



Standard Practice for Sampling Steel and Iron for Determination of Chemical Composition¹

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1. Scope

1.1 This practice covers the sampling of all grades of steel, both cast and wrought, and all types (grades) of cast irons and blast furnace iron for chemical and spectrochemical determination of composition. This practice is similar to ISO 14284.

1.2 This practice is divided into the following sections.

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1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific statements, see 6.4.3.5, 9.4.4.3, and 12.5.1 as well as Section 5.

2. Referenced Documents

2.1 ASTM Standards:²

- A48/A48M Specification for Gray Iron Castings
- A751 Test Methods, Practices, and Terminology for Chemical Analysis of Steel Products
- E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
- E415 Test Method for Atomic Emission Vacuum Spectrometric Analysis of Carbon and Low-Alloy Steel
- E1010 Practice for Preparation of Disk Specimens of Steel

¹ This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.01 on Iron, Steel, and Ferroalloys.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

and Iron for Spectrochemical Analysis by Remelting

2.2 *ISO Documents:*

ISO 9147 Pig irons—Definition and classification³

ISO 14284 Steel and iron—Sampling and preparation of samples for the determination of chemical composition³

3. Terminology

3.1 *Definitions*—For definitions of terms in this practice, refer to Terminology E135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *cast product, n*—item of iron or steel which has not been subject to deformation, for example, an ingot, a semi finished product obtained by continuous casting, or a shaped casting.

3.2.2 *consignment, n*—quantity of metal delivered at one time.

3.2.3 *grinding, n*—method of preparing a sample of metal for a spectrochemical method of analysis in which the surface of the sample for analysis is abraded using an abrasive wheel.

3.2.4 *increment, n*—in sampling, a portion of material removed from a lot by a single operation.

3.2.5 *linishing, n*—method of preparing a sample of metal for a spectrochemical method of analysis in which the surface of the sample for analysis is abraded using a rotating disk or continuous belt coated with an abrasive substance.

3.2.6 *lot, n*—in sampling, a collection of material regarded as a unit (see Specification A48/A48M).

3.2.7 *melt, n*—liquid metal from which a sample is removed.

3.2.8 *sample, probe, n*—sample taken from the melt using a sampling probe.

3.2.9 *sample, product, n*—specific item of iron or steel selected from a supplied quantity for the purpose of obtaining a sample.

3.2.10 *sample, spoon, n*—sample taken from the melt using a spoon and cast into a small mold.

3.2.11 *sampling, immersion, n*—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.

3.2.12 *sampling, probe, n*—method in which the sample is taken using a sampling probe inserted into the melt.

3.2.13 *sampling, spoon, n*—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 mold.

3.2.14 *sampling, stream, n*—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 force of metal flow.

3.2.15 *sampling, suction, n*—method of probe sampling in which the probe is inserted into the melt where the sample chamber in the probe fills by aspiration.

3.2.16 *test portion, n*—part of the sample for analysis, or part of the sample taken from the melt, actually analyzed.

3.2.16.1 *Discussion*—In certain cases, the test portion may be selected from the sample product itself.

3.2.17 *thermal method of analysis, n*—method for the determination of chemical composition in which the sample is submitted to a process of heating, combustion, or fusion.

3.2.18 *wrought product, n*—item of steel which has been subject to deformation by rolling, drawing, forging, or some other method, for example, a bar, billet, plate, strip, tube, or wire.

4. Significance and Use

4.1 This practice covers all aspects of sampling and preparing steel and iron for chemical analysis as defined in Test Methods, Practices, and Definitions A751 and Specification A48/A48M. Such subjects as sampling location and the sampling of lots are defined.

4.2 It is intended that this practice include most requirements for sampling steel and iron for analysis. Standard analytical methods that reference this practice need contain only special modifications and exceptions.

4.3 It is assumed that all who use these procedures will be trained samplers capable of performing common sampling operations skillfully and safely. It is expected that only proper sampling equipment will be used.

5. Hazards and Safety Precautions

5.1 Provide personal protective equipment 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 of liquid metal for use during the sampling of liquid metal; protective clothing and hand, eye, and hearing protection for use during the sampling and sample preparation of solid metal; and respiratory protection for use where necessary.

5.2 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.

5.3 Reference should be made to appropriate national regulations with respect to the use of solvents for the cleaning and drying of samples and test portions.

6. Requirements for Sampling and Sample Preparation

6.1 *General:*

6.1.1 This section describes 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 requirements are described in the relevant section.

6.1.2 The sequence of sampling and sample preparation of liquid iron and steel and cast iron and steel products is shown in Fig. 1. Special considerations apply to pig irons (see Section 10).

6.2 *Sample:*

6.2.1 *Quality:*

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

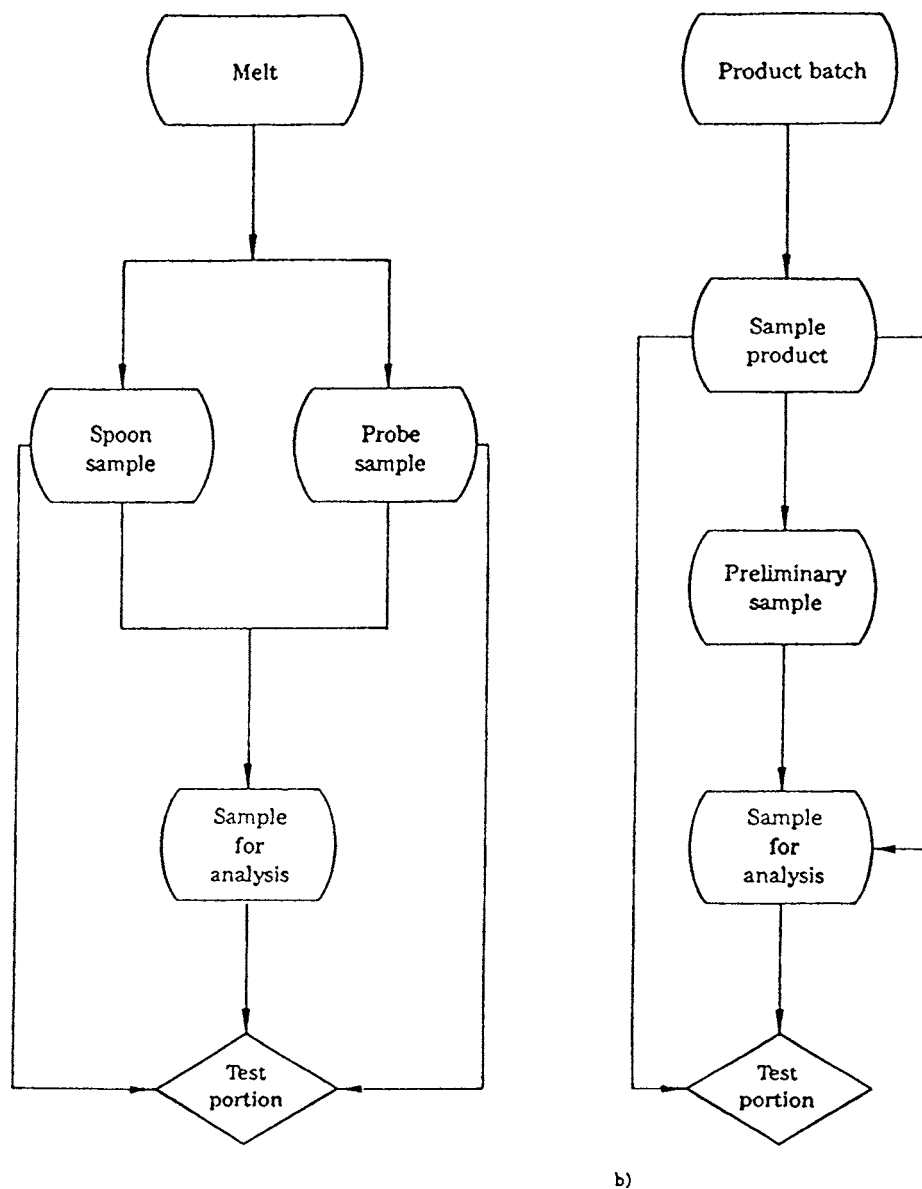


FIG. 1 Sequence of Sampling and Sample Preparation of (a) Liquid Iron and Steel and (b) Cast Iron and Steel Products

6.2.1.1 Sampling practices shall be designed to provide an analytical sample that is representative of the mean chemical composition of the melt or of the sample product.

6.2.1.2 Ensure that the sample is sufficiently homogeneous with respect to chemical composition so that inhomogeneity does not appreciably contribute to the error variability of the method of analysis. In the case of a sample taken from a melt, however, 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.

6.2.1.3 Ensure that the sample is free from surface coatings and from moisture, dirt, or other forms of contamination.

6.2.1.4 The sample should be free from voids, cracks, and porosity and from fins, laps, or other surface defects.

6.2.1.5 Take particular care when selecting and preparing the sample where a sample taken from a melt is expected to be heterogeneous or contaminated in any way.

6.2.1.6 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.

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

6.2.2 Size:

6.2.2.1 Ensure that the dimensions of a laboratory sample in the form of a solid mass are sufficient to permit additional samples for analysis to be taken for reanalysis when necessary using an alternative method of analysis.

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

6.2.2.3 The dimensional requirements for a sample in the form of a solid mass will depend upon the method selected for analysis. In the case of atomic 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 given in this practice are for guidance only. Refer to Test Method E415 for sample size requirements for atomic emission spectrometry.

6.2.3 Identification:

6.2.3.1 Assign the sample a unique identification to determine the melt or sample product from which it was taken, and if necessary, the process conditions of the melt or the location of the laboratory sample in the sample product. For pig iron, this shall identify the consignment or part of a consignment and the increment from which it was taken.

6.2.3.2 Use labeling or some equivalent method of marking to ensure that the assigned identification remains associated with the sample for analysis.

6.2.3.3 Record the identification of the sample to ensure that confusion cannot arise as to the identity of the item to which the analysis and records refer.

6.2.4 Conservation:

6.2.4.1 Provide adequate storage facilities to segregate and protect the sample. During and after preparation, store the sample in a manner which prevents contamination or chemical change.

6.2.4.2 Keep the sample, or the laboratory sample in the form of a solid mass, for a sufficient period of time to permit retesting if necessary.

6.2.5 Arbitration:

6.2.5.1 Samples intended for arbitration shall be prepared jointly by the supplier and purchaser or by their representatives. Keep a record of the methods used for preparing samples.

6.2.5.2 Both parties or their representatives seal containers with the samples intended for arbitration. Unless agreed to the contrary, the representatives of each party responsible for the preparation of samples will keep these containers.

6.3 Selection of a Sample:

6.3.1 Sample from a Melt:

6.3.1.1 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 analytical 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.

6.3.1.2 Sampling practices shall be designed for melts to provide samples during a particular manufacturing process in accordance with requirements for sample quality (see 6.2.1). The sample obtained from a melt usually is in the form of a small ingot, a cylindrical or rectangular block, or a chill-cast disk or a combination of a disk with one or more attached pins, in some cases small lugs are attached to a disk sample.

