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Determination of palladium in palladium jewellery alloys — Gravimetric determination with dimethylglyoxime

*Dosage du palladium dans les alliages de palladium pour la
bijouterie-joaillerie — Dosage gravimétrique par la diméthylglyoxime*



Reference number
ISO 11490:1995(E)

Foreword

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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.

International Standard ISO 11490 was prepared by Technical Committee ISO/TC 174, *Jewellery*.

Annex A of this International Standard is for information only.

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Determination of palladium in palladium jewellery alloys — Gravimetric determination with dimethylglyoxime

1 Scope

This International Standard specifies a gravimetric method for the determination of palladium in palladium jewellery alloys, preferably within the range of fineness stated in ISO 9202.

These alloys which may contain silver, indium, gallium, copper, cobalt, nickel, tin and ruthenium. Coprecipitated elements have to be determined by a suitable method and a correction applied.

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 sample is dissolved in *aqua regia*. Palladium is precipitated with dimethylglyoxime. If present, silver is separated as silver chloride. The palladium dimethylglyoxime compound is converted to metallic palladium by ignition and the latter is then determined gravimetrically.

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, 69 % (m/m), $\rho_{20} = 1,41 \text{ g/cm}^3$.

4.2 Hydrochloric acid, 38 % (m/m), $\rho_{20} = 1,19 \text{ g/cm}^3$.

4.3 Dilute hydrochloric acid, 8,5 % (m/m), $\rho_{20} = 1,04 \text{ g/cm}^3$.

4.4 Dimethylglyoxime solution.

Dissolve 10 g of dimethylglyoxime in 1 litre of ethanol.

4.5 Ammonium chloride.

4.6 Dilute nitric acid, 1,39 % (m/m), $\rho_{20} = 1,00 \text{ g/cm}^3$.

Cautiously add 10 ml of nitric acid (4.1) to 1 000 ml of water and mix.

4.7 Hydrofluoric acid, 40 % (m/m), $\rho_{20} = 1,13 \text{ g/cm}^3$.

4.8 Dilute sulfuric acid, 49 % (m/m), $\rho_{20} = 1,4 \text{ g/cm}^3$.

Cautiously add 100 ml of sulfuric acid [98 % (m/m), $\rho_{20} = 1,86 \text{ g/cm}^3$] to 100 ml of water and mix.

4.9 Reducing gas, such as hydrogen or a hydrogen/nitrogen mixture.

4.10 Inert gas under pressure, carbon dioxide or nitrogen are usual.

4.11 Aqua regia.

Mix 3 volumes of hydrochloric acid (4.2) and 1 volume of nitric acid (4.1).

WARNING — Aqua regia is potentially hazardous and safety glasses or goggles must be used. Dissolution should be carried out in a well-ventilated fume cupboard.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Reduction apparatus, see figure A.1 in annex A.

5.2 Platinum dishes, of volume 10 ml.

5.3 Atomic absorption or inductively coupled plasma (ICP) emission spectrometer, or other means of determining traces of metal.

5.4 Muffle furnace, capable of attaining at least 900 °C.

5.5 Ashless filter paper, capable of retaining particles greater than 3 µm.

6 Sampling

The sampling procedure for palladium alloys shall be agreed upon until a 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 Flatten the sample to less than 0,5 mm thick and weigh a sample containing 150 mg to 200 mg of palladium accurately to $\pm 0,01$ mg. Transfer it to a 800 ml tall-form beaker. Add 10 ml of nitric acid (4.1) and heat at 70 °C to 80 °C for 20 min in the beaker covered with a watch glass, before adding 30 ml of hydrochloric acid (4.2) to complete the dissolution.

7.2 If insoluble silver chloride forms, break this up with a glass rod to ensure that all the metal is dissolved.

7.3 Remove the watch glass and gently evaporate to dryness. Dissolve the residue in 10 ml of hydrochloric acid (4.2) and dilute to about 100 ml.

7.4 If a precipitate forms, allow it to settle for 12 h in a dark place. Filter and wash with dilute nitric acid (4.6), retaining the precipitate for the determination of traces of palladium using suitable apparatus (5.3).

7.5 Add 20 ml of hydrochloric acid to the clear solution from 7.3 (or filtrate and washings from 7.4). Dilute to approximately 400 ml, cool to 15 °C and add dimethylglyoxime solution in 5 ml portions up to a total of 30 ml for every expected 100 mg of palladium.

7.6 Leave to settle for 1 h, filter and wash with dimethylglyoxime solution (4.4) diluted 10 times with water. Retain the combined filtrate and washes for determination of palladium using suitable apparatus (5.3) and correct the final result.

7.7 Transfer the precipitate and filter to a tared porcelain crucible. Tap the filter to obtain a flat surface and dry in an oven at 110 °C to 120 °C for 3 h. Cover with a layer of ammonium chloride (4.5) about 3 mm thick (about 4 g for a crucible of diameter 40 mm) to prevent loss of palladium during ignition.

7.8 Heat the crucible gently (for about 40 min) first to char the paper and then to decompose the palladium complex and drive off the ammonium chloride. When all fuming has ceased, ignite at 800 °C $\pm$ 50 °C for 1 h.

NOTE 1 The ammonium chloride decomposes at 340 °C.

7.9 The partially oxidized palladium is reduced to the metallic state by heating in the presence of reducing gas (4.9) and allowing to cool in an inert gas atmosphere (4.10).

