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# INTERNATIONAL STANDARD 620

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

# Manganese ores — Determination of zinc content — Polarographic method (zinc content between 0,005 and 0,1 %)

*Minerais de manganèse — Dosage du zinc — Méthode polarographique (teneur en zinc comprise entre 0,005 et 0,1 %)*

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## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (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 set up 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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 65 has reviewed ISO Recommendation R 620 and found it technically suitable for transformation. International Standard ISO 620 therefore replaces ISO Recommendation R 620-1967 to which it is technically identical.

ISO Recommendation R 620 was approved by the Member Bodies of the following countries :

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

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

France

The Member Body of the following country disapproved the transformation of ISO/R 620 into an International Standard :

Bulgaria





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# Manganese ores — Determination of zinc content — Polarographic method (zinc content between 0,005 and 0,1 %)

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a polarographic method for the determination of the zinc content of manganese ores the zinc content of which is between 0,005 and 0,1 % (*m/m*).

## 2 REFERENCES

ISO 310, *Manganese ores — Determination of hygroscopic moisture in analytical samples — Gravimetric method.*

ISO . . ., *Manganese ores and concentrates — Sampling and sample preparation for chemical analysis and determination of moisture content.* [1)]

## 3 PRINCIPLE

Fusion of a test portion of the ore with sodium peroxide and extraction of the fused mass in water. Determination of the zinc in the solution by the polarographic method using ammonium chloride/ammonia background.

## 4 REAGENTS

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

**4.1 Ammonium chloride.**

**4.2 Metallic zinc.**

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

**4.4 Sodium peroxide.**

**4.5 Ethanol.**

**4.6 Ammonia** solution, $\rho$ 0,91 g/ml.

**4.7 Hydrochloric acid,** $\rho$ 1,19 g/ml.

**4.8 Sulphuric acid,** $\rho$ 1,84 g/ml, diluted 1 : 1.

**4.9 Agar-agar.**

**4.10 Ammonium chloride/ammonia mixture** (background).

Place in a 750 ml beaker 100 g of ammonium chloride (4.1), 150 ml of ammonia solution (4.6), 0,25 g of agar-agar (4.9) (dissolved by heating in 100 ml of water), 100 g of sodium sulphite (4.3) and 300 ml of water; mix until the salts are dissolved, transfer the solution obtained to a 1 l volumetric flask, dilute with water up to the mark and mix.

**4.11 Standard zinc solution.**

Dissolve, while heating, 0,1 g of metallic zinc (4.2) in 10 ml of sulphuric acid (4.8), cool, transfer the solution to a 1 l volumetric flask, add 20 ml of sulphuric acid (4.8), dilute with water up to the mark and mix.

1 ml of the solution contains 0,000 1 g of zinc.

**4.12 Methyl orange**, 1 g/l solution.

## 5 APPARATUS

Ordinary laboratory apparatus and

**5.1 Nickel or corundum crucibles.**

**5.2 Muffle furnace,** capable of being maintained at 600 to 650 °C.

**5.3 Polarograph.**

1) This document, at present at the stage of draft proposal, is intended to complete and replace ISO/R 309, *Methods of sampling manganese ores — Part I — Ore loaded in freight wagons.*

## 6 SAMPLE

Use a test sample which has been crushed to a size not exceeding 0,10 mm (checked on a sieve of appropriate size) and air-dried under laboratory conditions (see ISO . . .).

## 7 PROCEDURE

### 7.1 Number of analyses

Carry out the determination simultaneously on three test portions taken from the same test sample.

### 7.2 Blank test

In parallel with the determination and under the same conditions, carry out a blank test in duplicate to enable a corresponding correction in the result of the determination to be made.

### 7.3 Check test

In parallel with the determination and under the same conditions, carry out a check analysis of a standard sample of manganese ore of known zinc content and of the type of ore to which the sample being analysed belongs.

### 7.4 Test portion

Weigh, to the nearest 0,000 2 g, 2 g of the test sample into the nickel or corundum crucible (5.1).

### 7.5 Determination

NOTE – Glassware which contains zinc should not be used.

**7.5.1** To the test portion (7.4) in the nickel or corundum crucible, add 8 to 12 g of sodium peroxide (4.4), stir thoroughly and fuse in the muffle furnace (5.2) at a temperature of 600 to 650 °C for 15 to 20 min. After cooling the fused mass, extract it in 200 to 250 ml of water, heat to boiling, allow to boil for 10 to 15 min, add ethanol (4.5), drop by drop, and boil until the green colour disappears.

**7.5.2** 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 or 3 drops of the methyl orange solution (4.12), neutralize with hydrochloric acid (4.7) until the colour of the indicator changes and add 5 ml of the acid in excess.

**7.5.3** 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 (4.10), 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 the zinc content in the filtrate by the polarographic method. Carry out the polarographic measurement at 1,0 to 1,46 V.

Take for comparison a specified volume of the standard zinc solution (4.11) corresponding approximately to the zinc content of the sample being analysed and, parallel with the latter, take it through all the stages of the analysis including the polarographic measurement.

## 8 EXPRESSION OF RESULTS

### 8.1 Method of calculation

The zinc content of the absolutely dry ore is given, as a percentage by mass, by the formula

$$\frac{h_0 \times m_1 \times 100}{h_1 \times m_0} \times \frac{100}{100 - A}$$

where

$h_0$ is the height, in millimetres, of the polarographic wave of the solution being analysed;

$h_1$ is the height, in millimetres, of the polarographic wave of the standard zinc solution;

$m_0$ is the mass, in grams, of the test portion corresponding to the aliquot portion of the solution taken for the determination;

$m_1$ is the mass, in grams, of zinc in the volume of standard zinc solution used for the determination;

$A$ is the hygroscopic moisture content of the test sample, as a percentage by mass, determined in accordance with ISO 310.

Take as the result the arithmetic mean of the three determinations, provided that the requirement of repeatability (see 8.2) is satisfied.

### 8.2 Repeatability

The difference between the highest and the lowest results shall not exceed double the absolute value of the permissible tolerance on the result of the analysis (for the corresponding interval of zinc content) shown in the table below.

| Zinc content, % | | Permissible tolerance, % (in absolute value) |
|---|---|---|
| from (over) | to | |
| 0,005 | 0,010 | ± 0,001 0 |
| 0,010 | 0,050 | ± 0,002 0 |
| 0,050 | 0,100 | ± 0,003 0 |

The average result of the simultaneous check analysis of the standard sample of manganese ore for zinc content shall not differ from the result shown in the certificate by more than the ± value of the permissible tolerance (for the corresponding interval of zinc content) shown in the table.

## 9 TEST REPORT

The test report shall include the following information :

a) indications necessary for the identification of the sample;

b) reference to this International Standard;

c) results of the analysis;

d) the reference number of the results;

e) any characteristics noticed during the determination, and any operations not specified in this International Standard which may have had an influence on the results.