INTERNATIONAL STANDARD

ISO 11210

Second edition
2014-12-01

Jewellery — Determination of platinum in platinum jewellery alloys — Gravimetric method after precipitation of diammonium hexachloroplatinate

Joaillerie — Dosage du platine dans les alliages de platine pour la bijouterie-joaillerie — Méthode gravimétrique après précipitation de l'hexachloroplatinate de diammonium

Reference number
ISO 11210:2014(E)

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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).

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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 11210:1995), which has been technically revised with the following changes:

- addition of an analytical balance in Clause 5;
- change of requirement for sampling in Clause 6;
- addition in 7.1 to allow dissolution in a sealed container under pressure;
- addition in 7.1 to allow drying of the crucibles in a programmable furnace;
- deletion of the temperature for reduction in 7.1;
- International Standard editorially revised.

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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."

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INTERNATIONAL STANDARD **ISO 11210:2014(E)**

Jewellery — Determination of platinum in platinum jewellery alloys — Gravimetric method after precipitation of diammonium hexachloroplatinate

1 Scope

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

These alloys can contain palladium, iridium, rhodium, copper, cobalt, gold, ruthenium, gallium, chromium, indium, and less than 5 % tungsten. Some modifications are indicated where palladium, iridium, rhodium, gold, or ruthenium are present.

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. After converting the sample solution to a slightly acid medium, the platinum is precipitated as diammonium hexachloroplatinate. The precipitate is converted to metallic platinum. Coprecipitated alloying elements are tested in the re-dissolved platinum sponge and measured using, for example, an atomic absorption spectrometer (AAS) or an inductively coupled plasma optical emission spectrometer (ICP-OES), and a correction applied.

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

4.2 Dilute hydrochloric acid, 18 % HCl (mass fraction).

4.3 Nitric acid (HNO_3), approximately 65 % to 69 % HNO_3 (mass fraction).

4.4 Ammonium chloride solution (NH_4Cl), cold saturated solution.

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

4.6 Inert gas, such as carbon dioxide or nitrogen.

4.7 Aqua regia, mix three volumes of hydrochloric acid (4.1) and one volume of nitric acid (4.3).

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5 Apparatus

5.1 Ordinary laboratory apparatus.

5.2 Reduction apparatus, see Figure A.1.

5.3 AAS or ICP-OES, capable of determining traces of metals.

5.4 Muffle furnace, capable of attaining at least 1 000 °C.

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

5.6 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 Platinum jewellery alloys with less than 5 % iridium, rhodium, ruthenium, or tungsten

Weigh the sample containing approximately 250 mg to 300 mg of platinum, accurately to the nearest 0,01 mg, and transfer it to a 100 ml glass beaker. Dissolve the sample in 20 ml of aqua regia (4.7) in the glass beaker covered with a watch glass, while heating gently, or in a sealed container under pressure.

Any insoluble material remaining after this procedure shall be filtered off at this stage and its composition established for possible correction of the results.

Evaporate the solution five times without letting the residue become dry and without exceeding a temperature of 90 °C, each time adding 2 ml of dilute hydrochloric acid (4.2) before recommencing evaporation.

If this temperature is exceeded, the platinum can be reduced to Pt(II) or even Pt(I) and require re-oxidation with approximately 0,1 ml of nitric acid (4.3). After the last evaporation, dissolve the still moist platinum salt in 1 ml of dilute hydrochloric acid (4.2) and add 4 ml of water. Add 40 ml of saturated ammonium chloride solution (4.4) at (85 ± 5) °C to this clear solution. The platinum is precipitated as yellow $(NH_4)_2[PtCl_6]$. The solution with the precipitated $(NH_4)_2[PtCl_6]$ is evaporated almost to dryness at this temperature. Further gentle heating shall continue until hydrogen chloride is no longer emitted. Allow to cool. Add just sufficient water while agitating to dissolve the residual ammonium chloride crystals.

Immediately filter the $(NH_4)_2[PtCl_6]$ precipitate over a filter paper (5.5) which has been moistened with ammonium chloride solution (4.4). Thoroughly wash the precipitate with ammonium chloride solution. Wipe the glass beaker and watch glass with a second filter paper. Check the filtrate for residual platinum by suitable means, such as an AAS or ICP-OES (5.2).

