
International Standard



2597

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Iron ores — Determination of total iron content — Titrimetric methods

Minerais de fer — Dosage du fer total — Méthodes titrimétriques

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 2597 was prepared by Technical Committee ISO/TC 102, *Iron ores*.

ISO 2597 was first published in 1973. This second edition cancels and replaces the first edition of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Iron ores — Determination of total iron content — Titrimetric methods

1 Scope and field of application

This International Standard specifies two titrimetric methods for the determination of the total iron content in iron ores using potassium dichromate after reduction of the trivalent iron by tin(II) chloride (method 1) or hydrogen sulfide (method 2).

Both methods are applicable to a concentration range of 30 to 72 % (m/m) of iron in natural iron ores, and iron ore concentrates and agglomerates, including sinter products. For an unknown ore, it is recommended that the iron content be determined by both methods to check that the tin(II) chloride reduction method is satisfactory.

2 References

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

ISO 3081, *Iron ores — Increment sampling — Manual method.*¹⁾

ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method.*²⁾

ISO 3083, *Iron ores — Preparation of samples — Manual method.*³⁾

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

3 Principle

3.1 Method 1: Reduction by tin(II) chloride

Decomposition of the test portion:

- a) for samples containing less than 0,1 % (m/m) vanadium, 0,1 % (m/m) molybdenum or 0,1 % (m/m) copper: by treatment with hydrochloric acid, the residue

being filtered off, ignited, treatment with hydrofluoric and sulfuric acids to remove silica and fused 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 0,1 % (m/m) vanadium: by fusion with alkali, the melt being leached with water and filtered, the filtrate being discarded. 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 diphenylaminesulfonate as indicator.

3.2 Method 2: Reduction by hydrogen sulfide

Decomposition of the test portion by treatment with hydrochloric acid, the residue being filtered off, ignited, treated with hydrofluoric and sulfuric acids to remove silica, and fused with potassium pyrosulfate. Dissolution of the melt in the main solution, addition of sulfuric acid and evaporation to expel all chlorides. Dissolution of the sulfates in water.

After adjustment of the acidity, reduction of the trivalent iron in the solution, using hydrogen sulfide. Boiling to remove excess hydrogen sulfide. After cooling, titration of the reduced iron with potassium dichromate solution using sodium diphenylaminesulfonate as indicator.

4 Reagents

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

1) At present at the stage of draft. (Revision of ISO 3081-1973.)

2) At present at the stage of draft.

3) At present at the stage of draft. (Revision of ISO 3083-1973.)

| | Method |
|--|---------|
| 4.1 Hydrochloric acid , ρ 1,16 to 1,19 g/ml. | 1 and 2 |
| 4.2 Hydrochloric acid , ρ 1,16 to 1,19 g/ml, diluted 1 + 1. | 1 |
| 4.3 Hydrochloric acid , ρ 1,16 to 1,19 g/ml, diluted 1 + 2. | 1 |
| 4.4 Hydrochloric acid , ρ 1,16 to 1,19 g/ml, diluted 1 + 10. | 1 |
| 4.5 Hydrochloric acid , ρ 1,16 to 1,19 g/ml, diluted 1 + 50. | 1 and 2 |
| 4.6 Sulfuric acid , ρ 1,84 g/ml, diluted 1 + 1. | 1 and 2 |
| 4.7 Hydrofluoric acid , 40 % (m/m), ρ 1,13 g/ml, or 48 % (m/m), ρ 1,19 g/ml. | 1 and 2 |
| 4.8 Ammonia solution , ρ 0,90 g/ml. | 1 |
| 4.9 Hydrogen sulfide. | 2 |
| CAUTION — Hydrogen sulfide is highly toxic and shall be used only in a fume cupboard. | |
| The hydrogen sulfide may be taken from a cylinder. Alternatively a Kipp generator can be used. | |
| NOTE — The hydrogen sulfide gas from a Kipp generator should be passed through a water trap to remove any entrained iron salts. | |
| 4.10 Sodium carbonate (Na_2CO_3), anhydrous. | 1 |
| 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). | |
| 4.11 Sodium peroxide (Na_2O_2), dry powder. | 1 |
| 4.12 Potassium pyrosulfate ($\text{K}_2\text{S}_2\text{O}_7$), fine powder. | 1 and 2 |
| 4.13 Sulfuric acid-phosphoric acid mixture. | 1 and 2 |
| Cautiously pour 150 ml of sulfuric acid (ρ 1,84 g/ml) into about 300 ml of water while stirring, cool in a water-bath, add 150 ml of phosphoric acid (ρ 1,70 g/ml) and dilute with water to 1 litre. | |
| 4.14 Tin(II) chloride , 100 g/l solution. | 1 |
| Place 200 ml of the hydrochloric acid (4.1) into a 1 litre beaker and add 100 g of crystalline tin(II) chloride dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in small increments, while heating the solution on a water-bath. | |
| Cool the solution and dilute with water to 1 litre. | |

