
International Standard



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Manganese ores and concentrates — Determination of titanium content — 4,4'-Diantipyrilmethane spectrometric method

Minerais et concentrés de manganèse — Dosage du titane — Méthode spectrométrique au diantipyrilméthane-4,4'

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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.

International Standard ISO 7723 was developed by Technical Committee ISO/TC 65, *Manganese and chromium ores*, and was circulated to the member bodies in February 1983.

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

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

No member body expressed disapproval of the document.

Manganese ores and concentrates — Determination of titanium content — 4,4'-Diantipyrilmethane spectrometric method

1 Scope and field of application

This International Standard specifies a spectrometric method using 4,4'-diantipyrilmethane for the determination of the titanium content of manganese ores and concentrates.

The method is applicable to products having a titanium content of 0,01 to 0,5 % (m/m).

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

2 References

ISO 4296/1, *Manganese ores — Sampling — Part 1: Increment sampling*.

ISO 4296/2, *Manganese ores — Sampling — Part 2: Preparation of samples*.

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 sulfuric acids.

Separation of the insoluble residue, the filtrate being reserved as the main solution.

Ignition of the filter containing the residue and treatment with sulfuric and hydrofluoric acids.

Fusion of the ignited residue with potassium disulfate.

Dissolution of the melt in sulfuric acid and combination of the solution obtained with the main solution.

Reduction of iron with ascorbic acid.

Colour development with 4,4'-diantipyrilmethane and measurement of the absorbance at 385 nm.

4 Reagents

4.1 Potassium disulfate ($K_2S_2O_7$).

4.2 Hydrochloric acid, ρ 1,19 g/ml.

4.3 Hydrochloric acid, diluted 1 + 1.

4.4 Hydrochloric acid, diluted 1 + 50.

4.5 Nitric acid ρ 1,42 g/ml.

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

4.7 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 9.

4.8 Hydrofluoric acid, ρ 1,14 g/ml, 40 % (m/m) solution.

4.9 Ascorbic acid ($C_6H_8O_6$), 100 g/l solution.

This solution shall be freshly prepared.

4.10 4,4'-Diantipyrilmethane monohydrate ($C_{23}H_{24}O_2N_4 \cdot H_2O$), 50 g/l solution.

Dissolve 5 g of 4,4'-diantipyrilmethane monohydrate in 70 ml of hydrochloric acid, diluted 1 + 10. Filter, if necessary. Transfer the solution into a one-mark volumetric flask of capacity 100 ml, and dilute with the same acid to the mark.

This solution shall be freshly prepared.

4.11 Titanium, standard solution corresponding to 100 mg of Ti per litre.

Prepare this solution by one of the following two methods.

- Weigh 0,167 0 g of titanium dioxide previously calcined at 900 °C for 40 min, in a platinum crucible fitted with a lid. Fuse with 3 to 4 g of potassium disulfate (4.1) at 650 to 700 °C until a clear melt is obtained. Cool the crucible and place it with the melt in a beaker of capacity 200 ml, add 50 ml of sulfuric acid (4.7) and dissolve the melt using moderate heat. Remove the platinum crucible from the beaker after washing with sulfuric acid (4.7). Transfer the solution to a one-mark volumetric flask of capacity 1 000 ml, dilute with sulfuric acid (4.7) to the mark and mix.

b) Weigh 0,100 g of metallic titanium (purity 99,95 %) and dissolve it in 20 ml of the sulfuric acid (4.6). After dissolution add 2 to 3 drops of nitric acid (4.5) to the solution and evaporate until fumes of sulfuric acid appear. Cool and dissolve the residue in 50 ml of hot sulfuric acid (4.7). Cool the solution and transfer it to a one-mark volumetric flask of capacity 1 000 ml, dilute with sulfuric acid (4.7) to the mark and mix.

1 ml of either of these solutions contains 100 µg of Ti.

4.12 Titanium, standard solution corresponding to 20 mg of Ti per litre.

Pipette 50 ml of titanium standard solution (4.11) into a one-mark volumetric flask of capacity 250 ml, dilute with sulfuric acid (4.7) to the mark and mix.

1 ml of this solution contains 20 µg of Ti.

5 Apparatus

Usual laboratory apparatus and

5.1 Platinum crucible.

5.2 Spectrometer, with selectors for continuous or discontinuous variation, suitable for use at 385 nm.

6 Sample

For the sampling of manganese ores, see ISO 4296/1. For the preparation of samples, see ISO 4296/2.

Use a test sample which has been crushed to a size not exceeding 100 µm (checked on a sieve of appropriate aperture size) and air dried under laboratory conditions.

7 Procedure

7.1 Test portion

Weigh 1 g of the test sample.

7.2 Blank test

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

7.3 Decomposition of test portion

Place the test portion (7.1) in a beaker of capacity 200 ml, moisten with a few drops of water, add 30 ml of hydrochloric acid (4.2), cover the beaker with a watch-glass and heat to decompose the test portion completely. Add 5 ml of the nitric acid (4.5).