The filter paper containing the precipitate is folded into the second one, transferred to a porcelain crucible and covered with a thin layer (2 mm) of ammonium chloride. This crucible is then placed inside a covered crucible. Cautiously dry the contents of the crucibles on a hotplate which can be gradually adjusted or in a programmable furnace. By drying initially at 50 °C to 70 °C and heating subsequently to 340 °C, all ammonium chloride is lost. Ash the filter paper (5.5) and $(NH_4)_2[PtCl_6]$ residue at a temperature of 500 °C to 600 °C without a flame. Finally, remove the cover of the crucible and calcine in a muffle furnace

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(5.4) for 1 h to 3 h at a temperature of 900 °C to 1 000 °C. To ensure complete decomposition, the solid in the crucible might need breaking into several pieces during calcination.

NOTE The ammonium chloride decomposes at 340 °C.

Significant absorption of oxygen can take place during ignition if palladium, iridium, ruthenium, rhodium, or copper are present in the platinum sponge. Oxidation can be avoided by calcining under reducing gas (4.5) followed by cooling under inert or reducing gas (4.6).

Weigh the platinum sponge obtained.

Measure the co precipitated elements by suitable means such as AAS or ICP-OES (5.3).

7.2 Platinum jewellery alloys with more than 5 % iridium, rhodium, or ruthenium, or more than 0,5 % gold

The procedure described results in the complete precipitation of the platinum in the sample. However, under certain conditions, some palladium might precipitate as tetrachloropalladiate $(NH_4)_2[PdCI_4]$ with the $(NH_4)_2[PtCI_6]$ and iridium, rhodium, and ruthenium might co precipitate. In addition, gold, if present in quantities in excess of about 5 parts per thousand, can precipitate as diammonium trichloroaurate $(NH_4)_2[AuCl_3]$ with ammonium chloride. Copper also has a tendency to precipitate in solutions with a high concentration of ammonium chloride.

The presence of those impurities in the final platinum sponge weighed can be identified following dissolution in 20 ml of aqua regia (4.7), using, for example, AAS or ICP-OES (5.3).

If more than 0,5 % gold is present in the sample, it shall be separated before the precipitation of platinum. This is done by a reductive precipitation with sulfur dioxide in the hydrochloric acid solution after the nitric acid has been driven off. Sulfur dioxide gas is passed through the solution until no more gold precipitates. The precipitate is filtered off and can be weighed, if required.

If platinum alloys contain more than 10 % iridium or rhodium or more than 5 % ruthenium, dissolution of the sample in the aqua regia (4.7) can require operating in a sealed container under pressure.

Ruthenium shall be removed from the resulting solution, before the stage when platinum is precipitated, by passing chlorine through the solution and by filtration of the precipitate.

8 Methods of calculation and expression of results

8.1 Calculation

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

$$W_{\mathrm{Pt}} = \frac{m_3 + m_2}{m_1} \cdot 10^3 \qquad (1)$$

where

m_1 is the mass of the sample, in milligrams;

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

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

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If the final weighed mass contains other elements, calculate the platinum content, W_{Pt}, in parts by mass per thousand (‰) using Formula (2):

$$W_{\mathrm{Pt}} = \frac{m_3 + m_2 - m_{\mathrm{x}}}{m_1} \cdot 10^3 \tag{2}$$

where

m_{x} is the total mass of other elements contained in the platinum sponge, in milligrams.

8.2 Repeatability

The results of duplicate determinations shall correspond to better than three parts per mass per thousand (‰) of platinum. 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) method used by reference to this International Standard;

d) platinum 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 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.

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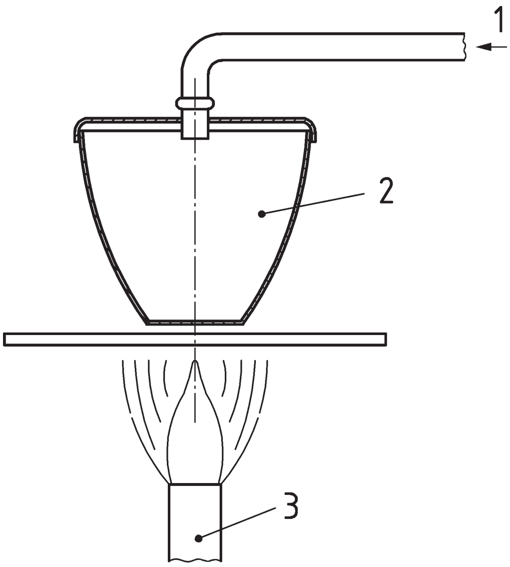
Annex A
(informative)

Reduction apparatus according to Rose



Key

1	gas flow
2	lidded Rose crucible
3	gas burner

Figure A.1 — Reduction apparatus

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