Method

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.

4.15 Sulfuric acid-hydrogen sulfide wash solution. 2

To 900 ml of water, add 10 ml of sulfuric acid (ρ 1,84 g/ml). Dilute to 1 litre and pass a rapid stream of hydrogen sulfide gas through the solution for at least 10 min.

4.16 Mercury(II) chloride (HgCl_2), 50 g/l solution. 1

4.17 Potassium permanganate (KMnO_4), 30 g/l solution. 1

4.18 Sodium diphenylaminesulfonate solution. 1 and 2

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 with water to 100 ml.

Store the solution in a brown glass bottle.

4.19 Ammonium iron(II) sulfate, approximately 0,1 mol/l solution. 1 and 2

Dissolve 40 g of ammonium iron(II) sulfate hexahydrate [$\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$] in sulfuric acid (ρ 1,84 g/ml, diluted 1 + 19).

Transfer to a 1 000 ml one-mark volumetric flask and dilute to the mark with sulfuric acid (ρ 1,84 g/ml, diluted 1 + 19). Standardize the solution against the potassium dichromate solution (4.21) by taking a 25 ml aliquot of the solution and titrating as shown in 7.5.3.

4.20 Sodium hydroxide, 20 g/l solution. 1

4.21 Potassium dichromate, standard volumetric solution, $c(\text{K}_2\text{Cr}_2\text{O}_7) = 0,01667$ mol/l. 1 and 2

Pulverize about 6 g of potassium dichromate standard reagent in an agate mortar, dry in an air-bath at 140 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 with water to exactly 1 000 ml.

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

5 Apparatus

Ordinary laboratory apparatus, and

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

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

5.3 Cylinder of hydrogen sulfide or Kipp generator (method 2).

5.4 Muffle furnace, capable of being maintained at between 500 ± 10 and 800 ± 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 with 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.

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 with 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) processed ores containing metallic iron;
- b) natural or processed ores in which the sulfur content is higher than 0,2 % (m/m);
- c) natural or processed ores in which the content of combined water is higher than 2,5 % (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 manner that it is representative of the whole contents of the container. Dry the test sample at 105 ± 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 accord with annex A, independently, on one test sample (6.2).

NOTE — The expression "independently" implies that the repetition of the procedure shall be carried out at a different time.

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). (See note 1.)

When 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.

When 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.

NOTES

- 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 would become necessary.
- 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

When 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 (7.4), for the determination of 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 (6.2) using a non-magnetic spatula (5.2).

NOTES

- 1 For samples of iron content higher than 68 % (m/m), weigh approximately 0,38 g.
- 2 When 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.

7.5 Determination — Method 1: Reduction by tin(II) chloride

7.5.1 Decomposition of the test portion

7.5.1.1 For samples containing less than 0,1 % (m/m) vanadium, 0,1 % (m/m) molybdenum and 0,1 % (m/m) copper

Place the test portion (7.4) in a 300 ml beaker, add 30 ml of the 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 1 and 2.)

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 the 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 but without boiling (main solution).

Place the filter paper and the residue in a platinum crucible, dry, char the paper and finally ignite at 750 to 800 °C. Allow the crucible to cool. Moisten the residue in the crucible with the sulfuric acid solution (4.6), add about 5 ml of the hydrofluoric acid (4.7), and heat gently to remove silica and sulfuric acid.

Add to the cool crucible 2 g of the potassium pyrosulfate (4.12), 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 the hydrochloric acid (4.1), and heat gently to dissolve the melt. Rinse and remove the platinum crucible.

