SLOVENSKI STANDARD
SIST EN 24159:1997

01-december-1997

Feromangan in ferosilikomangan - Določevanje mangana - Potenciometrična metoda (ISO 4159:1978)

Ferromanganese and ferrosilicomanganese - Determination of manganese content - Potentiometric method (ISO 4159:1978, ed. 1)

Bestimmung des Mangangehaltes von Ferromangan und Ferrosilicomangan - Potentiometrisches Verfahren (ISO 4159:1978, Ausg.1)

Ferromanganese et ferro-silico-manganese - Dosage du manganese - Méthode potentiométrique (ISO 4159:1978, éd. 1)

Ta slovenski standard je istoveten z: **EN 24159:1989**

ICS:

77.100	Železove zlitine	Ferroalloys

SIST EN 24159:1997 **en**

EUROPEAN STANDARD EN 24 159

NORME EUROPEENNE

EUROPAISCHE NORM May 1989

UDC 669.15-198:543.257.1:546.711

Key words: Iron- and steel products, Ferroalloys, Ferromanganese, Ferrosilicon, Silico-manganese, Chemical analysis, Determination of content, Manganese, Potentiometric method

English version

Ferromanganese and ferrosilicomanganese. Determination of manganese content. Potentiometric method (ISO 4159, 1st edition, 1978-12-15)

Ferromanganèse et ferro-silico-manganèse. Dosage du manganèse. Méthode potentiométrique (ISO 4159, 1ère édition, 1978-12-15)

Bestimmung des Mangangehaltes von Ferromangan und Ferrosilicomangan. Potentiometrisches Verfahren (ISO 4159, 1. Ausgabe, 1978-12-15)

This European Standard was accepted by CEN on 1989-05-15 and is identical to the ISO standard as referred to.
CEN members are bound to comply with the requirements of the CEN/CENELEC Common 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 may be 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

On the proposal of the Technical Committee ECISS/TC 20 "Methods of chemical analysis" the Coordinating Commission (COCOR) of the European Committee for Iron and Steel Standardization (ECISS) decided on 1987-11-24/25 to submit the International Standard

ISO 4159 - 1978 Ferromanganese and ferrosilicomanganese - Determination of manganese content - Potentiometric method (ISO 4159, 1st edition, 1978-12-15).

to the Formal Vote.

The standard was adopted by CEN on 1988-11-26.

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

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

Statement

The text of the International Standard ISO 4159, edition 1, 1978-12-15 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD 4159

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Ferromanganese and ferrosilicomanganese — Determination of manganese content — Potentiometric method

Ferro-manganèse et ferro-silico-manganèse — Dosage du manganèse — Méthode potentiométrique

First edition — 1978-12-15

UDC 669.15-198 : 543.257.1 : 546.711

Ref. No. ISO 4159-1978 (E)

Descriptors : ferroalloys, ferromanganese, chemical analysis, determination of content, manganese, potentiometric analysis.

Price based on 3 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

International Standard ISO 4159 was developed by Technical Committee ISO/TC 132, *Ferroalloys*, and was circulated to the member bodies in October 1977.

It has been approved by the member bodies of the following countries :

Australia	Iran	South Africa, Rep. of
Austria	Italy	Spain
Bulgaria	Japan	Sweden
Canada	Korea, Rep. of	Turkey
Czechoslovakia	Mexico	United Kingdom
France	Norway	U.S.A.
Germany, F.R.	Philippines	U.S.S.R.
India	Romania	Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

Poland

Printed in Switzerland

Ferromanganese and ferrosilicomanganese — Determination of manganese content — Potentiometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a potentiometric method for the determination of the manganese content of ferromanganese and ferrosilicomanganese.

The method is applicable to alloys containing from 55 to 95 % (*m/m*) of manganese.

2 REFERENCE

ISO 3713, *Ferroalloys — Sampling and preparation of samples — General rules.* [1]

3 PRINCIPLE

Dissolution of a test portion with hydrochloric, hydrofluoric and perchloric acids.

Potentiometric determination (see note) of manganese with potassium permanganate in a pyrophosphoric medium at a controlled pH, according to the reaction :

$$4\,Mn^{2+} + MnO_4^- + 8\,H^+ + 15\,(P_2O_7H_2)^{2-} \rightarrow 5\,Mn(P_2O_7H_2)_3^{3-} + 4\,H_2O$$

NOTE — The method may be completed by any other electrometric method : amperometric titration, dead-stop, etc.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity, except where otherwise specified.

