

Sampling and analysis of iron, steel and other ferrous metals —

Part 2: Sampling and sample preparation —

Section 2.1 Methods for iron and steel

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Committees responsible for this British Standard

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- BCIRA
- British Steel Industry
- Department of Trade and Industry (Laboratory of the Government Chemist)
- Ferro Alloys and Metals Producers' Association
- Ministry of Defence

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Foreword

This Section of BS 6200 has been prepared under the direction of the Iron and Steel Standards Policy Committee.

BS 6200 is a British Standard series covering all aspects of the sampling and analysis of iron, steel and other ferrous metals. A general introduction is given in BS 6200-1:1991, which lists the contents of all the existing and projected Parts, Sections and Subsections.

BS 6200-2 supersedes BS 1837:1970, which is withdrawn. Methods of sampling identical to those given in BS 1837 were included in BSI Handbook No. 19:1970, which has also been withdrawn.

This Section of BS 6200 describes methods for the sampling and sample preparation of iron and steel; BS 6200-2.2:1993 describes methods for use with ferroalloys and other alloying additives used in the manufacture of iron and steel.

This Section of BS 6200 takes account of international recommendations ISO 377-2:1989 and Euronorm 18:1979 for the selection and preparation of samples and test pieces for the determination of chemical composition. It also takes account of BS EN 10001:1991 which defines and classifies pig irons and specifies methods for sampling and sample preparation, and a method which is in course of preparation as a European Standard for the preparation of steel samples for oxygen determination.

In addition, information was obtained from ASTM standard practices for sampling iron and steel [1,2] and from BCIRA recommended practices for sampling cast iron [3,4].

Standard methods for sampling and sample preparation of direct reduced iron are being prepared as International Standards by ISO/TC 102 and will be published in due course.

This Section of BS 6200 calls for the use of substances and/or methods that may be injurious to health if adequate precautions are not taken. It refers to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

It has been assumed in the drafting of this Section of BS 6200 that the execution of its provisions is entrusted to appropriately qualified and experienced people.

Assessed capability. Users of this British Standard are advised to consider the desirability of quality system assessment and registration against the appropriate Part of BS 5750 by a third party certification body.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 34, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Subsection 1. General

1.1 Scope

This Section of BS 6200 describes methods for sampling and sample preparation for the determination of the chemical composition of pig iron, cast iron and steel. Methods are described for use with both liquid and solid metal.

1.2 References

1.2.1 Normative references

This Section of BS 6200 incorporates, by reference, provisions from specific editions of other publications. These normative references are cited at the appropriate points in the text and the publications are listed on page 34. Subsequent amendments to, or revisions of, any of these publications apply to this Section of BS 6200 only when incorporated in it by updating or revision.

1.2.2 Informative references

This Section of BS 6200 refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of the standard are listed page 34, but reference should be made to the latest editions.

1.3 Definitions

For the purposes of this Section of BS 6200, the following definitions apply.

1.3.1

chemical method of analysis

method for the determination of chemical composition in which the sample is submitted to chemical reaction

1.3.2

physical method of analysis

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

1.3.3

thermal method of analysis

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

1.3.4

melt

liquid metal from which a sample is removed

1.3.5

spoon sampling

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

1.3.6

spoon sample

sample taken from the melt using a spoon and cast into a small mould

1.3.7

probe sampling

method in which the sample is taken from the melt using a proprietary sampling probe inserted into the melt

1.3.8

immersion sampling

method of probe sampling in which the probe is immersed in the melt where the sample chamber in the probe fills by ferrostatic pressure or gravity

1.3.9

stream sampling

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

1.3.10

suction sampling

method of probe sampling in which the probe is inserted into the melt where the sample chamber in the probe fills by aspiration

1.3.11

probe sample

sample taken from the melt using a proprietary sampling probe

1.3.12

cast product

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

1.3.13

wrought product

item of steel which has been subject to deformation by rolling, drawing, forging or some other method, for example, a bar, a billet, a plate, a strip, a tube, a wire

1.3.14

sample product

specific item of iron or steel selected from a supplied quantity for the purpose of obtaining a sample

**1.3.15
preliminary sample**

sufficient quantity of metal selected from the sample product for the purpose of obtaining one or more samples for analysis

**1.3.16
sample for analysis**

part of the sample product, or part of the preliminary sample taken from the sample product, or part of the sample taken from the melt, brought to a required condition for submission to analysis
the sample for analysis may comprise the sample product itself or the sample taken from the melt

NOTE The following categories of sample for analysis are distinguished:

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

**1.3.17
test portion**

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

NOTE 1 The following special types of test portions in the form of a solid mass obtained from a probe sample are distinguished:

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

NOTE 2 When the sample for analysis is in the form of chips or powder, or when a sample in the form of a solid mass is analysed by a thermal method, the test portion is obtained by weighing. In the case of a physical method of analysis, the part actually analysed will constitute only a small mass of the sample for analysis. In an optical emission spectrometric method, the mass of metal consumed in an electrical discharge is about 0.5 mg to 1 mg; in an X-ray fluorescence spectrometric method, the characteristic radiation is produced from a very thin surface layer of the sample.

