
International Standard



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Manganese ores and concentrates — Determination of potassium and sodium content — Flame atomic emission spectrometric method

Minerais et concentrés de manganèse — Dosage du sodium et du potassium — Méthode par spectrométrie d'émission atomique de flamme

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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 4571 was developed by Technical Committee ISO/TC 65, *Manganese and chromium ores*, and was circulated to the member bodies in October 1979.

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

Austria	Hungary	South Africa, Rep. of
Bulgaria	India	Thailand
China	Italy	United Kingdom
Czechoslovakia	Japan	USSR
Egypt, Arab Rep. of	Poland	
France	Romania	

No member body expressed disapproval of the document.

Manganese ores and concentrates — Determination of potassium and sodium content — Flame atomic emission spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic emission spectrometric method for the determination of the potassium and/or sodium content of manganese ores and concentrates. The method is applicable to a concentration range of 0,08 to 3 % (*m/m*) of potassium and 0,02 to 1 % (*m/m*) of sodium.

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 of low iron content by treatment with nitric, hydrofluoric and sulphuric acids. Removal of hydrofluoric acid by evaporation of the solution until the fumes of sulphuric acid disappear completely. Treatment of the solution with sulphuric acid and hydrogen peroxide to dissolve manganese dioxide.

Decomposition of a test portion of high iron content by treatment with hydrochloric, nitric, hydrofluoric and perchloric acids. Removal of hydrofluoric acid by evaporation of the solution until dense fumes of perchloric acid are evolved. Treatment of the solution with hydrochloric acid to dissolve manganese dioxide.

Addition of a caesium nitrate solution and appropriate dilution. Aspiration of the solution into an air/acetylene flame and measuring the emission at wavelength 766,5 to 769,7 nm for potassium and at 589,0 to 589,6 nm for sodium.

The interference of calcium is eliminated by the use of monochromators and the mutual interference of alkali elements (sodium, lithium and others) by a caesium nitrate solution.

4 Reagents

4.1 Iron, purity 99,99 %.

4.2 Potassium chloride.

Dry at 110 °C to constant mass before use.

4.3 Electrolytic manganese, purity not less than 99,95 %.

Place 10 g of electrolytic manganese in a 400 ml beaker, treat the surface layer with a mixture of 50 ml of water and 5 ml of the nitric acid (4.5) for several minutes until a bright surface is obtained. Wash the manganese thus obtained six times with water and then with acetone, and dry at 100 °C for 10 min.

4.4 Sodium chloride.

Dry at 110 °C to constant mass before use.

4.5 Nitric acid, ρ 1,40 g/ml.

4.6 Hydrofluoric acid, ρ 1,14 g/ml.

4.7 Sulphuric acid, diluted 1 + 1.

4.8 Hydrogen peroxide, 30 % (*m/V*), free from alkali metals.

4.9 Hydrochloric acid, ρ 1,19 g/ml.

4.10 Perchloric acid, ρ 1,51 g/ml.

4.11 Caesium nitrate, 15 g/l solution.

4.12 Iron, standard solution, corresponding to 10 g of Fe per litre.

Place 2,5 g of the iron (4.1) in a 400 ml beaker. Dissolve in 15 ml of the nitric acid (4.5) and 30 ml of water, then add 15 ml of the sulphuric acid (4.7). Evaporate the solution to fumes of sulphuric acid, allow to cool and dilute with water. Transfer to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 10 mg of Fe.

4.13 Manganese, standard solution, corresponding to 25 g of Mn per litre.

Place 6,25 g of the manganese (4.3) in a 400 ml beaker and dissolve in 30 ml of the hydrochloric acid (4.9) and 30 ml of water. Then add 35 ml of the sulphuric acid (4.7) and evaporate to fumes of sulphuric acid, allow to cool and dilute with water. Transfer to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 25 mg of Mn.

4.14 Potassium, standard solution, corresponding to 0,1 g of K per litre.

Weigh 0,190 7 g of the potassium chloride (4.2) into a 400 ml beaker and dissolve in 200 ml of water. Transfer the solution into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,1 mg of K.

Store the solution in polyethylene flasks.

4.15 Sodium, standard solution, corresponding to 0,1 g of Na per litre.

Weigh 0,254 2 g of the sodium chloride (4.4) into a 400 ml beaker and dissolve in 200 ml of water. Transfer the solution to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,1 mg of Na.

Store the solution in polyethylene flasks.

5 Apparatus

NOTES

- 1 All apparatus should be rinsed with hydrochloric acid and then with distilled water.
- 2 All standard and calibration solutions should be stored in plastic bottles.

Ordinary laboratory apparatus and

5.1 Flame spectrometer, with a monochromator providing suitable sensitivity and precision.

5.2 Air-acetylene burner.

5.3 Dishes, of platinum or any other material suitable for treatment with hydrofluoric acid, capacity 300 ml.

5.4 Beakers, of fluoroplastic or polytetrafluoroethylene, capacity 250 to 300 ml.

6 Procedure

6.1 Blank test

Before proceeding to the treatment of the test portion, ensure that the reagents being used have produced a blank value for the sodium and potassium determinations in each case not greater than the equivalent of 0,002 % (*m/m*) alkali content in the ore or concentrate.

