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 - 2 : 1983 ‘Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 2: Determination of calcium, potassium, magnesium and sodium in contents from 0,001 to 0,02 % (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 2 - DETERMINATION OF CALCIUM, POTASSIUM, MAGNESIUM AND SODIUM IN CONTENTS FROM 0.001 TO 0.02 PERCENT (m/m)

(*First Revision*)

1 SCOPE

1.1 This standard (Part 2) specified the method to be used for the determination of calcium, potassium, magnesium and sodium contents in hardmetals within the range 0.001 to 0.02 *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.

> **NOTE** — In low concentration, the determination or these elements is very critical Every care should be taken to avoid contamination from atmosphere and reagents.

2 REAGENTS

During the analysis only reagents 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 Caesium Solution (1 ml = 1 ml of Ca)

Weigh (to the nearest 0.000 1 g) in a beaker 2.497 2 g of calcium carbonate previously dried at 110°C and cooled in a desiccator. Add 10 ml of concentrated hydrochloric acid. When dissolution is complete, transfer to 1 litre volumetric flask, dilute with water up to the mark and mix.

2.6 Standard Potassium Solution (1 ml = 1 mg of K)

Dry 3 g of potassium chloride at 110°C. Take 1.906 8 g of potassium chloride and dissolve in 100 ml of water. Transfer to 1 litre volumetric flask, dilute with water up to the mark and mix.

2.7 Standard Magnesium Solution (1 ml = 1 mg of Mg)

Weigh (to the nearest 0.000 1 g) 1 g of pure magnesium metal (purity 99.95 percent) in a 250 ml beaker. Add 25 ml dilute hydrochloric acid (1 : 1). Boil gently to dissolve the metal. Add 50 ml of dilute hydrochloric acid. Transfer to 1 litre volumetric flask, dilute with water up to the mark and mix. Store in a polyethylene bottle.

2.8 Standard Sodium Solution (1 ml = 1 mg of Na)

Weigh 3.089 1 g of anhydrous sodium sulphate previously dried at 110°C and cooled in 3 desiccators. Place it in a 250 ml beaker and add 10 ml of concentrated hydrochloric acid, transfer to 1 litre volumetric flask, dilute with water up to the mark and mix. Store in a polyethylene bottle.

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 of caesium chloride solution and 10 ml of ammonium fluoride solution. Then transfer the solution totally to a 100 ml polyethylene volumetric flask and dilute to the mark and mix.

3.3 Dilution Volume

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

3.3.1 *Dilution Volume* (100 ml = 1 g of test sample) — Use the solution **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 analyzed 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 solutions with a diluted matrix in accordance with **3.3.2** for dilution volumes of 1 000 ml, if necessary.

3.4.3 Prepare at least two blank solutions (*See* **3.4.1**) without the addition of 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 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.

TABLE 1 INSTRUMENTAL PARAMETERS AND CHARACTERISTICS OF CALIBRATION FUNTIONS

(*Clauses* 3.3 *and* 3.5)

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*
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Ca	100	N_2O	422.7	0.03	0.001 to 0.02
ii)	K	100	Air	769.9	0.04	0.000 5 to 0.02
iii)	Mg	100	N_2O	285.2	0.02	0.000 3 to 0.04
iv)	Na	100	Air	589.3	0.02	0.000 5 to 0.02

NOTES

1. For Ca - Sensitivity is greatly dependent on matrix composition.
2. For Na - Use the doublet.

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 it 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.

3.6.3 Convert the absorbance values of the test solutions corrected for the blank of 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 0.000 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 mean of acceptable determinations rounded to 0.001 *percent*. If the element content is below 0.001 *percent* or greater than 0.02 *percent*, report the result as less than 0.001 *percent* or greater than 0.02 *percent*, respectively.