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**Jewellery – Determination of Precious Metal Content –
Difference Method Using Inductively Coupled Plasma
Optical Emission Spectroscopy**

首饰 贵金属含量的测量 ICP 差减法

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Jewellery – Determination of Precious Metal Content – Difference Method Using Inductively Coupled Plasma Optical Emission Spectroscopy

1 Scope

This Document specifies a method for determining the content of precious metals by determining the content of impurity elements in gold, platinum, and palladium alloy jewellery.

This Document is applicable to gold alloy jewellery with gold content of 725‰~999‰, platinum alloy jewellery with platinum content of 800‰~999‰, and palladium alloy jewellery with palladium content of 800‰~999‰.

NOTE: The applicable measurement range of the precious metal content is the range covered by the precision test. It may also be applicable beyond this range, but the laboratory is required to verify the test beyond the range.

2 Normative References

The following documents are essential to the application of this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 12806-2011 Laboratory Glassware - One-Mark Volumetric Flasks

GB/T 12808-2015 Laboratory Glassware - One Mark Pipettes

3 Terms and Definitions

For the purposes of this Document, there are no terms and definitions that shall be given.

ratio of 3:1, and it is prepared immediately before use.

5.17 Gold standard stock solution (200µg/mL):

Weigh (100±0.01) mg of pure gold (5.2); place it in a 50mL beaker. Add 10mL of mixed acid (5.16) solution; slowly heat to dissolve. After cooling, transfer to a 500mL volumetric flask; add 105mL of hydrochloric acid (5.13); dilute to the mark with water and shake well.

5.18 Palladium standard stock solution (200µg/mL):

Weigh (100±0.01) mg of pure palladium (5.3); place it in a 50mL beaker. Add 10mL of mixed acid (5.16) solution; slowly heat to dissolve. After cooling, transfer to a 500mL volumetric flask; add 105mL of hydrochloric acid (5.13); dilute to the mark with water and shake well.

5.19 Silver standard stock solution (200µg/mL):

Weigh (100±0.01) mg of pure silver (5.4); place it in a 50mL beaker. Add 10mL of nitric acid (5.15) solution; slowly heat to dissolve. After cooling, transfer to a 500mL volumetric flask pre-filled with 92mL of hydrochloric acid (5.11). Wash the beaker with a small amount of water; incorporate the lotion into a volumetric flask; shake until the precipitate is dissolved. Dilute to the mark with hydrochloric acid (5.14), and shake well.

5.20 Copper standard stock solution (200µg/mL):

Weigh (100±0.01) mg of pure copper (5.5); place it in a 50mL beaker. Add 10mL of nitric acid (5.15) solution; slowly heat to dissolve. After cooling, transfer to a 500mL volumetric flask; add 115mL of hydrochloric acid (5.13); and dilute with water to the mark, and shake well.

5.21 Nickel standard stock solution (200µg/mL):

Weigh (100±0.01) mg of pure nickel (5.6); place it in a 50mL beaker. Add 10mL of nitric acid (5.15) solution; slowly heat to dissolve. After cooling, transfer to a 500mL volumetric flask; add 115mL of hydrochloric acid (5.13); dilute with water to the mark, and shake well.

5.22 Cobalt standard stock solution (200µg/mL):

Weigh (100±0.01) mg of pure cobalt (5.7); place it in a 50mL beaker. Add 10mL of nitric acid (5.15) solution; slowly heat to dissolve. After cooling, transfer to a 500mL volumetric flask; add 120mL of hydrochloric acid (5.13); dilute with water to the mark, and shake well.

5.23 Iron standard stock solution (200µg/mL):

Weigh (100 ± 0.01) mg of pure iron (5.8); place it in a 50mL beaker. Add 10mL of hydrochloric acid (5.13) solution; slowly heat to dissolve. After cooling, transfer to a 500mL volumetric flask; add 120mL of hydrochloric acid (5.13); dilute with water to the mark, and shake well.

5.24 Zinc standard stock solution (200 μ g/mL):

Weigh (100 ± 0.01) mg of pure zinc (5.9); place it in a 50mL beaker. Add 10mL of hydrochloric acid (5.13) solution; slowly heat to dissolve. After cooling, transfer to a 500mL volumetric flask; add 120mL of hydrochloric acid (5.13); dilute with water to the mark, and shake well.

5.25 Cadmium standard stock solution (200 μ g/mL):

Weigh (100 ± 0.01) mg of pure cadmium (5.10); place it in a 50mL beaker. Add 10mL of mixed acid (5.16) solution; slowly heat to dissolve. After cooling, transfer to a 500mL volumetric flask, add 115mL of hydrochloric acid (5.13); dilute with water to the mark, and shake well.

5.26 Platinum standard stock solution (200 μ g/mL):

Weigh (100 ± 0.01) mg of pure platinum (5.1); place it in a 50mL beaker. Add 10mL of mixed acid (5.16) solution; slowly heat to dissolve. After cooling, transfer to a 500mL volumetric flask; add 115mL of hydrochloric acid (5.13); dilute to the mark with water, and shake well.

5.27 Standard stock solutions or mixed standard stock solutions of iridium, rhodium, ruthenium, bismuth, manganese, lead, tin, magnesium, silicon, arsenic, antimony, and chromium:

National certified standard samples or standard substances may be purchased.

6 Instruments

6.1 Inductively coupled plasma emission spectrometer: wavelength resolution $\leq 0.01\text{nm}$. The detection limit of the measured element is better than 0.02mg/L, and it has a background correction function.

6.2 Micro-electronic balance: The actual graduation value is no more than 0.01mg; and the accuracy level is Level- II .

6.3 Single line pipette: Class-A in GB/T 12808-2015. Or adjustable pipette.

6.4 Single line volumetric flask: Class-A in GB/T 12806-2011.

ρ_i – concentration of the tested elements of the specimen solution, in $\mu\text{g/mL}$;

ρ_{i0} – concentration of the tested elements of the blank solution, in $\mu\text{g/mL}$;

V – volume of the specimen solution, in mL;

m – mass of the specimen, in mg.

If $\rho_i - \rho_{i0} \leq$ limit of detection, take the limit of detection as the $(\rho_i - \rho_{i0})$ value to introduce for calculation; thereof, the limit of detection is 3 times the standard deviation of the 11 test results of the concentration of each element in the corresponding reagent blank solution.

According to the relative intensity of the target element of the unknown sample, use the calibration curve to calculate the content of the element to be measured. The test result of the element to be tested shall be within the content range of a series of calibration samples used in the calibration curve.

8.2 Calculation of precious metal content

The precious metal content ω is expressed in‰; and calculated according to Formula (2):

$$\omega = 1\,000 - \sum A_i \dots\dots\dots (2)$$

The calculation results shall be expressed to one digit after the decimal point.

9 Repeatability

If the absolute difference of the total amounts of the tested elements (8.1) is no more than 40‰ of the arithmetic mean of the two tested values (8.1), for samples with a purity of more than 995‰, the absolute difference of the results of parallel determination of the precious metal content is no more than 0.2 ‰. If it is greater than this value, it shall be tested again.

10 Test Report

The test report shall include:

- a) The identification of the sample, including the source, the date of receiving the sample, the shape of the sample, and the date of the test;
- b) The number of the used standard;
- c) The permillage of the precious metal content of the specimen is calculated in

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