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Jewellery — Determination of precious metals in 999 ‰ gold, platinum and palladium jewellery alloys — Difference method using inductively coupled plasma optical emission spectroscopy (ICP-OES)

Joaillerie, bijouterie — Dosage des métaux précieux dans les alliages d'or, de platine et de palladium 999 ‰ pour la joaillerie, bijouterie — Méthode de la différence utilisant la spectrométrie d'émission à plasma induit par haute fréquence (ICP-OES)

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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 15093 was prepared by Technical Committee ISO/TC 174, *Jewellery*.

Jewellery — Determination of precious metals in 999 ‰ gold, platinum and palladium jewellery alloys — Difference method using inductively coupled plasma optical emission spectroscopy (ICP-OES)

1 Scope

This International Standard specifies an analytical procedure for the determination of either platinum in platinum jewellery alloys, gold in gold jewellery alloys or palladium in palladium jewellery alloys, with a nominal content of each precious metal of 999 ‰ (parts per thousand), by measuring specific elements listed in Tables A.1, A.2 and A.3.

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 Principle

The samples of the precious metal alloy are weighed and dissolved in aqua regia to prepare a 10 g/l solution. The impurities are determined by inductively coupled plasma optical emission spectroscopy (ICP-OES), and the precious metal content is obtained by subtraction of the total content of impurities in the sample from 1 000 ‰.

4 Sampling

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

NOTE Equivalent methods can be used, as described in ISO 5725-1.

For coated articles, appropriate precautions that have been agreed upon shall be taken to exclude the coating from the determination.

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); ρ_{20} = 1,19 g/cm^3; 37 % HCl (mass fraction).

5.2 Nitric acid (HNO_3); ρ_{20} = 1,40 g/cm^3; 67 % HNO_3 (mass fraction).

5.3 Aqua regia (to be prepared just before use).

To one part by volume of HNO_3 (5.2), add three parts by volume of HCl (5.1) and mix thoroughly.

5.4 Mixed stock solutions

Using appropriate certified reagents, the following mixed stock solutions shall be prepared in order to obtain the indicated concentrations.

5.4.1 Nitric stock solution (chloride free): Ag, Bi, Pb (100 mg/l each) in 2 mol/l of HNO_3 (5.2).

5.4.2 Hydrochloric stock solution (nitrate free): Sn, Ti (100 mg/l each) in 2 mol/l of HCl (5.1).

5.4.3 Acid stock solution (may contain both chlorides and nitrates): all remaining relevant elements (100 mg/l each) in 1 mol/l of HCl (5.1) and 1 mol/l of HNO_3 (5.2).

Any desired element may be added in the nitric stock solution (5.4.1) or the hydrochloric stock solution (5.4.2) instead of the acid stock solution (5.4.3), provided that no chlorides are introduced in the nitric stock solution (5.4.1) and no nitrates are introduced in the hydrochloric stock solution (5.4.2).

5.5 Precious metal wire or **sheet of gold, platinum or palladium** of 999,9 ‰ minimum purity. The content of each impurity shall be determined.

6 Apparatus

Customary laboratory apparatus and the following.

6.1 ICP optical emission spectrometer, with:

— fixed and/or scanning channels;

— an optical resolution of 0,02 nm for the relevant elements and a detection limit of 0,05 mg/l or better;

— the capability of background correction.

NOTE Annex A specifies preferably used wavelengths.

6.2 Analytical balance accurate to 0,01 mg.

7 Procedure

7.1 Test solution

For each sample to be analysed, two test solutions shall be prepared as follows.

Weigh (500 ± 2,5) mg of the test portion to the nearest 0,01 mg, transfer into a 50 ml volumetric flask and add 30 ml of aqua regia (5.3). Heat gently until complete dissolution of the sample and continue to heat to expel the nitrogen oxides. Allow to cool, make up with water and mix thoroughly.

The deviation in the volume of the volumetric flask caused by heating is acceptable for this method.

If insolubles are observed, they shall be weighed and their amount shall be added to the impurities.

