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**INTERNATIONAL STANDARD**



**316**

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

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**Manganese ores — Determination of cobalt content —  
Nitroso-R-salt photometric method**

*Minerais de manganèse — Dosage du cobalt — Méthode photométrique au sel nitroso-R*

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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 316 and found it technically suitable for transformation. International Standard ISO 316 therefore replaces ISO Recommendation R 316-1963 to which it is technically identical.

ISO Recommendation R 316 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 316 into an International Standard :

Hungary

# Manganese ores — Determination of cobalt content — Nitroso-R-salt photometric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method, by complexing with nitroso-R-salt, for the determination of the cobalt 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.*<sup>1)</sup>

## 3 PRINCIPLE

Reaction, in acetate solution, of the trivalent cobalt with nitroso-R-salt to form a coloured complex which tints the solution red. Elimination of the influence of interfering elements (iron, copper, nickel) by boiling the solution with nitric acid after addition of the nitroso-R-salt. Photometric measurement at a wavelength of 420 to 430 nm or 520 to 530 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 carbonate**, anhydrous.

**4.2 Ammonium hydroxide**,  $\rho$  0,91 g/ml.

**4.3 Nitric acid**, diluted 1 : 1.

**4.4 Hydrochloric acid**, diluted 1 : 1.

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

**4.6 Sulphuric acid**,  $\rho$  1,84 g/ml.

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

**4.8 Sodium acetate**, 300 g/l solution.

**4.9 Nitroso-R-salt**, 1 g/l solution.

**4.10 Standard cobalt solution.**

Dissolve 0,100 0 g of metallic cobalt in 20 ml of hydrochloric acid, diluted 1 : 4, in the presence of a few drops of nitric acid ( $\rho$  1,40 g/ml). Boil the solution until it ceases to evolve nitric oxides.

Place the solution thus obtained in a 1 l volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 0,000 1 g of cobalt.

## 5 APPARATUS

Ordinary laboratory apparatus and

**5.1 Platinum crucible.**

**5.2 Photoelectric absorptiometer** fitted with either a blue (wavelength 420 to 430 nm) or a green (wavelength 520 to 530 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 . . .).

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.*

## 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.

### 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 cobalt 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, 0,5 g of the test sample into a 50 ml conical flask.

### 7.5 Determination

**7.5.1** Add to the conical flask containing the test portion (7.4) 8 to 10 ml of hydrochloric acid (4.5) and heat to dissolve the test portion. Add 1 ml of nitric acid (4.3) and boil for 5 min. Add to the solution an equal volume of hot water (60 to 70 °C), and filter into a conical flask with a broad neck. Wash the filter and the insoluble residue 5 to 6 times with hot water (60 to 70 °C), place in the platinum crucible (5.1), and ignite at a temperature of 500 to 600 °C. Cool, and add to the residue in the crucible 1 to 2 drops of water, 1 to 2 drops of sulphuric acid (4.6) and 2 to 3 ml of hydrofluoric acid (4.7), and evaporate until dry.

Calcine the residue slightly, cool, add 1 to 2 g of anhydrous sodium carbonate (4.1) and fuse at a temperature of 900 to 1 000 °C. Extract the fusion in hydrochloric acid (4.4) and add it to the original filtrate. Evaporate the combined filtrate to a volume of 20 to 25 ml and, if necessary, filter as before and evaporate. Then add ammonium hydroxide (4.2) until turbidity develops; dissolve the turbidity by adding 1 to 2 drops of hydrochloric acid (4.4). Add to the solution 5 ml of sodium acetate solution (4.8) and 4 ml of nitroso-R-salt solution (4.9), and boil for 5 min.

Cool the solution, add 2 to 3 ml of nitric acid (4.3) and boil again for 1 min. Cool the solution, transfer it to a 50 ml volumetric flask, dilute with water to the mark, stir, and carry out the photometric measurement, using the blue or green light-filter.

**7.5.2** Determine the percentage of cobalt 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 cobalt solution, covering both the extreme (maximum and minimum) and the intermediate contents of cobalt in the given type of ore,

and take them through all the stages of the analysis, including the determination of the absorbance, simultaneously with the sample being analysed.

#### b) Comparison method

Take a known volume of the standard cobalt solution of a concentration approaching that of the sample being analysed, and pass it through all the stages of the analysis simultaneously with the sample.

## 8 EXPRESSION OF RESULTS

### 8.1 Method of calculation

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

a) as the value read directly from the calibration curve and multiplied by the factor  $\frac{100}{100 - A}$  or

b) 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 cobalt solution;

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

$m_1$  is the mass, in grams, of cobalt in the volume of standard cobalt 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 maximum 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 cobalt content) shown in the table below.

Cobalt content, %		Permissible tolerance, % (in absolute value)
from (over)	to	
	0,01	± 0,001
0,01	0,03	± 0,001 5
0,03	0,05	± 0,002 0
0,05	0,10	± 0,003

The average result of the simultaneous check analysis of the standard sample of manganese ore for cobalt 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 cobalt 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.