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# International Standard



# 4946

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## Steel and cast iron — Determination of copper content — 2,2'-Diquinolyl spectrophotometric method

*Aciers et fontes — Dosage du cuivre — Méthode spectrophotométrique au 2,2'-biquinolyle*

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## Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4946 was prepared by Technical Committee ISO/TC 17, *Steel*.

# Steel and cast iron — Determination of copper content — 2,2'-Diquinolyl spectrophotometric method

## 1 Scope and field of application

This International Standard specifies a 2,2'-diquinolyl spectrophotometric method for the determination of copper in steel and cast iron.

The method is applicable to copper contents between 0,02 and 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 with appropriate acids.

Fuming with perchloric acid to remove hydrochloric and nitric acids and dehydrate silicic acid.

Reduction of copper(II) to copper(I) in hydrochloric acid solution by means of ascorbic acid. Formation of a coloured compound of copper(I) with 2,2'-diquinolyl.

Spectrophotometric measurement at a wavelength of about 545 nm.

## 4 Reagents

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

**4.1 High-purity iron**, containing 0,001 % (m/m) or less of copper.

**4.2 Hydrochloric acid**,  $\rho$  approximately 1,19 g/ml.

**4.3 Nitric acid**,  $\rho$  approximately 1,40 g/ml.

**4.4 Perchloric acid**,  $\rho$  approximately 1,54 g/ml.

NOTE — Perchloric acid,  $\rho$  approximately 1,67 g/ml, may also be used. 100 ml of perchloric acid,  $\rho$  approximately 1,54 g/ml, is equivalent to 79 ml of perchloric acid,  $\rho$  approximately 1,67 g/ml.

**4.5 Perchloric acid**,  $\rho$  approximately 1,54 g/ml, diluted 1 + 7.

**4.6 Dimethylformamide** (*N,N*-dimethylformamide),  $\rho$  approximately 0,944 g/ml.

**4.7 Ascorbic acid**, 200 g/l solution.

Dissolve 20 g of ascorbic acid in water, dilute to 100 ml and mix.

Prepare this solution immediately before use.

**4.8 2,2'-Diquinolyl**, solution.

Dissolve 0,60 g of 2,2'-diquinolyl (cuproine) in the dimethylformamide (4.6), dilute to 1 litre with the same dimethylformamide and mix.

Keep this solution in a dark-coloured glass receptacle and protected from the light.

**4.9 Copper**, standard solutions.

**4.9.1 Copper**, standard solution, corresponding to 1,0 g of Cu per litre.

Weigh, to the nearest 0,000 1 g, 1,000 0 g of high purity copper and dissolve in a minimum of the nitric acid (4.3).

Heat to boiling to remove nitrous fumes. Cool and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 1 mg of Cu.

**4.9.2 Copper**, standard solution, corresponding to 0,050 g of Cu per litre.

Transfer 50,0 ml of the copper standard solution (4.9.1) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,05 mg of Cu.

## 5 Apparatus

Ordinary laboratory apparatus and

**5.1 Spectrophotometer.**