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Australian/New Zealand Standard[®]

Methods for the analysis of iron and steel

Part 1: Sampling iron and steel for chemical analysis

AS/NZS 1050.1:1996

This Joint Australian/New Zealand Standard was prepared by Joint Technical Committee CH/10, Analysis of Metals. It was approved on behalf of the Council of Standards Australia on 20 December 1995 and on behalf of the Council of Standards New Zealand on 18 December 1995. It was published on 5 May 1996.

The following interests are represented on Committee CH/10:

Aluminium Development Council, Australia
Australasian Institute of Mining and Metallurgy
Australasian Railway Association
Australian Chamber of Manufactures
Australian Lead Development Association
Bureau of Steel Manufacturers of Australia
Copper Technical Data Centre, Australia
National Association of Testing Authorities, Australia
University of New South Wales

Additional interests participating in preparation of Standard:

Analytical laboratories
Steel manufacturers

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STANDARDS AUSTRALIA/STANDARDS NEW ZEALAND

RECONFIRMATION

OF

AS/NZS 1050.1:1996

Methods for the analysis of iron and steel

Part 1: Sampling iron and steel for chemical analysis

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Technical Committee CH-010 has reviewed the content of this publication and in accordance with Standards Australia procedures for reconfirmation, it has been determined that the publication is still valid and does not require change.

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**Methods for the analysis of iron
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**Part 1: Sampling iron and steel
for chemical analysis**

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PREFACE

This Standard was prepared by the Joint Standards Australia/Standards New Zealand Committee CH/10 on the Analysis of Metals to supersede AS 1213 —1982, *Iron and steel—Methods of sampling*. This revision expands the first edition and includes additional detail of current Australian sampling methodology from molten and solidified steels and iron.

Cognizance was taken of BS 6200: Section 2.1:1993, *Sampling and analysis of iron, steel and other ferrous metals*, as well as the work being undertaken by ISO/TC 17/SC 1 which is currently developing a corresponding international standard.

The term ‘informative’ has been used in this Standard to define the application of the appendix to which it applies. An ‘informative’ appendix is only for information and guidance.

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STANDARDS AUSTRALIA/STANDARDS NEW ZEALAND

Australian/New Zealand Standard Methods for the analysis of iron and steel

Part 1: Sampling iron and steel for chemical analysis

SECTION 1 SCOPE AND GENERAL

1.1 SCOPE This Standard describes methods of sampling and sample preparation for determining the chemical composition of pig iron, cast iron and steel. Procedures are described for use with both liquid and solid metal.

1.2 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

ISO

9147 Pig-irons—Definition and classification

ASTM

E1257 Standard guide for evaluating grinding materials used for surface preparation in spectrochemical analysis

1.3 DEFINITIONS For the purposes of this Standard, the definitions below apply.

1.3.1 Cast product—item of iron or steel which has not been subjected to deformation. Examples include an ingot, a semi-finished product obtained by continuous casting and a shaped casting.

1.3.2 Chemical method of analysis—method for the determination of chemical composition in which the sample is submitted to chemical reaction.

1.3.3 Comminution—to reduce in size by crushing.

1.3.4 Consignment—quantity of metal delivered at one time.

1.3.5 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.

1.3.6 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.

1.3.7 Increment—quantity of metal obtained by sampling at one time from a consignment.

1.3.8 Linishing—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.

1.3.9 Melt—liquid metal from which a sample is removed.

1.3.10 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, e.g. an optical emission spectrometric method or an X-ray fluorescence spectrometric method.

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

1.3.12 Probe sampling—method in which the sample is taken from the melt using a commercially available sampling probe inserted into the melt.

1.3.13 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:

- (a) Sample in the form of a solid mass.
- (b) Sample that has been remelted.
- (c) Sample in the form of chips obtained by machining.
- (d) Sample in the form of fragments obtained by comminution.
- (e) Sample in the form of powder obtained by comminution.

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 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.

1.3.16 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.

1.3.17 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.

1.3.18 Test portion—part of the sample for analysis, or part of the sample taken from the melt, actually analysed. In certain cases, the test portion may be selected from the sample product itself.

