SLOVENSKI STANDARD
SIST EN 10071:1996

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Kemijska analiza železnih materialov - Ugotavljanje mangana v jeklih in železu - Elektrometrično-titracijska metoda

Chemical analysis of ferrous materials - Determination of manganese in steels and irons - Electrometric titration method

Chemische Analyse von Eisen- und Stahlwerkstoffen - Ermittlung des Mangangehalts von Stahl und Eisen - Elektrometrisches Titrierverfahren

Analyse chimique des matériaux sidérurgiques - Dosage du manganese dans les aciers et les fontes - Méthode par titrage électrométrique

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English version

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Determination of manganese in steels and irons
Electrometric titration method

Analyse chimique des matériaux sidérurgiques - Dosage du manganèse dans les aciers et les fontes
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Chemische Analyse von Eisen und Stahlwerkstoffen - Ermittlung des Mangangehalts von Stahl und Eisen - Elektrometrisches Titrierverfahren

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CEN

European Committee for Standardization
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Central Secretariat : Rue Bréderode 2, B-1000 Brussels

Brief History

This European Standard takes over the content of EURONORM 71-83 "Chemical analysis of ferrous materials - Determination of manganese in steels and irons - Electrometric titration 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 manganese in steels and irons
Electrometric titration method

CONTENTS

1 SCOPE AND FIELD OF APPLICATION

This EURONORM specifies an electrometric titration method for the determination of manganese in steels and irons.

The method is applicable to non-alloy or alloy steels and to irons with manganese contents greater than or equal to 0.5% (*m/m*).

2 REFERENCE

EURONORM 18-~~79~~ Selection and preparation of samples and test pieces for steel and iron and steel products.

3 PRINCIPLE

Dissolution of the test portion by a suitable acid and partial neutralization of the acid by sodium hydrogen carbonate.

Precipitation by zinc oxide of the cations which may interfere with the electrometric determination.

Titration of the Mn (II) by means of a potassium permanganate solution, in the presence of pyrophosphoric irons at a pH value of about 6.5.

The oxidation-reduction (oxidation of Mn (II) into Mn (III)) reaction is followed by an electrometric measurement.

4 REAGENTS

Only reagents of recognized analytical quality shall be used throughout the analysis.

4.1 Water free from reducing materials

Bring water acidified with 6 ml/l sulphuric acid (ρ approximately 1.84 g/ml) to the boil; add a few crystals of sodium periodate and maintain at boiling point for 10 minutes.

4.2 Zinc oxide, free from materials which could reduce the permanganate.

4.3 Nitric acid, ρ 1.40 g/ml approximately, diluted 1 + 1 (*V/V*) (7 mol/l approximately), with water (4.1).

4.4 Hydrochloric acid, ρ 1.19 g/ml approximately, diluted 1 + 1 (*V/V*) (6 mol/l approximately) with water (4.1).

4.5 Aqua regia

Mix three volumes of hydrochloric acid, ρ 1.19 g/ml approximately, with one volume of nitric acid, ρ 1.40 g/ml approximately.

4.6 Sodium hydrogen carbonate, saturated solution at the ambient temperature (about 100 g/l of $NaHCO_3$) in water (4.1).

4.7 Sodium pyrophosphate, 120 g/l solution

Dissolve in hot water (4.1) (the temperature shall not exceed about 70°C) 120 g of hydrated sodium pyrophosphate ($Na_4P_2O_7 \cdot 10\ H_2O$) and, after cooling, make up the volume to 1 000 ml with water (4.1).

4.8 Manganese, reference solution, 0.5000 g/l of Mn

Weigh to an accuracy of 0.1 mg, 1.4385 g of potassium permanganate ($KMnO_4$) and place in a conical flask of suitable capacity. Dissolve with 40 ml of hydrochloric acid (4.4) and evaporate to dryness. Then take up with 5 ml of hydrochloric acid (4.4) and 100 ml of water (4.1).

Transfer the solution quantitatively to a 1 000 ml volumetric flask, dilute with water (4.1) and after cooling, make up the volume with water (4.1). Mix.

1 ml of this reference solution contains 0.5000 mg of manganese.

4.9 Potassium permanganate, titred solution about 0.0022 mol/l

4.9.1 Preparation of the solution

Dissolve approximately 0.36 g of potassium permanganate in 1 000 ml of water (4.1), allow to stand out of the light for at least two weeks. Then, filter through sintered glass and keep the solution in a dark glass bottle.

4.9.2 Calibration of the solution

Place 350 ml of the sodium pyrophosphate solution (4.7) in a 600 ml beaker. Add 30 ml, measured by a burette or pipette, of the reference solution of manganese (4.8).

