BRITISH STANDARD

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1990
ISO 9507:1990

Analysis of iron ores —

Part 4: Method for the determination of total iron content —

Section 4.2 Titanium (III) chloride reduction methods

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Iron and Steel Standards Policy Committee (ISM/-) to Technical Committee ISM/58, upon which the following bodies were represented:

British Steel Industry
Institution of Mining and Metallurgy
Coopted members

The following body was also represented in the drafting of the standard, through subcommittees and panels:

British Ceramic Research Ltd.

This British Standard, having been prepared under the direction of the Iron and Steel Standards Policy Committee, was published under the authority of the Board of BSI and comes into effect on 31 October 1990

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Contents

National foreword

This Section of BS 7020 has been prepared under the direction of the Iron and Steel Standards Policy Committee. It is identical with ISO 9507:1990 "*Iron ores — determination of total iron content — Titanium (III) chloride reduction methods*" published by the International Organization for Standardization (ISO).

Cross-references

International Standard	Corresponding British Standard
ISO 1042:1981	BS 1792:1982 *Specification for one-mark volumetric flasks* (Identical)
	BS 7020 *Analysis of iron ores*
ISO 2596:1984	Part 2:1988 *Method for the determination of hygroscopic moisture in analytical samples* (Identical)
	BS 5660 *Methods of sampling iron ores*
ISO 3081:1986	Part 1:1987 *Manual method of increment sampling* (Identical)
ISO 3082:1987	Part 2:1987 *Mechanical method of increment sampling and sample preparation* (Identical)
ISO 3083:1986	BS 5661:1987 *Method for preparation of samples of iron ores by manual means* (Identical)
	BS 7020 *Analysis of iron ores*
ISO 7764:1985	Part 1:1988 *Method for the preparation of pre-dried test samples for chemical analysis*

The Technical Committee has reviewed the provisions of ISO 385-1 and ISO 648 to which reference is made in the text and has decided that they are acceptable for use in conjunction with this standard.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This International Standard 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 % (*m*/*m*) to 72 % (*m*/*m*) of iron in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 2596:1984, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric and Karl Fischer methods.*

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

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

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

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

3 Principle

3.1 Decomposition of the test portion

3.1.1 *Acid decomposition*

For samples containing not more than 0,05 % (*m*/*m*) of vanadium, 0,1 % (*m*/*m*) of molybdenum or 0,1 % (*m*/*m*) of copper: by treatment with hydrochloric acid, filtration of the residue, ignition, treatment with hydrofluoric and sulfuric acids, fusion with potassium pyrosulfate, leaching of the melt and combination with the main iron solution.

3.1.2 *Fusion-acidification*

For samples containing not more than 0,05 % (*m*/*m*) of vanadium, 0,1 % (*m*/*m*) of molybdenum or 0,1 % (*m*/*m*) of copper: by fusion with alkali, leaching of the cold melt with water and acidification with hydrochloric acid.

3.1.3 *Fusion-filtration*

For samples containing more than 0,05 % (*m*/*m*) of vanadium and/or 0,1 % (*m*/*m*) of molybdenum but not more than 0,1 % (*m*/*m*) of copper: by fusion with alkali, leaching of the cold melt with water followed by filtration. Dissolution of the precipitate in hydrochloric acid.

3.2 Titration of iron

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

4 Reagents

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

	Method
4.1 *Potassium pyrosulfate* ($K_2S_2O_7$), fine powder.	1 and 2
4.2 *Sodium carbonate* (Na_2CO_3), anhydrous, or pre-ignited at 500 °C.	1 and 2
4.3 *Sodium peroxide* (Na_2O_2), dry powder.	1 and 2
NOTE Sodium peroxide should be stored as dry as possible and should not be used once it has begun to agglomerate.	
4.4 *Hydrochloric acid,* ϱ 1,16 g/ml to 1,19 g/ml.	1 and 2
4.5 *Hydrochloric acid,* ϱ 1,16 g/ml to 1,19 g/ml, diluted 1 + 1.	1 and 2
4.6 *Hydrochloric acid,* ϱ 1,16 g/ml to 1,19 g/ml, diluted 1 + 10.	2
4.7 *Hydrochloric acid,* ϱ 1,16 g/ml to 1,19 g/ml, diluted 1 + 50.	1 and 2
4.8 *Hydrofluoric acid,* 40 % (*m*/*m*) (ϱ 1,13 g/ml) or 48 % (*m*/*m*) (ϱ 1,19 g/ml).	1 and 2
4.9 *Sulfuric acid,* ϱ 1,84 g/ml.	1 and 2
4.10 *Sulfuric acid,* ϱ 1,84 g/ml, diluted 1 + 1.	1 and 2
Carefully pour 1 volume of reagent **4.9** into 1 volume of cold water.	1 and 2
4.11 *Orthophosphoric acid,* ϱ 1,7 g/ml.	1 and 2

