BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

DETERMINATION OF GOLD CONTENT IN GOLD BULLION, GOLD ALLOYS AND GOLD JEWELLERY/ARTEFACTS — BY CUPELLATION (FIRE ASSAY) METHOD

[Fourth Revision of IS 1418]

ICS 39.060

Precious Metals Sectional Committee, MTD 10

Last date of comments

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FOREWORD

(Formal clauses of the foreword will be added later.)

This standard was first published in1962 and was subsequently revised in 1972, 1999 and 2009. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

In addition, following significant changes have been made:

- a) Extension the scope covers determination of gold content in multiple types of gold and gold bearing alloys including those used in jewellery/artefacts manufacturing;
- b) Purity of proof sample has been revised in clause 6.3;
- c) Specific procedure is described in section clause **9.8.3** for samples with large amount of base metals, containing platinum or palladium, or with silver/gold ratio higher than 3;
- d) Calculations have been adapted to take into account the addition of fine gold and the fineness of the gold used in the proof;
- e) Repeatability requirements have been changed; and
- f) The use of scorification has been removed.

For the purpose of deciding whether a 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 (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of specified value in the standard.

Draft Indian Standard DETERMINATION OF GOLD CONTENT IN GOLD BULLION, GOLD ALLOYS AND GOLD JEWELLERY/ARTEFACTS — BY CUPELLATION (FIRE ASSAY) METHOD (Fourth Revision)

1 SCOPE

1.1 This standard prescribes the cupellation method (fire assay) for the determination of gold content on a material considered homogeneous. The gold content of the sample shall lie between 100 and 999.9 parts per thousand (‰) by weight.

1.2 For fineness of gold above 999.5 (‰), a spectroscopic method by difference IS 16901 can be used and is a preferred optional method where trace element requirement is specified.

1.3 The procedure is applicable to most types of gold samples. Some modifications are indicated for specific cases (presence of large number of base metals, platinum or palladium, silver). It is not compatible with the presence of Ir, Ru, Rh, Os exceeding 0.25% for the sum of all three elements.

1.4 This method is also intended to be used as the recommended method for the determination of fineness in jewellery alloys covered by IS 1417.

NOTES

1 This standard does not purport to address safety concerns, if any, essential with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For few specific safety hazards (*see* **8**).

2 The presence of the following elements may cause difficulties in obtaining homogeneous sample, Iron, lead, antimony, arsenic, iridium or ruthenium and strontium. The presences of such elements affect the accuracy and precision of assay results.

2 REFERENCES

The following standards contain provisions which, through references in the text, constitute provisions of this standard. At the time of publication, the edition 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 standard indicated below:

IS No.	Title
IS 1070 : 1992	Reagent grade water — Specification (third revision)
IS 1417 : 2016	Gold and gold alloys, jewellery/artefacts — Fineness and marking — Specification (<i>fourth revision</i>)

IS 2790 : 1999	Guidelines for manufacture of 23, 22, 21, 18, 14 and 9 carat gold alloys (<i>second revision</i>)
IS 15820 : 2009	General requirements for competence of assaying and hallmarking centres
IS 16901 : 2022/ ISO 15093 : 2020	Jewellery and precious metals — Determination of high purity gold, platinum and palladium — Difference method using ICP-OES (<i>first revision</i>)
IS 17278 : 2019	Refined gold and silver bars for good delivery — Specification

3 TERMINOLOGY / DEFINITIONS

3.1 Bullion — Precious metals in the form of bars, ingots, plates, etc, (refined or unrefined) in various shapes and sizes containing gold and silver and variable amounts of one or more of the base metals.

3.2 Cupellation — An oxidizing fusion of lead, gold, silver and associated base metals in a cupel which absorbs the lead along with base metals oxides leaving a dore bead of gold and silver (along with any insoluble platinum group alloy if present) on the cupel.

3.3 Cornet — The alloy of gold and silver after it has been hammered / pressed in to flat shape, annealed and fashioned in to a roll, prior to parting.

3.4 Inquartation — The addition of silver to an assay sample to facilitate parting.

3.5 Parting — The selective separation of silver and other soluble base metals from gold by dissolution in an acid, usually nitric acid, in a chlorine free environment.

3.6 Gold Cornet — A gold shaped in a roll that has been parted.

3.7 Proof Sample — A synthetic reference sample having a chemical composition similar to the test sample included in every batch of assay in order to monitor the effect of prevailing assay conditions in an assay furnace.

3.8 Proof Correction — Analyzing the proof sample concurrently with the test samples and using the results to correct the final assay.

3.9 Test Portion — The portion of the metal taken from the test sample. The portion on which the test or assay is carried out.

3.10 Test Sample — A sample prepared from laboratory sample and from which test portion will be taken.

4 PRINCIPLE

An accurately weighed test portion is inquarted with silver, compounded with lead and cupelled in a cupellation furnace until a precious metal button/bead is obtained. The resultant bead of gold alloy is hammered, flattened, cleaned, rolled in to a cornet and then parted in nitric acid. The resultant gold cornet is washed, dried, annealed and weighed. Possible systematic errors are in the procedure are eliminated by assaying standard proof samples in parallel.

