International Standard 2598

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Iron ores — Determination of silicon content — Gravimetric methods

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Foreword

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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.

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Australia
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Iron ores — Determination of silicon content — Gravimetric methods

1 Scope and field of application

This International Standard specifies two gravimetric methods for the determination of the silicon content of iron ores.

These methods are applicable, with certain limitations, to a concentration range of 1 to 15 % of silicon in natural iron ores, and iron ore concentrates and agglomerates, including sinter products.

Method 1 — The method 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 with a high content of amphoteric elements.

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

NOTE — For samples having a silicon content less than 5 % (*m/m*), ISO 4686, *Iron ores — Determination of silicon content — Reduced molybdosilicate spectrophotometric method*, is preferable.

2 References

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples.*

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

ISO 3083, *Iron ores — Preparation of samples.*

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 sulphuric 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 the note in 7.3.1.2.

4.3 Sodium carbonate (Na_2CO_3), anhydrous.

4.4 Sodium hydrogen sulphate ($NaHSO_4$), anhydrous.

To be used as in clause 9.

4.5 Hydrochloric acid, (ϱ 1,19 g/ml).

4.6 Hydrochloric acid, (ρ 1,19 g/ml), diluted 1 + 9.

4.7 Hydrochloric acid, (ρ 1,19 g/ml), diluted 1 + 1.

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

4.9 Sulphuric acid, (ϱ 1,84 g/ml), diluted 1 + 1.

4.10 Sulphuric acid, (ϱ 1,84 g/ml), diluted 1 + 9.

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

4.12 Nitric acid, (ϱ 1,4 g/ml).

5 Apparatus

Ordinary laboratory apparatus and

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

5.2 Platinum crucibles, capacity approximately 40 ml.

5.3 Nickel spatula.

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

6 Sampling and samples

For analysis, a laboratory sample of minus 100 µm particle size which has been taken in accordance with ISO 3081[1] and prepared in accordance with ISO 3083[1] shall be used. In the case of ores having high contents of combined water and/or oxidizable compounds, the particle size shall be minus 160 µm.

7 Procedure

Carry out the analysis generally in duplicate, independently, on one ore sample.

NOTE — The expression "independently" implies the change of the person carrying out the analysis. If the same person must carry out the analysis, the procedure shall be carried out at different times.

Simultaneously with the analysis, determine the hygroscopic moisture in accordance with ISO 2596.

7.1 Test portion

Weigh, to the nearest 0,000 2 g, approximately the amount of the test sample according to table 1.

Table 1 — Mass of test portion

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

7.2 Blank test and check test

In each run, one analysis of a certified reference material of the same type of ore and one blank test shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions.

NOTE — The certified reference material should be of the same type as the sample to be analysed. Such certified reference material cannot, however, be considered as being of the same type if the properties of the sample to be analysed differ from those of the certified reference material to such an extent that the analytical procedure must be changed substantially.

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.

7.3 Determination

7.3.1 Decomposition of the test portion

If the decomposition is to be based on alkali sintering, proceed as instructed in 7.3.1.1; if it is to be based on acid attack, proceed as instructed in 7.3.1.2.

7.3.1.1 Alkali sinter attack

Place the test portion (7.1) in a nickel or vitreous carbon crucible (5.1), add 3 g of sodium peroxide (4.1), mix thoroughly using a nickel spatula (5.3) and tamp the mixture.

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

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

CAUTION — 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 add carefully 200 ml of water. Add 50 ml of hydrochloric acid (4.5) and 25 ml of perchloric acid (4.8) to dissolve the sintered mass.

Remove the crucible from the beaker and rinse it with water and hydrochloric acid (4.6). 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 completely the sintered products.

Add 1 ml of sulphuric acid (4.10) to prevent titanium precipitation. Remove the cover partially from the beaker and heat until dense white fumes of perchloric acid appear. Then cover the beaker completely and continue heating until there are no move fumes in the beaker. Maintain this stage until most of the perchloric acid has evaporated, but avoid evaporation to dryness.

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[1] A further International Standard now in preparation (ISO 3082) will specify mechanical methods of increment sampling and will also specify methods for preparation of samples.

Allow the solution to cool, then add about 25 ml of hydrochloric acid (4.7), agitate and heat gently to dissolve the soluble salts. Allow the precipitate to settle for some minutes, then rinse the walls of the beaker with about 30 ml of water. Continue in accordance with 7.3.2.

7.3.1.2 Acid attack

Place the test portion (7.1) in a 400 ml low-form beaker and moisten with 5 ml of water.

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

Add 50 ml of hydrochloric acid (4.5). 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 (4.12) and then 25 ml of perchloric acid (4.8). Add 1 ml of sulphuric acid (4.10) to prevent titanium precipitation.

Remove the cover partially from the beaker and heat until dense white fumes of perchloric acid appear. Cover the beaker completely and continue heating until there are no move 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 25 ml of hydrochloric acid (4.7), 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, 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 or reserve for the determination of other elements. (See clause 9.)