NOTE 1—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 Annex A1 and Annex A2, which include dimensions for information only.

6.3.2 Sample from a Product:

6.3.2.1 The laboratory sample 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.

6.3.2.2 In the case of an iron casting, the sample can be selected from a bar or block cast-on to the casting.

6.3.2.3 In the case of a forging, the sample 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.

6.3.2.4 In the absence of requirements given in the product standard or of a specification when ordering the product, the sample 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.

6.3.2.5 The laboratory sample 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.

6.4 Preparation of a Sample:

6.4.1 Preliminary Preparation:

6.4.1.1 If any part of the sample is liable to be nonrepresentative in chemical composition, remove those parts that have changed. Following this operation, the sample shall be protected from any change in composition.

6.4.1.2 Remove any coating that has been applied during manufacturing. If necessary, degrease the surface of the metal with a suitable solvent, taking care to ensure that the manner of degreasing does not affect the correctness of analysis.

6.4.2 Sample in the Form of Chips:

6.4.2.1 Obtain the sample by drilling or milling or turning to produce chips of a regular size and shape. Do not machine on a part of the sample that has been affected by the heat of a cutting torch.

6.4.2.2 Clean the tools, machines, and containers used during preparation of the sample beforehand to prevent any contamination of the sample for analysis.

6.4.2.3 Machine in such a way that the chips are not subject to overheating as indicated by a change in the color (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.

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

6.4.2.5 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.

6.4.2.6 Mix the chips thoroughly before weighing the test portion. For most purposes, it is satisfactory to mix the chips by rolling the container on a level surface, gently tumbling the container.

6.4.3 Sample in the Form of a Powder or Fragments:

6.4.3.1 Where drilling of the sample to obtain chips is impracticable, cut or break the sample into pieces. Crush the pieces using a percussion mortar or a vibratory grinding mill, also known as a disk mill or ring mill, to obtain a sample in the form of a powder the whole of which passes through a sieve of a specified aperture size.

6.4.3.2 In some applications the sample is crushed in a percussion mortar to obtain a sample for analysis in the form of fragments suitable for use with the selected method of analysis.

6.4.3.3 Equipment used for pulverization shall be constructed from material that does not alter the sample composition. Tests may be necessary to show that such equipment does not affect the composition of the sample.

6.4.3.4 Sieve the sample taking precautions to avoid contamination or loss of material. Take care when sieving hard materials to avoid damaging the fabric of the sieve.

6.4.3.5 Homogenize the sample before weighing the test portion. Powders can be homogenized by stirring. (**Warning**—Finely divided metals of particle size less than approximately 150 μm can present a fire risk. Ensure adequate ventilation during pulverization.)

6.4.4 *Sample in the Form of a Solid Mass:*

6.4.4.1 Obtain the sample by cutting from the sample product or laboratory sample a piece of suitable size and shape for the method of analysis. Cut samples by sawing, abrasive cutting, shearing, or punching.

6.4.4.2 In the absence of any indication in the product standard, perform a spectrochemical analysis on that part of the sample corresponding to a transverse section of the product, provided that the material has sufficient thickness.

6.4.4.3 Prepare the sample to expose a surface suitable for the analytical method. Do not prepare a surface for analysis on 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.

6.4.5 *Main Types of Equipment Used for Surface Preparation:*

6.4.5.1 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 should be capable of use, if required, with a sample taken from a melt where the sample is still hot.

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

6.4.5.3 A flatbed finishing machine with abrasive grinding disks, or machines with continuous abrasive belts, capable of preparing the surface of the sample for analysis to varying grades of finish.

6.4.5.4 A machine for blasting with sand or grit, or metal shot, capable of cleaning the surface of the sample for analysis or the test portion when required.

6.4.5.5 Ensure that the surface of the sample after preparation is flat and free from defects that affect correctness of analysis.

6.4.5.6 Cutting and surface preparation can be performed either manually or automatically. In the case of samples taken

from melts, commercially available systems may be used that perform each stage of preparation automatically. Systems for the automatic surface preparation of dualthickness probe samples (see A2.3) and for the punching of slugs as test portions may incorporate facilities for the sandblasting of the sample and for heat treatment to soften the sample before punching.

6.4.5.7 Select the abrasive substances used in the final stage of preparing the sample that do not contaminate the surface with elements that are to be determined by the analysis method. Ensure that the grit size of the abrasive is in accordance with the grade of surface finish required for the analysis method.

6.4.5.8 In the case of atomic emission spectrometric methods, an abrasive with a grade of 60 grit to 120 grit normally is 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 sufficiently smooth and reproducible from sample to sample. Also, there should be no smearing of the surface.

6.4.5.9 The effect of abrasive materials depends on the analytical method. When using atomic emission spectrometric methods, the action of pre-sparking normally will clean the surface of the sample for analysis by volatilizing any grinding contaminants. Particular care is required, however, to avoid surface contamination when using a new abrasive disk.

6.4.5.10 When using X-ray fluorescence spectrometric methods, examine all phases of surface preparation for potential surface contamination effects.

6.4.5.11 Examine the sample visually after preparation to establish that the surface is dry and free from particulate matter and that there are no defects. Resurface or discard the sample if defects are present. Protect the prepared surface from contamination.

6.4.6 *Preparation of a Sample for Analysis by Remelting:*

6.4.6.1 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 of the type described in Practice E1010. The sample is converted into a disk, 40-mm to 30-mm diameter by 6 mm thick, which is suitable for analysis by a spectrochemical method. Some types of remelting equipment incorporate facilities for the centrifugal casting of the disk sample.

6.4.6.2 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 occur are known quantitatively and do not significantly influence the analysis results. Carry out suitable tests to show that any change in composition is both small in magnitude and reproducible.

6.4.6.3 The equipment used and the method adopted for remelting shall be designed to prevent or minimize 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. Take into account when using the method for calibration of analytical measurement any change that does occur.

6.4.6.4 Not all ferrous metals can be remelted in this manner. Do not use remelting as a method of sample preparation for the determination of an element that is subject to a significant and nonreproducible change in composition.

7. Liquid Iron for Steelmaking and Pig Iron Production

7.1 General:

7.1.1 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 during the pouring of the melt 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.

7.1.2 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.

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

7.2 Spoon Sampling:

7.2.1 Hold Types:

7.2.1.1 A disk-shaped sample, commonly described as a coin sample, can be obtained using a two-piece steel mold. The dimensions of the sample are typically 35 mm to 40 mm in diameter with thickness varying from 6 mm to 12 mm. The mold is constructed in two pieces that are clamped together in use: one piece is a flat chill plate and the other is a block with the mold cavity. The edge of the mold cavity may be tapered, for example, from 38 mm to 32 mm, to facilitate removal of the sample from the mold.

7.2.1.2 A coin sample with one or more attached pins can be obtained using a combination-type mold. the pins are broken off from the disk and used, if required, as test portions for analysis by a thermal method. A combination-type mold for use with liquid iron intended for the production of cast iron is shown in Fig. 2.

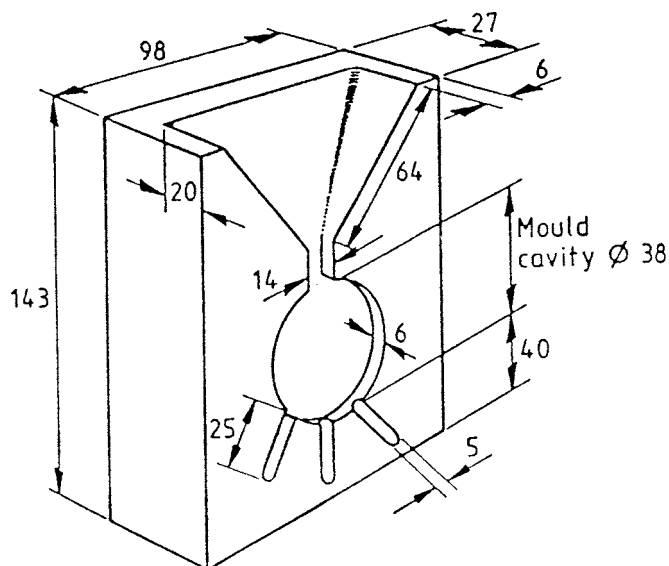
7.2.1.3 A thin slab-shaped sample with a rounded end can be obtained using a cast iron or steel split-mold. The dimensions of the sample are typically 70 mm by 35 mm with a thickness of 4 mm. The two halves of the mold are bevelled at the top to give a feeder head and are clamped together in use. This type of mold may be preferred for use with liquid iron containing high percentages of carbon.

7.2.2 Procedures:

7.2.2.1 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.

7.2.2.2 For sampling from a stream, introduce a pre-heated steel spoon into the stream from the ladle and fill with liquid iron.

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



NOTE 1—All dimensions are in millimetres.

NOTE 2—The flat chill plate (not shown) has similar overall dimensions.

FIG. 2 Combination-Type Vertical Mold Used for Sampling Liquid Iron Intended for Cast Iron Production

7.2.2.4 It is essential that the liquid iron should be poured into a mold that is cold to ensure adequate chilling. If necessary, the mold should be air-cooled before use. The mold should be free from moisture.

7.2.3 Maintenance of Equipment:

7.2.3.1 It is essential to maintain sampling spoons and molds in a clean and dry condition. After use, remove any slag and skull and brush the faces of the mold with a wire brush.

7.2.3.2 Molds should be re-machined 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:

7.3.1.1 The different types of probes used for sampling blast furnace iron are described in Annex A1. Probes are designed to provide a disk-shaped sample with a depth of white iron structure that is sufficient for the requirements of the spectrochemical method selected for analysis.

7.3.1.2 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 iron-making practice, and thereafter, strictly controlled to maintain the standard of quality of the sample for analysis.

7.3.2 Procedures:

7.3.2.1 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.

7.3.2.2 When sampling from the runner of a blast furnace, select the position of immersion to give 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.

7.3.2.3 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.

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

7.4 *Preparation of a Sample for Analysis:*

7.4.1 *Preliminary Preparation*—Remove any surface oxidation from a sample taken from the melt that may contaminate the sample during subsequent preparation.

7.4.2 *Sample for Analysis by a Chemical Method:*

7.4.2.1 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 less than 150 µm.

7.4.2.2 Alternatively, obtain chips by drilling the sample at a low speed as described in 10.3.1.2.

7.4.3 *Sample for Analysis by a Thermal Method:*

7.4.3.1 Break the pin of a disk-shaped sample into pieces of a suitable mass for use as test portions or use the lugs of a probe sample. Analyze a representative number of test portions to obtain an average value.

7.4.3.2 Alternatively, crush the pin or lugs using 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.

7.4.4 *Sample for Analysis by a Spectrochemical Method:*

7.4.4.1 In the case of a disk-shaped sample, remove any lugs or pin, as necessary, and grind the surface of the sample to expose a white iron structure that is representative of the sample. The amount of material to be removed in this way should 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.

