SLOVENSKI STANDARD

SIST ISO 5889:2001

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Manganove rude in koncentrati - Doloļ evanje aluminija, bakra, svinca in cinka - Metoda plamenske atomske absorpcijske spektrometrije

Manganese ores and concentrates -- Determination of aluminium, copper, lead and zinc contents -- Flame atomic absorption spectrometric method

Minerais et concentrés de manganèse -- Dosage de l'aluminium, du cuivre, du plomb et du zinc -- Méthode par spectrométrie d'absorption atomique dans la flamme

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International Standard ISO 5889

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Manganese ores and concentrates — Determination of aluminium, copper, lead and zinc contents — Flame atomic absorption spectrometric method

Minerais et concentrés de manganèse — Dosage de l'aluminium, du cuivre, du plomb et du zinc — Méthode par spectrométrie d'absorption atomique dans la flamme

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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 5889 was developed by Technical Committee ISO/TC 65, *Manganese and chromium ores*, and was circulated to the member bodies in May 1982.

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

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

The member body of the following country expressed disapproval of the document on technical grounds :

France

Printed in Switzerland

Manganese ores and concentrates — Determination of aluminium, copper, lead and zinc contents — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the aluminium, copper, lead and zinc contents of manganese ores and concentrates.

The method is applicable to a concentration range of

0,1 to 4,0 % (*m/m*) of aluminium;

0,005 to 1,0 % (*m/m*) of copper;

0,005 to 1,0 % (*m/m*) of lead;

0,005 to 0,25 % (*m/m*) of zinc.

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

2 References

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

ISO 4296/2, *Manganese ores — Sampling — Part 2 : Preparation of samples.*[1]

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

3 Principle

Decomposition of a test portion by treatment with hydrochloric and nitric acids.

Filtration of any insoluble residue after evaporation, the filtrate being reserved as the main solution. Ignition of the filter containing the residue. Treatment with hydrofluoric and sulfuric acids. Fusion with a fusion mixture. Dissolution of the melt in hydrochloric acid and combination of the solution obtained with the main solution.

Aspiration of the solution into the flame of an atomic absorption spectrometer using an air/acetylene burner for copper, lead and zinc and dinitrogen oxide/acetylene burner for aluminium.

Comparison of absorbance values obtained with those obtained from the calibration solutions.

4 Reagents

4.1 Fusion mixture : 3 parts of potassium carbonate and 1 part of boric acid.

4.2 Nitric acid, ϱ 1,40 g/ml.

4.3 Sulfuric acid, ϱ 1,84 g/ml.

4.4 Hydrochloric acid, ϱ 1,19 g/ml.

4.5 Hydrofluoric acid, 40 % (*m/m*), ϱ 1,14 g/ml.

4.6 Hydrochloric acid, diluted 1 + 1.

4.7 Hydrochloric acid, diluted 1 + 50.

4.8 Aluminium, standard solution corresponding to 1 g of Al per litre.

Dissolve 0,500 0 g of high purity aluminium (purity 99,99 %) by adding 25 ml of the hydrochloric acid (4.4) while heating. When dissolved, allow the solution to cool, transfer it to a 500 ml one-mark volumetric flask, dilute with water to the mark and mix.

1 ml of this standard solution contains 1 mg of Al.

4.9 Lead, standard solution corresponding to 1 g of Pb per litre.

Dissolve 1,000 0 g of high purity lead (purity 99,999 %) by adding 10 ml of the nitric acid (4.2) diluted 1 + 4 while heating. When dissolved, allow the solution to cool, transfer it to a 1 000 ml one-mark volumetric flask, dilute with water to the mark and mix.

1 ml of this standard solution contains 1 mg of Pb.

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[1] At present at the stage of draft.

4.10 Lead, standard solution corresponding to 0,05 g of Pb per litre.

Transfer 25 ml of the standard lead solution (4.9) to a 500 ml one-mark volumetric flask, dilute with water to the mark and mix.

1 ml of this standard solution contains 0,05 mg of Pb.

