to 5 mm by using a trepanning tool. Break the sample into small pieces and crush pieces using a percussion mortar or vibratory grinding mill to obtain a sufficient mass of test sample having particle size less than 150 μm .

- b) For malleable cast irons, the test sample should, wherever possible, be obtained prior to treatment by annealing.

Annealing causes major segregation, so the sample taken from an annealed casting shall represent the full cross-section of that casting. Particular care shall be taken when obtaining a sample from a casting with variable sectional thicknesses.

If annealed material must be analysed, remove a complete cross-section by machining, break into pieces and crush in a percussion mortar or disc mill. Separate the coarse and fine fractions using a 150 μm sieve and determine the mass of each fraction. Thoroughly mix each of the fractions separately and weigh proportionate amounts to obtain a representative test sample.

- c) For white cast irons and alloy cast irons, it can be possible to obtain the test sample by drilling as described in a).

Where drilling is impracticable, cut thin slices from the batch sample or laboratory sample, preferably complete cross-sections, using a saw or, if necessary, an abrasive cutting disc. If an abrasive cutting disc is used, remove any heat-affected zone.

Break the slices into pieces and crush in a percussion mortar or vibratory grinding mill to obtain a sufficient mass of test sample having particle size less than 150 µm.

NOTE Malleable cast iron products are particularly susceptible to segregation of manganese sulfide where the manganese to sulfur ratio exceeds 2:1.

9.2.3 Sample in the form of a solid block for analysis by thermal methods

Cut a thin slice from the batch sample or laboratory sample as described in 9.2.2.2 c).

In the case of a large casting, obtain a test sample of diameter 3 mm to 5 mm by using a trepanning tool. Break off pieces from the test sample, or cut using a saw to obtain a number of test portions of suitable mass for analysis. Analyse a representative number of pieces and obtain an average value. The mass of a piece selected as a test portion should not be less than approximately 0,3 g.

9.2.4 Test sample for physical methods

Use a saw or an abrasive cutting disc to cut a sample of suitable size from the batch sample or the laboratory sample.

Prepare the cut surface by grinding using a fixed-head machine, by finishing or by a combination of both methods. Air-cooling is recommended to avoid overheating the sample. Liquid coolants shall not be used.

The test sample is prepared by remelting (see 4.4.5). For this purpose, break pieces from the complete cross-section of the sample. Remelt a representative number of these pieces to obtain a test sample.

The method selected for remelting shall produce a chill-cast sample with a white cast iron microstructure. Particular attention shall be given to the requirements specified in 4.4.5 concerning partial losses of elements.

NOTE 1 A fixed-head grinder is preferable to a swing grinder for surface preparation. The swing grinder cannot give a flat surface to the test sample.

NOTE 2 Samples obtained from cast iron products that contain free graphite are not suitable for analysis by a physical method. In such cases, it is preferable to remelt the samples to obtain a white cast iron microstructure.

10 Steel products

10.1 General

The location and method of selecting a laboratory sample or a test sample from a batch sample should be agreed between the supplier and purchaser according to one of the methods described in 10.2 and 10.3.

The laboratory sample or test sample may be taken from the batch sample at the location indicated in the product standard for the selection of material for mechanical testing, or as specified in ISO 377. See also 4.3.2.

Special considerations apply to the sampling and sample preparation of leaded steel products (see 10.5), and of steel products for the determination of oxygen (see 10.6) and hydrogen (see 10.7).

10.2 Selection of a laboratory sample or a test sample from a cast product

From a cast product of large section, obtain a test sample in the form of chips at a point midway between the outside and centre of the section, by drilling parallel to the axis. If this is not practicable, obtain the test sample by drilling the section from the side and collecting chips that represent the part of the section midway between the outside and centre.

Alternatively, where a sample is needed in the form of a solid block, cut a laboratory sample from the product by machining or by any other appropriate means at the half or quarter of its cross-section.

10.3 Selection of a laboratory sample or a test sample from a wrought product

10.3.1 General

For a rolled product, the process of selecting a laboratory sample shall be carried out on a section of the product perpendicular to the direction of rolling and at one end of the product.

