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Iron ores — Determination of total iron content —

Part 2:
Titrimetric methods after titanium(III) chloride reduction

Minerais de fer — Dosage du fer total —

Partie 2: Méthodes titrimétriques après réduction au chlorure de titane(III)

Reference number
ISO 2597-2:2008(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 2597-2 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

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: Titrimetric methods after titanium(III) chloride reduction*

Iron ores — Determination of total iron content —

Part 2:
Titrimetric methods after titanium(III) chloride reduction

1 Scope

This part of ISO 2597 specifies two titrimetric methods, free from mercury pollution, for the determination of total iron content in iron ores, using potassium dichromate as titrant after reduction of the iron(III) by tin(II) chloride and titanium(III) chloride. The excess reductant is then oxidized by either dilute potassium dichromate (method 1) or perchloric acid (method 2).

Both methods are applicable to a concentration range of 30 % mass fraction to 72 % mass fraction of iron in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

WARNING — This part of ISO 2597 may involve hazardous materials, operations and equipment. This part of ISO 2597 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 2597 to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

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 31-0:1992, *Quantities and units — Part 0: General principles*

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

ISO 385, *Laboratory glassware — Burettes*

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

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

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

3 Principle

3.1 Decomposition of the test portion

3.1.1 Acid decomposition

For samples containing not more than 0,05 % mass fraction of vanadium, the test portion is treated with hydrochloric acid in the presence of tin chloride and the residue is filtered, ignited and treated with hydrofluoric and sulfuric acids. The mixture is fused with potassium disulfate and the cold melt is dissolved in water and hydrochloric acid then neutralized with ammonia solution. The precipitate is filtered, washed in water, dissolved in hydrochloric acid and combined with the main iron solution, which is treated with potassium permanganate and evaporated.

3.1.2 Fusion-filtration

For samples containing more than 0,05 % mass fraction of vanadium, the test portion is fused with a mixture of fluxes, the cold melt is leached with water and the precipitate is filtered, washed in sodium hydroxide solution, dissolved in hydrochloric acid and evaporated.

3.2 Titration of iron

The major portion of the iron(III) is reduced by tin(II) chloride and the remainder of the iron(III) is reduced by titanium(III) chloride. The excess reductant is oxidized with either dilute potassium dichromate solution (method 1) or dilute perchloric acid (method 2). The reduced iron is titrated with potassium dichromate solution using the sodium diphenylaminesulfonate indicator.

4 Reagents

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

	Method
4.1 Hydrochloric acid, ρ1,16 g/ml to 1,19 g/ml.	1 and 2
4.2 Hydrochloric acid, ρ1,16 g/ml to 1,19 g/ml, diluted 1 + 1.	1 and 2
4.3 Hydrochloric acid, ρ1,16 g/ml to 1,19 g/ml, diluted 1 + 12.	1 and 2
4.4 Hydrochloric acid, ρ1,16 g/ml to 1,19 g/ml, diluted 2 + 100.	1 and 2
4.5 Hydrofluoric acid, 40 % mass fraction (ρ1,13 g/ml) or 48 % mass fraction (ρ1,19 g/ml).	1 and 2
4.6 Sulfuric acid, ρ1,84 g/ml.	1 and 2
4.7 Sulfuric acid, ρ1,84 g/ml, diluted 1 + 1, carefully pour 1 volume of reagent 4.6 into 1 volume of cold water.	1 and 2
4.8 Orthophosphoric acid, ρ1,7 g/ml.	1 and 2
4.9 Perchloric acid, 72 % mass fraction (ρ1,7 g/ml), diluted 1 + 1.	2

