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#### **BUREAU OF INDIAN STANDARDS**

#### **Draft Revision**

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#### IS 3863 : METHODS OF CHEMICAL ANALYSIS OF COPPER-TELLURIUM ALLOYS

(First Revision)

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# भारतीय मानक प्रारूप

# ताँबा-टेलुरियम मिश्रधातुओं के रासायनिक विश्लेषण की पद्धतियाँ

( पहला पुनरीक्षण )

Draft Indian Standard

# METHODS OF CHEMICAL ANALYSIS OF COPPER-TELLURIUM ALLOYS

(First Revision)

ICS 77.120.30

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**Price Group** 

#### Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

#### FOREWORD

This draft Indian Standard (First Revision) subject to its finalization, is to be adopted by the Bureau of Indian Standards on recommendation of the Methods of Chemical analysis of Metals Sectional Committee and approval of the Metallurgical Engineering Division Council.

This standard was first published in 1966. Copper-tellurium alloys are used for the manufacture of nozzles for welding torches. Need was felt to prescribe standard methods of analysis which may be used to determine correctly the copper and tellurium contents of these alloys. While preparing this standard, views of producers, consumers and testing authorities relating to the technological methods followed in the country in this field, were taken into account.

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

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. 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

# METHODS OF CHEMICAL ANALYSIS OF COPPER-TELLURIUM ALLOYS

(First Revision)

### **1 SCOPE**

This standard prescribes methods for chemical analysis of copper-tellurium alloys in which tellurium content may vary between 0.5 to 0.8 percent.

#### **2 REFERENCE**

The following Indian Standards contain provisions, which through reference in this text, constitute provision of this standard. At the time of the publication, the editions indicated below were valid. All the standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards indicated below:

IS No.	Title
IS 264 : 2005	Nitric acid – Specification (third revision)
IS 266 : 1993	Sulphuric acid – Specification (third revision)
IS 1070 : 1992	Reagent grade water – Specification (third revision)
IS 1817 : 1961	Methods of sampling non-ferrous metals for chemical analysis

# **3 SAMPLING**

For the purpose of chemical analysis, samples shall be drawn and prepared in accordance with the procedure laid down in IS 1817.

#### **4** Quality of reagents

Unless otherwise specified, pure chemicals and distilled water (see IS 1070) shall be employed in the test.

NOTE – 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

# **5 DETERMINATION OF COPPER BY THE ELECTROLYTIC METHOD**

#### 5.1 Outline of the Method

Copper is deposited electrolytically on a platinum cathode. In order to prevent deposition of tellurium, manganese nitrate is added to the electrolyte before electrolysis.

#### **5.2 Apparatus**

The following platinum electrodes (*see* Fig. 1) are recommended but strict adherence to the shape and size of the electrodes is not essential.

#### 5.2.1 Cathode



It may be formed either from plain or perforated sheet or from wire gauze.

1A Cylindrical Platinum Cathode

1B Spiral Platinum Anode

All dimensions in millimetres



**5.2.1.1** Gauze cathodes preferably made from gauze containing  $400 \text{ mesh/cm}^2$  should be used. The wire used for making gauze should be approximately 0.20 mm in diameter. Cathode should be stiffened by doubling the gauze for about 3 mm on the top and the bottom or by reinforcing the gauze at the top and bottom with platinum ring or band.

**5.2.1.2** The diameter of the cylinder should be approximately 30 mm and the height 50 mm. The stem should be approximately 1.5 mm. It should be flattened and welded along the entire length of the gauze. The overall height of the cathode should be approximately 130 mm.

# **5.2.2** Anode

Either a spiral or a gauze anode should be used. The spiral of seven turns with a height of approximately 50 mm and diameter of 12 mm should he made from 1.0 mm or larger platinum wire, the, overall height being 130 mm. The gauze anode should be made of the same material and of the same general design as platinum gauze cathode mentioned under **5.2.1**. The diameter of the cylinder should be approximately 12 mm and the height 50 mm, the overall height of the anode being 130 mm.

# **5.3 Reagents**

5.3.1 Sulphuric Acid-Nitric Acid Solution

Add slowly, while stirring, 300 ml of concentrated sulphuric acid (sp gr 1.84; conforming to IS 266) to 750 ml of water. Cool and add 210 ml of concentrated nitric acid (sp gr 1.42; conforming to IS 264).

5.3.2 Potassium Permanganate Solution – 20 g/l.

Dissolve 2 g of potassium permanganate in water and dilute to 100 ml.

**5.3.3** *Manganese Nitrate Solution* – 20 g/l.

Dissolve 2 g of manganese nitrate in one litre of water.

**5.3.4** *Urea* – solid.

**5.3.5** *Sulphamic Acid* – solid.

**5.3.6** *Ethanol or Methanol* – 95 percent (v/v).

# **5.4 Procedure**

**5.4.1** Transfer 2.00 g of the sample to an electrolysis beaker. Add 30 ml of the sulphuric acid - nitric acid solution, cover, and allow to stand for a few minutes until reaction has nearly ceased. Heat the solution for complete dissolution of the sample and to expel the brown fumes of nitric acid. Wash down the cover glass and sides of the beaker, and dilute to 200 ml. Add a few drops of permanganate solution till faint permanent pink solution is obtained. Add 5 ml of manganese nitrate solution.