Methods of obtaining a test sample in the form of a solid block or chips are described in [10.3.2](#) by reference to products of differing cross-sections.

10.3.2 Sections

Cut a laboratory sample from the batch sample transversely in the form of a slice.

To obtain a test sample in the form of a solid block, cut a piece from the laboratory sample of a suitable size for the method of analysis.

To obtain a test sample in the form of chips, mill the whole of the cross-sectional area of the laboratory sample. Where milling is impracticable, drilling may be employed but is not recommended for rimming steels. The most suitable sampling position for block and chips sample depends on the shape of the section, as follows:

- a) For a section of symmetrical shape, for example a billet, a round, a slab, obtain chips by drilling a cross-sectional cut face in a direction parallel to the longitudinal axis at points midway between the centre and edges [see [Figures 4](#) a) and b)].
- b) For a section of complex shape, for example, an angle, a tee, a channel, a beam, obtain chips by drilling at points indicated in [Figures 4](#) c), d), e), f) and g) leaving a clearance of at least 1 mm around the drill.
- c) For a rail, obtain chips by drilling a hole, of diameter 20 mm to 25 mm, in the head of the rail at a position halfway between the centreline and edge of the rail [see [Figures 4](#) h) and i)].

Where drilling into the end or cut face of the section is impracticable, chips may be obtained by drilling inwards from the surface perpendicular to the main axis.

10.3.3 Plates or slabs

Cut a laboratory sample, of a suitable size for the preparation of a test sample in the form of a solid block or chips, from a point midway between the centreline and the outer edge of the plate. [in the example shown in [Figure 4](#) j), the laboratory sample is 50 mm wide.] Where this is not practicable, the sample shall be taken from a position agreed between the supplier and purchaser as being representative of the plate composition.

10.3.4 Light sections, bars, rods, sheets, strips and wires

Where the batch sample is of sufficient cross-sectional area, cut a slice transversely to constitute the laboratory sample and obtain a test sample as described in [10.3.2](#) or as follows:

Where the batch sample is of insufficient cross-section for machining, for example, a thin gauge sheet, a strip, a wire, mill over the combined transverse-sections obtained either by bundling the material after cutting into suitable lengths, or by folding.

Where the sample sheet or strip is thin but is wide enough, mill over the combined longitudinal or transverse sections in a zone midway between the centreline and the outer edge of a sheet or strip [in the example shown in [Figure 4 j\)](#)] obtained in the same way.

For a sheet or a strip where the rolling direction is unknown, take lengths of strip in two directions at right angles, and combine the resulting samples.

