Powder Metallurgical Materials and Products Sectional Committee, MTD 25

FOREWORD

This Indian Standard (First Revision) is to be adopted by the Bureau of Indian Standards, after the draft finalized by the Powder Metallurgical Materials and Products Sectional Committee has been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1988. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

This standard is published in six parts. Chemical analysis of hardmetals by flame atomic absorption spectrometric methods is covered in the following six parts:

	Percent Range of Each Element (m/m)
Part 1 General Requirement	-
Part 2 Determination of calcium potassium, magnesium and sodium	0.001 - 0.02
Part 3 Determination of cobalt iron, manganese and nickel	0.01 – 0.5
Part 4 Determination of molybdenum, titanium and vanadium	0.01 – 0.5
Part 5 Determination of cobalt, iron manganese, molybdenum, nickel, titanium and vanadium	0.5 - 2
Part 6 Determination of chromium	0.01 - 2

NOTE - The method for determination of chromium also permits determination of iron, nickel and manganese within the range of 0.01 to 2 percent

In the preparation of this standard, assistance has been derived from ISO 7627 - 4 : 1983 ‘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)’.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for Rounding Off Numerical Values’. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Draft Indian Standard

CHEMICAL ANALYSIS OF HARDMETALS BY FLAME ATOMIC ABSORPTION SPECTROMETRY

PART 4 DETERMINATION OF MOLYBDENUM, TITANIUM AND VANADIUM IN CONTENTS FROM 0.01 TO 0.5 PERCENT (m/m)

(First Revision)

1 SCOPE

1.1 The standard (part 4) specifics the method to be used for determination of molybdenum, titanium and vanadium content in hardmetals within the range 0.01 to 0.5 *percent* (*m/m*) by flame atomic absorption spectrometry.

1.2 General requirement concerning the field of application, principle, interfering elements apparatus, sampling and test report are given in part-1 of this standard.

2 REAGENTS

During analysis, only reagent of analytical grade and distilled water or water of equivalent purity shall be used.

2.1 Hydrofluoric Acid ($\rho = 1.12$ g/ml)

2.2 Concentrated nitric acid ($\rho = 1.42$ g/ml)

2.3 Ammonium Fluoride ($\rho = 0.1$ g/ml)

2.4 Caesium Chloride ($\rho = 0.01$ g/ml)

2.5 Standard Molybdenum Solution (1 ml = 1 mg of Mo)

Weight (to the nearest 0.000 1 g) 1.500 3 g of molybdenum trioxide previous dried at 110°C and cooled in a desiccator and dissolve in few ml of dilute sodium hydroxide solution and dilute with water . Add 10 ml of dilute hydrochloride acid (1: 1), When dissolution is complete, transfer to 1 litre volumetric flask, dilute with water up to the mark and mix.

2.6 Standard Titanium Solution (1 ml = 1 mg of Ti)

Fuse (to the nearest 0.000 1 g) 0.166 8 g of titanium dioxide in a covered platinum or silica crucible with 3 g of potassium pyrosulphate (potassium disulphate $K_2S_2O_7$) Cool the melt and dissolve in 50 ml hot dilute sulphuric acid (1: 1). Cool and transfer to 1 litre volumetric flask and make up the volume with dilute sulphuric acid (1 : 1) and mix.

2.7 Standard Vanadium Solution (1 ml = 1 mg of V)

Dissolve (to the nearest 0.000 1 g) l g of vanadium (purity 99.9 *percent*) in 20 ml of aquaregia (3 vol. of HCL and 1 vol. of HNO_3). Evaporate nearly to dryness and add 10 ml of hydrochloric acid. Cool and transfer to 1 litre volumetric flask and make up the volume and mix.

3 PROCEDURE

3.1 Test Portion

Weigh (to the nearest 0.001 g) approximately 1 g of the test sample. Transfer it to a 100 ml tetrafluoroethylene beaker. Cover the beaker.