**5.4.2** Add 1 g of urea or 0.1 g of sulphamic acid and boil to destroy any trace of nitrous acid present. Cool, insert the electrodes; the cathode having been accurately weighed. Cover with a pair of split watch-glasses and electrolyze for 16 hours at a current density of 0.6 A/dm<sup>2</sup>. When the solution becomes colourless reduce the current density to 0.3 A/dm<sup>2</sup>. Continue electrolysis until the deposition of copper is complete as indicated by absence of plating on the new surface of the electrode obtained by the raised level of the solution.

**5.4.3** Without interrupting the current, syphon off the electrolyte, at the same time adding water to maintain the level of the solution. Remove the cathode quickly, while washing with water; rinse it with water in a beaker and then dip it in two successive baths of ethanol or methanol. Dry the cathode in an air-oven at 110  $^{\circ}$ C for 3 to 5 minutes, cool and weigh for copper.

# 5.5 Calculation

Copper, percent = 
$$\frac{A}{B} \times 100$$

where

A = weight in g of copper deposited on the cathode, and

B = weight in g of the sample taken.

# **6 DETERMINATION OF TELLURIUM BY THE DICHROMATE METHOD**

# 6.1 Outline of the Method

Tellurium is precipitated from solution of the sample by sodium hypophosphite. Traces of copper co-precipitated with tellurium are removed by electrolysis and tellurium estimated by titration with standard potassium dichromate solution.

# 6.2 Reagents

- 6.2.1 Concentrated Sulphuric Acid sp gr 1.84 (conforming to IS 266).
- 6.2.2 Concentrated Nitric Acid sp gr 1.42 (conforming to IS 264).
- **6.2.3** Sodium Hypophosphite Solution 20 percent (w/v).
- **6.2.4** *Dilute Nitric Acid* 1 : 1 (*v*/*v*).
- 6.2.5 Potassium Permanganate Solution see 5.3.2.
- 6.2.6 Manganese Nitrate Solution see 5.3.3.
- **6.2.7** *Standard Potassium Dichromate Solution* 0.1 N.

Dry pure potassium dichromate in an oven at 105 °C to 110 °C for one and a half hours. Cool in a desiccator and transfer exactly 4.903 5 g to a one-litre volumetric flask carefully through a funnel using water at room temperature. Wash the funnel thoroughly and dissolve the salt by shaking until the solution is complete. Make up to the mark and mix well.

6.2.8 Ferrous Ammonium Sulphate Solution (0.1 N)

Dissolve 39.2 g of [ Fe (NH<sub>4</sub>)<sub>2</sub> (SO<sub>4</sub>)<sub>2</sub>, (6H<sub>2</sub>O) ] in 500 ml of cold dilute sulphuric acid (5:95), and dilute to one litre with dilute sulphuric acid (5:95). Standardize the solution with standard potassium dichromate solution (*see* **6.2.7**).

6.2.9 Phosphoric Acid – 85 percent.

# 6.2.10 Sodium Diphenylamine Sulphonate Indicator Solution

Dissolve 0.20 g of sodium diphenylamine sulphonate in 100 ml water and store the solution in a dark-coloured dropping bottle.

# 6.3 Procedure

**6.3.1** Transfer 5.00 g of the sample to a 250-ml beaker. To this add 50 ml of concentrated nitric acid, cover the beaker and allow to stand for a few minutes. Warm the solution for complete dissolution of the sample.

**6.3.2** Add 10 ml of concentrated sulphuric acid to the solution and then evaporate the solution to fumes. Cool and dilute the solution with 100 ml of water when a clear solution is obtained.

**6.3.3** To the warm solution, add 50 ml of sodium hypophosphite solution and boil to precipitate tellurium. Allow the precipitate to settle and filter through filter paper or sintered Gooch crucible. Wash the residue with hot water.

**6.3.4** Slowly dissolve the precipitate in 50 ml of hot dilute nitric acid and collect the solution in a beaker. Wash the filter paper or the crucible with hot water. Add 5 ml of concentrated sulphuric acid, evaporate the solution to fumes and then cool the solution. To this add 200 ml of water and boil till a clear solution is obtained. Cool the solution and add a few drops of permanganate solution

till the colour of the solution is pink. Add 5 ml of manganese nitrate solution and 7 ml of concentrated nitric acid. Boil the solution and cool.

**6.3.5** Remove the last traces of copper by electrolysis as under **5.4.2** and evaporate the solution to fumes. Cool and dilute the solution to 100 ml. Warm the solution and reprecipitate tellurium with sodium hypophosphite as under **6.3.3**. Filter off the residue and wash with water.

**6.3.6** Dissolve the precipitate in dilute nitric acid and evaporate to dryness. To this add 20 ml of concentrated sulphuric acid and evaporate to fumes, cool. Repeat the process of fuming as before to remove last traces of nitric acid. Dilute to 200 ml.

**6.3.7** Add 25 ml of standard potassium dichromate solution to the above solution, allow to stand for 30 minutes and add a measured excess of ferrous sulphate solution and add 5 ml of phosphoric acid. Stir and titrate the excess ferrous ammonium sulphate solution with standard dichromate solution using sodium diphenylamine sulphonate indicator solution.

# 6.4 Calculation

Tellurium, percent = 
$$\frac{(A - B) \times C \times 6.38}{D}$$

where

A = volume in ml of the standard dichromate solution added in 6.3.7,

B = volume in ml of the equivalent ferrous sulphate solution consumed by the excess dichromate solution,

C = normality of the standard dichromate solution, and

D = weight in g of the sample taken.