

---

---

**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*

Document Preview

ISO/TR 4688-1:2017

<https://standards.iteh.ai/catalog/standards/iso/fd0c8974-c783-4003-9b71-f90ac7f3e814/iso-tr-4688-1-2017>



iTeh Standards  
(<https://standards.iteh.ai>)  
Document Preview

[ISO/TR 4688-1:2017](https://standards.iteh.ai/catalog/standards/iso/fd0c8974-c783-4003-9b71-f90ac7f3e814/iso-tr-4688-1-2017)

<https://standards.iteh.ai/catalog/standards/iso/fd0c8974-c783-4003-9b71-f90ac7f3e814/iso-tr-4688-1-2017>



**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2017, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Ch. de Blandonnet 8 • CP 401  
CH-1214 Vernier, Geneva, Switzerland  
Tel. +41 22 749 01 11  
Fax +41 22 749 09 47  
[copyright@iso.org](mailto:copyright@iso.org)  
[www.iso.org](http://www.iso.org)

# Contents

Page

<b>Foreword</b> .....	<b>iv</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 Principle</b> .....	<b>1</b>
<b>5 Reagents</b> .....	<b>1</b>
<b>6 Apparatus</b> .....	<b>2</b>
<b>7 Sampling and samples</b> .....	<b>3</b>
7.1 Laboratory sample.....	3
7.2 Preparation of predried test samples.....	3
<b>8 Procedure</b> .....	<b>3</b>
8.1 Number of determinations.....	3
8.2 Test portion.....	4
8.3 Blank test and check test.....	4
8.4 Determination.....	4
8.4.1 Decomposition of the test portion.....	4
8.4.2 Treatment of the residue.....	4
8.4.3 Preparation of the test solution.....	4
8.4.4 Adjustment of the atomic absorption spectrometer.....	5
8.4.5 Atomic absorption measurements.....	5
<b>9 Expression of results</b> .....	<b>6</b>
9.1 Calculation of mass fraction of aluminium.....	6
9.2 General treatment of results.....	6
9.2.1 Repeatability and permissible tolerance.....	6
9.2.2 Determination of analytical result.....	7
9.2.3 Between-laboratories precision.....	7
9.2.4 Check for trueness.....	7
9.2.5 Calculation of final result.....	8
9.3 Oxide factor.....	9
<b>10 Test report</b> .....	<b>9</b>
<b>Annex A (informative) Flowsheet of the procedure for the acceptance of analytical values for test samples</b> .....	<b>10</b>
<b>Annex B (informative) Derivation of repeatability and permissible tolerance formulae</b> .....	<b>11</b>
<b>Annex C (informative) Precision data obtained by international analytical trials</b> .....	<b>12</b>
<b>Bibliography</b> .....	<b>13</b>

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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](http://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 ( $\text{Na}_2\text{CO}_3$ ), anhydrous.

**5.2 Hydrochloric acid**,  $\rho$  1,19 g/ml.

**5.3 Nitric acid**,  $\rho$  1,4 g/ml.

**5.4 Hydrochloric acid**,  $\rho$  1,19 g/ml, diluted 1 + 9.

**5.5 Hydrofluoric acid**,  $\rho$  1,13 g/ml, 40 % (mass fraction), or  $\rho$  1,185 g/ml, 48 % (mass fraction).

**5.6 Sulfuric acid**,  $\rho$  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  $\mu$ g 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.