IS 4548-1 (1967): Methods of chemical analysis of copper gold brazing alloys, Part 1: Analysis of gold and copper [MTD 8: Copper and Copper Alloys]

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER-GOLD BRAZING ALLOYS

PART I ANALYSIS OF GOLD AND COPPER

(First Reprint DECEMBER 1989)

UDC 621.791.36.04:669.35'21:543

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *May* 1968

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER-GOLD BRAZING ALLOYS

PART I ANALYSIS OF GOLD AND COPPER

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER-GOLD BRAZING ALLOYS

PART I ANALYSIS OF GOLD AND COPPER

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 November 1967, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Two grades of copper-gold brazing alloys have been specified in IS : 2927-1964*. The purpose of this standard is to prescribe methods of analysis of gold and copper which are the main elements present in the alloy. For the analysis of zinc and cadmium which have been restricted to 0·005 percent, investigation is called for to develop standard methods of analysis. These methods when finalized will be covered in Part II of this standard.

0.3 In the formulation of this standard due weightage has been given to the practices followed in the country in this field.

0.4 In reporting the result 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 : 2-1960†.

1. SCOPE

1.1 This standard covers the methods of chemical analysis of gold and copper in copper-gold brazing alloys as specified in IS : 2927-1964*.

2. QUALITY OF REAGENTS

2.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960‡) shall be employed in the tests.

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

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*Specification for brazing alloys.

†Rules for rounding off numerical values (*revised*).

‡Specification for water, distilled quality (*revised*).

3. DETERMINATION OF GOLD BY THE CUPELLATION METHOD

3.1 Outline of the Method — After the addition of definite proportion of silver, the sample is cupelled along with sufficient amount of lead to remove copper and zinc. The resulting gold-silver button is flattened, parted and gold cornet weighed.

3.2 Apparatus

3.2.1 *Cupellation Furnace* — It is a muffle type furnace, the muffle shall be provided with inlets and outlets for air so that a current of air, preferably preheated, may be constantly passing through it. The muffle shall be capable of being heated uniformly up to a temperature of 1 100°C.

3.2.2 *Assay Balance* — of the highest precision, capable of carrying one or two grams in each pan and sensitive to 0·01 mg. The balance should be so adjusted that one division of the scale traversed by the pointer corresponds to 0·05 mg.

3.2.3 *Gold Assay Weights* — shall be of the highest accuracy and preferably made of platinum-iridium. Figures shall be stamped on the weights to denote the number of half-milligrams or 'milliemes' contained in the weight, the 0·5 g weight shall be stamped '1 000'.

3.2.4 *Cupels* — made of bone-ash or magnesia.

3.2.5 *Rolling-Mill* — a small jewellers' rolling-mill.

3.2.6 *Balling Pliers* — consisting of a pair of steel pliers with smooth concave holes, the diameter of the cavity in the chops being about 10 mm.

3.2.7 *Parting Tray* — consisting of a number of small thimble like, perforated cups of platinum or fused silica supported in a frame of the same material.

3.2.8 *Hammer and Anvil* — with faces kept bright and clean.

3.2.9 *Trays* — for various forms of assay pieces, buttons, rolls, fillets, cornets, etc. Trays shall be of different kinds for holding the assay pieces according to different stages of operation.

3.2.10 *Tongs and Forceps* — of various forms, for charging the cupels and handling assay pieces, etc.

3.3 Reagents

3.3.1 *Check Gold* — The fineness of check gold shall be 999·9.

3.3.2 *Silver* — The metallic element silver free from any other element.

3.3.3 *Lead Foil* — commercial quality.

3.3.4 *Parting Acid No. 1* — consisting of dilute nitric acid (sp gr 1·2) containing a trace of silver nitrate.

3.3.5 *Parting Acid No. 2* — consisting of dilute nitric acid (sp gr 1·3) containing a trace of silver nitrate.

3.4 Procedure

3.4.1 *Weighing of Sample*

3.4.1.1 Weigh accurately 1 000 milliemes (0·5 g) of the sample within ± 0·3 milliemes. In case of alloys containing 58 percent gold, it is advisable to add 0·2 g of an accurately weighed check gold to the sample; the weight of the check gold added should be deducted-from the final weight of the cornet.