4.11 Copper, standard solution corresponding to 0,1 g of Cu per litre.

Dissolve 0,100 0 g of high purity copper (purity 99,99 %) by adding 5 ml of the nitric acid (4.2) while heating. When dissolved, allow the solution to cool, transfer it to a 1 000 ml one-mark volumetric flask, dilute with water to the mark and mix.

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

4.12 Zinc, standard solution corresponding to 0,1 g of Zn per litre.

Dissolve 0,100 0 g of high purity zinc (purity 99,99 %) by adding 10 ml of the hydrochloric acid (4.6) while heating. When dissolved, allow the solution to cool, transfer it to a 1 000 ml one-mark volumetric flask, dilute with water to the mark and mix.

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

4.13 Background solution.

Place 12,50 g of high purity manganese and 1,25 g of high purity iron in a 1 500 ml beaker and dissolve in 625 ml of the hydrochloric acid (4.6) and 25 ml of the nitric acid (4.2) while heating. Add 18,75 g of potassium carbonate and 6,25 g of boric acid and heat to dissolve. Allow the solution to cool, transfer it to a 1 000 ml one-mark volumetric flask, dilute with water to the mark and mix.

NOTE — The background solution should be absolutely free from the elements to be determined.

4.14 Copper, lead, zinc and aluminium calibrations solutions

From the standard copper (4.11), lead (4.9, 4.10), zinc (4.12) and aluminium (4.8) solutions, prepare calibration solutions as follows :

To each of a series of 100 ml one-mark volumetric flasks, transfer 40 ml of background solution (4.13). Add volumes of standard copper (4.11), lead (4.9, 4.10), zinc (4.12) and aluminium solutions (4.8) according to table 1, dilute with water to the mark and mix (see the note to table 1).

5 Apparatus

Ordinary laboratory apparatus and

5.1 Platinum crucible.

5.2 Atomic absorption spectrometer, equipped with an air/acetylene and a dinitrogen oxide/acetylene burner.

The atomic absorption spectrometer used in this method will be satisfactory if it meets the following criteria :

a) *Minimum sensitivity :* the absorbance of the highest calibration solution (see 4.14) shall be at least 0,3.

b) *Curve linearity :* the slope of the calibration curve covering the top 20 % concentration range (expressed as a change in absorbance) shall not be less than 0,7 of the value of the slope for the bottom 20 % concentration range determined in the same way.

c) *Minimum stability :* the standard deviation of the absorbance of the most concentrated calibration solution and the standard deviation of the absorbance of the zero calibration solution, each being calculated from a sufficient number of repetitive measurements, shall be less than 1,5 % and 0,5 %, respectively, of the mean value of the absorbance of the most concentrated solution.

Table 1 — Standard solutions to be added for preparation of calibration solutions

Al		Cu		Pb				Zn	
Standard solution 1 mg/ml (4.8)	Content in calibration solution	Standard solution 0,1 mg/ml (4.11)	Content in calibration solution	Standard solution 1 mg/ml (4.9)	Content in calibration solution	Standard solution 0,05 mg/ml (4.10)	Content in calibration solution	Standard solution 0,1 mg/ml (4.12)	Content in calibration solution
ml	µg/ml	ml	µg/ml	ml	µg/ml	ml	µg/ml	ml	µg/ml
0	0	0	0	0	0	0	0	0	0
1,0	10	1,0	1,0	1,0	10	1,0	0,5	1,0	1,0
2,5	25	2,5	2,5	2,5	25	2	1	1,5	1,5
5,0	50	5,0	5,0	5,0	50	6	3	2,0	2,0
10,0	100	7,5	7,5	7,5	75	14	7	2,5	2,5
15,0	150	10,0	10,0	10,0	100	20	10	3,0	3,0

NOTE — The range of the elements which can be covered may vary from instrument to instrument. Attention should be paid to the minimum criteria given in 5.2. For instruments with high sensitivity, smaller portions of the standard solution or a more diluted standard solution can be used.

