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

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

ISO 14284:1996

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Foreword

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

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 14284 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

It cancels and replaces ISO 377-2:1989, of which it constitutes a technical revision.

Annexes A and B of this International Standard are for information only.

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

1 Scope

This International Standard specifies methods for sampling and sample preparation for the determination of the chemical composition of pig iron, cast iron and steel. Methods are specified for use with both liquid and solid metal.

2 Normative references

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

ISO 377:—¹⁾, *Steel and steel products — Location of samples and test pieces for mechanical testing*.

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

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 chemical method of analysis: Method for the determination of chemical composition in which the sample is submitted to chemical reaction.

3.2 physical method of analysis: Method for the determination of chemical composition in which the determination of composition is carried out without submitting the sample to chemical reaction, for example an optical emission spectrometric method, an X-ray fluorescence spectrometric method.

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

3.4 melt: Liquid metal from which a sample is removed.

3.5 spoon sampling: Method in which the sample is taken from the melt, or during the pouring of the melt, using a long-handled spoon, and cast into a small mould.

3.6 spoon sample: Sample taken from the melt using a spoon and cast into a small mould.

3.7 probe sampling: Method in which the sample is taken from the melt using a commercially available sampling probe inserted into the melt.

3.8 immersion sampling: Method of probe sampling in which the probe is immersed in the melt where the sample chamber in the probe fills by ferrostatic pressure or gravity.

3.9 suction sampling: Method of probe sampling in which the probe is immersed in the melt where the sample chamber in the probe fills by aspiration.

1) To be published. (Revision of ISO 377-1:1989)

3.10 stream sampling: Method of probe sampling in which the probe is inserted into a stream of liquid metal where the sample chamber in the probe fills by the force of metal flow.

3.11 probe sample: Sample taken from the melt using a commercially available sampling probe.

3.12 cast product: Item of iron or steel which has not been subject to deformation, for example, an ingot, a semi-finished product obtained by continuous casting, a shaped casting.

3.13 wrought product: Item of steel which has been subject to deformation by rolling, drawing, forging or some other method, for example, a bar, a billet, a plate, a strip, a tube, a wire.

3.14 sample product: Specific item of iron or steel selected from a supplied quantity for the purpose of obtaining a sample.

3.15 preliminary sample: Sufficient quantity of metal selected from the sample product for the purpose of obtaining one or more samples for analysis.

3.16 sample for analysis: Part of the sample product, or part of the preliminary sample taken from the sample product, or part of the sample taken from the melt, brought to a required condition for submission to analysis.

The sample for analysis may comprise the sample product itself or the sample taken from the melt.

NOTE 1 The following categories of sample for analysis are distinguished:

- sample in the form of a solid mass;
- sample that has been remelted;
- sample in the form of chips obtained by machining;
- sample in the form of fragments obtained by comminution;
- sample in the form of powder obtained by comminution.

3.17 test portion: Part of the sample for analysis, or part of the sample taken from the melt, actually submitted to analysis. In certain cases, the test portion may be selected from the sample product itself.

NOTES

2 The following special types of test portions in the form of a solid mass obtained from a probe sample are distinguished:

- test portion in the shape of a small disc, commonly described as a slug, obtained by punching;
- test portion in the form of a small appendage, commonly described as a lug;
- test portion in the form of a small-diameter rod, commonly described as a pin, obtained by cutting.

3 When the sample for analysis is in the form of chips or powder, or when a sample in the form of a solid mass is analysed by a thermal method, the test portion is obtained by weighing. In the case of a physical method of analysis, the part actually analysed will constitute only a small mass of the sample for analysis. In an optical emission spectrometric method, the mass of metal consumed in an electrical discharge is about 0,5 mg to 1 mg; in an X-ray fluorescence spectrometric method, the characteristic radiation is produced from a very thin surface layer of the sample.

3.18 grinding: Method of preparing a sample of metal for a physical method of analysis in which the surface of the sample for analysis is abraded using an abrasive wheel.

3.19 finishing: Method of preparing a sample of metal for a physical method of analysis in which the surface of the sample for analysis is abraded using a flexible rotating disc or continuous belt coated with an abrasive substance.

3.20 milling: Method of preparing sample chips or the surface of a sample for a physical method of analysis in which the surface of the sample is machined using a rotating, multi-edged cutting tool.

3.21 consignment: Quantity of metal delivered at one time.

3.22 increment: Quantity of metal obtained by sampling at one time from a consignment.