Adjust this solution to slight alkalinity by the addition of ammonia solution (4.9), boil the solution for a few minutes and remove 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 over it 10 ml of the hydrochloric acid solution (4.3); wash the filter, first six to eight times with the warm hydrochloric acid solution (4.5), then twice with hot water, and follow the procedure given under 7.5.2.

NOTES

1 For samples which 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 7.5.1.1, first paragraph, first sentence, then add 10 ml of the tin(II) chloride solution 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 and continue the heating. If necessary, repeat the addition of tin(II) chloride solution until the test portion is completely decomposed. Add the potassium permanganate solution (4.17), 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, first paragraph, second sentence.

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

Place the test portion (7.4) in a porcelain crucible of 30 ml capacity, cover with the lid and roast at 700 to 750 °C for about 30 min. Allow the crucible to cool, 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 7.5.1.1 and subsequent subclauses.

7.5.1.2 For samples containing more than 0,1 % (m/m) vanadium

Place the test portion (7.4) in an alkali-resistant crucible (5.1), add about 4 g of fusion mixture [1 + 2 mixture of sodium carbonate (4.10) and sodium peroxide (4.11)] mix thoroughly and place in the muffle furnace (5.4) maintained at 500 ± 10 °C for 30 min. Remove from the furnace and heat on a burner to the melting point. Continue heating just above the melting point for approximately 1 1/2 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 with warm water, and reserve the crucible. Cool the solution and filter through a filter paper of dense texture. Wash the filter six to eight times with the sodium hydroxide solution (4.20) and discard the filtrate and washings.

Transfer the precipitate on the filter into the original beaker by washing with water, add 10 ml of the hydrochloric acid (4.1), and warm to dissolve the precipitate. Dissolve the remaining precipitate in the reserved crucible with the hot hydrochloric acid solution (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 the warm hydrochloric acid solution (4.3), several times with the warm hydrochloric acid solution (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 the potassium permanganate solution (4.17). 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 the hot hydrochloric acid solution (4.4). Immediately add the tin(II) chloride solution (4.14), drop by drop, while swirling the liquid in the beaker. When the yellow colour of the iron(III) chloride has disappeared, add an additional drop of the tin(II) chloride solution and cool the solution in a water cooler. Add all at once 8 ml of the mercury(II) chloride solution (4.16), 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 the sulfuric acid-phosphoric acid mixture (4.13), and titrate with the potassium dichromate solution (4.21), using 5 drops of the sodium diphenylaminesulfonate solution (4.18) as indicator. The end-point is obtained when the green colour of the solution changes to bluish green and a final drop of the titrant imparts a violet colour.

NOTE — Note the ambient temperature of the potassium dichromate solution. If this differs by more than 1 °C from the temperature at which it was prepared, make the appropriate volumetric correction: 0,02 % relative of each 1 °C of difference. (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.)

7.5.4 Blank test

Determine the value of the blank test concurrently with the test determination, using the same amounts of all reagents and following all the steps of the procedure. Immediately before titrating with the potassium dichromate solution (4.21), add 1,0 ml of the ammonium iron(II) sulfate solution (4.19) and make the appropriate correction for the iron addition.

NOTE — In the absence of iron, the diphenylaminesulfonate indicator does not react with the dichromate solution. The addition of ammonium iron(II) sulfate therefore is 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 solution (4.21).

7.6 Determination — Method 2: Reduction by hydrogen sulfide

7.6.1 Decomposition of the test portion

Place the test portion (7.4) in a 300 ml beaker, add 30 ml of the 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 the note.)

Wash the watch-glass with a jet of water, and dilute to 50 ml with warm water. Filter the insoluble residue on a close-texture paper. Wash the residue with the warm hydrochloric acid solution (4.5), until the yellow colour of iron(III) chloride is no longer observed. Then wash with warm water six to eight times. Collect the filtrate and washings in a 600 ml beaker (main solution).