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

7.4.4.3 Prepare the surface of the sample by grinding and finishing. Grinding should be carried out wet to avoid overheating the sample but the final surface preparation should be dry finishing. Alternatively, cool the sample after grinding by immersion in water, and then finish by dry finishing.

7.4.4.4 Particular care is required when preparing the surface of thin samples. A chuck should be specially designed to hold the sample securely during grinding and finishing operations.

8. Liquid Iron for Cast Iron Production

8.1 *General:*

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

8.1.2 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 analysis is representative of the melt as a whole.

8.1.3 In batch processes, two or more samples should 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 should be taken at regular intervals of time.

8.1.4 Methods of sampling normally are designed to chill the liquid metal of a sample cast from a spoon, as rapidly as possible, to produce a metallurgical structure of white iron free from graphite. A white iron structure obtained by chill-casting is required generally for analysis by spectrochemical methods.

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

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

8.1.7 Special considerations apply to the sampling and sample preparation of liquid iron for the determination of hydrogen, oxygen, and nitrogen (see 8.5).

8.2 *Spoon Sampling:*

8.2.1 *General:*

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

NOTE 2—Where sampling takes place before addition of inoculating agents, it should be recognized that the sample obtained will not be representative of the chemical composition of the cast product.

8.2.1.2 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 representivity of sampling.

8.2.1.3 Ductile iron particularly is 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 iron using a ceramic disk.

8.2.2 *Methods*—A graphite spoon or a steel spoon lined with a layer of a refractory, such as ganister, is suitable for use in accordance with one of the following methods.

8.2.2.1 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.

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

8.2.3 *Chilled Sample:*

8.2.3.1 Pour the liquid iron from the spoon without delay into a split mold made from graphite, hematite iron, or copper to obtain a sample in the form of a small, flat plate, 4 mm to 8 mm in thickness. Remove the sample from the mold, as soon as it is solid, to avoid overheating the mold and the risk of breakage of the sample. Break off any riser.

8.2.3.2 The sample, commonly described as a coin sample, may be circular, rectangular, or square in shape with typical sizes, respectively, 35-mm to 40-mm diameter, (50 by 27) mm and (50 by 50) mm. Generally, disk samples are cast vertically and rectangular and square samples are cast horizontally.

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

8.2.3.4 A coin sample with one or more attached pins can be obtained using a combination-type mold. The pins are broken off from the disk and used, if required, as test portions for analysis by a thermal method. A vertical mold of this type, commonly described as a book mold and made from low-phosphorus, high-carbon grey iron, or from copper or graphite, is shown in Fig. 2. The sample obtained is a disk, 38 mm in diameter by 6 mm thick, with three 5-mm diameter pins.

8.2.3.5 The temperature of the liquid iron in the spoon should be as high as possible compatible with the mold material. It is essential that the mold is cold to ensure adequate chilling for the production of a sample for analysis with a white iron structure. If necessary, the mold should be air-cooled before use. The mold should be free from moisture.

8.2.3.6 In the case of a process where samples are required to be taken at frequent intervals, provide several molds to ensure the availability for use of a mold that is cold.

8.2.3.7 Thermal stress due to overheating of the mold can cause breakage of the coin sample and should be avoided.

8.2.4 Non-Chilled Sample:

8.2.4.1 Pour the liquid iron from the spoon without delay into a sand mold to obtain a cylindrical block-shaped sample approximately 50 mm in diameter by 40 mm to 50 mm long.

8.2.4.2 Alternatively, a sample can 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 pouring spoon, or, if a small hand-ladle is used for pouring, directly from the ladle itself. Bars are typically 30 mm in diameter by 150 mm long and may be cast vertically or horizontally in a sand mold.

8.2.4.3 Allow samples to cool completely before removal from the mold.

8.2.5 Maintenance of Equipment:

8.2.5.1 It is essential to maintain pouring spoons and molds in a clean and dry condition. After use, remove any slag and skull and brush the faces of the mold.

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

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

8.4 Preparation of a Sample for Analysis:

8.4.1 Preliminary Preparation:

8.4.1.1 Remove any sand adhering to the surface of a sand-cast sample by scratch brushing or shot-blasting. Remove any surface oxidation by grinding.

8.4.1.2 Prepare the sample in accordance with one of the following procedures depending upon the method selected for analysis.

8.4.2 Sample for Analysis by a Chemical Method:

8.4.2.1 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 avoiding the production of fine particles. Avoid overheating the sample and the tool. Chips should be as solid and compact as possible with a mass of approximately 10 mg in order to prevent crumbling and loss of graphite. Chips should not be washed 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 use when obtaining chips by drilling.

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

8.4.2.3 When machining is not practicable, the sample can be broken into pieces and pieces crushed using a percussion mortar or vibratory grinding mill to obtain a sufficient mass of sample of particle size less than 150 μm . Use this method in cases where it can be shown that pulverization does not lead to contamination of the sample.

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

8.4.2.5 For a non-chilled sample, 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 center of the block to obtain the sample.

8.4.2.6 In the case of a test bar, either 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 or 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 center, or face turn a cross section of the bar, but 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 disk 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 sample for analysis of particle size less than 150 μm .

8.4.3 Sample in the Form of a Solid Mass for Analysis by a Thermal Method:

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

8.4.3.2 Alternatively, crush the pin in a percussion mortar to provide a sample for analysis of particle size range from approximately 1 mm to 2 mm. Avoid the production of fine material.

8.4.3.3 For a non-chilled sample, use a saw to cut a 3-mm disk or slice from the cross section of a cylindrical block or test bar and cut pieces of a suitable mass for use as test portions.

8.4.3.4 Analyze a representative number of test portions to obtain an average value. The mass of a piece selected as a test portion should not be less than approximately 0.3 g.

8.4.4 *Sample for Analysis by a Spectrochemical Method:*

8.4.4.1 For a chilled sample, remove any pins and then use a fixed-head grinder to expose a white iron structure which is representative of the sample. The amount of material to be removed in this way should be determined for the chemical composition of the particular iron and the conditions of sampling. The thickness of the layer to be removed is normally at least 1 mm.

8.4.4.2 Air-cooling is recommended during grinding. Grinding can be carried out wet to avoid overheating the sample, but the final treatment should be dry grinding or finishing. Excessive grinding may lead to analysis errors if the chill region of the sample is exceeded. Chilled samples should be examined regularly in routine practice to ensure the suitability of the metallurgical structure of the prepared sample for the analysis method.

8.4.4.3 For a non-chilled sample, use a grinding or finishing machine to remove a layer approximately 1 mm in thickness from the surface of the sample. Air cooling is recommended during grinding and, liquid coolants should not be used.

8.4.4.4 For irons that are subject to segregation effects, for example, high-phosphorus engineering iron, high-silicon ductile iron, malleable iron, prepare the surfaces of two sides of the sample for analysis to obtain an average value.

8.4.4.5 Avoid overheating of the sample during surface preparation as this can result in surface crazing that will affect correctness of analysis.

8.4.4.6 Care is required when preparing the surface of a thin coin-sample. A chuck should be designed specially to hold the sample securely during grinding operations.

NOTE 3—A fixed-head grinder is preferred to a swing grinder for surface preparation; the latter type of equipment may not give a flat surface to the sample for analysis.

8.5 *Sampling and Sample Preparation for the Determination of Oxygen, Nitrogen, and Hydrogen:*

8.5.1 *General*—The determination of oxygen, nitrogen, and hydrogen is required only to a limited extent in the production of castings. Sampling and sample preparation methods should minimize hydrogen losses and avoid contamination of the sample by oxygen, nitrogen, or hydrogen.

8.5.2 *Procedure:*

8.5.2.1 It is essential that a sample for the determination of hydrogen should be cooled very rapidly. It should be removed from the mold immediately after solidification and quenched without delay. A mixture of acetone and solid carbon dioxide in the form of a slurry is suitable for quenching. The sample should be stored by immersion in a refrigerant.

8.5.2.2 For the determination of oxygen and nitrogen, pins broken from a chill-cast sample normally are suitable. Such samples may be obtained from the melt using a spoon, then casting the liquid iron as described in 6.2 into a book combination-type mold to obtain pin-shaped samples of 6 mm to 8 mm in diameter. For this purpose, the construction of the mold shown in Fig. 2 should be modified by enlarging the three pin-shaped cavities to produce pins of the required diameter.

8.5.3 *Preparation of the Test Portion:*

8.5.3.1 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 to obtain a test portion of a suitable mass for analysis. Avoid overheating the pin during the preparation of test portions for the determination of hydrogen, and cool at frequent intervals using crushed, solid carbon dioxide.

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

9. Liquid Steel for Steel Production

9.1 *General:*

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

9.1.2 Special considerations apply to sampling and sample preparation of liquid steel for the determination of oxygen and hydrogen. See 9.5 and 9.6, respectively.

9.2 *Probe Sampling:*

9.2.1 *General:*

9.2.1.1 The main features of the different types of commercially available sampling probes for use with liquid steel are distinguished in Annex A1.

9.2.1.2 Probe sampling is influenced by such factors as the angle and depth of immersion of the sampler and the time of immersion in the melt. It is essential that these factors be determined for the particular conditions of composition and temperature of the steel involved, and thereafter, strictly controlled to maintain the standard of quality required for the analysis.

9.2.1.3 Take precautions 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 concentrations. The selection of materials used for construction of the sampling probe, the design of the capping and entrance system, and the method of deoxidation should be such as to minimize risk of contamination, other than from the deoxidant itself.

9.2.2 *Procedures:*

9.2.2.1 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 center of the melt, at an angle as near as possible to 90°.

9.2.2.2 For sampling from shallow melts, such as in tundishes, and from ingot mold tops and continuous casting molds, 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 approximately 2 s to fill the mold.

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

9.2.2.4 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. Care should be exercised when inserting the probe

sampler into the stream. It may be necessary to reduce the flow of metal during sampling.

9.2.2.5 Withdraw the probe sampler from the melt after a predetermined interval of time and break it apart. Allow the probe sample to cool somewhat in air, to a dull red color, 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.

9.3 Spoon Sampling:

9.3.1 Procedures:

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

9.3.1.2 For sampling from the stream, introduce the spoon into the stream from the ladle; fill with liquid steel; then withdraw the spoon.

9.3.1.3 Exercise care when introducing the spoon into the stream because of the force of the liquid metal emerging from the nozzle. It may be necessary to reduce the rate of metal flow during the sampling operation.

9.3.1.4 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 mold designed to produce a tapered cylindrical sample. The dimensions of 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.

NOTE 4—Aluminum wire is frequently used as the deoxidant in spoon sampling provided that aluminum does not cause interference in the analysis method and that the determination of the aluminum content of the melt is not required. The amount of aluminum added is usually between 0.1 % (m/m) and 0.2 % (m/m). Other deoxidants, such as, titanium or zirconium, can be used with similar restrictions.

9.3.1.5 Remove the sample from the mold and cool in a manner to prevent cracking and to ensure ease of machining.

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

9.3.2 Maintenance of Equipment:

9.3.2.1 Maintain sampling spoons and metal molds in a clean and dry condition.

9.3.2.2 Molds should be remachined if the internal surfaces become worn. This avoids the need for additional machining of the sample during surface preparation. After use, remove any slag and skull from the mold, and clean the surfaces with a wire brush.

