

## 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:

	<i>Percent Range of Each Element ( m/m )</i>
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 - 5 : 1983 'Hardmetals - Chemical analysis by flame atomic absorption spectrometry - Part 5 determination of cobalt, iron, manganese, molybdenum, nickel, titanium and vanadium in contents from 0.5 to 2 percent (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.

## Indian Standard

# Chemical analysis of hardmetals by flame atomic absorption spectrometry

**Part 5 - determination of cobalt, iron, manganese, molybdenum, nickel, titanium and vanadium in contents from 0.5 to 2 percent (M/m)**

( *First Revision* )

## 1 SCOPE

**1.1** This standard ( Part 5 ) specifies the method to be used for the determination of cobalt, iron, manganese, molybdenum, nickel, titanium and vanadium contents in hardmetals within the range 0.5 to 2 percent ( *m/m* ) by flame atomic absorption spectrometry.

**1.2** General requirements concerning the field of application, principle, interfering elements, apparatus, sampling and test report are given in Part 1 of this Indian Standard IS 12473.

**NOTE**— Determination of element contents in high concentration by the atomic absorption spectrometry method yields a wider scattering of results than are obtainable by other methods.

## 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 Cobalt Solution** ( 1 ml = 1 mg of Co )

Weigh ( to the nearest 0.000 1 g ) 1 g of pure cobalt metal and dissolve in minimum quantity of dilute nitric acid ( 1 : 1 ). Add 40 ml of dilute sulphuric acid ( 1 : 1 ). Heat to white fumes, cool and transfer to 1 litre volumetric flask and make up the volume and mix.

Alternatively, dissolve 4.769 8 g of cobalt sulphate (  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$  ) in water and transfer to 1 litre volumetric flask and make up the volume and mix.

**2.6 Standard Iron Solution** ( 1 ml = 1 mg of  $\text{Fe}^{2+}$  )

Dissolve ( to the nearest 0.000 1 g ) 7.021 5 g of ammonium ferrous sulphate in 200 ml of water containing 10 ml of concentrated sulphuric acid. Dilute to 1 litre and mix.

**2.7 Standard Manganese Solution** ( 1 ml = 1 mg of Mn )

Dissolve ( to the nearest 0.000 1 g ) 1 g of electrolytic manganese ( 99.9 percent, *Min* ) in 50 ml of dilute nitric acid ( 1 : 1 ). Cool and transfer to 1 litre volumetric flask, dilute with water up to the mark and mix.

### **2.8 Standard Molybdenum Solution** ( 1 ml = 1mg of Mo)

Weigh 1.5003 g of the molybdenum trioxide ( MoO<sub>3</sub> ) previously dried at 110°C and cooled in a desiccator and dissolve in few ml of dilute sodium oxide solution, and dilute with water. Add 10 ml of dilute hydrochloric acid ( 1 : 1 ). When dissolution is complete, transfer to 1 litre volumetric flask, dilute with water up to the mark and mix.

### **2.9 Standard Nickel Solution** ( 1 ml = 1 mg of Ni )

Dissolve ( to the nearest 0.0001 g ) 1 g of nickel ( 99.9 percent, Min ) in 100 ml of dilute nitric acid ( 1 : 1 ) and heat slowly. Cool and transfer to 1 litre volumetric flask, dilute with water up to the mark and mix.

### **2.10 Standard Titanium Solution** (1 ml = 1 mg of Ti)

Fuse 0.1668 g of titanium dioxide ( TiO<sub>2</sub> ) in a covered platinum or silica crucible with 3 g of potassium pyrosulphate ( potassium disulphate, K<sub>2</sub>S<sub>2</sub>O<sub>7</sub> ). Cool the melt and dissolve in 50 ml hot dilute sulphuric acid ( 1 : 1 ). Cool and transfer to 100 ml volumetric flask and make up the volume with dilute sulphuric acid ( 1 : 1 ) and mix.

### **2.11 Standard Vanadium Solution** (1 ml = 1 mg of V )

Dissolve ( to the nearest 0.0001 g ) 1 g of vanadium ( purity 99.9 percent, Min ) in 20 ml of aquaregia ( 3 vol of HCl and 1 vol of HNO<sub>3</sub> ). 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 polytetra-fluoroethylene 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 each of caesium chloride and 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. Add 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 **2.11** ) 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 solutions ( *see* **3.4.1** ) without the addition of the relevant element to be determined.

<b>TABLE 1 INSTRUMENTAL PARAMETER AND CHARACTERISTICS OF CALIBRATION FUNCTION</b> ( <i>Clauses 3.3 and 3.5</i> )							
Sl No.	Element	Dilution Volume (V) for 1 g Test Portion in ml	Oxidant	Wave length (nm)	Reciprocal Sensitivity for 1 percent absorption $\mu\text{g/ml}$	Linear Ranges <i>Percent</i>	NOTES
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Co	100	N <sub>2</sub> O	352.7	20	0.1 to 15	
ii)	Fe	1000	N <sub>2</sub> O	248.3	0.3	0.03 to 2.0	
iii)	Mn	1000	N <sub>2</sub> O	279.8	0.1	0.01 to 2.0	1
iv)	Mo	100	N <sub>2</sub> O	313.3	6	0.06 to 10.0	2
v)	Ni	1000	N <sub>2</sub> O	232.0	0.3	0.05 to 5.0	3 and 4
vi)	Ti	100	N <sub>2</sub> O	364.3	30	0.2 to 10	2
vii)	V	1000	N <sub>2</sub> O	318.4	1.0	0.1 to 5.0	1

**NOTES**

1. Mn & V - Use the triplet
2. Mo & Ti - Use a fuel rich flame, Adjust the burner head at right angles to the light beam or dilute the solutions approximately
3. Ni - Sensitivity is greatly depending on matrix composition.
4. Ni - use of a narrow-spectral hand pass and single element hollow-cathode lamp in mandatory.

### 3.5 Adjustment of the Atomic Absorption Spectrometer

Ignite the flame. Optimize the response of the instrument at the wavelength given in Table 1 for the element being determined. 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 blank against the concentration, in mg per litre, of the element.

**3.6.3** Convert the absorbance values of the test solutions corrected for the blank to mg of the element per litre by 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 0.1 times the element content in *percent* (absolute value).

### 4.3 Final Result

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

TABLE 2 ROUNDING OFF RESULTS (Clause 4.3)		
SI No.	Content percent	Round to the nearest percent
(1)	(2)	(3)
i)	From 0.01 to 0.1	0.02
ii)	Over 0.1 to 0.5	0.05

**NOTE** — If the element content is below 0.5 *percent*, use the relevant part of this standard, and if the element is greater than 2 *percent*, report it as greater than 2 *percent*.