The atomic absorption spectrometer shall be preferably attached to a chart recorder and/or digital readout device and capable of measuring absorbance at the following wavelengths :

Aluminium	309,3 nm
Copper	324,8 nm
Lead	283,3 nm
Zinc	213,8 nm

6 Sampling

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

7 Procedure

7.1 Safety precaution

Follow the manufacturer's instructions for igniting and extinguishing the dinitrogen oxide-acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the flame is burning.

7.2 Test portion

Weigh approximately 2 g of the test sample.

7.3 Determination

7.3.1 Decomposition of the test portion

Transfer the test portion (7.2) to a 200 ml breaker, moisten with water, dissolve in 40 ml of the hydrochloric acid (4.6) while heating, maintaining the volume constant by addition of water. After decomposition of the test portion, add 2 ml of the nitric acid (4.2), boil the solution till nitrous oxides cease to evolve, then evaporate the solution to dryness. Add 10 ml of the hydrochloric acid (4.4) to the dry residue and evaporate to dryness again. Redissolve the salts by heating with 10 ml of the hydrochloric acid (4.4) and add 20 ml of hot water. Filter the residue on a medium-texture filter paper containing some filter pulp [previously washed with hot hydrochloric acid (4.7) and water]. Carefully remove all adhering particles with a rubber-tipped glass rod, and wash the beaker with water. Wash the filter paper with the residue with hot hydrochloric acid (4.7), then with hot water till the yellow colour of the filter paper disappears. Reserve the filtrate (main solution).

7.3.2 Treatment of the residue

Transfer the filter paper and residue to a platinum crucible (5.1). Ignite the paper and residue at a temperature less than 600 °C. Allow to cool, moisten with two or three drops of water, add 1 ml of the sulfuric acid (4.3) and 5 to 10 ml of the hydrofluoric acid (4.5). Evaporate slowly to expel silica and then fume to remove the excess of sulfuric acid. Ignite at about 600 °C. Allow to cool, add 2,0 g of the fusion mixture (4.1) to the residue and fuse in a muffle furnace or over a Mecker burner, then heat at 1 000 °C for 5 min.

Allow to cool, transfer the crucible with the melt to the original 200 ml beaker, add 10 ml of the hydrochloric acid (4.6) and dissolve the melt while heating. Remove and wash the crucible (the volume of the solution shall not exceed 50 ml).

Combine the solution obtained with the main solution (7.3.1), filter, if necessary, through a medium-texture filter paper containing some filter pulp [previously washed with hot hydrochloric acid (4.7) and water].

7.3.3 Preparation of the test solution

Transfer the solution (7.3.2) to a 200 ml one-mark volumetric flask, dilute to the mark with water and mix.

Use this solution for aluminium contents up to 1,50 % (*m*/*m*), copper contents up to 0,1 % (*m*/*m*), lead in all concentration ranges and zinc contents up to 0,025 % (*m*/*m*).

For aluminium contents over 1,50 % (*m*/*m*), zinc contents over 0,025 % (*m*/*m*) and copper content over 0,1 % (*m*/*m*), transfer the aliquot portion of this solution (see table 2) to a 100 ml one-mark volumetric flask, add the corresponding amount of background solution (4.13), dilute to the mark with water and mix.

Table 2 — Dilution guide for test solution

Element	Expected content of element in sample	Aliquot portion	Background solution (4.13) to be added	Element content in the solution measured
	% (*m*/*m*)	ml	ml	µg/ml
Al	0,1 up to 1,5	—	—	10 to 150
	1,5 up to 3,0	50	20	75 to 150
	3,0 up to 4,0	25	30	75 to 100
Zn	0,005 up to 0,025	—	—	0,5 to 2,5
	0,025 up to 0,05	50	20	1,25 to 2,5
	0,05 up to 0,1	20	32	1,0 to 2,0
	0,1 up to 0,25	10	36	1,0 to 2,5
Cu	0,005 up to 0,1	—	—	0,5 to 10
	0,1 up to 1,0	10	36	1,0 to 10

To prepare the blank solution, dissolve 1 g of high purity manganese and 0,1 g of the high purity iron in 40 ml of the hydrochloric acid (4.6) in a 200 ml breaker. Carry out the blank test through all stages of the analysis.