NOTES

1 White gold alloys containing Platinum and/or Nickel as well as alloys with 990 or more parts per thousand (‰) of gold require some procedural changes (*see* **10.2**)

2 If the bullion contains any of the platinum group metals, the accuracy and precision cannot be achieved.

5 SAMPLING

5.1 The Following Methods of Sampling may be used Depending upon form of Gold

- a) Vacuum tube sampling;
- b) Dip sampling;
- c) Sampling by cutting or scrapping (not suitable for fine gold samples / suitable for jewellery artefacts); and
- d) Sampling by drilling.

5.2 Vacuum Tube Sampling of Molten Alloy

An evacuated, sealed glass tube having a thin-walled bulb section near to one end is plunged into a crucible filled with molten metal. The thin-walled bulb breaks upon contact with the molten metal, which is then drawn into the tube due to vacuum effect. After cooling, the glass is broken and the pin sample removed from the tube and inspected to ensure that it is satisfactory for assay.

NOTE — Samples containing slag or inclusions and piping are the cause of unreliable assay.

5.3 Dip Sampling of Molten Alloy

A preheated graphite sampler is dipped in to the molten metal and a sample of about 5 gm is drawn from middle of the pot just before casting the ingot. It is cleaned with a brush, hammered, rolled in to a strip and cut in to small pieces to prepare an assay sample.

5.3.1 When the bullion/jewellery is melted, it should be stirred well with a preheated graphite stirrer rod and a dip sample of about 5 gm taken with a preheated graphite sampler from the middle of the pot containing molten metal before it is cast in to a bar. The sample button be allowed to solidify and cooled in the sampler before being taken for further process. The sample may also be taken by granulation. The granule may be hammered, if necessary, to remove any trapped water. The sample so produced is cleaned, rolled and cut in to small piece for assaying.

5.4 Sampling by Cutting or Scrapping

Samples are cut from diagonally opposite corners of the upper and lower sides of the cast bar. Cutting to be done with a sharp, narrow, and rust-free steel chisel, the cut sample flattened, rolled and the resultant strip cut into small pieces, mixed to prepare an assay sample.

5.4.1 When the bullion is in the form of an ingot or a slab and is of higher fineness (high ductility), sample by cutting or drilling may be adopted.

5.4.2 Samples are cut from diagonally opposite corners of the upper and lower side of the bar. Cutting is to be done with a sharp narrow steel chisel. From each cut of 1 to 1.5 gm, sample may be taken. The cut piece should be flattened, rolled, cut in to small pieces and thoroughly mixed.

5.4.3 Samples for assaying of jewellery should be done by scrapping or cutting after removing the surface color/coatings or as agreed upon (for sampling of jewellery and artefacts refer to IS 15820).

5.5 Sampling by Drilling

The clean surface of the ingot is drilled, two from opposite top corners and two from bottom but diagonally opposite corners, using a steel drill bit, discarding first 3 mm of drillings from each hole. Then re-drill the same place and the drillings collected in a clean while paper before preparing an assay sample.

5.5.1 The ingot is drilled on the top corner with a 3 to 10 mm high speed tool steel drill. When it is known that the ingot is not of uniform composition or is of less fineness, the minimum of 2 drillings should be taken from the top and 2 from the bottom at a diagonally opposite corner.

5.5.2 The surface drilling resulting from the first few revolutions of the drill should be discarded till the hole in the drill is equal to the diameter of the drill. Preferably, the drilling should be carried out to a little more than half way through the ingot.

5.5.3 The minimum weight of the drillings from the above four borings shall not be less than 5 gm.

5.5.4 In case of fine gold, drillings should be cut in to small pieces and mixed thoroughly before taking up to assay.

5.5.5 In case of low fineness samples, 4 lots of drillings may be assayed separately or 2 lots from the top may be mixed to form one sample and 2 lots from the bottom mixed for a second sample.

5.5.6 When the material is in the form of sheet or wire, small pieces shall be cut preferably from opposite ends and thoroughly mixed.

5.6 The Following Points Needs to be Taken Care During Sampling

a) Drillings are not usually as representative of a melt as pin tube or in case when the sample is not

found homogeneous by assaying, other method such as pin tube should be used.

b) Shots or granulation method of sampling is not satisfactory for bullion of low fineness as during pouring of molten metal into water, some of the base metal present may oxidize, thus affecting the composition of silver.

c) Before taking samples by any of the above methods, all surface dirt from the surface of the ingot, bar, etc, shall be removed and all cutting, scarping, hammering, etc, shall be cleaned thoroughly. No oil shall be used in drilling.

d) When the bar has been pickled by dipping in acid after casting, the surface shall be well scrapped and cleaned before sampling. The first porting of the metal removed during preparation of the sample shall be rejected.

6 REAGENTS

6.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) or water of equivalent purity free from halides and suspended particles shall be used during the analysis.

NOTE — Pure Chemicals shall mean chemicals of recognized analytical grade, preferably AR grade that do not have impurities which affect results of analysis.

6.2 Borax (Na2B4O7) Anhydrous

6.3 Pure Gold for Proof Samples

6.3.1 For Test Method A, B and C — It is a gold metal with minimum purity 999.90 ‰ having the fineness determined to 3 significant digits (for example 999), with a previously determined accurate value. For test method D, gold purity of 999.95 ‰ and above, is preferred for gold determination value up to four significant digits. (for example 999.9).

