Designation: E 1806 – 96

Standard Practice for Sampling Steel and Iron for Determination of Chemical Composition[1]

This standard is issued under the fixed designation E 1806; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 *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.*

1.4 For specific precautions, see Note 2, Note 6, and Note 9 as well as Section 5.

2. Referenced Documents

2.1 *ASTM Standards:*

A 48 Specification for Gray Iron Castings[2]

A 751 Test Methods, Practices, and Definitions for Chemical Analysis of Steel Products[3]

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[4]

E 415 Test Method for Optical Emission Vacuum Spectrometric Analysis of Carbon and Low-Alloy Steel[5]

E 1010 Practice for Preparation of Disk Specimens of Steel and Iron for Spectrochemical Analysis by Remelting[5]

E 1087 Practice for Sampling Molten Steel from a Ladle Using an Immersion Sampler to Produce a Sample for Spectrochemical Analysis[5]

2.2 *ISO Documents:*

ISO 9147 Pig irons–Definition and classification[6]

ISO 14284 Steel and iron—Sampling and preparation of

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[1] This practice is under the jurisdiction of ASTM Committee E-1 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] *Annual Book of ASTM Standards*, Vol 01.02.

[3] *Annual Book of ASTM Standards*, Vol 01.03.

[4] *Annual Book of ASTM Standards*, Vol 03.05.

[5] *Annual Book of ASTM Standards*, Vol 03.06.

[6] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

samples for the determination of chemical composition[6]

3. Terminology

3.1 *Definitions*—For definitions of terms in this practice, refer to Terminology E 135.

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 A 48.

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

3.2.8 *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.

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

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

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

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

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. 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 A 751 and Specification A 48. 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*:

