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International Standard



7627/4

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**Hardmetals — Chemical analysis by flame atomic
absorption spectrometry —
Part 4: Determination of molybdenum, titanium and
vanadium in contents from 0,01 to 0,5 % (m/m)**

Métaux-durs — Analyse chimique par spectrométrie d'absorption atomique dans la flamme — Partie 4: Dosage du molybdène, du titane et du vanadium à des teneurs comprises entre 0,01 et 0,5 % (m/m)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (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 authorized 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 7627/4 was developed by Technical Committee ISO/TC 119, *Powder metallurgy*, and was circulated to the member bodies in August 1982.

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

Austria	Germany, F.R.	Spain
Brazil	Italy	Sweden
Bulgaria	Korea, Rep. of	Switzerland
China	Norway	United Kingdom
Czechoslovakia	Poland	USA
Egypt, Arab Rep. of	Romania	USSR
France	South Africa, Rep. of	

No member body expressed disapproval of the document.

Hardmetals — Chemical analysis by flame atomic absorption spectrometry —

Part 4: Determination of molybdenum, titanium and vanadium in contents from 0,01 to 0,5 % (m/m)

1 Scope and field of application

This part of ISO 7627 specifies the method to be used for the determination of molybdenum, titanium and vanadium contents of hardmetals within the range 0,01 to 0,5 % (m/m) by flame atomic absorption spectrometry.

General requirements concerning the field of application, principle, interfering elements, apparatus, sampling and test report are given in ISO 7627/1.

2 Reference

ISO 7627/1, *Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 1: General requirements.*

3 Reagents

3.1 Hydrofluoric acid, ρ 1,12 g/ml.

3.2 Nitric acid, ρ 1,42 g/ml.

3.3 Ammonium fluoride, 0,1 g/ml solution.

3.4 Caesium chloride, 0,01 g/ml solution.

3.5 High-purity stock solutions, for calibration purposes for each element to be determined, containing 1,000 g of the element per litre.

NOTE — This value is understood to establish a maximum limit of 1,000 5 g and a minimum limit of 0,999 5 g.

4 Procedure

4.1 Test portion

Weigh, to the nearest 0,001 g, approximately 1 g of the test sample. Transfer it to a 100 ml polytetrafluorethylene beaker or a beaker of other suitable material. Cover the beaker.

4.2 Dissolution of the test portion

Add 10 ml of water, 5 ml of the hydrofluoric acid (3.1), and then 5 ml of the nitric acid (3.2), drop by drop, to the beaker containing the test portion (4.1) and heat gently until the test portion is completely dissolved. Add 10 ml of the caesium chloride solution (3.4) and 10 ml of the ammonium fluoride solution (3.3). Then transfer the solution totally to a 100 ml polypropylene one-mark volumetric flask and dilute to the mark.

4.3 Dilution volume

Prepare the relevant dilution volume for the analysis according to table 1 as follows:

4.3.1 Dilution volume 100 ml: use the solution in 4.2.

4.3.2 The concentration of the solution may be reduced by a factor of 10 for instruments of higher sensitivity by transferring 10 ml of the solution in 4.2 to a 100 ml polypropylene one-mark volumetric flask. Add 10 ml of the caesium chloride solution (3.4). Add 10 ml of the ammonium fluoride solution (3.3) and dilute to the mark.

4.4 Preparation of calibration and blank solutions

4.4.1 Prepare at least six solutions according to 4.2 with a matrix composition as similar as possible to the test portion to be analysed, but without making up to volume. Then add increasing volumes of properly diluted stock solutions of the elements to be determined according to the concentration ranges to be covered. Make up to 100 ml and mix.

4.4.2 Also prepare calibration solutions with a diluted matrix in accordance with 4.3 for dilution volumes of 1 000 ml, if necessary.

4.4.3 Prepare one or more blank solutions (see 4.4.1) without the addition of the relevant element to be determined.

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Table 1 — Instrumental parameters and characteristics of calibration functions

Element	Dilution volume (<i>V</i>) for 1 g test portion ¹⁾ ml	Oxidant	Wavelength nm	Reciprocal sensitivity, for 1 % absorption ¹⁾ µg/ml	Linear range ¹⁾ %	Notes
Mo	100	N ₂ O	313,3	0,3	0,003 to 0,1	1
	100	N ₂ O	313,3	6	0,06 to 10,0	1 and 2
Ti	100	N ₂ O	364,3	1,0	0,03 to 0,5	1
V	100	N ₂ O	318,4	0,8	0,008 to 0,5	3

1) Guidelines for information only.

NOTES

- 1 Use a fuel-rich flame.
- 2 Adjust the burner head at right angles to the light beam or dilute the solutions appropriately.
- 3 Use of the triplet.

4.5 Adjustment of the atomic absorption spectrometer

SAFETY PRECAUTION: Follow the manufacturer's recommendation on igniting and extinguishing the flame.

Optimize the response of the instrument at the wavelength given for the element being determined. See table 1.

Preheat the burner for about 5 min and then adjust the fuel and correct the burner to obtain maximum absorption while aspirating a calibration solution. Make sure that the absorbance reading is not drifting. Aspirate water and set the initial reading to zero absorbance.

4.6 Atomic absorbance measurements

4.6.1 Aspirate first the blank solution and then the calibration and test solutions consecutively and record the readings. Aspirate water between each solution. Make at least two measurements for each solution. If a build-up of solids occurs on the burner slit, it will cause a decrease of sensitivity and must be removed.

4.6.2 Prepare a calibration curve by plotting the obtained absorbance values of the calibration solutions corrected for the blank against the concentration, in milligrams per litre, of the element.

4.6.3 Convert the absorbance values of the test solutions corrected for the blank to milligrams of the element per litre by means of the calibration curve.

5 Expression of results

5.1 Calculation

The element content, expressed as a percentage by mass, is given by the formula

$$\frac{c \times V}{10^4 \times m}$$

where

c is the concentration, in milligrams per litre, of the element of the test solution;

V is the dilution volume, in millilitres;

m is the mass, in grams, of the test portion.

5.2 Permissible tolerances

The deviations between three independent determinations shall not exceed 0,2 times the element content in per cent (absolute value).

5.3 Final result

Report the arithmetical mean of acceptable determinations rounded to the nearest value as shown in table 2.

Table 2 — Rounding of results

Content % (m/m)	Round to the nearest % (m/m)
from 0,01 to 0,1	0,01
over 0,1 to 0,5	0,05

If the element content is below 0,01 % (m/m), report it as less than 0,01 % (m/m).