4.1 Perchloric acid[2], ρ 1,61 g/ml.

4.2 Hydrofluoric acid, ρ 1,14 g/ml.

4.3 Hydrochloric acid, ρ 1,19 g/ml.

4.4 Hydrochloric acid, ρ 1,19 g/ml, diluted 1 + 2.

4.5 Hydrochloric acid, ρ 1,19 g/ml, diluted 1 + 4.

4.6 Sodium pyrophosphate, saturated solution.

Dissolve 145 g of sodium pyrophosphate ($P_2O_7Na_4.10H_2O$) in a 2 litre conical flask, with about 1 litre of hot water; heat without exceeding, even locally, the temperature of 60 °C, until the salt is completely dissolved. Cool.

Prepare this solution at the time of use.

4.7 Potassium permanganate, recrystallized.

Place 50 g of pure analytical grade potassium permanganate in the conical flask (5.1), and dissolve it in 200 ml of warm distilled water (70 to 80 °C).

Fit the reflux condenser (5.2) to the flask and boil the solution for 20 min. Filter the warm solution quickly under vacuum through a sintered glass funnel (5.3).

Cool the filtrate in iced water, stirring vigorously, and allow the fine, crystalline precipitate to settle for 10 min.

Decant the solution; then, using a glass spatula, transfer the crystalline mass into a funnel with a filter plate, porosity 4, and place under suction for a few minutes to remove most of the mother liquor.

Dissolve the crystalline mass in 160 ml of distilled water (deionized water is not permitted for this phase), and repeat the recrystallization.

After filtering and placing under suction for approximately 5 min, transfer the crystalline mass onto a 150 mm diameter watch glass using a glass spatula, and dry it in air, protected from light and dust. When the crystalline mass no longer agglomerates when crushed with the spatula, dry it at 110 °C for 2 h, then transfer it to a weighing bottle fitted with a ground glass stopper.

Store in the dark.

This salt contains 34,76 % (*m/m*) of Mn.

1) At present at the stage of draft.

2) Attention is drawn to the hazards associated with perchloric acid when heated to fuming.

4.8 Potassium permanganate, about 0,1 N standard volumetric solution.

4.8.1 *Preparation*

Dissolve 3,20 g of potassium permanganate in 1 000 ml of water. Allow to stand for 6 days. Filter through glass wool or a sintered glass filter, then transfer to a brown glass bottle. Mix.

4.8.2 *Standardization*

Place about 2,5 g of the recrystallized potassium permanganate (4.7), weighed to the nearest 0,000 2 g, in a 250 ml conical flask, and follow exactly the procedure specified in 7.3. It is recommended that this standardization be carried out in parallel with the determination.

The manganese equivalent T of the potassium permanganate solution (4.8), expressed in grams of manganese corresponding to 1 ml of solution, is given by the formula

$$T = \frac{m_1 \times 34{,}76}{100\,(V_1 - V_0)} \times \frac{50}{250} = \frac{0{,}069\,52\,m_1}{V_1 - V_0}$$

where

m_1 is the mass, in grams, of the crystallized potassium permanganate (4.7) used;

V_0 is the volume, in millilitres, of the potassium permanganate solution (4.8) used for the blank test;

V_1 is the volume, in millilitres, of the potassium permanganate solution (4.8) used for the standardization.

5 APPARATUS

Usual laboratory equipment, and in particular :

5.1 Erlenmeyer conical flask with ground neck.

5.2 Reflux condenser with ground glass joint, to fit the flask (5.1).

5.3 Filter crucibles (porosity 4) or **filter funnels,** capacity 40 ml.

5.4 Volumetric flask, capacity 250 ml.

5.5 Beaker, capacity 600 ml.

5.6 Conical flask, capacity 250 ml.

5.7 Dish, capacity 250 ml, or beaker, capacity 400 ml, of polytetrafluoroethylene.

5.8 Magnetic stirrer.

5.9 pH meter, fitted with glass and calomel electrodes.

5.10 Potentiometer, fitted with platinum and calomel electrodes.

6 SAMPLE

Use powder which will pass through a sieve with a mesh size of 160 µm, prepared in accordance with ISO 3713.

7 PROCEDURE

7.1 Test portion

Take a test portion of 1 ± 0,000 2 g.

7.2 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same reagents.

7.3 Determination

7.3.1 Attack the test portion as specified in 7.3.1.1 or 7.3.1.2, as appropriate.

7.3.1.1 *Ferromanganese*

Transfer the test portion (7.1) to the conical flask (5.6) and attack it with 20 ml of the hydrochloric acid (4.3) and 0,2 ml of the hydrofluoric acid (4.2), then add 10 ml of the perchloric acid (4.1).[1)]

Proceed as specified in 7.3.2.

7.3.1.2 *Ferrosilicomanganese*

Transfer the test portion (7.1) to a dish or beaker (5.7). Attack it with 10 ml of the hydrochloric acid (4.3), add 10 ml of the perchloric acid (4.1) then, slowly, about 20 ml of the hydrofluoric acid (4.2).[1)]

Proceed as specified in 7.3.2.

1) Oxidize the solution with nitric acid prior to adding the perchloric acid.