TECHNICAL REPORT

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Iron ores — Determination of aluminium —

Part 1: Flame atomic absorption spectrometric method

Minerais de fer — Dosage de l'aluminium —

Partie 1: Méthode par spectrométrie d'absorption atomique dans la flamme

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This first edition Technical Report cancels and replaces the second edition (ISO 4688-1:2006), which has been technically revised. It has been converted to a Technical Report as it is no longer suitable for determination of aluminium as a referee method.

Iron ores — Determination of aluminium —

Part 1:
Flame atomic absorption spectrometric method

WARNING — This document may involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices.

1 Scope

This document describes a flame atomic absorption spectrometric method for the determination of the mass fraction of aluminium in iron ores.

This method is applicable to mass fractions of aluminium between 0,1 % and 5,0 % in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

The test portion is decomposed by treatment with hydrochloric acid and a small amount of nitric acid.

The mixture is evaporated to dehydrate silica, followed by dilution and filtration.

The residue is ignited and silica is removed by evaporation with hydrofluoric and sulfuric acids. The residue is then fused with sodium carbonate and the cooled melt is dissolved in the filtrate.

The solution obtained is aspirated into the flame of an atomic absorption spectrometer using a dinitrogen oxide/acetylene burner.

The absorbance values obtained for aluminium are compared with those obtained from the calibration solutions.

5 Reagents

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

5.1 Sodium carbonate (Na_2CO_3), anhydrous.

5.2 Hydrochloric acid, ρ 1,19 g/ml.

5.3 Nitric acid, ρ 1,4 g/ml.

5.4 Hydrochloric acid, ρ 1,19 g/ml, diluted 1 + 9.

5.5 Hydrofluoric acid, ρ 1, 13 g/ml, 40 % (mass fraction), or ρ 1,185 g/ml, 48 % (mass fraction).

5.6 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 1.

5.7 Background solution

Dissolve 10 g of high purity iron [minimum purity 99,9 % (mass fraction)] of mass fraction of aluminium less than 0,002 %, in 50 ml of hydrochloric acid (5.2) and oxidize by adding nitric acid (5.3) drop by drop.

Evaporate until a syrupy consistency is obtained. Add 20 ml of hydrochloric acid (5.2) and dilute to 200 ml with water. Dissolve 17 g of sodium carbonate (5.1) in water and add it to the iron solution. Transfer the solution to a 1 000 ml one-mark volumetric flask and dilute to volume with water.

5.8 Aluminium standard solution, 500 µg Al/ml.

Dissolve 0,5 000 g of high purity aluminium [minimum purity 99,9 % (mass fraction)] in 25 ml of hydrochloric acid (5.2). Cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

5.9 Aluminium calibration solutions

Transfer 2,0 ml; 5,0 ml; 10,0 ml; 20,0 ml; 40,0 ml; and 50,0 ml portions of aluminium standard solution (5.8) to 200 ml volumetric flasks. Dilute to about 100 ml. Add 6 ml of hydrochloric acid (5.2) and 60 ml of background solution (5.7) to each flask. Prepare a zero aluminium calibration solution by transferring 60 ml of the background solution to a 200 ml volumetric flask, and add 6 ml of hydrochloric acid (5.2). Dilute all the solutions to 200 ml with water and mix. (For an atomic absorption spectrometer having high sensitivity, smaller portions of the standard solution may be used.)

6 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 and ISO 1042, respectively, and the following.

6.1 Platinum crucible, of capacity 30 ml.

6.2 Muffle furnace, capable of maintaining a temperature of approximately 1 100 °C.

6.3 Atomic absorption spectrometer, equipped with a dinitrogen oxide/acetylene burner.

WARNING — Follow the manufacturer's instructions for igniting and extinguishing the dinitrogen oxide/acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the flame is burning.

The atomic absorption spectrometer used in this method should meet the following criteria.

a) Minimum sensitivity: the absorbance of the most concentrated aluminium calibration solution (5.9) should be at least 0,3.