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Manganese ores — Determination of zinc content — Zinc mercurithiocyanate gravimetric method

Minerais de manganèse — Dosage du zinc — Méthode gravimétrique à l'état de thiocyanatomercurate de zinc

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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 551 and found it technically suitable for transformation. International Standard ISO 551 therefore replaces ISO Recommendation R 551-1966 to which it is technically identical.

ISO Recommendation R 551 was approved by the Member Bodies of the following countries :

Australia	Hungary	Poland
Austria	India	Romania
Chile	Iran	Spain
Czechoslovakia	Ireland	United Kingdom
Egypt, Arab Rep. of	Italy	U.S.S.R.
France	Japan	Yugoslavia

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

Germany*

The Member Bodies of the following countries disapproved the transformation of ISO/R 551 into an International Standard :

Bulgaria
Poland
United Kingdom

* Subsequently, this Member Body approved the Recommendation.

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Manganese ores — Determination of zinc content — Zinc mercurithyocyanate gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a zinc mercurithiocyanate gravimetric method for the determination of the zinc content of manganese ores.

2 REFERENCES

ISO 310, *Manganese ores — Determination of hygroscopic moisture content 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

Separation of zinc from the accompanying elements in the form of sulphide in formic medium, followed by its precipitation in the form of the complex salt zinc mercurithiocyanate, $Zn[Hg(CNS)_4]$.

4 REAGENTS

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

4.1 Potassium pyrosulphate.

4.2 Iron(II) sulphide (FeS) (for the production of hydrogen sulphide).

4.3 Nitric acid, ρ 1,40 g/ml.

4.4 Hydrochloric acid, ρ 1,19 g/ml.

4.5 Hydrochloric acid, diluted 1 : 1.

4.6 Sulphuric acid, diluted 1 : 1.

4.7 Sulphuric acid, diluted 1 : 9.

4.8 Sulphuric acid, diluted 1 : 20.

4.9 Sulphuric acid, diluted 1 : 50.

4.10 Hydrofluoric acid, 40 % (*m/m*).

4.11 Citric acid, 200 g/l solution.

4.12 Ammonia solution, ρ 0,91 g/ml.

4.13 Formic mixture.

To 200 ml of formic acid (ρ 1,22 g/ml), add 250 g of ammonium sulphate dissolved in 500 ml of water, and 30 ml of ammonia solution (4.12) and dilute with water to 1 l.

4.14 Mercury(II) chloride, 50 g/l solution.

4.15 Ammonium mercurithiocyanate, $(NH_4)_2[Hg(CNS)_4]$, solution.

Dissolve 39 g of ammonium thiocyanate in 200 ml of water. Dissolve 27 g of mercury(II) chloride in 200 ml of water heated to 50 °C. Mix the solutions obtained, dilute with water to 1 l, allow to stand for 2 days and then filter.

4.16 Washing solution.

Dilute 4 ml of formic acid with water to 1 l.

4.17 Washing solution.

Dilute 15 ml of the ammonium mercurithiocyanate solution (4.15) with water to 1 l.

4.18 Methyl orange, 1 g/l solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Platinum crucible.

5.2 Hot-plate.

5.3 Filter crucible (pore size 10 to 20 μm).

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, in order to allow 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, 5 g of the test sample into a 500 ml beaker.

7.5 Determination

7.5.1 Add 50 ml of hydrochloric acid (4.4) to the beaker containing the test portion and dissolve while heating. Add 2 to 3 ml of nitric acid (4.3) and boil for 5 min. Add 20 ml of sulphuric acid (4.6), and evaporate until sulphuric acid vapours appear. The salt having been cooled, dissolve it in 50 ml of water, then filter and wash the residue on the filter with hot water (60 to 70 °C). Place the filter with the residue in the platinum crucible (5.1), ignite and calcinate at a temperature of 500 to 600 °C. Moisten the residue with several drops of water, add 3 or 4 drops of sulphuric acid (4.6) and 5 to 6 ml of hydrofluoric acid (4.10) and evaporate until dry. Fuse the residue with 2 g of potassium pyrosulphate (4.1), extract the fusion in 40 to 50 ml of sulphuric acid (4.8), and add the solution thus obtained to the main solution.

7.5.2 Dilute the solution with water to a volume of 250 ml, heat to boiling, add 2 or 3 drops of mercury(II) chloride solution (4.14) and pass hydrogen sulphide for 35 to 40 min.

7.5.3 Digest the solution with the residue of lead, copper and other sulphides on the hot-plate (5.2) for 30 to 40 min and then filter the residue on a filter of average density.

7.5.4 Wash the residue on the filter eight to ten times with sulphuric acid (4.9) saturated with hydrogen sulphide and discard the residue.

7.5.5 Boil the filtrate until hydrogen sulphide is removed, cool, add 25 ml of citric acid solution (4.11), 2 or 3 drops of methyl orange solution (4.18) and then ammonia solution (4.12) until the colour changes to yellow. Add to the solution 25 ml of formic mixture (4.13), and 3 or 4 drops of mercury(II) chloride solution (4.14). Dilute the solution with water to a volume of 300 ml, heat to boiling and pass hydrogen sulphide for 35 to 40 min; digest the solution with the residue of zinc sulphides on the hot-plate for 30 to 40 min, then filter it on a dense filter and wash eight to ten times with washing solution (4.17) saturated with hydrogen sulphide.

7.5.6 Place the filter with the residue in a porcelain crucible, dry, ignite and calcinate at a temperature of 500 to 600 °C under a hood. Then cool the crucible with the residue, dissolve the residue in the crucible in 4 to 6 ml of sulphuric acid (4.7), while heating, transfer the solution to a 100 ml beaker, filter off the insoluble residue on a filter of average density, wash five or six times with hot water (60 to 70 °C) and discard the residue.

7.5.7 Add to the filtrate (volume of 20 to 30 ml) 5 ml of ammonium mercurithiocyanate solution (4.15), stir with a glass rod and allow to stand for 12 h.

Filter off the residue into the weighed filter crucible (5.3) and wash eight to ten times with washing solution (4.17). Dry the filter crucible with the residue at a temperature of 105 to 110 °C for 1 to 2 h, cool and weigh to the nearest 0,000 2 g.

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

$$\frac{m_1 \times 0{,}131\,2 \times 100}{m_0} \times \frac{100}{100 - A}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the dried residue of zinc mercurithiocyanate;

0,131 2 is the factor for converting zinc mercurithiocyanate to zinc;

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.