BRITISH STANDARD

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1993
ISO 2598-1:
1992

Analysis of iron ores —

Part 5: Methods for the determination of silicon content —

Section 5.1 Gravimetric methods

UDC 553.31:543.21:546.28

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 Standards Board and comes into effect on 15 October 1993

The following BSI references relate to the work on this standard:
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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 and is identical with ISO 2598-1:1992 *Iron ores — Determination of silicon content — Part 1: Gravimetric methods* published by the International Organization for Standardization (ISO).

It supersedes BS 4158-3:1972, which is withdrawn.

Cross-references

International standard	Corresponding British Standard
ISO 1042:1983	BS 1792:1982 *Specification for one-mark volumetric flasks* (Identical)
ISO 2596:1984	BS 7020 *Analysis of iron ores* 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)
ISO 7764:1985	BS 7020 *Analysis of iron ores* Part 1:1988 *Method for the preparation of pre-dried test samples for chemical analysis* (Identical)

The Technical Committee has reviewed the provisions of ISO 648, to which normative reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard. A related British Standard to ISO 648:1977 is BS 1583:1986 *Specification for one-mark pipettes.*

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.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, 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 part of ISO 2598 specifies two gravimetric methods for the determination of the silicon content of iron ores.

These methods are applicable, with certain limitations, to silicon contents between 1 % (*m*/*m*) and 15 % (*m*/*m*) in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

Method 1 is not applicable to iron ores having a content of reducing agents greater than 2 % (*m*/*m*), for instance pyrite, or to ores having a fluorine content greater than 0,1 % (*m*/*m*). It is recommended for lower grade ores having a high content of amphoteric elements.

Method 2 can be used for ores having a fluorine content greater than 0,1 % (*m*/*m*). It is recommended for high grade ores having a low content of gangue.

NOTE 1 For ores having a silicon content less than 5 % (*m*/*m*), the method specified in ISO 2598-2:—[1], *Iron ores — Determination of silicon content — Part 2: Reduced molybdosilicate spectrophotometric method,* is preferable.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 2598. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 2598 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 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

Decomposition of the test portion, by either method 1 or method 2.

Method 1: Decomposition by sintering with sodium peroxide, followed by treatment with hydrochloric and perchloric acids, or

Method 2: Decomposition by treatment with hydrochloric, nitric and perchloric acids (with inclusion of boric acid, if necessary) and evaporation to fumes of perchloric acid. Filtration of silica together with any residue, fusion with sodium carbonate and dissolution in hydrochloric and perchloric acids.

Evaporation of the solution from either method 1 or method 2 to fumes of perchloric acid and filtration of the precipitated silica. Ignition of the impure silica and weighing. Treatment of the ignited residue with hydrofluoric and sulfuric acids, followed by ignition and reweighing.

4 Reagents

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

4.1 *Sodium peroxide* (Na_2O_2), powder.

4.2 *Boric acid* (H_3BO_3).

To be used as in note 7.

4.3 *Sodium carbonate* (Na_2CO_3), anhydrous.

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

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

4.6 *Hydrochloric acid,* ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 9.

4.7 *Perchloric acid,* ρ 1,67 g/ml, 70 % (*m*/*m*), or ρ 1,54 g/ml, 60 % (*m*/*m*).

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

4.9 *Sulfuric acid,* ρ 1,84 g/ml, diluted 1 + 9.

4.10 *Hydrofluoric acid,* ρ 1,13 g/ml, 40 % (*m*/*m*), or ρ 1,185 g/ml, 48 % (*m*/*m*).

4.11 *Nitric acid,* ρ 1,4 g/ml.

5 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 or ISO 1042 respectively, and

5.1 *Nickel, zirconium or vitreous carbon crucibles,* of capacity approximately 40 ml.

5.2 *Platinum crucibles,* of capacity approximately 40 ml.

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[1] To be published. (At present published as ISO 4686:1980.)

5.3 *Nickel spatula*

5.4 *Muffle furnaces,* adjustable to 400 °C ± 20 °C and to temperatures up to 1 050 °C.

6 Sampling and samples

6.1 Laboratory sample

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

NOTE 2 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 having significant quantities of combined water or oxidizable compounds and silica contents above*** 10 % (*m*/*m*)

Where the silica content is above 10 % (*m*/*m*), prepare an air-equilibrated test sample in accordance with ISO 2596 for 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.

NOTE 3 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, in either case, appropriate recalibration.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, approximately the amount of the predried test sample indicated in Table 1.

