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## Iron ores — Determination of metallic iron content — Iron(III) chloride titrimetric method

*Minerais de fer — Dosage du fer métallique — Méthode titrimétrique au chlorure de fer(III)*

ICS: 73.060.10

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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  
www.iso.org

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## 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)).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#).

ISO 16878 was prepared by Technical Committee ISO/TC 102, *Iron Ore and Direct Reduced Iron*, Subcommittee SC 02, *Chemical Analysis*.

This first edition of ISO 16878 cancels and replaces ISO/TS 16878:2010, which has been technically revised.

# Iron ores — Determination of metallic iron content — Iron(III) chloride titrimetric method

**CAUTION** — This standard may involve hazardous operations and equipment. This standard does not purport to address all of the safety issues associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

## 1 Scope

This International Standard specifies a titrimetric method for the determination of the metallic iron content of reduced iron ores.

This method is applicable to a concentration range of 57,5 % mass fraction to 90,5 % mass fraction of the metallic iron.

**NOTE** The term "metallic iron" means those forms of iron not bonded to oxygen or not present as pyrite.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

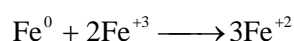
ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 10835, *Direct reduced iron — Sampling and sample preparation — Manual methods for reduced pellets and lump ores*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

## 3 Principle

Iron present in the oxidation state  $\text{Fe}^0$  in the sample is oxidized to  $\text{Fe}^{+2}$  by the action of  $\text{FeCl}_3$  according to the following reaction:



The  $\text{Fe}^{+2}$  is titrated with potassium dichromate solution using the sodium diphenylaminesulfonate indicator.

## 4 Definitions

For the purposes of this International Standard, the definitions given in ISO 11323 apply.

## 5 Reagents

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

### 5.1 Iron(III) chloride solution.

To a 2 000 ml container holding 600 ml of water add 250 g of iron(III) chloride hexahydrate and agitate until it is completely dissolved. Dilute with water to a volume of 1 l.

NOTE For better dissolution of the  $\text{FeCl}_3$ , it is recommended to use a warm water bath. The ideal condition is a complete dissolution of the salt resulting in a translucent solution.

### 5.2 Sulfuric acid, 1,84 g/ml.

### 5.3 Phosphoric acid, 1,7 g/ml.

### 5.4 Solution 15% sulfuric acid/15 % phosphoric acid mixture.

To a 3 000 ml beaker add 1 000 ml of water. Place the beaker in a cool place and add slowly with stirring 300 ml of phosphoric acid (5.3). Allow cooling. Add slowly with stirring 300 ml of sulfuric acid (5.2) and allow cooling. Dilute this mixture to 2 000 ml with stirring. Store the solution in a 2 000 ml high-density polyethylene bottle or equivalent container.

### 5.5 Sodium diphenylaminesulfonate.

Dissolve 0,2 g of powdered sodium diphenylaminesulfonate ( $\text{C}_6\text{H}_5\text{NHC}_6\text{H}_4\text{SO}_3\text{Na}$ ) in a small volume of water and dilute to 100 mL.

Store this solution in a brown glass bottle.

### 5.6 Standard potassium dichromate solution, 0,01667 mol/l.

Dry potassium dichromate at 150 °C for 3 h. Remove potassium dichromate from the oven and cool to room temperature in a desiccator or over silica gel. For a minimum purity 99,9 % (m/m) potassium dichromate weigh 9,808 g into a weighing scoop and transfer qualitatively to a 2 000 ml volumetric flask. Add 1 500 ml of water and dissolve potassium dichromate. When dissolution is complete, make up to volume and mix thoroughly.

### 5.7 Inert gas, carbon dioxide ( $\text{CO}_2$ ), argon (Ar) or nitrogen ( $\text{N}_2$ ).

## 6 Apparatus

One mark A-grade volumetric flasks complying with the specifications of ISO 648 and ISO 1042 respectively, and the following.

### 6.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

### 6.2 Erlenmeyer flask, widemouth (500 ml).

### 6.3 Stopper, to fit erlenmeyer flask, modified to allow the inert gas to pass through the beaker.

### 6.4 Magnet stirrer, with magnetic **stirring** bar.

### 6.5 Measuring cylinder, of appropriate volume.

### 6.6 A grade burette, mark A, complying with the specifications of ISO 648.

## 6.7 Non-magnetic spatula.

## 7 Sampling and sample preparation

For analysis, use a laboratory sample of – 160 µm particle size which has been taken and prepared in accordance with ISO 10835. Metallized fines should be dried in an inert atmosphere and stored where possible in an airtight container flushed with a dry inert gas to prevent re-oxidation of the sample.

Thoroughly mix the laboratory sample using non-magnetic materials. Taking multiple increments with a non-magnetic spatula, extract a test sample of not less than 25 g in such a manner that it is representative of the whole of the contents of the container. When taking the test portion for the purpose of analysis, expose the sample to air for as short a time as practical. After weighing immediately replace the remaining material in its container, flush with an inert gas and store.