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4 Requirements for sampling and sample preparation

4.1 General

This clause covers the general requirements for the sample, and for the sampling and sample preparation of iron and steel. Special requirements apply to each category of liquid and solid metal and these are considered in the relevant subclause.

The sequence of sampling and sample preparation of liquid iron and steel, and cast iron and steel products is shown in figure 1. Special considerations apply to pig irons (see clause 8).

4.2 Sample

4.2.1 Quality

Sampling practices shall be designed to provide a sample for analysis that is representative of the mean chemical composition of the melt or the sample product.

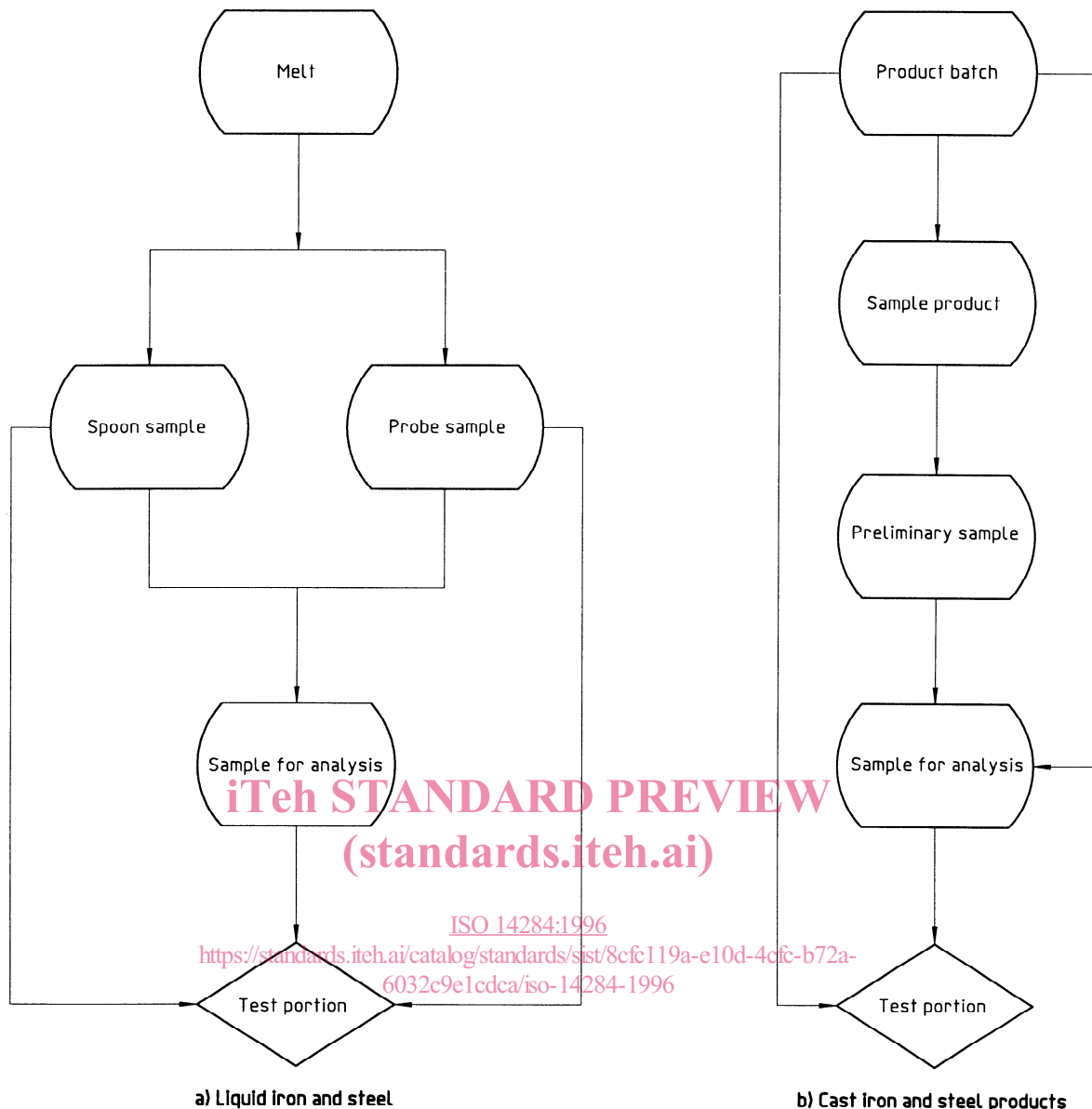


Figure 1 — Sequence of sampling and sample preparation

The sample for analysis shall be sufficiently homogeneous with respect to chemical composition such that inhomogeneity does not appreciably contribute to the error variability of the method of analysis. However, in the case of a sample taken from a melt, some variability in analysis, both within and between samples for analysis, may be unavoidable; this variability will form an inherent part of the repeatability and reproducibility of the analysis.

