International Standard 629

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Steel and cast iron — Determination of manganese content — Spectrophotometric method

Aciers et fontes — Dosage du manganèse — Méthode spectrophotométrique

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Foreword

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International Standard ISO 629 was developed by Technical Committee ISO/TC 17, *Steel*.

It was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 629-1967, which had been approved by the member bodies of the following countries :

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

Portugal

Printed in Switzerland

Steel and cast iron — Determination of manganese content — Spectrophotometric method

1 Scope and field of application

This International Standard specifies a spectrophotometric method for the determination of manganese in steel and cast iron.

The method is applicable to products having manganese contents between 0,001 and 4 % (m/m).

2 Reference

ISO 377/2, *Selection and preparation of samples and test pieces of wrought steels — Part 2 : Samples and test pieces intended for the determination of the chemical composition.*[1]

3 Principle

Dissolution of a test portion in sulphuric-phosphoric acid mixture and nitric acid. Treatment of the test solution with perchloric acid. Formation of the permanganate ion by sodium periodate.

Spectrophotometric measurement of the test solution at 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.

NOTE — Verify by blank tests that relevant reagents are free from manganese. Wherever necessary, the results should be corrected accordingly.

4.1 **Water,** free from organic matter.

Bring to the boil water acidified with 10 ml/l of sulphuric acid (ϱ about 1,83 g/ml), add several crystals of sodium periodate ($NaIO_4$) and keep boiling for 10 min.

4.2 **Pure iron,** free from manganese.

4.3 **Hydrochloric acid,** ϱ about 1,19 g/ml.

4.4 **Nitric acid,** ϱ about 1,40 g/ml.

4.5 **Perchloric acid,** ϱ about 1,67 g/ml.

NOTE — It is also possible to use perchloric acid, ϱ about 1,54 g/ml. 127 ml of perchloric acid, ϱ about 1,54 g/ml is equivalent to 100 ml of perchloric acid, about 1,67 g/ml.

4.6 **Perchloric acid,** diluted 1 + 499.

4.7 Sulphuric/phosphoric acid mixture.

To 600 ml of water add in the following order, carefully and stirring at the same time, 100 ml of sulphuric acid (ϱ about 1,83 g/ml) and 150 ml of phosphoric acid (ϱ about 1,71 g/ml). Cool and dilute to 1 000 ml with water.

4.8 **Sodium periodate,** 50 g/l solution.

4.9 **Manganese,** standard solution

4.9.1 Stock solution

Weigh, to the nearest 0,001 g, 2,877 g of potassium permanganate, dissolve in 500 ml of water and add 10 ml sulphuric acid (ϱ about 1,83 g/ml). Decolorize the solution with hydrogen peroxide [about 30 % (m/m)] or with a stream of sulphur dioxide. In the latter case, eliminate the excess of reagent by boiling.

After cooling, transfer the solution to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this stock solution contains 1,00 mg of Mn.

4.9.2 Standard solution A.

Transfer 100 ml of the stock solution (4.9.1) to a 1 000 ml one-mark volumetric flask and dilute to the mark with water and mix.

1 ml of standard solution A contains 0,1 mg of Mn.

4.9.3 Standard solution B.

Transfer 250 ml of the standard solution A (4.9.2) to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of standard solution B contains 0,025 mg of Mn.

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[1] At present at the stage of draft. (Partial revision of ISO/R 377-1964.)

5 Apparatus

Ordinary laboratory apparatus and

5.1 Spectrophotometer.

6 Sampling

Sampling shall be carried out in accordance with ISO 377/2 or appropriate national standards for cast iron.

7 Procedure

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

7.1 Test portion

According to the presumed manganese content, weigh, to the nearest 0,001 g, the following mass (*m*) of the test portion :

a) for Mn contents less than 0,05 % (*m*/*m*) : *m* approximately 2 g;

b) for Mn contents between 0,05 and 2 % (*m*/*m*) : *m* approximately 1 g;

c) for Mn contents greater than 2 % (*m*/*m*) : *m* approximately 0,5 g.

7.2 Determination

7.2.1 Preparation of test solution

Place the test portion (7.1) in a 250 ml conical flask, add 50 ml of the sulphuric/phosphoric acid mixture (4.7) and heat gently until effervescence ceases. When the attack is complete, oxidize with a few drops of the nitric acid (4.4) and add 20 ml of the perchloric acid (4.5). Evaporate until white perchloric acid fumes are given off and keep at this temperature for 10 min.

After cooling, take up with water, dilute to 70 to 80 ml and boil. If necessary, filter and wash with the hot perchloric acid (4.6).

Collect the filtrate in a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix. This gives solution S_1.

NOTE — For samples that do not dissolve readily in the sulphuric/phosphoric acid mixture (4.7), add also 10 ml of the hydrochloric acid (4.3) and 10 ml of the nitric acid (4.4) to facilitate the dissolution.

7.2.2 Formation of coloured compound for test portion

Introduce into a 250 ml conical flask the following quantities of solution S_1, and reagents specified in table 1, depending on the presumed manganese content.

Bring to the boil, add 10 ml of the sodium periodate solution (4.8), boil for 2 min and maintain at 90 °C for 10 min. Cool to ambient temperature. Transfer into a 100 ml volumetric flask, dilute to the mark with water (4.1) and mix.

7.2.3 Preparation of the compensation solution

Take the same volumes of solution S_1, water and sulphuric/phosphoric acid mixture (4.7) as for the formation of the coloured compound for the test portion (7.2.2). Add 2 or 3 drops of the hydrochloric acid (4.3) and boil for several minutes.

After cooling, transfer into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

7.2.4 Spectrophotometric measurement

Carry out the spectrophotometric measurements on the test solution (7.2.2), using the spectrophotometer (5.1) at a wavelength about 545 nm, after having adjusted the instrument to zero absorbance against the compensation solution (7.2.3).

7.2.5 Establishment of calibration curve

7.2.5.1 Preparation of reference solutions

7.2.5.1.1 For manganese contents less than 0,05 % (*m*/*m*)

Weigh, to the nearest 0,001 g, 2,000 g of the pure iron (4.2). Transfer into a series of six 250 ml conical flasks, add 50 ml of the sulphuric/phosphoric acid mixture (4.7) and heat gently until effervescence ceases. When the attack is complete, oxidize with a few drops of the nitric acid (4.4) and add 20 ml of the perchloric acid (4.5). Evaporate until white perchloric acid fumes are given off and keep at this temperature for 10 min.

After cooling, take up with water, dilute to 70 to 80 ml and boil. If necessary, filter and wash with the hot perchloric acid (4.6).

Collect the filtrate in a 100 ml one-mark volumetric flask. After cooling, dilute to the mark with water and mix. This gives solution S_2.

Introduce into a series of six 250 ml conical flasks the quantities of manganese standard solution B (4.9.3) specified in table 2.

Dilute the above volumes to 25 ml with water, add 40,0 ml of solution S_2 and 5 ml of the sulphuric/phosphoric acid mixture (4.7). Continue in accordance with sub-clause 7.2.2, beginning at "Bring to the boil...".

7.2.5.1.2 For manganese contents between 0,05 and 4 % (*m*/*m*)

Weigh, to the nearest 0,001 g, approximately 1 g of the pure iron (4.2). Transfer into a 250 ml conical flask, add 50 ml of the sulphuric/phosphoric acid mixture (4.7) and heat gently until effervescence ceases. When the attack is complete, oxidize with a few drops of the nitric acid (4.4) and add 20 ml of the perchloric acid (4.5). Evaporate until white perchloric acid fumes are given off and keep at this temperature for 10 min.