3.2 Dissolution of the test portion

Add 10 ml of water, 5 ml of hydrofluoric acid and then 5 ml of concentrated nitric acid drop by drop, to the beaker containing the test portion and heat gently until the test portion is completely dissolved. Add 10 ml of caesium chloride solution and 10 ml of ammonium fluoride solution. Then, transfer the solution to a 100 ml polyethylene volumetric flask and dilute to the mark and mix.

3.3 Dilution Volume

Prepare the relevant dilution volume for the analysis according to Table 1 as follows.

3.3.1 *Dilution Volume* (100 ml = 1 g of test sample) — Use the solution in **3.2.**

3.3.2 *Dilution Volume* (1 000 ml = 1 g of test sample) — 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 **3.2** to a 100 ml polyethylene volumetric flask. Add 10 ml of caesium chloride solution, 10 ml of ammonium fluoride solution and dilute to the mark.

3.4 Preparation of Calibration and Blank Solutions

3.4.1 Prepare at least six solutions according to **3.2** 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 standard solutions (**2.5** to **2.7**) of the elements to be determined according to the concentration ranges to be covered. Make up to 100 ml and mix.

3.4.2 Also prepare calibration solutions with a diluted matrix in accordance with **3.3.2** for dilution volumes of 1 000 ml, if necessary,

3.4.3 Prepare two blank solution (*see* **3.4.1**) without the addition of the relevant element to be determined.

3.5 Adjustment of the Atomic Absorption Spectrometer

3.5.1 Ignite the flame. Optimize the response of the instrument at the wavelength given for the element being determined (see Table 1).

3.5.2 Preheat the burner for about 5 minutes 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.

3.6 Atomic Absorbance Measurements

3.6.1 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. Solids which build up on the burner slit, must be removed, otherwise they will cause a decrease of sensitivity.

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

TABLE 1 INSTRUMENTAL PARAMETER AND CHARACTERTICS OF CALIBRATION FUNCTION

(*Clauses* 3.3 *and* 3.5.1)

Sl No.	Element	Dilution Volume (*V*) for 1 g Test Portion in ml	Oxidant	Wave length (nm)	Reciprocal Sensitivity for 1 percent absorption µg/ml	Linear Ranges *Percent*	NOTE
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Mo	100	N_2O	313.3	0.3	0.003 – 0.1	1
		100	N_2O	313.3	6	0.06 – 10.0	1 and 2
ii)	Ti	100	N_2O	364.3	1.0	0.03 – 0.5	1
iii)	V	100	N_2O	318.4	0.8	0.08 – 0.5	3

NOTES

1. Mo & Ti - Use a fuel rich flame
2. Mo - Adjust the burner head at right angles to the light beam or dilute the solutions approximately.
3. V - Use the triplet

3.6.3 Convert the absorbance values of the test solution corrected for the blank to mg of the element per litre by means of means of the calibration curve.

4 TEST RESULTS

4.1 Calculation

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

$$\text{Element content} = \frac{C \times V}{10^4 \times m}$$

where,

C = concentration, in mg per litre, of the element in the test solution;
V = dilution volume, in ml; and
m = mass, in g, of the test portion.

4.2 Permissible Tolerances

The deviations between three independent determinations shall not exceed 0.000 5 *percent* (absolute value) at the lower limit of determination. It shall not exceed 0.002 *percent* at the 0.02 *percent* level.

4.3 Final Result

Report the arithmetical means of acceptable determinations rounded to the nearest value as shown in Table 2.

4.3.1 If the element content is below 0.01 *percent*, report it as less than 0.01 *percent*.

TABLE 2 ROUNDING OFF RESULTS

(*Clause* 4.3)

Sl No.	**Content percent**	**Round to the nearest percent**
(1)	(2)	(3)
i)	From 0.01 to 0.1	0.01
ii)	Over 0.1 to 0.5	0.05