

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

Part 3: Methods of analysis —

Section 3.28 Determination of sulfur —

Subsection 3.28.3 Steel and iron: spectrophotometric method

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee ISE/18, Sampling and analysis of iron and steel, upon which the following bodies were represented:

- BCIRA
- British Iron and Steel Producers' Association
- Ministry of Defence
- TWI
- Coopted members

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National foreword

This Subsection of BS 6200 has been prepared by Technical Committee ISE/18. It is identical with ISO 10701:1994 *Steel and iron — Determination of sulfur content — Methylene blue spectrophotometric method*, published by the International Organization for Standardization (ISO).

Cross-references

Publication referred to	Corresponding British Standard
ISO 1042:1983	BS 1792 <i>Specification for one-mark volumetric flasks</i> (Identical)
ISO 3696:1987	BS 3978:1987 <i>Specification for water for laboratory use</i> (Identical)
ISO 5725:1986	BS 5497 <i>Precision of test methods</i> 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 377-2, 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 385-1:1984 and ISO 648:1977 are, respectively, BS 846:1985 *Specification for burettes* and BS 1583:1986 *Specification for one-mark pipettes*. Appropriate procedures from ISO 377 are incorporated in BS 6200-2:1993 *Methods of sampling and sample preparation*.

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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 methylene blue spectrophotometric method for the determination of sulfur in steel and iron.

The method is applicable to sulfur contents between 0,000 3 % (*m/m*) and 0,010 % (*m/m*).

However, niobium, silicon, tantalum and titanium interfere in the determination of sulfur.

Depending on the concentration of the interfering elements, the application ranges and test portion masses given in Table 1 apply.

Table 1

Maximum allowable content of the interfering elements % (<i>m/m</i>)				Test portion g	Application ranges Δw_s % (<i>m/m</i>)
Nb	Si	Ta	Ti		
0,5	1,0	0,3	1,0	1,0	0,000 3 to 0,001 0
1,0	2,0	0,6	2,0	0,50	0,001 0 to 0,010

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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 377-2:1989, *Selection and preparation of samples and test pieces of wrought steels — Part 2: Samples for the determination of the chemical composition*.

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 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

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 a mixture of hydrochloric and nitric acids.

Evaporation with perchloric acid until white fumes appear to remove hydrochloric and nitric acids.

Dissolution of the salts in hydrochloric acid.

Evolution of hydrogen sulfide by reducing with a mixture of hydroiodic and hypophosphorous acids in a nitrogen atmosphere, distillation, and absorption into zinc acetate solution.

Formation of methylene blue by reacting with *N,N*-dimethyl-*p*-phenylenediamine and iron(III) solution.

Spectrophotometric measurement at a wavelength of about 665 nm.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade with a very low sulfur content, and only freshly prepared grade 2 water as specified in ISO 3696.

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

4.2 Hydrochloric acid, ρ about 1,19 g/ml, diluted 1 + 15.

4.3 Perchloric acid, ρ about 1,54 g/ml.

4.4 Hydrobromic acid, ρ about 1,48 g/ml.

4.5 Mixture of hydrochloric and nitric acids

Mix one volume of hydrochloric acid (4.1) and one volume of nitric acid, ρ about 1,40 g/ml.

Prepare immediately before use.

4.6 Reducing reagent solution

Transfer 200 ml of hydriodic acid [about 57 % (*m/m*)] and 50 ml of hypophosphorous acid [about 50 % (*m/m*)] into the purifying apparatus (see Figure 1). Purge with nitrogen (4.12) at a flowrate of 100 ml/min for 10 min, to mix the acids and expel air from the system. Switch on the electric heating mantle. Heat to boiling and boil gently for about 120 min at a temperature of about 115 °C in a current of nitrogen. When purification is completed (see 10.3), switch off the electric heating mantle. Then cool the solution and keep it in a brown bottle.

4.7 Absorbing solution

Dissolve 5 g of zinc acetate dihydrate $[(CH_3COO)_2Zn \cdot 2H_2O]$ in 400 ml of water. Add 200 ml of sodium hydroxide solution, 30 g/l, and 70 g of ammonium chloride, and then dilute to 1 000 ml with water.