INTERNATIONAL STANDARD ISO 4503

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

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FOREWORD

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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.

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 SO_3 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.

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.

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