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

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

Reference number
ISO 11210:1995(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11210 was prepared by Technical Committee ISO/TC 174, *Jewellery*.

Annex A of this International Standard is for information only.

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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 may 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 reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 9202:1991, *Jewellery — Fineness of precious metal alloys*.

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 for in the redissolved platinum sponge and measured by, for example, atomic absorption and inductively coupled plasma (ICP) emission spectrometry, 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, 36 % (*m/m*) to 38 % (*m/m*), ρ_{20} = 1,19 g/cm^3.

4.2 Dilute hydrochloric acid, 18 % (*m/m*), ρ_{20} = 1,09 g/cm^3.

4.3 Nitric acid, 69 % (*m/m*), ρ_{20} = 1,41 g/cm^3.

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 3 volumes of hydrochloric acid (4.1) and 1 volume of nitric acid (4.3).

WARNING — *Aqua regia* is potentially hazardous and safety glasses or goggles must be used. Dissolution should be carried out in a well-ventilated fume cupboard.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Reduction apparatus, see figure A.1 in annex A.

5.2 Atomic absorption or inductively coupled plasma (ICP) emission spectrometer, capable of determining traces of metals.

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

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

6 Sampling

The sampling procedure for platinum jewellery alloys shall be agreed upon until a corresponding standard method has been published.

7 Procedure

7.1 Platinum jewellery alloys with not more 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.

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 °C ± 5 °C to this clear solution. The platinum is precipitated as yellow $(NH_4)_2PtCl_6$. The solution with the precipitated $(NH_4)_2PtCl_6$ is evaporated almost to dryness at this temperature. Further gentle heating should 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 $(NH_4)_2PtCl_6$ precipitate over a filter paper (5.4) 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 atomic absorption or ICP emission spectrometer (5.2), after decomposition of the residual ammonium chloride, and correct the final result accordingly.

NOTE 1 The ammonium chloride decomposes at 340 °C.

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. By drying initially at 50 °C to 70 °C and heating subsequently to 340 °C, all ammonium chloride is lost. Without a flame, ash the filter paper (5.4) and $(NH_4)_2PtCl_6$ residue at a temperature of 500 °C to 600 °C . Finally remove the cover of the crucible and calcine in a muffle furnace (5.3) 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 may need breaking into several pieces during calcination.

Significant absorption of oxygen can take place during ignition if palladium, iridium, rhodium or copper are present in the platinum sponge. Reduction at 600 °C to 700 °C for 10 min in a reduction apparatus (5.1), followed by cooling under inert gas (4.6), will ensure that the sponge contains only metallic elements.

Weigh the resulting metallic platinum.

Measure the coprecipitated elements by suitable means such as an atomic absorption or ICP emission spectrometer (5.2).

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 may precipitate as tetrachloropalladiate $[(NH_4)_2PdCl_4]$ with the $(NH_4)_2PtCl_6$, and iridium, rhodium and ruthenium may coprecipitate. In addition, gold, if present in quantities in excess of about 5 parts per thousand, may precipitate as diamonium trichloroaurate $[(NH_4)_2AuCl_3]$ 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 sample weighed may be identified following dissolution in 20 ml of *aqua regia* (4.7), using, for example, atomic absorption spectroscopy.

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) may 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 Expression of results

8.1 Method of calculation

8.1.1 If the final weighed mass contains exclusively platinum, calculate the platinum content w_{Pt}, in parts by mass per thousand (‰), using the formula

$$w_{Pt} = \frac{m_3 + m_2}{m_1} \times 10^3$$

where

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

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

m_3 is the final mass, in milligrams.

8.1.2 If the final weighed mass contains other elements, calculate the platinum content w_{Pt}, in parts by mass per thousand (‰), using the formula

$$w_{Pt} = \frac{m_3 + m_2 - m_X}{m_1} \times 10^3$$

where m_X is the total mass, in milligrams, of other elements.

8.2 Repeatability

The results of duplicate determinations shall correspond to better than 3 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) the method used by reference to this International Standard;

d) platinum 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

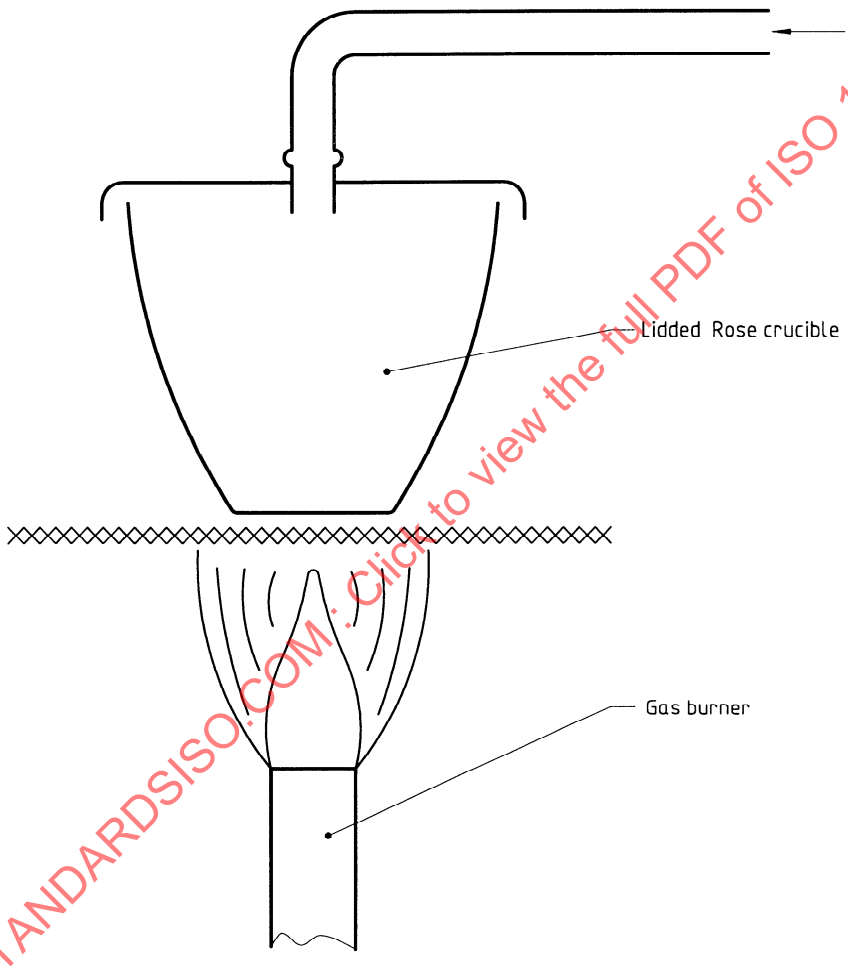

Figure A.1 — Reduction apparatus