SLOVENSKI STANDARD
oSIST prEN ISO 11210:2022

01-julij-2022

Nakit in plemenite kovine - Določevanje platine v zlitinah platine - Gravimetrično določanje po obarjanju z amonijevim kloridom (ISO/DIS 11210:2022)

Jewellery and precious metals - Determination of platinum in platinum alloys - Gravimetric determination after precipitation using ammonium chloride (ISO/DIS 11210:2022)

Schmuck und Edelmetalle - Bestimmung von Platin in Platinlegierungen - Gravimetrisches Verfahren nach Fällung mittels Ammoniumchlorid (ISO/DIS 11210:2022)

Joaillerie, bijouterie et métaux précieux - Dosage du platine dans les alliages de platine - Méthode gravimétrique après précipitation par le chlorure d’ammonium (ISO/DIS 11210:2022)

Ta slovenski standard je istoveten z: **prEN ISO 11210**

ICS:

39.060	Nakit	Jewellery

oSIST prEN ISO 11210:2022 **en,fr,de**

DRAFT INTERNATIONAL STANDARD
ISO/DIS 11210

ISO/TC **174** Secretariat: **DIN**

Voting begins on:
2022-05-25

Voting terminates on:
2022-08-17

Jewellery and precious metals — Determination of platinum in platinum alloys — Gravimetric determination after precipitation using ammonium chloride

ICS: 39.060

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 11210:2022(E)

COPYRIGHT PROTECTED DOCUMENT

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

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 11210:2014), 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;
- Addition of an alternative method for the filtration in Clause 8;
- Harmonization of method with ISO 11490.

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 platinum in platinum alloys — Gravimetric determination after precipitation using ammonium chloride

1 Scope

This document specifies a gravimetric method for the determination of platinum in platinum alloys. The platinum content of the sample lies preferably between 50 and 999 parts per thousand (‰) by weight. 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
platinum sponge
platinum obtained after calcination of the diammonium hexachloroplatinate precipitate

3.2
vacuum system
vacuum filtration system for the filtration of one or several samples simultaneously

EXAMPLE Buchner filtration system.

4 Principle

The sample is dissolved in aqua regia. Platinum is precipitated as diammonium hexachloroplatinate. The precipitate is converted to metallic platinum 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 platinum 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 (HCI), approximately mass fraction of 30 % to 37 % HCI.

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

5.5 Ammonium chloride (NH_4CI)

5.6 Saturated ammonium chloride solution, mix ammonium chloride (5.5) in water until ammonium chloride does not dissolved anymore and let stay overnight.

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

5.8 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.

NOTE For complex alloys containing particular elements (such as tungsten, silicon, titanium), dissolution of the sample can be difficult. The following procedure can be adapted to ensure a complete dissolution of the sample.

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 to 1 g of platinum accurately to 0,01 mg and transfer it to a 150 ml glass beaker.

Add 20 ml of aqua regia (5.4), cover the glass beaker with a watch glass, dissolve by heating gently. Allow to cool.

NOTE Dissolution can be performed in a sealed container under pressure. In that case, the volume of aqua regia (5.4) 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 to 1 g of platinum accurately to 0,01 mg, and transfer it to a 150 ml glass beaker.

Add 10 ml of 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 1-2 ml of hydrochloric diluted acid (5.2), 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 predigestion with nitric acid (5.3) can be performed as an alternative method.

8.3 Precipitation of platinum with ammonium chloride

Evaporate the solution (from 8.1) or the filtrate (from 8.2) at least three times without letting the residue become dry and without exceeding a temperature of 100 °C , each time adding 2 ml of dilute hydrochloric acid (5.2) 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 (5.3).

After the last evaporation, dissolve the still moist platinum salt in 1 ml of dilute hydrochloric acid (5.2) and add 4 ml of water.

Add approximately 1 ml of the saturated ammonium chloride solution (5.6) for the equivalent of 7 mg of platinum at (85 ± 10) °C to this clear solution obtained in 8.1 or the filtrate obtained in 8.2. Ammonium chloride shall be added in excess.

To facilitate the filtration in case the volume of saturated ammonium chloride solution (5.6) is large, ammonium chloride can be added as powder. Add approximately 1 g of ammonium chloride (5.5) for the equivalent of 50 mg of platinum, and dissolve it in water (add at least 3 ml of distilled water for 1 g of ammonium chloride powder) and heat the solution.

The platinum is precipitated as yellow $(NH_4)_2[PtCI_6]$. The solution with the precipitated $(NH_4)_2[PtCI_6]$ is evaporated almost to dryness at this temperature. Allow to cool. Add just sufficient water while agitating to dissolve the residual ammonium chloride crystals.

NOTE 1 In the presence of palladium in significant concentration, co-precipitation of platinum and palladium can occur. Palladium can be removed before the addition of ammonium chloride. Otherwise, it will be deducted after the analysis of the platinum sponge.

Immediately filter the $(NH_4)_2[PtCI_6]$ precipitate over a filter paper (6.6) which has been moistened with ammonium chloride solution (5.4). Collect the filtrate in a glass beaker. Thoroughly wash the precipitate with ammonium chloride solution (5.4).

The precipitated $(NH_4)_2[PtCI_6]$ can also be filtered while still warm, without evaporating until dryness, but only using a vacuum system.

NOTE 2 Without a vacuum system, filtration would take too long and the ammonium chloride remaining in the solution would crystallized.

Wipe the glass beaker and watch glass with a second filter paper (6.6). If a concentration of the filtrate or reduction of quantity of ammonium chloride is necessary, it is possible to evaporate the filtrate by heating gently (the solution can foam and splash) and add aqua regia (5.4) until the solution is clear. Analyse this clear solution to check for residual platinum by suitable means, such as ICP-OES (6.4), using calibration solutions with a composition similar to the composition of the clear solution. The mass of platinum contained in the filtrate cannot exceed 0.5 % of the mass of the initial sample. Otherwise, the assays shall be repeated.

Transfer the filter paper containing the precipitate into a porcelain crucible (6.3). Cautiously dry the content of the crucible on a hotplate which can be gradually adjusted or in a programmable furnace.

By drying initially at 50 °C to 70 °C and heating gradually to 340 °C, all remaining ammonium chloride is lost. Ash the filter paper (6.6) and $(NH_4)_2[PtCI_6]$ residue at a temperature of 500 °C to 600 °C without a flame (at least 30 min). Finally, calcine in a muffle furnace (6.5) for 1 h to 3 h at a temperature of 900 °C.

NOTE 3 The ammonium chloride decomposes at 340 °C.

Significant absorption of oxygen can take place during ignition if palladium, iridium, ruthenium, or rhodium are present in the platinum sponge. Oxidation can be avoided by calcining under reducing gas (5.7) followed by cooling under inert or reducing gas (5.7).

Weigh the platinum sponge obtained.

NOTE 4 The filter paper can be transferred into a previously weighed crucible placed into a larger crucible. The mass of the platinum sponge can be determined by weighing the crucible with the sponge and by deducting the crucible weight.

NOTE 5 The crucible with the sponge is placed in a desiccator before weighing the sponge.

Dissolve the platinum sponge in 20 ml of aqua regia (5.4). 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 platinum sponge.

NOTE 6 If solid material is still observed in the solution, then dissolve it using dissolve it using alkaline melting method and measure the co-precipitated elements by ICP-OES or other methods.

9 Calculation and expression of results

9.1 Calculation

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

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

where

m_1 is the mass of the sample, in milligrams;