9.4 Preparation of a Sample for Analysis:

9.4.1 Preliminary Preparation—From a sample taken from the melt, remove any surface oxidation that may contaminate the sample for analysis during subsequent preparation.

9.4.2 Sample for Analysis by a Chemical Method:

9.4.2.1 In the case of a spoon sample, drill the cylindrical sample at a point one third from the bottom through to the

center 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 may be necessary to soften the sample sufficiently for machining.

9.4.2.2 In the case of a probe sample, obtain chips from the disk section of the sample by drilling or milling as described in 11.2.2.

9.4.3 Sample for Analysis by a Thermal Method:

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

9.4.3.2 In the case of a dual-thickness probe sample, punch a slug from the thin section of the disk to constitute a test portion. Heat treatment may be required to soften the probe sample sufficiently for ease of punching if the hardness of the sample exceeds approximately Rockwell HRC 25.

9.4.3.3 In the case of a disk-and-pin probe sample, from the pin cutoff a test portion of a suitable mass for analysis.

9.4.3.4 Particular care is required in the case of samples for the determination of carbon in low-carbon steels to prevent contamination during preparation of the test portion. Use tweezers for all manipulations.

9.4.4 Sample for Analysis by a Spectrochemical Method:

9.4.4.1 In the case of a spoon sample, cut off the base of the cylindrical sample, using an abrasive cutting-off disk to constitute the sample for analysis, usually 20 to 30 mm in thickness. Finish the cut surface before analysis.

9.4.4.2 In the case of a probe sample, remove any lugs or pin, as necessary, and then mill or finish the surface of the disk to expose a surface representative of the sample. The amount of material to be removed in this way should 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. In the case of a dual-thickness probe sample, prepare the thick section of the disk.

9.4.4.3 In the case of samples of leaded steels, enclose equipment used for surface preparation and fit it with dust extraction equipment. (**Warning**—Swarf arising from the surface preparation of leaded-steels, and dust from dust extraction filter systems, shall be collected and disposed of safely in accordance with local regulations for lead-containing waste materials.)

9.5 Sampling and Sample Preparation for the Determination of Oxygen:

9.5.1 Methods of Sampling:

9.5.1.1 Methods of sampling liquid steel for the determination of oxygen are based upon the use of commercially available sampling probes. The main features of the different types of probes are distinguished in Annex A1. Methods of use should be designed to ensure that the operation of sampling does not influence the equilibrium between carbon and oxygen in the melt. It is essential to avoid contamination of the sample and to remove all surface oxidation at each stage in sample preparation.

9.5.1.2 Small appendages to probe samples, such as a pin or a lug, generally are not suitable for preparation as a test portion

free from surface oxidation. A slug obtained by punching from a dual thickness probe sample may be satisfactory. For some applications, it may be preferred to obtain a sample of larger mass by using a sampling probe filled by gravity.

9.5.2 *Preparation of the Test Portion:*

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

9.5.2.2 Cut a slice from the disk of a probe sample, then cut a cube-shaped test portion from this slice of a mass suitable for analysis.

9.5.2.3 Place the test portion in a stainless steel holding block or some other device to hold it firmly and abrade each surface using a fine-cut file or abrade all surfaces with water-cooled silicon-carbide abrasive media. Use tweezers for all manipulations.

9.5.2.4 Immerse the test portion in acetone and dry in air or by exposure to a rough vacuum. Analyze immediately. There should be no delay between preparation of the test portion and analysis.

9.6 *Sampling and Sample Preparation for the Determination of Hydrogen:*

9.6.1 *General:*

9.6.1.1 Methods of sampling liquid steel for the determination of hydrogen are based upon the use of commercial sampling probes. The main features of the different types of probes are distinguished in [Annex A2](#). Methods of use should minimize and control the rapid diffusion of hydrogen from the probe sample that occurs during storage of the sample, and preparation of the test portion. Losses by diffusion may be large at ambient temperatures especially from small-diameter samples.

9.6.1.2 The probe sample should be free from cracks and surface porosity, and from moisture, particularly entrapped water. The condition of the test portion can influence strongly the analytical measurement. Methods of analysis may differ in their sensitivity to the presence of water in the sample. If a suction sampling probe is used, the method of operation should avoid risk of introduction of moisture into the sample.

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

9.6.1.4 It is essential to 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 should be stored in a refrigerant, either liquefied nitrogen or a mixture of acetone and solid carbon dioxide in the form of a slurry is suitable.

9.6.1.5 The probe sample and the test portion should be kept cold when cutting the sample and during preparation of the test portion. Cooling may be carried out by immersion in water that is ice-cold or preferably in a refrigerant. Remove any moisture present on the surface of the test portion after cooling; immerse

the test portion in acetone; then dry by exposure to a rough vacuum for a few seconds.

9.6.1.6 Discard samples that have been cooled or stored inadequately.

9.6.1.7 Preparation of the surface of the test portion by abrading should be kept to a minimum consistent with the need to remove all oxidation products and surface defects.

9.6.1.8 Analyze the test portion immediately after preparation.

9.6.2 *Methods of Sampling:*

9.6.2.1 A range of commercially available sampling probes designed to provide pin-shaped or pencil-shaped samples of varying diameter is available (see [Annex A2](#)). Use the selected sampling probe in accordance with the manufacturer's guidance.

9.6.2.2 It is essential that the probe sample be quenched in ice water and that the ice water be stirred vigorously and continuously during quenching. There should be no delay, and quenching should take place within an interval not greater than 10 s after taking the sample. To allow rapid cooling, quickly remove the silica sheath used as the sample mold.

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

9.6.2.4 If the probe is designed to capture diffusible hydrogen, quench it to cool sufficiently for handling purposes.

9.6.3 *Preparation of the Test Portion:*

9.6.3.1 Cut a test portion of a suitable mass for analysis from the central sections of the probe sample. Cutting should be carried out in such a manner 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.

NOTE 5—Cutting without heating the sample can be accomplished by rapidly breaking pieces from the probe sample with manual or hydraulic bolt cutters.

9.6.3.2 Prepare the surface of the test portion by filing, grit blasting, acid etching, 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 should be reserved exclusively for this purpose to avoid contamination from the grit. If grinding is used, cool the test portion at frequent intervals. It is difficult to prevent the sample from being heated with any of these surface preparation methods. If the test portion is of good quality, no surface preparation using any of these methods should be necessary.

9.6.3.3 Degrease the test portion by immersion in acetone, dry by exposure to a rough vacuum, and analyze immediately. Alternatively, prepare the test portion by immersion in propanol-2 (isopropyl alcohol), and then dry it with diethyl ether.

10. *Pig Irons*

10.1 *General:*

10.1.1 The following methods are applicable to the sampling of iron from the blast furnace that has been cast into pieces of a simple shape, known as a pig, usually in the form of a double lozenge or some other similar shape. The various

types of pig irons are classified in ISO 9147. Other types of iron may be used in the manufacture of cast iron, for example, iron produced in a cupola furnace or an electric melting furnace.

10.1.2 Use special care to ensure the collection of a representative sample from pig irons.

10.2 Increment Sampling:

10.2.1 *Number of Increments*—Ensure that the number of pigs taken as incremental samples are 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 the purchaser, the minimum number of pigs to be taken from a consignment shall be in accordance with ISO 9147 (see [Table 1](#)).

10.2.2 Methods:

10.2.2.1 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.

10.2.2.2 In the case of a consignment supplied in wagons or trucks, the points of sampling shall be prescribed in a definite order, for example, from five positions, one at the center of the wagon and four others at one sixth of the distance from each of the corners of the wagons along the two diagonals.

10.2.2.3 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.

10.2.2.4 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 points of sampling shall be prescribed in a definite order on the surface of the stockpile.

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

10.2.3 Consignment of Mixed Pig Irons:

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

10.2.3.2 Incremental samples then should 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.

10.3 Preparation of a Sample for Analysis:

10.3.1 General:

10.3.1.1 If the pigs taken as incremental samples contain remnant magnetism as a result of handling with magnetic grabs, demagnetize them by means of a demagnetizing coil to prevent separation of coarse and fine particles during drilling.

10.3.1.2 To obtain chips by machining, drill at a low speed (100 r/min to 150 r/min) using a freshly sharpened tool and adjust the speed and feed to produce chips of uniform size with a minimum of fine material. A drill with a diameter of 12 mm to 14 mm is suitable for obtaining chips. Resharpener the drill at frequent intervals and avoid overheating both the sample and the tool.

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

10.3.1.4 Chips should 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 should be approximately from 1 mm to 2 mm.

10.3.1.5 Do not employ milling because of the high proportion of fines produced.

10.3.1.6 Do not wash the prepared sample with a solvent or treat it magnetically because of the risk of altering the distribution of metal and graphite.

10.3.2 *Sample for Analysis by a Chemical Method*—Prepare each of the incremental samples using one of the following methods:

10.3.2.1 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, and cease drilling at a point approximately 5 mm from the opposite face. If necessary drill a further hole parallel to the first hole. (See [Fig. 3\(a\)](#), [Fig. 3\(b\)](#), [Fig. 3\(c\)](#), and [Fig. 3\(d\)](#).)

10.3.2.2 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; then grind using a vibratory grinding mill to a particle size of less than 150 μm .

10.3.2.3 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.

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

10.3.3 Sample for Analysis by a Thermal Method:

10.3.3.1 To prepare samples in the form of chips or fragments from machinable iron, at the center 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, so that large chips, approximately 1 mm to 2 mm in size, are obtained.

10.3.3.2 To prepare samples in the form of chips or fragments from non-machinable iron, obtain small pieces from the pig as described in [7.4.2.1](#), and then crush these pieces using a percussion mortar to a particle size between approximately 1 mm and 2 mm.

TABLE 1 Minimum Number of Pigs to Be Taken as Incremental Samples from a Consignment of Pig Iron

Consignment, Tons	Number of Pigs
< 10	9
10–20	11
20–40	12
40–80	14
80–160	16
160–300	18
300–600	21
> 600	24

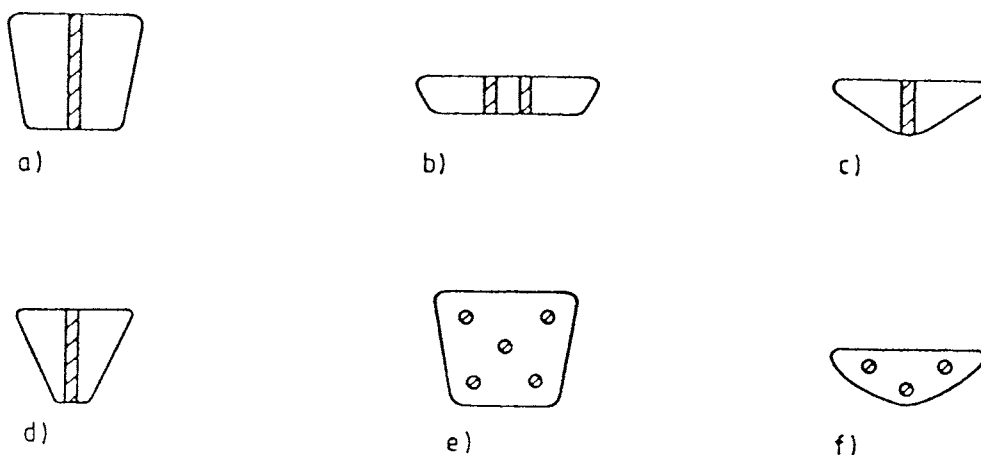


FIG. 3 Sampling Positions for Pig Irons

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

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

10.3.3.5 To prepare samples in the form of a solid mass, 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 Fig. 3(e) or Fig. 3(f) to constitute test portions of a mass suitable for analysis. Alternatively, saw or break the pig at a position midway along its length. Using a trepanning tool, drill either three or five holes at positions corresponding to those shown in Fig. 3(e) and Fig. 3(f) to obtain pins of approximately 3 mm in diameter. Break the pins into pieces to constitute test portions of a mass suitable for analysis.

