

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 621

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES
DETERMINATION OF METALLIC IRON CONTENT

(Photometric method for metallic iron content up to and including 2%)

1st EDITION
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BRIEF HISTORY

The ISO Recommendation R 621, *Methods of Chemical Analysis of Manganese Ores — Determination of Metallic Iron 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. 780) 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	Hungary	Spain
Austria	India	Switzerland
Chile	Italy	Turkey
Czechoslovakia	Korea, Rep., of	U.A.R.
France	Netherlands	United Kingdom
Germany	Poland	U.S.S.R.
Greece	Romania	Yugoslavia

No Member Body opposed the approval of the Draft.

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.

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF METALLIC IRON CONTENT

(Photometric method for metallic iron content up to and including 2%)

(Atomic mass Fe : 55.85)

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 metallic iron, take three more test samples for the determination of hygroscopic moisture.

Calculate the content of metallic iron in ore on the dry basis, by multiplying the numerical results of the determination of metallic iron 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 metallic iron 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.

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

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 metallic iron content, shown in the Table under clause 5.2 "Accuracy of method".

1.3 If an average sample of manganese ore contains metallic iron, the necessary corrections should be made to the result of the analysis.

1.4 The test samples should be weighed to an accuracy of ± 0.0002 g.

1.5 Distilled water should be used during the procedure and for preparation of the solutions.

1.6 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.

1.7 The following abbreviations and symbols are used:

CP	= chemically pure
<i>d</i>	= relative density
g	= gramme
l	= litre
ml	= millilitre
nm	= nanometre
PFA	= pure for analysis

2. PRINCIPLE OF METHOD

The method is based on the selective solution of the metallic iron in methyl alcohol solution of mercuric chloride and sodium salicylate. The latter eliminates completely the formation of insoluble basic ferric salt and methanol prevents the solution of ferric oxides.

The metallic iron in the solution is determined by the photometric method in the presence of an acetate buffer against the yellow-brown colour of its complex with sulphosalicylic acid, the determination being unaffected by manganese dioxide or other oxidants.

3. REAGENTS

- 3.1 *Sodium salicylate*, PFA.
- 3.2 *Crystalline sodium acetate* ($\text{CH}_3\text{CO}_2\text{Na} \cdot 3\text{H}_2\text{O}$), CP.
- 3.3 *Ferric oxide*, CP.
- 3.4 *Mercuric chloride*, PFA.
- 3.5 *Methanol*, PFA.
- 3.6 *Sulphosalicylic acid*, PFA.
- 3.7 *Ammonia, aqueous solution*, PFA (d 0.91).
- 3.8 *Ammonia, aqueous solution*, PFA (d 0.91), diluted 1: 1.
- 3.9 *Hydrochloric acid*, PFA (d 1.19).
- 3.10 *Hydrochloric acid*, PFA, (d 1.19), diluted 1: 2.
- 3.11 *Hydrochloric acid*, PFA, (d 1.19), diluted 1: 4.
- 3.12 *Ammonium sulphosalicylate solution*. Dissolve 100 g of sulphosalicylic acid (3.6) in 500 to 600 ml of water, neutralize against an indicator paper (pH approximately 5) with aqueous ammonia (3.8), filter, cool, dilute with water up to 1 litre and mix.
- 3.13 *Buffer solution*. Dissolve 500 g of crystalline sodium acetate (3.2) in 500 ml of hot hydrochloric acid (3.11), filter, cool and dilute with water up to 1 litre.
- 3.14 *Solvent*. Dissolve 2.5 g of mercuric chloride (3.4), and 3 g of sodium salicylate (3.1) in 100 ml of methanol (3.5).
- 3.15 *Standard iron solution*. Dissolve 0.1430 g of ferric oxide (3.3), previously calcinated at a temperature of 750 to 800 °C, in 15 ml of hydrochloric acid (3.9), evaporate to 10 ml, transfer to a 1 litre volumetric flask, dilute with water up to the mark and mix. 1 ml of the solution contains 0.0001 g of iron.

4. PROCEDURE

- 4.1 Weigh accurately about 1 g of manganese ore into a 50 to 100 ml dry conical flask, add 20 ml of solvent (3.14) and boil for 10 minutes while mixing, the flask being covered with a watch glass.
- 4.2 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.
- 4.3 Transfer a 20 ml aliquot portion of the solution to a 100 ml volumetric flask, add 3 drops of hydrochloric acid (3.10) and mix. Add 10 ml of the ammonium sulphosalicylate solution (3.12) and 10 ml of the buffer solution (3.13), dilute with water up to the mark, mix and carry out the photometric determination in 10 to 15 minutes using a blue light filter (wave-length, 420 to 430 nm).
- 4.4 The percentage content of metallic iron is obtained from the optical density found for the solution being tested by the calibration curve method or by the comparative method.

(a) Calibration curve method

To construct the calibration curve, take corresponding volumes of the standard solution of iron (3.15) covering both the limits (maximum and minimum) and the intermediate degrees of its concentration in the given type of ore and take them through all the stages of the analysis, including the determination of the optical density, parallel with the sample being analysed.

The calibration curve is constructed on the basis of the values of the optical density of the standard solutions and the content of metallic iron in the sample being analysed is determined from the calibration curve on the basis of the optical density of the solution of the sample.

(b) Comparative method

Take a specified volume of the standard solution of iron (3.15) corresponding to the same iron content as in the sample being analysed and, parallel with the latter, take it through all the stages of the analysis, including the determination of the optical density.