

DRAFT INTERNATIONAL STANDARD

ISO/DIS 11426

ISO/TC 174

Secretariat: DIN

Voting begins on:
2020-08-25

Voting terminates on:
2020-11-17

Jewellery and precious metals — Determination of gold — Cupellation method (fire assay)

ICS: 39.060

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ISO/CEN PARALLEL PROCESSING



Reference number
ISO/DIS 11426:2020(E)

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Published in Switzerland

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 174, *Jewellery and precious metals*.

This fourth edition cancels and replaces the third edition (ISO 11426:2014), which has been technically revised.

The main changes compared to the previous edition are as follows:

- Extension of the scope to cover determination of gold in multiple types of alloys, not only in jewellery ones;
- Purity of proof samples was re-defined in 5;
- Specific procedures are described in 8 for samples with large amount of base metals, containing platinum or palladium, or with a silver/gold ratio higher than 3;
- Calculation were adapted to take into account the addition of fine gold and the fineness of the gold used in the proof;
- Repeatability requirements were changed;
- The use of scorification was removed.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Jewellery and precious metals — Determination of gold — Cupellation method (fire assay)

1 Scope

This document specifies a cupellation method (fire assay) for the determination of gold on a material considered homogeneous. The gold content of the sample should preferably lie between 100 and 999,5 parts per thousand (‰). Finenesses above 999,5 ‰ can be determined using a spectroscopy method by difference (e.g. ISO 15093).

The procedure is applicable to most types of gold samples. Some modifications are indicated for specific cases (presence of large amount of base metals, platinum or palladium, silver). It is not compatible with the presence above trace levels of iridium, rhodium and ruthenium (more than 0.25 ‰ for the sum of all three elements).

This method is also intended to be used as the recommended method for the determination of fineness in jewellery alloys covered by ISO 9202.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 15093, *Jewellery and precious metals — Determination of high purity gold, platinum and palladium — Difference method using ICP-OES*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

cornet

alloy of gold and precious metals shaped in a roll, prior the parting process

3.2

gold cornet

gold shaped in a roll, after the parting process

3.3

parting

separation of silver and other metals from gold by digestion of those metals with nitric acid, in a chloride-free environment

3.4

proof sample

synthetic reference sample whose composition is as similar as possible to the sample; cupellation of the proof sample is performed together with the sample, and its result is used to correct the final assay

3.5

inquartation

addition of silver to gold alloys in a specific ratio in order to enable the parting of gold from silver by means of nitric acid

4 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 weighed. Possible systematic errors in the procedure are eliminated by assaying standard proof samples in parallel.

[Annex A](#) gives information on metals, that can influence the result of gold testing.

5 Reagents

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

5.1 Nitric acid (HNO_3), approximately 33 % (mass fraction), with low content of halides (approximately < 2 mg/l; the presence of halides can be detected with silver nitrate test).

5.2 Nitric acid (HNO_3), approximately 49 % (mass fraction), with low content of halides (approximately < 2 mg/l; the presence of halides can be detected with silver nitrate test).

5.3 Lead, assay grade, free of gold and platinum group metals, containing less than 0,1 ‰ of bismuth, as foil, beads or tablets.

5.4 Pure silver, for inquartation, minimum purity 999,9 ‰, with low content of gold and platinum group metals ($\leq 0,01$ ‰ per element).

5.5 Pure gold, for proof samples, minimum purity 999,90 ‰, with a fineness determined to 5 significant digits; a purity of 999,99 ‰ is preferred.

5.6 Pure platinum, for proof samples, minimum purity 999,5 ‰, with low content of gold ($\leq 0,01$ ‰).

5.7 Pure palladium, for proof samples, minimum purity 999,5 ‰, with low content of gold ($\leq 0,01$ ‰).

5.8 Base metals, for proof samples, in a form of an appropriate pre-alloy (free of precious metals).

5.9 Copper (foil, wire, beads or tablets), for proof samples, minimum purity 999 ‰, free of gold and platinum group metals.

NOTE 1 The term “free of” corresponds a concentration below $\leq 0,002$ ‰ of each element.

NOTE 2 For fineness determination of metals according to [5.3](#) to [5.9](#), the oxygen content is not taken into account.

6 Apparatus

6.1 Cupellation furnace, capable of reaching a relatively homogeneous temperature of 1 050 °C to 1 150 °C, and in which an oxidizing atmosphere can be maintained. A standard muffle furnace is not satisfactory for this purpose.

6.2 Magnesia cupels, in form of single or block cupels, capable of absorbing the lead and base metals during the cupellation.

6.3 Parting flasks or nitric acid resistant basket with thimbles.

6.4 Annealing crucibles, made of refractory or other non-contaminating materials.

6.5 Cupellation tongs.

6.6 Assay pliers, forceps / tweezers.

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

6.8 Polished hammer, which may be replaced by a press, polished and reserved for this purpose.

6.9 Jewellers' rolls.

6.10 Assay cleaning brush.

6.11 Analytical balance, with a reading accuracy of at least 0,01 mg; for determination of gold fineness above 995 ‰, a reading accuracy of 0,001 mg is preferred.