3.4.1.2 It is always preferable to make duplicate assay.

3.4.2 *Preparation of the Assay-Piece*

3.4.2.1 In making up the assay-piece, add enough silver to the sample so as to make up the total silver content between 2 to 2·5 times the amount of gold present in the sample.

3.4.2.2 Wrap the weighed sample together with the required amount of silver in a lead foil weighing 8 g and squeeze them in the balling pliers to form small ball. In the case of alloys containing about 58 percent and about 37 percent gold, an extra amount of two grams and 4 grams respectively of lead shall be added towards the end of cupellation. The assay-piece shall now be considered ready for charging in the cupellation furnace.

3.4.3 *Preliminary Assay* — If the approximate content of gold in the sample is not known, it is necessary to make a preliminary assay. For this purpose, weigh 100 mg of the alloys, add 300 mg of silver and then wrap it in three grams of lead. Cupel it and flatten the button. Boil in 15 ml of parting acid No. 2 for 15 minutes, wash the resulting gold, dry and heat to redness, and weigh. The weight in milligrams gives directly the percentage of gold.

3.4.4 *Cupellation* — Arrange the cupels carefully in the muffle, preferably on a removable tray. When the cupels have attained the furnace temperature of about 1 000°C, place in them the assay pieces prepared under **3.4.2.2**, each in its proper cupel, by means of a pair of long cupellation tongs. The charging-in should be done carefully, but as rapidly as possible, so as not to cool the muffle unduly; close muffle door and allow the cupellation to proceed for 20 to 30 minutes depending on the amount of lead used, the temperature being raised to about

1 050°C towards the end. The end of the cupellation is shown by the appearance of bright globules of gold-silver alloy. The cupels may be withdrawn while the metal is still molten or may be cooled in the furnace until the buttons are solidified and then removed from the furnace.

3.4.5 *Preparation of the Assay-Piece for Parting* — Remove the buttons from the cupels by means of a pair of forceps and clean with a stiff brush. Flatten the buttons on a polished anvil with a polished hammer. Anneal the flattened buttons at about 800°C and pass in succession through the rolls to form elongated fillets of thickness 0·25 mm. After being rolled, anneal again to soften them and then separately roll up between the finger and thumb into a ‘ cornet ’ or spiral.

3.4.6 *Parting and Annealing the Cornets* — Place the cornets in the respective cups of the parting tray and immerse the entire tray in boiling parting acid No. 1 and boil for 10 to 15 minutes. Take out the tray and drain from acid liquor, wash by dipping in hot water, drain again, and immerse in a second lot of boiling parting acid No. 2. Boil the cornets in this way for 15 to 20 minutes, drain and wash thoroughly. Dry the tray with the cornets by gently heating and then place in a muffle heated to about 800°C. The cornets, which before annealing are of a dull-brown colour and friable, now assume a bright gold-yellow colour, diminished in size and hardened. The cornets, when cold, are ready for weighing.

3.4.7 *Check-Assays*

3.4.7.1 Certain losses and gains are incurred in the various operations of assay mainly due to losses of gold and retention of silver by cornet. The net sum of these losses and gains is called ‘ surcharge ’. The surcharge shall be determined by means of check-assays or checks of similar composition as sample or samples being assayed. The checks shall be made according to the known composition of the sample or from that obtained by preliminary assay according to **3.4.3**, if the approximate composition of the sample is not known. Check-gold shall always be used for the preparation of the checks and taken as the standard against which all assays shall be compared.

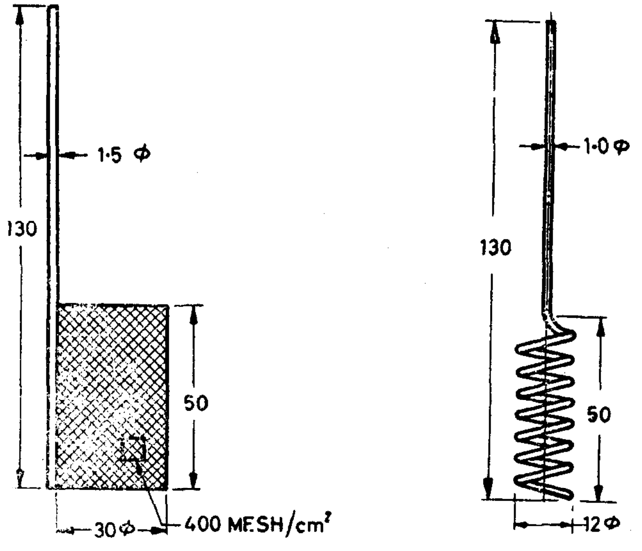
3.4.7.2 The checks shall be subjected to the same operations side by side and under identical conditions with the assay-pieces. The number of checks shall be not less than two for each group of assays and shall be positioned even in the group.

