SLOVENSKI STANDARD
oSIST prEN ISO 11490:2016

01-junij-2016

Nakit - Določevanje paladija v zlitinah za nakit iz paladija - Gravimetrična metoda z dimetil glioksimom (ISO 11490:2015)

Jewellery - Determination of palladium in palladium jewellery alloys - Gravimetric determination with dimethylglyoxime (ISO 11490:2015)

Schmuck - Bestimmung von Palladium in Palladium-Schmucklegierungen - Gravimetrische Bestimmung mit Dimethylglyoxim (ISO 11490:2015)

Joaillerie - Dosage du palladium dans les alliages de palladium pour la bijouterie-joaillerie - Dosage gravimétrique par la diméthylglyoxime (ISO 11490:2015)

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39.060	Nakit	Jewellery

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INTERNATIONAL STANDARD

ISO 11490

Second edition
2015-02-01

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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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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).

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 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.