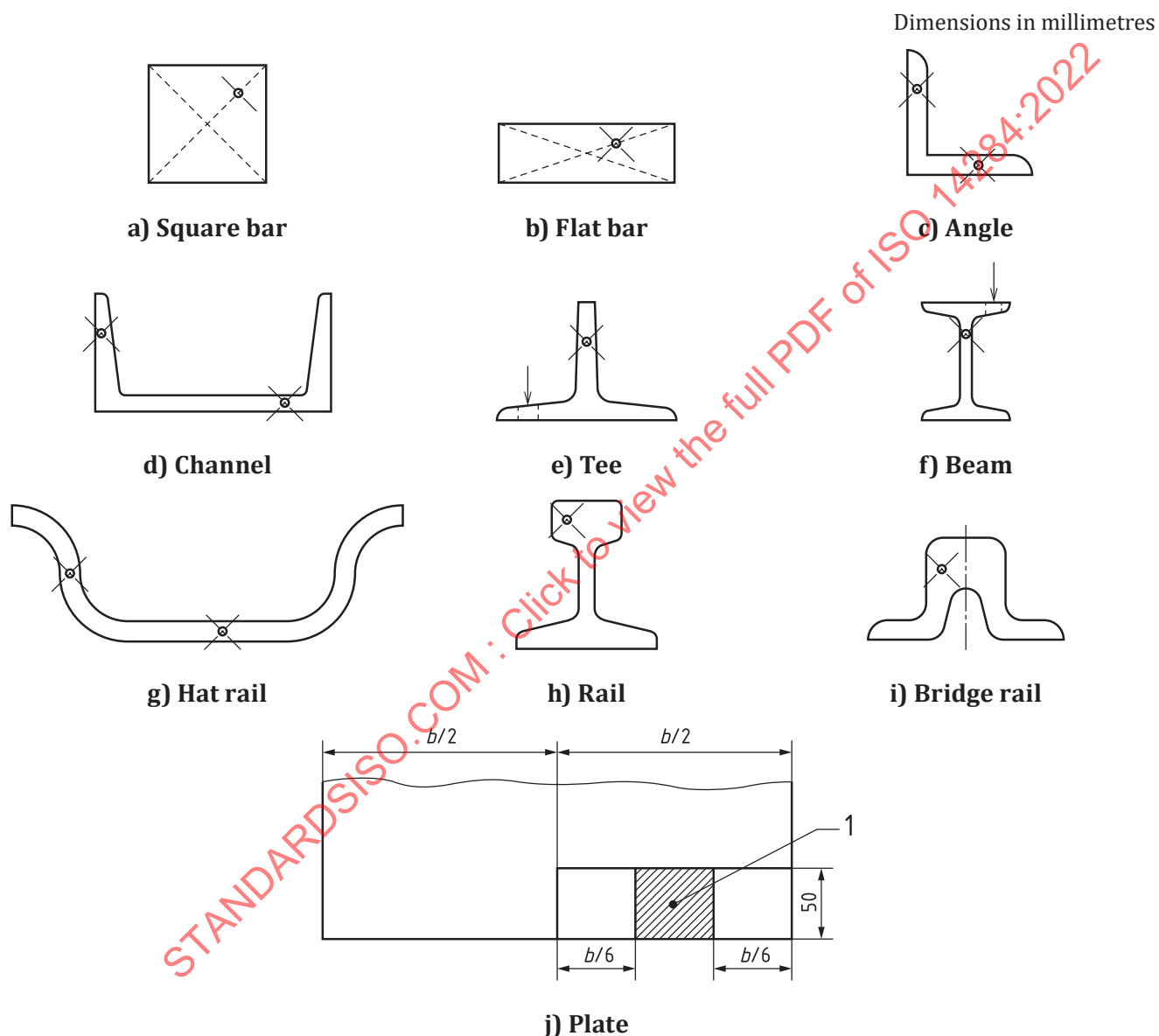


Figure 4 — Sampling positions for steel sections

10.3.5 Tubes and pipes

Sampling may be in accordance with one of the following methods:

- a) Cut a laboratory sample at a position 90° from the weld in a welded product.
- b) Cut across the tube and turn or mill across the cut face to obtain a test sample in the form of chips. Tubes with a small cross-section may be flattened before milling.
- c) Remove oxidized layer on the sample surface and drill through the wall of the tube or of the pipe, at several positions around its circumference, to obtain a test sample in the form of chips.

10.4 Preparation of a test sample

10.4.1 General

Methods for the sample preparation of steel products shall be in accordance with the general requirements specified in [4.4](#). Specific requirements are described in [10.4.2](#) and [10.4.3](#).

10.4.2 Test sample in the form of chips

Chips obtained by machining shall be sufficiently small to avoid or minimize the need for subsequent fragmentation to prepare the test sample. The size of the chips should be such that the mass of individual chips is approximately 10 mg (100 chips per gram) for unalloyed and low-alloy steels, and approximately 2,5 mg (400 chips per gram) for high-alloy steels.

If the chips are not small enough for analysis, crush them in a percussion mortar.

NOTE Crushing is not possible for ductile steel grades.

Machining shall be carried out in such a manner as to avoid the production of fine material. In the case of a test sample which contains fine powder, that is particles of size less than approximately 50 µm (500 µm for graphite carbon, sulfur and other elements that tend to segregate depending on chip size), separate the coarse from the fine particles and determine the mass of each size fraction. Weigh proportionate parts of each fraction to obtain a representative test sample.

