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Iron ores -- Determination of silicon content -- Part 2: Reduced molybdosilicate
spectrophotometric method

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Minerais de fer -- Dosage du silicium -- Partie 2: Méthode spectrophotométrique au
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INTERNATIONAL STANDARD

**ISO
2598-2**

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Iron ores — Determination of silicon content —

Part 2:
Reduced molybdosilicate spectrophotometric method

Minerais de fer — Dosage du silicium —
Partie 2: Méthode spectrophotométrique au molybdosilicate réduit

Reference number
ISO 2598-2:1992(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, govern-mental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

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International Standard ISO 2598-2 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 4686:1980, 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.

INTERNATIONAL STANDARD
ISO 2598-2:1992(E)

Iron ores — Determination of silicon content —

Part 2:
Reduced molybdosilicate spectrophotometric method

1 Scope

This part of ISO 2598 specifies a reduced molybdosilicate spectrophotometric method for the determination of the silicon content of iron ores.

This method is applicable to silicon contents between 0,1 % (m/m) and 5,0 % (m/m) in natural iron ores, iron ore concentrates and agglomerates, including sinter products, especially for ores containing fluorine.

The fluorine content of the sample does not affect the determination.

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 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 fusion with sodium tetraborate or a fusion mixture (carbonate and tetraborate) and treatment with dilute nitric acid.

Addition of ammonium molybdate to convert the silicate into a molybdosilicate complex and reduction to molybdenum blue with ascorbic acid.

Spectrophotometric measurement of the absorbance of the molybdenum blue complex at a wavelength of approximately 600 nm.

4 Reagents

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

4.1 Sodium tetraborate ($Na_2B_4O_7$), anhydrous.

4.2 Potassium nitrate (KNO_3).

To be used as in note 6 of 7.4.1.

4.3 Fusion mixture.

Mix 100 g of anhydrous sodium carbonate (Na_2CO_3)/anhydrous potassium carbonate (K_2CO_3) mixture (1 + 1) with 30 g of anhydrous sodium tetraborate (4.1) and 0,5 g of potassium nitrate (4.2).

4.4 Nitric acid, ρ 1,4 g/ml, diluted 1 + 9.

1

4.5 Ammonium molybdate $[(NH_4)_6Mo_7O_{24}.4H_2O]$, solution, 120 g/l.

4.6 Oxalic acid $(C_2H_2O_4.2H_2O)$, solution, 50 g/l.

4.7 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 3.

4.8 Ascorbic acid $(C_6H_8O_6)$, solution, 20 g/l.

Prepare a fresh solution on the day of use.

4.9 Hydrogen peroxide, solution, 3 % (V/V).

Prepare by 1 + 9 dilution of hydrogen peroxide solution [30 % (V/V)].

To be used as in note 9 of 7.4.1.

4.10 Silicon standard solution, 50 µg Si/ml.

Calcine an appropriate quantity of silicon dioxide at 1 050 °C, cool, and weigh 53,5 mg ± 0,1 mg into a platinum crucible. Add 400 mg of iron(III) oxide and 4 g of sodium tetraborate (4.1) or fusion mixture (4.3) and mix.

Heat the mixture gently to a temperature of 1 100 °C (see 7.4.1) until a clear melt is obtained.

Cool and dissolve in 200 ml of nitric acid (4.4), transfer to a 500 ml one-mark volumetric flask, dilute to volume with water and mix.

4.11 Background solution.

In parallel with the silicon standard solution (4.10), prepare a background solution from 4 g of sodium tetraborate (4.1) or fusion mixture (4.3) and 400 mg of iron(III) oxide as described in 4.10, but without adding silicon dioxide.

5 Apparatus

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

5.1 Platinum crucibles, of capacity 30 ml.

5.2 Muffle furnace, capable of maintaining a temperature of approximately 1 100 °C.

5.3 Spectrophotometer, suitable for measurement of absorbance at approximately 600 nm.

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 1   A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

6.2 Preparation of predried test samples

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a way 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 predried test sample.

NOTE 2   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 0,5 g of the predried test sample obtained in accordance with 6.2.

NOTE 3   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.

NOTE 4   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

Mix the test portion (7.2) with 4 g of sodium tetraborate (4.1) or fusion mixture (4.3) in a platinum crucible (5.1). (See notes 5 and 6.)

Cover the platinum crucible and heat gently in a muffle furnace (5.2) up to a temperature of 1 100 °C. Maintain this temperature until the test portion is completely dissolved. (See note 7.)

Cool the platinum crucible (see note 8), place it in a 600 ml glass beaker containing 200 ml of nitric acid (4.4), and dissolve the cooled melt at a temperature below 90 °C. (See note 9.)

Cool the solution, wash into a 500 ml one-mark volumetric flask, dilute to volume with water and mix. (This is the test solution.)

NOTES

5 To minimize corrosive attack on the platinum crucible, it is recommended that 2 g of sodium tetraborate (4.1) or fusion mixture (4.3) be pre-melted in the crucible and swirled while cooling to cover the lower walls of the crucible. The remaining 2 g of sodium tetraborate or fusion mixture is mixed with the test portion on the surface of the cooled melt and then fused.

