## Kõvasulamid. Metalliliste elementide sisalduse määramine röntgenfluorestsentsmeetodil. Sulatusmeetod

Hardmetals - Determination of contents of metallic elements by X-ray fluorescence - Fusion method



### **EESTI STANDARDI EESSÕNA**

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Käesolev Eesti standard EVS-EN 24503:2000 sisaldab Euroopa standardi EN 24503:1993 ingliskeelset teksti.

Standard on kinnitatud Eesti Standardikeskuse 11.01.2000 käskkirjaga ja jõustub sellekohase teate avaldamisel EVS Teatajas.

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This Estonian standard EVS-EN 24503:2000 consists of the English text of the European standard EN 24503:1993.

This standard is ratified with the order of Estonian Centre for Standardisation dated 11.01.2000 and is endorsed with the notification published in the official bulletin of the Estonian national standardisation organisation.

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ICS 77.160

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### EN 24503:1993

### NORME EUROPÉENNE

### **EUROPÄISCHE NORM**

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Descriptors:

Powder metallurgy, hard metals, chemical analysis, metals, spectrophotometric analysis, X ray fluorescense spectrometry

English version

Hardmetals - Determination of contents of metallic elements by X-ray fluorescence - Fusion method (ISO 4503:1978)

Métaux-durs - Dosage des éléments métalliques par fluorescence de rayons X - Méthode par fusion (ISO 4503:1978) Hartmetalle – Bestimmung des Gehaltes metallischer Elemente durch Röntgenfluoreszenz in fester Lösung (ISO 4503:1978)

This European Standard was approved by CEN on 1993-04-02. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard in 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.

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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**

In 1992 ISO 4503:1978 "Hardmetals - Determination of contents of metallic elements by X-ray fluorescense - Fusion method" was submitted to the CEN Primary Questionnaire procedure.

Following the positive result of the CEN/CS Proposal ISO 4503:1978 was submitted to the CEN 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 October 1993, and conflicting national standards shall be withdrawn at the latest by October 1993.

According to the Internal Regulations of CEN/CENELEC, 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 4503:1978 was approved by CEN as a European Standard without any modification.

### INTERNATIONAL STANDARD



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION●MEЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ●ORGANISATION INTERNATIONALE DE NORMALISATION

# Hardmetals – Determination of contents of metallic elements by X-ray fluorescence - Fusion method

Descriptors: hardmetals, carbides, chemical analysis, determination of content, metals, cobalt, chromium, iron, manganese, molybdenum, nickel, niobium, tantalum, titanium, tungsten, vanadium, zirconium, spectrophotometric analysis, X-ray fluorescence spectrometry

Métaux-durs — Dosage des éléments métallagues par fluorescence de rayons X — Méthode par fusion

First edition - 1978-06-15

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UDC 621.762.661.665.2:546.3:543.422.8

Ref. No. ISO 4503-1978 (E)

### **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 4503 was developed by Sechnical 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

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

No member body expressed disapproval of the document.

## Hardmetals — Determination of contents of metallic elements by X-ray fluorescence — Fusion method

### 1 SCOPE

This International Standard specifies an X-ray fluorescence fusion method for the determination of cobalt, chromium, iron, manganese, molybdenum, nickel, niobium, tantalum, titanium, tungsten, vanadium and zirconium contents of carbides and hardmetals.

### 2 FIELD OF APPLICATION

The method is applicable to

- carbides of niobium, tantalum, titanium, vanadium, tungsten and zirconium,
- mixtures of these carbides and binder metals,
- all grades of presintered or sintered hardment produced from these carbides,

with the element contents shown in table 1.

TABLE 1

Element	Content, % (m/m)	
	min.	max.
Со	0,05	50
Cr	0,05	2,0
Fe	0,05	2,0
Mn	0,05	2,5
Мо	0,05	5,0
Nb	0,05	15
Ni	0,05	5,0
Та	0,10	30
Ti	0,3	30
V	0,15	4,0
W	45	95
Zr	0,05	2,0

### 3 PRINCIPLE

Measurement of the intensity of the characteristic X-ray spectrum of the elements being determined. To eliminate the effects of particle size and inter-element effects, the test portion is dissolved in a suitable mixture of acids and converted to sulphates, or is directly oxidized. Either the sulphates or the oxides are then fused with a mixture of sodium tetraborate and a barium compound.

### **4 INTERFERING ELEMENTS**

The effect of interfering elements, such as line interference of titanium and tungsten on vanadium, shall be taken into account.

#### 5 REAGENTS

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

- **5.1 Barium peroxide**, anhydrous, or **barium carbonate**, anhydrous.
- 5.2 Sodium tetraborate, anhydrous.

To ensure freedom from water, heat the sodium tetraborate to approximately 400  $^{\circ}$ C.

- 5.3 Hydrofluoric acid,  $\rho$  1,12 g/ml.
- Nitric acid,  $\rho$  1,24 g/ml (nitric acid,  $\rho$  1,42 g/ml, diluted 1  $\pm$  1).
- **5.5 Subturic acid**,  $\rho$  **1**,54 g/ml (sulphuric acid,  $\rho$  **1**,84 g/ml, diluted **1 1**).

### 6 APPARATUS

Ordinary laboratory apparatus and

- 6.1 X-ray spectrometer
- **6.2 Furnaces** for oxidation of the test portion at 700 to  $900\,^{\circ}\text{C}$  and for preparation of the borate melt at approximately 1  $100\,^{\circ}\text{C}$ .
- 6.3 Platinum dishes, 50 to 100 ml.

NOTE - Dishes of 95 % Pt + 5 % Au are to be preferred.

**6.4** Plate, with polished surface, of platinum alloy, for example 85 % Pt + 10 % Rh + 5 % Au or 95 % Pt + 5 % Au.

The plate shall be maintained at a surface temperature between 300 and  $400\,^{\circ}$ C so that the borate disk (see 8.4) loosens easily and does not crack.