NOTES:

- 1 The following types of test portions in the form of a solid mass obtained from a probe sample are distinguished:
 - (a) Test portion in the shape of a small disc, commonly described as a slug, obtained by punching.
 - (b) Test portion in the form of a small appendage, commonly described as a lug.
 - (c) Test portion in the form of a small-diameter rod, commonly described as a pin, obtained by cutting.
- 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.

1.3.19 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.

1.3.20 Wrought product—item of steel which has been subjected to deformation by rolling, drawing, forging or some other method. Examples include bars, billets, plates, strips, tubes and wire.

SECTION 2 REQUIREMENTS FOR SAMPLING AND SAMPLE PREPARATION

2.1 GENERAL This Section 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 clause.

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 Section 6).

2.2 SAMPLING

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 of the 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, 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 some 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 to be taken if re-analysis is required using an alternative method of analysis.

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 analysis method selected. In the case of optical emission and X-ray fluorescence spectrometric methods, the shape and size of the sample will be determined by the dimensions of the sample chamber. The dimensions for samples for analysis given in this Standard should be regarded as indicative 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 of pig iron for analysis 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.

A method of marking shall be used to ensure that the assigned identification remains associated with the sample for analysis.

The unique identification, status and condition of the sample shall be recorded to confirm the identity of the item to which analysis and records refer.