Table 1 — Mass of test portion

Si content % (*m*/*m*)	**Mass of test portion** g
1 to 10	1,0
10 to 15	0,5

NOTE 4 The test portion should be taken and weighed quickly to avoid reabsorption of moisture.

7.3 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 predried test sample of the certified reference material shall be prepared as specified in **6.2.2**.

NOTE 5 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.

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 that 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.

7.4 Determination

7.4.1 ***Decomposition of the test portion***

If the decomposition is to be based on alkali sintering, proceed as specified in **7.4.1.1**; if it is to be based on acid attack, proceed as specified in **7.4.1.2**.

7.4.1.1 *Alkali sinter attack (Method 1)*

Place the test portion (**7.2**) in a nickel, zirconium or vitreous carbon crucible (**5.1**), add 3 g of sodium peroxide (**4.1**), mix thoroughly using the nickel spatula (**5.3**) and tamp the mixture.

Place the crucible in the entrance of the muffle furnace (**5.4**), set at 400 °C ± 20 °C, and leave for 1 min to 2 min. Place the crucible in the furnace, maintained at the same temperature, and leave for 1 h. Remove the crucible from the furnace and allow to cool in a desiccator.

NOTE 6 The mixture should not reach the melting point. Should this happen, it is recommended that the operation be repeated at a lower temperature.

WARNING — Protective goggles should be worn during the following operation.

Transfer the crucible containing the sintered mass to a 600 ml low-form beaker. Cover the beaker with a watch-glass and carefully add 200 ml of water. Add 50 ml of hydrochloric acid (**4.4**) and 25 ml of perchloric acid (**4.7**) to dissolve the sintered mass. Remove the crucible from the beaker and rinse it with hydrochloric acid (**4.6**) and water. Scrape out the rest of the sintered mixture adhering to the walls of the crucible, using a rubber-tipped glass rod. Place the beaker on a hot-plate and heat the solution gently to decompose the sintered products completely.

Add 1 ml of sulfuric acid (**4.9**) to prevent precipitation of titanium.

Partially uncover the beaker and heat until dense white fumes of perchloric acid appear. Cover the beaker completely and continue heating until there are no more fumes in the beaker. Maintain this stage until most of the perchloric acid has evaporated, but avoid evaporation to dryness.

Allow the solution to cool, then add about 25 ml of hydrochloric acid (**4.5**). Agitate and heat gently to dissolve the soluble salts. Allow the precipitate to settle for several minutes, then rinse the walls of the beaker with about 30 ml of water. Continue immediately, in accordance with **7.4.2**.

7.4.1.2 *Acid attack (Method 2)*

Place the test portion (**7.2**) in a 400 ml low-form beaker and moisten with 5 ml of water.

NOTE 7 For ores having fluorine contents greater than 0,1 % (*m*/*m*) or where the fluorine content is unknown, 0,8 g of boric acid (**4.2**) should be added to the beaker containing the test portion before the addition of 5 ml of water.

Add 50 ml of hydrochloric acid (**4.4**). Cover the beaker with a watch-glass and heat gently without boiling, until decomposition of the test portion is complete. Add 1 ml of nitric acid (**4.11**) and then 25 ml of perchloric acid (**4.7**).

Add 1 ml of sulfuric acid (**4.9**) to prevent precipitation of titanium.

Partially uncover the beaker and heat until dense white fumes of perchloric acid appear. Cover the beaker completely and continue heating until there are no more fumes in the beaker. Maintain this stage until most of the perchloric acid has evaporated, but avoid evaporation to dryness.

Allow the solution to cool, then add about 25 ml of hydrochloric acid (**4.5**). Agitate and heat gently to dissolve the soluble salts. Add about 30 ml of water, mix and collect the precipitate on a close-texture filter paper containing small pieces of filter paper or a small amount of filter pulp.

Rinse the beaker with water, and rub the walls of the beaker using a rubber-tipped glass rod. Wash the residue three or four times with hot hydrochloric acid (**4.6**) and finally with hot water, until free from acids. Discard the filtrate and washings (see note 8).

NOTE 8 For sinters, the silica should be recovered from this filtrate and washings, by the following procedure:
Add 10 ml of perchloric acid (**4.7**) and 1 ml of sulfuric acid (**4.9**) to the filtrate and washings, and heat until dense white fumes of perchloric acid appear. Cover the beaker completely and continue heating until there are no more fumes in the beaker. Maintain this stage until most of the perchloric acid has evaporated, but avoid evaporation to dryness. Repeat the procedure specified in paragraphs 4 and 5, and then continue in accordance with the second paragraph of **7.4.2**.

