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Jewellery and precious metals — Determination of palladium — Gravimetry using dimethylglyoxime

*Joellerie, bijouterie et métaux précieux — Dosage du palladium —
Méthode gravimétrique après précipitation par la diméthylglyoxime*

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Contents

	Page
Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	1
5 Reagents	1
6 Apparatus	2
7 Sampling	2
8 Procedure	2
8.1 Preliminary analysis.....	2
8.2 Preparation of samples free of silver.....	3
8.3 Preparation of samples containing silver.....	3
8.4 Precipitation of palladium with dimethylglyoxime.....	3
9 Calculation and expression of results	4
9.1 Calculation.....	4
9.2 Repeatability.....	4
10 Test report	4
Annex A (informative) Reduction apparatus example	5
Bibliography	6

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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 the European Committee for Standardization (CEN) Technical Committee CEN/SSM21, *Precious metals — Applications in jewellery and associated products*, in collaboration with ISO Technical Committee TC 174, *Jewellery*, in accordance with the agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 11490:2015), which has been technically revised.

The main changes are as follows:

- extension of the scope of application to all precious metal alloys beyond the jewellery sector;
- clarification of the fineness for which the test is suitable;
- addition of a specific preparation for samples containing a significant amount of silver in [Clause 8](#);
- change of precipitation method in [Clause 8](#);
- suppression of the use of hydrofluoric acid and sulfuric acid;
- harmonization of method with ISO 11210.

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 palladium — Gravimetry using dimethylglyoxime

1 Scope

This document specifies a gravimetric method for the determination of palladium on a material considered homogeneous. The palladium content of the sample lies preferably between 50 and 999 parts per thousand (‰) by mass. Fineness above 999 ‰ can be determined using a spectroscopy method by difference (e.g. ISO 15093).

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

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <https://www.electropedia.org/>

3.1

palladium sponge

palladium obtained after calcination of the palladium dimethylglyoxime precipitate

4 Principle

The sample is dissolved in aqua regia. Palladium is precipitated with dimethylglyoxime. The palladium dimethylglyoxime complex precipitate is converted by ignition to metallic palladium which is weighed.

If present, silver is separated as silver chloride.

Co-precipitated alloying elements are tested in the re-dissolved palladium sponge and measured using, for example, an inductively coupled plasma optical emission spectrometer (ICP-OES), and a correction applied.

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 Hydrochloric acid (HCl), with a mass fraction of approximately 30 % to 37 % of HCl.

5.2 Diluted hydrochloric acid, consisting of a mix of one volume of hydrochloric acid (5.1) and one volume of water.

- 5.3 **Nitric acid (HNO₃)**, with a mass fraction of approximately 65 % to 70 % of HNO₃.
- 5.4 **Diluted nitric acid**, consisting of a mix of one volume of nitric acid (5.3) and one volume of water.
- 5.5 **Aqua regia**, consisting of a mix of three volumes of hydrochloric acid (5.1) and one volume of nitric acid (5.3).
- 5.6 **Ethanol**, with a mass fraction of 96 %.
- 5.7 **Dimethylglyoxime**.
- 5.8 **Saturated aqueous solution of dimethylglyoxime**, obtained by mixing dimethylglyoxime (5.7) in boiling water until dimethylglyoxime does not dissolve anymore, and letting the solution stay overnight.
- 5.9 **Saturated ethanol solution of dimethylglyoxime**, obtained by mixing dimethylglyoxime (5.7) in ethanol (5.6) until dimethylglyoxime does not dissolve anymore, and letting the solution stay overnight.
- 5.10 **Reducing gas**, such as hydrogen or a hydrogen/nitrogen mixture.
- 5.11 **Inert gas** such as carbon dioxide or nitrogen.

6 Apparatus

- 6.1 **Customary laboratory apparatus**.
- 6.2 **Reduction apparatus**, see [Figure A.1](#).
- 6.3 **Porcelain crucibles**, of preferably 20 ml to 45 ml volume.
- 6.4 **ICP-OES**, capable of determining traces of metals.
- 6.5 **Muffle furnace**, capable of attaining at least 900 °C.
- 6.6 **Ashless filter paper**, capable of retaining particles greater than 3 µm.
- 6.7 **Analytical balance**, with a reading accuracy of 0,01 mg.

7 Sampling

The sampling procedure should be performed in accordance with ISO 11596.

8 Procedure

8.1 Preliminary analysis

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 (X-ray fluorescence) analysis.

8.2 Preparation of samples free of silver

Flatten the sample (if necessary) to less than 0,5 mm thick, weigh at least two samples of the alloy containing approximately 250 mg of palladium accurately to 0,01 mg and transfer it to a glass beaker (preferably of 150 ml).

Sample mass may be increased to contain up to 1 g of palladium, provided all other masses and volumes are adapted. The palladium precipitate is voluminous and this aspect should be considered while deciding the mass of the sample.

Add 10 ml to 15 ml of aqua regia (5.5), dissolve on a hot plate. After dissolution, evaporate to 5 ml to 7 ml and add few drops of hydrochloric acid (5.1). Allow to cool.

Dissolution may be performed in a sealed container under pressure. In that case, the volume of aqua regia (5.5) may be adapted to optimize the dissolution.

8.3 Preparation of samples containing silver

Flatten the sample (if necessary) to less than 0,5 mm thick weigh at least two samples of the alloy containing approximately 250 mg of palladium accurately to 0,01 mg and transfer it to a glass beaker (preferably of 150 ml).

