

INTERNATIONAL STANDARD



621

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Manganese ores — Determination of metallic iron content — Sulphosalicylic acid photometric method (metallic iron content not exceeding 2 %)

Minerais de manganèse — Dosage du fer métallique — Méthode photométrique à l'acide sulfosalicylique (teneur en fer métallique inférieure ou égale à 2 %)

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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 621 and found it technically suitable for transformation. International Standard ISO 621 therefore replaces ISO Recommendation R 621-1967 to which it is technically identical.

ISO Recommendation R 621 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 621 into an International Standard.

Manganese ores — Determination of metallic iron content — Sulphosalicylic acid photometric method (metallic iron content not exceeding 2 %)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method, by complexing with sulphosalicylic acid, for the determination of the metallic iron content of manganese ores the metallic iron content of which does not exceed 2 % (m/m).

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 moisture determination.*¹⁾

3 PRINCIPLE

Selective solution of the metallic iron in a methanolic solution of mercury(II) chloride and sodium salicylate. (The latter eliminates completely the formation of insoluble basic iron(III) salt and methanol prevents the solution of iron(III) oxides. The determination is unaffected by manganese oxide or other oxidants.)

Reaction, in buffer acetate solution, of the trivalent iron with sulphosalicylic acid to form a coloured complex which tints the solution yellow-brown.

Photometric measurement of the coloured complex at a wavelength of 420 to 430 nm.

4 REAGENTS

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

4.1 Sodium salicylate.

4.2 Sodium acetate, crystalline ($\text{CH}_3\text{CO}_2\text{Na} \cdot 3\text{H}_2\text{O}$).

4.3 Iron(III) oxide.

4.4 Mercury(II) chloride.

4.5 Methanol.

4.6 Sulphosalicylic acid.

4.7 Ammonia solution, ρ 0,91 g/ml.

4.8 Ammonia solution, diluted 1 : 1.

4.9 Hydrochloric acid, ρ 1,19 g/ml.

4.10 Hydrochloric acid, diluted 1 : 2.

4.11 Hydrochloric acid, diluted 1 : 4.

4.12 Ammonium sulphosalicylate solution.

Dissolve 100 g of sulphosalicylic acid (4.6) in 500 to 600 ml of water, neutralize against an indicator paper (pH approximately 5) with ammonia solution (4.8), filter, cool, dilute with water up to 1 l and mix.

4.13 Buffer solution.

Dissolve 500 g of sodium acetate (4.2) in 500 ml of hot hydrochloric acid (4.11) (60 to 70 °C), filter, cool, dilute with water up to 1 l and mix.

4.14 Solvent.

Dissolve 2,5 g of mercury(II) chloride (4.4), and 3 g of sodium salicylate (4.1) in 100 ml of methanol (4.5).

4.15 Standard iron solution.

Dissolve 0,143 0 g of iron(III) oxide (4.3), previously calcinated at a temperature of 750 to 800 °C, in 15 ml of hydrochloric acid (4.9), evaporate to 10 ml, transfer to a 1 l volumetric flask, dilute with water up to the mark and mix.

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

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.*

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Photoelectric absorptiometer fitted with a blue (wavelength 420 to 430 nm) light-filter.

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 metallic iron 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, about 1 g of the test sample into a 50 to 100 ml dry conical flask.

7.5 Determination

7.5.1 To the test portion (7.4) in the conical flask, add 20 ml of solvent (4.14) and boil for 10 min while mixing, the flask being covered with a watch-glass.

7.5.2 When the solution has cooled, transfer it to a 100 ml volumetric flask, dilute with water up to the mark and mix. Allow the precipitate to settle and filter the solution through a dense dry filter into a dry beaker, discarding the first 10 to 15 ml of the filtrate.

7.5.3 Transfer a 20 ml aliquot portion of the solution to a 100 ml volumetric flask, add 3 drops of hydrochloric acid (4.10) and mix. Add 10 ml of the ammonium sulphosalicylate solution (4.12) and 10 ml of the buffer

solution (4.13), dilute with water up to the mark, mix and carry out the photometric determination in 10 to 15 min using a blue light-filter.

7.5.4 Determine the percentage of metallic iron in the test sample, from the absorbance of the solution being tested, using either of the following methods :

a) Calibration curve method

To construct the calibration curve, take corresponding volumes of the standard iron solution (4.15), covering both the limits (maximum and minimum) and the intermediate content of metallic iron in the given type of ore, and take them through all the stages of the analysis, including the determination of the absorbance, parallel with the sample being analysed.

b) Comparison method

Take a specified volume of the standard iron solution (4.15) corresponding approximately to the metallic iron content of the sample being analysed and, parallel with the latter, take it through all the stages of the analysis including the determination of the absorbance.

8 EXPRESSION OF RESULTS

8.1 Method of calculation

The metallic iron content of the absolutely dry ore is calculated, as a percentage by mass, either

- as the value read directly from the calibration curve and multiplied by the factor $100/(100 - A)$, or
- by the comparison method, using the formula

$$\frac{D_x \times m_1 \times 100}{D_{st} \times m_0} \times \frac{100}{100 - A}$$

where

D_x is the absorbance of the solution being analysed;

D_{st} is the absorbance of the standard iron 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 iron in the volume of standard iron 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 metallic iron content) shown in the table below.

Metallic iron content, %		Permissible tolerance, % (in absolute value)
from (over)	to	
	0,2	$\pm 0,02$
0,2	0,5	$\pm 0,03$
0,5	1,0	$\pm 0,04$
1,0	2,0	$\pm 0,05$

The average result of the simultaneous check analysis of the standard sample of manganese ore for metallic iron content shall not differ from the result shown in the certificate by

more than the $\pm$ value of the permissible tolerance (for the corresponding interval of metallic iron content) shown in the table.

9 TEST REPORT

The test report shall include the following information :

- indications necessary for the identification of the sample;
- reference to this International Standard;
- results of the analysis;
- the reference number of the results;
- any characteristics noticed during the determination, and any operations not specified in this International Standard which may have had an influence on the results.

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