Place the residue with the filter in a platinum crucible (**5.2**), dry, then ash the paper and finally ignite in the muffle furnace (**5.4**), set at 750 °C to 800 °C. Allow the crucible to cool. Add 2 g to 3 g of sodium carbonate (**4.3**), mix with the nickel spatula (**5.3**) and heat in the muffle furnace, set at 900 °C to 1 000 °C, to effect complete fusion.

WARNING — Protective goggles should be worn during the following operation.

Allow the crucible to cool, then place it in a 600 ml low-form beaker. Cover the beaker with a watch-glass. Add 200 ml of water, then 50 ml of hydrochloric acid (**4.4**) and 25 ml of perchloric acid (**4.7**). Remove the crucible from the beaker and rinse it with hydrochloric acid (**4.6**) and water. Scrape out the rest of the fused mixture adhering to the walls of the crucible, using a rubber-tipped glass rod. Place the beaker on a hot-plate and heat the solution gently to dissolve the fused products.

Add 1 ml of sulfuric acid (**4.9**) to prevent precipitation of titanium.

Partially uncover the beaker and heat until dense white fumes of perchloric acid appear. Cover the beaker completely and continue heating until there are no more fumes in the beaker. Maintain this stage until most of the perchloric acid has evaporated, but avoid evaporation to dryness.

Allow the solution to cool, then add about 25 ml of hydrochloric acid (**4.5**). Agitate and heat gently to dissolve the soluble salts. Allow the precipitate to settle for several minutes, then rinse the walls of the beaker with about 30 ml of water. Continue immediately, in accordance with **7.4.2**.

7.4.2 *Treatment of silica*

Filter the solution containing the insoluble silica obtained as in **7.4.1.1** or **7.4.1.2** through a close-texture filter paper containing small pieces of filter paper or a small amount of filter pulp. Rinse the beaker with water, and rub the walls of the beaker using a rubber-tipped glass rod. Wash the residue with hot hydrochloric acid (**4.6**), then with hot water to effect complete elimination of the perchloric acid, and finally with warm water. Reserve the residue on the filter, and discard the filtrate and washings (see note 9).

NOTE 9 For ores having a silicon content in excess of 5 % (*m*/*m*) [about 10 % (*m*/*m*) as silica], or where the silicon content is unknown, the silica should be recovered from this filtrate and washings by the procedure given in note 8.

Place the residue and filter in a platinum crucible (**5.2**). Heat gently until dry, then ash the filter paper and ignite for 30 min in the muffle furnace (**5.4**), set at 1 050 °C ± 20 °C.

Cool in a desiccator and weigh the crude silica to the nearest 0,000 1 g. Repeat the heating as above until constant mass is obtained (mass m_1). Moisten the residue in the crucible with a few drops of water, add 5 drops of sulfuric acid (**4.8**) and, according to the silica content, 5 ml to 15 ml of hydrofluoric acid (**4.10**). Heat gently in a fume cupboard to remove silica and sulfuric acid. Finally heat the crucible for 15 min in the muffle furnace set at 1 050 °C ± 20 °C. Cool in a desiccator and weigh the impurities to the nearest 0,000 1 g. Repeat the treatment with sulfuric and hydrofluoric acids, and also the ignition, until constant mass is obtained (mass m_2).

8 Expression of results

8.1 Calculation of silicon content

The silicon content, w_{Si}, as a percentage by mass, is calculated to four decimal places using the following equation

$$w_{\mathrm{Si}} = \frac{m_1 - m_2}{m_3} \times 0{,}467\ 4 \times 100 \times K$$

$$= \frac{m_1 - m_2}{m_3} \times 46{,}74 \times K \qquad \text{...(1)}$$

where

m_1 is the mass, in grams, of the platinum crucible containing the crude silica;

m_2 is the mass, in grams, of the platinum crucible containing the impurities;

m_3 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

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

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 tolerances*

The precision of this analytical method is expressed by the following regression equations[2]:

$$r = 0{,}009\ 0\ X + 0{,}051\ 1 \qquad \text{...(3)}$$

$$P = 0{,}009\ 5\ X + 0{,}083\ 1 \qquad \text{...(4)}$$

$$\sigma_{\mathrm{r}} = 0{,}003\ 2\ X + 0{,}018\ 1 \qquad \text{...(5)}$$

$$\sigma_{\mathrm{L}} = 0{,}002\ 7\ X + 0{,}024\ 8 \qquad \text{...(6)}$$

where

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

- within-laboratory equations (3) and (5): the arithmetic mean of the duplicate values,
- between-laboratories equations (4) and (6): 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.