6.4 Lead Foils — Lead foils, beads or tablets and beads, assay grade with minimum purity 999 ‰ free of gold, Bismuth and platinum group metals for method A, B and C. For method D, the lead foils, beads or tablets and beads, assay grade with minimum purity 999.9 ‰ free of gold, Bismuth and platinum group metals.

6.5 Copper Metal — in form foil, beads, tablets, wire, or disc having a minimum purity 999 ‰ and free of gold and platinum group metals.

6.6 Nickel — Minimum 999 ‰ pure, free of gold and platinum group metal

6.7 Palladium — for proof sample, minimum purity 999. 5(%) with low content of gold (<0.01‰).

6.8 Platinum — for proof sample, minimum purity 999.5 ‰ with low gold content (<0.01‰).

6.9 Base Metals — for proof samples, in a form of an approximate pre-alloy (free from precious metals).

6.10 Nitric Acid Part 1 (1:2) — It is dilute nitric acid with specific gravity 1.2 (gm/cm3), by volume, it is 1 part of nitric acid (sp. gr. 1.42 gm/cm3) to 2 parts of water properly stirred and free from suspended matter and free from halides. The presence of halides to be checked with silver nitrate test).

6.11 Nitric Acid Part 2 (1.3) — It is dilute nitric acid with specific gravity 1.3 (gm/cm3), by volume, it is 1 part of nitric acid (sp. gr. 1.42 gm/cm 3) to 1 part of water properly stirred and free from suspended matter and free from halides. The presence of halides to be checked with silver nitrate test).

6.12 Silver — for inquartation minimum purity 999 ppt (‰), free of gold and platinum group metals. For analysis of gold above 999 ppt (‰), 999.9 ppt (‰) shall be used.

6.12.1 For test method A, B and C, It is a silver containing at least 999 ppt (‰) and free from gold and platinum group metals.

6.12.2 For test method D, it is a silver containing at least 999.9 ppt (‰) purity and less than 0.0005 % gold (5 mg per kilogram of gold).

NOTES

1 The term "free of" corresponds a concentration below <0.002 ‰ of each element.

2 Otherwise, the presence of elements beyond three decimals of purity may be considered as free from impurity.

7 APPARATUS

7.1 Assay Balance — It is a balance with resolution up to 0.002 mg for assaying gold content up to 990.0 ppt (∞) (for test method – A and B), for assaying of gold alloy above 990 fineness, the resolution of balance shall be minimum 0.001 mg or better (for test methods C and D), preferably with 0.001 mg resolution).

NOTE — Determination of gold up to 99.00 % purity balance with 5th significant digits is considered adequate and for purity above 99.00 %, balance with higher resolution with 6th significant digit is required.

7.2 Assay Cleaning Brush — it is brush with stiff bristles of nylon but not of any metal.

7.3 Balling Pliers — A pair of half sphere-shaped steel pliers with smooth concave cavity in the chops being about 10 mm - 15 mm in diameter.

7.4 Cupel — Made of calcium phosphate or magnesia usually of diameter 16 mm to absorb 4 gm of lead, 22 mm diameter to absorb 6 gm lead and or 26 mm diameter to absorb 10 gm lead or a block of cupel of similar lead and base metals absorption capacity.

7.5 Cupellation Furnace — It is a muffle furnace with inlets and outlets for air for maintaining an oxidizing atmosphere constantly during the cupellation period. The muffle shall be capable of being heated with relative uniformly up to a temperature of 1050 °C to 1150 degree centigrade being controlled to \pm 50 degree centigrade. A standard muffle furnace is not satisfactory for this purpose.

7.6 Hammer and Anvil — These shall have a bright and clean striking face. The hammer shall be of polished steel and a minimum weight of 400 gm. This may be replaced by a press, with polished striking faces and reserved for this purpose only.

7.7 Parting Basket and Parting Cups — It is a made of number of small perforated cup shaped thimbles made of Pt or Pt/Ir or Pt/Rh or unglazed silica/fused quartz supported in a frame (basket) of the same material.

7.8 Rolling Mill — Constructed of polished steel and capable of reducing the cupelled button in to a flat strip of 0.12 mm thickness.

7.9 Annealing Crucibles — made of refractory material (preferably unglazed silica or quarts).

7.10 Tongs and Forceps — of various forms and sizes for charging the cupel and handling assay pieces, etc.

7.11 Trays — of various forms for keeping the assay pieces, buttons, rolls, fillets, cornets, etc.

8 HAZARDS

For Precautions to be observed in the use of certain reagents and equipment.

8.1 Use care when handling hot crucibles, cupels and operating furnace to avoid personal injury by either burn or electrical shock.

8.2 Lead and litharge (PbO) are toxic in nature and are volatile at low temperatures. Avoid inhalation, ingestion, or skin contact.

8.3 Take care while handling acids and chemicals by use of PPE (personal protection equipment).

8.4 Scrubber to control the exit of toxic fumes needs to be installed in the assay laboratory employing fire assay

9 TEST PROCEDURE

The test methods appear in the following order:

Test Method A — Gold content between 100 to 990.0 ‰ (ppt) for gold bullion and alloys.

Test Method B — Gold content between 330 to 990 ‰ (ppt) for gold jewellery and artefacts.

