

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

Part 3: Methods of analysis —

Section 3.34 Determination of vanadium —

Subsection 3.34.3 Steel and cast iron: flame atomic absorption spectrometric method

Committees responsible for this British Standard

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- BCIRA
- British Forging Industry Association
- 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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National foreword

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

It is identical with ISO 9647:1989 “*Steel and iron — Determination of vanadium content — Flame atomic absorption spectrometric method*”, published by the International Organization for Standardization (ISO).

Cross-references

International Standard	Corresponding British Standard
ISO 1042:1983	BS 1792:1982 <i>Specification for one-mark volumetric flasks</i> (Identical) BS 5497 <i>Precision of test methods</i>
ISO 5725:1986	Part 1:1987 <i>Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory tests</i> (Identical)

The Technical Committee has reviewed the provisions of ISO 377, ISO 385-1 and ISO 648, to which reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard. Related British Standards for ISO 377:1985, ISO 385-1:1984 and ISO 648:1977 are, respectively, BS 1837:1970 “*Methods for the sampling of iron, steel, permanent magnet alloys and ferro-alloys*”, BS 846:1985 “*Specification for burettes*” and BS 1583:1986 “*Specification for one-mark pipettes*”. Appropriate procedures from ISO 377 will be incorporated in BS 6200-2 “*Methods of sampling and sample preparation*”, which will be published in due course and which will supersede BS 1837.

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.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, 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.

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of vanadium in steel and iron.

The method is applicable to vanadium contents between 0,005 % (*m/m*) and 1,0 % (*m/m*), provided that the tungsten content in the test portion is not higher than 10 mg and/or the titanium content is not higher than 5 mg.

2 Normative references

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

ISO 377:1985, *Wrought steel — Selection and preparation of samples and test pieces*.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

3 Principle

Dissolution of a test portion in hydrochloric, nitric and perchloric acids.

Addition of aluminium solution as spectrochemical buffer.

Spraying the solution into a dinitrogen monoxide-acetylene flame.

Spectrometric measurement of the atomic absorption of the 318,4 nm spectral line emitted by a vanadium hollow cathode lamp.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Pure iron, containing less than 0,000 5 % (*m/m*) of vanadium, or of low known vanadium content.

4.2 Hydrochloric acid, ρ about 1,19 g/ml.

4.3 Nitric acid, ρ about 1,40 g/ml.

4.4 Perchloric acid, ρ about 1,67 g/ml.

4.5 Hydrochloric acid, ρ about 1,19 g/ml, diluted 1 + 50.

4.6 Aluminium, solution, 20 g/l.

Dissolve 90 g of aluminium chloride hexahydrate ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$) in about 300 ml of water, add 5 ml of hydrochloric acid (4.2), dilute to 500 ml with water and mix.

4.7 Vanadium, standard solutions.

4.7.1 Stock solution, corresponding to 2 g of V per litre.

4.7.1.1 Preparation using vanadium metal

Weigh, to the nearest 0,001 g, 1,000 g of high purity vanadium [purity > 99,9 % (*m/m*)] and dissolve in 30 ml of aqua regia [mix three volumes of hydrochloric acid (4.2) with one volume of nitric acid (4.3)].

Heat to evaporate to just before dryness and add 20 ml of hydrochloric acid (4.2). Cool and transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 2 mg of V.

4.7.1.2 Preparation using ammonium metavanadate

Dry several grams of ammonium metavanadate (NH_4VO_3) [purity > 99,9 % (*m/m*)] in an air oven at 100 °C to 105 °C (see the note) for at least 1 h and cool to room temperature in a desiccator.

Weigh 2,296 g of the dried product into a 600 ml beaker, add about 400 ml of hot water and gently simmer to dissolve. Cool, transfer to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 2 mg of V.

NOTE A drying temperature over 110 °C will cause decomposition of ammonium metavanadate. Keep strictly to the drying temperature specified.

4.7.2 Standard solution, corresponding to 0,08 g of V per litre.

Transfer 10,0 ml of the stock solution (4.7.1) into a 250 ml one-mark volumetric flask, dilute to the mark with water and mix. Prepare this standard solution immediately before use.

1 ml of this standard solution contains 0,08 mg of V.

5 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042 as appropriate.

Ordinary laboratory apparatus, and