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Draft Indian Standard

#### METHODS OF CHEMICAL ANALYSIS OF SILVER-COPPER BRAZING ALLOYS

#### PART 1 ANALYSIS FOR SILVER AND COPPER

[First Revision of IS 4667 (Part 1)]

ICS 77.120.30

Methods of Chemical Analysis of Metals	Last date of comments
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#### FOREWORD

#### (Formal foreword clause will be added later)

This standard was first published in 1968. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

In IS 2927 : 1975, 22 grades of silver-copper brazing alloys were specified. In order to determine correctly the composition of different silver-copper brazing alloys, standard methods of analysis of elements specified in these grades have been described in various parts of this standard. This standard (Part 1) covers analysis of silver and copper in silver-copper brazing alloys. The analysis of silver and copper in presence of tin in Grades BA-CuAg7 and BA-CuAg8 have been covered in Part 2.

The other parts in the series are:

- Part 2 Determination of silver, copper and tin
- Part 3 Determination of nickel
- Part 4 Determination of silver, copper, cadmium and zinc Electolytic method

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

# Draft Indian Standard

# METHODS OF CHEMICAL ANALYSIS OF SILVER-COPPER BRAZING ALLOYS

### PART 1 ANALYSIS FOR SILVER AND COPPER

## (First Revision)

### **1 SCOPE**

This standard (Part 1) prescribes methods of chemical analysis for silver and copper in silvercopper brazing alloys (excluding Grades BA-CuAg7 and BA-CuAg8) as specified in IS 2927.

### 2 REFERENCES

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

IS No.	Title
IS 264 : 2005	Nitric acid — Specification (third revision)
IS 266 : 1993	Sulphuric acid — Specification ( <i>third revision</i> )
IS 1070 : 2023	Reagent grade water — Specification (fourth revision)
IS 1817 : 1961	Methods of sampling non-ferrous metals for chemical analysis
IS 2927 : 1975	Specification for brazing alloys (first revision)

### **3 SAMPLING**

### **3.1 Laboratory Sample**

It shall be drawn and prepared in accordance with IS 1817.

### **4 QUALITY OF REAGENTS**

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

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

### **5 DETERMINATION OF SILVER BY THE GRAVIMETRIC METHOD**

### **5.1 Outline of the Method**

The sample is dissolved in nitric acid and silver is precipitated as silver chloride by means of hydrochloric acid. The precipitate is filtered, washed, dried and weighed.

## 5.2 Reagents

**5.2.1** *Dilute Nitric Acid* — 1 : 1 and 3 : 97 (*v*/*v*).

**5.2.2** *Dilute Hydrochloric Acid* — 1 : 9 (*v*/*v*).

# **5.3 Procedure**

**5.3.1** Transfer 2 g of an accurately weighed sample to a 250 ml beaker. Add 25 ml of dilute nitric acid (1 : 1). Heat gently to dissolve and boil to remove the brown fumes. Cool to room temperature. Transfer to a 250 ml volumetric flask and make up to the volume. Take a suitable aliquot.

**5.3.2** Add 25 ml of dilute hydrochloric acid slowly with constant stirring. Let stand for about an hour.

**5.3.3** Decant the solution through a weighed sintered glass crucible. Wash the precipitate with warm dilute nitric acid (3:97) till free from chloride and decant through the crucible. Transfer the filtrate and washings to a 400 ml beaker and pressure for the determination of copper.

**5.3.4** Dry the crucible and precipitate at about 110 °C. Cool in a desiccator to room temperature and reweigh.

# 5.4 Calculation

Silver, percent = 
$$\frac{A}{B} \times 75.26$$

where

A = weight in g of silver chloride; and

B = weight in g of the sample represented by the aliquot taken.

