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Hardmetals — Determination of cobalt — Potentiometric method

Métaux durs — Détermination du cobalt — Méthode potentiométrique

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FOREWORD

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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.

International Standard ISO 3909 was drawn up by Technical Committee ISO/TC 119, *Powder metallurgical materials and products*, and was circulated to the Member Bodies in July 1975.

It has been approved by the Member Bodies of the following countries :

Austria	Morocco	Turkey
Czechoslovakia	Poland	United Kingdom
France	Romania	U.S.A.
Germany	South Africa, Rep. of	U.S.S.R.
Ireland	Spain	
Italy	Sweden	

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Australia
Canada

Hardmetals – Determination of cobalt – Potentiometric method

1 SCOPE

This International Standard specifies a potentiometric method for the determination of the cobalt content of hardmetals.

2 FIELD OF APPLICATION

This method is applicable to

- carbides and binder metal powder mixtures, free of lubricant,
- all grades of presintered or sintered hardmetals,

having a cobalt content exceeding 1 % (*m/m*).

3 PRINCIPLE

Oxidation of cobalt to the trivalent state in a strongly ammoniacal solution with an excess of potassium hexacyanoferrate(III). Potentiometric back-titration of the excess with cobalt sulphate solution.

4 INTERFERING ELEMENTS

The effect of interfering elements, for example vanadium and manganese, shall be taken into account. Vanadium and manganese are also oxidized by hexacyanoferrate(III). Vanadium reacts stoichiometrically and a correction can be applied if the vanadium content is below 0,5 % (*m/m*).

Manganese reacts almost stoichiometrically up to 0,2 % (*m/m*) only if the sum of tantalum and niobium in the hardmetal is less than 0,02 % (*m/m*). Above this level the method is applicable only if the manganese content is less than 0,03 % (*m/m*).

5 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Ammonium sulphate.

5.2 Hydrochloric acid, ρ 1,19 g/ml.

5.3 Hydrofluoric acid, ρ 1,12 g/ml.

5.4 Nitric acid, ρ 1,42 g/ml.

5.5 Sulphuric acid, ρ 1,84 g/ml.

5.6 Ammonia solution, ρ 0,91 g/ml.

5.7 Ammonium citrate solution.

Dissolve 100 g of citric acid in 900 ml of water and add 100 ml of the ammonia solution (5.6).

5.8 Cobalt sulphate, standard reference solution containing 3 g of cobalt per litre.

Prepare the solution from compact metallic cobalt, or from diammonium cobalt sulphate, or from cobalt sulphate. If necessary, establish the titre gravimetrically with 1-nitroso-naphthol-2, or electrolytically.

5.9 Potassium hexacyanoferrate(III), standard volumetric solution.

1 l should correspond to approximately 3 g of cobalt.

Dissolve 17 g of $K_3[Fe(CN)_6]$ in 1 l of water.

Determine the relationship between the volumes of the cobalt sulphate solution (5.8) and the potassium hexacyanoferrate(III) solution (5.9) in the following way.

5.9.1 Transfer into a 400 ml beaker 5 g of the ammonium sulphate (5.1), 40 ml of the ammonium citrate solution (5.7), 50 to 100 ml of water, 80 ml of the cooled ammonia solution (5.6), and from a burette approximately 9 ml of the potassium hexacyanoferrate(III) solution (5.9). Volume used : V_3 ml.

Immediately titrate with the cobalt sulphate solution (5.8). Volume used : V_4 ml.

5.9.2 Calculate the relationship according to the formula

$$K = \frac{V_4}{V_3}$$

5.10 Methyl red indicator, 1 g/l solution, or indicator paper for pH 3 to 5.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Potentiometric titration apparatus with stirrer or some other arrangement for agitating.

6.2 Platinum electrode.

6.3 Tungsten wire or any other reference electrode.

6.4 Burettes, of capacity 10 ml.

7 SAMPLING

7.1 The sample shall be crushed to a powder in a mortar made of a material which does not alter the sample composition. The powder shall pass a 0,18 mm sieve.

7.2 The analysis shall be carried out on two or three test portions.

8 PROCEDURE

8.1 Test portion

Weigh, to the nearest 0,000 1 g, 0,1 to 0,5 g of the test sample, depending on the cobalt content.

8.2 Attack

Transfer the test portion into a 400 ml beaker, add 10 to 15 ml of the sulphuric acid (5.5), 3 ml of the hydrochloric acid (5.2) and 5 g of the ammonium sulphate (5.1). Cover the beaker with a watch-glass and heat until complete dissolution is achieved. Cool.

Add, in small portions, 40 to 50 ml of the ammonium citrate solution (5.7). Rinse the watch-glass and the beaker walls with 20 to 50 ml of water. Heat the solution gently until all salts are dissolved. Cool.

The following alternative method of attack may be used. Transfer the test portion into a 400 ml polypropylene beaker or into a platinum dish, add 10 ml of water and 5 ml of the hydrofluoric acid (5.3) and cover the beaker with a polypropylene cover. Warm to approximately 60 °C. Add the nitric acid (5.4), drop by drop, until dissolution is complete. Cool. Add, in small portions, 10 ml of the sulphuric acid (5.5) and 40 to 50 ml of the ammonium citrate solution (5.7). (If vanadium is present, heat the solution and cool again.) Rinse the cover and the beaker walls with 20 to 50 ml of water.

8.3 Neutralization

Add 1 drop of the methyl red indicator solution or use the indicator paper (5.10). Carefully neutralize most of the acid with the ammonia solution (5.6) to a weakly acid solution (pH 3 to 5). Avoid over-neutralizing. Cool.

8.4 Titration

Add 80 ml of the ammonia solution (5.6) preferably cooled to 10 °C. Immediately add the potassium hexacyanoferrate(III) solution (5.9) from a burette to an excess of a few millilitres. Volume used : V_1 ml.

Back-titrate with the cobalt sulphate solution (5.8). Volume used : V_2 ml.

9 EXPRESSION OF RESULTS

9.1 Calculation

The cobalt content, expressed as a percentage by mass, is given by the formula

$$\frac{(K \times V_1 - V_2) \times T \times 100}{m}$$

where

K is as defined in 5.9.2;

T is the mass, in grams, of cobalt in 1 ml of the cobalt sulphate solution;

m is the mass, in grams, of the test portion.

The result shall be rounded to the nearest 0,01 %.

9.2 Corrections

As a tentative guide, the following correction values may be used.

Vanadium : 0,100 % (m/m) vanadium is equal to 0,116 % (m/m) cobalt.

Manganese : 0,100 % (m/m) manganese is equal to 0,107 % (m/m) cobalt.

9.3 Tolerances

The deviations between two or three independent determinations shall not exceed the values shown in the table.

Cobalt content %	Range for two determinations %	Range for three determinations %
from 1 to 5	0,10	0,12
over 5 to 8	0,15	0,18
over 8 to 20	0,20	0,25
over 20	0,30	0,35

9.4 Final result

Report the arithmetical mean of acceptable determinations rounded to the nearest 0,1 %.

10 TEST REPORT

The test report shall include the following information :

- a) reference to this International Standard;
- b) all details necessary for identification of the test sample;
- c) the result obtained;
- d) all operations not specified by this International Standard, or regarded as optional;
- e) details of any occurrence which may have affected the result.

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