Add 10 ml of diluted nitric acid (5.4), cover the beaker with a watch glass and heat at 70 °C to 80 °C for 20 min. Add 30 ml of hydrochloric acid (5.1). An insoluble silver chloride is formed. Heat for good coagulation of the precipitate and evaporate the solution to 20 ml. Allow it to settle for 12 h in a dark place.

Filter the supernatant solution and keep the precipitate into the beaker. Collect the filtrate in a 250 ml beaker. If the silver chloride precipitate is coloured yellow, add the 1 ml to 2 ml of diluted hydrochloric acid (5.2), boil for 1 min to 2 min, filter the solution and repeat until the precipitate is white. Wash the precipitate with water.

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8.4 Precipitation of palladium with dimethylglyoxime

Transfer the solution (from 8.1) or the filtrate (from 8.2) into a 1 000 ml Erlenmeyer flask. Add 100 ml of diluted hydrochloric acid (5.2) and mix well. Add 100 ml of saturated aqueous dimethylglyoxime solution (5.8) and mix well. Add hot water to an approximate volume of 450-500 ml and mix well. Add 100 ml of saturated ethanol dimethylglyoxime solution (5.9) and mix well. Add hot water to an approximate volume of 750 to 800 ml.

Leave to settle for 1 h, filter over a filter paper (6.6) and wash with 600 ml to 700 ml of hot water. Collect the filtrate for determination of palladium by ICP-OES (6.4). Alternative filtration methods may be used, such as vacuum filtration on a Gooch funnel. Wipe the Erlenmeyer flask with a second filter paper (6.6). Transfer the precipitate and the filter paper to a porcelain crucible (6.3). Tap the filter paper to obtain a flat surface and dry in an oven at 110 °C to 120 °C for 3 h.

Heat the crucible gently (for about 40 min) first to char the paper and then to decompose the palladium complex. When all fuming has ceased, ignite at 800 °C ± 50 °C for 1 h.

Significant absorption of oxygen can take place during ignition. Oxidation can be avoided by calcining under reducing gas (5.10), using the device or an equivalent presented in Annex A, followed by cooling under inert gas (5.11) or reducing gas (5.10). Not using a reducing gas can lead to the presence of a large quantity of oxides. In that case, the partially oxidized palladium shall be reduced to the metallic state by heating it in presence of reducing gas (5.10).

Weigh the palladium sponge obtained.

NOTE 1 The filter paper can be transferred into a previously weighed crucible placed into a larger crucible. The mass of the palladium sponge can be determined by weighing the crucible with the sponge and by deducting the crucible weight.

NOTE 2 The crucible with the sponge should be stored in a desiccator before weighing the sponge.

Dissolve the palladium sponge in 20 ml of aqua regia (5.5). Measure the co-precipitated elements by suitable means such as ICP-OES (6.4), using calibration solutions with a composition similar to the composition of the palladium sponge.

9 Calculation and expression of results

9.1 Calculation

If the final weighed mass contains exclusively palladium, calculate the palladium content, W_{Pd} , in parts by mass per thousand (‰) using [Formula \(1\)](#):

$$W_{Pd} = \frac{m_3 + m_2}{m_1} \cdot 10^3 \quad (1)$$

where

m_1 is the mass of the sample, in milligrams;

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

m_3 is the final mass of the palladium sponge, in milligrams.

If the final weighed mass contains other elements, calculate the palladium content, W_{Pd} , in parts by mass per thousand (‰) using [Formula \(2\)](#):

$$W_{Pd} = \frac{m_3 + m_2 - m_x}{m_1} \cdot 10^3 \quad (2)$$

where m_x is the total mass of other elements contained in the palladium sponge, in milligrams. [77/iso-fdis-11490](#)

9.2 Repeatability

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

10 Test report

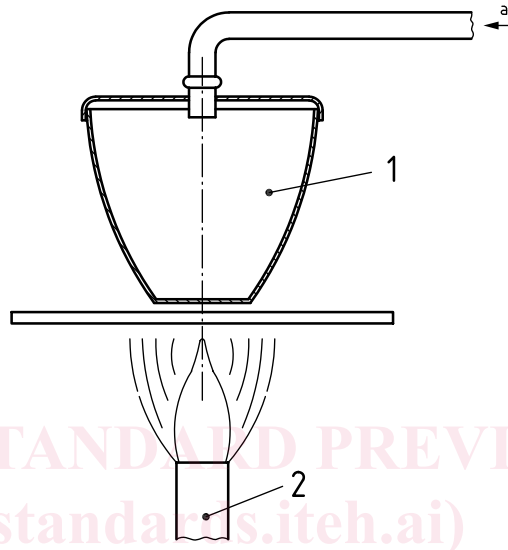
The test report shall include at least the following information:

- a) identification of the sample including source, date of receipt, form of sample;
- b) sampling procedure;
- c) method used by reference to this document, i.e. ISO 11490:—;
- d) palladium content of the sample, in parts per thousand (‰), by mass, as single values and mean values;
- e) if relevant, any deviations from the method specified in this document;
- f) any unusual features observed during the determination;
- g) date of test;
- h) identification of the laboratory carrying out this analysis;
- i) signature of the laboratory manager and operator.

Annex A (informative)

Reduction apparatus example

Figure A.1 shows an example of a reduction apparatus.



Key

1 crucible

2 gas burner

a Gas flow.

Figure A.1 — Reduction apparatus

Bibliography

- [1] ISO 9202, *Jewellery and precious metals — Fineness of precious metal alloys*
- [2] ISO 11596, *Jewellery and precious metals — Sampling of precious metals and precious metal alloys*
- [3] ISO 15093, *Jewellery and precious metals — Determination of high purity gold, platinum and palladium — Difference method using ICP-OES*

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