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

CAUTION — 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 (4.5), and 25 ml of perchloric acid (4.8). Remove the crucible from the beaker and rinse it with water and hydrochloric acid (4.6). 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 sulphuric acid (4.10) to prevent titanium precipitation.

Remove the cover partially from 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 beaker to cool and add 25 ml of hydrochloric acid (4.7). Agitate and heat gently to dissolve the soluble salts. Allow the precipitate to settle for some minutes, then rinse the walls of the beaker with about 30 ml of water. Continue in accordance with 7.3.2.

7.3.2 Treatment of silica

Filter the solution containing the insoluble silica from 7.3.1.1 or 7.3.1.2 through a close-texture filter paper containing small pieces of filter paper or a small amount of filter pulp. Rinse the beaker with water, 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. Discard the filtrate and washings, or reserve for the determination of other elements. (See clause 9.)

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

Cool in a desiccator and weigh, to 0,000 1 g, as crude silica. 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 sulphuric acid (4.9) and, in accordance with the silica content, 5 to 15 ml of hydrofluoric acid (4.11). Heat gently in a fume cupboard to remove silica and sulphuric acid. Finally heat the crucible for 15 min in a muffle furnace controlled at 1 050 ± 20 °C. Cool in a desiccator and weigh, to 0,000 1 g, as the impurities. Repeat the treatment with sulphuric and hydrofluoric acids and the ignition, until constant mass is obtained (mass m_2).

8 Expression of results

8.1 Calculation of silicon content

The silicon content, as a percentage by mass, is calculated from the formula

$$\text{Si } \% \ (m/m) = \frac{m_1 - m_2}{m_3} \times 0{,}467\,4 \times 100 \times K$$

$$= \frac{m_1 - m_2}{m_3} \times 46{,}74 \times K$$

where

m_1 is the mass, in grams, of the platinum crucible containing 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 the conversion factor found from the formula

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

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 Acceptance of analytical values

The analytical values for the test sample shall be accepted when the respective analytical values obtained for the corresponding certified reference material agree with the certified value within the limit of permissible tolerance for certified reference materials, and when the difference between the two values for the test sample does not exceed the permissible tolerance for test samples (table 2).

When the analytical value obtained for the certified reference material is outside the limit of the permissible tolerance (table 2, second column), an analysis shall be carried out simultaneously on one test sample and one certified reference material, with one blank test. The analytical value obtained for the certified reference material shall be examined for the acceptance of the value for the test sample, as above. If the value obtained for the certified reference material is again outside the limit, the procedure shall be repeated with a different reference material of the same type of ore, until two acceptable values for the test sample are obtained.

When the range (absolute difference) of the two values for the test sample is outside the limit of the permissible tolerance (table 2, third column), additional analyses shall be carried out on one test sample with one blank test, as required by the procedure specified in annex A, simultaneously with an analysis of a certified reference material of the same type of ore. Acceptance of such additional values for the test sample shall be subject, in each case, to the acceptability of the value obtained for the certified reference material.

8.2.2 Calculation of final result

The arithmetic mean of the acceptable values of the test sample is the final result.

The arithmetic mean of the acceptable analytical values calculated to the fourth decimal place shall be 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 no figure other than 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 Permissible tolerances

The permissible tolerances described in 8.2.1 are shown in table 2.1)

Table 2 — Permissible tolerances within the laboratory

Silicon content	Certified reference material	Test sample Si (%)	
Si (%)	Si (%) 1,96 s_w	2,77 s_w	3,31 s_w
1 to 1,5	± 0,042	0,058	0,070
1,5 to 2,5	± 0,072	0,100	0,120
2,5 to 5	± 0,108	0,149	0,180
5 to 10	± 0,144	0,200	0,241
10 to 15	± 0,160	0,222	0,268

8.4 Oxide factor

$$SiO_2\,(\%) = 2{,}139 \times Si\,(\%)$$

9 Notes on procedure

The filtrate and washings from 7.3.1.2 and 7.3.2 can be used for the determination of other elements if they are combined with the residue after volatilization of the silica (7.3.2).

The residue can be fused with 2 g of sodium hydrogen sulphate (4.4). Attention should be paid to the fusion temperature, which should be kept as low as possible. After cooling, add 5 ml of hydrochloric acid (4.5) and 10 ml of water to dissolve the cooled melt.

Combine this solution with the solutions from 7.3.1.2 and 7.3.2 and evaporate to about 150 ml. Transfer to a 200 ml volumetric flask, dilute to the mark with water and mix. This solution can be used for determination of a number of elements.

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1) The derivation of the figures in the table is given in annex B.