**1.3.18
grinding**

method of preparing a sample of metal for a physical method of analysis in which the surface of the sample for analysis is abraded using an abrasive wheel

**1.3.19
finishing**

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

**1.3.20
consignment**

quantity of metal delivered at one time

**1.3.21
increment**

quantity of metal obtained by sampling at one time from a consignment

1.4 Quality systems

Sampling methods have to provide adequate protection for the purchaser, it being recognized that any sampling method has limitations.

For the purpose of demonstrating the fulfilment of contractual requirements the supplier is obliged to provide valid confidence that the required quality will be achieved.

NOTE Requirements for quality systems are specified in BS 5750-2:1987 for use when a contract requires demonstration of a supplier's capability¹⁾ to control the processes that determine the acceptability of the product supplied.

BS 5750-3:1987 specifies requirements for use when a contract requires demonstration of capability to detect and control the disposition of any product nonconformity during final inspection and test.

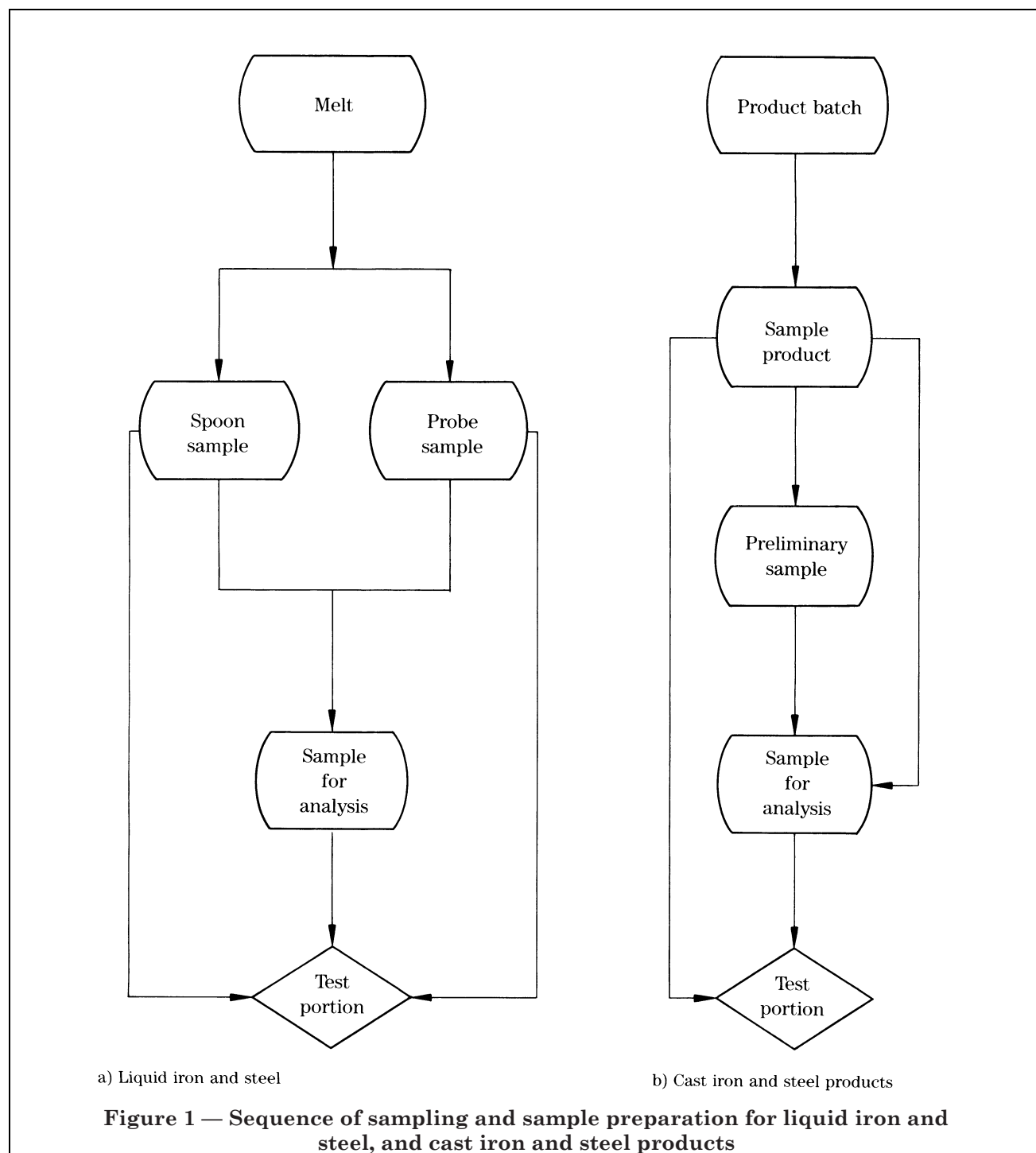
¹⁾ See foreword.