	Method
4.12 *Perchloric acid,* 72 % (*m*/*m*) (ϱ 1,7 g/ml), diluted 1 + 1.	2
4.13 *Sulfuric acid-orthophosphoric acid mixture*	1 and 2
Pour 150 ml of orthophosphoric acid (**4.11**) into about 400 ml of water while stirring, add 150 ml of sulfuric acid (**4.9**). Cool in a water bath and dilute with water to 1 litre.	
4.14 *Sodium hydroxide* (NaOH) solution, 20 g/l.	1 and 2
4.15 *Hydrogen peroxide* (H_2O_2), 30 % (*V*/*V*) solution.	1 and 2
4.16 *Hydrogen peroxide* (H_2O_2), 3 % (*V*/*V*) solution.	1
Dilute 1 volume of reagent **4.15** with 9 volumes of water.	
4.17 *Potassium permanganate* ($KMnO_4$) solution, 25 g/l.	1 and 2
4.18 *Potassium dichromate* ($K_2Cr_2O_7$) solution, 0,5 g/l.	1
4.19 *Tin(II) chloride* solution, 100 g/l.	1 and 2
Dissolve 100 g of crystalline tin(II) chloride ($SnCl_2\ 2H_2O$) in 200 ml of hydrochloric acid (**4.4**) by heating the solution in a water bath. Cool the solution and dilute with water to 1 litre. This solution should be stored in a brown glass bottle with a small quantity of granular tin metal.	
4.20 *Titanium(III) chloride* ($TiCl_3$) solution, 15 g/l	1 and 2
Dilute 1 volume of titanium(III) chloride solution (about 15 % $TiCl_3$) with 9 volumes of hydrochloric acid (**4.5**). Alternatively, dissolve 1 g of titanium sponge in about 30 ml of hydrochloric acid (**4.4**) 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.	
4.21 *Iron,* standard solution, 0,1 mol/l.	1 and 2
Transfer 5,58 g of pure iron to a 500 ml Erlenmeyer flask and place a small filter funnel in the neck. Add 75 ml of hydrochloric acid (**4.5**) in small increments and heat until dissolved. Cool and oxidize with 5 ml of hydrogen peroxide (**4.15**) 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 volumetric flask and dilute to volume.	
1,00 ml of this solution is equivalent to 1,00 ml of the standard potassium dichromate solution.	
4.22 *Potassium dichromate* (99,9 % minimum purity), standard solution, 0,016 67 mol/l.	1 and 2
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.	
Dissolve 4,904 g of this material in water, transfer to a 1 000 ml volumetric flask, make up to volume with water after cooling to 20 °C and mix.	
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 Record the temperature at which this dilution was made (20 °C) on the stock bottle.	
4.23 *Indigo carmine* [indigo-5,5′-disulfonic acid disodium salt ($C_{16}H_8O_8N_2S_2Na_2$)] solution, 0,1 g/100 ml.	1
Dissolve 0,1 g of indigo carmine in a cold mixture of 50 ml sulfuric acid (**4.10**) and 50 ml of water.	
4.24 *Sodium diphenylaminesulfonate indicator,* solution, 0,2 g/100 ml.	1 and 2
Dissolve 0,2 g of sodium diphenylaminesulfonate ($C_6H_5NHC_6H_4SO_3Na$) in a small volume of water and dilute to 100 ml.	
Store the solution in a brown glass bottle.	

5 Apparatus

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

Ordinary laboratory apparatus, and

5.1 *Zirconium* or *vitreous carbon crucible*, capacity 25 ml to 30 ml.

5.2 *Burette,* class A, conforming with ISO 385-1.

5.3 *Platinum crucible*

Platinum crucibles should be precleaned, before use, to avoid iron contamination.

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

5.5 *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 less than 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 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 with significant contents of combined water or oxidizable compounds, use a particle size of less than 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 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 °C ± 2 °C as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

7.1 Number of determinations

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

NOTE 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 each 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**). (See note 1 below.)

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.