Test Method C — Gold content above 990 up to 999.5 % (ppt) for gold alloy/artefacts and refined gold.

Test Method D — Gold content above 999.5 ‰ up to 999.9 ‰ for refined gold bullion.

TEST METHOD A

This test method covers cupellation analysis of gold in unrefined gold bullion and gold alloys containing 100 ‰ to 990 ‰ gold.

9.1 Procedure for Assaying Gold in Gold Bullion and Gold Alloys

9.2 Weighing of Sample

Weigh at least two samples of the alloy each of 500 mg to an accuracy of 0.01 mg or better. If sample mass is insufficient, take preferably 250 mg (in duplicate) weighing to the same accuracy. In case of white gold containing nickel, take 125-250 mg sample in duplicate. Run a proof assay in duplicate adding same quantity of nickel as present in the test sample.

The mass of lead foil (or foil + lead beads) should be 8 g (approximately) for a sample of 500 mg and 4 g for 250 mg sample. In case of white gold, containing nickel, effective cupellation requires an addition of 4 g of lead. Larger cupels and additional time for completion. In case of 375 and 585 carat gold, take 250 mg sample in duplicate and extra amount of lead 4 g and 2 g respectively towards the end of cupellation and allow sufficient time for completion of cupellation. Proof assays are also to be treated in the same manner.

NOTE — Sample weight can be adjusted between 125 and 500 mg, provided all other weights are adapted and the cupel can absorb the number of oxides generated.

9.3 Preparation of the Assay Piece

9.3.1 In making up the assay-piece add enough silver to the sample to make up the total silver content between 2.3 and 3.0 times the mass of gold present in the sample allowing for the silver already present in the sample.

9.3.2 Weigh as in **9.2** at least two proof assay samples of proof gold (*see* **6.3**) and pure silver in masses which correspond to the expected gold and silver content (including inquartation addition) of the assay sample. The total content of base metals in the assay samples is taken into consideration by addition of corresponding quantity of copper (*see* **6.5**).

9.3.3 Wrap the weighed sample together with the required amount of silver and copper in the lead-foil and squeeze them in the balling pliers to form small balls. The assay piece shall now be considered ready for charging in the cupellation furnace.

9.4 Preliminary Assay

If the approximate composition of the sample of gold alloy is not known, it is necessary to make a preliminary assay. For this purpose, weigh 2 lots of 100 mg of the alloy, add 300 mg of silver to one lot and then wrap each lot separately in 3 g of lead. Cupel both side by side. Flatten the button containing the added silver and boil in 15 ml of parting acid No .2 (*see* **6.11**) for 15 min. Wash the resulting gold, dry and heat to redness and weigh. The weight in milligrams gives directly the percentage of gold and the weight in milligrams of other button gives the percentage of gold and silver together the difference between the two gives the percentage of silver. The

remainder will be base metal. When the approximate composition of the alloy has been ascertained, determine the amount of silver to be added for assay. Alternatively, preliminary assay can be done by other method such as XRF, ICP-OES, AAS, etc.

9.5 Cupellation

Arrange the cupels carefully in the muffle furnace, preferably on a removable tray. When the furnace temperature is of about 1050 - 1100 °C, place in them the assay-pieces and the proof assay sample each in proper cupel, by use of a long cupellation tongs. Ensure that proof samples are as close as possible to corresponding assay samples. The charging should be done carefully, but as rapidly as possible, so as not to cool the muffle furnace unduly. A mechanical device may be used for charging all the samples at a time. Close the muffle door and allow the cupellation to proceed for 20 to 30 min depending on the amount of lead used, the temperature being raised to about 1100°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 temperature comes down to approximately 850 °C in the furnace after the buttons are solidified and then remove from the furnace. Allow buttons to cool down sufficiently before further process.

NOTE — Alternately, samples can be placed outside the furnace in the cupels that have been previously and cooled to room temperature.

9.6 Preparing the Precious Metal Buttons (Assay-Piece) for Parting

9.6.1 Remove the buttons from the cupels by means of 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 700 °C and pass in succession through the rolls to form elongated fillets of thickness 0.22 to 0.25 mm and number them serially. After being rolled, anneal again to soften them and then separately roll up between the finger and thumb into a cornet or spiral making the lower side of the button the outer face of the cornet.

9.6.2 Place the cornets in the respective cups of the parting tray and immerse the entire tray in parting acid No.1 at a temperature of 90-95 °C and boil for 25 to 30 min or until no more nitrous fumes are observed. Take out the tray and drain from acid liquor, then wash by dipping in and out of a vessel of hot distilled water drain again and immerse in a second lot of boiling parting acid No.2. Boil the cornets in this way for 20 to 25 min, drain and wash thoroughly till the washing is free from silver nitrate. Dry the tray with the cornets by gently heating and then anneal in a muffle furnace heated to 700 °C for about 5 min.

9.7 Proof Assays

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

9.8 Parting of the Silver / Gold Samples

NOTE — For the parting operations with nitric acid, fume hood should be kept clean and used.

