
International Standard



2598

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Iron ores — Determination of silicon content — Gravimetric methods

Minerais de fer — Dosage du silicium — Méthodes gravimétriques

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

International Standard ISO 2598 was developed by Technical Committee ISO/TC 102, *Iron ores*, and was circulated to the member bodies in June 1978.

It has been approved by the member bodies of the following countries :

Australia	India	Poland
Austria	Iran	Romania
Bulgaria	Italy	South Africa, Rep. of
Canada	Japan	Sweden
Czechoslovakia	Korea, Dem. P. Rep. of	United Kingdom
Egypt, Arab Rep. of	Mexico	USA
France	Netherlands	USSR
Germany, F. R.	New Zealand	

No member body expressed disapproval of the document.

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 car-

bonate 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 3081¹⁾ and prepared in accordance with ISO 3083¹⁾ 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 more fumes in the beaker. Maintain this stage until most of the perchloric acid has evaporated, but avoid evaporation to dryness.

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.