
**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 2597-1:2006(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 2597-1 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This second edition cancels and replaces the first edition (ISO 2597-1:1994), which has been editorially revised.

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 method after titanium(III) chloride reduction*
- *Part 3: Calculation method*

Introduction

This part of ISO 2597 has been updated to correct errors in the presentation of statistical information in the previous edition.

This is considered to be an editorial revision.

Iron ores — Determination of total iron content —

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

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 issues associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

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 % (mass fraction) and 72 % (mass fraction) in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

NOTE An equivalent International Standard that does not use mercury(II) chloride is ISO 9507:1990, *Iron ores — Determination of total iron content — Titanium(III) chloride reduction methods*.

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 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 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — One-mark 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*

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

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

3 Principle

The test portion is decomposed by one of the following methods:

- a) For samples containing not more than 0,05 % (mass fraction) vanadium, 0,1 % (mass fraction) molybdenum or 0,1 % (mass fraction) 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.

The melt is dissolved in hydrochloric acid, iron is precipitated with ammonia solution, the precipitate is re-dissolved in hydrochloric acid and this solution is added to the main solution.

- b) For samples containing more than 0,05 % (mass fraction) vanadium: fusion with alkali, the melt being leached with water and filtered, the filtrate being discarded. The residue is dissolved in hydrochloric acid.

Trivalent iron in the solution is reduced using tin(II) chloride. Excess reductant is oxidized with mercury(II) chloride.

The reduced iron is titrated with potassium dichromate solution, using sodium diphenylaminesulfonate as the 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**, $\rho = 1,16$ g/ml to 1,19 g/ml.
- 4.2 **Hydrochloric acid**, $\rho = 1,16$ g/ml to 1,19 g/ml, diluted 1 + 1.
- 4.3 **Hydrochloric acid**, $\rho = 1,16$ g/ml to 1,19 g/ml, diluted 1 + 2.
- 4.4 **Hydrochloric acid**, $\rho = 1,16$ g/ml to 1,19 g/ml, diluted 1 + 10.
- 4.5 **Hydrochloric acid**, $\rho = 1,16$ g/ml to 1,19 g/ml, diluted 1 + 50.
- 4.6 **Sulfuric acid**, $\rho = 1,84$ g/ml.
- 4.7 **Sulfuric acid**, $\rho = 1,84$ g/ml, diluted 1 + 1.
- 4.8 **Hydrofluoric acid**, $\rho = 1,13$ g/ml, [40 % (mass fraction)], or $\rho = 1,19$ g/ml, [48 % (mass fraction)].
- 4.9 **Orthophosphoric acid**, $\rho = 1,7$ g/ml.
- 4.10 **Ammonia solution**, $\rho = 0,90$ 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 % (mass fraction) (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 after it has begun to agglomerate.
- 4.13 **Potassium pyrosulfate** ($\text{K}_2\text{S}_2\text{O}_7$), 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 ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) 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 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 % (mass fraction)] 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 has dissolved. Cool and oxidize with 5 ml of hydrogen peroxide [30 % (volume fraction)] 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, $c(\text{K}_2\text{Cr}_2\text{O}_7) = 0,016\ 67\ \text{mol/l}$.

Pulverize about 6 g of potassium dichromate standard reagent [minimum purity 99,9 % (mass fraction)] 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 ($\text{C}_6\text{H}_5\text{NHC}_6\text{H}_4\text{SO}_3\text{Na}$) in a small volume of water and dilute to 100 ml.

Store this solution in a brown glass bottle.

5 Apparatus

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

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 $500\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ and $800\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$.

6 Sampling and samples

6.1 Laboratory sample

For the 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 minus 160 μm .

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

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 % (mass fraction);
- b) processed ores containing metallic iron;
- c) natural or processed ores in which the sulfur content is higher than 0,2 % (mass fraction).

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 $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, 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 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).

NOTE For samples of iron content higher than 68 % (mass fraction), weigh approximately 0,38 g.

If predried test samples are being 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).

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.

NOTE 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 % (mass fraction) vanadium, 0,1 % (mass fraction) molybdenum or 0,1 % (mass fraction) 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 note and second-last paragraph.)

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 with warm water six to eight times. 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.

For samples that are difficult to decompose with hydrochloric acid alone, tin(II) chloride may be used as follows.

