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Determination of platinum in platinum jewellery alloys — Gravimetric determination by reduction with mercury(II) chloride

*Dosage du platine dans les alliages de platine pour la
bijouterie-joaillerie — Dosage gravimétrique par réduction au chlorure de
mercure(II)*



Foreword

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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 11489 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 determination by reduction with mercury(II) chloride

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.

The procedure applies specifically to platinum alloys incorporating 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 elimination of all nitrates by evaporation, the residue is dissolved in hydrochloric acid. The platinum is then precipitated from this solution by reduction with mercury(II) chloride. The mercury is eliminated by ignition and the platinum is weighed. If present, gold

and palladium will also be precipitated by this reduction procedure. Their content shall be determined separately by, for example, atomic absorption or 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), $\rho_{20} = 1,19 \text{ g/cm}^3$.

4.2 Dilute hydrochloric acid, 18 % (m/m), $\rho_{20} = 1,09 \text{ g/cm}^3$.

4.3 Dilute hydrochloric acid, 8,5 % (m/m), $\rho_{20} = 1,04 \text{ g/cm}^3$.

4.4 Nitric acid, 69 % (m/m), $\rho_{20} = 1,41 \text{ g/cm}^3$.

4.5 Mercury(II) chloride (Hg_2Cl_2), in suspension.

Dissolve 200 g of mercury(II) nitrate dihydrate [$\text{Hg}_2(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$] in 300 ml of water in a beaker and add approximately 50 ml of nitric acid; just sufficient to ensure that the basic mercury(II) nitrate is redissolved. Dilute the solution with water to 4 litres and add 400 ml of cold saturated ammonium chloride solution. Allow the precipitate of mercury(II) chloride to settle, decant and wash about 20 times to ensure that it is nitrate free. Add 2 litres of water and store in a closed flask.

NOTE 1 This suspension is stable and can be used even after storage for a few months.

4.6 Hydrofluoric acid,
40 % (m/m), $\rho_{20} = 1,13 \text{ g/cm}^3$.

4.7 Dilute sulfuric acid,
50 % (m/m), $\rho_{20} = 1,42 \text{ g/cm}^3$.

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

4.9 Inert gas, such as carbon dioxide or nitrogen.

4.10 Aqua regia.

Mix 3 volumes of hydrochloric acid (4.1) and 1 volume of nitric acid (4.4).

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 Platinum dishes, of volume 10 ml.

5.3 Atomic absorption or inductively coupled plasma (ICP) emission spectrometer, or other means of determining traces of metals.

5.4 Muffle furnace, capable of attaining at least 900 °C.

5.5 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 or ruthenium

Weigh the sample containing approximately 250 mg to 300 mg of platinum, accurately to the nearest 0,01 mg, and transfer it to a glass beaker. Dissolve the sample in 20 ml of *aqua regia* (4.10) 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.

Heat the solution on a hotplate and evaporate almost to dryness. Dissolve the still moist salt residue in 10 ml of dilute hydrochloric acid (4.2) and evaporate almost to dryness again. Repeat this dissolution in hydrochloric acid and evaporation three times, finally dissolving the residue in 10 ml of dilute hydrochloric acid (4.2), and then diluting the solution with hot water to a total volume of 300 ml.

Heat the solution to boiling and, while continuing to boil, add the shaken mercury(II) chloride suspension in portions of 5 ml to 10 ml. Continue to add the suspension until the initially black precipitate turns grey due to excess addition. Allow the precipitate to settle for about 15 min at a temperature above 85 °C and filter through a double filter paper (5.5). Wash the precipitate and beaker thoroughly with dilute hydrochloric acid (4.3). Retain the filtrate for testing for platinum by suitable means and, if detected, a correction shall be applied.

Fold the filter paper containing the precipitate, place it in a preweighed glazed porcelain crucible and dry at 120 °C under flowing air. Gradually increase the temperature to 400 °C over approximately 30 min. It is important to control the rate of heating to 400 °C, as rapid mercury distillation can cause losses of platinum.

Once a temperature of 400 °C is achieved, the crucible and contents can be heated at 900 °C for 60 min in a muffle furnace (5.4) to ignite the precipitate.

WARNING — Considering the health hazards associated with mercury vapour, it must be ensured that all appropriate precautions are taken to prevent it entering the atmosphere.