Mix carefully, and add nitric acid (4.3), in small amounts, to bring the pH to about 6.5 as determined with a pH meter or indicator paper (4.10).

Titrate with the permanganate solution (4.9) following the progress of the reaction by an electrometric measurement.

4.9.3 Calculation of the titre of the solution

The manganese (Mn) content (B) of the titrated solution expressed in mg/ml is given by the formula:

$$B = \frac{0.5000 \times 30}{\text{ml of the permanganate solution (4.9) used for the titration}}$$

4.10 pH indicator paper, range 5 to 7.

5 APPARATUS

The apparatus consists of ordinary laboratory equipment, and equipment suitable for electrometric titration.

6 SAMPLING

Sampling shall be carried out in accordance with EURONORM 18-79

7 PROCEDURE

7.1 Test portion

Weigh to the nearest 0.001 g, 1.25 g (m) of the sample.

7.2 Determination

7.2.1 Dissolution of the test portion

7.2.1.1 Non-alloy steels and irons

Place the test portion (7.1) in a 500 ml conical flask and dissolve with 30 ml of nitric acid (4.3).

7.2.1.2 Alloy steels

Place the test portion (7.1) in a 500 ml conical flask and attack with 20 ml (or a larger quantity if necessary) of hydrochloric acid (4.4). Oxidize with 10 ml of nitric acid (4.3) or with 15 ml of aqua regia (4.5).

7.2.2 Preparation of the test portion

Boil the solution from 7.2.1 in order to eliminate the nitrous fumes. Dilute to about 80 ml with water (4.1) and add slowly, whilst stirring, 80 ml of sodium hydrogen carbonate solution (4.6).

Note
The addition of the sodium hydrogen carbonate serves to neutralize part of the attack acid, with the object of reducing to a minimum the quantity of zinc which passes into solution during the precipitation of the hydroxides by the zinc oxide.

Before adding the zinc oxide, the solution shall be completely limpid. The precipitates which may be formed during the addition of the hydrogen carbonate lead to a loss of manganese. Therefore, if the solution is not completely limpid, dissolve the precipitates by adding a small quantity of nitric acid (4.3), boil to remove the nitrous fumes and add the zinc oxide as described below.

Bring to the boil once more, adding about 5 g of zinc oxide (4.2) whilst stirring vigorously. Transfer the solution and the precipitates quantitatively to a 250 ml volumetric flask, cool, dilute to the mark with water (4.1) and mix. Filter the solution through a dry, fluted filter paper, collecting the filtrate in a dry container, after having removed the first fractions of the filtrate (a few millilitres).

7.2.3 Titration

Pour 350 ml of the pyrophosphate solution (4.7) into a 600 ml beaker.

Add an aliquot portion of the filtrate according to the estimated manganese content, as indicated in the following table, whilst all the time stirring vigorously by means of a stirrer to prevent the precipitation of zinc phosphate.

Add nitric acid (4.3) to bring the pH of the solution to approximately 6.5 as determined with a pH meter or the indicator paper (4.10).

Mn content % (*m*/*m*)	Aliquot portion to be taken	
	Test portion (7.2.2) ml	Ratio *D*
0.5 ⩽ Mn ⩽ 4	100.0	2.5
4 < Mn ⩽ 8	50.0	5
8 < Mn	20.0	12.5

Titrate with the titred permanganate solution (4.9) following the progress of the titration by an electrometric measurement (volume *V* ml).

Note

In the presence of an excess of pyrophosphate, the pH of the solution after the addition of the aliquot portion of the test solution is greater than 6.5. The correction of the pH value may therefore be carried out with an acid solution, thus avoiding the formation of precipitates which are difficult to redissolve, which tends to happen when the pH value has to be increased by the addition of an alkaline solution.

8 EXPRESSION OF RESULTS

The percentage by mass of manganese (Mn) is given by the expression:

$$\text{Mn (\%)} = \frac{V \times B \times D}{10 \times m}$$

where

V is the volume, in ml, of the titred permanganate solution (4.9) used for the titration;

B is the manganese content, in mg/ml, of the titrated solution (4.9);

D is the ratio between the volume of the test solution and the aliquot portion taken for titration (7.2.3);

m is the mass, in g, of the test portion (7.1).

9 TEST REPORT

The test report shall contain the following particulars:

(a) the method of analysis used by reference to this EURONORM;

(b) the results obtained, as well as the form in which they are expressed;

(c) any particular details which may have been noted during the determination;

(d) any operations not specified in this EURONORM or any optional operations, which could have had an influence on the result.