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 both cases no significant changes in the analytical procedure would become 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

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 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 (**6.2**) using a non-magnetic spatula (**5.4**).

NOTE 1 For samples with an iron content higher than 68 % (*m*/*m*), weigh approximately 0,38 g.

NOTE 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

7.5.1 *Decomposition of the test portion*

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

Place the test portion (**7.4**) in a 400 ml beaker, add 30 ml of hydrochloric acid (**4.4**), cover the beaker with a watch glass, and heat the solution gently without boiling to decompose the ore.

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

Wash the watch glass with a jet of water, and dilute to 50 ml with warm water. Filter the insoluble residue on a close-textured filter paper. Wash the residue with warm hydrochloric acid (**4.7**) 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. Begin the evaporation of this main solution without boiling.

Place the filter paper and residue in a platinum crucible (**5.3**), dry, char the paper and finally ignite at 750 °C to 800 °C. Allow the crucible to cool, moisten the residue with sulfuric acid (**4.10**), add about 5 ml of hydrofluoric acid (**4.8**) and heat gently to remove silica and sulfuric acid. Add 2 g of potassium pyrosulfate (**4.1**) to the cold crucible, heat gently at first then strongly until a clear melt is obtained. Cool, place the crucible in a 250 ml beaker, add about 25 ml of water and about 5 ml of hydrochloric acid (**4.4**) and warm to dissolve the melt. Remove and wash the crucible. Combine this solution with the main solution, evaporate without boiling to about 100 ml and follow the procedure specified in **7.5.2**.

7.5.1.2 Fusion-acidification [for samples containing not more than 0,05 % (*m*/*m*) of vanadium and 0,1 % (*m*/*m*) of both molybdenum and copper]

Place the test portion (**7.4**) in a zirconium or vitreous carbon crucible (**5.1**), add 1,3 g of sodium carbonate (**4.2**) and 2,7 g of sodium peroxide (**4.3**) and mix thoroughly. Place in a muffle furnace (**5.5**) at 500 °C ± 10 °C for 30 min. 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.

Allow the melt to cool, place the crucible in a 600 ml beaker, add about 100 ml of warm water and heat for a few minutes to leach the melt. Add 20 ml of hydrochloric acid (**4.4**) and heat until effervescence from decomposition of sodium carbonate and peroxide ceases. Remove and wash the crucible while adding the washings to the solution, evaporate without boiling to about 100 ml and follow the procedure specified in **7.5.2**.

7.5.1.3 Fusion-filtration [for samples containing more than 0,05 % (*m*/*m*) of vanadium and/or 0,1 % (*m*/*m*) of molybdenum, but not more than 0,1 % (*m*/*m*) of copper]

Place the test portion (**7.4**) in a zirconium or vitreous carbon crucible (**5.1**), add 1,3 g of sodium carbonate (**4.2**) and 2.7 g of sodium peroxide (**4.3**) and mix thoroughly. Place in a muffle furnace (**5.5**) at 500 °C ± 10 °C for 30 min. 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.

Allow the melt to cool, place the crucible in a 600 ml beaker, add about 100 ml of water and heat for a few minutes to leach 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 paper twice with sodium hydroxide solution (**4.14**) and discard the filtrate.

Transfer the precipitate on the filter to the original beaker by washing with water, add 10 ml of hydrochloric acid (**4.4**) and warm to dissolve the precipitate. Pour the solution through the original filter paper, wash the filter three times with warm hydrochloric acid (**4.5**), and several times with hydrochloric acid (**4.7**). Finally, wash with warm water until the washings are no longer acid. Collect the filtrate and washings in a 600 ml beaker (this is the main solution). Dissolve any iron retained by the reserved crucible in hot hydrochloric acid (**4.5**) and wash with hot water into the main solution. Evaporate without boiling to about 100 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.

To the solution obtained in **7.5.1** add three to five drops of potassium permanganate solution (**4.17**) and heat the solution to just below the boiling point. Maintain at this temperature for 5 min to oxidize any arsenic or organic matter. Wash the cover and inside wall of the beaker with a small amount of hot hydrochloric acid (**4.6**). Immediately add tin(II) chloride solution (**4.19**) 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.

NOTE 1 If the solution is made colourless by the excessive addition of tin(II) chloride solution, add hydrogen peroxide solution (**4.16**) dropwise, until the solution changes to a light yellow colour. 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 (**4.22**) to 100 ml with water.

