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Jewellery — Determination of platinum in platinum jewellery alloys — Inductively coupled plasma (ICP) solution-spectrometric method using yttrium as internal standard element

Joaillerie, bijouterie — Dosage du platine dans les alliages de platine pour la joaillerie, bijouterie — Méthode par spectrométrie d'émission à plasma induit en solution, utilisant l'yttrium comme étalon interne

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Foreword

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ISO 11494 was prepared by Technical Committee ISO/TC 174, *Jewellery*.

Jewellery — Determination of platinum in platinum jewellery alloys — Inductively coupled plasma (ICP) solution-spectrometric method using yttrium as internal standard element

1 Scope

This International Standard describes a method for the determination of platinum in platinum jewellery alloys, preferably within the range of fineness specified in ISO 9202, by means of inductively coupled plasma (ICP) emission spectrometry.

The preferred platinum content of the alloys lies between 850 ‰ (parts per thousand) and 950 ‰ platinum.

NOTE This method can be used to analyse other contents of platinum.

Platinum jewellery alloys can contain silver, indium, gallium, copper, cobalt, nickel, tin and ruthenium. The presence of these alloying elements has not been observed to interfere with the determination method. If other elements are alloyed, a check is made as to whether any interference occurs.

2 Normative references

The following referenced documents are indispensable for the application 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 — Sampling of precious metal alloys for and in jewellery and associated products*

3 Terms and definitions

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

3.1
bracketing
running of standards and samples in the following sequence: low standard – sample – high standard – sample – low standard – sample – high standard – sample – low standard – sample – high standard

4 Short description of method

At least two accurately weighed samples are dissolved in aqua regia and made up to an exactly weighed mass. Exactly weighed portions (aliquots) of these sample solutions are mixed with the internal standard and made up to the standard measuring volume.

Using an ICP emission spectrometer, the platinum content of the sample solution is measured by comparison of the ratio intensities of the spectral emission of platinum (at 265,95 nm, 214,42 nm, 299,80 nm or 306,47 nm) and yttrium (at 371,03 nm) with the ratios for solutions containing known masses of platinum and yttrium, using the bracketing method.

Minor modifications are required when the alloy contains ruthenium, rhodium, iridium, chromium or tungsten.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity. All reagents shall be platinum free.

5.1 Hydrochloric acid (HCl); $\rho_{20} = 1{,}16\ \text{g/cm}^3$; 32 % HCl (mass fraction).

5.2 Nitric acid (HNO_3); $\rho_{20} = 1{,}41\ \text{g/cm}^3$; 65 % HNO_3 (mass fraction).

5.3 Pure platinum (Pt).

The platinum content shall be at least 99,99 %.

5.4 Internal standard solution.

Approximately 680 mg $YCl_3 \cdot 6H_2O$ are dissolved in 200 ml water and made up to 1 000 ml with water. Due to the sensitivity of the instrument, this concentration may be changed to achieve optimum performance.

This also applies to the calibration solutions (see 8.1) and the sample solutions (see 8.2).

5.5 Pure copper.

The copper content shall be 99,99 % and platinum free.

5.6 Orthophosphoric acid (H_3PO_4); $\rho_{20} = 1{,}71\ \text{g/cm}^3$; 85 % (mass fraction).

6 Apparatus

Customary laboratory apparatus and the following.

6.1 ICP emission spectrometer capable of simultaneously measuring the platinum emission lines (at 265,95 nm, 214,42 nm, 299,80 nm or 306,47 nm) and the emission line of the internal standard yttrium (at 371,03 nm), with a minimum optical resolution of 0,02 nm.

6.2 Analytical balance accurate to 0,01 mg.

7 Sampling

The sampling procedure shall be performed in accordance with ISO 11596.

8 Procedure

8.1 Calibration solutions

Weigh approximately 100 mg of platinum (5.3) accurately to at least 0,01 mg and dissolve in 30 ml of hot HCl (5.1) and 10 ml of HNO_3 (5.2) in a tared 100 ml volumetric flask. Add water until the mass of the solution is approximately 100 g, weighed accurately to at least 0,01 g. This platinum stock solution is used to prepare the calibration solutions.