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[2] Additional information is given in Annex B and Annex C.

8.2.2 *Acceptance of analytical values*

The result obtained for the certified reference material shall be such that the difference between this result and the certified 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 in precision with this method, the following condition may be used to test the significance of the difference

$$|A_c - A| \leqslant 2\sqrt{\frac{s_{Lc}^2 + \frac{s_{Wc}^2}{n_{Wc}}}{N_c} + \sigma_L^2 + \frac{\sigma_r^2}{n}} \qquad \text{...(7)}$$

where

A_c	is the certified value;
A	is the result or the mean of results obtained for the certified 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 (7) 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 reference material of the same type of ore.

When the range of the two values for the test sample is outside the limit for *r* calculated according to equation (3) in **8.2.1**, one or more additional tests shall be carried out in accordance with the flowsheet presented in Annex A, simultaneously with a corresponding blank test and 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 10 The following procedure should be used when the information on the reference material certificate is incomplete:

a) if there are insufficient 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 interlaboratory 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 condition

$$|A_c - A| \leqslant 2\sqrt{2\sigma_L^2 + \frac{\sigma_r^2}{n}} \qquad \text{...(8)}$$

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 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 factor

The oxide factor is given by the following equation:

w_{SiO_2} (%) = 2,139 w_{Si} (%)

9 Test report

The test report shall include the following information:

a) name and address of the testing laboratory;

b) date of issue of the test report;

c) reference to this part of ISO 2598;

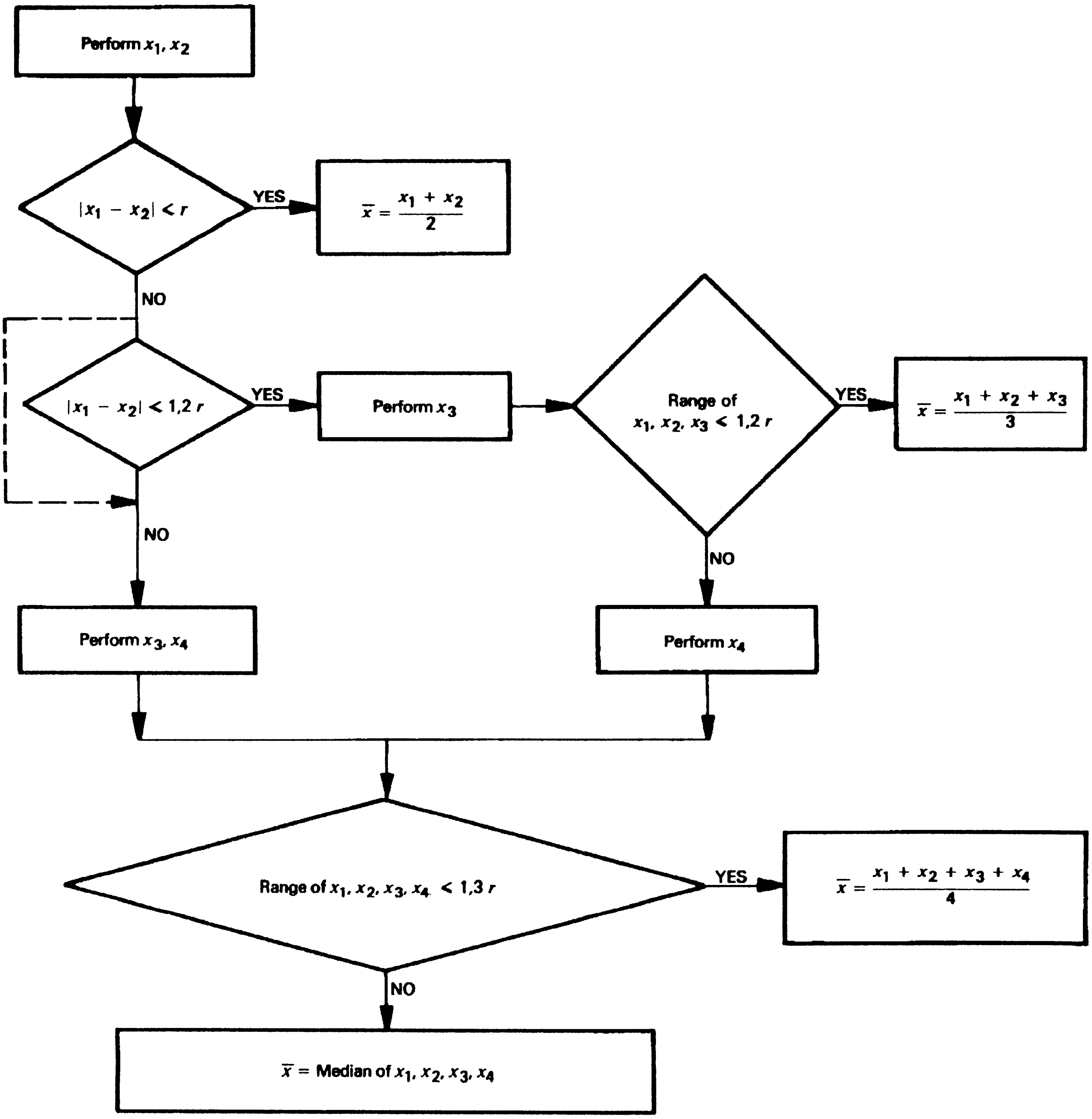
d) details necessary for the identification of the sample;