In the case of a test sample for the determination of nitrogen, contamination of the chips can occur during machining caused by atmospheric nitrogenation of fine particles. Obtain the test sample by machining the laboratory sample in such a manner as to avoid, as far as possible, the production of particles of a size less than approximately 50 µm and preferably under argon atmosphere. Store the chips in an airtight container.

In the case of a test sample for the determination of very low carbon contents (ULC), for example, in interstitial-free steels, the chips may be contaminated by carbonaceous materials present in the atmosphere or from other sources. The chips shall be stored in an airtight container or preferably under inert atmosphere. It is preferable to remove surface carbon, for example by preheating just before the determination, or to determine the surface and internal carbon separately. Alternatively, a test portion in a solid form may be selected, such as a slug obtained by punching.

10.4.3 Test sample in the form of a solid block

In the case of products of thin section, such as strips or sheets, test portions for analysis by thermal methods may be produced by nibbling small pieces from the edge of the product. Alternatively, slugs of thickness 4 mm to 6 mm may be obtained by punching.

In the case of a batch sample, laboratory sample or test sample for analysis by physical methods, the sample shall be prepared by using an abrasive cutting-off tool to obtain about 10 mm to 30 mm thickness. The surface of the sample shall be finished before analysis.

In the case of batch samples with a thickness of approximately 1,5 mm or less, it is necessary to reduce the local heating that occurs when an electrical discharge is produced when using an optical emission spectrometric method. For example, the edges of the test sample may be electrically welded to a small block of steel, the sample may be embedded, for example in tin, leaving one surface exposed, or overheating of sample may be avoided by placing the sample on a copper piece with thickness of 3 mm to 5 mm.

10.5 Sampling of leaded steel

Care shall be taken to minimize the creation of dust particles during all sampling and sample preparation operations.

Select a laboratory sample from the batch sample by sawing.

Chips shall be obtained by milling at a low speed to avoid overheating the sample and the creation of dust.

Equipment used for the surface preparation of a test sample by a physical method shall be enclosed and fitted with dust-extraction equipment.

CAUTION — Swarf arising from machining and surface preparation of leaded steels and dust from dust-extraction filter systems shall be collected and disposed of safely. It is presupposed that it is done in accordance with local applicable regulations for waste materials containing lead.

10.6 Sampling and sample preparation for the determination of oxygen

10.6.1 General

It is essential to avoid contamination of the sample and to remove any surface oxidation at each stage of sampling and sample preparation.

Avoid any contact of the fingers with the test portion; use tweezers for manipulation. In the case of steels with very low oxygen contents, machining of the test portion shall be carried out under the protection of an inert gas.

10.6.2 Methods of sampling

Sampling should be in accordance with one of the methods described below.

- a) Cut a suitably shaped laboratory sample using a mechanical saw. The sample may, for example, be in the form of a small plate or disc. By using a hand saw, cut a test portion from this sample of a mass that is suitable for analysis.
- b) Cut a laboratory sample in the form of a slice of thickness 3 mm to 4 mm. Finish the surfaces of the sample using at least 60 grit abrasive paper made of silicon carbide and then abrade by using a burr, that is a rotary tool with cutting teeth, at a speed of approximately 30 000 r/min.

The condition of the surfaces of the sample after preparation shall be smooth, metallicly bright and free from imperfections.

Punch a slug from the sample of a suitable mass for analysis to constitute a test portion using a punch of diameter 4 mm to 6 mm. Carry out the punching operation in such a manner that the test portion falls into a glass container that is purged with argon or nitrogen and may be closed with a cap or stopper.

- c) Cut a rectangular laboratory sample, approximately 10 mm wide and 100 mm long. Turn the sample using a lathe at a speed of approximately 1 000 r/min to obtain a diameter of approximately 7 mm; continue turning at a controlled feed rate of approximately 0,1 mm to 0,15 mm per revolution at a speed of 800 r/min to 1 000 r/min to reduce the diameter of the sample to 6 mm. The condition

of the surfaces of the sample after preparation shall be smooth, metallicly bright and free from imperfections. Cooling lubricants shall not be used during the machining process.

