

# इंटरनेट

# मानक

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IS 12483 (1988): Determination of Titanium in Hardmetals by Spectrophotometric Method [MTD 25: Powder Metallurgical Materials and Products]



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*Indian Standard*

# DETERMINATION OF TITANIUM IN HARDMETALS BY SPECTROPHOTOMETRIC METHOD

**1. Scope** — This standard covers a spectrophotometric method for the determination of the titanium content in carbides and hardmetals.

**1.1** This method is applicable to the following having titanium content exceeding 0.2 percent (*m/m*):

- a) Carbides and binder metal powder mixtures, free of lubricant; and
- b) All grades of presintered or sintered hardmetals.

**2. Principle** — Formation of a yellow complex of pertitanic acid. Determination of the absorbance of the complex.

**3. Interfering Elements** — The effect of interfering elements which also form coloured complexes with hydrogen peroxide under the conditions specified, for example vanadium and molybdenum shall be taken into account. A correction can be applied if the content is below 5 percent of each.

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

**4.1 Ammonium Hydrogen Fluoride**

**4.2 Ammonium Sulphate**

**4.3 Sodium Disulphite**

**4.4 High Purity Titanium Metal or Titanium Dioxide**

**4.5 Citric Acid Solution** — 30 percent (*m/m*).

**4.6 Perchloric Acid** —  $\rho = 1.54$  or  $1.67$  g/ml.

**4.7 Concentrated Sulphuric Acid** —  $\rho = 1.84$  g/ml.

**4.8 Dilute Sulphuric Acid** —  $\rho = 1.54$  g/ml (concentrated sulphuric acid,  $\rho = 1.84$  g/ml, diluted 1+1).

**4.9 Hydrogen Peroxide** — 30 percent (*m/m*).

**4.10 Hydrofluoric Acid** —  $\rho = 1.12$  g/ml.

**4.11 Concentrated Nitric Acid** —  $\rho = 1.42$  g/ml.

## 5. Apparatus

**5.1 Ordinary Laboratory Apparatus**

**5.2 Spectrophotometer or Filter Photometer**

**6. Sample Preparation** — The sample shall be crushed to a powder in a mortar made of a material which does not alter the sample composition. The powder shall pass through a  $180 \mu\text{m}$  sieve.

**Note** — For sintered hardmetals, the preparation of sample may be as agreed to between the purchaser and the manufacturer.

**6.1** The analysis shall be carried out on two or three test portions.

## 7. Procedure

**7.1** Select the mass of the test portion, the volume of the volumetric flask and the cell length in accordance with Table 1.

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**TABLE 1 TEST PORTION, FLASK VOLUME AND CELL LENGTH**  
( Clause 7.1 )

Titanium Content Percent ( m/m )	Test Portion Mass g	Flask Volume ml	Cell Length mm
0.2 to 4	0.2	250	20
2 to 8	0.2	250	10
5 to 15	0.2	500	10
10 to 30	0.1	500	10

**Note** — Approximately 17 mg of titanium in 250 ml of final solution gives unit absorbance with a 10 mm cell.

**7.2 Test Portion** — Weigh, to the nearest 0.0001 g, 0.1 or 0.2 g of the test sample.

**7.3 Dissolution** — Transfer the test portion into a 100 to 200 ml conical flask or a 250 ml beaker. Add 5 g ammonium sulphate and 10 ml concentrated sulphuric acid. Cover the beaker or flask with a watch glass. Heat to boiling until complete dissolution is achieved.

**7.3.1 Alternative dissolution method** — Transfer the test portion into a platinum dish. Add 10 ml of water and 5 ml of hydrofluoric acid. Cover the dish with a polypropylene or platinum cover. Heat to approximately 800°C. Add the concentrated nitric acid drop by drop until dissolution is complete. Cool, add 10 ml of concentrated sulphuric acid and 5 g of ammonium sulphate. Heat until fumes of SO<sub>3</sub> are observed, and cool again.

**7.3.2** If any undissolved particles or carbonaceous matter are left, cool to below 100°C, and cautiously add 1 ml of perchloric acid. Heat to fume off the perchloric acid, then cool to room temperature.

**CAUTION** : When using perchloric acid, avoid contact with organic matter.

**7.4 Preparation of the Solution for Analysis** — Add, in small portions, 1 ml of hydrogen peroxide, mix by shaking. Add, in small portions, 30 ml of citric acid solution while mixing. Add 40 ml of water.

**7.4.1 Using a 250 ml volumetric flask** — Transfer the solution into 250 ml volumetric flask, rinsing copiously with water. Add 25 ml of dilute sulphuric acid ( 1 : 1 ), fill nearly to the mark and mix. Cool, add 1 ml hydrogen peroxide, mix, make up to volume and mix again.

