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

National foreword

This British Standard is the UK implementation of ISO 16878:2016. It supersedes DD ISO/TS 16878:2010 which is withdrawn.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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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)

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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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The committee responsible for this document is ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *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 International Standard may involve hazardous operations and equipment. This International 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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

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

ISO 10835, *Direct reduced iron and hot briquetted iron — Sampling and sample preparation*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

4 Principle

Iron present in the oxidation state Fe^0 in the sample is oxidized to Fe^{+2} by the action of $FeCl_3$ according to the following reaction:

$$Fe^{0} + 2Fe^{+3} \longrightarrow 3Fe^{+2}$$

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

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**.

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

NOTE For better dissolution of the $FeCl_3$, 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**.

Add 1 000 ml of water to a 3 000 ml beaker. 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 ($C_6H_5NHC_6H_4SO_3Na$) 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**, 0,016 67 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 % (mass fraction) 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 (CO_2), argon (Ar) or nitrogen (N_2).

6 Apparatus

One mark A-grade volumetric flasks complying with the specifications of ISO 1042 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 385.

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 can 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.

In each run, one blank test shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions.

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

For every new batch of iron (III) chloride solution, a blank should be performed.

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):

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

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 according to the reaction quoted in Clause 4;

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:

— $R_d = 0{,}349$;

— $P = 1{,}015$;

— $S_d = 0{,}123$;

— $S_L = 0{,}344$.

where

R_d is the independent duplicate limit;

P is the permissible tolerance between laboratories;

S_d is the independent duplicate standard deviation;

S_L is the between-laboratories standard deviation.

NOTE Additional information is given in Annex B.

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:

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

where

μ_1 is the final result reported by laboratory 1;

μ_2 is the final result reported by laboratory 2;

μ_{12} is the mean of final results.

If $\left|\mu_1 - \mu_2\right| \leq P$, 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:

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

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

where

μ_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 given in Formula (3):

$$C = 2\sqrt{\frac{S_C^2}{N_C} + \sigma_L^2 + \frac{\sigma_d^2}{n}} \qquad (3)$$

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 given in Formula (4):

$$C = 2\sqrt{2\sigma_{\mathrm{L}}^{2} + \frac{\sigma_{\mathrm{d}}^{2}}{n}} \qquad (4)$$

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

9.2.5 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, or as otherwise determined by the operations specified in Annex A, calculated to four decimal places and rounded off to the second decimal place as follows:

a) where the figure in the third decimal place is less than 5, it is discarded and the figure in the second decimal place is kept unchanged;

b) where the figure in the third decimal place is 5 and there is a figure other than 0 in the fourth decimal place, or where the figure in the third decimal place is greater than 5, the figure in the second decimal place is increased by one;

c) where the figure in the third decimal place is 5 and there is the figure 0 in the fourth decimal place, the 5 is discarded and the figure in the second decimal place is kept unchanged if it is 0, 2, 4, 6 or 8 and is increased by one if it is 1, 3, 5, 7 or 9.

10 Test report

The test report shall include the following information:

a) name and address of testing laboratory;

b) date of the test report;

c) reference to this International Standard, i.e. ISO 16878;

d) details necessary for the identification of the sample;

e) results of the test, to one decimal place;