Wash the inside wall of the beaker with a small amount of hot water. Add 6 drops of indigo carmine solution (**4.23**) as indicator, then titanium(III) chloride solution (**4.20**) drop by drop, while swirling the solution, until it turns blue then colourless. Add 2 to 3 drops in excess. 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 greater than 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 with cold water. Follow the procedure specified in **7.5.3**.

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

To the solution obtained in **7.5.1** add 3 to 5 drops of potassium permanganate solution (**4.17**) and heat the solution to just below the boiling point. Maintain at this temperature for 5 min to oxidize any arsenic or organic matter. Wash the cover and inside wall of the beaker with a small amount of hot hydrochloric acid (**4.6**). Immediately add tin(II) chloride solution (**4.19**) drop by drop, while swirling the solution until only a light yellow colour remains.

NOTE It is essential that some iron(III) remain unreduced. If all the iron is inadvertently reduced, add hydrogen peroxide solution (**4.16**) drop by drop, until the solution changes to a light yellow colour (see **7.5.2.1**, note 1).

Reduce the remaining iron(III) by adding titanium(III) chloride solution (**4.20**), 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 (**4.12**). Mix well by swirling for about 5 s. Dilute immediately with cold (below 10 °C) water to 300 ml. Cool rapidly to below 15 °C and follow the procedure specified in **7.5.3**.

7.5.3 *Titration*

To the cold solution obtained in **7.5.2.1** or **7.5.2.2** add 30 ml of sulfuric acid-orthophosphoric acid mixture (**4.13**) and titrate with the potassium dichromate standard solution (**4.22**) using five drops of the sodium diphenylaminesulfonate solution (**4.24**) as 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 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.

7.5.4 *Blank test*

Determine the blank test value (**7.2**) 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 (**4.19**), add by means of a one-mark pipette, 1,00 ml of iron standard solution (**4.21**) and titrate the solution as described in **7.5.3**. 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 by mass, is calculated from the following equation:

$$w_{\mathrm{Fe}} = \frac{V_1 - V_2}{m} \times 0{,}005\,584\,7 \times 100 \times K \qquad \ldots (1)$$

where

V_1 is the volume, in millilitres, of potassium dichromate standard solution (**4.22**) 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 **7.5.4**;

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 (**6.2.2**), and for air-equilibrated test samples (**6.2.1**) is the conversion factor found from the equation

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

where 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 in Table 1[1].

Table 1 — Precision

	Method 1 (Dilute potassium dichromate oxidation)		**Method 2 (Perchloric acid oxidation)**
	a) Acid decomposition	b) Fusion decomposition	
r	0,156	0,170	0,208
P	0,239	0,267	0,336
σ_r	0,055	0,062	0,074
σ_L	0,074	0,085	0,105

r is the permissible tolerance within 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 material (**7.2**) shall be such that the difference between this result and the certified value of the certified reference material is statistically insignificant. For a certified 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:

$$|A_c - A| < 2\sqrt{\frac{s_{Lc}^2 + \dfrac{s_{Wc}^2}{n_{Wc}}}{N_c} + \sigma_L^2 + \frac{\sigma_r^2}{n}} \qquad \ldots (3)$$

where

A_c is the certified 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**.

If condition (3) 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 value for r in Table 1, an analysis shall be carried out in accordance with the flowsheet given in Annex A, simultaneously with an analysis of a certified reference material of the same 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:

a) 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;

b) if the certification has been made by only one laboratory or if the inter-laboratory results are missing, it is advisable that this material not be used in the application of the standard. In case its use is unavoidable, use the formula

$$|A_c - A| < 2\sqrt{2\sigma_L^2 + \frac{\sigma_r^2}{n}} \qquad \ldots (4)$$

8.2.3 *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) 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.

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[1] Additional information is given in Annex B.

8.3 Oxide factors

$w_{Fe_2O_3}$ = 1,430 wFe

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:

a) name and address of the testing laboratory;

b) date of issue of the test report;

c) reference to this International Standard (including reference to the method used);

