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Iron ores — Determination of silicon content —

Part 1:

Gravimetric methods

Sample Document

Minerais de fer — Dosage du silicium —

Partie 1: Méthodes gravimétriques

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 2598-1 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Sub-Committee SC 2, *Chemical analysis*.

This part of ISO 2598 cancels and replaces ISO 2598:1980, of which it constitutes a technical revision.

ISO 2598 consists of the following parts, under the general title *Iron ores — Determination of silicon content*:

- *Part 1: Gravimetric methods*
- *Part 2: Reduced molybdosilicate spectrophotometric method*

Annex A forms an integral part of this part of ISO 2598. Annexes B and C are for information only.

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Iron ores — Determination of silicon content —

Part 1: Gravimetric methods

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.

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

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.

5.3 Nickel spatula.

5.4 Muffle furnaces, adjustable to $400\text{ }^\circ\text{C} \pm 20\text{ }^\circ\text{C}$ and to temperatures up to $1\ 050\text{ }^\circ\text{C}$.

6 Sampling and samples

6.1 Laboratory sample

For analysis, use a laboratory sample of minus $100\ \mu\text{m}$ 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 $160\ \mu\text{m}$.

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:

- processed ores containing metallic iron;
- natural or processed ores in which the sulfur content is higher than 0,2 % (m/m);
- 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 $105\text{ }^\circ\text{C} \pm 2\text{ }^\circ\text{C}$ as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

7.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with annex A, independently, on one test sample.

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 $400\text{ }^{\circ}\text{C} \pm 20\text{ }^{\circ}\text{C}$, 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.