SLOVENSKI STANDARD
oSIST prEN ISO 11490:2022

01-julij-2022

Nakit in plemenite kovine - Določevanje paladija v zlitinah paladija - Gravimetrično določanje po obarjanju z dimetilglioksimom (ISO/DIS 11490:2022)

Jewellery and precious metals - Determination of palladium in palladium alloys - Gravimetric determination after precipitation using dimethylglyoxime (ISO/DIS 11490:2022)

Schmuck und Edelmetalle - Bestimmung von Palladium in Palladiumlegierungen - Gravimetrische Bestimmung nach Fällung mit Dimethylglyoxim (ISO/DIS 11490:2022)

Joaillerie, bijouterie et métaux précieux - Dosage du palladium dans les alliages de palladium - Méthode gravimétrique après précipitation par la diméthylglyoxime (ISO/DIS 11490:2022)

Ta slovenski standard je istoveten z: **prEN ISO 11490**

ICS:

39.060	Nakit	Jewellery

oSIST prEN ISO 11490:2022 **en,fr,de**

DRAFT INTERNATIONAL STANDARD

ISO/DIS 11490

ISO/TC **174**

Secretariat: **DIN**

Voting begins on:
2022-05-25

Voting terminates on:
2022-08-17

Jewellery and precious metals — Determination of palladium in palladium alloys — Gravimetric determination after precipitation using dimethylglyoxime

Joaillerie, bijouterie et métaux précieux — Dosage du palladium dans les alliages de palladium— Méthode gravimétrique après précipitation avec de la diméthylglyoximeue

ICS: 39.060

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 11490:2022(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

The committee responsible for this document is ISO/TC 174, *Jewellery.*

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

— 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 in palladium alloys — Gravimetric determination after precipitation using dimethylglyoxime

1 Scope

This document specifies a gravimetric method for the determination of palladium in palladium alloys. The palladium content of the sample lies preferably between 50 and 999 parts per thousand (‰). 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

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 11596, *Jewellery and precious metals — Sampling of precious metals and precious metal alloys*

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 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 to metallic palladium by ignition and the latter is then determined gravimetrically.

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), approximately mass fraction of 30 % to 37 % HCl.

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

5.3 Nitric acid (HNO_3), approximately mass fraction of 65 % to 70 % HNO_3.

5.4 Diluted nitric acid, mix one volume of nitric acid (5.3) and one volume of water.

5.5 Aqua regia. Mix three volumes of hydrochloric acid (5.1) and one volume of nitric acid (5.3).

5.6 Ethanol, mass fraction of 96 %.

5.7 Dimethylglyoxime.

5.8 Saturated aqueous solution of dimethylglyoxime, mix dimethylglyoxime (5.7) in boiling water until dimethylglyoxime does not dissolve anymore and let stay overnight.

5.9 Saturated ethanol solution of dimethylglyoxime, mix dimethylglyoxime (5.7) in ethanol (5.6) until dimethylglyoxime does not dissolve anymore and let 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 20 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

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.

8.1 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 150 ml glass beaker.

NOTE 1 Sample weight can be increased to 1 g, provided all other weights and volumes are adapted. The palladium precipitate is voluminous and this aspect should be taken into account while deciding the weight 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.

NOTE 2 Dissolution can be performed in a sealed container under pressure. In that case, the volume of aqua regia (5.5) can be adapted to optimize the dissolution.

8.2 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 150 ml glass beaker.

Add 10 ml of diluted nitric acid (5.3), 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 forms has 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 to 2 ml of hydrochloric diluted acid (5.4), boil for 1 to 2 min, filter the solution and repeat until the precipitate is white. Wash the precipitate with water.

NOTE For alloys containing a significant amount of silver, a pre-digestion with nitric acid (5.3) can be performed as an alternative method.

8.3 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 (5.4) 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). 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.

NOTE Alternative filtration methods can be used, such as vacuum filtration on a Gooch funnel.

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 (5.11) or 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 is placed in a desiccator before weighing the sponge.

If contamination of the palladium is suspected, 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.

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 this document, ISO/DIS 11490:2022;

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;