By using a hand saw, cut a test portion from turned sample of a mass that is suitable for analysis.

10.6.3 Preparation of a test portion

In the case of 10.6.2 b), if the test portion and laboratory sample have no oxidation, the test portion may be used directly after it is picked up from the punching machine (after storage of limited duration in a glass flask). In the case of 10.6.2 a) and c), place the test portion on a stainless steel holding block or some other device to hold the test portion firm; abrade the surfaces using a fine-cut file or a burr [see 10.6.2 b)].

In the case of a test portion obtained using the method described in 10.6.2 c), the cylindrical surface of the test portion shall be sufficiently smooth to dispense with the need for filing. However, each of the two end faces should be prepared using a file. Immerse the test portion in acetone and dry in air or by exposure to a rough vacuum; analyse immediately.

There should be no delay between the preparation of the test portion and analysis.

10.7 Sampling and sample preparation for the determination of hydrogen

10.7.1 General

Methods shall be designed to minimize and control the rapid diffusion of hydrogen from the sample, which occurs during sampling, storage of the sample and preparation of the test portion. It is emphasised to take account that in case of austenitic matrixes, less losses of hydrogen occurs. The sample shall be free from cracks, surface porosities and moisture. The condition of the test portion can strongly influence the analysis; methods of analysis may differ in their sensitivity regarding the presence of water.

Procedural detail should be rigidly adhered to in order to obtain consistency in the quality of analysis.

Losses of hydrogen from the sample by diffusion may be large at ambient temperatures, especially from samples with thin cross-sections. Maintain the laboratory sample, test sample and test portion at a temperature that is as low as possible at all stages during sampling, storage and sample preparation. The test sample shall be stored in a refrigerant: either liquefied nitrogen or a mixture of acetone and solid carbon dioxide in the form of slurry are suitable.

The sample and the test portion shall be kept cold when cutting the sample and during preparation of the test portion. A copious flow of cooling fluid should be used during all machining operations, the sample and test portion shall be cooled at frequent intervals or a combination of both methods shall be used. Cooling may be carried out by immersion in a refrigerant. Samples of large section shall be packed with solid carbon dioxide in a manner that ensures the maintenance of good thermal contact with the sample. During intervals between machining, rough-cut pieces shall be returned to storage in a refrigerant.

Any moisture present on the surface of the test portion after cooling shall be removed. The test portion shall be immersed in acetone and then dried by exposure to a rough vacuum for a few seconds.

Samples that have been inadequately cooled or stored shall be discarded.

Preparation of the surface of the test portion by abrading shall be kept to a minimum, consistent with the need to remove oxidation products and surface imperfections. The test portion shall be analysed immediately after preparation.

10.7.2 Methods of sampling

According to the geometric form of a piece or product, use suitable machining tools to prepare the laboratory sample by turning, milling, sawing, slicing or trepanning, etc.

From a cast product or forging, obtain a test sample of a suitable size in the centred part.

Obtain a laboratory sample from a long wrought product at a position midway between the product centreline and the edge and at a distance from the end of at least half the cross-section using a saw or an abrasive cutting disc. In order to obtain a test sample, cut a piece from the laboratory sample of a size suitable for turning using a lathe.

Store the test sample in a refrigerant.

10.7.3 Preparation of a test portion

Cut a test portion from the test sample of a suitable mass in such a manner as to minimize heating the sample. Cool the sample at frequent intervals.

Prepare the surface of the test portion by filing, grit-blasting or light grinding. If filing is used, abrade the surface by hand with a fine-cut file. If grit-blasting is used, a grit-blasting machine shall be reserved exclusively for this purpose to avoid contamination of the test portion from grit. If grinding is used, cool the test portion at frequent intervals.

Degrease the test portion by immersion in acetone, dry by exposure for a few seconds to a rough vacuum and analyse immediately. Alternatively, the test portion may be degreased by immersion in 2-propanol (isopropyl alcohol) and then dried with diethyl ether.