10 Test report

The test report shall include the following information :

a) reference to this International Standard;

b) details necessary for the identification of the sample;

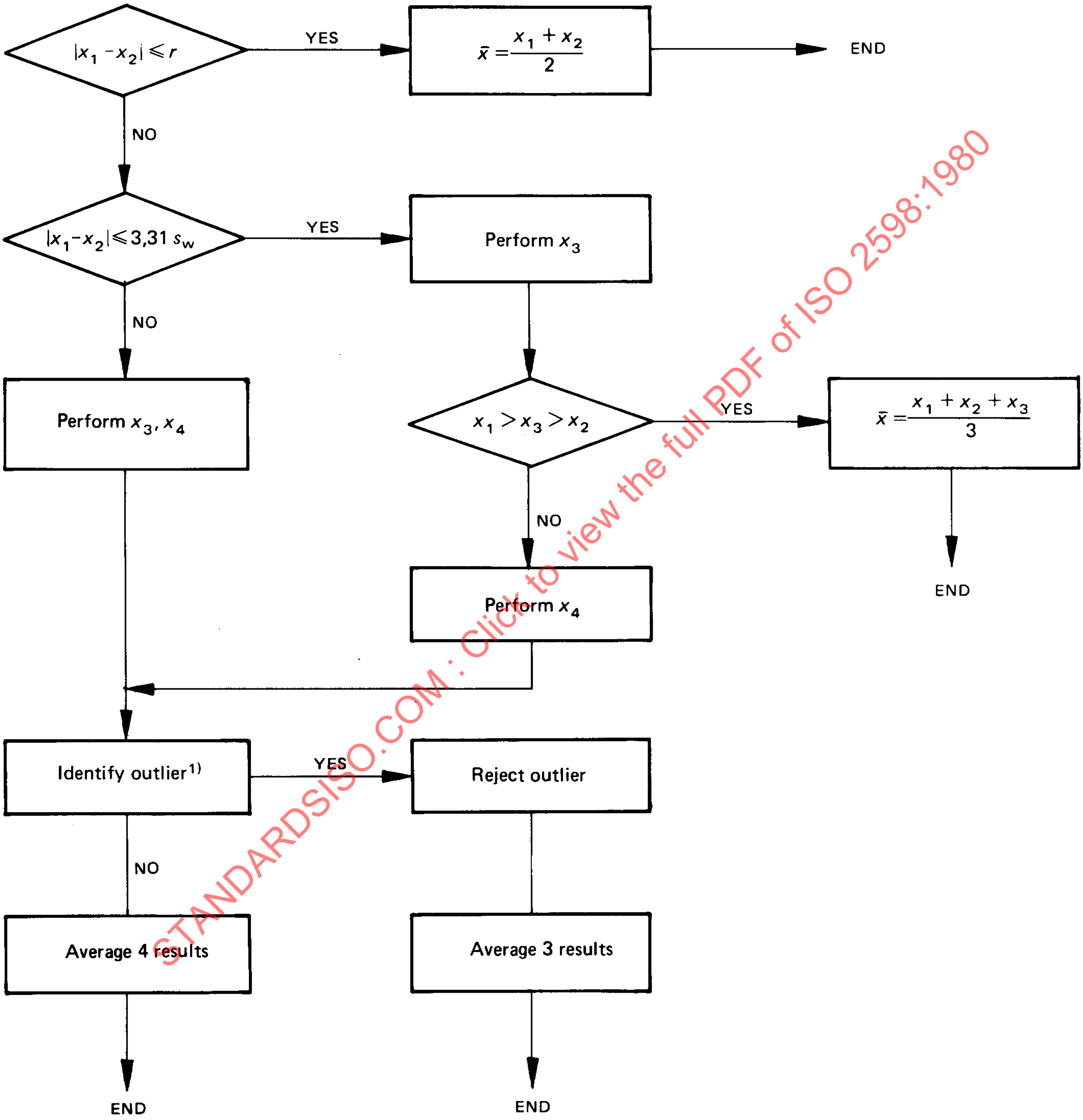
c) results of the analysis;

d) reference number of the results;

e) any characteristics noticed during the determination, and any operations not specified in this International Standard which may have had an influence on the results.

Annex A

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



r = repeatability

$= 2{,}77\ s_w$

$\bar{x}$ = average

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[1] Use Dixon's criteria for outlier.

Annex B

Derivation of permissible tolerances

The standard deviations within laboratories (s_w) were derived from a comparison of the results of international analytical trials carried out in 1967/1968 and 1970/1971 on several iron ores samples. The results of these trials were reported in Documents 102/2N 148 E, January 1969, and 102/2N 224 F, June 1971.

The permissible tolerances for the certified reference material (table 2, second column) are calculated by multiplying the standard deviations by the statistical factor 1,96 and relate to the upper limit of each range of contents respectively.

The permissible tolerances for the test sample (table 2, third column) are calculated by multiplying the standard deviations by the statistical factors 2,77 and 3,31 ($n = 2$ with 95 % confidence) and relate to the upper limit of each range of contents respectively.