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

As far as possible, the sample for analysis should be free from voids, cracks and porosity, and from fins, laps or other surface defects.

Particular care shall be taken when selecting and preparing the sample for analysis, where a sample taken from a melt is expected to be heterogeneous or contaminated in any way.

A sample taken from a melt shall be cooled in such a manner that the chemical composition and metallurgical structure of the sample is consistent from sample to sample.

It is important to recognize that analysis by some physical methods may be influenced by the metallurgical structure of the sample, particularly in the case of irons with white and grey iron structures, and in the case of steels in the as-cast and wrought conditions.

4.2.2 Size

The dimensions of a preliminary sample in the form of a solid mass shall be sufficient to permit additional samples for analysis to be taken for re-analysis, where necessary using an alternative method of analysis.

A sample for analysis, of a sufficient mass to provide for any re-analysis necessary, shall be prepared. Generally, a mass of 100 g will be sufficient for a sample in the form of chips or powder.

The dimensional requirements for a sample for analysis in the form of a solid mass will depend upon the method selected for analysis. In the case of optical emission and X-ray fluorescence spectrometric methods, the shape and size of the sample will be determined by the dimensions of the sample chamber. The dimensions for samples for analysis given in this International Standard should be regarded as indicative only.

4.2.3 Identification

A sample for analysis shall be assigned a unique identification in order to determine the melt of sample product from which it was taken and, if necessary, the processing conditions of the melt or the location of the preliminary sample or the sample for analysis in the sample product.

A sample for analysis of pig iron shall be assigned a unique identification in order to determine 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 sample for analysis.

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 Conservation

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

It is permitted to conserve the preliminary sample in the form of a solid mass, and a sample for analysis may then be prepared when required.

The sample for analysis, or the preliminary sample in the form of a solid mass, shall be kept for a sufficient period of time to protect the integrity of the test laboratory.

4.2.5 Arbitration

In the case of a sample intended for arbitration, the sample for analysis shall be prepared jointly by the supplier and purchaser, or by their representatives. A record shall be kept of the methods used for preparing the sample for analysis.

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

4.3 Selection of a sample

4.3.1 Sample from a melt

Melts are sampled at various stages in the manufacturing process for the purposes of monitoring and controlling the process. Samples can 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 sample for analysis 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 for sample quality (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 or a combination of a disc with one or more attached pins; in some cases small lugs are attached to a disc sample.

NOTE 4 Sampling probes for use with liquid iron and steel may be obtained from a number of suppliers; the main features of the different types of probe are distinguished in annexes A and B, which include dimensions for information only.

4.3.2 Sample from a product

The preliminary sample or the sample for analysis can be selected from the sample product 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 sample for analysis can be selected from a bar or block cast onto the casting.

In the case of a forging, the sample for analysis can 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 sample for analysis may, following agreement between the supplier and the purchaser, be selected from the sample for mechanical testing or from the test piece, or directly from the sample product.

The preliminary sample or the sample for analysis can be obtained from the sample product by machining or by using a cutting torch. 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

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. Following this operation, the sample shall then be protected from any change in composition.

If necessary, the surface of the metal shall be laid completely bare at the point 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, taken care to ensure that the manner of degreasing does not affect the correctness of analysis.

4.4.2 Sample for analysis in the form of chips

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

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

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, can be minimized by selection of appropriate tools and cutting speeds.

Heat treatment may be required to soften the sample for machining.

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

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

4.4.3 Sample for analysis in the form of a powder or 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 sample for analysis in the form of a powder, 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 sample for analysis in the form of fragments with a particle size range of approximately 1 mm to 2 mm.

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

Comminution shall not be used for the preparation of samples of graphite-bearing 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 sample for analysis shall be homogenized before weighing the test portion. Powders can 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 Sample for analysis in the form of a solid mass

4.4.4.1 Selection of the sample for analysis

The sample for analysis shall be obtained by cutting, from the sample product or preliminary 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 sample for analysis

The sample for analysis 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 affected by the heat of a cutting torch. The equipment used for sample preparation shall be designed to minimize overheating the sample and, where appropriate, shall incorporate systems of cooling.

Four main types of equipment used for surface preparation may be distinguished.

- a) A milling machine capable of removing a pre-selected depth of metal in a reproducible manner, for use with samples that are within a hardness range suitable for milling. The equipment should 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 sample for analysis 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 sample for analysis or the test portion.