Subsection 2. Requirements for sampling and sample preparation

2.1 General

This subsection considers 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 considered in the relevant subsection.

The sequence of sampling and sample preparation of liquid iron and steel, and cast iron and steel products is shown in Figure 1. Special considerations apply to pig irons (see subsection 6).



2.2 Sample

2.2.1 Quality

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

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

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

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

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

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

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

2.2.2 Size

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

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

The dimensional requirements for a sample for analysis in the form of a solid mass will depend upon the method selected for analysis. In the case of optical emission and X-ray fluorescence spectrometric methods, the shape and size of the sample will be determined by the dimensions of the sample chamber.

NOTE The dimensions for samples for analysis given in this Section of BS 6200 should be regarded as examples only.

2.2.3 Identification

A sample for analysis shall be assigned a unique identification in order 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 preliminary sample or the sample for analysis in the sample product.

A sample for analysis of pig iron shall be assigned a unique identification in order to determine the consignment or part of a consignment and the increment from which it was taken.

Labelling or some equivalent method of marking shall be used to ensure that the assigned identification remains associated with the sample for analysis.

The identification, status and condition of the sample shall be recorded so as to ensure that confusion cannot arise as to the identity of the item to which analysis and records refer.

2.2.4 Conservation

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

A container used for storage shall be completely dry and made from material that is chemically inert and resistant to abrasion by iron and steel. A sample for analysis intended for the determination of an element present in a low concentration shall be stored in a container that is airtight.

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

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

2.2.5 Arbitration

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

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

2.3 Selection of a sample

2.3.1 Sample from a melt

Melts are sampled at various stages in the manufacturing process for the purposes of monitoring and controlling the process. Samples can be taken during the casting of the melt to verify chemical composition in accordance with the specification of the cast product. In the case of liquid metal intended for the production of a casting, the sample for analysis may be selected from test bars or blocks specially cast from the same metal as that of the casting for purposes of mechanical testing in accordance with the product standard.

Sampling practices for melts shall be designed to provide samples during a particular manufacturing process in accordance with requirements for sample quality (see 2.2.1). The sample obtained from a melt is usually in the form of a small ingot, a cylindrical or rectangular block, or a chill-cast disc or a combination of a disc with one or more attached pins; in some cases small lugs are attached to a disc sample.

NOTE 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 A and Annex B.

2.3.2 Sample from a product

The preliminary sample or the sample for analysis can be selected from the sample product at the location indicated in the product specification for the selection of material for mechanical testing when available.

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

In the case of a forging, the sample for analysis can be selected from the initial starting material from which the forging has been made, or from prolongations of the forging or from additional forgings.

In the absence of requirements given in the product standard, or of a specification when ordering the product, the sample for analysis may, following agreement between the supplier and the purchaser, be selected from the sample for mechanical testing or from the test piece, or directly from the sample product.

The preliminary sample or the sample for analysis can be obtained from the sample product by machining or by using a cutting torch. Special considerations apply in the case of sampling for the determination of certain elements.

2.4 Preparation of a sample

2.4.1 Preliminary preparation

If any part of the sample is liable to be non-representative in chemical composition, for example, due to oxidation, it may be agreed, following investigation to establish the nature and extent of any change in composition, to remove from the sample those parts of the sample that have changed. Following this operation, the sample shall then be protected from any change in composition.

If necessary, the surface of the metal shall be laid completely bare at the point of machining by any suitable means to remove any coating that has been applied during manufacture; if necessary, the surface of the metal shall be degreased by means of a suitable solvent taking care to ensure that the manner of degreasing does not affect the correctness of analysis.

2.4.2 Sample for analysis in the form of chips

The sample for analysis shall be obtained by drilling or milling or turning to produce chips of a regular size and shape. Machining shall not be carried out on a part of the sample that has been affected by the heat of a cutting torch.

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

Machining shall be carried out in such a way that the chips are not subject to overheating as indicated by a change in the colour (blueing or blackening) of the chips. Unavoidable coloration of chips obtained from some types of alloy steels, for example manganese and austenitic steels, can be minimized by selection of appropriate tools and cutting speeds.

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

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

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

2.4.3 Sample for analysis in the form of a powder or fragments

Where drilling of the sample to obtain chips is impracticable, the sample shall be cut or broken into pieces and the pieces then crushed using a percussion mortar or a vibratory grinding mill, also known as a disc mill or ring mill, to obtain a sample for analysis in the form of a powder the whole of which passes through a sieve of a specified aperture size.