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

11. Cast Iron Products

11.1 General:

11.1.1 The location and method of taking a laboratory sample from a cast iron product shall be agreed upon between the supplier and the purchaser in relation to one of the methods described in the following paragraphs.

11.1.2 The sample can be selected from a test bar or block cast onto the casting for the purpose of mechanical testing.

11.1.3 Take special care to ensure the collection of a representative sample from cast iron products. There may be differences in chemical composition, particularly in the contents of carbon, sulphur, phosphorus, manganese, and magnesium between the selected sample and that of the casting or castings as a whole. Segregated elements may concentrate towards the upper surfaces of a casting and under cores. These regions should be avoided when selecting a laboratory sample. Sectional dimensions and areas of differential heating or cooling require special attention. Careful consideration is necessary in the design of sampling strategies for high phosphorus engineering iron, and malleable and ductile irons. Pay

particular attention when sampling grey irons to ensure that the sample is representative of the chemical composition of the product especially where segregation is suspected.

11.1.4 Samples obtained from cast iron products that contain free graphite may not be suitable for accurate analysis by using atomic emission or X-ray fluorescence spectrometric methods. In such cases, it is preferable to use other methods of analysis, obtaining suitable samples as described in 11.2.2 and 11.2.3.

11.2 Sampling and Sample Preparation:

11.2.1 General:

11.2.1.1 Sample and prepare samples in accordance with the grade of iron, type of casting, and the method selected for analysis.

11.2.1.2 Clean the sample product or laboratory sample by scratch-brushing, grinding, or shot-blasting to remove adhering particles of sand and to expose areas of metallic surface. Ensure that both the inner and outer surfaces of hollow castings are clean.

11.2.2 Sample for Analysis by a Chemical Method:

11.2.2.1 Machine the sample to obtain chips 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. Take care 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 should be discarded. Milling shall not be employed owing to the high proportion of fines produced.

11.2.2.2 Chips should be as solid and compact as possible with a mass of approximately 10 mg in order to prevent crumbling of graphite. Do not wash chips with a solvent or treat them magnetically because of the risk of altering the distribution of metal and graphite. A 10-mm diameter tool is suitable for obtaining chips by drilling. The size range of chips intended for the determination of carbon or nitrogen should be approximately from 1 mm to 2 mm.

11.2.2.3 When machining is not practical, the sample can be broken into pieces and pieces crushed using a percussion mortar or a vibratory grinding mill to obtain a sufficient mass

of sample for analysis of particle size less than 150 μm . Use this method only when it can be shown that pulverization does not lead to contamination of the sample.

11.2.2.4 For grey irons, obtain chips from the central section of a casting, that is, from a region that represents approximately one third of a complete section of the casting. Do not use chips obtained from the as-cast surface. 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 sample for analysis. For a casting of large section, it may be impracticable to drill through the casting. In such a case, drill halfway 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 3 mm to 5 mm in diameter 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 sample of particle size less than 150 μm .

11.2.2.5 For malleable irons, wherever possible, obtain the sample prior to treatment by annealing. Annealing causes major segregation. It is essential that a sample taken from an annealed casting represents the full cross section of a casting. Particular care is required in obtaining a sample from a casting with variable sectional thickness. If annealed material has to be analyzed, remove a complete cross section by machining; break into pieces; crush in a percussion mortar or disk 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 sample for analysis.

NOTE 6—Malleable iron products are particularly susceptible to segregation of manganese sulfide where the manganese to sulfur ratio exceeds 2:1.

11.2.2.6 For white irons and alloy irons, it may be possible to obtain the sample by drilling as described in 11.2.2.4. Where drilling is impractical, cut thin slices from the sample product or laboratory sample, preferably complete cross sections, using a saw, or if necessary, an abrasive cutting disk. If an abrasive cutting disk 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 sample for analysis of particle size less than 150 μm .

11.2.3 *Sample in the Form of a Solid Mass for Analysis by Thermal Methods:*

11.2.3.1 Cut a thin slice from the sample product or laboratory sample as described in 11.2.

11.2.3.2 In the case of a large casting, obtain a sample for analysis of 3 mm to 5 mm in diameter by using a trepanning tool. Break off pieces from the sample or cut using a saw to obtain a number of test portions of suitable mass for analysis. Analyze 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 0.3 g.

11.2.4 *Sample for Analysis by a Spectrochemical Method:*

11.2.4.1 Use a saw or abrasive cutting disk to cut a sample of suitable size from the sample product or laboratory sample.

11.2.4.2 Prepare a cut surface by grinding using a fixed-head machine or by finishing, or by a combination of both methods. Air cool the sample to avoid overheating. Do not use liquid coolants.

NOTE 7—A fixed-head grinder is preferred to a swing grinder for surface preparation, the latter type of equipment may not produce a flat surface on the sample for analysis.

11.2.4.3 The method selected for remelting should produce a chill cast sample with a white iron structure. Particular attention should be given to avoid partial losses of elements.

12. Steel Products

12.1 General:

12.1.1 The location and method of selecting a laboratory sample from a sample product shall be agreed upon between the purchaser and the supplier using one of the methods in the following paragraphs.

12.1.2 The laboratory sample can be taken from the sample product at the location indicated in the product standard for the selection of material for mechanical testing. See also ISO 14284.

12.1.3 Special considerations apply to the sampling and sample preparation of leaded steel products (12.5), and of steel products for the determination of oxygen (12.6), and hydrogen (12.7).

12.2 Selection of a Laboratory Sample from a Cast Product:

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

12.2.2 Alternatively, and where a sample is required in the form of a solid mass, cut a laboratory sample from the product by machining or by using a cutting torch at the half or quarter cross section of the product.

12.3 Selection of a Laboratory Sample from a Wrought Product:

12.3.1 General:

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

12.3.1.2 Methods of obtaining a sample for analysis in the form of a solid mass or chips are illustrated in the following by reference to products of differing cross sections.

12.3.2 Sections:

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

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

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

12.3.2.4 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 center and edges (see Fig. 4(a) and Fig. 4(b)).

12.3.2.5 For a section of complex shape, for example, an angle, a tee, a channel, or a beam, obtain chips by drilling at points indicated in Fig. 4(c), Fig. 4(d), Fig. 4(e), Fig. 4(f), and Fig. 4(g) leaving a clearance of at least 1 mm around the drill.

12.3.2.6 For a rail, obtain chips by drilling a hole, 20-mm to 25-mm diameter in the head of the rail at a position halfway between the centerline and edge of the rail (see Fig. 4(h) and Fig. 4(j)).

12.3.2.7 Where drilling into the end or cut face of the section is impractical, obtain chips by drilling inwards from the surface perpendicular to the main axis.

12.3.3 *Plates or Slabs*—Cut a laboratory sample of a suitable size for analysis in the form of a solid mass or chips from a point midway between the centerline and the outer edge of

the plate. In the example shown in Fig. 4(k), the laboratory sample is 50 mm wide. Where this is not practical, take the sample from a position agreed upon between the supplier and the purchaser as being representative of the plate composition.

12.3.4 *Light Sections, Bars, Rods, Sheets, Strips, and Wires:*

12.3.4.1 Where the sample product is of sufficient cross-sectional area, cut a slice transversely to constitute the laboratory sample and obtain a sample for analysis as described in 12.3.2.

12.3.4.2 Where the sample product is of insufficient cross section for machining, for example, a thin gage 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.

12.3.4.3 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.

12.3.5 *Tubes and Pipes*—Sampling may be in accordance with one of the following methods:

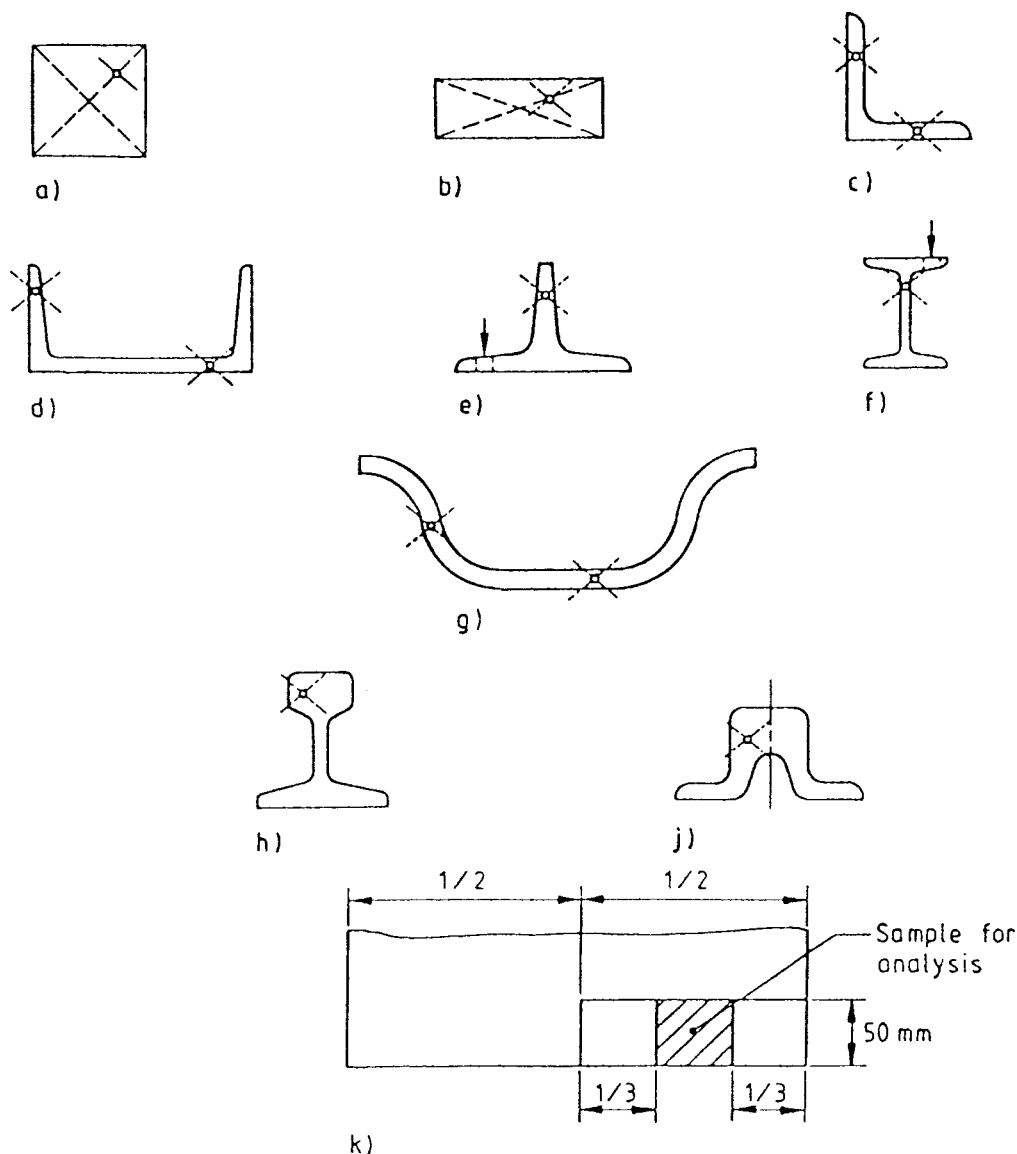


FIG. 4 Sampling Positions for Steel Sections

12.3.5.1 Cut a laboratory sample at a position 90° from the weld on a welded product.

12.3.5.2 Cut across the tube and turn or mill across the cut face to obtain a sample in the form of chips. Thin section tubes may be flattened before milling.