## 8 Procedure

### 8.1 Number of determinations

All determinations should be carried out in duplicate. If necessary, make further determinations in accordance with the flowsheet presented in Annex A.

### 8.2 Preparation of test portions

Accurately weigh 0,200 0 g ± 0,005 0 g of the test sample. Record the sample mass (*m*).

### 8.3 Determination

Transfer the sample to a 500 ml erlenmeyer flask and fit the stopper to the flask. Eliminate air from the flask by **flushing** with a controlled flow of inert gas (approximately 3 l/min) for 5 min. Remove the stopper and dispense 35 ml of iron(III) chloride solution (5.1) into the flask. Add sufficient water to the sample to cover the stirring bar (at least 15 ml). Adjust the inert gas flow to a gentle stream (approximately 5 l/min) and fit the stopper to the flask.

**WARNING — Adjust the inert gas flow while the stopper is not fitted to the flask. Adjusting the inert gas flow while the stopper is fitted to the beaker may cause the solution to be forced out of the beaker.**

Stir the solution with a magnetic agitator for at least 45 min maintaining the inert gas flow.

Remove the stopper and add 50 ml of sulfuric acid-phosphoric acid solution (5.4), 200 ml of water and 1 ml of sodium diphenylamine sulfonate indication (5.5).

Titrate the solution with potassium dichromate (5.6). The end point is reached when a final drop of the titrant imparts a permanent purple colour.

Perform a blank test by following the procedure without the sample. Record the blank titration volume as  $V_0$ .

NOTE A blank needs to be performed every time a new batch of iron (III) chloride solution is prepared.

## 9 Expression of results

### 9.1 Calculation of the metallic iron content

Determine the metallic iron content in the sample,  $Fe_{(met)}$ , as a percentage by mass, using Formula (1):

$$\% Fe_{(\text{met})} \text{ m/m} = \frac{(V - V_0) \times 0,005\,584\,7}{m \times 3} \times 100 \quad (1)$$

where

$V$  is the volume of potassium dichromate consumed during the titration of the sample, in millilitres;

$V_0$  is the volume of potassium dichromate consumed during the titration of the blank, in millilitres;

$m$  is the sample mass, in grams;

3 is the constant of stoichiometric relation between  $Fe^{+2}$  and  $Fe^0$ ;

0,005 584 7 is the multiple of atomic mass of iron.

## 9.2 General treatment of result

### 9.2.1 Repeatability and permissible tolerances

The precision of this analytical method is expressed by the following values:

NOTE Additional information is given in Annex B.

$$R_d = 0,349$$

$$P = 1,015$$

$$\sigma_d = 0,351$$

$$\sigma_L = 0,586$$

where

$R_d$  is the independent duplicate limit;

$P$  is the permissible tolerance between laboratories;

$\sigma_d$  is the independent duplicate standard deviation;

$\sigma_L$  is the between-laboratories standard deviation.

### 9.2.2 Determination of analytical result

Having computed the independent duplicate results according to Formula (1), compare them with the independent duplicate limit ( $R_d$ ), using the procedure given in Annex A.

### 9.2.3 Between-laboratories precision

Between-laboratories precision is used to determine the agreement between the final results reported by two laboratories. The assumption is that both laboratories followed the same procedure described in 8.3.

Compute the following quantity:

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \quad (2)$$



where

$\mu_1$  is the final result reported by laboratory 1;

$\mu_2$  is the final result reported by laboratory 2;

$\mu_{12}$  is the mean of final results.

If  $|\mu_1 - \mu_2| \leq P$ , the final results are in agreement.

#### 9.2.4 Check for trueness

The trueness of the analytical method shall be checked by applying it to a certified reference material (CRM) or a reference material (RM). The procedure is the same as that described above. After confirmation of the precision, the final laboratory result is compared with the reference or certified value  $A_c$ . There are two possibilities:

- $|\mu_c - A_c| \leq C$ , in which case the difference between the reported result and the reference/certified value is statistically insignificant.
- $|\mu_c - A_c| > C$ , in which case the difference between the reported result and the reference/certified value is statistically significant.

Where

$\mu_c$  is the final result for the certified reference material;

$A_c$  is the reference/certified value for the CRM/RM;

$C$  is a value dependent on the type of CRM/RM used.

Certified reference materials used for this purpose should be prepared and certified in accordance with ISO Guide 35.

$C$  shall be calculated as follows:

$$C = 2 \sqrt{\frac{S_C^2}{N_C} + \sigma_L^2 + \frac{\sigma_d^2}{n}}$$

where

$S_C$  is the standard deviation of laboratory means (each data for calculating the standard deviation is the average data in each certifying laboratory) of the CRM/RM.

$N_C$  is the number of certifying laboratories.

$n$  is the number of replicate determinations carried out on the CRM/RM.

For CRMs certified by only one laboratory  $C$  shall be calculated as follows:

$$C = 2 \sqrt{2\sigma_L^2 + \frac{\sigma_d^2}{n}}$$

A CRM certified by only one laboratory should be avoided unless it is known to have an unbiased certified value.