Cool the crucible which now contains the platinum sponge. The sponge absorbs oxygen if palladium is present, so heat it with reducing gas (4.8) in a reduction apparatus (5.1) and cool under an inert atmosphere (4.9). A check weighing can be made at this stage to confirm that the platinum content approximates that expected.

Transfer the sponge to a platinum dish and purify it by moistening with a little hydrofluoric acid (4.6) and three drops of dilute sulfuric acid (4.7). Heat until fuming commences and then cool, add a little hot

water, filter and wash with hot dilute hydrochloric acid (4.3). Ash the filter and sponge in a porcelain crucible and ignite at 700 °C in a muffle furnace (5.4). The sponge should then be treated again in the reduction apparatus (5.1) and cooled under an inert atmosphere (4.9) before finally weighing.

Any gold and palladium in the sponge is then determined by suitable means, such as atomic absorption or ICP emission spectrometry (5.3) and a correction applied. It is also possible that iridium, rhodium and ruthenium may still be present in the final sponge. It is recommended that the sponge be checked for the presence of these elements by suitable means, such as atomic absorption or ICP emission spectrometry. If they are detected, a correction shall be applied.

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

Follow the procedure described in 7.1. However, for dissolving use a pressure dissolution procedure.

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

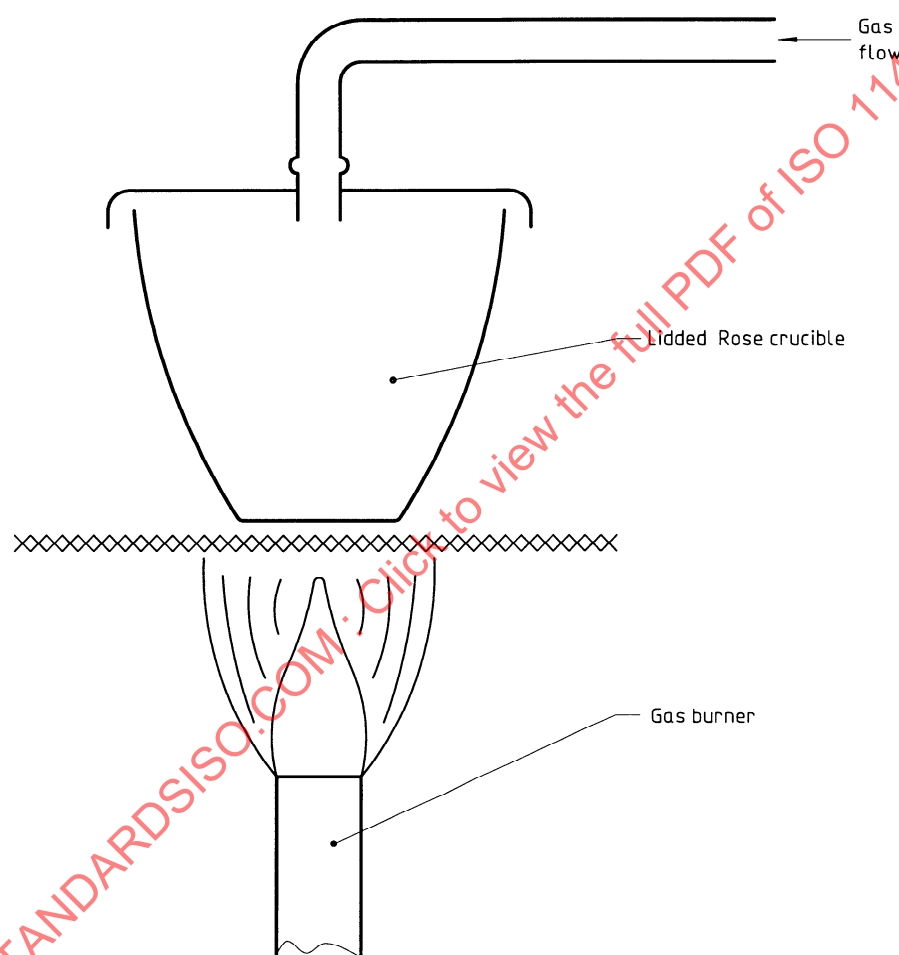


Figure A.1 — Reduction apparatus