INTERNATIONAL STANDARD 4883

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

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

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

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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 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.

Printed in Switzerland

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, ρ 1,12 g/ml.

5.2 Nitric acid, ρ 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 µm 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 ± 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.

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 :

a) reference to this International Standard;

b) all details necessary for identification of the test sample;

c) the result obtained;

d) all operations not specified by this International Standard, or regarded as optional;

e) details of any occurrence which may have affected the result.