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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.
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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.