	Method
4.10 Sulfuric acid–orthophosphoric acid mixture, pour 150 ml of orthophosphoric acid (4.8) into about 400 ml of water while stirring, add 150 ml of sulfuric acid (4.6), cool in a water bath, dilute with water to 1 l and mix well.	1 and 2
4.11 Ammonia solution, 28 % mass fraction (ρ 0,90 g/ml) to 30 % mass fraction (ρ 0,96 g/ml).	1
4.12 Sodium hydroxide (NaOH) solution, 20 g/l.	1 and 2
4.13 Hydrogen peroxide (H_2O_2), 30 % by volume solution.	1 and 2
4.14 Hydrogen peroxide (H_2O_2), 30 % by volume solution, diluted 1 + 9.	1
4.15 Tin(II)–hydrochloric acid solution, dissolve 130 g of tin metal in about 500 ml of hydrochloric acid (4.1) and dilute with hydrochloric acid to 1 l. This solution should be stored in a brown glass bottle. Use supernatant liquid as needed.	1 and 2
4.16 Tin(II) chloride solution, 100 g/l, dissolve 100 g of crystalline tin(II) chloride ($SnCl_2 \cdot 2H_2O$) in 200 ml of hydrochloric acid (4.1) by heating the solution in a water bath. Cool the solution and dilute with water to 1 l. This solution should be stored in a brown glass bottle with a small quantity of granular tin metal.	1 and 2
4.17 Potassium permanganate ($KMnO_4$) solution, 25 g/l.	1 and 2
4.18 Potassium dichromate ($K_2Cr_2O_7$) solution, 1 g/l.	1
4.19 Titanium(III) chloride ($TiCl_3$) solution, 20 g/l, dilute one volume of titanium(III) chloride solution (about 20 % $TiCl_3$) with nine volumes of hydrochloric acid (4.2). Alternatively, dissolve 1,3 g of titanium sponge in about 40 ml of hydrochloric acid (4.1) in a covered beaker by heating in a water bath. Cool the solution and dilute with water to 200 ml. Prepare fresh solution as needed.	1 and 2
4.20 Potassium disulfate ($K_2S_2O_7$), fine powder.	1 and 2
4.21 Flux mixture, mix one portion of anhydrous sodium carbonate (Na_2CO_3) and two portions of sodium peroxide (Na_2O_2).	1 and 2
4.22 Iron standard solution, 0,1 mol/l, transfer 5,58 g of pure iron (purity greater than 99,9 % mass fraction) to a 500 ml Erlenmeyer flask and place a small filter funnel in the neck. Add 75 ml of hydrochloric acid (4.2) in small increments and heat until dissolved. Cool and oxidize with 5 ml of hydrogen peroxide (4.13) added in small portions. Heat to boiling and boil to decompose the excess hydrogen peroxide and to expel chlorine. Cool, transfer to a 1 000 ml volumetric flask and mix well.	1 and 2
1,00 ml of this solution is equivalent to 1,00 ml of the standard potassium dichromate solution (4.23).	

Method

4.23 Potassium dichromate (99,9 % minimum purity), standard solution, 0,016 67 mol/l, pulverize about 6 g of potassium dichromate reagent in an agate mortar, dry at 140 °C to 150 °C for 2 h, and cool to room temperature in a desiccator.

1 and 2

Transfer 4,903 g of this material to a 300 ml beaker, dissolve in about 100 ml of water, transfer quantitatively to a 1 000 ml volumetric flask, make up to volume with water after cooling to 20 °C and mix well. Record the temperature at which this dilution was made (20 °C) on the stock bottle. Measure the temperature at each use to correct the volume of titrant used.

NOTE 1 The volumetric flask should previously be calibrated by weighing the mass of water contained at 20 °C and converting to volume.

NOTE 2 Water used for preparation should previously be equilibrated at room temperature.

NOTE 3 A calibrated mercury thermometer, graduated in 0,1 °C divisions and having a marked dipping line, should be used. Take a sufficient volume of standard solution for dipping the thermometer and transfer to a suitable beaker. Measure the temperature of the solution to the nearest 0,1 °C, after dipping for more than 60 s.

4.24 Indigo carmine [indigo-5,5'-disulfonic acid disodium salt $C_{l6}H_8O_8N_2S_2Na_2$)] solution, 0,1 g/100 ml, dissolve 0,1 g of indigo carmine in a cold mixture of 50 ml sulfuric acid (4.7) and 50 ml of water.

1

4.25 Sodium diphenylaminesulfonate indicator solution, 0,2 g/100 ml, dissolve 0,2 g of sodium diphenylaminesulfonate ($C_6H_5NHC_6H_4SO_3Na$) in a small volume of water and dilute to 100 ml.

1 and 2

Store the solution in a brown glass bottle.

