



# SLOVENSKI STANDARD

## SIST EN 24883:2000

01-december-2000

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### Hardmetals - Determination of contents of metallic elements by X-ray fluorescence - Solution method (ISO 4883:1978)

Hardmetals - Determination of contents of metallic elements by X-ray fluorescence -  
Solution method (ISO 4883:1978)

Hartmetalle - Bestimmung des Gehaltes metallischer Elemente durch  
Röntgenfluoreszenz - Lösungsverfahren (ISO 4883:1978)

Métaux-durs - Dosage des éléments métalliques par fluorescence de rayons X -  
Méthode par solution (ISO 4883:1978)

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Ta slovenski standard je istoveten z: **EN 24883:1993**

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#### **ICS:**

77.160

Metalurgija prahov

Powder metallurgy

**SIST EN 24883:2000**

**en**

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EUROPEAN STANDARD

EN 24883:1993

NORME EUROPÉENNE

EUROPÄISCHE NORM

April 1993

UDC 669.018.25:620.1:543.422.8:546.3.047

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

English version

**Hardmetals - Determination of contents of metallic elements by X-ray fluorescence - Solution method (ISO 4883:1978)**

**iTeh STANDARD PREVIEW**

Métaux-durs - Dosage des éléments métalliques par fluorescence de rayons X - Méthode par solution (ISO 4883:1978)

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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

## Foreword

In 1992 ISO 4883:1978 "Hardmetals - Determination of contents of metallic elements by X-ray fluorescence - Solution method" was submitted to the CEN Primary Questionnaire procedure.

Following the positive result of the CEN/CS Proposal ISO 4883:1978 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 4883:1978 was approved by CEN as a European Standard without any modification.

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**INTERNATIONAL STANDARD****4883**

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

*Métaux-durs — Dosage des éléments métalliques par fluorescence de rayons X — Méthode par solution*

First edition — 1978-06-15

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UDC 621.762 : 661.665.2 : 546.3 : 543.422.8

Ref. No. ISO 4883-1978 (E)

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

Price based on 2 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 4883 was developed by Technical Committee ISO/TC 119, *Powder metallurgical materials and products*, and was circulated to the member bodies in December 1977.

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

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

No member body expressed disapproval of the document.

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

## 1 SCOPE

This International Standard specifies an X-ray fluorescence solution method for the determination of cobalt, 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 minimum element contents shown in table 1.

TABLE 1

Element	Minimum content % (m/m)
Co	0,05
Fe	0,05
Mn	0,05
Mo	0,05
Nb	0,07
Ni	0,05
Ta	0,10
Ti	0,2
V	0,05
W	0,10
Zr	0,05

## 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 mixture of hydrofluoric and nitric acids.

## 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 Hydrofluoric acid**,  $\rho$  1,12 g/ml.

**5.2 Nitric acid**,  $\rho$  1,42 g/ml.

**5.3 Solvent solution.**

Mix 2 parts of the hydrofluoric acid (5.1), 1 part of the nitric acid (5.2) and 2 parts of distilled water.

**5.4 Tartaric acid solution**, 200 g/l.

## 6 APPARATUS

Ordinary laboratory apparatus and

**6.1 X-ray spectrometer**, suitable for solution analysis.

**6.2 Sample cells**, resistant to hydrofluoric-nitric acid mixture, with a window consisting of 6  $\mu$ m thick film of polyethylene terephthalic acid ester.

## 7 SAMPLING

**7.1** The sample shall be crushed in a mortar made of a material which does not alter the sample composition. The crushed material shall pass a 2 mm sieve.

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

## 8 PROCEDURE

**8.1** Weigh  $2 \pm 0,001$  g of the test sample into a 150 ml polypropylene beaker.

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

**8.2** Add 20 ml of the solvent solution (5.3). Dissolve the test portion completely by heating on a water bath for 30 min.

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8.3 Cool and transfer the solution into a 50 ml polypropylene volumetric flask containing 10 ml of the tartaric acid solution (5.4).

Make up to volume with water and mix.

8.4 Filter the solution through a dry filter paper into a polypropylene beaker.

8.5 Fill the cell (6.2) to a height of at least 10 mm with the solution.

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

9.3 Background corrections shall be made when necessary.

## 10 PREPARATION OF CALIBRATION CURVES

10.1 The calibration shall be performed with at least five calibration samples prepared according to clause 8 from mixtures of accurately known amounts of pure metals or their suitable compounds. It is essential to construct separate calibration curves for different types of hardmetals.

A calibration sample of approximately the same composition as the sample to be analysed shall be used as an external standard.

A calibration curve shall be drawn for each element by plotting its concentration versus the ratio of the count rate of each calibration sample to that of the external standard.

10.2 Elements in the test sample shall be determined by calculating the ratio of the count rates in the test sample to the external standard, and taking the concentration from the appropriate calibration curve.

## 11 EXPRESSION OF RESULTS

### 11.1 Tolerances

The deviations between two or three independent determinations shall not exceed the values shown in table 3.

TABLE 3

Content %	Range for two determinations %	Range for three determinations %
from 0,05 to 0,4	0,04	0,05
over 0,4 to 2	0,20	0,25
over 2 to 10	0,30	0,35
over 10 to 30	0,4	0,5
over 30 to 95	1,0	1,2

### 11.2 Final result

Report the arithmetical mean of acceptable determinations rounded to the nearest value as shown in table 4.

TABLE 4

Content %	Round to the nearest %
from 0,05 to 0,4	0,01
over 0,4 to 30	0,1
over 30 to 95	1

## 12 TEST REPORT

The test report shall include the following information :

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