Decompose the test portion as much as possible by treatment with hydrochloric acid according to the procedure outlined in the first sentence of this subclause, then add 10 ml of tin(II) chloride solution (4.16) and heat in the low-temperature zone of the hotplate until the yellow colour of iron(III) chloride appears in the solution. Again add a small excess of tin(II) chloride solution (4.16) and continue the heating. If necessary, repeat the addition of tin(II) chloride solution (4.16) until the test portion is completely decomposed. Add potassium permanganate solution (4.18), drop by drop, to oxidize the excess of tin(II) chloride and restore the yellow colour of iron(III) chloride. Proceed as specified in 7.5.1.1, second sentence.

Samples that are more easily decomposed by roasting should be processed as follows.

Place the test portion (7.2) in a porcelain crucible (5.3), cover with the lid and roast at 700 °C to 750 °C for about 30 min. Allow the crucible to cool and transfer the test portion, as far as possible in solid form, into a 300 ml beaker. Dissolve the remainder in the crucible in hydrochloric acid (4.1) and transfer the solution to the beaker. Add hydrochloric acid (4.1) up to a total volume of 30 ml, and proceed as specified in the first eight paragraphs of this subclause.

7.5.1.2 Fusion acidification [for samples containing more than 0,05 % (mass fraction) vanadium]

Place the test portion (7.2) in an alkali-resistant crucible (5.1), add about 4 g of fusion mixture [1 + 2 mixture of sodium carbonate (4.11) and sodium peroxide (4.12)], mix thoroughly and place in the muffle furnace (5.5) maintained at $500^{\circ}\text{C} \pm 10^{\circ}\text{C}$ for 30 min. Remove from the furnace, heat over a burner to melt the sinter within 30 s and, swirling gently, continue heating to allow a total heating time of 2 min. Allow the melt to cool, then place the crucible in a 300 ml beaker, add about 100 ml of warm water, and boil for a few minutes to leach the melt. Take out the crucible, wash it with warm water and reserve it. Cool the solution and filter through a filter paper of dense texture. Wash the filter twice with sodium hydroxide solution (4.15) and discard the filtrate and washings.

Transfer the precipitate on the filter into the original beaker by washing with water, add 10 ml of hydrochloric acid (4.1), and warm to dissolve the precipitate. Dissolve the remaining precipitate in the reserved crucible with hot hydrochloric acid (4.2) and wash the crucible with hot water, transferring the solution and crucible washings to the original beaker. Place a 500 ml beaker under the filter and wash the filter three times with warm hydrochloric acid (4.3), several times with warm hydrochloric acid (4.5), and finally with warm water until the washings are no longer acid. Combine the solution and washings in the 500 ml beaker, evaporate this solution, without boiling, to about 30 ml and follow the procedure specified in 7.5.2.

7.5.2 Reduction

Heat the solution, now containing all the iron, to just below the boiling point and add three to five drops of potassium permanganate solution (4.18). Maintain at this temperature for 5 min to oxidize any arsenic and organic matter, and evaporate to about 30 ml. Wash the cover and inside wall of the beaker with a small amount of hot hydrochloric acid (4.4). Immediately add tin(II) chloride solution (4.16), drop by drop, while swirling the solution in the beaker. When the yellow colour of the iron(III) chloride has disappeared, add an additional drop of the tin(II) chloride solution (4.16) and cool the solution in a water cooler. Add, all at once, 8 ml of mercury(II) chloride solution (4.17), mix gently, and allow the solution to stand for 5 min.

7.5.3 Titration

Dilute the solution to about 300 ml with cold water, add 30 ml of sulfuric acid-orthophosphoric acid mixture (4.14), and titrate with potassium dichromate standard volumetric solution (4.20), using 5 drops of sodium diphenylaminesulfonate solution (4.21) as indicator. The endpoint is obtained when the green colour of the solution changes to bluish green and a final drop of the titrant imparts a violet colour.

The ambient temperature (t_2) of the potassium dichromate solution should be noted. If this differs by more than 1°C from the temperature at which it was prepared (t_1), make a volumetric correction such as that given in ISO 4787:1984, *Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity*. For example, the titre should be decreased if 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.

7.5.4 Blank test

Determine the blank test value (7.4), using the same amounts of all reagents and following all the steps of the procedure. Immediately before reduction (7.5.2) with tin(II) chloride solution (4.16), add 1,00 ml of iron standard solution (4.19) with a one-mark pipette and titrate the solution as described in 7.5.3. Record the volume of this titration (V_0). The blank test value of this titration (V_2) is calculated using $V_2 = V_0 - 1,00$.

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

NOTE In the absence of iron, the diphenylaminesulfonate indicator does not react with the potassium dichromate solution. The addition of 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 potassium dichromate standard volumetric solution.