5 Apparatus

The pipette and volumetric flask specified shall conform with ISO 648 and ISO 1042 respectively.

Ordinary laboratory apparatus, and

5.1 Alumina, **zirconium or vitreous carbon crucible**, capacity 25 ml to 30 ml, crucibles should be cleaned before use to avoid contamination with iron.

5.2 Burette, class A, conforming with ISO 385.

5.3 Weighing bottle, of approximate volume 10 ml and approximate mass 6 g.

5.4 Platinum crucible, capacity 25 ml to 30 ml and having a lid.

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

5.6 Muffle furnace, suitable for operation in the range 500 °C to 800 °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 and prepared in accordance with ISO 3082. In the case of ores having significant contents of combined water or oxidizable compounds, use a particle size of less than 160 μm.

NOTE 1 A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

NOTE 2 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 less than 160 μm.

6.2 Preparation of test samples

6.2.1 General

Depending on the ore type, proceed in accordance with either 6.2.2 or 6.2.3.

6.2.2 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 ore:

a) processed ores containing metallic iron;

b) natural or processed ores in which the sulfur content is higher than 0,2 % mass fraction;

c) natural or processed ores in which the content of combined water is higher than 2,5 % mass fraction.

6.2.3 Ores outside the scope of 6.2.2

Prepare a predried test sample as follows.

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container. Prepare test portions in accordance with the Japanese weighing method (see Annex C).

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 (see 6.2).

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

7.2 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 the manner appropriate to the type of ore involved (see 6.2 and NOTE 1 below.)

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.

NOTE 1 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 be necessary.

NOTE 2 The certified reference material is used only to validate the performance of the analytical procedure and expressly not to standardize the potassium dichromate solution.

7.3 Determination of hygroscopic moisture content

Where the ore type conforms to the specifications of 6.2.2, determine the hygroscopic moisture content in accordance with ISO 2596, simultaneously with the taking of the test portion (see 7.4) for the determination of the iron content.

7.4 Test portion

Taking several increments, weigh to the nearest 0,000 2 g, approximately 0,4 g of the test sample (see 6.2), using a non-magnetic spatula (5.5).

NOTE For samples having an iron content higher than 68 % mass fraction, weigh approximately 0,38 g.

Where the ore type is outside the scope of 6.2.2, transfer the portion to a weighing bottle and determine the mass of the test portion to the nearest 0,000 2 g in accordance with the Japanese weighing method (see Annex C).

7.5 Determination

7.5.1 Decomposition of the test portion

7.5.1.1 Acid decompostion (for samples containing $\leqslant$ 0,05 % mass fraction vanadium)

Place the test portion (see 7.4) in a dried 300 ml or 400 ml beaker, project about 10 ml of water on the inside wall of the beaker and suspend the test portion in water while swirling.

Add 20 ml of hydrochloric acid (4.1) and 10 drops of tin(II)-hydrochloric acid solution (4.15), cover the beaker with a watch glass, heat the solution gently at about 80 °C for 1 h and continue heating without boiling at a higher temperature for about 10 min to decompose the portion.

NOTE Boiling should be avoided to prevent volatilization loss of iron(III) chloride.

Remove the beaker from the source of heat, wash the watch glass in a jet of water, and dilute to 50 ml with warm water. Filter the insoluble residue on a filter paper. Scrub the remainder of the residue on the beaker wall by using a policeman, and transfer the remainder on to the filter paper with a small volume of warm hydrochloric acid (4.4). Wash the residue with warm hydrochloric acid (4.4) until the yellow colour of iron(III) chloride is no longer observed, and then wash in warm water six to eight times. Collect the filtrate and washings in a 500 ml or 600 ml beaker. Evaporate this main solution without boiling to about 70 ml (see NOTE above).

Place the filter paper and residue in a platinum crucible (5.4), dry then char the paper and finally ignite at 750 °C to 800 °C for 1 h. Allow the crucible to cool, moisten the residue with several drops of sulfuric acid (4.7), add about 5 ml of hydrofluoric acid (4.5), and heat gently to remove silica and sulfuric acid (until white fumes are no longer observed).