Place the filter paper and residue in a platinum crucible, dry and ignite at 750 to 800 °C. Allow the crucible to cool, moisten the residue with sulfuric acid solution (4.6), add about 5 ml of the hydrofluoric acid (4.7), and heat gently to remove silica and sulfuric acid. Add to the cool crucible 2 g of the potassium pyrosulfate (4.12), heat gently at first then strongly until a clear melt is obtained. Cool the melt, place the crucible in a 250 ml beaker, add about 25 ml of water and about 5 ml of the hydrochloric acid (4.1), and warm to dissolve the melt. Remove and wash the crucible, and add this solution to the main solution.

NOTE — Samples which are more easily decomposed by roasting should be processed as follows.

Place the test portion (7.4) in a porcelain crucible of 30 ml capacity, cover, and roast at 700 to 750 °C for about 30 min. Allow to cool, wash the test portion in a 300 ml beaker, and proceed as directed in 7.6.1.

7.6.2 Reduction

Add 10 ml of the sulfuric acid (4.6) to the main solution and evaporate to copious fumes of sulfur trioxide. (See the note.)

Dilute to about 350 ml, and heat until a clear solution is obtained. Pass the hydrogen sulfide (4.9) through the warm solution for 15 min at a rate of about 1 l/min. Digest at about 60 °C for a further 15 min, filter through a small filter paper into a 1 litre conical flask and wash 15 times with the hydrogen sulfide-sulfuric acid wash solution (4.15). Add to the filtrate 10 ml of the sulfuric acid solution (4.6) and boil until free from hydrogen sulfide as tested by lead acetate paper. Boil for an additional 10 min, fit a Bunsen valve to the flask, then cool to room temperature.

NOTE — If the sample contains much calcium, prolonged fuming with sulfuric acid may lead to the formation of calcium salts that are difficult to dissolve. Therefore, in the presence of considerable calcium, fume just long enough to expel the chlorides.

7.6.3 Titration

Dilute to about 300 ml with water, add 30 ml of the sulfuric acid-phosphoric acid mixture (4.13) and titrate this solution with potassium dichromate solution (4.21), using 5 drops of the sodium diphenylaminesulfonate solution (4.18) as indicator. The end-point is obtained when the green colour of the solution changes to bluish green and a final drop of the titrant imparts a violet colour.

NOTE — Note the ambient temperature of the potassium dichromate solution. If this differs by more than 1 °C from the temperature at which it was prepared, make the appropriate volumetric correction: 0,02 % relative for each 1 °C of difference. (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.)

7.6.4 Blank test

Determine the value of the blank test concurrently with the test determination, using the same amounts of all reagents and following all the steps of the procedure. Immediately before titrating with the potassium dichromate solution (4.21), add 1,0 ml of ammonium iron(II) sulfate solution (4.19) and make the appropriate correction for the iron addition.

NOTES

1 In the absence of iron, the diphenylaminesulfonate indicator does not react with the dichromate solution. The addition of ammonium iron(II) sulfate 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 solution (4.21).

2 In order to comply with local environmental specifications it may be necessary to remove mercury from the waste solutions before discharge to effluent drains. A suggested procedure is given in annex D.

8 Expression of results

8.1 Calculation of total iron content

The total iron content, w_{Fe} , as a percentage by mass, is calculated to four decimal places using the equation

$$w_{Fe} (\%) = \frac{V_1 - V_2}{m} \times 0,005\,584\,7 \times 100 \times K \quad \dots (1)$$

where

V_1 is the volume, in millilitres, of potassium dichromate solution (4.15) used for the determination;

V_2 is the volume, in millilitres, of potassium dichromate standard solution (4.15) used for the blank test, corrected for the addition of ammonium iron(II) sulfate solution;

m is the mass, in grams, of the test portion;

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

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

in which A is the hygroscopic moisture content, as a percentage by mass, 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:¹⁾

Tin(II) chloride reduction (Method 1)

$$r = 0,001\,1 X + 0,171\,6 \quad \dots (3)$$

$$P = 0,005\,3 X + 0,218\,9 \quad \dots (4)$$

$$\sigma_r = 0,000\,4 X + 0,061\,9 \quad \dots (5)$$

$$\sigma_L = 0,002\,2 X + 0,054\,1 \quad \dots (6)$$

Hydrogen sulfide reduction (Method 2)

$$r = 0,000\,4 X + 0,234\,8 \quad \dots (7)$$

$$P = 0,003\,1 X + 0,322\,8 \quad \dots (8)$$

$$\sigma_r = 0,000\,2 X + 0,084\,5 \quad \dots (9)$$

$$\sigma_L = 0,001\,2 X + 0,100\,2 \quad \dots (10)$$

where

X is the total iron content, expressed as a percentage by mass, of the test sample, calculated as follows:

within laboratory equations (3, 5, 7, 9); the arithmetic mean of the duplicate values,

between laboratories equations (4, 6, 8, 10); the arithmetic mean of the final results (8.2.3) of the two laboratories;

r is the permissible tolerance within a laboratory (repeatability);

