International Standard 5890

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Manganese ores and concentrates — Determination of silicon content — Gravimetric method

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Foreword

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Australia	Germany, F.R.	Romania
Austria	Hungary	South Africa, Rep. of
China	India	United Kingdom
Czechoslovakia	Italy	USSR
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France	Poland	

No member body expressed disapproval of the document.

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Manganese ores and concentrates — Determination of silicon content — Gravimetric method

1 Scope and field of application

This International Standard specifies a gravimetric method for the determination of the silicon content of manganese ores and concentrates.

The method is applicable to products having a silicon content greater than 0,5 % (*m/m*) and a fluorine content not more than 0,1 % (*m/m*).

This International Standard should be read in conjunction with ISO 4297.

2 Reference

ISO 4297, *Manganese ores and concentrates — Methods of chemical analysis — General instructions.*

3 Principle

Decomposition of a test portion by treatment with hydrochloric, nitric and perchloric acids. Filtration of the residue containing the silicon and reservation of the filtrate as the main solution. Ignition of the filter paper with the residue. Fusion of the residue with sodium carbonate; leaching of the melt with hydrochloric acid and combination with the main solution.

Separation of the silica by evaporation of the solution with perchloric acid and weighing of the silica with impurities. Volatilization of the silica with hydrofluoric and sulphuric acids, weighing of the residue and calculation of the silica content by the difference in mass.

4 Reagents

4.1 Sodium carbonate, anhydrous.

4.2 Nitric acid, ϱ 1,40 g/ml.

4.3 Sulphuric acid, diluted 1 + 1.

4.4 Hydrochloric acid, ϱ 1,19 g/ml.

4.5 Hydrochoric acid, diluted 1 + 4.

4.6 Hydrochloric acid, diluted 1 + 9.

4.7 Hydrofluoric acid, ϱ 1,14 g/ml.

4.8 Perchloric acid, ϱ 1,51 g/ml.

4.9 Hydrogen peroxide, 30 g/l solution.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Muffle furnace, capable of being controlled at a temperature between 1 000 and 1 100 °C.

5.2 Platinum crucibles.

6 Procedure

6.1 Test portion

Weigh a mass of the test sample, chosen from table 1 in accordance with the expected silicon content.

Table 1

Expected silicon content	Mass of test portion
% (*m/m*)	g
From 0,5 to 2	2,0
From 2 to 10	1,0
From 10 to 20	0,5

6.2 Decomposition of test portion

Place the test portion (6.1) in a 250 or 300 ml beaker, moisten with a few drops of water, add 15 to 30 ml of the hydrochloric acid (4.4) and heat gently to decompose the ore. Add 2 to 3 ml of the nitric acid (4.2), heat until nitrous fumes cease to be evolved, and allow to cool. Add 10 ml of the perchloric acid (4.8), and heat gently until dense white fumes of perchloric acid appear.

Allow the solution to cool, add 30 to 40 ml of hot water and a few drops of the hydrogen peroxide solution (4.9). Heat to clear the solution and filter it through a medium-texture filter paper containing a small amount of paper pulp. Transfer the residue from the beaker to the filter using a rubber-tipped glass rod.

Wash the residue and the beaker with hot hydrochloric acid (4.6) 4 to 6 times, then with hot water 3 to 4 times. Reserve the filtrate as the main solution.

6.3 Dissolution of residue

Transfer the filter paper containing the residue to a platinum crucible (5.2). Heat gently to dry, ignite and ash in the muffle furnace (5.1), controlled at a temperature between 700 and 750 °C. Allow to cool, add 3 g of the sodium carbonate (4.1), mix and fuse at a temperature between 1 000 to 1 100 °C, until a homogeneous melt is obtained.

Allow the crucible with the melt to cool, and place it in the beaker in which the test portion has been decomposed. Add 50 ml of hot hydrochloric acid (4.5) and heat to dissolve the melt. Remove the crucible from the beaker and rinse it with water. Add the solution thus obtained to the main solution.

6.4 Separation of silica

Add 30 ml of the perchloric acid (4.8) to the solution obtained in 6.3 and heat until white fumes of perchloric acid appear. Cover the beaker with a watch-glass and continue heating to maintain this stage until most of the perchloric acid has evaporated, but avoid evaporation to dryness.

Allow the solution to cool, add 40 to 50 ml of hot water and 2 or 3 drops of the hydrogen peroxide solution (4.9). Heat to dissolve the soluble salts until the solution is clear, then filter it immediately through a medium-texture filter paper containing a small amount of paper pulp. Transfer the silica precipitate from the beaker to the filter paper using a rubber-tipped glass rod.

Wash the precipitate on the filter paper and the beaker with cold water 2 or 3 times, then with hot hydrochloric acid (4.6) 6 to 8 times, and finally with cold water 2 or 3 times. For samples having a silicon content up to 10 % (m/m), discard the filtrate and washings. For samples having a silicon content greater than 10 % (m/m), add 20 ml of the perchloric acid (4.8) to the filtrate and repeat the evaporation. Filter through a new filter paper and treat the combined precipitates as in 6.5.

6.5 Treatment of silica precipitate

Place the filter paper with the residue in a platinum crucible (5.2). Heat gently to dry, ignite and ash in the muffle furnace (5.1), controlled at a temperature between 700 and 750 °C, then raise the temperature to 1 000 to 1 100 °C. Hold at this temperature for 1 h (until constant mass is obtained). Allow the crucible containing crude silica to cool in a desiccator. Weigh the crucible with the precipitate (m_1). Moisten the precipitate in the crucible with a few drops of water, add 5 drops of the sulphuric acid (4.3) and 5 to 10 ml of the hydrofluoric acid (4.7), and evaporate the solution to remove silicon and sulphuric acid.

Then place the crucible containing the impurities in the muffle furnace and ash at 1 000 to 1 100 °C until constant mass is obtained.

Allow the crucible to cool, and weigh (m_2).

6.6 Blank test

Carry out a blank test following all the stages of the analysis.

7 Expression of results

7.1 Calculation

The silicon (Si) content, expressed, as a percentage by mass, is given by the formula

$$\frac{0{,}467\,4 \times [(m_1 - m_2) - (m'_1 - m'_2)] \times 100}{m_0} \times K$$

where

m_0 is the mass, in grams, of the test portion;

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

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

m'_1 is the mass, in grams, of the crucible containing the crude silica in the blank test;

m'_2 is the mass, in grams, of the crucible containing the impurities in the blank test;

K is the conversion factor for the expression of the silicon content on the dry basis.

NOTE — If it is required to express the silicon content as a percentage by mass as silica (SiO_2), multiply the result obtained by the factor 2,139.

7.2 Permissible tolerances on results of parallel determinations

Table 2

Silicon content	Permissible tolerance	
	Two parallel determinations	Three parallel determinations
% (m/m)	% (m/m)	% (m/m)
From 0,5 to 1,0	0,06	0,07
From 1,0 to 2,0	0,08	0,10
From 2,0 to 5,0	0,13	0,15
From 5,0 to 10,0	0,18	0,20
From 10,0 to 20,0	0,26	0,30