7.2 Calibration solutions

Weigh two portions of (500 ± 2,5) mg of the pure precious metal (5.5) and dissolve each one as specified in 7.1.

Calibration solution 1 (blank solution): dilute the first pure precious metal solution up to 50 ml and mix thoroughly.

Calibration solution 2: add 5 ml of each mixed stock solution (5.4), dilute the solution up to 50 ml and mix thoroughly.

7.3 Measurement

Set up the instrument in accordance with the manufacturer's instructions, and choose appropriate background correction positions and an appropriate matrix line for establishing the identity. A clean torch, spray chamber and sample uptake tubes shall be used, and the plasma shall be stabilized at least 30 min before use.

Spray the calibration solutions 1 and 2 in accordance with the defined instrument calibration procedure and then run the analytical procedure for the sample solutions. The result shall be displayed to enough decimal places to provide an accurate indication of concentrations at the detection limits of the relevant elements.

Each solution shall have a preintegration time of at least 30 s, followed by five integrations of at least 5 s each for the determination of the net intensities (i.e. background-corrected).

The intensity of the chosen matrix line (see Tables A.1, A.2 and A.3) shall not be included in the calculation described in 8.2.

8 Expression of results

8.1 Calibration curves

Set the concentration in calibration solutions 1 and 2, taking into account impurities introduced in the solution by the reference material (5.5), and calculate the calibration curve for each element, using the net intensities obtained for the calibration solutions 1 and 2.

8.2 Method of calculation

By means of the calibration curves (see 8.1), convert the net intensity values into concentration values and use Equation (1) to calculate the mass ratio of each relevant element, W_i:

$$W_i = \frac{c_i \times V_S}{m_S} \tag{1}$$

where

c_i is the concentration of element i in the sample solution or the detection limit of element i, whichever is higher, in milligrams/litre;

V_S is the volume of the sample solution, in litres;

m_S is the mass of the metallic sample, in milligrams.

The detection limit is defined as three standard deviations of the concentration of each individual element measured in the calibration solution 1.

The specific precious metal fineness, W_{sp}, expressed in parts per thousand, is thus calculated as follows:

$$W_{sp} = 1000 - \left(\sum W_i \times 1000\right) \quad (2)$$

where $\sum W_i$ is the sum of the mass ratio of each relevant element.

8.3 Repeatability

The results of duplicate determinations shall not deviate by more than 0,2 ‰ of the precious metal. If the variation is greater than this, the assays shall be repeated.

9 Test report

The test report shall contain at least 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) precious metal content of the sample, in parts per thousand, as single values and mean values, with the result reported to one decimal place only;

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 the analysis;

i) signature of the laboratory manager and operator.

Annex A
(normative)

Wavelengths

Other wavelengths than those specified in Tables A.1, A.2 and A.3 may be used. In such cases, attention shall be paid to optical interferences.

Table A.1 — Platinum

Element	Wavelength	Alternative	Element	Wavelength	Alternative
Ag	328,068	—	Ni	352,454	231,604
Au	242,795	—	Pb	168,220	220,353
Bi	223,061	—	Pt [a]	224,552	273,396
Cd	226,502	—	Pd	340,458	355,308
Co	228,616	238,892	Rh	343,489	—
Cu	324,754	—	Ru	240,272	—
Fe	259,94	—	Sn	189,989	—
Ir	215,278	—	Ti	334,941	—
Mn	257,610	—	Zn	213,856	—

[a] Suggested matrix line (see 7.3).

Table A.2 — Gold

Element	Wavelength	Alternative	Element	Wavelength	Alternative
Ag	328,068	—	Ni	352,454	231,604
Au [a]	389,789	302,920	Pb	168,220	220,353
Bi	223,061	—	Pd	340,458	355,308
Cd	228,802	226,502	Pt	306,471	203,646
Co	228,616	238,892	Rh	343,489	—
Cu	324,754	—	Ru	240,272	—
Fe	259,94	—	Sn	189,989	189,927
Ir	215,278	—	Ti	334,941	—
Mn	257,610	—	Zn	213,856	—

[a] Suggested matrix line (see 7.3).