P is the permissible tolerance between laboratories;

σ_r is the within laboratory standard deviation;

σ_L is the between laboratories standard deviation.

8.2.2 Acceptance of analytical values

The result obtained for the certified reference materials (7.2) shall be such that the difference between this result and the certificate value of the reference material is statistically insignificant. For a reference material that has been analysed by at least 10 laboratories using method(s) that are comparable both in accuracy and precision with this method, the following condition may be used to test the significance of the difference:

$$|A_c - A| < 2 \sqrt{\frac{s_{Lc}^2 + \frac{s_{Wc}^2}{n_{Wc}}}{N_c} + \sigma_L^2 + \frac{\sigma_r^2}{n}} \quad \dots (11)$$

where

A_c is the certificate value;

A is the result or the mean of results obtained for the reference material;

s_{Lc} is the between laboratories standard deviation of the certifying laboratories;

s_{Wc} is the within laboratory standard deviation of the certifying laboratories;

n_{Wc} is the average number of replicate determinations in the certifying laboratories;

N_c is the number of certifying laboratories;

n is the number of replicate determinations on the reference material (in most cases $n = 1$);

σ_L and σ_r are as defined in 8.2.1.

1) Additional information is given in annex B and annex C.

If condition (11) is satisfied, i.e. if the left-hand side is less than or equal to the right-hand side, then the difference $|A_c - A|$ is statistically insignificant; otherwise, it is statistically significant.

When the difference is significant, the analysis shall be repeated, simultaneously with an analysis of the test sample. If the difference is again significant, the procedure shall be repeated using a different certified material of the same type of ore.

When the range of the two values for the test sample is outside the limit calculated according to either equation (3) or equation (7), an analysis shall be carried out in accordance with the flowsheet presented in annex A, simultaneously with an analysis of a certified reference material of the sample type of ore.

Acceptability of the results for the test sample shall in each case be subject to the acceptability of the results for the certified reference material.

NOTE — The following procedure should be used when the information on the reference material certificate is incomplete:

- if there are sufficient data to enable the between laboratories standard deviation to be estimated, delete the expression s_{WC}^2/n_{WC} and regard s_{LC} as the standard deviation of the laboratory means;
- if the certification has been made by only one laboratory or if the interlaboratory results are missing, use the condition

$$|A_c - A| < 2 \sqrt{2\sigma_L^2 + \frac{\sigma_r^2}{n}} \quad \dots (12)$$

8.2.3 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, calculated to four decimal places and rounded off to the second decimal place as follows:

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

c) when 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.

