UDC 669.74

Ref. No.: ISO / R 553 - 1966 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 553

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES
DETERMINATION OF VANADIUM CONTENT

1st EDITION
December 1966

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Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

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BRIEF HISTORY

The ISO Recommendation R 553, *Methods of Chemical Analysis of Manganese Ores—Determination of Vanadium Content*, 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 (GOST).

Work on this question by the Technical Committee began in 1957 and led, in 1959, to the adoption of a Draft ISO Recommendation.

In November 1962, this Draft ISO Recommendation (No. 542) 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:

Australia
Austria
Burma
Chile
Czechoslovakia
France
Germany
Hungary
India
Iran
Ireland
Italy
Japan
Poland
Romania
Spain
U.A.R.
United Kingdom
U.S.S.R.
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 December 1966, to accept it as an ISO RECOMMENDATION.

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ISO Recommendation R 553 December 1966

METHODS OF CHEMICAL ANALYSIS OF MANGANESE ORES

DETERMINATION OF VANADIUM CONTENT

(Atomic mass V : 50.95; molecular mass V_2O_5: 181.90)

This ISO Recommendation contains three parts:

I. INTRODUCTION

1. GENERAL INSTRUCTIONS

1.1 In the following analyses, 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 the samples for the determination of vanadium, take three more test samples for the determination of hygroscopic moisture.

Calculate the content of vanadium in ore which is absolutely dry by multiplying the numerical results of the determination of vanadium 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 vanadium 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 vanadium content.

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

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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 vanadium content), shown in the tables under clauses 6.2 and 10.2, " Accuracy of method ".

The average result of the simultaneous check analysis of the standard sample of manganese ore for vanadium 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 vanadium content), shown in the tables under clauses 6.2 and 10.2, " 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 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:

CP	chemically pure
d	relative density
g	gramme
g/l	grammes per litre
l	litre
ml	millilitre
N	standard solution of normal concentration
nm	nanometer

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II. VOLUMETRIC METHOD OF DETERMINATION OF VANADIUM CONTENT

(Vanadium content exceeds 0.05%)

2. PRINCIPLE OF METHOD

Oxidize tetravalent vanadium in sulphate solution by potassium permanganate to the pentavalent form. Titrate pentavalent vanadium with double ammonium ferrous sulphate solution in the presence of the indicator, phenylantranilic acid.

3. REAGENTS REQUIRED

3.1 *Urea*, CP.

3.2 *Sodium carbonate*, anhydrous, CP.

3.3 *Hydrochloric acid*, CP (*d* 1.19).

3.4 *Sulphuric acid*, CP (*d* 1.84).

3.5 *Sulphuric acid*, CP, diluted (1 : 1).

3.6 *Sulphuric acid*, CP, diluted (1 : 20).

3.7 *Phosphoric acid*, CP (*d* 1.70).

3.8 *Hydrofluoric acid*, CP (40%).

3.9 *Phenylantranilic acid*, CP, solution (2 g/l):

0.2 g of the acid is dissolved while boiling in 100 ml of water, containing 0.2 g of sodium carbonate.

3.10 *Potassium permanganate*, CP, solution (10 g/l).

3.11 *Sodium nitrite* ($NaNO_2$) CP, solution (50 g/l).

3.12 *Sodium dyphenylamine sulphonate*, CP, solution (0.8 g/l).

3.13 *Standard solution 0.01 N of ammonium ferrous sulphate*

$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$: 4 g of ammonium ferrous sulphate, CP, are dissolved in one litre of sulphuric acid, diluted (1 : 20).

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4. DETERMINING THE TITRE OF DOUBLE AMMONIUM FERROUS SULPHATE SOLUTION AGAINST THE STANDARD SAMPLE OF ORE

4.1 To determine the titre of ammonium ferrous sulphate solution, take three weighed quantities of standard sample of manganese ore, the vanadium content of which is close to that of the analysed sample, and pass them through all the stages of the analysis.

4.2 The titre of ammonium ferrous sulphate solution expressed in grammes of vanadium (T) is calculated from the following formula:

$$T = \frac{AG}{V \times 100}$$

where A = vanadium content in the standard sample of ore, in per cent;

G = mass of the standard sample of ore, in grammes;

V = volume of ammonium ferrous sulphate solution, used for titration of vanadium, expressed in millilitres.

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5. PROCEDURE

5.1 Place 1 to 2 g of manganese ore into a 300 ml beaker and dissolve in 30 ml of hydrochloric acid (d 1.19). After dissolving add 25 ml of sulphuric acid, diluted (1 : 1), evaporate until sulphuric acid vapour appears, cool, wash the walls of the flask with water and evaporate again until the sulphuric acid vapours appear. Dissolve the salts in 50 ml of water, filter off the insoluble residue, wash six to eight times with sulphuric acid, diluted (1 : 20), place the filter with the residue into a platinum crucible, ignite at a temperature of 500 to 600 °C. Moisten the residue in the crucible with water, add one or two drops of sulphuric acid (d 1.84), 5 to 7 ml of hydrofluoric acid (40 %) and evaporate until dry. Ignite the residue at a temperature of 500 to 600 °C, cool, add 2 to 3 g of sodium carbonate (anhydrous), cover the crucible with a lid and fuse at a temperature of 800 to 900 °C. Extract the fusion in 40 to 50 ml of sulphuric acid, diluted (1 : 20), wash the crucible and the lid with water and remove them; add the solution thus obtained to the main one.

5.2 Add 10 ml of phosphoric acid (d 1.70) and several drops of potassium permanganate (10 g/l) to the cool combined solution until pink colouring appears which does not disappear for 1 to 2 min. Reduce the excess of potassium permanganate by adding drop by drop potassium nitrite solution (50 g/l); introduce 2 g of urea, stir, allow to stand for 1 min and add 80 ml of sulphuric acid, diluted (1 : 1). Cool the solution, add six drops of phenylantranilic acid solution (2 g/l) or 2 ml of sodium dyphenylamine sulphonate solution (0.8 g/l), stir for 0.5 to 1 min, and titrate with 0.01 N ammonium ferrous sulphate solution until crimson colouring of the solution changes into green.

6. EXPRESSION OF RESULTS

6.1 Method of calculation

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

$$V = \frac{VT \times 100}{G} \text{ per cent}$$

where V = volume, in millilitres, of ammonium ferrous sulphate solution used in titration;

T = titre of ammonium ferrous sulphate solution, expressed in grammes of vanadium;

G = mass of test sample of ore, in grammes.

6.2 Accuracy of method

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

Vanadium content		Permissible tolerance (absolute value)
from	to	
0.05%	0.1%	±0.010%
(over) 0.1%	0.3%	±0.015%
„ 0.3%	0.5%	±0.020%

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