9.8.1 *Parting in Individual Flasks*

Place the precious metal cornets in parting flasks **6.3**, immerse the cornet in 20 ml of nitric acid (33%. *see* **6.10**) and bring to boil. Continue heating for 15 minutes or until the evolution of nitrous fumes has ceased whichever is longer, and decant. Repeat the treatment using 20 ml of nitric acid (49%, *see* **6.11**). Rinse well with water, and transfer the gold cornets to annealing crucibles. Dry them and anneal at 650-750 °C for at least 5 minutes. Allow to cool and weigh the gold cornets.

9.8.2 *Parting in a Basket*

When assaying a series of samples of similar composition, a nitric acid resistant basket equipped with a number of thimbles with perforated bottoms can be used for the parting the precious metal cornets.

Place the cornets in the thimbles and immerse the basket into nitric acid (33%, acid 1) at 60 °C to 80 °C. Bring acid to the boil and allow to boil gently for about 15 minutes or until the evolution of nitrous fumes has ceased. Remove the basket from the acid flask. Repeat the treatment in the second bath of nitric acid (49 % acid 2). Rinse well with water and weigh the gold cornets.

9.8.3 Sample with Large Amount of Base Metals

Some base metals containing in a sample are difficult to oxidize depending on their concentration. Indicative concentration is given in Annex 1. This procedure involves using additional lead and/or starting with lower sample weight.

Effective cupellation typically requires an additional 2 gm to 4 gm of lead and possibly the use of large cupels. The extra lead is incorporated at the start of the test. This procedure can be improved by decreasing the weight of the sample to 125 mg.

The proof sample should contain approximately the same proportion of base metals, which may be replaced with copper, as the assay sample.

9.8.4 Sample Containing Platinum or Palladium

For sample containing platinum with a Pt/Au ratio > 1/15 or palladium with a Pd/Au ratio > 1/3, traces of those metals can remain in the gold cornet after cupellation and parting. In these cases, follow either of the three procedures:

- a) The amount of sample shall be decreased to 125 mg, and pure gold shall be added to bring the Pt/Au and Pd/Au ratios in the acceptable range. The cupellation is performed normally;
- b) The gold cornets from the sample and the proof assay shall be re cupelled with 4 gm of lead, silver equal to 2.5 times the mass of gold and 25 to 50 mg of copper. Repeat the parting process and weigh the final gold cornet; and
- c) Place the gold cornet from the sample and the proof assays in the third bath of nitric acid (49%, acid 2).

The proof sample should contain approximately the same amount of platinum and palladium as the sample.

9.8.5 Sample with a Silver/Gold Ratio Higher than 3

If the Silver content of a sample is higher than 3 times the gold content, an addition of fine gold is required in order to respect the 2.3:1 to 3.1 silver/gold ratio.

Alternatively, parting of these alloys can be done in individual flasks without addition of fine gold. In this case, the broken gold cornet shall be quantitatively recovered for weighing.

TEST METHOD B

10 SCOPE

This test method covers cupellation analysis of gold in gold jewelry or artefacts containing 375 ‰ to 990 ‰ gold.

10.1 Procedure for Assaying Gold in Gold Jewellery/Artefacts (not Containing Nickel, Platinum and Palladium)

10.1.1 Analysis Samples

Transfer at least two samples of the alloy, preferably between 125 mg and 250 mg weighed to the nearest ± 0.01 mg into assay-grade lead-foil. In case of white gold containing nickel, take 125-250 mg sample in duplicate. The mass of the foil (or foil + beads) should be at least 4 g for gold samples up to 125 mg and 6 g for samples weighing 250 mg. Add pure silver equivalent to 2.3 to 3 times the mass of fine gold present. Roll and compress the lead foil into a tight ball.

NOTE — This method is developed to assist hallmarking of jewellery as per relevant Indian standard to validate fineness value of all jewellery / artefacts as determined by XRF method (IS xxxxx). Hence the use of assay balance of 0.01/002mg may be sufficient to achieve desired accuracy of a three-digit fineness marking up to 995.0 ppt (24 KS) Refer **7.1**.

10.1.2 *Proof Assay Samples*

Weigh, as in **10.1.1** at least two proof assay samples of proof gold and pure silver in masses which correspond to the expected gold and silver contents (including the inquartation addition) of the assay sample. The total content of base metals in the assay samples is taken into consideration by the addition of a corresponding quantity of copper. Treat the proof assay samples and the assay samples in **10.1.2** and **10.1.1** in the same manner.

10.1.3 Cupellation and Treatment of Precious Metal Buttons

Place the assay and the proof assay samples (*see* **10.1.2**) tightly wrapped in lead foil, on magnesium oxide cupels which have been preheated to at least 1 100 °C in the cupellation furnace. Place the cupels with the proof assay samples as close as possible to the corresponding assay samples in the cupellation furnace maintained at 1050 °C – 1100 °C. Continue heating until this stage is completed (about 20 min) under oxidizing conditions. Remove the cupels from the furnace. Allow the precious metal buttons to cool down before lifting them from the cupels with the forceps. Squeeze the buttons and brush their undersides carefully with a brush to remove any adhering material. Flatten the beads on the polished anvil and polished hammer / press meant for the purpose and anneal by heating just to red heat 700 °C.

Roll them into 0.12 to 0.15 mm thick strips and anneal again. Roll the strips into cornets without contamination or loss of gold. It may be noted that the cupel should be examined carefully to ensure that the precious metal bead contains all the sample gold. Small droplet residues indicate the need for a repeat determination in a smaller cupel.