6.2 Decomposition of test portion

- a) For materials of low iron content

Place 0,1 to 0,5 g (depending on the element content to be determined) of the test portion in a dish (5.3) and dissolve in the following acid mixture :

3 ml of the nitric acid (4.5), 10 ml of the hydrofluoric acid (4.6) and 10 ml of the sulphuric acid (4.7).

Evaporate the solution until fumes of sulphuric acid are no longer evolved, then allow to cool. Wash down the sides of the dish with 10 to 15 ml of water, add 1 ml of the sulphuric acid (4.7) and 10 to 20 drops of the hydrogen peroxide (4.8). Heat the solution to dissolve manganese dioxide and to decompose completely the excess of hydrogen peroxide.

- b) For materials of high iron content

Place 0,1 to 0,5 g (depending on the element content to be determined) of the test portion in a beaker (5.4) and dissolve in 10 ml of the hydrochloric acid (4.9). Then add 1 to 2 ml of the nitric acid (4.5) and 5 to 10 ml of the hydrofluoric acid (4.6), depending on the silica content. Add 5 ml of the perchloric acid (4.10), heat the solution until white fumes of perchloric acid are evolved and allow the solution to stand for 2 min. Wash down the sides of the beaker with distilled water and heat again until dense fumes of perchloric acid are evolved. Allow to cool. Dissolve the residue in 5 ml of the hydrochloric acid (4.9) and in 10 to 15 ml of water, while heating.

6.3 Preparation of solutions for spectrometric measurement

Transfer the solutions obtained to 100 ml one-mark volumetric flasks, add 2 ml of the caesium nitrate solution (4.11), dilute to the mark with water and mix.

If the solutions are turbid, filter them through a dry, medium-texture filter paper into dry flasks, discarding the first portions of the filtrate.

Carry out a blank test throughout the analysis, in order to make corrections for potassium and sodium contents.

6.4 Spectrometric measurement

Aspirate the solutions obtained into the air/acetylene flame of the spectrometer (5.1) and measure the emission at a wavelength of 766,5 to 769,7 nm for potassium and 589,0 to 589,6 nm for sodium.

To eliminate the effect of the gas pressure, repeat the entire series of measurements and take an average value of the readings obtained.

Convert the emission reading of the test solution to micrograms of sodium and potassium per millilitre by means of the calibration graph or by the bracketing method with subtraction of the emission reading of the blank solution.

6.5 Preparation of calibration graph

6.5.1 Simultaneously with the analysis, analyse a series of standard solutions. For their preparation, transfer iron solutions

(4.12) and manganese solutions (4.13) in quantities corresponding to their contents in the analysed material to dishes (5.3) or beakers (5.4), then add the standard potassium solution (4.14) and the standard sodium solution (4.15) within the concentration range from 0 to 40 µg/ml.

The blank solution is obtained without addition of standard potassium and sodium solutions.

Prepare calibration graphs by plotting the emission readings, after subtraction of the emission readings of the blank solution, against the corresponding potassium and sodium content.

6.5.2 For the determination of potassium and sodium content by the bracketing method, compare the emission of the test solution with that of two standard solutions obtained as stated in 6.5.1, the emission of one being not more than 10 % higher and the emission of the second not more than 10 % lower than the emission of the test solution.

7 Expression of results

7.1 Calculation

The potassium (K) or sodium (Na) content is given, as a percentage by mass, by the formula (1) :

$$\frac{c \times V \times K \times 100}{m \times 10^6} \dots (1)$$

where

c is the concentration, in micrograms per millilitre, of potassium or sodium in the test solution, obtained from the calibration graph or by formula (2);

V is the volume, in millilitres, of the test solution;

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

K is the conversion factor for the expression of the potassium or sodium content on the dry basis.

The value of *c* for the bracketing method is given by the formula (2) :

$$c = c_1 + \frac{(c_2 - c_1) \times (b - b_1)}{b_2 - b_1} \dots (2)$$

where

*c*₁ is the content, in micrograms per millilitre, of potassium or sodium in the standard solution the concentration of which is less than that of the test solution;

*c*₂ is the content, in micrograms per millilitre, of potassium or sodium in the standard solution the concentration of which is higher than that of the test solution;

b is the emission of the test solution, corrected for the emission of the blank solution;

*b*₁ is the emission of the standard solution containing iron and manganese and having concentration *c*₁, corrected for the emission of the blank solution;

*b*₂ is the emission of the standard solution containing iron and manganese and having concentration *c*₂, corrected for the emission of the blank solution.

7.2 Permissible tolerances on results of parallel determinations

7.2.1 Determinations of potassium content

Table 1

Potassium content	Permissible tolerance	
	Three parallel determinations	Two parallel determinations
% (m/m)	% (m/m)	% (m/m)
From 0,05 to 0,10	0,013	0,011
From 0,10 to 0,20	0,021	0,018
From 0,20 to 0,40	0,032	0,027
From 0,40 to 0,80	0,050	0,042
From 0,80 to 1,50	0,076	0,064
Over 1,50	0,120	0,100

7.2.2 Determinations of sodium content

Table 2

Sodium content	Permissible tolerance	
	Three parallel determinations	Two parallel determinations
% (m/m)	% (m/m)	% (m/m)
From 0,02 to 0,05	0,008	0,007
From 0,05 to 0,10	0,013	0,011
From 0,10 to 0,20	0,021	0,018
From 0,20 to 0,40	0,032	0,027
From 0,40 to 0,80	0,050	0,042
From 0,80 to 1,50	0,076	0,064

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