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

Hardmetals - Determination of contents of metallic elements by X-ray fluorescence -
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Hartmetalle - Bestimmung des Gehaltes metallischer Elemente durch
Röntgenfluoreszenz in fester Lösung (ISO 4503:1978)

Métaux-durs - Dosage des éléments métalliques par fluorescence de rayons X -
Méthode par fusion (ISO 4503:1978)

[SIST EN 24503:2000](#)

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EUROPEAN STANDARD

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Descriptors: Powder metallurgy, hard metals, chemical analysis, metals, spectrophotometric analysis, X ray fluorescence spectrometry

English version

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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 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.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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

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

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Foreword

In 1992 ISO 4503:1978 "Hardmetals - Determination of contents of metallic elements by X-ray fluorescence - 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.

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Endorsement notice

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The text of the International Standard ISO 4503:1978 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD 4503

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

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

Métaux-durs – Dosage des éléments métalliques par fluorescence de rayons X – Méthode par fusion

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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

Price based on 3 pages

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

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

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

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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 hardmetals produced from these carbides.

with the element contents shown in table 1.

TABLE 1

Element	Content, % (m/m)	
	min.	max.
Co	0,05	50
Cr	0,05	2,0
Fe	0,05	2,0
Mn	0,05	2,5
Mo	0,05	5,0
Nb	0,05	15
Ni	0,05	5,0
Ta	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 °C.

5.3 Hydrofluoric acid, ρ 1,12 g/ml.

5.4 Nitric acid, ρ 1,24 g/ml (nitric acid, ρ 1,42 g/ml, diluted 1 + 1).

5.5 Sulphuric acid, ρ 1,54 g/ml (sulphuric acid, ρ 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 °C and for preparation of the borate melt at approximately 1 100 °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 °C so that the borate disk (see 8.4) loosens easily and does not crack.

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6.5 Brass rings or heat-resisting steel cylinders or graphite cylinders.

NOTE — Instead of the plate (6.4) and brass rings (6.5), moulds of graphite or heat-resisting steel may be used.

6.6 Wet or dry grinding device.**7 SAMPLING**

7.1 The sample shall be crushed in a mortar made of material which does not alter the sample composition. The crushed material shall pass a 2 mm sieve when using the acid solution method or a 0,18 mm sieve when using the oxidation method.

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

8 PROCEDURE

8.1 Weigh, to the nearest 0,001 g, approximately 0,5 g of the test sample.

NOTE — If the sample includes lubricant, a correction for the lubricant content must be applied.

8.2 Transform the test portion in a platinum dish by the preferred procedure of dissolution (8.2.1) or by oxidation (8.2.2). The oxidation procedure shall not be used if molybdenum may be present in quantities above 0,1 % (*m/m*).

8.2.1 Add 15 ml of the nitric acid (5.4) to the unoxidized or partially oxidized test portion, heat the dish moderately, add 2 ml of the hydrofluoric acid (5.3), drop by drop, and maintain the dish at moderate temperature.

Following complete dissolution of the test portion, add 1 to 2 ml of the sulphuric acid (5.5). Take to dryness and heat at approximately 600 °C until no more SO₃ vapour is evolved. Cool.

There is a possibility of transfer of molybdenum from one crucible to another, so care should be taken to avoid such transfer during the heating procedure. Therefore, do not heat samples with high and low molybdenum contents in the same furnace batch.

8.2.2 Oxidize the test portion in air in a furnace at 700 to 900 °C for about 1 h. If complete oxidation is achieved, continue as in 8.3. If complete oxidation is not achieved (as in the case of hardmetals with a high content of titanium), continue as in 8.2.1.

8.3 Mix the test portion well with the fused buffer mixture consisting of 15 ± 0,01 g of the sodium tetraborate (5.2) and 4 ± 0,01 g of the barium peroxide (5.1) or 4,5 ± 0,01 g of the barium carbonate (5.1), and heat the dish moderately until the contents are completely fused and the reaction has subsided. Cover the dish with a lid. Dissolve the oxides by melting at approximately 1 100 °C for 10 to 15 min. Swirl the melt to obtain a homogeneous mass.

8.4 Pour the melt into the preheated ring (6.5) placed on the support plate (6.4) lying on a heating device at about 300 to 400 °C. Allow to cool until the disk loosens from the plate. Place the disk with the ring on an asbestos plate and allow it to cool to room temperature.

8.5 Grind the disk on the wet or dry grinding device (6.6) until an even and smooth surface is obtained. Immediately rinse and dry the surface.

NOTE — Final wet or dry grinding should be done on an abrasive paper N 220. Care must be taken to avoid contamination of one specimen by another from the abrasive paper, when dry grinding is used.

8.6 Analyse with the X-ray spectrometer.

9 X-RAY FLUORESCENCE ANALYSIS

9.1 All measuring parameters, including the target material of the X-ray tube, shall be chosen to obtain the optimal number of impulses.

9.2 The analytical lines shown in table 2 shall be used.

TABLE 2

Element	Co, Cr, Fe, Mn, Mo, Nb, Ni, Ti, V, Zr	Ta, W
Analytical line	$K_{\alpha 1,2}$	$L_{\alpha 1}$

NOTE — It is recommended that all elements be measured in the first order of reflexion by a LiF (200) crystal. For titanium, a pentaerythritol crystal can be used, with measurements in the second order.

9.3 Background corrections shall be made when necessary.

10 PREPARATION OF CALIBRATION CURVES

The calibration curves shall be plotted on the basis of synthetically prepared samples with compositions as given in clause 2 and roughly corresponding to the grades of hardmetals to be analysed. These samples shall be prepared by mixing accurately known amounts of metals or their suitable compounds. The mixture shall be analysed as described in 8.2 to 8.6.