In some applications for the determination of carbon using a thermal method of analysis, the sample is crushed in a percussion mortar to obtain a sample for analysis in the form of fragments with a particle size range of approximately 1 mm to 2 mm.

Equipment used for comminution shall be constructed from material which does not alter the sample composition.

NOTE For comminution suitable tests may be necessary to show that the use of equipment does not affect the composition of the sample for analysis in this way.

Comminution shall not be used for the preparation of samples of graphite-bearing irons.

The sieving operation shall be performed taking all precautions necessary to avoid contamination or loss of material. Care shall be taken when sieving hard materials to avoid damaging the fabric of the sieve.

The sample for analysis shall be homogenized before weighing the test portion. Powders can be homogenized by stirring.

CAUTION. Finely-divided metals of particle size less than approximately 150 µm can present a fire risk. Ensure adequate ventilation during comminution.

2.4.4 Sample for analysis in the form of a solid mass

2.4.4.1 Selection of the sample for analysis

The sample for analysis shall be obtained by cutting from the sample product or preliminary sample a piece of suitable size and shape for the method of analysis. Samples shall be cut by sawing, abrasive cutting, shearing or punching.

In the absence of any indication in the product standard, analysis by a physical method shall be carried out on that part of the sample corresponding to a transverse section of the product, provided that the material has sufficient thickness.

2.4.4.2 Surface preparation of the sample for analysis

The sample for analysis shall be prepared to expose a surface suitable for the analysis method.

Preparation of a surface for analysis shall not be carried out 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.

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

- a) A milling machine capable of removing a preselected depth of metal in a reproducible manner, for use with samples that are within a hardness range suitable for milling. The equipment should be capable of use, if required, with a sample taken from a melt where the sample is still hot.
- b) A grinding machine with a fixed, rotating or oscillating head capable of removing a preselected depth of metal in a reproducible manner.
- c) A flat-bed finishing machine with abrasive grinding discs, or machines with continuous abrasive-belts, capable of being used to prepare the surface of the sample for analysis to varying grades of finish.
- d) A machine for blasting with sand or grit, or metal shot, capable of being used in special applications to clean the surface of the sample for analysis or the test portion.

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

Cutting and surface preparation can be performed either manually or automatically. In the case of samples taken from melts, proprietary systems may be used which perform each stage of preparation automatically. Systems for the automatic surface preparation of dual-thickness probe samples (see **A.2.3**) and for the punching of slugs as test portions may incorporate facilities for the sand-blasting of the sample and for heat treatment to soften the sample before punching.

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

In the case of optical emission spectrometric methods, an abrasive with a grade of 60 grit to 120 grit is normally suitable. In the case of X-ray fluorescence spectrometric methods, it is essential to ensure that the method selected for surface preparation produces a grade of surface finish that is reproducible from sample to sample; also, there should be no smearing of the surface.

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

When using X-ray fluorescence spectrometric methods, all phases of surface preparation shall be examined for potential surface contamination effects.

The sample for analysis shall be examined visually after preparation to establish that the surface is free from particulate matter and that there are no defects; the sample shall be re-surfaced or discarded if defects are present. The sample for analysis shall be dry and care shall be taken to protect the prepared surface from contamination.

2.4.5 Preparation of a sample for analysis by remelting

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

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 quantitatively known and do not significantly influence the analysis results. Suitable tests shall be carried out to show that any change in composition is both small in magnitude and reproducible.

The equipment used and the method adopted for remelting shall be designed to prevent or minimize change in composition and to ensure that any change is reproducible. A deoxidant, for example, 0.1 % (m/m) zirconium, should be used during remelting. The method used for calibration of analytical measurement shall take account of any change that does occur.

Not all ferrous metals can be remelted in this manner. Remelting shall not be used as a method of sample preparation for the determination of an element which is subject to a significant and non-reproducible change in composition.

2.5 Safety precautions

2.5.1 Personal protection

Personal protective equipment shall be provided to minimize the risk of injury during sampling and sample preparation methods. Provisions shall include protective clothing, hand protection and face visors resistant to splashes of liquid metal for use during the sampling of liquid metal. Provisions shall also include protective clothing, hand, eye and hearing protection for use during the sampling and sample preparation of solid metal; and respiratory protection for use where necessary.

2.5.2 Machinery

The use of machinery for sampling and sample preparation shall be in accordance with BS 5304:1988. Grinding operations used for surface preparation are specifically covered by the *Abrasive Wheels Regulations* [5], which include a requirement for special training, and by the *Control of Substances Hazardous to Health Regulations* [6].

2.5.3 Hazardous substances

Attention is drawn to the provisions of the *Control of Substances Hazardous to Health Regulations* [6] with respect to the use of solvents for the cleaning and drying of samples and test portions.