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

Joaillerie, bijouterie — Dosage du palladium dans les alliages de palladium 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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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 11495 was prepared by Technical Committee ISO/TC 174, *Jewellery*.

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Jewellery — Determination of palladium in palladium 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 palladium in palladium jewellery alloys, preferably within the range of fineness specified in ISO 9202, by means of inductively coupled plasma (ICP) emission spectrometry.

The preferred palladium content of the alloys lies between 500 ‰ (parts per thousand) and 950 ‰ palladium.

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

Palladium 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.

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2 Normative references

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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 palladium content of the sample solution is measured by comparison of the ratio intensities of the spectral emission of palladium (at 340,45 nm) and yttrium (at 371,03 nm) with the ratios for solutions containing known masses of palladium and yttrium, using the bracketing method.

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Other palladium emission lines may be used, but need to be checked for spectral interference and instrumental performance.

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 palladium free.

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

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

5.3 Pure palladium (Pd).

The palladium 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).

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6 Apparatus

Customary laboratory apparatus and the following.

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6.1 ICP emission spectrometer capable of simultaneously measuring the palladium emission line (at 340,45 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 palladium (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 palladium stock solution is used to prepare the calibration solutions.

Weigh approximately 4,5 g, 5,5 g, 6,5 g, 7,5 g, 8,5 g, 9,5 g and 9,8 g of the palladium stock solution accurately to at least 0,001 g, each into a 100 ml volumetric flask. Add 10 g of the internal standard solution (5.4) accurate to at least 0,01 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.

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8.2 Sample solutions

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 0,01 g. Add 10 ml of HCl (5.1) and make up to 100 ml. 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.

8.3 Measurements

The data processing unit of the ICP spectrometer is used to establish a measuring programme in which the intensities of the palladium emission lines (at 340,45 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 palladium 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)].

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8.4 Calculations

8.4.1 The method of internal standardization is based on the linear relation between the intensity ratios I_{Pd}/I_Y and the concentration ratios C_{Pd}/C_Y or, better, mass ratios m_{Pd}/m_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_{Pd} instead of m_{Pd}/m_Y, nominal.

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In general, the data processing unit provides the quotients from the simultaneously registered single measurements of the palladium 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:

$$\overline{Q} = \frac{1}{5}\left(\sum_{n=1}^{5} \frac{I_{Pd}}{I_Y}\right) \tag{1}$$

then this mean value shall have an 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_{IS} = 10,000 g), each intensity quotient belonging to a measuring solution shall be corrected by the corresponding real mass of internal standard solution $W_{IS,n}$, in grams, used to prepare this measuring solution. The corrected quotient, Q_C, is calculated as follows:

$$Q_C = Q \times \frac{W_{IS,n}}{m_{IS}} \tag{2}$$

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

$$m_{\mathrm{Pd,Cs},n} = \frac{W_{\mathrm{Pd,SS}}}{m_{\mathrm{SS,Pd}}} \times W_{\mathrm{SS,Pd},n} \tag{3}$$

where

$W_{\mathrm{Pd,SS}}$ is the mass, in milligrams, of palladium used to prepare the palladium stock solution;

$m_{\mathrm{SS,Pd}}$ is the mass, in grams, of the prepared palladium stock solution;

$W_{\mathrm{SS,Pd},n}$ is the mass, in grams, of palladium stock solution used to prepare the calibration solution n.

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

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

where

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

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

$Q_{\mathrm{C},a}$ is the corrected intensity ratio $I_{\mathrm{Pd}}/I_{\mathrm{Y}}$ of the "low standard";

$Q_{\mathrm{C},b}$ is the corrected intensity ratio $I_{\mathrm{Pd}}/I_{\mathrm{Y}}$ of the "high standard";

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

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

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

The RSD of $\bar{m}_{\mathrm{Pd}}$ shall not exceed 0,30 %.

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

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

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where

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

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

$W_{SS,Sa}$ 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 palladium. 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) palladium 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) palladium 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.

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