7.10 Weigh the product to obtain an approximate mass of palladium.

7.11 Transfer the impure palladium to a platinum dish (5.2). Moisten with hydrofluoric acid (4.7), and add 3 drops of dilute sulfuric acid (4.8). Heat until fumes start to evolve from the solution. Cool, then extract the residue with a little hot water. Filter and wash with water. Combine the filtrate and washes with those from the previous filtration. Transfer the palladium and filter to a crucible, ignite at approxi-

mately 700 °C and reduce as described in 7.9. Re-weigh to obtain the final mass.

7.12 If contamination of the palladium is suspected, it shall be dissolved in *aqua regia* (4.11), the elements determined by a spectrometric method and their mass subtracted from the final mass of palladium; or the palladium shall be cleaned by repeating the process in 7.1 to 7.11.

7.13 The combined filtrates and washes are tested for palladium by instrumental means, usually an atomic absorption or ICP emission spectrometer (5.3). Excess dimethylglyoxime is destroyed by evaporation to dryness then treating the residue with *aqua regia*. The resulting solution is compared with standard palladium solutions containing equivalent quantities of acids and spectroscopic buffers.

8 Expression of results

8.1 Method of calculation

8.1.1 If the final weighed mass contains exclusively palladium, calculate the palladium content w_{Pd} , in parts by mass per thousand (‰), using the formula

$$w_{\text{Pd}} = \frac{m_3 + m_2}{m_1} \times 10^3$$

where

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

m_2 is the mass, in milligrams, of the filtrate;

m_3 is the final mass, in milligrams.

8.1.2 If the final weighed mass contains other elements, calculate the palladium content w_{Pd} , in parts by mass per thousand (‰), using the formula

$$w_{\text{Pd}} = \frac{m_3 + m_2 - m_X}{m_1} \times 10^3$$

where

m_X is the total mass, in milligrams, of other elements.

8.1.3 The palladium content w_{Pd} , in parts by mass per thousand (‰), is corrected for the mass, m_Y , of palladium in the silver chloride precipitated in 7.4 using the formula

$$w_{\text{Pd}} = \frac{m_3 + m_2 - m_X + m_Y}{m_1} \times 10^3$$

where m_Y is the mass, in milligrams, of palladium in the silver chloride.

8.2 Repeatability

The results of duplicate determinations shall correspond to better than 5 parts per mass per thousand (‰) of palladium. If the variation is greater than this, the assays shall be repeated.

9 Test report

The test report shall include the following information:

- identification of the sample including source, date of receipt, form of sample;
- sampling procedure;
- the method used by reference to this International Standard;
- palladium content of the sample, in parts by mass per thousand (‰) as single values and mean values;
- if relevant, any deviations from the method specified in this International Standard;
- any unusual features observed during the determination;
- date of test;
- identification of the laboratory carrying out this analysis;
- signature of the laboratory manager and operator.

Annex A

(informative)

Reduction apparatus according to Rose

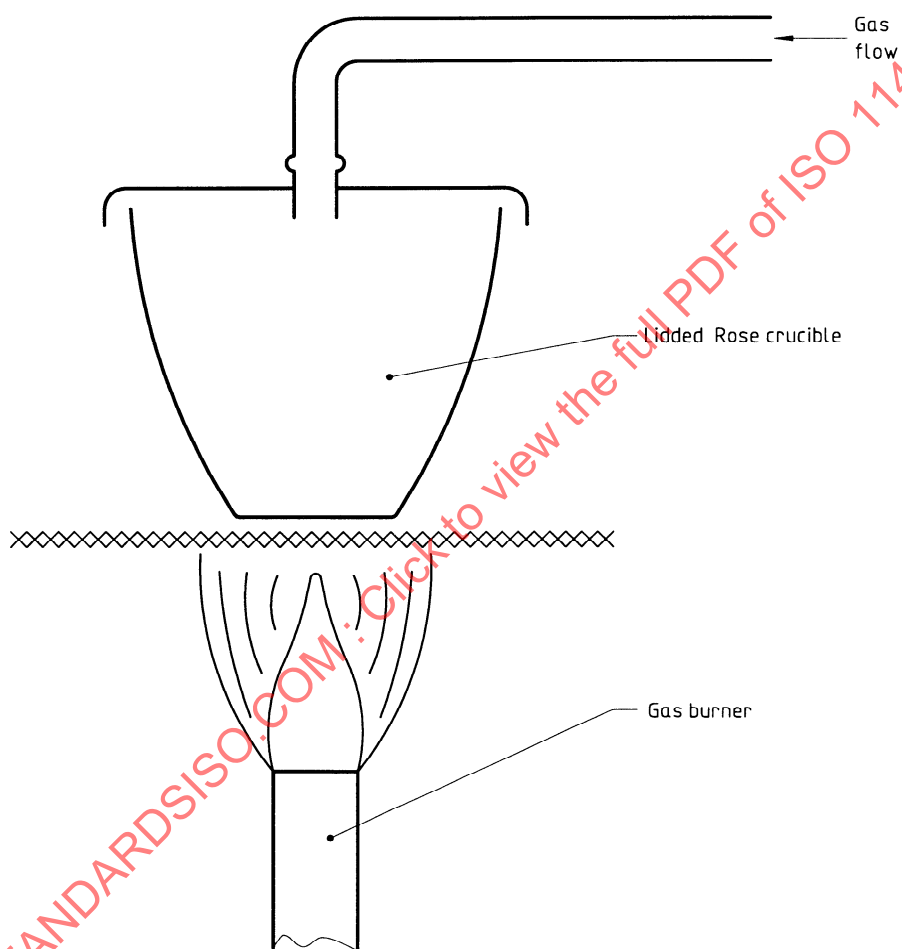


Figure A.1 — Reduction apparatus