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Handläggande organ

SIS - MATERIALNORMCENTRALEN

SVENSK STANDARD SS-EN 10 179

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Kemiska analysmetoder för järn och stål — Bestämning av kvävehalt (spårämneshalter) i stål — Spektrofotometrisk metod

Orientering

Denna standard utgörs av den engelska versionen av den europeiska standarden EN 10 179:1989. Standarden har en direkt motsvarighet i den internationella standarden ISO 4945-1977, Steel – Determination of nitrogen content – Spectrophotometric method. ISO 4945 omfattar halter på 0,002–0,050 %.

Chemical analysis of ferrous materials — Determination of nitrogen (trace amounts) in steel — spectrophotometric method

Introduction

This Swedish standard consists of the English version of the European standard EN 10 179:1989. The corresponding ISO standard is ISO 4945-1977, Steel – Determination of nitrogen content – Spectrophotometric method. The application range for ISO 4945 is 0,002–0,050%.

UDK 543.42:062:546.17:669.14

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English version

Chemical analysis of ferrous materials Determination of nitrogen (trace amounts) in steels Spectrophotometric method

Analyse chimique des matériaux sidérurgiques — Dosage du l'azote (à l'état de traces) dans les aciers Méthode spectrophotométrique

Chemische Analyse von Eisen- und Stahlwerkstoffen — Bestimmung von Stickstoff (Spuren-Gehalte) in Stahl — Photometrisches Verfahren

This European Standard was accepted by CEN on 1989-01-15. CEN members are bound to comply with the requirements of the CEN/CENELEC Rules 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 Central Secretariat 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 CEN Central Secretariat has the same status as the official versions.

CEN members are the national standards organizations of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxemburg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN
European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: Rue Bréderode 2, B-1000 Brussels

Brief History

This European Standard takes over the content of EURONORM 179-85 "Chemical analysis of ferrous materials – Determination of nitrogen (trace amounts) in steels – Spectrophotometric method", prepared by ECISS/TC 20 "Methods of chemical analysis"; the Secretariat of which is allocated to the Dansk Standardiseringsrad (DS).

It has been submitted to the CEN Formal Vote following the decision of the Coordinating Commission (COCOR) of the European Committee for Iron and Steel Standardization on 1987-11-24/25.

It has been adopted and ratified by CEN BT on 1988-11-05.

According to the Common CEN/CENELEC Rules, following countries are bound to implement this European Standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxemburg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

Note in clauses 1 and 9 EURONORM shall read EUROPEAN STANDARD.

Chemical analysis of ferrous materials
Determination of nitrogen (trace amounts) in steels
Spectrophotometric method

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1. SCOPE AND FIELD OF APPLICATION

This EURONORM specifies a method for the spectrophotometric determination of nitrogen in steels.

The method is primarily intended for the determination of total nitrogen in very low nitrogen non-alloy steels. It may be used, however, for any low nitrogen ferrous alloy that is soluble in hydrochloric acid provided that the acid-resistant form of silicon nitride is not present. This highly resistant nitride has been found only in samples of silicon steels manufactured without aluminium addition and then only in sheet material.

The method is applicable to nitrogen contents from 0.0005 to 0.005% (m/m).

NOTE – The method has also been successfully applied to a pure iron with a nitrogen content of 0.0038% (m/m) – see *Annex*.

2. REFERENCE

EURONORM 18 – Selection and preparation of samples and test pieces for steel and iron and steel products.

3. PRINCIPLE

Dissolution of the test portion with hydrochloric acid and separation of the acid-insoluble residue by means of a centrifuge.

Decomposition of the acid-insoluble residue by intense fuming with sulphuric acid and addition of the extract to the solution of the test portion containing the acid-soluble nitrogen.

Recovery of the total nitrogen as ammonia by steam distillation over sodium hydroxide.

Spectrophotometric measurement of the coloured complex produced by the indophenol blue reaction.

4. REAGENTS

During the analysis use only reagents of recognized analytical reagent quality and which are known to give a very low nitrogen blank. The same batch of each reagent shall be used for every test and blank determination in a given series of tests.

All references to 'water' relate to ammonia-free water. Ammonia-free water shall be prepared by passing distilled water through the cation exchange column (5.1). It is essential that the resin column shall be acid washed before use to ensure its conversion to the hydrogen form (H^+). This is most conveniently done by passing 2 litres of hydrochloric acid ρ 1.19 g/ml approximately, diluted 1 + 9 (V/V), through the column, then washing with water until freed from acid.