After cooling, add about 3 g of potassium disulfate (4.20) to the crucible, cover the crucible with a lid, heat gently at first then strongly (dull red) until a clear melt is obtained. Cool, place the crucible and lid in a 250 ml or 300 ml beaker, add about 100 ml of warm water and 5 ml of hydrochloric acid (4.1) and warm to dissolve the melt. Remove the crucible and lid, wash with a small volume of warm water, and add the washings to the solution. Adjust this solution to slight alkalinity by the incremental addition of ammonia solution (4.11). Heat the solution to boiling, continue the boiling for a few minutes and remove from the source of heat.

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

Place the beaker (without the remaining filtrate and washings) under the funnel and dissolve the precipitate on the filter by pouring over it about 10 ml of hot hydrochloric acid (4.3). Wash the filter, first several times using warm hydrochloric acid (4.4), then in warm water until the washings are no longer acid. Collect the filtrate and washings in the beaker and dissolve the remainder of the precipitate on the beaker wall. Combine this solution with the main solution.

Add 5 drops of potassium permanganate solution (4.17), heat the solution to just below the boiling point and maintain at this temperature for 5 min to oxidize any arsenic and organic matter. Evaporate without boiling to about 70 ml, and follow the procedure specified in 7.5.2. (See NOTE above).

7.5.1.2 Fusion – filtration (for samples containing more than 0,05 % mass fraction of vanadium and/or samples not being decomposed by the acid-decomposition)

Place the test portion in an alumina, zirconium or vitreous carbon crucible (5.1), add about 4 g of flux mixture (4.21) and mix thoroughly. Place in a muffle furnace (5.6) at 500 °C ± 10 °C for 30 min (see NOTE 1). Remove from the furnace, heat over a burner to melt the sinter within 30 s and, swirling gently, continue heating allowing a total heating time of 2 min.

NOTE 1 Alternatively, a burner can be used instead of the furnace. In this case, fusion of the test portion is carried out as follows: heat the crucible over a burner, first at low temperature until the contents in the crucible begin to melt, then at high temperature (dull red) while swirling until a clear melt is obtained.

Cool, place the crucible in a 300 ml or 400 ml beaker, add about 100 ml of warm water and heat for a few minutes to dissolve the melt. Remove the crucible, wash and add the washings to the solution. Reserve the crucible. Cool the solution and filter through a close-textured filter paper. Wash the filter twice in sodium hydroxide solution (4.12) and discard the filtrate and washings.

NOTE 2 A filter paper, which is used for a fine precipitate such as barium sulfate, is more suitable than a close-textured filter paper.

Transfer the precipitate on the filter to the original beaker by washing in a jet of water, add 10 ml of hydrochloric acid (4.1), and warm to dissolve the precipitate. Place a 500 ml or 600 ml beaker under the funnel, pour the solution through the original filter paper to dissolve the remaining precipitate. Wash the filter three times in warm hydrochloric acid (4.3), and several times in warm hydrochloric acid (4.4). Finally wash with warm water until the washings are no longer acid. Combine the solution and washings in the 500 ml or 600 ml beaker. Place the reserved crucible in the beaker to dissolve any retained iron, remove the crucible, wash and add the washings to the solution. Evaporate this solution without boiling (see NOTE beneath the second paragraph in 7.5.1.1) to about 70 ml and follow the procedure specified in 7.5.2.

7.5.2 Reduction

7.5.2.1 Method 1: Oxidation of excess titanium(III) chloride with dilute dichromate solution using indigo carmine indicator solution

Maintain the solution obtained in 7.5.1 at 90 °C to 95 °C and wash the cover and inside wall of the beaker in a small amount of hot water. Immediately add tin(II) chloride solution (4.16) dropwise to reduce iron(III), while swirling the solution in the beaker until only a faint yellow tint of the iron(III) chloride solution remains.

It is essential that some iron(III) remain unreduced. If the solution is made colourless by the excessive addition of tin(II) chloride solution, add hydrogen peroxide (4.14) dropwise until the solution changes to a faint yellow tint.

NOTE 1 It is convenient to use dilute potassium dichromate solution as a reference solution for establishing the desired slight yellow tint of the iron solution. The solution is prepared by diluting 5 ml of potassium dichromate standard solution (4.23) to 100 ml with water.

