

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 7627-3 : 1983 'Part 3: Determination of cobalt, iron, manganese and nickel 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.

Indian Standard

**CHEMICAL ANALYSIS OF HARDMETALS BY FLAME ATOMIC ABSORPTION
SPECTROMETRY**

**PART 3 - DETERMINATION OF COBALT IRON MANGANESE AND NICKEL IN CONTENTS
FROM 0.01 TO 0.5 PERCENT (min)**

(*First Revision of IS 12473 Part - 3*)

1 SCOPE

1.1 This standard (Part 3) specifies the method to be used for the determination of cobalt, iron, manganese and nickel contents in hardmetals within the range **0.01 to 0.5 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 standard.

2 REAGENTS

During the analysis, use only reagents of analytical grade, and distilled water or water of equivalent purity.

2.1 Hydrofluoric Acid — $\rho = 1.12\text{g/ml}$.

2.2 Concentrated Nitric Acid ($\rho = 1.42\text{ g/ml}$)

2.3 Ammonium Fluoride (0.1 g/ml solution)

2.4 Caesium Chloride (0.01 g/ml solution)

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. Dilute to 1 litre and mix.

Alternatively, dissolve 4.769 8 g of cobalt sulphate ($\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$) in water and dilute to 1 litre.

2.6 Standard Iron solution (1 ml = 1 mg of Fe^{+2})

Dissolve 7.021 5 g of ammonium ferrous sulphate in 200 ml of water containing 10 ml 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 nickel (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 Nickel Solution (1 ml = 1 mg of Ni)

Dissolve (to the nearest 0.000 1 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.

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 polytetrafluoroethylene 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.

3.3 Dilution Volume

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

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 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 **2.8**) 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 solution 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.

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 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.

<p style="text-align: center;">TABLE 1 INSTRUMENTAL PARAMETERS AND CHARACTERISTICS OF CALIBRATION FUNCTIONS (<i>Clauses 3.3 and 3.5</i>)</p>

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
i)	Co	100	N ₂ O	240.7	0.7	0.007 to 0.5	1
ii)	Fe	100	N ₂ O	248.3	0.3	0.003 to 0.05	
		1 000	N ₂ O	248.3	0.3	0.03 to 2.0	
iii)	Mn	100	N ₂ O	279.8	0.1	0.001 to 0.03	2
		1 000	N ₂ O	279.8	0.1	0.01 to 2.0	2
iv)	Ni	100	N ₂ O	232.0	0.3	0.003 to 0.05	3
		1 000	N ₂ O	232.0	0.3	0.05 to 5.0	3

NOTES

- Co - When broad spectral band pass is used, a single – element hollow cathode lamp is mandatory.
- Mn - Use triplet.
- Ni - Sensitivity is greatly dependent on matrix composition. Use of a narrow spectral band pass and a single element hollow cathode lamp is mandatory.

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 concentrations, 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 celebration 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 Tolerance

The deviation between three independent determinations shall not exceed 0.2 times the element content in percent (absolute value)

4.3 Final Result

Report the arithmetical mean of acceptable determination rounded to the nearest value as shown in Table 2.

TABLE 2 ROUNDING OFF OF RESULTS (<i>Clause 4.3</i>)		
SI No.	Content	Round to the nearest percent
i)	From 0.01 to 0.1	0.01
ii)	Over 0.1 to 0.5	0.05

NOTE — If the element content is below 0.01 percent, report it as less than 0.01 percent.