International Standard 4943

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Steel and cast iron — Determination of copper content — Flame atomic absorption spectrometric method

Aciers et fontes — Dosage du cuivre — Méthode par spectrométrie d'absorption atomique dans la flamme

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Foreword

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Steel and cast iron — Determination of copper content — Flame atomic absorption spectrometric method

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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 µg 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.