Wash the inside wall of the beaker using a small amount of hot water. Add 3 to 4 drops of indigo carmine solution (4.24) as indicator, then titanium(III) chloride solution (4.19) drop by drop, while swirling the solution, until it turns blue then colourless. Add 2 to 3 drops in excess. Immediately add dilute potassium dichromate solution (4.18) drop by drop, to oxidize the excess of titanium(III) chloride, until the solution changes to a persistent blue colour which lasts for 5 s.

NOTE 2 The temperature of the solution at this time should be > 70 °C; if the temperature is 60 °C to 70 °C, permanence of the blue colour should be extended to 15 s.

Place in a cooling bath for several minutes, then dilute the solution to about 300 ml using cold water. Follow the procedure specified in 7.5.2.3.

NOTE 3 For samples containing $\leqslant$ 0,02 % mass fraction of copper, a cooling bath need not be used.

7.5.2.2 Method 2: Oxidation of excess titanium(III) chloride solution with perchloric acid.

Maintain the solution obtained in 7.5.1 at 90 °C to 95 °C and wash the cover and inside wall of the beaker with a small amount of hot water. Immediately add tin(II) chloride solution (4.16) dropwise to reduce iron(III), while swirling the solution in the beaker until only a light yellow colour remains (see 7.5.2.1, NOTE 1).

Reduce the remaining iron(III) by adding titanium(III) chloride solution (4.19), drop by drop, until the yellow colour has disappeared, then add an additional 3 to 5 drops. Wash the inside wall of the beaker with a small amount of water and reheat rapidly to an incipient boil. Remove from the source of heat and immediately add, all at once, 5 ml of perchloric acid (4.9). Mix well by swirling for about 5 s. Dilute immediately with cold ($<$ 10 °C) water to 300 ml. Cool rapidly to $<$ 15 °C and follow the procedure specified in 7.5.2.3.

7.5.2.3 Titration

Titrate the cold solution obtained in accordance with 7.5.2.1 or 7.5.2.2 with the potassium dichromate standard solution (4.23), while swirling, until the amount of the titrate reaches 10 ml. Add 30 ml of sulfuric acid-orthophosphoric acid mixture (4.10) and continue to titrate with the potassium dichromate standard solution (4.23) using 0,5 ml of the sodium diphenylaminesulfuonate solution (4.25) as an indicator. The end point is reached when the green colour of the solution changes to a bluish green and a final drop of the titrant imparts a violet colour.

NOTE Barium diphenylaminesulfonate can be used as an indicator.

The ambient temperature of the potassium dichromate solution should be noted. If this differs by more than 1 °C from the temperature at which it was prepared (20 °C), make the appropriate volumetric correction: 0,02 % relative for each 1 °C of difference (for example, the titre should be decreased when the ambient temperature during the titration is higher than the temperature during preparation of the standard volumetric solution). A correction for the temperature difference, if any, is essential.

7.5.2.4 Blank test

Determine the blank test value (see 7.2) using the same amounts of all reagents and following all the steps of the procedure. Immediately before reduction (see 7.5.2) with tin(II) chloride solution (4.16) add, by means of a one-mark pipette, 1,00 ml of iron standard solution (4.22) and titrate the solution as described in 7.5.2.3. It is not necessary to add the first 10 ml of potassium dichromate standard solution. Record the volume of this titration as V_0. The blank test value of this titration, V_2, is calculated as $V_2 = V_0 - 1{,}00$.

NOTE 1 In the absence of iron, the sodium diphenylaminesulfonate indicator does not react with the dichromate solution. The addition of the iron solution is therefore necessary to promote indicator response in the blank solution and thus allow a suitable correction for the blank in terms of its equivalent in millilitres of the potassium dichromate standard solution.

NOTE 2 The 1 ml one-mark pipette should previously be calibrated by weighing the mass of water delivered and converting to volume.