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Annex A (informative)

Sampling probes for use with liquid iron and steel

A.1 General

Disposable probes for taking samples from liquid iron and steel consist of a small mould made from pressed steel, ceramic material or silica tubing, and which are mounted in a thick-walled protective cardboard tube.

Several types of sampling probe are available commercially. The main features are described in [Clauses A.2 to A.4](#) with examples shown in [Figures A.1 to A.7](#).

A.2 Immersion sampling probes

A.2.1 Immersion probes may be inserted into the melt either by hand or by mechanical means using a lance of steel piping fitted into the cardboard tube or attached directly to the probe assembly. Immersion time may vary depending on the design of the probe and the conditions of sampling, in particular the temperature of the melt, but normally lie between 3 s and 8 s.

The lance is constructed so that air from the mould cavity and gases produced during the combustion of cardboard can easily escape. A handlebar is incorporated for controlling the lance during immersion and withdrawal.

When sampling from ladles and secondary treatment vessels, a mechanical system may be used to lower and raise the lance.

Some types of probe combine a thermocouple in a silica tube adjacent to the sample chamber for the measurement of temperature. Where a measurement substance is used in a basic oxygen converter furnace, a mould assembly may be incorporated with the measurement sensors in the substance to obtain a test sample.

There are some probes made from a thermocouple and a zirconium oxide cell in order to online control the oxygen in the melt (for example see [Figure A.7](#)).

A.2.2 Probes in which the sample chamber is filled by ferrostatic pressure consist of a steel split-mould held in a cardboard tube by a collar of refractory material. The mould has a bottom-entry silica tube with a protective cap of thin steel to prevent ingress of slag and any other contaminant. Cardboard tubes, which vary in length from 200 mm to 1 500 mm or longer, may be partially coated with refractory material to minimize splashing during immersion.

This type of probe is mainly used to obtain samples from liquid steel in furnaces and ladles; two different designs are shown in [Figure A.1](#).

A.2.3 The samples obtained by using immersion probes filled by ferrostatic pressure may differ in configuration. Three main types of probe samples are as follows:

- a) Disc-and-pin probe samples shown in [Figure A.2 a](#)); the disc is suitable for analysis by a physical method and the pin may be used, if required, for analysis by a thermal method. The disc may be oval, circular or some other similar shape.

- b) Disc-and-pin probe samples with a number of lugs attached to the disc; the lugs have a mass of 0,5 g or 1 g and may be easily broken off from the disc and may be used, if required, as test portions for thermal methods of analysis.
- c) Dual-thickness probe samples shown in [Figure A.2](#) b), in which a part of the disc is reduced in thickness and is suitable for the punching out of slugs. The slugs are 4 mm to 6 mm in diameter and are used as test portions in thermal methods of analysis. The thicker section of the disc of a dual-thickness probe sample is used for analysis by physical methods. A probe sample with a Rockwell hardness greater than approximately 25 HRC may require heat treatment under an adequate atmosphere or environment (to ensure that the chemical composition is not changed) such as annealing before punching, provided that the product has been submitted to the same heat treatment.

A.2.4 Probes of the type shown in [Figure A.3](#), in which the sample chamber is filled by gravity, consist of a two-piece or four-piece steel cylindrical mould, which is contained in a refractory assembly and attached to a cardboard tube.

The mould has a side entrance port that is protected in some way to prevent ingress of slag. The assembly may be sleeved with refractory to minimize splashing during immersion. The overall length of the assembly and cardboard tube is 400 mm to 800 mm. Immersion times are usually 2 s or 3 s.

This type of probe is used in situations where the disc-and-pin type of sample is not satisfactory for analysis. It is used to obtain samples from liquid iron and steel in ladles, and from liquid steel in ingot moulds and continuous casting tundishes.

NOTE Typically, the sample is 30 mm in diameter and 70 mm long.

