SVENSK STANDARD
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Stål och gjutjärn – Bestämning av kopparhalt – Spektrofotometrisk metod med 2,2'-Diquinolyl (ISO 4946:2016)

Steel and cast iron – Determination of copper – 2,2'-Biquinoline spectrophotometric method (ISO 4946:2016)

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Europastandarden EN ISO 4946:2016 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN ISO 4946:2016.

Denna standard ersätter SS-EN 24946, utgåva 1 och SS-EN 24946/AC:1991, utgåva 1.

The European Standard EN ISO 4946:2016 has the status of a Swedish Standard. This document contains the official English version of EN ISO 4946:2016.

This standard supersedes the Swedish Standard SS-EN 24946, edition 1 and SS-EN 24946/AC:1991, edition 1.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

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Denna standard är framtagen av kommittén för Kemiska analysmetoder för metaller, SIS/TK 122.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 4946

March 2016

ICS 77.080.01

Supersedes EN 24946:1990

English Version

Steel and cast iron - Determination of copper - 2,2'-Biquinoline spectrophotometric method (ISO 4946:2016)

Aciers et fontes - Détermination du cuivre - Méthode spectrophotométrique au 2,2'-biquinolyle (ISO 4946:2016)

Stahl und Gusseisen - Bestimmung des Kupferanteils - Spektrophotometrisches Verfahren mit 2,2'-Biquinoline (ISO 4946:2016)

This European Standard was approved by CEN on 21 November 2015.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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European foreword

This document (EN ISO 4946:2016) has been prepared by Technical Committee ISO/TC 17 “Steel” in collaboration with Technical Committee ECISS/TC 102 “Methods of chemical analysis for iron and steel” the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2016, and conflicting national standards shall be withdrawn at the latest by September 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 24946:1990.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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Endorsement notice

The text of ISO 4946:2016 has been approved by CEN as EN ISO 4946:2016 without any modification.

Steel and cast iron — Determination of copper — 2,2'-Biquinoline spectrophotometric method

1 Scope

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

The method is applicable to the determination of copper mass fraction in the range of 0,02 % and 5 %.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

3 Principle

Dissolution of a test portion in 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'-biquinoline.

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 grade 2 water as specified in ISO 3696.

4.1 High-purity iron, containing copper 0,001 % (mass fraction) or less.

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

4.3 Nitric acid, ρ approximately 1,40 g/ml.

4.4 Perchloric acid, ρ approximately 1,54 g/ml.

WARNING — Perchloric acid vapour might cause explosions in the presence of ammonia, nitrous fumes or organic material in general.

Perchloric acid, ρ approximately 1,67 g/ml, may also be used. 100 ml of perchloric acid, ρ approximately 1,54 g/ml is equivalent to 79 ml of perchloric acid, ρ approximately 1,67 g/ml.

4.5 Perchloric acid, ρ approximately 1,54 g/ml, diluted 1 + 7.

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

WARNING — Dimethylformamide is a hazardous substance and can cause birth defects. It should be handled with safety gloves in a fume hood.

4.7 Ascorbic acid, 200 g/l solution.

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

Prepare this solution immediately before use.

4.8 2,2'-Biquinoline solution.

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

Keep this solution in a dark-coloured glass flask and protect it from the light.

4.9 Copper standard solution, 1 g/l.

Weigh, to the nearest 0,001 g, 1,000 g of high purity copper and dissolve in the minimum of 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 with water and mix.

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

4.10 Copper standard solution, 0,05 g/l.

Transfer 25,0 ml of the copper standard solution (4.9) to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

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

5 Apparatus

All volumetric glassware shall be Class A, in accordance with ISO 648 and ISO 1042.

Ordinary laboratory apparatus and the following shall be used.

Spectrophotometer, suitable for measuring the absorbance of the solution at a wavelength of 545 nm with cells of 2 cm or 4 cm optical path length.

6 Sampling

Carry out sampling in accordance with ISO 14284 or appropriate national standards for steel and cast iron.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,001 g, approximately 0,5 g of the test sample.

7.2 Blank test

In parallel with the determination and following the same procedure, carry out two blank tests using the same quantities of all the reagents but using, to the nearest 0,001 g, approximately 0,5 g of pure iron (4.1) instead of test portion.

7.3 Determination

7.3.1 Preparation of the test solution

Introduce the test portion (7.1) into a 250 ml beaker. Add 10 ml of hydrochloric acid (4.2) and 5 ml of nitric acid (4.3). Cover the beaker with a watch-glass and heat until acids action ceases.

NOTE For samples of high chromium content, first dissolve in hydrochloric acid (4.2) and then, when all effervescence has ceased, oxidize by adding nitric acid (4.3), drop by drop.

Add 10 ml of perchloric acid (4.4) and evaporate to fuming. Continue fuming for 3 min.

Cool, dissolve the salts with 20 ml of water, transfer the solution quantitatively to a one-mark volumetric flask of suitable capacity (see Table 1), dilute to the mark with water and mix.

Filter by decantation through a dry filter to remove any residue or precipitate, e.g. graphite, silica, tungstic acid. Collect the filtrate in a dry beaker, discarding the first fractions of the filtrate.

7.3.2 Colour development

Take an aliquot portion, according to the expected copper content, as indicated in Table 1.

Table 1 — Volume of test solution and aliquot portion

Copper content % (mass fraction)	Volume of test solution ml	Volume of aliquot portion ml
0,02 to 0,3	100	10
0,3 to 0,6	100	5
0,6 to 1,5	250	5
1,5 to 5,0	500	5

Transfer the selected aliquot portion to a 50 ml one-mark volumetric flask. If the aliquot portion is 5 ml, add 5 ml of perchloric acid (4.5).

Add, in the following order, shaking after each addition:

— 5 ml of ascorbic acid solution (4.7);

— 25 ml of 2,2'-biquinoline solution (4.8).

Dilute to the mark with water and mix. Cool for 5 min in a water-bath at about 20 °C.

Finally, readjust the volume and mix again.