SLOVENSKI STANDARD
SIST EN 24501:2000

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Hardmetals - Determination of titanium - Photometric peroxide method (ISO 4501:1978)

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Hartmetalle - Bestimmung des Titangehaltes - Photometrisches Wasserstoffsuperoxidverfahren (ISO 4501:1978)

Métaux durs - Détermination du titane - Méthode photométrique au peroxyde (ISO 4501:1978)

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English version

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This European Standard was approved by CEN on 1993-10-25. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

On the proposal of the CEN Central Secretariat, the Technical Board decided to submit the International Standard:

"Hardmetals - Determination of titanium - Photometric peroxide method (ISO 4501:1978)"

to the formal vote.

The result of the formal vote was positive.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 1994, and conflicting national standards shall be withdrawn at the latest by April 1994.

In accordance with the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of the International Standard ISO 4501:1978 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD 4501

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

Hardmetals — Determination of titanium — Photometric peroxide method

Métaux-durs — Détermination du titane — Méthode photométrique au peroxyde

First edition — 1978-08-01

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Descriptors : hardmetals, chemical analysis, determination of content, titanium, spectrophotometric analysis.

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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.

International Standard ISO 4501 was developed by Technical Committee ISO/TC 119, *Powder metallurgical materials and products*, and was circulated to the member bodies in June 1977.

It has been approved by the member bodies of the following countries :

Australia	Germany	Spain
Austria	Italy	Sweden
Bulgaria	Japan	Turkey
Canada	Mexico	United Kingdom
Czechoslovakia	Poland	U.S.A.
Egypt, Arab Rep. of	Romania	U.S.S.R.
France	South Africa, Rep. of	Yugoslavia

No member body expressed disapproval of the document.

Printed in Switzerland

Hardmetals — Determination of titanium — Photometric peroxide method

1 SCOPE

This International Standard specifies a photometric peroxide method for the determination of the titanium content of carbides and hardmetals.

2 FIELD OF APPLICATION

This method is applicable to

— carbide and binder metal powder mixtures, free of lubricant,

— all grades of presintered or sintered hardmetals,

with a titanium content exceeding 0,2 % (*m*/*m*).

3 PRINCIPLE

Formation of a yellow complex of pertitanic acid. Determination of the absorbance of the complex.

4 INTERFERING ELEMENTS

The effect of interfering elements which also form coloured complexes with hydrogen peroxide under the conditions specified, for example vanadium and molybdenum, shall be taken into account. A correction can be applied if the content is below 5 % (*m*/*m*) of each.

5 REAGENTS

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

5.1 Ammonium hydrogen fluoride.

5.2 Ammonium sulphate.

5.3 Sodium disulphite.

5.4 High-purity titanium metal or titanium dioxide, analysed for all impurities exceeding 0,01 % (*m*/*m*) each.

5.5 Citric acid solution, 30 % (*m*/*m*).

5.6 Perchloric acid, ρ 1,54 or 1,67 g/ml.

5.7 Sulphuric acid, ρ 1,84 g/ml.

5.8 Sulphuric acid, ρ 1,54 g/ml (sulphuric acid, ρ 1,84 g/ml, diluted 1 + 1).

5.9 Hydrogen peroxide, 30 % (*m*/*m*).

5.10 Hydrofluoric acid, ρ 1,12 g/ml.

5.11 Nitric acid, ρ 1,40 g/ml.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Spectrophotometer or **filter photometer.**

7 SAMPLE PREPARATION

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 Preparation

Select the mass of the test portion, the volume of the volumetric flask and the cell length in accordance with table 1.

TABLE 1

Titanium content % (*m*/*m*)	Test portion mass g	Flask volume ml	Cell length mm
0,2 to 4	0,2	250	20
2 to 8	0,2	250	10
5 to 15	0,2	500	10
10 to 30	0,1	500	10

NOTE — Approximately 17 mg of titanium in 250 ml of final solution gives unit absorbance with a 10 mm cell.

8.2 Test portion

Weigh, to the nearest 0,000 1 g, 0,1 or 0,2 g of the test sample.

8.3 Attack

Transfer the test portion into a 100 to 200 ml conical flask or a 250 ml beaker. Add 5 g of the ammonium sulphate (5.2) and 10 ml of the sulphuric acid (5.7). Cover the beaker or flask with a watch glass. Heat near the boiling point until complete dissolution is achieved.