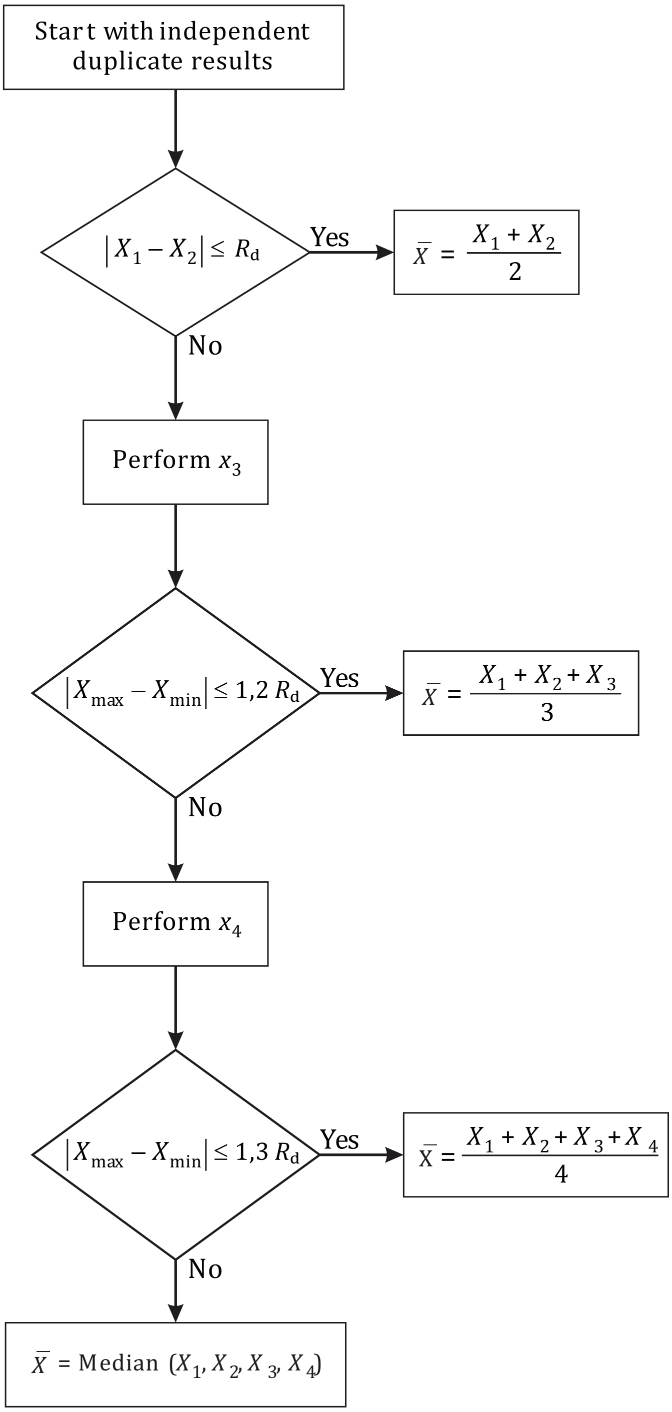
f) any characteristics noticed during the test and any operations which may have had an influence on the results, e.g. drying time, titration end-point;

g) all operations not specified by this International Standard or regarded as optional;

h) reference to verification activities.

Annex A
(normative)

Flowsheet of the procedure for the acceptance of test results



NOTE R_d is independent duplicate limit, defined in 9.2.1.

Figure A.1 — Flowsheet of the procedure for the acceptance of test results

Annex B
(informative)

Derivation of precision statements

The precision statements in 9.2.1 were obtained by statistical evaluation of the results of international analytical trials carried out in 2013 on thirteen metallic iron samples (see Table B.1) involving ten laboratories in seven countries.

Table B.1 — Total metallic iron contents of test samples

Sample	Metallic iron content % (mass fraction)
SG23-01	57,56
SG23-02	74,95
SG23-03	85,14
SG23-04	88,43
SG23-05	79,82
SG23-06	90,34
SG23-07	82,20
SG23-08	87,31
SG23-09	82,16
SG23-10	89,95
SG23-11	57,77
SG23-12	71,37
SG23-13	65,97

NOTE 1 A report of the international trials and a statistical analysis of the results (Document ISO/TC 102/SC 2 N2056) is available from the secretariat of ISO/TC 102/SC 2.

NOTE 2 The statistical analysis has been performed in accordance with the principles embodied in ISO 5725-2.

Bibliography

[1] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[2] ISO Guide 35, *Reference materials — General and statistical principles for certification*

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