10.1.4 Parting of the Silver/Gold Samples

Place the precious metal comets in parting flasks. First immerse the comet in 20 ml of nitric acid (see 6.8) at a temperature at least 5 °C below the boiling point and bring to the boil. Continue heating for 15 min or until the evolution of nitrous fumes has ceased, whichever is longer, decant, wash with warm water and immerse in 20 ml of nitric acid (see 6.9) and boil for 10 min. Decant and wash the gold comet with warm water at 60° to 70 °C until it is free of silver nitrate. Transfer the gold comets to small porous parting cups (porcelain crucible). Dry them and anneal at 700° to 750 °C for about 5 min, cool, and then weigh the gold comet. When assaying a series of samples of similar composition, instead of using a number of parting flasks the precious metal comet can be parted with the aid of a dissolution basket consisting of Pt/lr or Pt/Rh which is equipped with a number of quartz, Pt/lr or Pt/Rh thimbles with perforated bottoms. The cornets are placed in the thimbles and basket is lowered slowly into nitric acid (see 6.8) at about 90 °C. The acid is brought to the boil and allowed to boil gently for 15 min or until the evolution of nitrous fumes has ceased, whichever is longer. Remove the basket from the acid, rinse in water repeat the treatment in a second batch of nitric acid (see 6.9) and boil again for 15 min. Remove the basket from the acid, rinse it with warm (60 °C to 70 °C) water until it is completely free of silver nitrate and allow to dry. Finally place the basket with the gold samples for about 5 min in muffle furnace, heated to approximately 700 °C to 750 °C. After cooling, the gold samples shall be weighed.

NOTE — For the parting operations with nitric acid, fume hood should be kept clean and used exclusively for this determination.

10.2 Procedure for Assaying Gold in Gold Jewellery/Artefacts Containing Nickel (White Gold)

It is difficult to extract all the nickel in the alloy into the cupel by using the standard quantity of lead . Effective cupellation requires an additional 4 g of lead and the use of larger cupels. This extra lead may be incorporated at the start of the test, if the cupel is large enough to contain the increased volume of melt. Alternatively a button of lead is added to the hot precious metal bead in the cupel after the lead oxide fumes from the initial operation have ceased. Care is needed if the cupellation furnace is not adopted for this addition. The proof assays should contain approximately the same proportion of nickel as the sample.

10.3 Procedure for Assaying Gold in Gold Jewellery/Artefacts Containing Platinum or Palladium

For sample containing platinum with a Pt/Au ratio > 1/15 or palladium with a Pd/Au ratio > 1/3, traces of those metals can remain in the gold cornet after cupellation and parting. In these cases, follow either of the three procedures:

a) The amount of assay sample shall be decreased to 125 mg, and pure gold (*see* **5.5**) shall be added to bring the Pt/Au and Pd/Au ratios in the accepted range. The cupellation is performed normally.

- b) The gold cornets from the assay sample and the proof sample shall be recupelled with 4 g of lead, silver equal to 2.5 times the mass of gold and 25 to 50 mg of copper (**5.9**). Repeat the parting process and weigh the final gold cornets.
- c) Place the gold cornets from the assay sample and the proof sample in a third bath of nitric acid (49 %, *see* 5.2). The proof samples should contain approximately the same amount of platinum and palladium as the assay samples.

10.4 Procedure for Assaying Gold in Gold Jewellery/Artefacts with a Silver/Gold Ratio Higher than 3

If the silver content of a sample is higher than 3 times the gold content, an addition of pure gold is required in order to respect the 2.3:1 to 3:1 silver/gold ratio. Alternatively, parting of these alloys can be done in individual flasks without addition of pure gold. In that case, the broken gold cornet shall be quantitatively recovered for weighing.

TEST METHOD C

11 SCOPE

This test method covers cupellation analysis of gold in refined bullion containing 990 and up to 999.5 fineness of gold.

NOTE — The marking of refined gold bar (**8.1** of IS 17278) is to be of four significant digits. Hence the method covers gold fineness up to 999.5 ppt allowing use of proof gold of 999.9 ppt (‰).

11.1 Procedure for Analysis of Gold in Refined Bullion Containing 990 ppt and up to 999.5 ppt (‰) of Gold.

When analyzing samples containing approximately 990 ppt (‰) gold, still increased accuracy in operation and parameter control is needed.

In order to achieve the best results, proceed as stated in (*see* **10.1**), introducing the following modifications:

a) Weigh at least 250 mg of alloy; add 20 ± 5 mg of copper to the sample and amount of inquartation silver as stated in **9.3.1**;

NOTE — For determination of gold content above 999 ppt and up to 999.5 ppt (%) fineness, sample in quadruplicate is preferred.

- b) For the proof assay samples, proceed exactly in the same way as for the assay sample. Use gold of purity 999.9 ppt (‰) and take care the mass of the added inquartation silver is in the same range ± 10 mg) as for the assay samples. Always run in parallel at least two proof assay samples;
- c) Carry out the cupellation with a total amount of at least 2 g of lead;
- d) After cupellation, flatten all beads so that they have approximately the same shape and thickness anneal the flattened beads in a muffle to red heat (700 °C) to obtain the same condition of recrystallization; and
- e) Proceed to the parting of the samples as stated in **10.1.4**. Take care that the quantity of acid and the parting time are the same for all samples of the same series. Finally, dry and anneal in parallel all fine gold comets. The use of a basket for the parting will be advantageous for this purpose.

