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

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

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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary Information.

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

This second edition cancels and replaces the first edition (ISO 11490:1995), which has been technically revised with the following changes:

a) addition of an analytical balance in Clause 5;

b) change of requirement for sampling in Clause 6;

c) addition of a warning in Clause 7 that suitable health and safety procedures should be followed;

d) standard editorially revised.

Introduction

The following definitions apply in understanding how to implement an ISO International Standard and other normative ISO deliverables (TS, PAS, IWA):

— “shall” indicates a requirement;

— “should” indicates a recommendation;

— “may” is used to indicate that something is permitted;

— “can” is used to indicate that something is possible, for example, that an organization or individual is able to do something.

ISO/IEC Directives, Part 2 (sixth edition, 2011), 3.3.1 defines a requirement as an “expression in the content of a document conveying criteria to be fulfilled if compliance with the document is to be claimed and from which no deviation is permitted.”

ISO/IEC Directives, Part 2 (sixth edition, 2011), 3.3.2 defines a recommendation as an “expression in the content of a document conveying that among several possibilities, one is recommended as particularly suitable without mentioning or excluding others, or that a certain course of action is preferred, but not necessarily required, or that (in the negative form) a certain possibility or course of action is deprecated, but not prohibited.”

Jewellery — 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 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 references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 — Sampling of precious metal alloys for and in jewellery and associated products*

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

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

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

4.3 Hydrochloric acid (HCl), 30 % to 37 % HCl (mass fraction).

4.4 Diluted hydrochloric acid, 8,5 % (mass fraction).

4.5 Dimethylglyoxime solution.

Dissolve 10 g of dimethylglyoxime in 1 000 ml of ethanol.

4.6 Ammonium chloride.

4.7 Diluted nitric acid, 1,39 %.

Cautiously add 10 ml of nitric acid (4.2) to 1 000 ml of water and mix.

4.8 Hydrofluoric acid, 40 % (mass fraction).

4.9 Diluted sulphuric acid, 50 % (mass fraction).

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

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

4.12 Aqua regia.

Mix three volumes of hydrochloric acid (4.3) and one volume of nitric acid (4.2).

5 Apparatus

5.1 Customary laboratory apparatus.

5.2 Reduction apparatus, see Figure A.1.

5.3 Platinum dishes, of volume 10 ml.

5.4 AAS or **ICP-OES**, or other means of determining traces of metal.

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

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

5.7 Analytical balance, with a reading accuracy of 0,01 mg.

6 Sampling

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

7 Procedure

WARNING — Suitable health and safety procedures should be followed.

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 0,01 mg. Transfer it to an 800 ml tall-form beaker. Add 10 ml of nitric acid (4.2) 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 (4.3) 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 (4.3) 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 (4.7), retaining the precipitate for the determination of traces of palladium using suitable apparatus (5.4).

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 (4.5) diluted 10 times with water. Retain the combined filtrate and washes for determination of palladium using suitable apparatus (5.4) 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 (4.6) 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 ± 50 °C for 1 h.

NOTE 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 (4.10) and allowing to cool in an inert gas atmosphere (4.11).

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

7.11 Transfer the impure palladium to a platinum dish (5.3). Moisten with hydrofluoric acid (4.8) and add three drops of dilute sulfuric acid (4.9). 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 approximately 700 °C, and reduce as described in 7.9. Reweigh to obtain the final mass.

7.12 If contamination of the palladium is suspected, it shall be dissolved in aqua regia (4.12). 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 AAS or ICP-OES (5.4). Excess dimethylglyoxime is destroyed by evaporation to dryness, then treating the residue with aqua regia (4.12). The resulting solution is compared with standard palladium solutions containing equivalent quantities of acids and spectroscopic buffers.

8 Calculation and expression of results

8.1 Calculation

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

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

where

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

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

m_3 is the final mass, 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).

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

where

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

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 Formula (3).

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

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 five 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:

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, i.e. ISO 11490;

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

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

f) any unusual features observed during the determination;

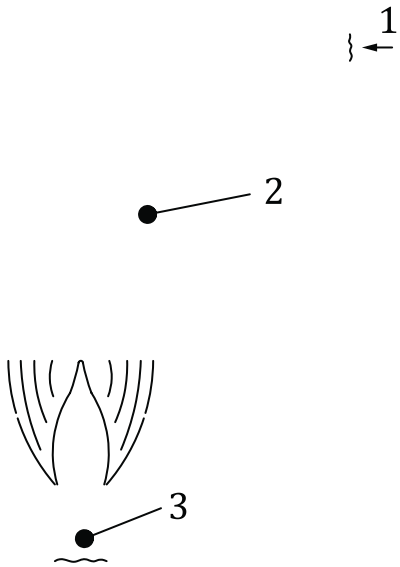
g) date of test;

h) identification of the laboratory carrying out this analysis;

i) signature of the laboratory manager and operator.

Annex A
(informative)

Reduction apparatus according to Rose



Key

1 gas flow
2 lidded Rose crucible
3 gas burner

Figure A.1 — Reduction apparatus

Bibliography

[1] ISO 9202:1991, *Jewellery — Fineness of precious metal alloys*

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