7 Sampling

The sampling procedure for jewellery gold alloys and associated products should be performed in accordance with ISO 11596.

8 Procedure

8.1 Method selection

WARNING — Suitable health and safety procedures should be followed

When the composition of the samples is unknown, a preliminary analysis by suitable means shall be used to determine the approximate composition of the material, e.g. XRF analysis.

Based on the preliminary analysis, the appropriate method shall be selected:

For samples containing large amounts of base metals (including white gold alloys with nickel; see [Annex A](#) for maximum concentration of each element), [8.3](#) shall be applied;

For samples containing large amount of platinum and palladium, [8.4](#) shall be applied;

For samples containing large amount of silver, [8.5](#) shall be applied;

For all other samples, the general method [8.2](#) shall be applied.

8.2 General method

8.2.1 Assay samples

Transfer at least two samples of the alloy, approximatively 250 mg, weighed to the nearest 0,01 mg, into assay-grade lead foil ([5.3](#)). The mass of the lead (foils, beads, tablets or a mixture of them) should be 4 g to 6 g. Add pure silver ([5.4](#)) to reach a total mass of silver equivalent to 2,3 to 3 times the mass of fine

gold present. If the expected sum of all precious metals is above 900 ‰, 25 mg to 50 mg of copper (5.9) should be added. Roll and compress the lead foil into a tight ball.

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

8.2.2 Proof samples

Weigh, as in 8.2.1, at least two proof samples of pure gold (5.5) and pure silver (5.4) in masses which correspond to the expected gold content and the total silver content (including the inquartation addition) of the assay sample. The total content of base metals in the assay samples (including added copper) is taken into consideration by the addition of a corresponding quantity of copper.

8.2.3 Cupellation and treatment of precious metal buttons

Treat the assay samples (8.2.1) and the proof samples (8.2.2) in the same manner.

Place the assay and the proof samples on magnesia cupels (6.2) that have been preheated to 1 050 to 1 150 °C in the cupellation furnace (6.1).

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

Place the cupels with the proof samples as close as possible to the corresponding assay samples in the cupellation furnace maintained at 1 050 °C to 1 150 °C. Continue heating under oxidizing conditions until the cupellation process is completed (typically 20 min to 40 min). Remove the cupels from the furnace. Allow the precious metal buttons to cool down before lifting them from the cupels with the forceps/tweezers (6.6). Squeeze the buttons and brush their undersides carefully with a brush (6.10) to remove any adhering cupel material. Flatten the beads on the polished anvil (6.7) with a polished hammer (6.8) or by a press polished and reserved for this purpose, and anneal by heating at 500 °C to 900 °C.

Roll them into 0,12 mm to 0,15 mm thick strips using the jewellers' rolls (6.9), anneal again to red heat and then roll the strips into cornets with the underside outwards.

The cupel should be examined carefully to ensure that the precious metal bead contains all gold of the sample. If small droplet residues are present a full assay has to be repeated.

8.2.4 Parting of the silver/gold samples

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

8.2.5 Parting in individual flasks

Place the cornets in parting flasks (6.3). Immerse the cornet in 20 ml of nitric acid (33 %, see 5.1) and bring to the boil.

Continue heating for 15 min 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 5.2). Rinse with water, and transfer the gold cornets to annealing crucibles (6.4). Dry them and anneal at 500 °C to 900 °C for at least 5 minutes. Allow to cool and weigh the gold cornets.

8.2.6 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 (6.3) can be used for parting the cornets.

Place the cornets in the thimbles and immerse the basket into the nitric acid (33 %, see 5.1) at 60 °C to 90 °C. Bring the acid to the boil and allow to boil gently for about 15 min or until the evolution of nitrous

fumes has ceased. Remove the basket from the acid. Repeat the treatment in a second bath of nitric acid (49 %, see 5.2). Rinse with water, and allow to dry.

Finally, place the basket containing the gold cornets in a muffle furnace at 500 °C to 900 °C for at least 5 min. Allow to cool and weigh the gold cornets.

NOTE Presence of iridium, rhodium and ruthenium can be detected by observing the cornets and the gold cornets.

8.3 Sample with large amount of base metals

Some base metals contained in samples are difficult to oxidise depending on their concentration. Indicative concentrations are given in Annex A. This procedure involves using additional lead and/or starting with a lower sample weight.

Effective cupellation typically requires an additional 2 g to 4 g of lead (5.3) and possibly the use of larger cupels. This extra lead is incorporated at the start of the test. This procedure can be improved by decreasing the weight of 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.

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:

- The amount of sample shall be decreased to 125 mg, and pure gold (5.5) shall be added to bring the Pt/Au and Pd/Au ratios in the accepted range. The cupellation is performed normally.
- The gold cornets from the sample and the proof assays 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.
- Place the gold cornets from the sample and the proof assays 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 sample.

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 that case, the broken gold cornet shall be quantitatively recovered for weighing.