Alternative dissolution method : Transfer the test portion into a platinum dish. Add 10 ml of water and 5 ml of the hydrofluoric acid (5.10). Cover the dish with a polypropylene or platinum cover. Heat to approximately 80 °C. Add the nitric acid (5.11) drop by drop until solution is complete. Cool. Add 10 ml of the sulphuric acid (5.7) and 5 g of the ammonium sulphate (5.2). Heat until fumes (SO_3) are observed. Cool.

If any unattacked particles or carbonaceous matter are left, cool to below 100 °C, and cautiously add 1 ml of the perchloric acid (5.6). Heat to fume off the perchloric acid then cool to room temperature.

CAUTION – When using perchloric acid avoid contact with organic matter.

8.4 Preparation of the solution for analysis

Add, in small portions, 1 ml of the hydrogen peroxide (5.9), mixing by shaking. Add, in small portions, 30 ml of the citric acid solution (5.5), while mixing. Add 40 ml of water.

8.4.1 *Using a* 250 ml *volumetric flask*

Transfer the solution into the volumetric flask, rinsing copiously with water. Add 25 ml of the sulphuric acid (5.8), fill nearly to the mark and mix. Cool. Add 1 ml of the hydrogen peroxide (5.9), mix, make up to volume and mix again.

8.4.2 *Using a* 500 ml *volumetric flask*

Transfer the solution into the volumetric flask, rinsing copiously with water. Add 30 ml of the citric acid solution (5.5) and 50 ml of the sulphuric acid (5.8), fill nearly to the mark and mix. Cool. Add 2 ml of the hydrogen peroxide (5.9), mix, make up to volume, and mix again.

8.5 Compensating solution

Transfer approximately 30 ml of the coloured solution (8.4) into a 50 ml beaker. Add approximately 0,2 g of the sodium disulphite (5.3) to decolorize the solution. If necessary, repeat the addition of sodium disulphite.

8.6 Determination of absorbance

Choose appropriate cells. Measure the absorbance of the coloured and the compensating solutions at a wavelength of 420 nm using the same cell or a matched pair of cells.

9 ELIMINATION OF INTERFERENCES

9.1 Vanadium

Transfer 30 ml of the coloured solution (8.4) into a 50 ml beaker and add approximately 0,1 g of the ammonium hydrogen fluoride (5.1), which destroys the coloured titanium complex. Wait for 3 min. The colour left is due to vanadium.

Measure the absorbance and subtract it from the absorbance due to titanium plus vanadium.

9.2 Molybdenum

The colour of the molybdenum peroxide complex is weak. It cannot be compensated for chemically but a correction can be calculated from the molybdenum content. 1 % of molybdenum in the sample corresponds to approximately 0,08 % of titanium at 420 nm, but the correction should be determined with the spectrophotometer used.

10 PREPARATION OF THE CALIBRATION CURVE

10.1 Standard titanium solution

The standard titanium solution can be prepared from titanium metal or titanium dioxide.

10.1.1 *Standard titanium solution from titanium metal*

Weigh approximately 250 mg of the titanium metal (5.4) and transfer it into a 200 ml conical flask. Add 30 ml of water and 20 ml of the sulphuric acid (5.8). Cover the flask. Warm gently and keep the volume constant by adding water until all metal has dissolved. Cool. Add approximately 1 ml of the hydrogen peroxide (5.9) to oxidize the titanium. Evaporate the water by heating to fumes. Cool. Add 10 g of the ammonium sulphate (5.2) and heat gently until deposits on the walls of the flask have dissolved. Cool. Add about 50 ml of water. Transfer into a 250 ml volumetric flask and add 25 ml of the sulphuric acid (5.8). Cool. Make up to volume with water and mix. Calculate the titanium content in grams of Ti per litre.

10.1.2 *Standard titanium solution from titanium dioxide*

Weigh a quantity of the titanium dioxide (5.4) calculated to contain approximately 250 mg of titanium and transfer it into a 200 ml conical flask. Add 10 g of the ammonium sulphate (5.2) and 10 ml of the sulphuric acid (5.7). Cover the flask. Heat to near boiling until a clear solution is obtained. Cool. Add, in small portions, 1 ml of the hydrogen peroxide (5.9), mixing by shaking. Add about 50 ml of water. Transfer into a 250 ml volumetric flask and add 25 ml of the sulphuric acid (5.8). Cool. Make up to volume with water and mix. Calculate the titanium content in grams of Ti per litre.