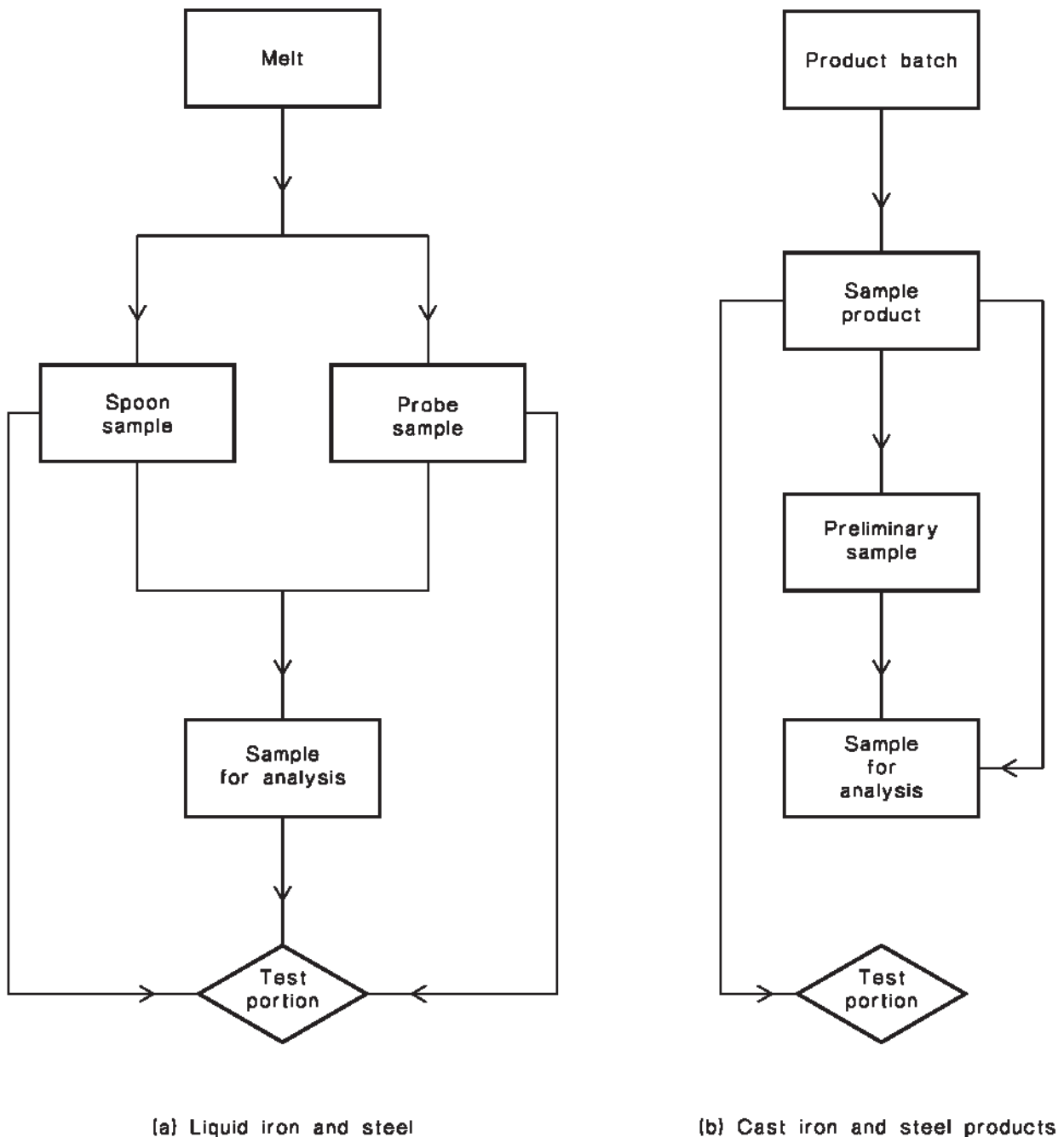


FIGURE 1 SEQUENCE OF SAMPLING AND SAMPLE PREPARATION

2.2.4 Conservation Adequate storage facilities shall be provided to segregate and protect the sample. During and after preparation, it shall be stored in a way that prevents contamination or chemical change.

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

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 can be sampled at various stages for monitoring and controlling the manufacturing process. Samples are taken during the casting of the melt to verify that the chemical composition complies 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 the cast product.

Sampling practices for melts during a particular manufacturing process shall be designed to provide samples in accordance with requirements for sample quality (see Clause 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 suppliers; the main features of the different types of probes are distinguished in Appendices A and B, which include dimensions provided for information only.

2.3.2 Sample from a product The preliminary sample or the sample for analysis can be selected from the sample product from the location indicated in the product specification for the selection of material for mechanical testing.

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

In the case of a forging, the sample for analysis can be selected from the starting material from which the forging has been made, from extensions 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, e.g. due to oxidation, it may be agreed by the supplier and the purchaser to establish the nature and extent of any change in composition and to remove those parts of the sample that have changed. The sample shall then be protected from any further 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, milling, turning or punching. The chips shall not be taken from a part of the sample that has been affected by the heat of a cutting torch.

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

Machining shall be carried out in such a way that the chips are not subject to overheating, as indicated by blueing or blackening. Unavoidable colouration 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 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 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, 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 such as the determination of carbon using a thermal method of analysis, the sample is crushed in a percussion mortar to obtain a sample in the form of fragments with a particle size range of approximately 1 mm to 2 mm.

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

Comminution shall not be used for preparing 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 COMMUNITION.

2.4.4 Sample for analysis in the form of a solid mass

2.4.4.1 Selection 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 specific instruction 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 The surface of the sample for analysis shall be prepared for analysis. 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.

The following four main types of equipment used for surface preparation may be distinguished:

- (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 with a hot sample taken from a melt.
- (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 of the test portion.

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

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 which automatically perform each stage of preparation. Systems for the automatic surface preparation of dual-thickness probe samples (see Appendix A, Paragraph A2.3) 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 36 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 by volatilizing any grinding contaminants. However, particular care is required to avoid surface contamination when using a new abrasive disc. The sample preparation should be uniform for standard and test sample preparation.

When using X-ray fluorescence spectrometric methods, all phases of surface preparation shall be examined for potential surface contamination effects. A suitable method is contained in ASTM E1257.

The sample shall be examined 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 shall be dry and care shall be taken to protect the prepared surface from any future contamination.

2.4.5 Preparation of a sample 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, 30 mm to 40 mm in diameter and 6 mm thick, which is suitable for analysis by a physical method. Some types of remelting equipment incorporate facilities for the centrifugal casting of the disc sample.

Partial losses of some elements may be experienced during the remelting process. It is essential to ensure that any selective volatilization or segregation of elements, or any other change in composition which occurs are quantitatively determined to show that any change in composition is both small in magnitude and reproducible.