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Jewellery and precious metals — Determination of platinum — Gravimetry using diammonium chloride

*Joaillerie, bijouterie et métaux précieux — Dosage du platine
— Méthode gravimétrique après précipitation par le chlorure
d'ammonium*

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Foreword

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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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This document was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/SSM21, *Precious metals — Applications in jewellery and associated products*, in collaboration with ISO Technical Committee TC 174, *Jewellery*, in accordance with the agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 11210:2014), which has been technically revised.

The main changes are as follows:

- 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 — Gravimetry using diammonium chloride

1 Scope

This document specifies a gravimetric method for the determination of platinum on a material considered homogeneous. The platinum content of the sample lies preferably between 50 and 999 parts per thousand (‰) by mass. 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

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology 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

4 Principle

The sample is dissolved in aqua regia. Platinum is precipitated with ammonium chloride. The diammonium hexachloroplatinate precipitate is converted by ignition to metallic platinum which is weighed.

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

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

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

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

5.5 Ammonium chloride (NH_4Cl).

5.6 Saturated ammonium chloride solution, obtained by mixing ammonium chloride (5.5) in water until ammonium chloride does not dissolved anymore, and letting the solution 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 preferably 20 ml 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

8.1 Preliminary analysis

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.

8.2 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 glass beaker (preferably of 150 ml).

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

Dissolution may be performed in a sealed container under pressure. In that case, the volume of aqua regia (5.4) may be adapted to optimize the dissolution.

8.3 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 glass beaker (preferably of 150 ml).

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 is 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 ml to 2 ml of diluted hydrochloric acid (5.2), boil for 1 min to 2 min, filter the solution and repeat until the precipitate is white. Wash the precipitate with water.

8.4 Precipitation of platinum with ammonium chloride

Evaporate the solution from 8.2 or the filtrate from 8.3 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 8.2 or the filtrate obtained in 8.3. 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 $(\text{NH}_4)_2[\text{PtCl}_6]$. The solution with the precipitated $(\text{NH}_4)_2[\text{PtCl}_6]$ 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 with dimethylglyoxime before the addition of ammonium chloride. Otherwise, it will be deducted after the analysis of the platinum sponge.

Immediately filter the $(\text{NH}_4)_2[\text{PtCl}_6]$ precipitate over a filter paper (6.6) which has been moistened with ammonium chloride solution (5.6). Collect the filtrate in a glass beaker. Thoroughly wash the precipitate with ammonium chloride solution (5.6).

The precipitated $(\text{NH}_4)_2[\text{PtCl}_6]$ 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 crystallize.

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 $(\text{NH}_4)_2[\text{PtCl}_6]$ 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. Oxidation can be avoided by calcining under reducing gas (5.7), using the device or an equivalent presented in Annex A, followed by cooling under inert gas (5.8) or reducing gas (5.7). Not using a reducing gas (5.7) can lead to the presence of a large quantity of oxides. In that case, the partially oxidized palladium shall be reduced to the metallic state by heating it in presence of 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 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):

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

where

- m_1 is the mass of the sample, in milligrams;
- m_2 is the mass of platinum in the filtrate, in milligrams;
- m_3 is the final mass of the platinum sponge, in milligrams.

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

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

where m_x is the total mass of other elements contained in the platinum sponge, in milligrams.

9.2 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.

10 Test report

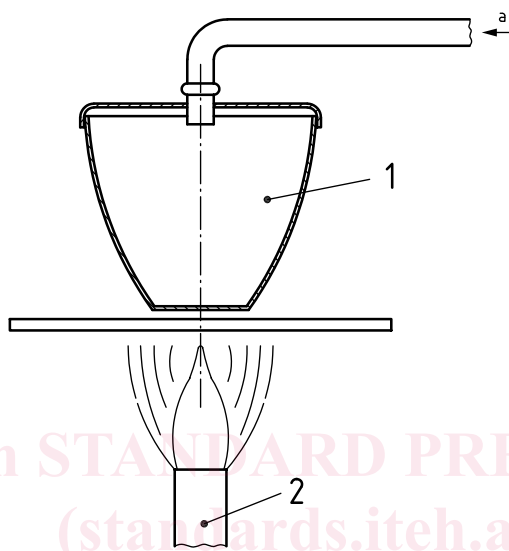
The test report shall include at least the following information:

- a) identification of the sample including source, date of receipt, form of sample;
- b) sampling procedure;
- c) method used by reference to this document, i.e. ISO 11210:—;
- 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 document;
- 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 example

Figure A.1 shows an example of a reduction apparatus.



Key

- 1 crucible
- 2 gas burner
- a Gas flow.

Figure A.1 — Reduction apparatus