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METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF ZINC CONTENT

(Polarographic method, for zinc content from 0.005 to 0.1% inclusive)

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BRIEF HISTORY

The ISO Recommendation R 620, *Methods of Chemical Analysis of Manganese Ores — Determination of Zinc Content*, was drawn up by Technical Committee ISO/TC 65, *Manganese Ores*, the Secretariat of which is held by Komitet Standartov Mer i Izmeritel'nyh Priborov pri Sovete Ministrov S.S.S.R. (GOST).

Work on this question by the Technical Committee began in 1954 and led, in 1964, to the adoption of a Draft ISO Recommendation.

In April 1965, this Draft ISO Recommendation (No. 779) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Argentina	India	Switzerland
Austria	Italy	Turkey
Chile	Korea, Rep. of	U.A.R.
Czechoslovakia	Netherlands	United Kingdom
Germany	Poland	U.S.S.R.
Greece	Romania	Yugoslavia
Hungary	Spain	

One Member Body opposed the approval of the Draft:

France

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in September 1967, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 620 September 1967

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF ZINC CONTENT

(Polarographic method, for zinc content from 0.005 to 0.1 % inclusive)

(Atomic mass Zn : 65.38 ; molecular mass ZnO : 81.38)

1. GENERAL INSTRUCTIONS

1.1 In the following analysis use a sample for chemical analysis of air-dried manganese ore, sampled and prepared in accordance with ISO Recommendation R 309, *Methods of Sampling Manganese Ores*.

Simultaneously with the collection of test samples for the determination of zinc, take three more test samples for the determination of hygroscopic moisture.

Calculate the content of zinc in ore on the dry basis by multiplying the numerical results of the determination of zinc by the conversion factor K, as found from the following formula:

$$K = \frac{100}{100 - A}$$

where

A is the percentage hygroscopic moisture content.

1.2 The determination of zinc in manganese ore should be carried out by analysing three parallel-weighed test samples of the ore together with two blank tests to allow the necessary corrections to be made to the results of the determination.

Simultaneously and under the same conditions carry out a check analysis of a standard sample of manganese ore for the determination of its zinc content. The standard sample for analysis should be taken from the same type of ore as that from which the analysed sample was taken.

The arithmetical mean of the three results should be accepted as the final result.

The following conditions should be observed:

The difference between the highest and the lowest results should not exceed double the absolute value of the permissible tolerance for the corresponding interval of zinc content shown in the table under clause 5.2 "Accuracy of method".

The average result of the simultaneous check analysis of the standard sample of manganese ore for zinc content should not differ from the result shown in the certificate by more than the $\pm$ value of the permissible tolerance for the corresponding interval of zinc content, shown in the table under clause 5.2 "Accuracy of method".

1.3 The test samples and the residues should be weighed to an accuracy of $\pm$ 0.0002 g.

1.4 Water which has been double distilled in quartzware or de-ionized water should be used during the procedure and for preparation of the solutions.

1.5 Meanings of particular expressions:

hot water (or solution)	implies a temperature of the liquid of 60 to 70 °C;
warm water (or solution)	implies a temperature of the liquid of 40 to 50 °C;
diluted 1 : 1, 1 : 2, 1 : 5 etc.	the first figure gives the number of parts by volume of concentrated acid or some other solution; the second figure gives the number of parts by volume of water.

1.6 Indications as to the concentration of solutions show the quantity of solute, in grammes, in the corresponding volume of the solvent.

1.7 The following abbreviations and symbols are used:

CP	chemically pure
d	relative density
g	gramme
g/l	grammes per litre
l	litre
ml	millilitre
mm	millimetre
PFA	pure for analysis

2. PRINCIPLE OF METHOD

A test sample of the ore is fused with sodium peroxide and the fused mass is extracted in water. Zinc is determined in the solution by the polarographic method using ammonium chloride ammonia background.

3. REAGENTS

3.1 *Ammonium chloride*, CP.

3.2 *Metallic zinc*, CP.

3.3 *Sodium sulphite, crystalline*, ($Na_2SO_3.7H_2O$), CP.

3.4 *Sodium peroxide*, CP.

3.5 *Ethanol*, PFA.

3.6 *Ammonia*, aqueous solution CP (*d* 0.91).

3.7 *Hydrochloric acid*, CP (*d* 1.19).

3.8 *Sulphuric acid*, CP (*d* 1.84), diluted (1 : 1).

3.9 *Agar-agar*.

3.10 *Ammonium chloride ammonia mixture (background)*.

Place in a 750 ml beaker 100 g of ammonium chloride (3.1), 150 ml of aqueous ammonia (3.6), 0.25 g of agar-agar (3.9), (dissolved by heating in 100 ml of water), 100 g of sodium sulphite (3.3) and 300 ml of water; mix until the salts are dissolved, transfer the solution obtained to a 1 litre volumetric flask, dilute with water up to the mark and mix.

3.11 *Standard solution of zinc*.

Dissolve, while heating, 0.1 g of metallic zinc (3.2) in 10 ml of sulphuric acid (3.8), cool, transfer the solution to a 1 litre volumetric flask, add 20 ml of sulphuric acid (3.8), dilute with water up to the mark and mix. 1 ml of the solution contains 0.0001 g of zinc.

3.12 *Methyl-orange*, aqueous solution (1 g/l).

4. PROCEDURE

4.1 Weigh accurately about 2 g of manganese ore into a nickel or corundum crucible, add 8 to 12 g sodium peroxide (3.4), stir thoroughly and fuse in a muffle furnace at a temperature of 600 to 650 °C for 15 to 20 minutes. After cooling the fused mass, extract it in 200 to 250 ml of water, heat to boiling, allow to boil for 10 to 15 minutes, add ethanol (3.5), drop by drop, and boil until the green colour disappears.

4.2 After cooling the solution, transfer it with the precipitate to a 500 ml volumetric flask, dilute with water up to the mark, mix and allow to stand until the supernatant liquid becomes clear. Filter the solution through a dry filter into a dry beaker. Take a 250 ml aliquot portion, transfer it to a 500 ml beaker, add 2 to 3 drops of the methyl-orange solution (3.12), neutralize with hydrochloric acid (3.7) until the colour of the indicator changes and add 5 ml of the acid in excess.

4.3 Evaporate the solution to 40 to 50 ml, cool and transfer to a 100 ml volumetric flask. Wash the sides of the beaker with the ammonium chloride ammonia mixture (3.10), dilute the solution with this mixture up to the mark and mix well. Filter a part of the solution through a dry filter into a dry beaker and determine zinc content in the filtrate by the polarographic method. Carry out the polarographic determination at 1.0 to 1.46 V.

Use for comparison a standard solution of zinc (3.11) taken in a quantity corresponding approximately to the same zinc content as in the ore being analysed. Place the exact standard solution of zinc in a nickel or corundum crucible and take it through all the stages of the analysis.

NOTE. — Glassware which contains zinc should not be used.

5. EXPRESSION OF RESULTS

5.1 Method of calculation

The percentage content of zinc (Zn) is calculated from the following formula:

$$Zn = \frac{B_x \, C_{st} \times 100}{B_{st} \, G} \, \%$$

where

B_x is the height of polarographic wave of the test solution, expressed in millimetres;

C_{st} is the quantity of the standard solution (3.11) used, expressed in grammes of zinc;

B_{st} is the height of polarographic wave of the standard solution, expressed in millimetres;

G is the mass of a test sample of the ore, expressed in grammes, corresponding to the aliquot portion of the solution taken for the polarographic determination.