12.3.5.3 Drill through the wall of the tube or pipe at several positions around its circumference to obtain a sample in the form of chips.

12.4 *Preparation of a Sample:*

12.4.1 *General*—Methods for the sample preparation of steel products shall be in accordance with the general requirements specified in 6.4. Special requirements are considered in the following.

12.4.2 *Sample in the Form of Chips:*

12.4.2.1 Chips obtained by machining shall be of a sufficiently small size to avoid or minimize the need for subsequent fragmentation to prepare the sample for analysis. The size of the chips should be such that the mass of individual chips is approximately 10 mg (100 chips/g) for unalloyed and low-alloy steels and approximately 2.5 mg (400 chips/g) for high-alloy steels. If the size of the chips is not sufficiently small for analysis, crush the chips in a percussion mortar.

12.4.2.2 Machine in such a manner to avoid the production of fine material. In the case of a sample that does contain fine powder, that is, particles of size less than approximately 50 µm, separate the coarse and the fine particles and determine the mass of each size fraction. Weigh proportionate parts of each fraction to obtain a representative sample for analysis.

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

12.4.2.4 In the case of a sample for the determination of carbon where the carbon is present in very small amounts, for example, in interstitial free steels, the chips may be subject to contamination from carbonaceous materials present in the atmosphere or from other sources. The chips should be stored in a manner that prevents contamination. Alternatively prepare a test portion in a solid form such as a slug obtained by punching.

12.4.3 *Sample for Analysis in the Form of a Solid Mass:*

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

12.4.3.2 When using an atomic emission spectrometric method for sample of products with a thickness of approximately 1.5 mm or less, it is necessary to reduce the local heating that occurs when the electrical discharge is produced. For example, the edges of the sample can be electrically welded to a small block of steel or the sample can be embedded in another material such as, tin, leaving one surface exposed.

12.5 *Sampling of Lead Steel:*

12.5.1 Take care to minimize the creation of dust particles during all sampling and sample preparation operations. (**Warning**—Swarf arising from machining and surface preparation of leaded steels, and dust from dust extraction filter systems, shall be disposed of safely in accordance with local regulations for lead-containing waste materials.)

12.5.2 Select a laboratory sample from the sample product by cutting using a saw.

12.5.3 Obtain chips by milling at a low speed to avoid overheating the sample and the creation of dust.

12.5.4 Enclose the equipment used for the surface preparation of a sample by a spectrochemical method and fit it with dust extraction equipment.

12.6 *Sampling and Sample Preparation for the Determination of Oxygen:*

12.6.1 *General:*

12.6.2 Avoid contamination of the sample and remove any surface oxidation at each stage in sampling and sample preparation.

12.6.2.1 Do not allow the fingers to come into contact with the test portion. Use tweezers for manipulation. For steels containing very low oxygen contents of approximately < 10 ppm, machine the test portion under the protection of an inert gas.

12.6.3 *Methods of Sampling*—Sample using one of the following methods:

12.6.3.1 Cut a suitably shaped laboratory sample by using a mechanical or abrasive saw. The sample can, for example, be in the form of a small plate or disk. Using a hand or water-cooled abrasive saw, cut a test portion from this sample of suitable mass for analysis.

12.6.3.2 Cut a laboratory sample in the form of a slice, 3 mm to 4 mm thick, finish the surfaces of the sample using 60-grit to 80-grit silicon-carbide abrasive paper and then abrade by using a burr, 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 should be smooth, metallically bright, and free from defects. Punch a slug from the sample of a suitable mass for analysis to constitute a test portion using a 4-mm to 6-mm diameter punch. Perform the punching operation so that the test portion falls into a container that can be closed with a cap or stopper.

NOTE 8—If the surfaces of the sample are finished with water cooled silicon-carbide abrasive media, no further surface preparation should be necessary before punching slugs for use as test portions.

12.6.3.3 Cut a rectangular-shaped laboratory sample, approximately 10-mm section by 100 mm long. Turn the sample using a lathe at a speed of approximately 1000 r/min to a diameter of approximately 7 mm; continue turning at a controlled feed rate of approximately (0.1 to 0.15) mm/revolution at a speed of (800 to 1000) r/min to reduce the diameter of the sample to 6 mm. The condition of the surfaces of the sample after preparation should be smooth, metallically bright and free from defects. Do not use cooling lubricants during the final stage of the matching process. Using a hand saw, cut a test portion from the turned sample of a suitable mass for analysis.

12.6.4 *Preparation of a Test Portion:*

12.6.4.1 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.

12.6.4.2 If the test portion was obtained using the method described in 12.6.3.1, abrade each of the surfaces of the test portion.

12.6.4.3 If the test portion was obtained using the method described in 12.6.3.3, the cylindrical surface of the test portion should be sufficiently smooth to dispense with the need for filing but 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; analyze immediately.

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

12.7 *Sampling and Sample Preparation for the Determination of Hydrogen:*

12.7.1 *General:*

12.7.1.1 Methods should be designed to minimize and control the rapid diffusion of hydrogen from the sample that occurs during sampling, storage of the sample, and preparation of the test portion. The sample should be free from cracks and surface porosity and from moisture. The condition of the test portion can strongly influence the analytical measurement. Methods of analysis may differ in their sensitivity to the presence of water.

12.7.1.2 Rigidly adhere to procedural detail in order to obtain consistency in the quality of analysis.

12.7.1.3 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 and test portion at a temperature that is as low as possible at all stages during sampling, storage, and sample preparation. Store the sample in a refrigerant; either liquid nitrogen or a slurry of acetone and solid carbon dioxide is suitable.

12.7.1.4 Keep the sample and the test portion cold when cutting the sample and during preparation of the test portion. Either use a copious flow of cooling fluid during all machining operations or cool the sample and test portion at frequent intervals, or use a combination of both methods. Cool by immersion in ice-cold water or, preferably, in a refrigerant. Pack samples of large section with solid carbon dioxide in a manner that ensures the maintenance of good thermal contact

between the sample and the solid carbon dioxide. During intervals between machining, return rough-cut pieces to storage in a refrigerant.

12.7.1.5 Remove any moisture present on the surface of the test portion after cooling. Immerse the test portion in acetone and then dry it by exposure to a rough vacuum for a few seconds.

12.7.1.6 Discard samples that have been cooled or stored inadequately.

12.7.1.7 Abrading the surface of the test portion should be kept to a minimum consistent with the need to remove oxidation products and surface defects.

12.7.1.8 Analyze the test portion immediately after preparation.

12.7.2 *Methods of Sampling:*

12.7.2.1 From a cast product, obtain a sample of a suitable size by using a trepanning tool.

12.7.2.2 From a wrought product or a large forging, using a saw or an abrasive cutting disk, obtain a laboratory sample at a position mid-width from the product centerline. From the laboratory sample, cut a piece of a size suitable for turning using a lathe in order to obtain a sample for analysis.

12.7.2.3 Store the sample in a refrigerant.

12.7.3 *Preparation of a Test Portion:*

12.7.3.1 From the sample, cut a test portion of a suitable mass in such a manner to minimize heating the sample, then cool the sample at frequent intervals.

12.7.3.2 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 blasting machine should be reserved exclusively for this purpose to avoid contamination of the test portion from contaminated grit. If grinding is used, cool the test portion at frequent intervals. It is difficult to prevent the sample from being heated with any of these surface preparation methods. If the test portion is of good quality, no surface preparation using any of these methods should be necessary.

12.7.3.3 Degrease the test portion by immersion in acetone, dry by exposure to a rough vacuum for a few seconds, then analyze immediately. Alternatively, prepare the test portion for analysis by immersion in propanol-2 (isopropyl alcohol), and then dry it with diethyl ether.

13. **Keywords**

13.1 castings; iron sample; sample lot; steel sample

A1. SAMPLING PROBES FOR USE WITH LIQUID IRON AND STEEL

A1.1 General

A1.1.1 Disposable probes for taking samples from liquid iron and steel consist of a small mold made from pressed steel, ceramic material, or silica tubing, and which are mounted in a thick-walled protective cardboard tube.

A1.1.2 A range of different types of sampling probes is available commercially. The main features are described in the following paragraphs with illustrative examples shown in Figs. A1.1-A1.6. The dimensions given in this annex are for example only.

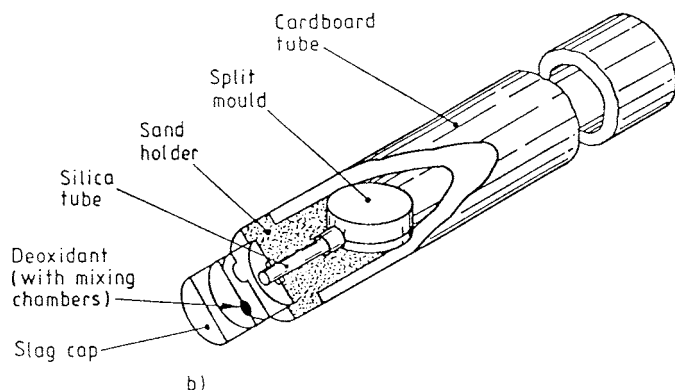
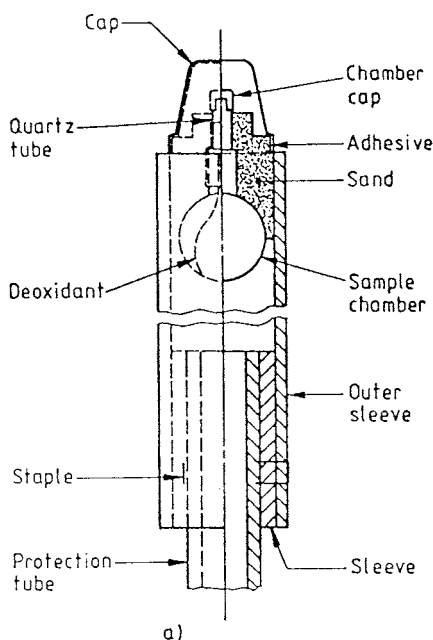


FIG. A1.1 Examples of Immersion Probes Filled by Ferrostatic Pressure (a) With the Deoxidant in the Sample Chamber and (b) with the Deoxidant in a Separate Mixing Chamber

A1.2 Immersion Sampling Probes

A1.2.1 Immersion probes can 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 times vary depending on the design of the probe and the conditions of sampling, in particular the temperature of the melt, but are normally between 3 s and 8 s.