Weigh approximately 8,25 g, 8,75 g, 9,25 g and 9,75 g of the platinum stock solution accurate to at least 0,001 g, each into a 100 ml volumetric flask. Add 10 g of the internal standard solution (5.4) accurately to at least 0,001 g. Add 10 ml of HCl (5.1) and make up to 100 ml with water. Mix thoroughly.

In the presence of certain other elements (e.g. silver), it can be necessary to increase the HCl concentration to a maximum of 50 ml. The acid concentration of calibration solutions and sample solutions shall be consistent.

8.2 Sample solutions

8.2.1 General

Weigh approximately 100 mg of the sample accurately to at least 0,01 mg, dissolve and treat the sample as described in 8.1. Weigh approximately 10 g of this “sample stock solution” accurate to at least 0,001 g into a 100 ml volumetric flask, and add 10 g of the internal standard solution (5.4) accurate to at least 0,001 g. Add 10 ml of HCl (5.1) and make up to 100 ml with water. Mix thoroughly.

In the presence of certain other elements (e.g. silver), it can be necessary to increase the HCl concentration to a maximum of 50 ml. The acid concentration of calibration solutions and sample solutions shall be consistent.

NOTE Attention is drawn to the possibility that smaller samples will also be more affected by any variation of homogeneity in the material sampled.

8.2.2 Platinum alloys containing ruthenium, rhodium, iridium or tungsten

Platinum alloys containing more than 5 % of the cited elements can require a pressure dissolution or involve a preliminary alloying stage with a quantity of 10 times the platinum-free copper (5.5).

In the presence of tungsten, 200 µl of orthophosphoric acid (5.6) shall be added.

Calibration solutions and sample solutions shall be matrix matched.

8.2.3 Platinum alloys containing ruthenium, rhodium, iridium or chromium

It is useful to compare the analytical results obtained with the four platinum emission lines (at 265,95 nm, 214,42 nm, 299,80 nm and 306,47 nm). The platinum emission line at 265,95 nm can be influenced by ruthenium, rhodium and chromium, and the platinum emission lines at 299,80 nm and 306,47 nm can be influenced by iridium and chromium; therefore, caution is necessary.

8.3 Measurements

The data processing unit of the ICP spectrometer is used to establish a measuring programme in which the intensities of the platinum emission lines (at 265,95 nm, 214,42 nm, 299,80 nm or 306,47 nm) and of the internal standard element yttrium (at 371,03 nm) can be measured simultaneously. After ignition, allow the ICP torch at least 15 min to stabilize before use. Aspirate the calibration solutions and the sample solutions sequentially.

Each standard and sample solution shall have a minimum 30 s preintegration period, followed by the integration times and the number of integrations required to obtain a maximum relative standard deviation (RSD) of 0,2 % [see 8.4.1, Equation (1)]. The accurate mass of platinum of the sample solution is derived from the measurement of the two calibration solutions bracketing the rough value of the sample solution [see 8.4.2, Equation (4)].

8.4 Calculations

8.4.1 The method of internal standardization is based on the linear relation between the intensity ratios $I_{\mathrm{Pt}}/I_{\mathrm{Y}}$ and the concentration ratios $C_{\mathrm{Pt}}/C_{\mathrm{Y}}$ or, better, mass ratios $m_{\mathrm{Pt}}/m_{\mathrm{Y}}$. Using the same mass of yttrium (internal standard solution) to prepare all solutions, it is not necessary to have an exact volume of the measuring solutions. The accuracy of the 100 ml volumetric flask is satisfactory. The other important advantage of always referring to the same mass of the internal standard is that all calculations can be done with m_{Pt} instead of $m_{\mathrm{Pt}}/m_{\mathrm{Y}}$, nominal.

In general, the data processing unit provides the quotients from the simultaneously registered single measurements of the platinum and the yttrium intensities.

If the mean value, $\overline{Q}$, of the five intensity quotients (Q_1, Q_2, Q_3, Q_4, Q_5) belonging to each solution is calculated as follows:

$$\overline{Q} = \frac{1}{5}\left(\sum_{n=1}^{5}\frac{I_{\text{Pt}}}{I_{\text{Y}}}\right) \tag{1}$$

then this mean value shall have a RSD from Q not larger than 0,2 %.