3.4.8 Weigh the cornets accurately in the assay balance in terms of milliemes and apply the surcharge as determined under **3.4.7** to the weight obtained. Record the mean value of the duplicate test results, provided the difference is not more than 0·4 milliemes. If the duplicate results do not show this agreement, repeat the test in duplicate and report the mean value of four results provided that the maximum difference between any two of them is not more than 0·8 milliemes.

4. DETERMINATION OF COPPER BY THE ELECTROLYTIC METHOD

4.1 Outline of the Method — From the solution of the sample, copper is deposited electrolytically on cathode and weighed.

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



1A CYLINDRICAL PLATINUM CATHODE

1B SPIRAL PLATINUM ANODE

All dimensions in millimetres.

FIG. 1 CYLINDRICAL PLATINUM CATHODE AND SPIRAL PLATINUM ANODE

4.2.1 *Cathode* — It may be formed either from plain or perforated sheet or wire gauze.

4.2.1.1 Gauze cathodes preferably made from gauze containing 400 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.

4.2.1.2 The diameter of the cylinder should be approximately 30 mm and the height 50 mm. 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 along the entire length of the gauze. The overall height of the cathode should be approximately 130 mm.

4.2.2 *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 be 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 **4.2.1**. The diameter of the cylinder should be approximately 12 mm and the height 50 mm, the overall height of the anode being 130 mm.

4.3 Reagents

4.3.1 *Aqua Regia* — Mix concentrated hydrochloric acid and concentrated nitric acid in the ratio of 3 : 1.

4.3.2 *Concentrated Hydrochloric Acid* — sp gr 1·16 (conforming to IS : 265-1962*).

4.3.3 *Sulphur Dioxide Solution* — saturated.

4.3.4 *Concentrated Sulphuric Acid* — sp gr 1·84 (conforming to IS : 266-1961†).

4.3.5 *Concentrated Nitric Acid* — sp gr 1·42 (conforming to IS : 264-1950‡).

4.3.6 *Dilute Nitric Acid* — 1 : 1 (*v/v*).

4.3.7 *Urea* — solid.

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

4.4 Procedure

4.4.1 Transfer 0·5 g of an accurately weighed sample rolled and cut into small pieces into a 250-ml beaker. Add 30 ml of aqua regia and after vigorous reaction is over, heat on a hot plate until the alloy is decomposed. Evaporate the solution to a small volume but do not dry. Add 20 ml of concentrated hydrochloric acid and evaporate to syrupy

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*Specification for hydrochloric acid (*revised*).

†Specification for sulphuric acid (*revised*).

‡Specification for nitric acid.

consistency. Repeat evaporation with concentrated hydrochloric acid twice more using 20 ml of concentrated hydrochloric acid each time. Dilute to 250 to 500 ml and heat nearly to boiling. To the hot solution, add with stirring sulphur dioxide solution in slight excess and digest on a steam bath for two hours. Add a further 10 ml of sulphur dioxide solution or sufficient amount to provide a strong odour of sulphur dioxide. Let aside to cool.

4.4.2 Filter the precipitate through a sintered crucible and wash with hot water containing a little concentrated hydrochloric acid, until free from all soluble salts. Add five millilitres of concentrated sulphuric acid to the filtrate and washings and evaporate to strong fumes.

4.4.3 Take up the residue with dilute nitric acid and boil to expel brown fumes. Cool, dilute to 200 ml. Add 0·5 g of urea and boil for few minutes. Insert the electrodes, cover the beaker with split watch glasses. Electrolyse with a current of 5 A/dm^2 with constant stirring. When the solution is colourless, wash down the cover glasses, electrodes and sides of the beaker, raising the level of liquid slightly. 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.

4.4.4 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°C for 3 to 5 minutes, cool and weigh for copper.

4.5 Calculation

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

where

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

B = weight in g of the sample taken.

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