8 Expression of results

8.1 Calculation of total iron content

The total iron content, as a percentage mass fraction, is calculated from the following equation:

$$w_{\text{Fe}} = \frac{V_1 - V_2}{m} \times 0{,}005\,584\,7 \quad 100 \quad K \qquad \times \qquad \times \tag{1}$$

where

V_1	is the volume, in millilitres, of potassium dichromate standard solution (4.23) used for the titration of the test sample;
V_2	is the volume, in millilitres, of potassium dichromate standard solution used for the titration in the blank test, corrected for the iron added in 7.5.2.4;
m	is the mass, in grams, of the test portion;
0,005 584 7	is a multiple of the atomic mass of iron;
K	is 1,00 for predried test samples (6.2.3), and for air-equilibrated test samples (6.2.2) is the conversion factor found from the equation:

$$K = \frac{100}{100 - A} \tag{2}$$

where A is the hygroscopic moisture content, as a percentage mass fraction, determined in accordance with ISO 2596.

8.2 General treatment of results

8.2.1 Repeatability and permissible tolerance

The precision of this analytical method is expressed by the following regression equations (see Annex B):

$$R_{\text{d}} = 0{,}003\,4\,X - 0{,}018\,7 \tag{3}$$

$$P = 0{,}37 \tag{4}$$

$$\sigma_{\text{d}} = 0{,}001\,2\,X - 0{,}006\,6 \tag{5}$$

$$\sigma_{\text{L}} = 0{,}12 \tag{6}$$

8.2.2 Determination of analytical result

Having computed the independent duplicate results according to Equation (1), compare them with the independent duplicate limit, R_{d}, using the procedure given in Annex A, and obtain the final laboratory result μ (see 8.2.5).

8.2.3 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 procedure described in 8.2.2.

Compute the following quantity:

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \qquad (7)$$

where

μ_1 is the final result reported by laboratory 1;

μ_2 is the final result reported by laboratory 2;

$\mu_{1,2}$ is the mean of the final results.

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

8.2.4 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) (see NOTE 1 of 7.2). Calculate the analytical result, μ, for the CRM/RM using the procedures in 8.1 and 8.2, and compare it with the reference or certified value A_c. There are two possibilities:

a) $\mu_\mathrm{c} - A_\mathrm{c} < C$

in which case the difference between the reported result and the certified/reference value is statistically insignificant.

b) $\mu_\mathrm{c} - A_\mathrm{c} > C$

in which case the difference between the reported result and the certified/reference value is statistically significant.

where

μ_c is the final result for the certified reference material;

A_c is the certified/reference 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.

For a CRM certified by an interlaboratory test programme

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

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

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

8.2.5 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 according to Rule A in ISO 31-0:1992, Annex B.3. 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 a 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.

8.3 Oxide factors

$$w_{\text{Fe}_2\text{O}_3} = 1{,}430 w_{\text{Fe}}$$

$$w_{\text{FeO}} = 1{,}286 w_{\text{Fe}}$$

$$w_{\text{Fe}_3\text{O}_4} = 1{,}382 w_{\text{Fe}}$$

9 Test report

The test report shall include the following information:

a) name and address of the testing laboratory;

b) date of issue of the test report;

c) reference to this part of ISO 2597, i.e., ISO 2597-2:2008 (including reference of the method used);

d) details necessary for the identification of the sample;

