INTERNATIONAL STANDARD

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Iron ores — Determination of total iron content —

Part 1:
Titrimetric method after tin(II) chloride reduction

Minerais de fer — Dosage du fer total —

Partie 1: Méthode titrimétrique après réduction au chlorure d'étain(II)

Reference number
ISO 2597-1:1994(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 2597-1 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 2, *Chemical analysis*.

This first edition of ISO 2597-1 cancels and replaces ISO 2597:1985, of which it constitutes a technical revision.

ISO 2597 consists of the following parts, under the general title *Iron ores — Determination of total iron content*:

— *Part 1: Titrimetric method after tin(II) chloride reduction*

— *Part 2: Titanium(III) chloride reduction methods*

Parts 2 and 3 will cancel and replace ISO 9507:1990 and ISO 9508:1990, respectively.

Annex A forms an integral part of this part of ISO 2597. Annexes B and C are for information only.

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Iron ores — Determination of total iron content —

Part 1:
Titrimetric method after tin(II) chloride reduction

1 Scope

This part of ISO 2597 specifies a titrimetric method for the determination of the total iron content of iron ores using potassium dichromate after reduction of the trivalent iron by tin(II) chloride.

The method is applicable to total iron contents between 30 % (*m/m*) and 72 % (*m/m*) in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

NOTE 1 Equivalent International Standards that do not use mercury(II) chloride are ISO 9507:1990, *Iron ores — Determination of total iron content — Titanium(III) chloride reduction methods*, and ISO 9508:1990, *Iron ores — Determination of total iron content — Silver reduction titrimetric method*.

A suggested procedure for removal of mercury from the waste solutions before discharge to effluent drains is given in annex C.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 2597. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 2597 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 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 2596:1994, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric and Karl Fischer methods.*

ISO 3081:1986, *Iron ores — Increment sampling — Manual method.*

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method.*

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 7764:1985, *Iron ores — Preparation of predried test samples for chemical analysis.*

3 Principle

Decomposition of the test portion.

a) For samples containing not more than 0,05 % (*m/m*) vanadium, 0,1 % (*m/m*) molybdenum or 0,1 % (*m/m*) copper: treatment with hydrochloric acid, the residue being filtered off and ignited, treatment with hydrofluoric and

sulfuric acids to remove silica, and fusion with potassium pyrosulfate.

Dissolution of the melt in hydrochloric acid, precipitation of iron with ammonia solution, redissolution of the precipitate in hydrochloric acid and addition of this solution to the main solution.

b) For samples containing more than 0,05 % (*m/m*) vanadium: fusion with alkali, the melt being leached with water and filtered, the filtrate being dicarded. Dissolution of the residue in hydrochloric acid.

Reduction of trivalent iron in the solution, using tin(II) chloride. Oxidation of excess reductant with mercury(II) chloride.

Titration of the reduced iron with potassium dichromate solution, using sodium diphenylamine-sulfonate as indicator.

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water that complies with grade 2 of ISO 3696.

4.1 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml.

4.2 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 1.

4.3 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 2.

4.4 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 10.

4.5 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 50.

4.6 Sulfuric acid, ρ 1,84 g/ml.

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

4.8 Hydrofluoric acid, ρ 1,13 g/ml [40 % (*m/m*)], or ρ 1,19 g/ml [48 % (*m/m*)].

4.9 Orthophosphoric acid, ρ 1,7 g/ml.

4.10 Ammonia solution, ρ 0,90 g/ml.

4.11 Sodium carbonate (Na_2CO_3), anhydrous powder.

Heat for 30 min at 500 °C, or confirm that the water content is not more than 1 % (*m/m*) (by heating a test portion for 30 min at 500 °C and measuring the loss in mass).

4.12 Sodium peroxide (Na_2O_2), dry powder.

Store this reagent in a dry environment and do not use it after it has begun to agglomerate.

