

British Standard

Sampling and analysis of iron, steel and other ferrous metals

Part 3. Methods of analysis

Section 3.12 Determination of copper

Subsection 3.12.3 Steel and cast iron: flame atomic absorption
spectrometric method

Echantillonnage et analyse du fer, de l'acier et d'autres métaux ferreux

Partie 3. Méthodes d'analyse

Section 3.12 Dosage du cuivre

Sous-section 3.12.3 Aciers et fontes: méthode par spectrométrie d'absorption atomique
dans la flamme

Probenahme und Analyse von Eisen, Stahl und anderen Eisenwerkstoffen

Teil 3. Analysenverfahren

Abschnitt 3.12 Kupferbestimmung

Unterabschnitt 3.12.3 Stahl und Gußeisen: Flammenatomabsorptionsspektrometrie

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National foreword

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

It is identical with ISO 4943 'Steel and cast iron — Determination of copper content — Flame atomic absorption spectrometric method' published by the International Organization for Standardization (ISO).

Terminology and conventions. The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'British Standard'.

Cross-reference. The Technical Committee has reviewed the provisions of ISO/R 377, to which reference is made in clause 6, and has decided that they are acceptable for use in conjunction with this standard.

A related standard to ISO/R 377 is BS 1837 'Methods for the sampling of iron, steel, permanent magnet alloys and ferro-alloys'. Appropriate procedures from ISO/R 377 will be incorporated in BS 6200 : Part 2 'Methods of sampling and sample preparation', which will be published in due course and which will supersede BS 1837.

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

1 Scope and field of application

This International Standard specifies a method for the determination of copper in steel and cast iron by means of flame atomic absorption spectrometry.

The method is applicable to copper contents in the range 0,004 to 0,5 % (m/m).

2 Reference

ISO/R 377, *Selection and preparation of samples and test pieces for wrought steel*.

3 Principle

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

Spraying of the solution into an air-acetylene flame. Spectrometric measurement of the atomic absorption of the 324,7 nm spectral line emitted by a copper hollow cathode lamp.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and having a very low copper content, and only distilled water or water of equivalent purity.

If possible, use only freshly prepared distilled or de-ionized water.

4.1 Iron of high purity,
copper content < 0,000 5 % (m/m).

4.2 Hydrochloric acid-nitric acid mixture.

Mix three parts by volume of hydrochloric acid (ρ about 1,19 g/ml), one part by volume of nitric acid (ρ about 1,40 g/ml), and two parts by volume of water.

Prepare this mixture immediately before use.

4.3 Hydrochloric acid-nitric acid-perchloric acid mixture.

Mix 20 ml of hydrochloric acid (ρ about 1,19 g/ml) with 55 ml of nitric acid (ρ about 1,40 g/ml) and 75 ml of perchloric acid (ρ about 1,54 g/ml).

NOTE — Perchloric acid (ρ about 1,67 g/ml) may also be used. 100 ml of perchloric acid (ρ about 1,54 g/ml) is equivalent to 79 ml of perchloric acid (ρ about 1,67 g/ml).

4.4 Copper, standard solution.

4.4.1 Stock solution, corresponding to 1 g of Cu per litre.

Weigh, to the nearest 0,000 1 g, 1,000 0 g of high purity copper [$> 99,95$ % (m/m) Cu].

Transfer to a 400 ml beaker and dissolve in 25 ml of nitric acid (ρ about 1,40 g/ml, diluted 1 + 4). Cover with a watch-glass. When dissolution is complete, evaporate on a water-bath until the onset of crystallization. Dissolve the residue in water, cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

4.4.2 Standard solution, corresponding to 20 mg of Cu per litre.

Transfer 20,0 ml of the stock solution (4.4.1) into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 20 μ g of Cu.

Prepare this standard solution immediately before use.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Atomic absorption spectrometer.

A copper hollow cathode lamp; supplies of air and acetylene sufficiently pure to give a steady clear fuel-lean flame, free from water and oil, and free from copper.