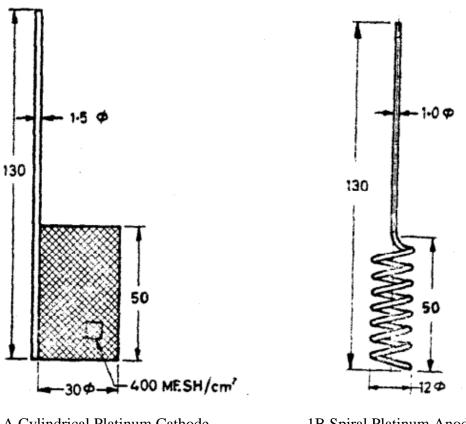
# **6 DETERMINATION OF COPPER BY THE ELECTROLYTIC METHOD**

# 6.1 Outline of the Method

After removal of silver, copper in the filtrate is deposited electrolytically and weighed.

# 6.2 Apparatus

The platinum electrodes mentioned in **6.2.1** and **6.2.2** (*see* Fig. 1) are recommended but strict adherence to the shape and size of the electrodes is not essential. In order to decrease the time of deposition, one of the types of rotating forms of electrodes generally available for agitation of electrolyte may be employed.



1A Cylindrical Platinum Cathode

**1B Spiral Platinum Anode** 

All dimensions in millimetres.



### **6.2.1** *Cathode*

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

**6.2.1.1** Gauze cathodes made preferably from gauze containing 400 mesh/cm<sup>2</sup> should be used. The wire used for making gauze should be approximately 0.20 mm in diameter. Cathodes should be stiffened by doubling the gauze for about 3 mm on the top and bottom or by reinforcing the gauze at the top and bottom with a platinum ring or brand.

6.2.1.2 The diameter and height of the cylinder should be approximately 30 mm and 50 mm respectively. The stem should be made from platinum alloy wire, such as platinum-iridium, platinum-rhodium or platinum-ruthenium having diameter of approximately 1.5 mm. It should be flattened and welded to the entire height of the gauze. The overall height of the cathode including the stem should approximately be 130 mm.

### 6.2.2 Anode

Either a spiral or a gauze anode should be used. The spiral anode should be made from 1.0 mm or larger platinum wire formed into a spiral of seven coils with a height of approximately 50 mm and diameter of 12 mm, the overall height including the stem being 130 mm. The gauze anode should

be made of the same material and of the same general design as platinum gauze cathode specified under.

### 6.3 Reagents

6.3.1 Concentrated Sulphuric Acid — specific gravity 1.84 (conforming to IS 266).

6.3.2 Concentrated Nitric Acid — specific gravity 1.42 (conforming to IS 264).

**6.3.3** *Urea* — solid.

**6.3.4** *Ethanol or Methanol* — 95 percent (v/v).

## 6.4 Procedure

**6.4.1** To the filtrate and washings preserved under **5.3.3**, add 5 ml of concentrated sulphuric acid and evaporate to fumes. Cool, dilute to 250 ml and add 10 ml of concentrated nitric acid.

**6.4.2** Weigh the cathode, adjust the electrodes in the solution, and cover with a pair of split watchglasses. Add 2 g of urea. Electrolyze at a current density of  $0.6 \text{ A/dm}^2$  (at this current density, the electrolysis is conveniently carried on overnight), or at a current density of 4 A/dm<sup>2</sup> for a short period (about 2.5 hours). In the latter case, one of the types of rotating forms of electrodes generally available may be used. When the solution becomes colourless reduce the current density to 0.3 A/dm<sup>2</sup> and continue electrolysis until the deposition of copper is complete as indicated by failure of copper to plate on the newly exposed cathode surface when the solution level is raised.

**6.4.3** Without interrupting the current, lower the beaker slowly while rinsing the electrodes with water and collecting the washing in the electrolyte. Remove the cathode quickly, 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 three to five minutes, cool and reweigh the cathode. The difference in weight gives the weight of copper deposited.

### 6.5 Calculation

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

where

A = weight in g of copper deposited; and

B = weight in g of the sample taken.