8.3 Oxide factors

$$w_{Fe_2O_3} = 1,430 w_{Fe}$$

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

$$w_{Fe_3O_4} = 1,382 w_{Fe}$$

9 Test report

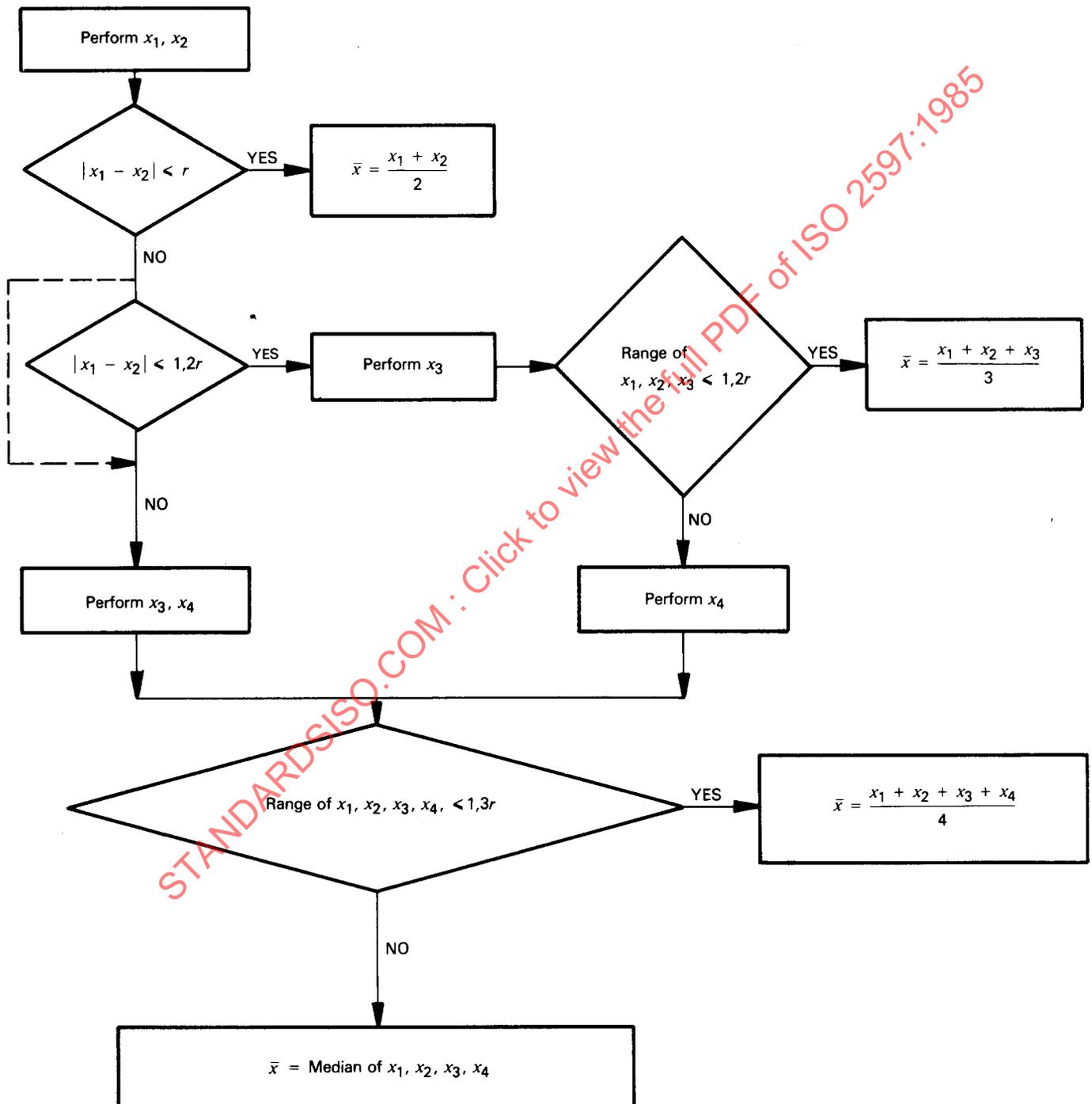
The test report shall include the following information:

- reference to this International Standard (including reference of the method used);
- details necessary for the identification of the sample;
- result of the analysis;
- reference number of the result;
- any characteristics noticed during the determination and any operations not specified in this International Standard which may have had an influence on the results, either for the test sample or the certified reference material(s).

Annex A

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

(An integral part of this International Standard.)



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r: as defined in 8.2.1.

Annex B

Derivation of repeatability and permissible tolerance equations

(This annex is for information only, and is not an integral part of this International Standard.)

The regression equations in 8.2.1 were obtained by statistical evaluation of the results of international analytical trials carried out in 1966/67 on eight iron ore samples (see the table) involving 30 laboratories in various countries.

Graphical treatment of the precision data is given in annex C.

Table – Total iron contents of test samples

| Sample | Iron content [% (m/m)] |
|----------------------|------------------------|
| 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 |

NOTES

- 1 A report of the statistical analysis of the international trial (Document ISO/TC 102/SC 2 N 147E, November 1968) is available from the secretariat of ISO/TC 102/SC 2.
- 2 The statistical analysis has been performed in accordance with the principles embodied in ISO 5725 (at present at the stage of draft).