4.1 Iron of high purity or steel of very low nitrogen content

NOTE — High purity iron powder or very low nitrogen mild steel (< 0.001% nitrogen) may be used for this purpose. It should be washed in the same way as the sample.

4.2 Hydrochloric acid, ρ 1.19 g/ml approximately (12 mol/l approximately)

4.3 Sulphuric acid, ρ 1.84 g/ml approximately, (18 mol/l approximately)

Sulphuric acid supplies shall be tested individually and selected for a low content of combined nitrogen in any form (less than 0.5 ppm). Nitrogen as ammonia will usually be the major source of contamination and this may be tested by the normal methods, but nitrates may also be present and may be detected by the following method.

Add 6 ml of the sulphuric acid to 2 ml of water and cool to 60 °C. Add one drop of hydrochloric acid (4.2) and one drop of diphenylamine reagent (4.8). No blue colour should develop. Less than 0.1 ppm can be detected by this test.

4.4 Sulphuric acid, ρ 1.84 g/ml approximately, diluted 1 + 4 (V/V) (3.6 mol/l approximately)

4.5 Sodium hydroxide, 400 g/l solution

Dissolve 400 g of sodium hydroxide in water, dilute to 1 000 ml and mix. This solution should be prepared in a polyethylene beaker (water-cooled if necessary) and stored in a polyethylene bottle.

4.6 Barium chloride, 100 g/l solution

Dissolve 100 g of barium chloride ($BaCl_2.2H_20$) in water, dilute to 1 000 ml and mix.

4.7 Chromic-sulphuric acid*

Dissolve 2 g of chromium trioxide in 50 ml of water, and add slowly 100 ml of sulphuric acid (4.3) with constant stirring.

4.8 Diphenylamine reagent*

To 25 ml of water, whilst stirring, cautiously add 75 ml of sulphuric acid (4.3) then add 0.1 g diphenylamine, stir until dissolved and cool.

4.9 Phenol, 50 g/l solution

Dissolve 50 g of phenol in water, transfer to a 1 000 ml volumetric flask, dilute to the mark with water and mix. (Store out of direct sunlight in an amber coloured bottle).

4.10 Sodium hydroxide–sodium hypochlorite solution

4.10.1 Determination of available chlorine in commercial sodium hypochlorite solution

By means of a burette, transfer 10 ml of the sodium hypochlorite solution to a 250 ml volumetric flask, dilute to the mark with water and mix.

Transfer, by means of a safety pipette, 10 ml of this solution to a 100 ml conical beaker, add 2 g of potassium iodide and 10 ml of glacial acetic acid, ρ 1.048-1.050 g/ml approximately. Titrate the liberated iodine using sodium thiosulphate (4.12) until the colour is almost discharged. Add 2 ml of starch solution (4.13), and continue the titration until the blue colour is discharged.

Available chlorine per cent (w/v) = 0.886 . V

where V is the volume in millilitres of sodium thiosulphate (4.12) used in the titration.

4.10.2 Calculation

The volume of sodium hypochlorite solution required for the preparation of 1 l of sodium hydroxide-sodium hypochlorite solution (4.10) is

$$\frac{2.1 \,.\, 100 \,.\, 70.91}{0.886 \,.\, V. \, 74.44} = \frac{225.7}{V} \text{ ml}$$

This volume is equivalent to 2.1 g of available chlorine.

4.10.3 Preparation of solution 4.10

Dissolve 25 g of sodium hydroxide in about 400 ml of water. Add the calculated volume of sodium hypochlorite solution (4.10.2) from a burette, dilute to 1 000 ml and mix. (Store out of direct sunlight in an amber bottle).

4.11 Sodium pentacyanonitrosylferrate (sodium nitroprusside) (Na_2 [$Fe(CN)_5NO$] $2H_2O$)

Dissolve 0.1 g of sodium pentacyanonitrosylferrate in water, dilute to 100 ml and mix. Prepare freshly each day, store in an amber coloured bottle but do not use until at least 60 min. after preparation.

4.12 Sodium thiosulphate, 0.05 mol solution*

Dissolve 24.821 g of sodium thiosulphate ($Na_2S_2O_3.5H_2O$) in water containing 0.1 g of sodium carbonate. Transfer to a 1 000 ml volumetric flask, dilute to the mark with water and mix.

4.13 Starch, 5 g/l solution*

Make a suspension of 0.5 g of starch in 10 ml of water. Add to 90 ml of boiling water. Cool, dilute to 100 ml and mix.

4.14 Ammonium chloride reference solution corresponding to 1 μg of nitrogen per millilitre

Dissolve 0.382 g of ammonium chloride (previously dried to constant weight at 105 °C) in water, transfer to a 1 000 ml