4.13 Potassium pyrosulfate ($K_2S_2O_7$), fine powder.

4.14 Sulfuric acid-orthophosphoric acid mixture.

Cautiously pour 150 ml of sulfuric acid (4.6) into about 300 ml of water while stirring, cool in a water bath or running water, add 150 ml of orthophosphoric acid (4.9) and dilute to 1 litre with water.

4.15 Sodium hydroxide, 20 g/l solution.

4.16 Tin(II) chloride, 100 g/l solution.

Dissolve 100 g of crystalline tin(II) chloride dihydrate ($SnCl_2 \cdot 2H_2O$) in 200 ml of hydrochloric acid (4.1) by heating the solution in a water bath.

Cool the solution and dilute to 1 litre with water.

Store this solution in a brown glass bottle with the addition of a small quantity of granular or mossy tin metal.

NOTE 2 The tin(II) chloride solution may be prepared in 250 ml lots.

4.17 Mercury(II) chloride ($HgCl_2$), 50 g/l solution.

4.18 Potassium permanganate ($KMnO_4$), 30 g/l solution.

4.19 Iron standard solution, 0,1 mol/l.

Weigh, to the nearest 0,002 g, 5,58 g of pure iron [minimum purity 99,9 % (*m/m*)] into a 500 ml conical flask and place a small filter funnel in the neck. Add 75 ml of hydrochloric acid (4.2) in small increments and heat until the iron is dissolved. Cool and oxidize with 5 ml of hydrogen peroxide [30 % (*V/V*)] added in small portions. Heat to boiling and boil to decompose the excess hydrogen peroxide and to remove chlorine. Transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

1,00 ml of this solution is equivalent to 1,00 ml of potassium dichromate standard volumetric solution.

4.20 Potassium dichromate, standard volumetric solution, $c(K_2Cr_2O_7) = 0{,}016\ 67$ mol/l.

Pulverize about 6 g of potassium dichromate standard reagent [minimum purity 99,9 % (*m/m*)] in an agate mortar, dry in an air bath at 140 °C to 150 °C for 2 h, and cool to room temperature in a desiccator. Dissolve 4,904 g of this dried and pulverized potassium dichromate in water and dilute the solution to exactly 1 000 ml.

Record on the stock bottle the temperature at which this dilution was made (t_1).

4.21 Sodium diphenylaminesulfonate, solution.

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 Apparatus

Ordinary laboratory apparatus, including burettes, one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 385-1, ISO 648 or ISO 1042 respectively (unless otherwise indicated), and

5.1 Zirconium, vitreous carbon or **alkali-resistant sintered alumina crucibles**, of capacity 25 ml to 30 ml.

5.2 Platinum crucibles, of capacity 25 ml to 30 ml.

5.3 Porcelain crucibles, of capacity 25 ml to 30 ml.

5.4 Weighing spatula, of non-magnetic material or demagnetized stainless steel.

5.5 Muffle furnace, capable of being maintained at between 500 °C ± 10 °C and 800 °C ± 10 °C.

6 Sampling and samples

6.1 Laboratory sample

For analysis, use a laboratory sample of minus 100 µm particle size which has been taken in accordance with ISO 3081 or ISO 3082 and prepared in accordance with ISO 3082 or ISO 3083. In the case of ores having significant contents of combined water or oxidizable compounds, use a particle size of minus 160 µm.

NOTES

3 A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

4 If the determination of total iron relates to a reducibility test, prepare the laboratory sample by crushing and pulverizing, to less than 100 µm particle size, the whole of one of the reducibility test portions which has been reserved for chemical analysis. In the case of ores having significant contents of combined water or oxidizable compounds, use a particle size of minus 160 µm.

6.2 Preparation of test samples

Depending on the ore type, proceed in accordance with either 6.2.1 or 6.2.2.

6.2.1 Ores having significant contents of combined water or oxidizable compounds

Prepare an air-equilibrated test sample, in accordance with ISO 2596, with the following types of ores:

a) natural or processed ores in which the content of combined water is higher than 2,5 % (*m/m*);

b) processed ores containing metallic iron;

c) natural or processed ores in which the sulfur content is higher than 0,2 % (*m/m*).