8 Expression of results

8.1 Calculation of total iron content

The total iron content, w_{Fe} , as a percentage by mass (see last 2 paragraphs and Equation 3), is calculated using the equation

$$w_{\text{Fe}} = \frac{(V_1 - V_2) [1 - (t_2 - t_1) \times 0,000 2]}{m} \times 0,005 584 7 \times 100 \times K \quad (1)$$

where

V_1 is the volume, in millilitres, of potassium dichromate standard volumetric solution (4.20) used for the determination;

V_2 is the volume, in millilitres, of potassium dichromate standard volumetric solution (4.20) used for the blank test, corrected for the addition of iron solution;

t_1 is the temperature, in degrees Celsius, at which the potassium dichromate was prepared;

t_2 is the temperature, in degrees Celsius, at which the potassium dichromate was used;

m is the mass, in grams, of the test portion (7.2);

K is 1,00 for predried test samples (6.2.2), and, for air-equilibrated test samples (6.2.1), is the conversion factor found from the equation

$$K = \frac{100}{100 - A} \quad (2)$$

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

The atomic mass of iron $A_r(\text{Fe})$ should be verified by inserting the final test result into the following equation:

$$A_r(\text{Fe}) = \frac{w_{\text{Fe}} \times 100m}{K(V_1 - V_2)[1 - (t_2 - t_1) \times 0,000\ 2]} \quad (3)$$

If the difference between the theoretical atomic mass of iron (55,847) and the calculated value is greater than 0,01, the reason for the discrepancy should be examined and the test result should be amended accordingly. If a discrepancy cannot be identified, the test result should be rejected and the determination repeated.

8.2 General treatment of results

8.2.1 Repeatability and permissible tolerance

The precision of this analytical method is expressed by the following equations ¹⁾:

$$\sigma_d = 0,084 \quad (4)$$

$$\sigma_L = 0,178 \quad (5)$$

$$R_d = 0,235 \quad (6)$$

$$P = 0,53 \quad (7)$$

where

σ_d is the independent duplicate standard deviation;

σ_L is the between-laboratories standard deviation;

R_d is the independent duplicate limit;

P is the permissible tolerance between laboratories.

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 described in Annex A.

8.2.3 Between-laboratories precision

The 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:

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \quad (8)$$

1) Additional information is given in Annex B.

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 $|\mu_1 - \mu_2| \leq P$, 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). 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

μ_c is the final result for the certified reference material;

A_c is the certified value for the CRM or the reference value for the RM;

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

Certified reference materials used for this purpose should be prepared and certified in accordance with ISO Guide 35:2006, *Reference materials — General and statistical principles for certification*.

For a CRM certified by an interlaboratory test programme

$$C = 2 \left[\sigma_L^2 + \frac{\sigma_d^2}{n} + V(A_c) \right]^{1/2}$$

where

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

$V(A_c)$ is the variance of the certified value A_c .

For a CRM certified by only one laboratory

$$C = 2 \left[\sigma_L^2 + \frac{\sigma_d^2}{n} \right]^{1/2}$$

This type of CRM 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 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 the figure 0 is 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 in percent