NOTE — The cupellation will take about 15 min.

12 CALCULATION AND EXPRESSION OF RESULTS (FOR TEST METHOD A, B AND C)

12.1 Proof Sample Factor

The proof sample factor (F) is calculated using formula

$$F = \frac{(m_1 \times T_{Au})}{m_2}$$

where

 $m_1 = \text{mass of the proof sample in milligrams};$

- $m_2 = mass$ of the proof gold cornet after assay in milligrams; and
- T_{Au} = fineness of pure gold (*see* **6.3**) in proof sample (T_{Au} = 999.9 and 1 for fineness of above 999.99 ppt).

12.2 Calculation of Gold Content

The gold content W_{Au} in parts per thousand ((‰) by mass of the alloy is calculated using formula.

$$W_{A_u} = \frac{[(m_4 \ F_{mean}) - m_5 \times T_{Au})]}{m_3} \times 10^3$$

where

 $m_3 = mass$ of the sample, in milligrams;

 m_4 = mass of the sample in god cornet after cupellation in milligrams;

- $m_5 = \text{mass of the added pure gold (used in see 9.8.5), otherwise m_5 = 0) in milligrams;$
- F_{mean} = mean value of proof sample factors (F), see 13.1; and
- T_{Au} = fineness of pure gold (*see* 6.3) in proof sample {T_{Au}= 999.9 and 1 for fineness of above 999.99 (ppt) ((‰))}

13 REPEATABILITY

Duplicate determination shall give results differing by maximum 0.5 ppt (‰) by mass for sample with a fine gold content below 990 ppt and not containing platinum, palladium and Nickel and by maximum 1.0 ppt (‰) by mass for sample with a fine gold content below 990 ppt containing platinum, palladium and Nickel (white gold) alloys and maximum 0.2 ppt (‰) by mass for gold alloys containing above 990 and up to 999.5 (‰) or more gold. If the difference is greater than this, the assay shall be repeated.

When analyzing alloys with a fine gold content up to 990 (‰) or more, the values of F (Proof correction Factor), for the proof samples which run in parallel shall not differ by more than 0.04 mg. If the difference is greater than this, the assay shall be repeated.

NOTE — If the duplicate results do not provide the repeatability even after repeat assays (maximum two repeat assay), the sample is considered as non-homogeneous and resampling shall be done to produce a homogeneous sample.

TEST METHOD D

14 SCOPE

This test method covers cupellation analysis of gold in refined bullion containing gold content above 999.5 ppt and up to 999.9 ppt fineness.

14.1 Procedure for Assaying Gold in Gold Bullion and Gold Alloys

14.2 Number of Determinations

A minimum of four and maximum of 8 determinations shall be carried out on each test sample, where the gold content of the test sample is 999 ppt (‰) and above.

14.3 Proof Test

A minimum of two and maximum of four proof tests shall be carried out with each batch of samples and shall be subjected to the same procedure as the test portions. The proofs shall contain a known mass of gold (*see* 6.3) equivalent to the mass of the sample.

14.4 Determination

14.4.1 *Preparation of the Test Portion and Proof*

The procedure shall be as follows:

a) For each test portion and proof, cut and weigh an appropriate mass of lead foil (*see* **6.4**). Fold each sheet into a cone;

NOTE -2.5 g to 5 g of lead foil is recommended. Do not record the mass but the same amount of lead should be used for each test portion and proof.

- b) Weigh and record to 0.001 mg, accuracy. 500 to 501 mg of each test portion (m₁) and proof (m₂) (*see* **6.3**) and transfer each to a separate lead con; and
- c) To each cone add between 1240 to 1260 mg of silver (*see* **6.10**) and 20 to 30 mg of copper (*see* **6.5**); and

NOTE — A small amount of copper added to a sample containing no copper, acts as a deoxidant and helps prevent cracking and splitting of the cornet edges when it is hammered and rolled.

d) Fold the cones tightly around the contents and carefully squeeze to a compact form using ball plier.

14.5 Cupellation

The cupellation procedure shall be as follows:

- a) Preheat the cupellation furnace to 1150 ± 50 °C;
- b) Place cupels into the pre-heated cupellation furnace, one cupel for each test portion and proof and allow the cupels to attain the furnace temperature;
- c) To minimize the temperature loss in the cupellation furnace, quickly place the folded cones on to pre-heated cupels in order from left to right and from the back to the front;

NOTE — The test portions and proofs are to be arranged so that they are evenly distributed throughout the furnace.

- d) Maintain the samples at a temperature of $1150 \pm 50^{\circ}$ until cupellation is complete, (approximately 25 min) ensuring that adequate air flow is maintained through the furnace to promote oxidation;
- e) Remove the cupels from the furnace, maintaining them in the original spatial order, and allow them to cool in a breeze-free environment;
- f) As soon as the material has solidified, loosen each bead from its cupel. Check that the beads are smooth, hard and shiny. Reject any that are dull, flat or fragmented; and
- g) Carefully brush off the bead with cleaning brush. Place each cooled bead on its side on the anvil or press, and hammer or press it once, firmly, to dislodge adhering cupel material.