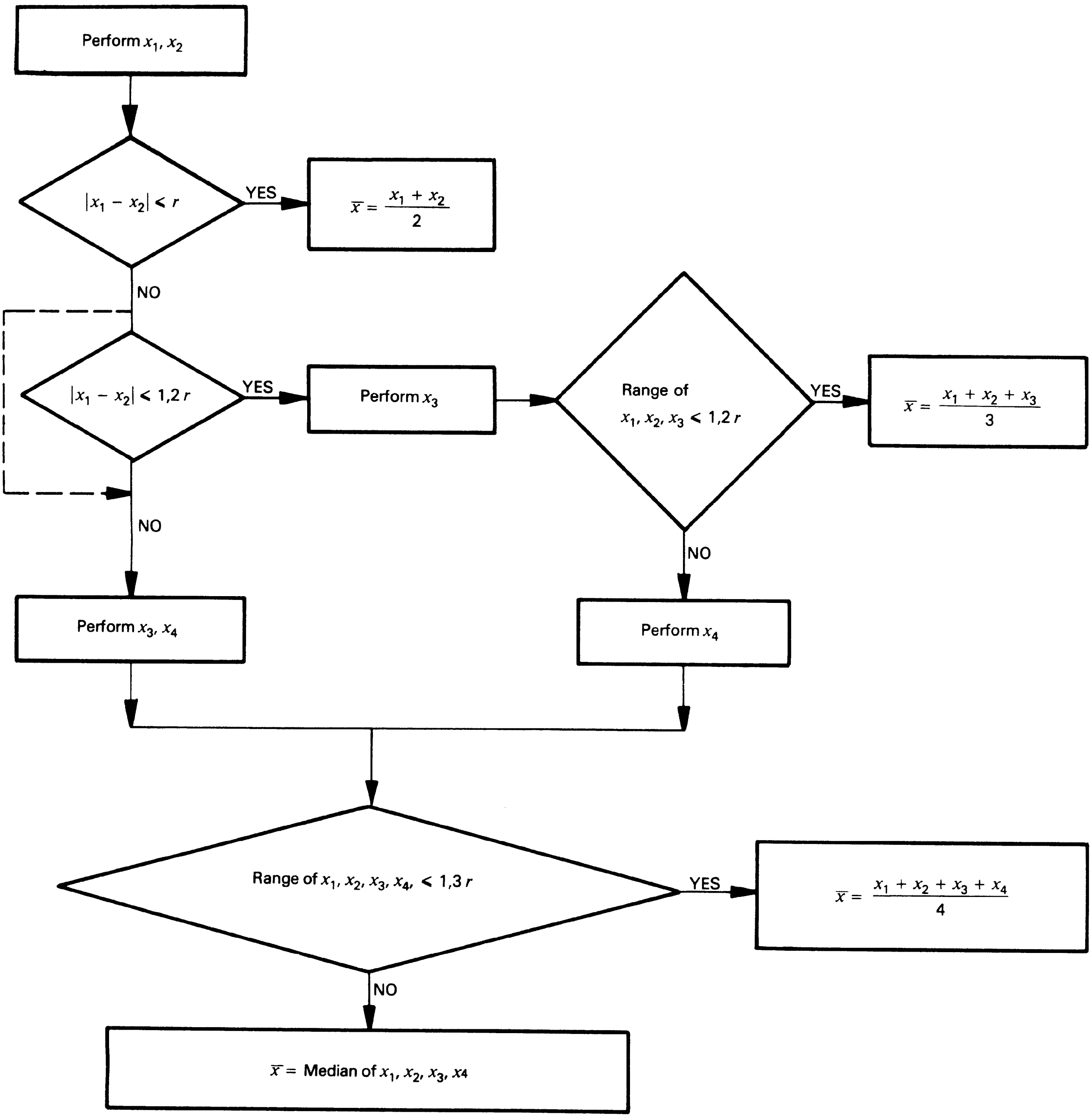
d) details necessary for the identification of the sample;

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 International Standard 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



r: see Table 1.

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 1981/84 on 5 iron ore samples (see Table B.1) involving 35 laboratories in 9 countries.

Table B.1 — Total iron contents of test samples

Sample	Iron content [% (*m*/*m*)]	Used in method
Savage River pellets	67,1	1 b), 2
Schefferville iron ore	60,7	1 a), 1 b), 2
Brazilian iron ore	45,9	1 a), 2
Labrador iron ore	59,6	1 a), 1 b), 2
Marcona pellets	66,8	1 a), 1 b), 2

NOTE 1 Reports of the international trials and statistical analysis of the results (Documents ISO/TC 102/SC 2 N692E, August 1982, ISO/TC 102/SC 2 N754E, March 1984 and ISO/TC 102/SC 2 N760E, August 1984) are available from either the secretariat of ISO/TC 102/SC 2 or the secretariat of ISO/TC 102.

NOTE 2 The statistical analysis has been performed in accordance with the principles embodied in ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

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Publication(s) referred to

See national foreword.

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