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

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

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

9 Test report

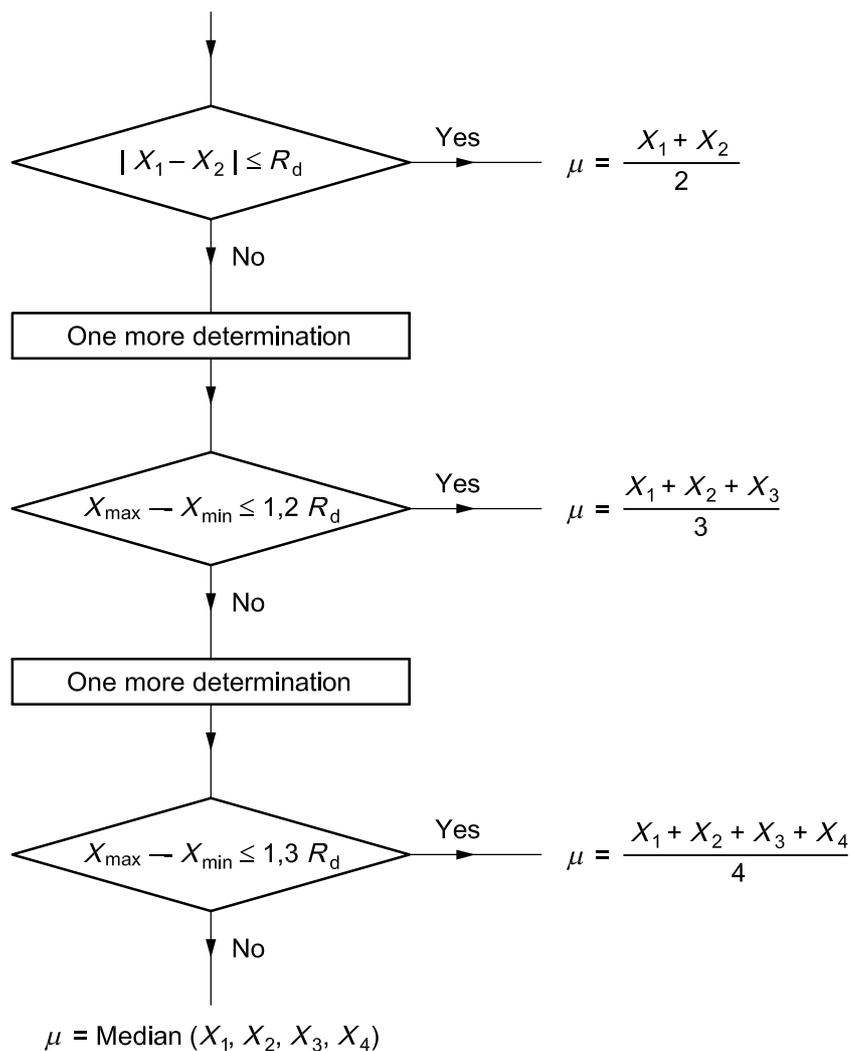
The test report shall include the following information:

- a) name and address of the testing laboratory;
- b) date of issue of the test report;
- c) a reference to this part of ISO 2597;
- 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 result, for either the test sample or the certified reference material(s).

Annex A (normative)

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

Start with independent duplicate results



Annex B (informative)

Derivation of precision statements

The precision statements in 8.2.1 were derived from the results of international analytical trials carried out in 1966 to 1967 on eight iron ore samples, involving 30 laboratories in various countries.

The test samples are listed in Table B.1.

Table B.1 — Total iron contents of test samples

Sample	Iron content % (mass fraction)
Sweden-2	64,8
Sweden-7	61,7
UK Sinter	35,9
Canadian	65,3
Minette	31,8
Philippine iron sand	60,6
Krivoj Rog	47,4
Marcona	62,7

NOTE 1 A report of the international trials and a statistical analysis of the results (Document ISO/TC 102/SC 2 N147E, November 1968) is available from the Secretariat of ISO/TC 102/SC 2.

NOTE 2 The statistical analysis was performed in accordance with the principles given in ISO 5725-2:1994, *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*.

Annex C (informative)

Decontamination of the waste solutions from the analysis

To avoid discharging to the environment the mercury contained in the waste solutions remaining after the determination of iron, such solutions should be collected and processed for the removal of the mercury that they contain.

A suitable treatment system may be constructed as follows.

Three 10 litre plastics bottles are connected as illustrated in Figure C.1. (The expansion chamber incorporated in the connecting lines minimizes the risk of pressure increase in the case of a vigorous reaction.) The first two bottles each contain 3 kg of aluminium or iron rods to induce the electrochemical precipitation of mercury.

The solution discharged from the third bottle is fed to a neutralization vessel before being finally discarded. Accumulated mercury sludge is removed from time to time and concentrated by decantation before being transferred to a recovery plant. The aluminium or iron rods are replaced as necessary.

(Reference: ANSMANN, W. Arch. Eisenhüttenw. 53(10) 1982: p. 390.)

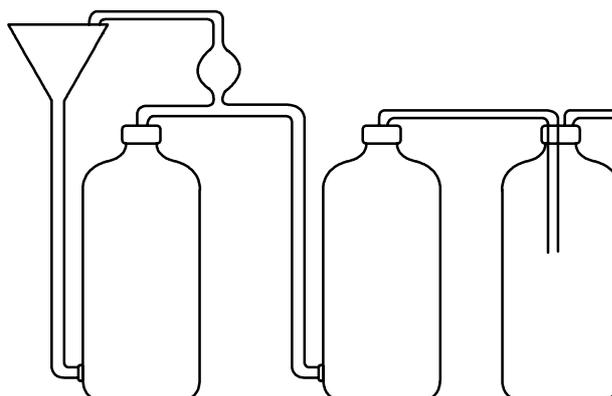


Figure C.1 — Apparatus for the removal of mercury from waste solutions

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