17

TECHNICAL REPORT

ISO TR 10281

First edition
1990-11-01

Steel and iron — Determination of manganese content — Flame atomic absorption spectrometric method

Aciers et fontes — Dosage du manganèse — Méthode par spectrométrie d'absorption atomique dans la flamme

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Reference number
ISO/TR 10281:1990(E)

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Foreword

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The main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

— type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;

— type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;

— type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 10281, which is a Technical Report of type 2, was prepared by Technical Committee ISO/TC 17, *Steel*.

Annex A forms an integral part of this Technical Report. Annexes B and C are for information only.

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International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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Full document details: ISO_TR 10281_1990

Introduction

At the 10th ISO/TC 17/SC 1 meeting held in Chicago in 1984, WG 16 was created to develop a method for the determination of manganese content by flame atomic absorption spectrometry. Afterwards, in relation to this working group, three methods concerned with the determination of manganese were submitted for consideration. These were the ECSC/WG 20 working programme method (ECSC/WG 20 N 656), French standard method (NF A 08-331) and ASTM method (E 350), which were all similar. Coincidentally, at the 11th ISO/TC 17/SC 1 meeting held in Rome in 1986, delegates discussed whether the use of perchloric acid in the atomic absorption spectrometric technique was dangerous or not.

Taking into consideration the above position, the first working draft of WG 16 was therefore prepared as a method without using perchloric acid, based on the ECSC/WG 20 working programme. In accordance with the first working draft, an international co-operative experiment was conducted. The experimental results were reported at the 12th ISO/TC 17/SC 1 meeting held in Sydney in 1988.

That is to say, this method was developed at a time when there were grave doubts as to the safety of perchloric acid in the atomic absorption spectrometric technique and that the need to avoid its use had resulted in a relatively cumbersome method involving fusion.

However, by the time of the Sydney meeting, following further investigations, the use of perchloric acid had gained wider acceptance as the hazards associated with its use in atomic absorption spectrometric techniques became more clearly understood. It was agreed that perchloric acid could be used in atomic absorption spectrometric methods, provided that appropriate safety precautions were observed. In addition, it was suggested that coexisting silicon may interfere in the determination of manganese content. If this were the case, since perchloric acid attack could exclude silicon from the test solution, it would be better than hydrochloric/nitric acid attack which was used in the WG 16 experiments.

On the basis of the discussion described above, it was concluded at the 12th ISO/TC 17/SC 1 meeting that a new Working Group should be set up to develop a simpler method, of wider application, using perchloric acid in place of hydrochloric/nitric acid, thus avoiding the need for a fusion step, and to publish the present method in the form of a Technical Report, type 2.

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Steel and iron — Determination of manganese content — Flame atomic absorption spectrometric method

1 Scope

This Technical Report specifies a flame atomic absorption spectrometric method for the determination of manganese content in non-alloy and low alloy steels and iron.

The method is applicable to manganese contents between 0,002 % (*m/m*) and 3,0 % (*m/m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Technical Report. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Technical Report are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 377-2:1989, *Selection and preparation of samples and test pieces of wrought steels — Part 2: Samples for the determination of the chemical composition.*

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

3 Principle

Dissolution of a test portion in hydrochloric acid followed by oxidation with nitric acid. Ignition of the residue and removal of silica with hydrofluoric acid. Fusion of the residue with potassium hydrogen sulfate.

Determination of the manganese content by means of the spectrometric measurement of the atomic absorption of the 403,1 nm line emitted by a manganese hollow cathode lamp when the solution is sprayed into an air acetylene flame.

For low levels of manganese the more sensitive line of 279,5 nm may be used.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Pure iron, free from manganese.

4.2 Potassium hydrogen sulfate ($KHSO_4$).

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

4.4 Nitric acid, ρ about 1,40 g/ml.

4.5 Hydrofluoric acid, ρ about 1,15 g/ml.

4.6 Sulfuric acid, ρ about 1,84 g/ml.

4.7 Ammonia water, ρ about 0,88 g/ml.

4.8 Sulfuric acid, ρ about 1,84 g/ml, diluted 1 + 3.

To 75 ml of water add cautiously, with stirring, 25 ml of sulfuric acid (4.6).

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