Heat until fumes of nitrogen oxides cease to evolve. Cool, add 20 ml of sulfuric acid (4.6) and evaporate until dense white fumes appear. Cool, add 20 ml of hydrochloric acid (4.3), 50 ml

of hot water and heat to dissolve the salts. Filter the insoluble residue on a medium-texture filter paper containing a small amount of paper pulp. Remove adherent particles of precipitate from the walls of the beaker with a rubber-tipped glass rod and transfer the residue to the filter paper. Wash the beaker and the filter with the residue with hot hydrochloric acid (4.4) two or three times and then with hot water eight to ten times. Reserve the filtrate as the main solution.

7.4 Treatment of residue

Transfer the filter containing the residue to a platinum crucible (5.1). Dry and ignite at 500 to 600 °C. Cool the crucible, moisten the residue with 2 or 3 drops of water, add 5 or 6 drops of sulfuric acid (4.6), 5 to 7 ml of hydrofluoric acid (4.8) and heat gently until the fumes of sulfuric acid cease to evolve.

Allow the crucible to cool, add 1 g of potassium disulfate (4.1), cover the crucible with a platinum lid and fuse at 650 to 700 °C. Place the cooled crucible and lid in a beaker of capacity 200 ml, add 30 ml of sulfuric acid (4.7) and heat to dissolve the melt. Remove the crucible, rinse it with sulfuric acid (4.7) over the beaker. Combine the solution with the main solution (7.3) and evaporate to about 150 ml, if necessary. Transfer the obtained solution to a one-mark volumetric flask of capacity 200 ml, dilute with water to the mark and mix.

7.5 Preparation of solution for spectrometric measurement

Before taking aliquots, if necessary (i.e. when precipitates of barium and lead sulfates are present), filter the test solution through a dry, fine-texture paper into a dry flask, discarding the first runnings of filtrate.

Introduce a suitable aliquot (see table 1) of test solution into a one-mark volumetric flask of capacity 100 ml. Add 10 ml of ascorbic acid solution (4.9) and shake. Allow to stand for 5 min. Add 15 ml of hydrochloric acid (4.3) and 10 ml of 4,4'-dianthiprylmethane solution (4.10). Dilute the solution with water to the mark and mix.

Allow the solution to stand for 20 min until a stable coloration is obtained.

Table 1

Expected titanium content % (m/m)	Aliquot ml	Titanium content in the aliquot µg
≥ 0,01 to 0,10	20	≥ 10 to 100
> 0,10 to 0,30	10	> 50 to 150
> 0,30 to 0,50	5	> 75 to 125

7.6 Spectrometric measurement

Measure the absorbance of the solution in a cell of thickness 10 to 30 mm (depending on the titanium content), using the spectrometer (5.2) equipped with a filter giving maximum transmission in the wavelength range 380 to 400 nm or using the spectrometer with selectors for continuous variation at a wavelength of 385 nm against water as reference.

7.7 Preparation of calibration graph

Into a series of eight one-mark volumetric flasks of capacity 100 ml, introduce, using a burette 0,0; 0,5; 1,0; 2,0; 4,0; 6,0; 8,0 and 10 ml of titanium standard solution (4.12), corresponding to 0,0; 10; 20; 40; 80; 120; 160 and 200 µg of titanium. The first volumetric flask serves for the preparation of the calibration compensation solution. Add 10 ml of ascorbic acid solution (4.9) and shake. Allow to stand for 5 min, add 15 ml of hydrochloric acid (4.3) and 10 ml of the 4,4'-dianthipyrylmethane solution (4.10), dilute the solution with water to the mark, mix and after 20 min, measure the absorbance according to 7.6 using the cell which was taken for measurement of the absorbance of the test solutions.

Prepare a calibration graph by plotting the absorbance values (deducting the absorbance value of the calibration compensation solution) against the nominal titanium contents of the calibration solutions.

8 Expression of results

8.1 Calculation

Convert the net absorbance reading for the test solution (obtained by subtracting the absorbance reading of the blank test solution from that of the test solution) to titanium content by means of the calibration graph (7.7).

The titanium content (Ti), expressed as a percentage by mass, is given by the formula

$$\frac{m_1 \times 100}{m_0 \times 1\,000} \times K = \frac{m_1}{m_0 \times 10} \times K$$

where

m_0 is the mass, in grams, of the test portion corresponding to the aliquot portion of the test solution;

m_1 is the mass, in milligrams, of titanium in the aliquot portion of the test solution, obtained from the calibration graph;

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

8.2 Permissible tolerances on results of parallel determinations

Table 2

Titanium content	Permissible tolerance	
	Three parallel determinations	Two parallel determinations
% (m/m)	% (m/m)	% (m/m)
> 0,01 to 0,02	0,010	0,009
> 0,02 to 0,04	0,015	0,014
> 0,04 to 0,08	0,020	0,019
> 0,08 to 0,15	0,030	0,025
> 0,15 to 0,25	0,040	0,035
> 0,25 to 0,50	0,050	0,045

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