**7.4.2 Using a 500 ml volumetric flask** — Transfer the solution into 500 ml volumetric flask, rinsing copiously with water. Add 30 ml citric acid solution and 50 ml dilute sulphuric acid ( 1 : 1 ), fill nearly to the mark and mix. Cool, add 2 ml hydrogen peroxide, mix, make up to volume and mix again.

**7.5 Compensating Solution** — Transfer approximately 30 ml of the coloured solution ( 7.4 ) into a 50 ml beaker. Add approximately 0.2 g of sodium disulphite to decolourize the solution. If necessary, repeat the addition of sodium disulphite.

**7.6 Determination of Absorbance** — Choose appropriate cells. Measure the absorbance of the coloured and the compensating solutions at a wavelength of 420 nm using the same cell or a matched pair of cells.

## 8. Elimination of Interference

**8.1 Vanadium** — Transfer 30 ml of the coloured solution ( 7.4 ) into a 50 ml beaker and add approximately 0.1 g ammonium hydrogen fluoride ( 1.1 ), which destroys the coloured titanium complex. Wait for 3 min. The colour left is due to vanadium.

**8.1.1** Measure the absorbance and subtract it from the absorbance due to titanium plus vanadium.

**8.2 Molybdenum** — The colour of the molybdenum peroxide complex is weak. It cannot be compensated for chemically but correction can be calculated from the molybdenum content. 1 percent of molybdenum in the sample corresponds to approximately 0.08 percent of titanium at 420 nm, but the correction should be determined with the spectrophotometer used.

## 9. Preparation of the Calibration Curve

**9.1 Standard Titanium Solution** — The standard titanium solution can be prepared from titanium metal or titanium dioxide.

**9.1.1 Standard titanium solution from titanium metal** — Weigh approximately 250 mg of the titanium metal (4.4) and transfer it into a 200 ml conical flask. Add 30 ml water and 20 ml sulphuric acid (1 : 1). Cover the flask. Warm gently and keep the volume constant by adding water until all metal has dissolved. Cool and add approximately 1 ml hydrogen peroxide to oxidize the titanium. Evaporate the water by heating to fumes. Cool, add 10 g ammonium sulphate and heat gently until deposits on the walls of the flask have dissolved. Cool and add about 50 ml of water, transfer into a 250 ml volumetric flask and add 25 ml dilute sulphuric acid (1 : 1). Cool, make up to volume with water and mix. Calculate the titanium content in grams of titanium per litre.

**9.1.2 Standard titanium solution from titanium dioxide** — Weigh a quantity of the titanium dioxide calculated to contain approximately 250 mg of titanium and transfer it into a 200 ml conical flask. Add 10 g ammonium sulphate and 10 ml concentrated sulphuric acid. Cover the flask. Heat to near boiling until a clear solution is obtained. Cool, add, in small portions, 1 ml hydrogen peroxide while mixing by shaking. Add about 50 ml water, transfer into a 250 ml volumetric flask and add 25 ml dilute sulphuric acid (1 : 1). Cool, make up to volume with water and mix. Calculate the titanium content in grams of titanium per litre.

**9.2 Calibration** — Determine the slope of the absorbance-titanium curve, that is the absorbance for 1.0 mg of titanium per 250 ml of solution, for example, as follows:

Into four conical flasks, transfer 5 g ammonium sulphate and 10 ml concentrated sulphuric acid. Add separately to three of the flasks, 5.0, 10.0 and 15.0 ml standard titanium solution. Use the remaining flask for the blank test. Continue as in 7.4 to 7.6.

## 10. Test Results

**10.1 Calculation** — The titanium content, expressed as a percentage by mass, is given by the formula

$$\text{Titanium content (percent by mass)} = \frac{100 \times A \times V}{25 \times a \times m \times L}$$

where

$A$  = absorbance from the titanium in the hardmetal;

$a$  = absorbance when the concentration is 1 mg of titanium per 250 ml with a 10 mm cell;

$m$  = mass, in mg, of the test portion;

$V$  = volume, in ml, of the coloured solution; and

$L$  = cell length, in mm.

**10.1.1** The result shall be rounded to the nearest 0.01 percent.

**10.2 Tolerances** — The deviations between two or three independent determinations shall not exceed the values shown in Table 2.

TABLE 2 PERMISSIBLE DEVIATIONS

Titanium Content Percent	Range for Two Determinations Percent	Range for Three Determinations Percent
From 0.2 to 5	0.10	0.12
Over 5 to 8	0.15	0.18
Over 8 to 15	0.20	0.25
Over 15	0.30	0.35

**10.3 Final Results** — Report the arithmetical mean of acceptable determinations rounded to the nearest 0.1 percent:

- Reference to this standard,
- All details necessary for identification of the test sample,
- Result obtained, and
- Details of any occurrence which may have affected the result.

## EXPLANATORY NOTE

In the preparation of this standard, assistance has been derived from ISO 4501-1978 'Hardmetals — Determination of titanium — Photometric peroxide method', issued by the International Organization for Standardization (ISO).