8.4.2 In view of deviations from the nominal mass, m_{IS}, in grams, of the internal standard solution ($m_{\text{IS}} = 10{,}000$ g), each intensity quotient belonging to a measuring solution shall be corrected by the corresponding real mass of internal standard solution $W_{\text{IS},n}$, in grams, used to prepare this measuring solution. The corrected quotient, Q_{C}, is calculated as follows:

$$Q_{\text{C}} = Q \times \frac{W_{\text{IS},n}}{m_{\text{IS}}} \tag{2}$$

To determine the platinum content of the sample using the corrected intensity quotient, the exact masses of platinum in the calibration solutions $m_{\text{Pt,Cs},n}$, in milligrams, are required. These masses shall be calculated individually for each calibration solution or calibration point as follows:

$$m_{\text{Pt,Cs},n} = \frac{W_{\text{Pt,SS}}}{m_{\text{SS,Pt}}} \times W_{\text{SS,Pt},n} \tag{3}$$

where

$W_{\text{Pt,SS}}$ is the mass, in milligrams, of platinum used to prepare the platinum stock solution;

$m_{\text{SS,Pt}}$ is the mass, in grams, of the prepared platinum stock solution;

$W_{\text{SS,Pt},n}$ is the mass, in grams, of platinum stock solution used to prepare the calibration solution n.

The two calibration points nearest to the platinum sample content, corresponding to the low mass a and to the high mass b, are used to determine the platinum mass in the sample solution as follows:

$$m_{\text{Pt}} = a + \frac{(b-a)\times\left(Q_{\text{C,s}} - Q_{\text{C},a}\right)}{\left(Q_{\text{C},b} - Q_{\text{C},a}\right)} \tag{4}$$

where

a is the mass, in milligrams, of platinum in the calibration solution used as “low standard”, according to Equation (3);

b is the mass, in milligrams, of platinum in the calibration solution used as “high standard”, according to Equation (3);

$Q_{\text{C},a}$ is the corrected intensity ratio $I_{\text{Pt}}/I_{\text{Y}}$ of the “low standard”;

$Q_{\text{C},b}$ is the corrected intensity ratio $I_{\text{Pt}}/I_{\text{Y}}$ of the “high standard”;

$Q_{\text{C,s}}$ is the corrected intensity ratio $I_{\text{Pt}}/I_{\text{Y}}$ of the sample measuring solution.

The final mass of platinum of the sample solution corresponds to the mean value of five measuring cycles and evaluations of this type, $\bar{m}_{\text{Pt}}$, and is calculated as follows:

$$\bar{m}_{\text{Pt}} = \frac{1}{5}\left(\sum_{n=1}^{5} m_{\text{Pt}}\right) \tag{5}$$

The RSD of m_{Pt} shall not exceed 0,30 %.

Once $\bar{m}_{\text{Pt}}$ has been determined from the five single determinations of the sample solution, the platinum content of the sample, X_{Pt}, expressed in parts per thousand, is calculated as follows:

$$X_{\text{Pt}} = \frac{\bar{m}_{\text{Pt}} \times m_{\text{SS,Sa}}}{W_{\text{Sa}} \times W_{\text{SS,Sa}}} \times 1\,000 \tag{6}$$

where

W_{Sa} is the mass, in milligrams, of sample used to prepare the sample stock solution;

$m_{\text{SS,Sa}}$ is the mass, in grams, of the prepared sample stock solution;

$W_{\text{SS,Sa}}$ is the mass, in grams, of sample stock solution used to prepare the sample measuring solution.

9 Repeatability

Duplicate determinations shall give results differing by less than 3 ‰ for platinum. If the difference is greater than this, the assay shall be repeated.

10 Test report

With reference to this method, the test report shall contain at least the following information:

a) identification of the sample including source, date of receipt, form;

b) sampling procedure;

c) the reference of the method used;

d) platinum content of the sample, in percent or parts per thousand, as single values and mean values;

e) deviations from this standard method, if relevant;

f) platinum line and internal standard line used;

g) any unusual features observed during the determination;

h) date of test;

i) identification of the laboratory carrying out the test;

j) signature of the laboratory manager and operator.