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

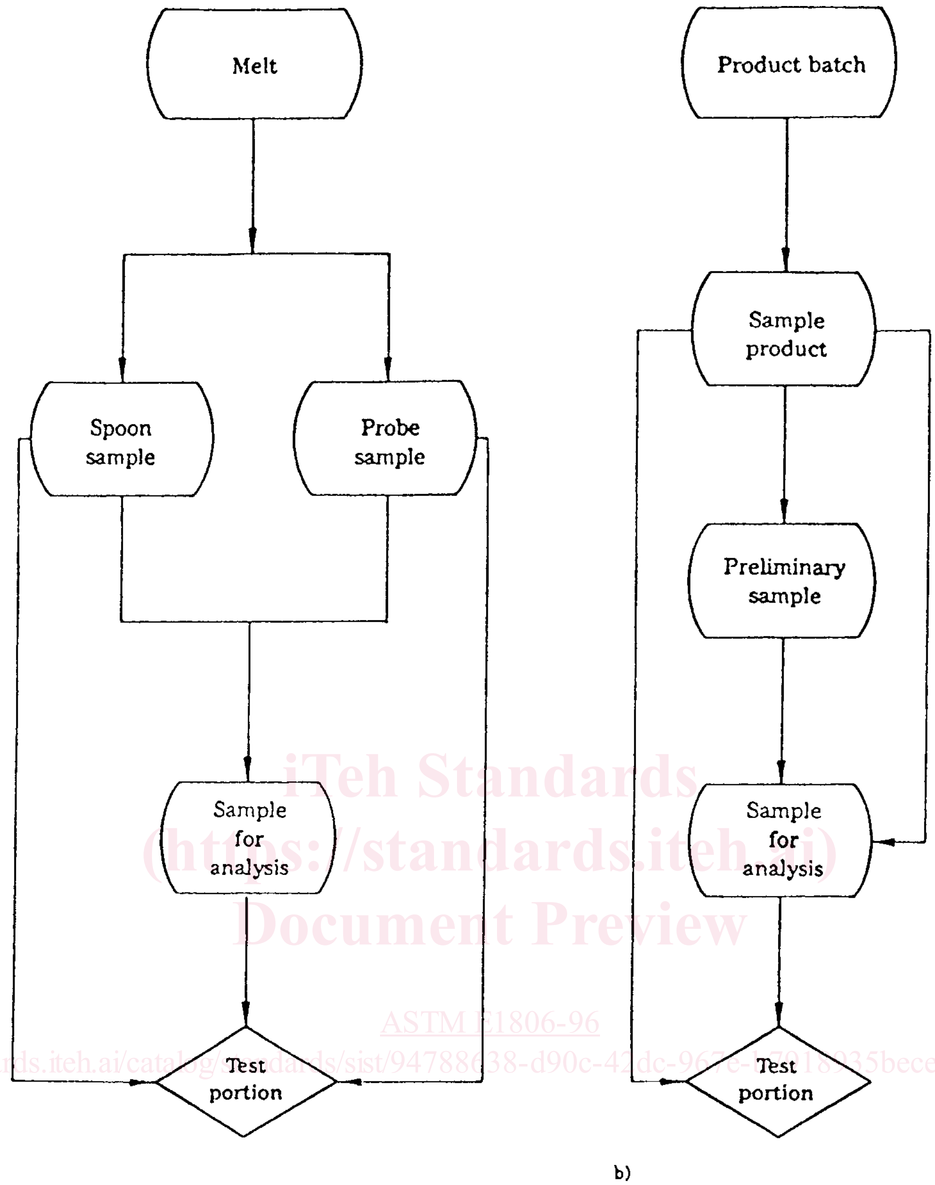


FIG. 1 Sequence of Sampling and Sample Preparation of (a) Liquid Iron and Steel and (b) Cast Iron and Steel Products

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 optical emission and X-ray fluorescence spectrometric methods, the shape and size of the sample will be determined by the dimensions of the sample chamber. The dimensions for samples given in this practice are for guidance only. Refer to Test Method E 415 for sample size requirements for optical 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, status, and condition 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.

NOTE 2—**Caution:** 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 linishing 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 optical emission spectrometric methods, an abrasive with a grade of 60 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 optical 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 E 1010. The sample is converted into a disk, 40 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 to 40 mm in diameter with thickness varying from 6 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 to 32 mm, to facilitate removal of the sample from the mold.

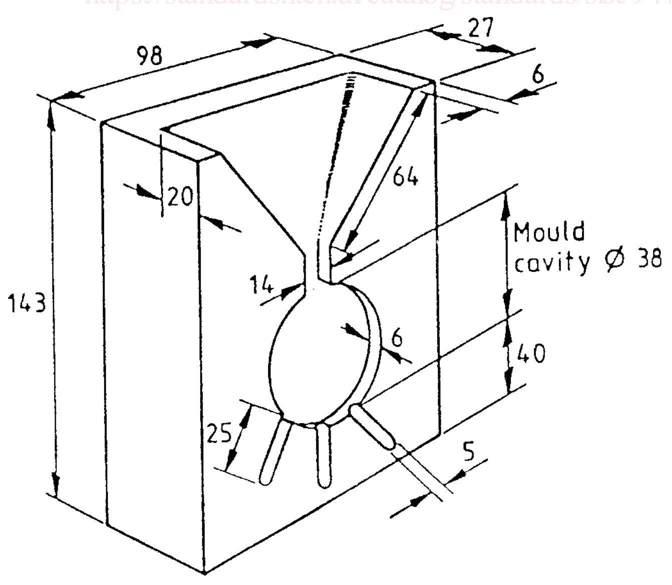
7.2.1.2 A coin sample with one or more attached pins can be obtained using a combination-type mold. the pins are broken off from the disk and used, if required, as test portions for analysis by a thermal method. A combination-type mold for use with liquid iron intended for the production of cast iron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



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

7.2.2.3 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.

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

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 linishing 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 3—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 seriously will 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 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 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 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 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 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. 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 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 linishing. 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 linishing 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 4—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 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 to 40 mm in diameter at the top and 20 to 35 mm in diameter at the base, and 40 to 75 mm long.

NOTE 5—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 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. Linish 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 linish 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 iron and the conditions of sampling. The thickness of the layer to be removed normally lies between 1 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.

NOTE 6—**Caution:** 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. 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