A1.2.2 The lance is constructed so that air from the mold cavity and gases produced during the combustion of cardboard can escape easily. A handle bar is incorporated for controlling the lance during immersion and withdrawal.

A1.2.3 When sampling from ladles and secondary treatment vessels, a mechanical system can be used to lower and raise the lance.

A1.2.4 Some types of probe combine a thermocouple in a silica tube adjacent to the sample chamber for measurement of temperature. Where a measurement substance is used in a basic oxygen converter furnace, a mold assembly can be incorporated with the measurement sensors in the substance to obtain a sample for laboratory analysis.

A1.2.5 Probes in which the sample chamber is filled by ferrostatic pressure consist of a steel split mold held in a cardboard tube by a collar of refractory material. The mold 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 1500 mm or longer, may be coated partially with refractory material to minimize splashing during immersion.

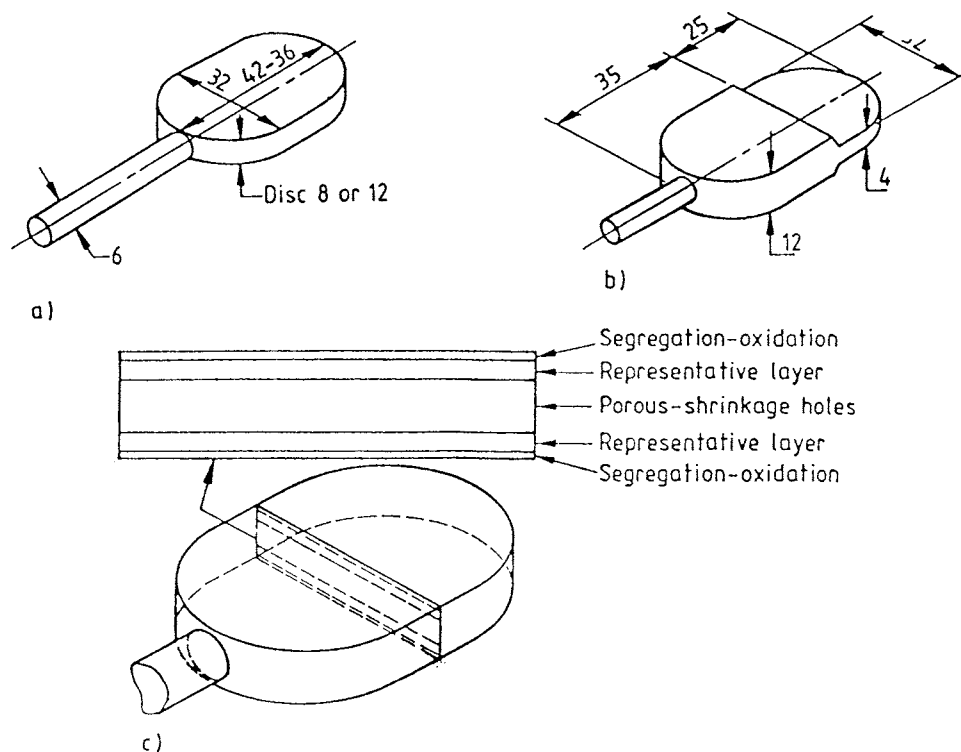
A1.2.5.1 This type of probe is used mainly to obtain samples from liquid steel in furnaces and ladles. Two designs are shown in Fig. A1.1.

A1.2.6 The samples obtained by using immersion probes filled by ferrostatic pressure can differ in configuration.

A1.2.6.1 The disk-and-pin probe samples are exemplified in Fig. A1.2. The disk is suitable for analysis by a spectrochemical method, and the pin may be used, if required, for analysis by a thermal method. The disk may be oval or circular or some other similar shape.

A1.2.6.2 For disk-and-pin probe samples with a number of lugs attached to the disk, the lugs have a mass of 0.5 g or 1 g, and may be easily broken off from the disk and can be used, if required, as test portions in thermal methods of analysis.

A1.2.6.3 The dual-thickness probe samples are illustrated in Fig. A1.2(b), in which part of the disk 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 disk of a dual-thickness probe sample is used for analysis by spectrochemical methods. A probe sample with a hardness of greater



NOTE 1—All dimensions are in millimetres.

FIG. A1.2 Disk-and-Pin Samples Showing (a) the Basic Design, (b) the Dual-Thickness Variant, and (c) Layer Stratification of the Disk of the Sample

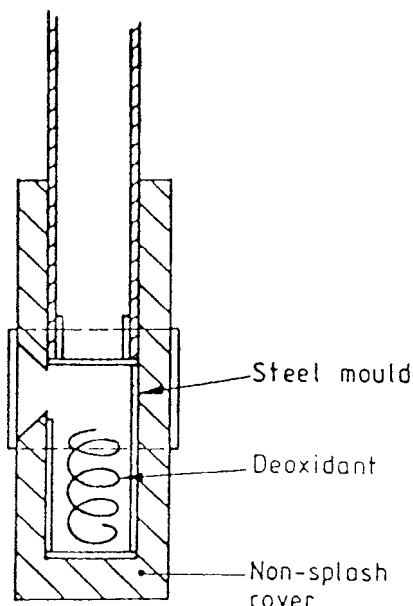


FIG. A1.3 Example of an Immersion Sampling Probe Filled by Gravity

than approximately Rockwell 25 HRC may require heat treatment before punching.

A1.2.7 Probes of the type shown in Fig. A1.3 in which the sample chamber is filled by gravity consist of a two-piece or four-piece steel cylindrical mold that is contained in a refractory assembly and attached to a cardboard tube.

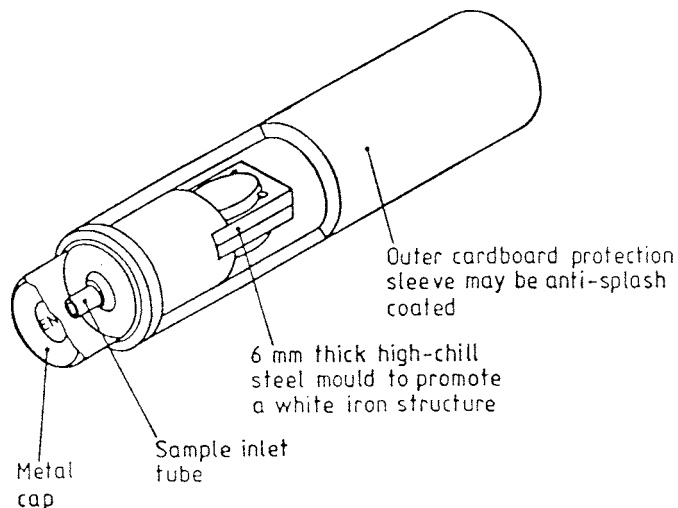


FIG. A1.4 Example of an Immersion Probe Used for Sampling Liquid Iron from a Blast Furnace

A1.2.7.1 The mold 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; overall length of the assembly and cardboard tube is 400 mm to 800 mm. Immersion times are usually 2 s or 3 s.

A1.2.7.2 This type of probe is used in situations where the disk-and-pin type of sample is not satisfactory for analysis. It is used to obtain samples from liquid iron and steel in ladles

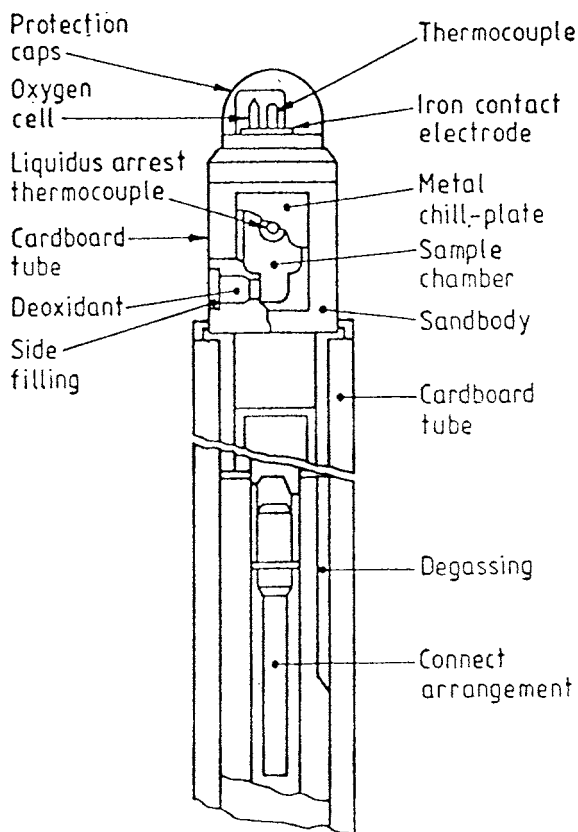


FIG. A1.5 Example of a Sublimation Probe Assembly Showing the Sample Chamber

and from liquid steel in ingot molds and continuous casting tundishes. The sample is typically 30 mm in diameter by 70 mm long.

A1.2.8 Probes of 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 types are illustrated as follows:

A1.2.8.1 The first type is a variant of the probe filled by ferrostatic pressure (A1.2.6) with a design based on the use of either a thick-walled, steel split mold or steel chill plates to provide a disk-and-pin sample with a number of lugs attached to the disk. This type of probe is shown in Fig. A1.4. The disk of the sample can vary in thickness from 8 mm to 12 mm and the pin is typically 4 mm in diameter. Immersion times vary from 5 s to 9 s depending upon the application.

A1.2.8.2 The second type is a probe designed to eliminate the risk of drainage of liquid iron when sampling very fluid melts. The side-entry mold has one or more steel chill-plates and is held in a sand body attached to a cardboard tube. This type of probe can combine a separate or linked pin-shaped mold. The sample is typically 35 mm in diameter with a thickness varying from 4 mm to 12 mm depending on the metallurgical structure required. The optional pin sample is 6 mm in diameter by 45 mm long.

A1.2.9 Probes of special design are used to obtain samples from liquid steel in a vacuum induction furnace.

A1.2.9.1 In one example, the sampling mechanism in the form of tube made from a refractory material is located in the furnace charging system and suspended on a wire rope to permit vertical access to the melt by gravitational means. The sample is cylindrical in shape, 35 mm in diameter.