6.2.2 Ores outside the scope of 6.2.1

Prepare a predried test sample as follows.

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a way that it is representative of the whole contents of the container. Dry the test sample at 105 °C ± 2 °C, as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

7.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with annex A, independently, on one test sample (6.2).

NOTE 5 The expression "independently" means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure is carried out either by the same operator at a different time or by a different operator, including appropriate recalibration in either case.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, approximately 0,4 g of the test sample (6.2) using the non-magnetic spatula (5.4).

NOTES

6 For samples of iron content higher than 68 % (*m/m*), weigh approximately 0,38 g.

7 If predried test samples are used, the test portion should be taken and weighed quickly on the day of predrying, to avoid reabsorption of moisture.

7.3 Determination of hygroscopic moisture content

Where the ore type conforms to the specifications of 6.2.1, determine the hygroscopic moisture content in accordance with ISO 2596, simultaneously with the taking of the test portion (7.2) for the determination of iron content.

7.4 Blank test and check test

In each run, one blank test and one analysis of a certified reference material of the same type of ore shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions. A test sample of the certified reference material shall be prepared in a manner appropriate to the type of ore involved (see 6.2).

NOTES

8 The certified reference material should be of the same type as the sample to be analysed, and the properties of the two materials should be sufficiently similar to ensure that, in either case, no significant changes in the analytical procedure will become necessary.

9 The certified reference material is used only to validate the performance of the analytical procedure and expressly not to standardize the potassium dichromate solution.

Where the analysis is carried out on several samples at the same time, the blank value may be represented by one test, provided that the procedure is the same and the reagents used are from the same reagent bottles.

Where the analysis is carried out on several samples of the same type of ore at the same time, the analytical value of one certified reference material may be used.

7.5 Determination

7.5.1 Decomposition of the test portion

7.5.1.1 Acid decomposition [for samples containing not more than 0,05 % (*m/m*) vanadium, 0,1 % (*m/m*) molybdenum or 0,1 % (*m/m*) copper]

Place the test portion (7.2) in a 300 ml beaker, add 30 ml of hydrochloric acid (4.1), cover the beaker with a watch glass, and heat the solution in the low temperature zone (about 80 °C) of a hotplate for about 1 h. Transfer to a higher temperature zone and heat just below boiling for about 10 min or until the ore is substantially decomposed. (See notes 10 and 11.)

Wash the watch glass and dilute the solution to about 50 ml with warm water. Filter the insoluble residue on a close-texture paper and wash with warm hydrochloric acid (4.5) until the yellow colour of iron(III) chloride is no longer observed. Then wash it six to eight times with warm water. Collect the filtrate and washings in a 600 ml beaker and concentrate to about 30 ml by heating without boiling. (This is the main solution.)

Place the filter paper and the residue in a platinum crucible (5.2), dry, char the paper and finally ignite at 750 °C to 800 °C. Allow the crucible to cool. Moisten the residue in the crucible with sulfuric acid (4.7), add about 5 ml of hydrofluoric acid (4.8), and heat gently to remove silica and sulfuric acid.

Add 2 g of potassium pyrosulfate (4.13) to the contents of the cooled crucible and heat gently at first and then more strongly, minimizing any tendency for the flux to creep, until a clear melt is obtained. Allow the melt to cool, place the platinum crucible in a 300 ml beaker, add about 100 ml of warm water and about 5 ml of hydrochloric acid (4.1), and heat gently to dissolve the melt. Rinse and remove the platinum crucible from the beaker.

Adjust this solution to slight alkalinity by the addition of ammonia solution (4.10), boil the solution for a few minutes and remove it from the source of heat.

When the precipitate has settled, collect the precipitate of iron hydroxide on a rapid filter paper, and wash six to eight times with warm water. Discard the filtrate and washings.

Place the beaker containing the main solution under the funnel and dissolve the precipitate on the filter paper by pouring 10 ml of hydrochloric acid (4.3) over it; wash the filter, first six to eight times with warm hydrochloric acid (4.5), then twice with hot water, and follow the procedure specified in 7.5.2.