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Annex C

Precision data obtained by international analytical trial

(This annex is for information only, and is not an integral part of this International Standard.)

NOTE — This figure is a graphical presentation of the equations in 8.2.1.

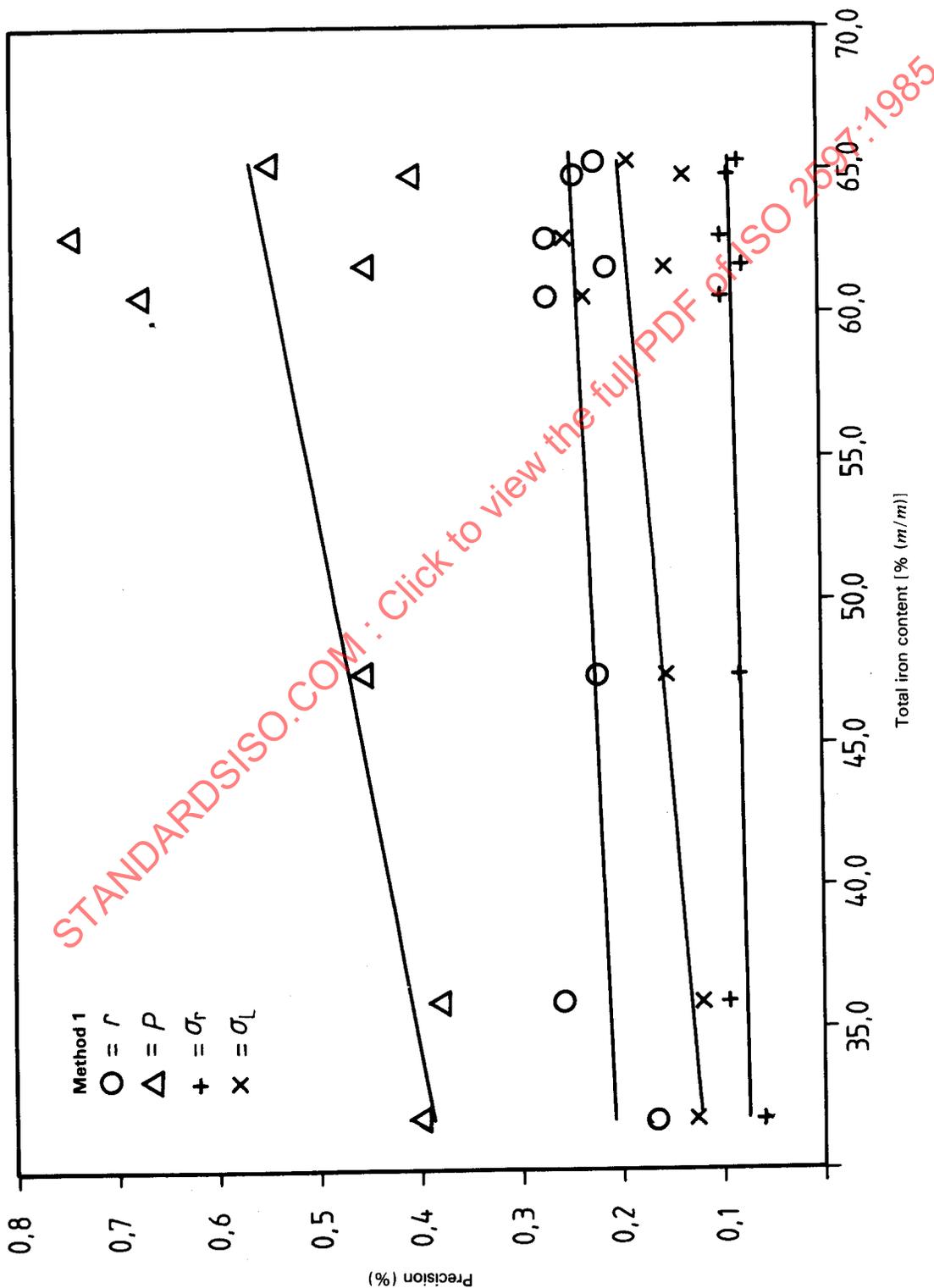


Figure 1 — Least-squares fit of precision against X for total iron, for the tin(II) chloride reduction method