A1.2.10 Measurement sensor assemblies used with substances in basic oxygen steelmaking may incorporate molds for obtaining samples from liquid steel. Molds of the types described in A1.2.2 may be used both in the cases of a substance used during oxygen blowing (in-blow operation) and when oxygen is not being blown (end-blow operation). In the case of in-blow operation a mold of different design can be used to obtain a sample of a rectangular shape, (40 by 30 by 20) mm thick.

A1.2.10.1 A typical assembly, shown in Fig. A1.5 includes sensors for the measurement of liquidus arrest, temperature and oxygen potential, and incorporates a rectangular-shaped mold with a side entry to obtain a sample during the measurement process.

A1.3 Stream Sampling Probes

A1.3.1 Probes of the type shown in Fig. A1.6(a) consist of a steel split mold with an exposed entry tube of silica held by a plug in a cardboard tube of length 100 mm to 225 mm. Samples are of the disk-and-pin type.

A1.3.2 Differing designs of mold construction are used for the sampling of liquid iron.

A1.3.3 Lances for stream sampling probes are designed so that the probe can be positioned in the metal stream at an angle of 45°. Some means of supporting the lance may be provided. Sampling time is usually 2 s.

A1.3.3.1 This type of probe is used to obtain samples from liquid iron and steel flowing from ladles.

A1.4 Suction Sampling Probes

A1.4.1 Probes of the type shown in Fig. A1.6(b) consist of a steel split mold held in position by a plug in a cardboard tube, about 125 mm in length. The mold has an exposed entry tube of silica with a protective cap to prevent ingress of slag or casting powder. Air is removed from the mold to create a partial vacuum by using a hand-operated pump or a compressed-air venturi pump. Sampling time is usually 2 s.

A1.4.1.1 This type of probe is used to obtain samples from liquid steel in small furnaces, ingot molds, continuous casting molds, and tundishes. Samples are of the disk-and-pin type.

A1.5 Deoxidation Systems for Sampling Probes

A1.5.1 Probes used for the sampling of liquid steel that is oxidized and effervescent contain a deoxidant usually in the form of wire or a pellet inserted in the probe in such a manner that it becomes uniformly dispersed in the liquid steel. Various methods are used to incorporate the deoxidant in the probe, for example:

A1.5.1.1 Deoxidant in the sample chamber itself as shown in Fig. A1.1(a) and Fig. A1.3.

A1.5.1.2 Deoxidant in the entry tube to the sample chamber:

A1.5.1.3 Deoxidant in a separate chamber. The deoxidant and liquid steel are thoroughly mixed before the steel enters the

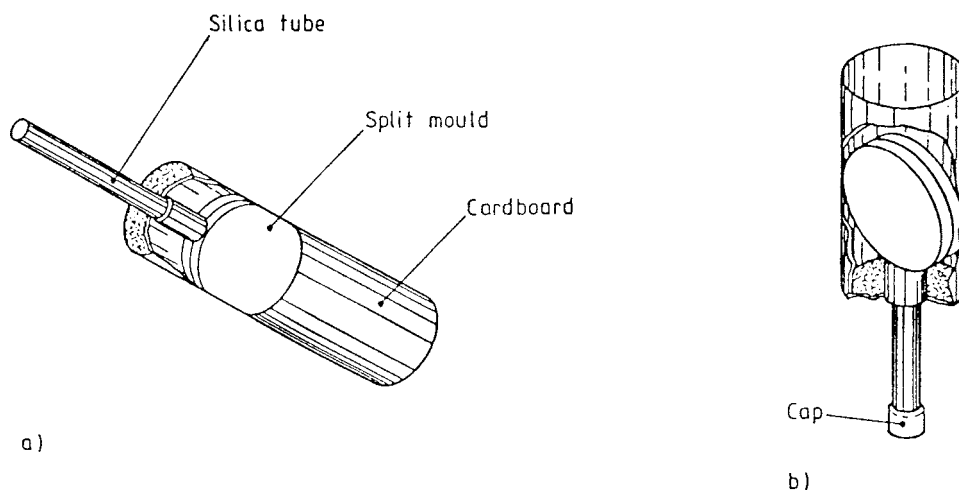


FIG. A1.6 Examples of (a) Stream Sampling and (b) Suction Sampling Probes

sample chamber, as shown in Fig. A1.1(b). Some probes have a second mixing chamber.

A1.5.1.4 Aluminum, zirconium, or titanium are commonly used as deoxidants dependent upon the type of melt and the analytical requirements.

A1.6 Quality of Sample

A1.6.1 Apart from the presence of defects and oxides on the surfaces of the disk sample, the surface layers of the disk may be subject to segregation and the central portion may be porous and subject to shrinkage or other thermal effects as shown in Fig. A1.2(c). Special care is necessary to ensure that procedures for preparing the surface of the disk for analysis by spectrochemical methods expose a layer of metal that is representative of the chemical composition of the sample.

A1.6.1.1 It is usually necessary to remove a layer of 1 mm to 2 mm from the surface of a disk sample obtained from liquid

steel to expose a part of the sample that is suitable for the analysis method selected.

A1.6.2 The amount of material removed from the surface of chill samples taken from liquid iron is determined by the metallurgical structure of the sample that may vary through the thickness of the disk. The type of sampling probe used and the method of preparing the disk sample should be selected to provide a surface structure of either white or grey iron in accordance with the requirements of the analysis method.

A1.6.2.1 In the case of a disk sample obtained from liquid iron, it is usually necessary to remove a layer of thickness approximately 0.5 mm to 1 mm from the surface of the disk sample.

A1.6.3 In routine practice, probe samples should be examined regularly to ensure the suitability of the prepared sample for the analysis method.

A2. SAMPLING PROBES FOR USE WITH LIQUID STEEL FOR THE DETERMINATION OF HYDROGEN

A2.1 General

A2.1.1 Disposable probes for taking samples from liquid steel for the determination of hydrogen usually consist of a mold made from pressed steel or silica tubing mounted in a thick-walled protective cardboard tube. Probes are designed to obtain a pin-shaped or pencil-shaped sample, 7 mm to 12 mm in diameter by 75 mm to 150 mm long, from liquid steel in ladles, ingot molds, and continuous casting tundishes and molds.

A2.1.2 Several types of sampling probes are available commercially; the main features of which are described in the following sections with illustrative examples shown in Fig. A2.1.

A2.1.3 The dimensions given in this annex are for information only.

A2.2 Immersion-Sampling Probes

A2.2.1 Two Types of Probes for Immersion Sampling:

A2.2.1.1 Probes of the type shown in Fig. A2.1(a) consist of a silica tube, 7 mm to 9 mm in internal diameter contained in a protective cardboard tube. The top end of the tube is open and the bottom end is capped with aluminum foil to prevent ingress of contaminants. The cardboard tube is 250 mm or 400 mm in length depending upon the application and has a refractory anti-splash coating. This type of probe is used for taking samples from liquid steel at temperatures near to the liquidus point of the steel.

A2.2.1.2 Probes of the type shown in Fig. A2.1(b) consist of an exposed silica tube, 10 mm to 12 mm in internal diameter, held in a cardboard tube. The top end of the tube is open or may be covered with aluminum foil. The tube has a side entrance port covered with aluminum foil. The tube may contain

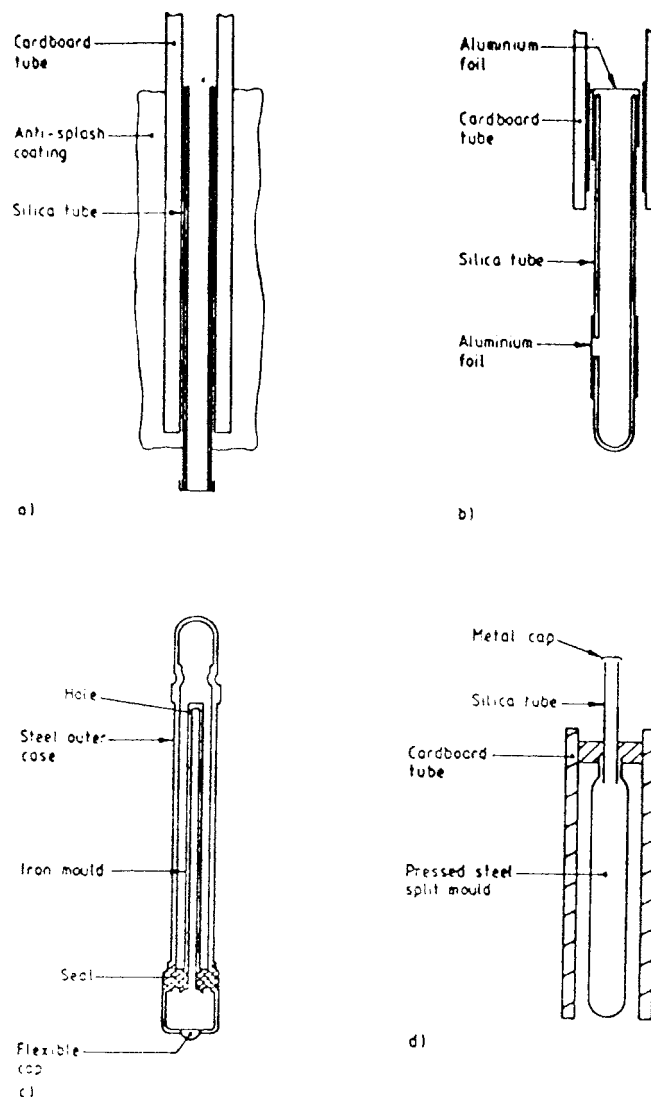


FIG. A2.1 Examples of Probes Used for Sampling Liquid Steel for the Determination of Hydrogen: (a) and (b) Immersion Sampling Probes, (c) an Evacuated Sampling Probe, and (d) a Suction Sampling Probe

aluminum wire as a deoxidant, typically a mass of approximately 0.1 g. This type of probe is used in a wide range of applications for taking samples from liquid steel.

A2.3 Suction Sampling Probes

A2.3.1 Types of Probes for Suction Sampling:

A2.3.1.1 An evacuated probe of the type shown in Fig. A2.1(c) consists of a steel sleeve and a sample chamber made from high-purity iron with an internal diameter of 4 mm. The probe is mounted in a cardboard tube and sheathed with a heat-resistant material. The probe may have a protective slag cap. On immersion in the melt, the fusible cap melts and liquid steel is aspirated into the evacuated sample chamber, which

then closes as the metal solidifies, thus sealing the probe. Hydrogen, which diffuses from the sample, is captured in the outer evacuated chamber and is measured when the probe is inserted into specially designed analytical equipment and the probe pierced. Any residual hydrogen remaining in the sample can be measured separately after removal of the sample together with the sample chamber.

A2.3.1.2 Probes of the type shown in Fig. A2.1(d) consist of a pressed steel split mold, 7 mm to 9 mm in internal diameter by 75 mm long, with a silica entry tube. The mold is held in a cardboard tube by a collar. Air is removed from the mold to create a partial vacuum by using a compressed air venturi pump.

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