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 2598 which may have had an influence on the result, for either 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: as defined in **8.2.1**

Annex B (informative) Derivation of repeatability and permissible tolerance equations

The equations in **8.2.1** were derived from the results of international analytical trials carried out in 1967/1968 and 1970/1971 on five iron ore samples, involving 28 laboratories in 12 countries.

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

The test samples are listed in Table B.1.

NOTE 11 A report of the international trials and a statistical analysis of the results (Documents ISO/TC 102/SC 2 N 148, January 1969 and N 224, June 1971) are available from either the Secretariat of ISO/TC 102/SC 2 or the Secretariat of ISO/TC 102.

NOTE 12 The statistical analysis was 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.*

Table B.1 — Silicon contents of test samples

Sample	Silicon content % (*m*/*m*)
Palabora ore	0,19
Philippine iron sand	1,05
Swedish ore	3,88
UK sinter	7,73
Krivoï-Rog ore	12,85

Annex C (informative) Precision data obtained by international analytical trials

NOTE 13 Figure C.1 is a graphical representation of the equations in **8.2.1**.

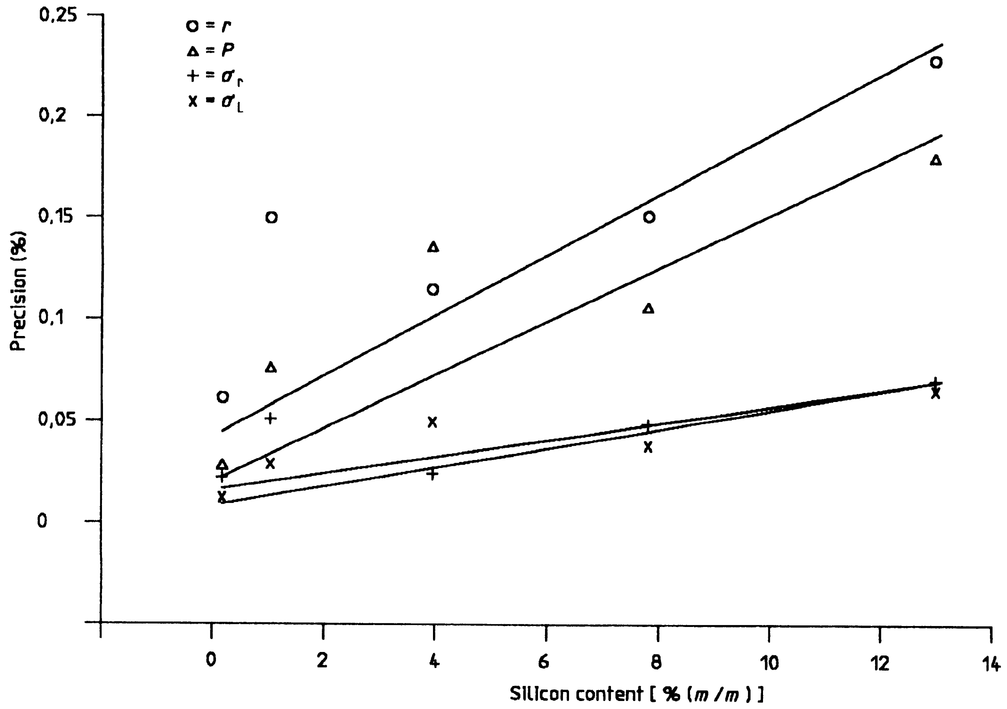


Figure C.1 — Least-squares fit of precision against *X* for silicon *blank*

List of references

See national foreword.

BSI — British Standards Institution

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