7.3.4 Adjustment of atomic absorption spectrometer

Optimize the response of the instrument as described in 5.2. Set the wavelength for aluminium (309,3 nm), copper (324,8 nm), lead (283,3 nm) or zinc (213,8 nm) to obtain minimum absorbance. A gas mixture of dinitrogen oxide/acetylene (with the appropriate burner) is used for the determination of copper, lead and zinc. Adjust the fuel and burner after 10 min preheating to obtain maximum absorbance while aspirating the highest calibration solution (see 4.14).

Aspirate water and the calibration solution to establish that the absorbance reading is not drifting and then set the initial reading for water to zero absorbance.

7.3.5 Spectroscopic measurements

Aspirate water until the initial reading is again obtained. Aspirate the calibration solutions and the final test solution in the order of increasing absorption, starting with the reagent blank solution, the zero calibration solution and the final test solution being aspirated at the proper points in the series. When a stable response is obtained for each solution, record the readings. Aspirate water between each calibration and test solution. Repeat the measurements at least twice more.

If necessary, convert the average of the readings for each calibration solution to absorbance. Obtain the net absorbance of each calibration solution by substracting the average absorbance of the zero calibration solution. In a similar manner, obtain the net absorbance of the test solution by subtracting the absorbance of the reagent blank solution.

Prepare calibration curves by plotting the net absorbance values of the calibration solutions against the masses of Al, Cu, Pb, Zn, in micrograms per millilitre. Convert the net absorbance values of the test solution to micrograms of Al, Cu, Pb, Zn per millilitre by means of the calibration curve.

8 Expression of results

8.1 Calculation

The aluminium (Al), copper (Cu), lead (Pb) or zinc (Zn) contents, expressed as a percentage by mass, are given by the formula

$$\frac{C \times V}{m_1 \times 10^4} \times K$$

where

C is the concentration, in micrograms per millilitre, of aluminium, copper, lead or zinc in the test solution, obtained from the calibration graph;

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

m_1 is the mass, in grams, of the test portion corresponding to the aliquot portion of the test solution;

K is the conversion factor for the expression of the aluminium, copper, lead or zinc content on the dry basis.

8.2 Permissible tolerances on results of parallel determinations

Table 3 — Permissible tolerances on copper or lead content

Copper or lead content	Permissible tolerance	
	Three parallel determinations	Two parallel determinations
% (*m*/*m*)	% (*m*/*m*)	% (*m*/*m*)
From 0,005 to 0,01	0,004	0,003
From 0,01 to 0,02	0,006	0,005
From 0,02 to 0,05	0,008	0,007
From 0,05 to 0,10	0,015	0,010
From 0,10 to 0,20	0,025	0,020
From 0,20 to 0,50	0,040	0,030
From 0,50 to 1,0	0,050	0,040

Table 4 — Permissible tolerances on zinc content

Zinc content	Permissible tolerance	
	Three parallel determinations	Two parallel determinations
% (*m*/*m*)	% (*m*/*m*)	% (*m*/*m*)
From 0,005 to 0,01	0,004	0,003
From 0,01 to 0,02	0,006	0,005
From 0,02 to 0,05	0,008	0,007
From 0,05 to 0,10	0,015	0,010
From 0,10 to 0,25	0,025	0,020

Table 5 — Permissible tolerances on aluminium content

Aluminium content	Permissible tolerance	
	Three parallel determinations	Two parallel determinations
% (*m*/*m*)	% (*m*/*m*)	% (*m*/*m*)
From 0,10 to 0,20	0,025	0,02
From 0,20 to 0,50	0,04	0,03
From 0,50 to 1,0	0,05	0,04
From 1,0 to 2,0	0,07	0,06
From 2,0 to 4,0	0,15	0,10

8.3 Oxide factor

Al_2O_3 [% (*m*/*m*)] = 1,889 5 × Al [% (*m*/*m*)]