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Determination of gold in gold jewellery alloys — Cupellation method (fire assay)

Dosage de l'or dans les alliages d'or pour la bijouterie-joaillerie — Méthode de coupellation (essai au feu)

Reference number
ISO 11426:1993(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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INTERNATIONAL STANDARD **ISO 11426:1993(E)**

Determination of gold in gold jewellery alloys — Cupellation method (fire assay)

1 Scope

This International Standard specifies a cupellation method (fire assay) for the determination of gold in gold jewellery alloys. Preferably the gold content of the alloys lies between 333 and 930 parts by mass per thousand (‰) of gold. The procedure applies specifically to gold alloys incorporating silver, copper and zinc. Some modifications are indicated where nickel and/or palladium are present in the so-called white gold alloys.

NOTE 1 This method is intended to be used as the referee method for the determination of fineness in the alloys covered by ISO 9202.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 9202:1991, *Jewellery — Fineness of precious metal alloys.*

3 Principle

The gold alloys are inquarted with silver, compounded with lead and cupelled in a cupellation furnace until a precious metal button is obtained. After flattening and rolling, the silver is extracted (parted) in nitric acid and the gold is weighed. Possible systematic errors in the procedure are eliminated by assaying standard proof samples in parallel. White gold alloys containing palladium and/or nickel require some procedural changes.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Nitric acid, 33 % (*m/m*), ρ_{20} = 1,2 g/cm^3, free of halide.

4.2 Nitric acid, 49 % (*m/m*), ρ_{20} = 1,3 g/cm^3, free of halide.

4.3 Lead, assay grade, free of precious metals and bismuth, as foil, beads or tablets.

4.4 Silver, for inquartation, minimum purity 999,9 parts by mass per thousand (‰), free of gold and platinum group metals.

4.5 Pure gold, for proof samples, minimum purity 999,9 parts by mass per thousand (‰).

4.6 Palladium, for proof samples, minimum purity 999,9 parts by mass per thousand (‰), free of gold and other platinum group metals.

4.7 Nickel, for proof samples, minimum purity 999 parts by mass per thousand (‰), free of gold and platinum group metals.

4.8 Copper, foil or wire, minimum purity 999 parts by mass per thousand (‰), free of gold and platinum group metals.

4.9 Borax, ($Na_2B_4O_7$), anhydrous.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Cupellation furnace, in which an oxidizing atmosphere can be maintained. N.B. A standard muffle furnace is not satisfactory for this purpose.

5.2 Magnesium oxide cupels, usually of diameter 22 mm to absorb 6 g of lead, usually of diameter 26 mm to absorb 10 g of lead, or blocks of cupels of similar absorption.

5.3 Parting flasks, or platinum basket.

5.4 Parting cups, unglazed porcelain crucibles.

5.5 Cupellation tongs.

5.6 Assay pliers.

5.7 Polished anvil, may be replaced by a press, polished and reserved for this purpose.

5.8 Polished hammer, of minimum mass 400 g, may be replaced by a press, polished and reserved for this purpose.

5.9 Scorification dishes, usually of diameter 50 mm.

5.10 Jewellers' rolls.

5.11 Platinum-tipped crucible tongs.

5.12 Assay cleaning brush, of stiff bristle or nylon but not brass.

6 Sampling

The sampling procedure for gold jewellery alloys shall be agreed upon until a corresponding standard method has been published.

For coated articles, appropriate precautions that have been agreed upon shall be taken, to exclude the coating from the determination.

7 Procedure

7.1 Yellow gold alloys

7.1.1 Analysis samples

Transfer at least two samples of the alloy, preferably between 125 mg and 250 mg weighed accurately to the nearest 0,01 mg, into assay grade lead foil (4.3). The mass of foil (or foil plus beads) should be at least 4 g for yellow gold samples up to 200 mg, and 6 g for samples from 201 mg to 300 mg (250 mg). Add pure silver (4.4), equivalent to 2,3 to 3 times the mass of fine gold present. Roll and compress the lead foil into a tight ball.

7.1.2 Proof assay samples

Weigh, as described in 7.1.1, at least two proof assay samples of proof gold (4.5) and pure silver (4.4) 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 7.1.3 and 7.1.4 in the same manner.

7.1.3 Cupellation and treatment of precious metal buttons

Place the assay and the proof assay samples (7.1.2), tightly wrapped in lead foil, on magnesium oxide cupels (5.2) which have been preheated to at least 1 000 °C in the cupellation furnace (5.1).

Place the cupels, with the proof assay samples as close as possible to the corresponding assay samples, in the cupellation furnace controlled at 1 050 °C to 1 150 °C. Continue heating until this stage is completed (about 25 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 assay pliers (5.6). Squeeze the buttons and brush their undersides carefully with a brush (5.12) to remove any adhering cupel material. Flatten the beads on the polished anvil (5.7) with a polished hammer (5.8) and anneal by heating just to red heat. Roll them into 0,12 mm to 0,15 mm thick strips and anneal again. Roll the strips into cornets without contamination or loss of gold.