A.2.5 Probes of a special design are used to obtain samples from liquid iron in the blast-furnace runner and from the torpedo car and transfer ladle. These probes incorporate chill-plates of varying thickness to ensure that the liquid iron of the sample is chilled very rapidly. Two different types are described below:

- a) A variant of the probe filled by ferrostatic pressure (see [A.2.2](#)) with a design based on the use of either a thick-walled, steel split-mould or steel chill-plates, to provide a disc-and-pin sample with a number of lugs attached to the disc.

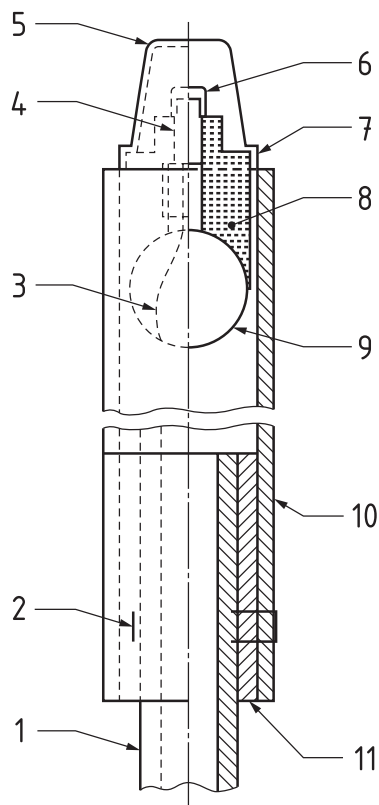
This type of probe is shown in [Figure A.4](#). The disc of the sample may vary in thickness from 5 mm to 6 mm. Immersion time may vary from 5 s to 9 s depending upon the application.

NOTE 1 Typically, the pin is 4 mm in diameter.

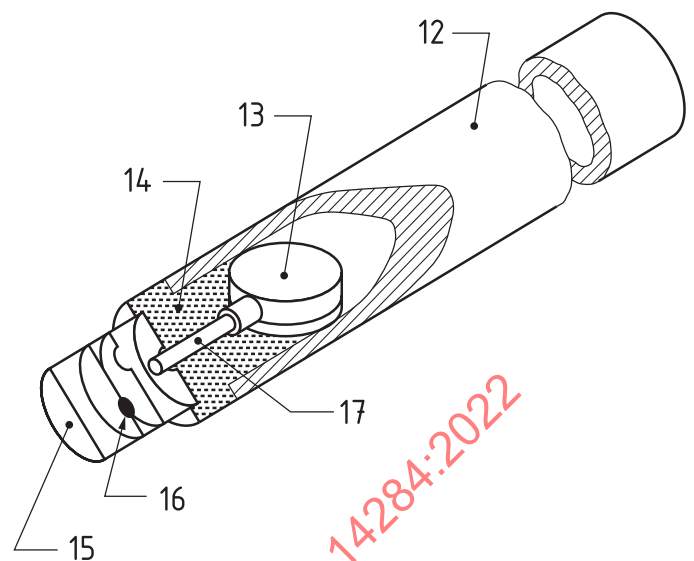
- b) A probe is designed to avoid the risk of drainage of liquid iron when sampling very fluid melts. The side-entry mould has one or more steel chill-plates and is held in a body of sand attached to a cardboard tube.

This type of probe may combine a separate or linked pin-shaped mould. The optional pin sample is 6 mm in diameter and 45 mm long.

NOTE 2 Typically, the sample is 35 mm in diameter with a thickness varying from 4 mm to 12 mm depending on the metallurgical microstructure required.



a) Deoxidant in the sample chamber



b) Deoxidant in a separate mixing chamber

Key

- 1 protection tube
- 2 staple
- 3 deoxidant
- 4 quartz tube
- 5 cap
- 6 chamber cap
- 7 adhesive
- 8 sand
- 9 sample chamber

- 10 outer sleeve
- 11 sleeve
- 12 cardboard
- 13 split mould
- 14 sand holder
- 15 slag cap
- 16 deoxidant (in mixing chambers)
- 17 silica tube

Figure A.1 — Examples of immersion sampling probes filled by ferrostatic pressure