14.6 Parting

The procedure for parting shall be as follows:

- a) After hammer/press, each flattened bead into a flat oval sheet, ensuring that a thin edge is produced to facilitate rolling;
- b) Anneal the sheets at 700-800 °C for 5 min in an annealing/muffle furnace;
- c) Pass each sheet through rollers reducing the roller clearance with each pass, until the resultant sheets are of equal thickness, between 0.15 mm and 0.2 mm;
- d) Anneal each strip as in Step *see* **9.3.3**(b);
- e) Crimp over the end and carefully roll each strip into a compact cornet. Sufficient clearance should be allowed between each fold to allow acid penetration of the cornet;
- f) Preheat the following solutions to approximately 90 °C:
 - 1) Sufficient dilute nitric acid (*see* **6.8**) as will enable the complete submersion of the parting tray in Step **8.3.3** (g);
 - 2) Sufficient dilute nitric acid (*see* **6.9**) as will enable the complete submersion of the parting tray in Step **8.3.3** (h); and

3) For better accuracy and precision 3rd parting is suggested with sufficient dilute nitric acid (*see* **6.9**).

NOTE — Anti-bumping beads may be used while heating the acid in bath.

- g) Carefully place each cornet into separate thimbles of a parting tray. Submerge the parting tray in a vessel containing dilute nitric acid (Step 8.3.3 (f) (i)). Allow the parting reaction to subside and boil gently until the fumes of oxides of nitrogen have disappeared or approx. 20 min after boiling;
- h) Transfer the parting tray, after DI water washing, to the second vessel containing preheated dilute nitric acid (Step **8.3.3** (f) (ii)) and allow to boil and keep for 20 min after boiling;
- i) Transfer the parting tray to a container of hot water (Step **8.3.3** (f) (iii)) and wash in three changes of hot water by agitating the tray;
- j) Remove excess hot water from the parting tray by draining or drying on tissue paper and then carefully dry it on a hot plate. Anneal the cornets at a temperature of 700-800 °C for 5 min in annealing furnace;
- k) Cool and allow the parting tray to come to equilibrium with the balance room temperature; and
- 1) Weigh and record to 0.001 mg, the gold cornet from each test portion (m3) and each proof (m4).

15 CALCULATION AND EXPRESSION OF RESULTS

15.1 Proof Sample Factor

The proof sample factor (F) is calculated using formula

$$F = \frac{m_1 \times T_{Au}}{m_2}$$

where

 $m_1 = \text{mass of the proof sample in milligrams;}$

- $m_2 = mass$ of the proof gold cornet after assay in milligrams; and
- TAu = fineness of pure gold (6.3) in proof sample (T_{Au} = 999.9 and 1 for fineness of above 999.99 ppt).

15.2 Calculation of Gold Content

The gold content W_{Au} in parts per thousand (‰) by mass of the alloy is calculated using formula (2).

$$W_{Au} = \frac{[(m_4 \ F_{means} - (m_5 \times T_{Au})]]}{m_3} \times 10^3$$

Where

 $m_3 = mass$ of the sample, in milligrams;

 $m_4 = \text{mass of the sample in god cornet after cupellation in milligrams;}$

 $m_5 = \text{mass of the added pure gold (used in$ **9.8.5**), otherwise m5 = 0) in milligrams;

 F_{mean} = mean value of proof sample factors (F), see 13.1; and

 T_{Au} = fineness of pure gold (6.3) in proof sample {TAu= 999.9 and 1 for fineness of above 999.99(ppt) (‰).

16 REPEATABILITY

< 999.5 ‰ the acceptance criteria should be:

Difference between replicates shall be not more than 0.05 parts per thousand (‰), One outliner can be omitted.

> 999.5 ‰ the acceptance criteria should be:

Difference between replicates shall be not more than 0.15 parts per thousand (‰), One outliner can be omitted.

17 TEST REPORT

The test report shall include at least the following information:

- a) Identification of sample including source, date of receipt, date of testing;
- b) Description of sample;
- c) Sampling Procedure;
- d) The methods used as described by referenced to this standard;
- e) The Gold content of the test sample, expressed as parts per thousand (‰) by mass, as single and mean value to one decimals space as applicable;
- f) Any unusual observation features observed during the test, which may have had an effect on the reported values;

- g) Identification of the laboratory carrying out the analysis; and
- h) Signature of the laboratory chemist and the lab manager.

ANNEX A

(*Clause* 9.8.3)

CONCENTRATION OF OTHER METALS, AFFECTING THE GOLD RESULT

The elemental concentration below represents the concentrations above which some elements will affect the gold assay result (based upon a starting weight of 250 mg).

Table A.1 – Maximum	Concentration of base metals fo	for which the general method appli	es

Sl	Element	Maximum Concentration
No.		
		‰ (ppt)
(1)	(2)	(3)
i)	Al	10
ii)	Bi	30
iii)	Cr	10
iv)	Fe	30
v)	Ni	40
vi)	Sb	30
vii)	Se	50
viii)	Sn	20
ix)	Te	30
x)	Ti	10
xi)	W	10
xii)	Zn	200

Appropriate measures as described in 9.8.3 allow to work (assay) beyond these concentration.