After preparation, the surface of the sample for analysis shall be flat and free from defects which affect the correctness of analysis.

Cutting and surface preparation can 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) in annex A], and for the punching of slugs forming test portions, may incorporate facilities for the sand-blasting of the sample and for heat treatment to soften the sample before punching.

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

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 is essential to ensure 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 sample for analysis by volatilizing any grinding contaminants. However, particular care is required 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 sample for analysis shall be examined visually after preparation to establish that the surface is free from particulate matter and that there are no defects; the sample shall be resurfaced or discarded if defects are present. The sample for analysis shall be dry and care shall be taken to protect the prepared surface from contamination.

4.4.5 Preparation of a sample for analysis by remelting

A sample in the form of small pieces or chips, or a part of the sample product itself, can be remelted in an atmosphere of argon using commercially available melting equipment. The sample is converted into a disc, 40 mm to 30 mm in diameter and 6 mm thick, which is suitable for analysis by a physical method. Some types of remelting equipment incorporate facilities for the centrifugal casting of the disc.

Partial losses of some elements can be experienced 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 analysis results. 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 % (*m/m*) zirconium, should be used during remelting. The method used for calibration of the analytical measurement shall take account of any change that does occur.

Not all ferrous metals can be remelted in this manner. This method shall not be used for sample preparation for the determination of an element which is subject 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 methods. Provisions shall include protective clothing, hand protection and face visors resistant to splashes, for use during the sampling of liquid metal. Provisions shall also include protective clothing, hand, eye and hearing protection, for use during the sampling and sample preparation of solid 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. Grinding operations used for surface preparation may be covered by national legislation.

4.5.3 Hazardous substances

Reference shall be made to appropriate national regulations 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, and commonly described as hot metal, 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 pig-iron.

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

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

5.2 Spoon sampling

5.2.1 Methods

For sampling from a melt, immerse a preheated steel 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 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.

It is essential that the liquid iron should be poured into a mould that is cold 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, can be obtained using a two-piece steel mould; the sample is typically 35 mm to 40 mm in diameter with a thickness varying from 6 mm to 12 mm. The mould is constructed in two pieces which 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, for example, from 38 mm to 32 mm, to facilitate removal of the sample from the mould. A coin sample is vertically or horizontally cast in the mould.

A coin sample with one or more attached pins can 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. (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 can be obtained using a cast iron or steel split-mould; the dimensions of the sample are typically 70 mm x 35 mm with a thickness of 4 mm. 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 may be preferred for use with liquid iron containing high percentages of carbon.

5.2.2 Maintenance of equipment

It is essential to keep sampling 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.

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5.3 Probe sampling

5.3.1 General

The different types of probes used for sampling blast furnace iron are described in annex A. Probes should be designed to provide a disc-shaped sample with a white iron structure which is sufficiently deep for the requirements of the physical method selected for analysis.

Probe sampling is influenced by such factors as the angle and depth of immersion of the sampler in the melt, and immersion times can vary depending on the temperature of the liquid iron. These factors should be determined for the particular ironmaking practice and thereafter strictly controlled to maintain the standard of quality of the sample for analysis.

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 sample for analysis

5.4.1 Preliminary preparation

Remove any surface oxidation from a sample taken from the melt, that may contaminate the sample for analysis during subsequent preparation.

5.4.2 Sample for analysis by a chemical method

Break the sample into small pieces and crush pieces using a percussion mortar or a vibratory grinding mill to obtain a sufficient mass of sample for analysis of particle size preferably less than about 150 µm.

Alternatively, obtain chips by drilling the sample at a low speed as described in 8.3.1.

5.4.3 Sample for analysis by 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. Analyse a representative number of test portions to obtain an average value.

Alternatively, crush the pin or lugs in a percussion mortar to obtain a sufficient mass of sample for analysis 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 Sample for analysis by a physical method

In the case of a disc-shaped sample, remove any lugs or pins, as necessary, and then grind the surface of the sample to expose a white iron structure 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 iron and the conditions of sampling; the thickness of the layer to be removed normally lies between 0,5 mm and 1 mm (see 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 surface of the sample by grinding and finishing. Grinding shall be carried out wet to avoid overheating the sample but the final surface preparation shall be dry finishing. Alternatively, cool the sample after grinding by immersion in water and then finish by dry finishing.

Particular care is required when preparing the surface of thin samples. A chuck shall be specially designed to hold the sample securely grinding and finishing 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 may be subject to inhomogeneity, and particular care is required 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 should ensure that the analysis is representative of the melt as a whole.