

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 315

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES
DETERMINATION OF NICKEL

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 315, *Methods of Chemical Analysis of Manganese Ores—Determination of Nickel*, was drawn up by Technical Committee ISO/TC 65, *Manganese Ores*, the Secretariat of which is held by the Komitet Standartov, Mer i Izmeritel'nyh Priborov pri Sovete Ministrov SSSR.

Work on this question by the Technical Committee began in 1954 and led, in 1957, to the adoption of a Draft ISO Recommendation.

In October 1958, this Draft ISO Recommendation (No. 248) was circulated to all the ISO Member Bodies for enquiry. It was approved by the following Member Bodies:

Austria	Hungary	Republic of
Bulgaria	Ireland	South Africa
Burma	Italy	Romania
Chile	Japan	Spain
Czechoslovakia	Netherlands	United Kingdom
France	Poland	U.S.S.R.
Germany	Portugal	

One Member Body opposed the approval of the Draft: India.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in July 1963, to accept it as an ISO RECOMMENDATION.

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF NICKEL

(Atomic mass Ni: 58.69; molecular mass NiO: 74.69)

This ISO Recommendation contains three parts:

- | | |
|---|------------------|
| I. Introduction | section 1, |
| II. Gravimetric method, for nickel content exceeding 0.1 per cent | sections 2 to 5, |
| III. Colorimetric method, for nickel content below 0.1 per cent | sections 6 to 9. |

I. INTRODUCTION

1. GENERAL INSTRUCTIONS

- 1.1 In the following analysis, use a sample for chemical analysis of air-dried manganese ore, which has been crushed to a size not exceeding 0.10 mm and checked on a sieve of appropriate size.

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

Calculate the content of nickel in ore which is absolutely dry by multiplying the numerical results of the determination of nickel by the conversion factor K , as found from the following formula:

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

where

A = hygroscopic moisture content, per cent.

- 1.2 The determination of nickel in manganese ore is carried out by simultaneously analysing three samples of ore, with two blank determinations to enable a corresponding correction in the result of the determination to be made.

Simultaneously and under the same conditions, carry out a check analysis of a standard sample of manganese ore, for nickel content.

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

The following conditions should be observed:

The maximum difference between the highest and the lowest results should not exceed double the absolute value of the permissible tolerance on the result of the analysis (for the corresponding interval of nickel content), shown in the tables under clauses 5.2 and 9.2, "Accuracy of method".

The average result of the simultaneous check analysis of the standard sample of manganese ore for nickel 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 nickel content), shown in the tables under clauses 5.2 and 9.2, "Accuracy of method".

For the analysis, take a standard sample of the type of ore to which the sample being analysed belongs.

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

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

1.5 Meanings of the following 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. means that

the first figure gives the number of parts by volume of concentrated acid or some other solution, and

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 symbols and abbreviations are used:

<i>d</i>	relative density
g	gramme
g/l	grammes per litre
ml	millilitre
mm	millimetre
nm	nanometre
PFA	pure for analysis

II. GRAVIMETRIC METHOD

FOR NICKEL CONTENT EXCEEDING 0.1 PER CENT

2. PRINCIPLE OF METHOD

The method is based on the precipitation of nickel with dimethyl-glyoxime. Iron, manganese and other elements are converted beforehand into tartrate or citrate complexes.

The precipitate of nickel glyoxime is dried and weighed.

3. REAGENTS REQUIRED

- 3.1 *Ammonium hydroxide*, PFA (*d* 0.91).
- 3.2 *Dimethyl-glyoxime*, PFA, alcohol solution (10 g/l).
- 3.3 *Hydrochloric acid*, PFA (*d* 1.19).
- 3.4 *Hydrochloric acid*, PFA, diluted 1 : 1.
- 3.5 *Hydrochloric acid*, PFA, diluted 1 : 5.
- 3.6 *Hydrochloric acid*, PFA, diluted 1 : 20.
- 3.7 *Tartaric (citric) acid*, PFA, solution (250 g/l).
- 3.8 *Hydrofluoric acid*, PFA (40 per cent).
- 3.9 *Sulphuric acid*, PFA (*d* 1.84).
- 3.10 *Sodium carbonate*, PFA, anhydrous.

4. PROCEDURE

- 4.1 Weigh 2 g of manganese ore into a 300 ml beaker and dissolve while heating in 25 ml of hydrochloric acid (*d* 1.19). Evaporate the solution until dry, add 10 to 15 ml of hydrochloric acid (*d* 1.19) to the dry residue, and evaporate again to dryness.
- 4.2 Add 10 to 15 ml of hydrochloric acid (*d* 1.19) to the dry residue, dilute with hot water to 30 to 40 ml, boil until salts are dissolved and then filter off the insoluble residue.
- 4.3 Wash the filter and residue 5 or 6 times with hot hydrochloric acid, diluted 1 : 20, and 2 or 3 times with hot water. Place in a platinum crucible, and ignite at a temperature of 500 to 600 °C.
- 4.4 Moisten the residue in the crucible with water, add 1 or 2 drops of sulphuric acid (*d* 1.84) and 5 to 10 ml of hydrofluoric acid (40 per cent), and evaporate until dry. Heat the residue to a temperature of 500 to 600 °C, cool, add 1 to 2 g of anhydrous sodium carbonate, and fuse the mixture at a temperature of 900 to 1000 °C. Extract the fusion with hydrochloric acid, diluted 1 : 5.

- 4.5 Remove the crucible from the beaker, rinsing it beforehand with water, and add the resultant solution to the original filtrate.

Add to the combined solutions 25 ml of the solution of citric or tartaric acid (250 g/l), then add ammonium hydroxide until a slight odour is detected. Dilute with water to 150 to 200 ml, add 10 ml of dimethyl-glyoxime solution, stir carefully and leave standing for from 1 to 2 hours at room temperature.

- 4.6 Filter off the precipitate of nickel glyoxime on a medium filter, wash it with warm water, and then dissolve it in hot hydrochloric acid, diluted 1 : 1, collecting the solution in the beaker in which the precipitation was effected. To dissolve the residue, pour in three portions of 10 ml each of hot hydrochloric acid, diluted 1 : 1, and then wash the filter 3 or 4 times with hot water.
- 4.7 Add 3 ml of the solution of citric acid or tartaric acid (250 g/l) and ammonium hydroxide until a slight odour is detected, adding an excess of 1 ml of ammonia. Pour in 10 ml of the alcohol solution of dimethyl-glyoxime (10 g/l), stir carefully, allow the precipitate to settle for 20 to 30 min, filter off into a weighed sintered glass crucible, and wash 6 to 8 times with warm water. Dry the sintered glass crucible with the precipitate at a temperature of 110 to 120 °C to constant mass.

5. EXPRESSION OF RESULTS

5.1 Method of calculation

The percentage content of nickel is calculated from the following formula:

$$\text{Ni} = \frac{A \times 0.2032 \times 100}{G} \text{ per cent}$$

where

A = mass of dried residue of nickel glyoxime, in grammes;

G = mass of sample of ore, in grammes;

0.2032 = conversion factor for nickel glyoxime to nickel.

5.2 Accuracy of method

The permissible tolerances, per cent (absolute value), are given in the table below:

Nickel content		Permissible tolerance (in absolute value)
from	to	
0.10 per cent	0.30 per cent	± 0.010 per cent
(over) 0.30 per cent	0.50 per cent	± 0.020 per cent