6 When fusing with sodium tetraborate (4.1), 0,1 g of potassium nitrate (4.2) should be added to samples containing reducing elements or sulfides, to accelerate the decomposition and to prevent any attack on the crucible.

7 Swirling the melt during the heating period greatly helps dissolution of the test portion. In general, 15 min will be sufficient; however, the fusion should be continued until the test portion is completely dissolved.

8 To accelerate the subsequent dissolution of the cooled melt, it is recommended that the melt be gently swirled during cooling, to enable it to solidify as a thin uniform film on the inner wall of the crucible. Slight heating during the swirling may help this process.

9 High manganese contents of the sample may cause cloudiness. In this case, add a few drops of hydrogen peroxide solution (4.9) and heat to boiling for the minimum time required to decompose the excess hydrogen peroxide.

7.4.2 Preparation of the blank test solution

In parallel with the fusion of the test portion, make a second fusion using all the reagents and following the procedure described in 7.4.1, but omitting the test portion.

Dissolve the cooled melt, treat it in the same way as the test solution, dilute it in a volumetric flask to 500 ml with water and mix.

7.4.3 Spectrophotometric determination

Transfer 5,0 ml of the test solution to a 100 ml volumetric flask. Add 2 ml of nitric acid (4.4) and 5,0 ml of ammonium molybdate solution (4.5).

Mix the solution thoroughly and allow to stand for 5 min.

Add the following reagents, in quick succession, shaking well after each addition: 10,0 ml of oxalic acid solution (4.6), 5,0 ml of sulfuric acid (4.7) and 5,0 ml of ascorbic acid solution (4.8). The maximum time allowed for the complete addition shall not exceed 3 min. After the final thorough mixing, allow the solution to stand for 1 min, dilute to volume with water and mix.

After a further 5 min, measure, in cells of 10 mm optical pathlength, the absorbances of the test solution and the blank test solution, against water as a reference. The portion of the absorption curve suitable for measurement is located at approximately 600 nm. Correct the absorbance of the test solution with the absorbance of the blank test solution.

NOTE 10 As an indication of the sensitivity of the method, the following information is provided: 0,250 mg Si/100 ml determined with a mercury vapour lamp and an Hg 578 filter results in an absorbance of 0,55 for an optical pathlength of 1 cm.

7.5 Calibration

To plot the calibration graph, measure aliquots of the silicon standard solution (4.10) and make up to 5,0 ml with appropriate quantities of the background solution (4.11) in accordance with table 1.

NOTE 11 During the preparation of the test solution and calibration solutions, the same fusion reagents should be used.

ISO 2598-2:1992(E)

Table 1 — Aliquots

Silicon standard solution (4.10) ml	Background solution (4.11) ml	Mass of Si in 5 ml aliquot mg	Content of Si in 0,50 g sample % (m/m)
0,00	5,0	0,00	0,0
0,10	4,9	0,005 0	0,10
0,20	4,8	0,010	0,20
0,50	4,5	0,025	0,50
1,00	4,0	0,050	1,00
3,00	2,0	0,150	3,00
5,00	0,0	0,25	5,00

Treat these solutions as indicated in 7.4.3, correcting the absorbances with the absorbance obtained for the zero calibration solution. Plot the absorbance calculated for an optical pathlength of 1 cm on the abscissa, against the known mass of silicon on the ordinate. Use the slope Z of the graph to calculate the silicon content as a percentage by mass.

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 equation

$$w_{Si} = \frac{A \times 10}{m} \qquad \ldots (1)$$

where

A is the absorbance of the test solution, corrected with the absorbance of the blank test solution (7.4.2) and reduced to an optical pathlength of 1 cm;

m is the mass, in grams, of the test portion.

8.2 General treatment of result

8.2.1 Repeatability and permissible tolerance

The precision of this analytical method is expressed by the following regression equations[1]:

$$r = 0,020\ 9\ X + 0,014\ 8 \qquad \ldots (2)$$

$$P = 0,026\ 4\ X + 0,115\ 1 \qquad \ldots (3)$$

$$\sigma_r = 0,007\ 4\ X + 0,005\ 2 \qquad \ldots (4)$$

$$\sigma_L = 0,007\ 8\ X + 0,041\ 7 \qquad \ldots (5)$$

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1) Additional information is given in annexes B and C.

where

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

— within-laboratory equations (2) and (4): the arithmetic mean of the duplicate values,

— between-laboratories equations (3) and (5): the arithmetic mean of the final result (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.

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 shall be used to test the significance of the difference:

$$|A_c - A| \leqslant 2\sqrt{\frac{s_{Lc}^2 + \frac{s_{Wc}^2}{n_{Wc}}}{N_c} + \sigma_L^2 + \frac{\sigma_r^2}{n}} \qquad \ldots (6)$$

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 (6) 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 (2) in 8.2.1, one or more additional tests shall be carried out in accordance with the flowsheet presented 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 12 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}^2 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

$$|A_c - A| \leqslant 2\sqrt{2\sigma_L^2 + \frac{\sigma_r^2}{n}} \qquad \ldots (7)$$

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.

The arithmetic mean of the acceptable analytical values, calculated to the fourth decimal place, is 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) results 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).