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METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF COBALT

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BRIEF HISTORY

The ISO Recommendation R 316, *Methods of Chemical Analysis of Manganese Ores—Determination of Cobalt*, 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. 249) was circulated to all the ISO Member Bodies for enquiry. It was approved by the following Member Bodies:

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

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 July 1963, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 316 July 1963

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF COBALT

(Atomic mass Co: 58.94; molecular mass CoO: 74.94)

This ISO Recommendation contains two parts:

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 cobalt, take three more test samples for the determination of hydroscopic moisture.

Calculate the content of cobalt in ore which is absolutely dry by multiplying the numerical results of the determination of cobalt 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 cobalt 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 cobalt 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 cobalt content), shown in the table under clause 5.2, " Accuracy of method ".

The average result of the simultaneous check analysis of the standard sample of manganese ore for cobalt content should not differ from the result shown in the certificate by more than the ± value of the permissible tolerance (for the corresponding interval of cobalt content), shown in the table under clause 5.2, " 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:

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

II. COLORIMETRIC METHOD OF DETERMINATION OF COBALT IN THE FORM OF A COMPLEX WITH NITROSO-R-SALT

2. PRINCIPLE OF METHOD

In an acetate solution, the trivalent cobalt reacts with nitroso-R-salt to form a coloured complex which tints the solution red. The influence of the interfering elements (iron, copper, nickel) is eliminated by boiling the solution with nitric acid after addition of nitroso-R-salt. The solution is then subjected to a colorimetric analysis.

3. REAGENTS REQUIRED

3.1 *Ammonium hydroxide*, PFA (*d* 0.91).

3.2 *Nitric acid*, PFA, diluted 1 : 1.

3.3 *Hydrochloric acid*, PFA, diluted 1 : 1.

3.4 *Hydrochloric acid* (*d* 1.19).

3.5 *Sulphuric acid* (*d* 1.84).

3.6 *Hydrofluoric acid* (40 per cent).

3.7 *Sodium acetate*, PFA, solution (300 g/l).

3.8 *Sodium carbonate anhydrous*, PFA.

3.9 *Nitroso-R-salt*, PFA, solution (1 g/l).

3.10 *Standard solution of cobalt.* Dissolve 0.1000 g of metallic cobalt in 20 ml of hydrochloric acid, PFA, diluted 1 : 4, in the presence of a few drops of nitric acid, PFA (*d* 1.40). Boil the solution until it ceases to evolve nitric oxides.

Place the solution thus obtained in a 1 litre measuring flask, add water until it reaches the mark and mix.

1 ml of this solution contains 0.0001 g of cobalt.

4. PROCEDURE

4.1 Weigh 0.5 g of manganese ore into a 50 ml conical flask and dissolve it, while heating, in 8 to 10 ml of hydrochloric acid (*d* 1.19). Add 1 ml of nitric acid, diluted 1 : 1, and boil for 5 min. Add to the solution an equal volume of hot water, and filter into a conical flask with a broad neck. Wash the filter and the insoluble residue 5 to 6 times with hot water, place in a platinum crucible, 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 (*d* 1.84) and 2 to 3 ml of hydrofluoric acid (40 per cent), and evaporate until dry.

4.2 Calcine the residue slightly, cool, add 1 to 2 g of anhydrous sodium carbonate and fuse at a temperature of 900 to 1000 °C. Extract the fusion in hydrochloric acid, diluted 1 : 1, 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 (*d* 0.91) until turbidity develops; dissolve the turbidity by adding 1 to 2 drops of hydrochloric acid, diluted 1 : 1. Add to the solution 5 ml of sodium acetate solution (300 g/l) and 4 ml of nitroso-R-salt solution (1 g/l), and boil for 5 min.