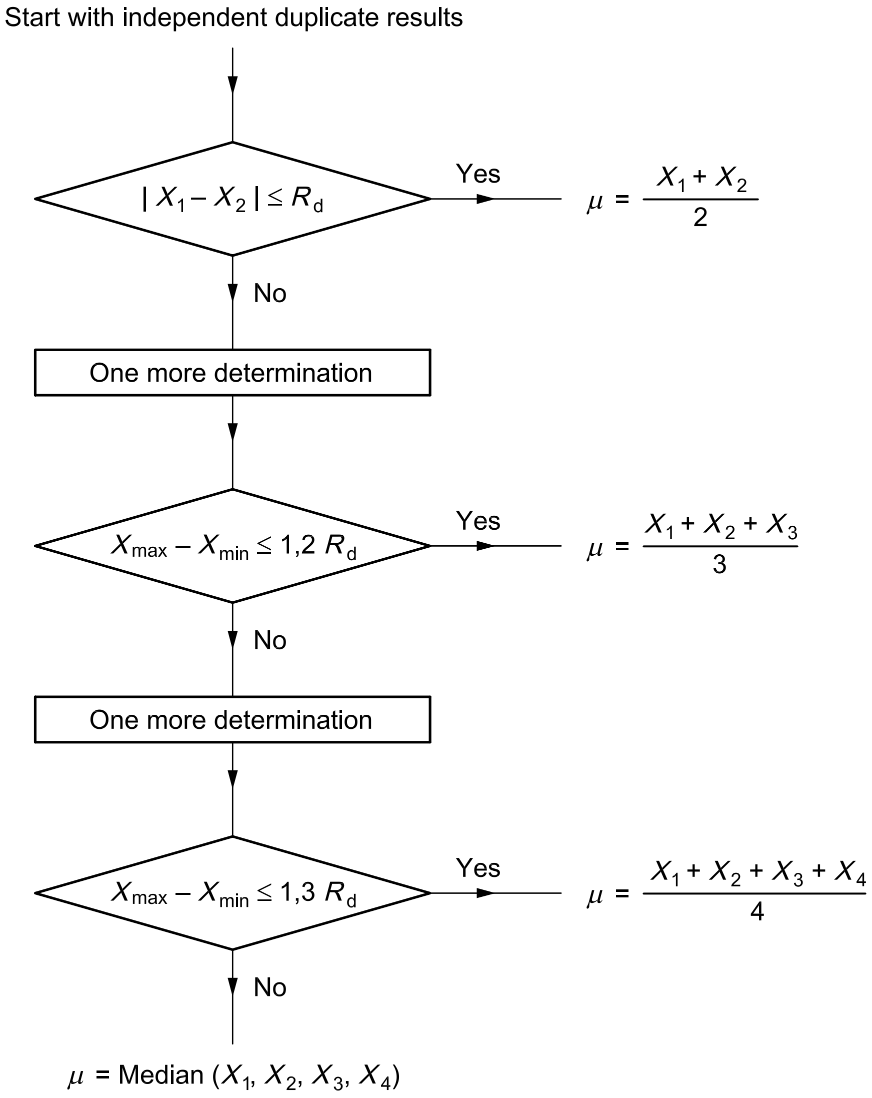
e) result of the analysis;

f) reference number of the result;

g) any characteristics noticed during the determination and any operations not specified in this part of ISO 2597 which may have had an influence on the results, either for the test sample or the certified reference material(s).

Annex A
(normative)

Flowsheet of the procedure for the acceptance of analytical values for test samples



Annex B
(informative)

Derivation of precision statements

The precision statements in 8.2.1 were obtained by statistical evaluation of the results of international analytical trials carried out in 1999/2000 on eight iron samples (see Table B.1) involving 19 laboratories in 10 countries.

Table B.1 — Total iron contents of test samples

Sample	**Iron content** % (mass fraction)
JSS 009-2	69,63
JSS 805-1	68,04
JSS 831-1	57,03 [a]
JSS 851-4	56,16
ASCRM 004	62,50
ECRM 676-1	39,76 [a]
ECRM 680-1	59,98
JK 29	71,09 [a]

[a] Fusion method applied.

NOTE 1 A report of the international trials and a statistical analysis of the results (documents ISO/TC 102/SC2 N1424 and ISO/TC 102/SC2 N1421,) are 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.

Annex C
(normative)

Procedure of the Japanese weighing method

Dry a weighing bottle (5.3) and a well-fitting lid at a temperature of 105 °C ± 2 °C and cool in desiccator. Take from the sample a test portion of 0,4 g and transfer to the dried weighing bottle (see NOTE). Spread the test sample evenly in the weighing bottle.

NOTE The test sample should be taken in multiple increments in such a way that it is representative of the entire contents of the container.

Dry the open weighing bottle with the test portion and the lid in a laboratory oven for 2 h at 105 °C ± 2 °C. Close the weighing bottle with the lid, transfer to a desiccator and cool to room temperature (20 min to 30 min). Slightly remove the lid and quickly replace it again, then weigh the bottle with the lid on to the nearest 0,000 1 g (m_1).

Quickly transfer the test portion to a beaker or a crucible described in 7.5.1 and then weigh the weighing bottle with the lid on (m_2).

The mass of the test portion, m, is the difference between the two weighings, m_1 and m_2.

Bibliography

[1] ISO 3696, *Water for analytical laboratory use — Specification and test methods*

[2] 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*

[3] ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*

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