7.1.4 Parting of the silver/gold samples

The precious metal cornets are placed in parting flasks (5.3). First immerse the cornet in 20 ml of nitric acid (4.1) at a temperature at least 5 °C below boiling temperature 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 (4.2), boil gently for 15 min and decant. Wash, immerse in 20 ml of nitric acid (4.2) and boil for 10 min. Decant and wash the undissolved remaining gold with warm (60 °C to 70 °C) water until it is free of silver nitrate. Transfer the gold cornets to small porous porcelain parting cups (gold crucibles) (5.4), dry, anneal at 700 °C to 750 °C for about 5 min, cool and weigh the gold cornet.

When assaying a series of samples of similar composition, instead of using a number of parting flasks, the precious metal cornets can be parted with the aid of a dissolution basket consisting of Pt/Ir or Pt/Rh which is equipped with a number of quartz thimbles with perforated bottoms. The cornets are placed in the thimbles and the basket is lowered slowly into nitric acid (4.1) 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, and repeat the treatment in a second bath of nitric acid (4.2), boiling again for 15 min. Remove the basket from the acid, rinse it with 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 a muffle furnace, heated to approximately 700 °C to 750 °C. After cooling, the gold samples can be weighed.

7.2 White gold alloys containing nickel

If nickel is present, two equivalent variations on the method specified in this International Standard are acceptable. These involve either the use of additional lead or scorification.

7.2.1 Cupellation with additional lead

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 (4.3) 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 (preferably) 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 (5.1) is not adapted for this addition.

The proof assays should contain approximately the same proportion of nickel as the sample.

7.2.2 Scorification

For white gold alloys containing nickel, pretreatment of the sample by scorification involves wrapping it in 2 g of lead foil (4.3). The sample consists of 125 mg to 250 mg of gold, and is inquarted with silver equivalent to 2,3 to 3 times the mass of fine gold present. Place this capsule in a scorification dish (5.9), together with 15 g of lead and 1,5 g to 2 g of anhydrous borax (4.9) and heat to 1 000 °C in the furnace. An increased air supply may be needed to oxidize the large quantity of lead. After 20 min to 30 min, when a liquid slag covers the surface of the dish, the temperature is raised briefly to 1 100 °C (for about 2 min). Remove the dish with the tongs, cool, and separate the lead button from the slag. This button, which contains the original gold and silver, is cupelled as described in 7.1.3.

Proof gold samples made with the appropriate amount of added nickel are treated in a similar way.

7.3 White gold alloys containing palladium (nickel absent)

For white gold alloys containing palladium, traces of this metal may remain in the cornet after a single cupellation and parting. With these alloys, the cornets from the sample and the proof assays should be recupelled with 4 g of lead, silver equal to 2,5 times the weight of gold and a small piece (about 50 mg) of copper (4.8). The parting process is repeated and the final cornets are weighed.

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

7.4 Gold alloys incorporating more than 40 % silver

These alloys shall be treated as yellow gold alloys, with proper allowance being made for the higher silver content, when determining the inquartation addition.

8 Expression of results

8.1 Method of calculation

Calculate the gold content w_{Au}, in parts by mass per thousand (‰), of the alloy using the formula

$$w_{\text{Au}} = \frac{m_2 + \Delta T}{m_1} \times 10^3$$

where

m_1 is the mass, in milligrams, of the sample;

m_2 is the mass, in milligrams, of sample cornet;

ΔT is the average value of the proof assay gold mass minus cornet mass of proof assay sample, in milligrams.

8.2 Repeatability

Duplicate determinations shall give results differing by less than 0,5 part by mass per thousand (‰) for yellow and red gold alloys and less than 1,0 part by mass per thousand (‰) for white gold alloys. If the difference is greater than this, the assay shall be repeated.

9 Notes on procedure

9.1 When the composition of the samples is unknown, use a preliminary assay for the estimation of the fineness of gold. For the distinction between palladium and nickel white golds, the touch stone test can also be used. If the cornet breaks up during the parting process, this is often an indication of excess silver.

9.2 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 Test report

The test report shall include the following information:

a) identification of the sample including source, date of receipt, form of sample;

b) sampling procedure;

c) the method used by reference to this International Standard;

d) gold content of the sample, in parts by mass per thousand (‰) as single and mean values;

e) if relevant, any deviations from the method specified in this International Standard;

f) any unusual features observed during the determination;

g) date of test;

h) identification of the laboratory carrying out the analysis;

i) signature of the laboratory manager and operator.