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Jewellery — Determination of silver in silver jewellery alloys — Volumetric (potentiometric) method using potassium bromide

Joaillerie, bijouterie — Dosage de l'argent dans les alliages d'argent pour la bijouterie-joaillerie — Méthode volumétrique (potentiométrique) utilisant le bromure de potassium

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 174, *Jewellery*.

This second edition cancels and replaces the first edition (ISO 11427:1993) which has been technically revised with the following changes:

a) change of requirement for sampling in Clause 6;

b) addition of a warning in Clause 7 that suitable health and safety procedures should be followed;

c) addition of the possibility to use watch glasses in 7.1.1 to cover the beaker;

d) deletion of the specified volume of water in 7.1.1;

e) addition in 7.1.3 that a potassium bromide standard solution is used;

f) change in 8.1 from potassium chloride to potassium bromide;

g) standard editorially revised.

Introduction

The following definitions apply in understanding how to implement an ISO International Standard and other normative ISO deliverables (TS, PAS, IWA).

— “shall” indicates a requirement

— “should” indicates a recommendation

— “may” is used to indicate that something is permitted

— “can” is used to indicate that something is possible, for example, that an organization or individual is able to do something

3.3.1 of the ISO/IEC Directives, Part 2 (sixth edition, 2011) defines a requirement as an “expression in the content of a document conveying criteria to be fulfilled if compliance with the document is to be claimed and from which no deviation is permitted.”

3.3.2 of the ISO/IEC Directives, Part 2 (sixth edition, 2011) defines a recommendation as an “expression in the content of a document conveying that among several possibilities one is recommended as particularly suitable, without mentioning or excluding others, or that a certain course of action is preferred but not necessarily required, or that (in the negative form) a certain possibility or course of action is deprecated but not prohibited.”

Jewellery — Determination of silver in silver jewellery alloys — Volumetric (potentiometric) method using potassium bromide

1 Scope

This International Standard method describes a volumetric method for the determination of silver in jewellery alloys, preferably within the range of fineness stated in ISO 9202.

These alloys may contain copper, zinc, cadmium, and palladium. Apart from palladium, which must be precipitated before commencing titration, these elements do not interfere with this method of determination.

This method is intended to be used as the referee method for the determination of fineness in alloys covered by ISO 9202.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 sample is dissolved in dilute nitric acid. The silver content of the resulting solution is determined by titration with standard potassium bromide solution using a potentiometric indication of the equivalence point.

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 Nitric acid; 33 % HNO_3 (mass fraction), with sufficiently low content of halides (check with silver nitrate test).

4.2 Potassium bromide, solution, *c* (KBr) = 0,1 mol/l.

Dissolve 11,901 g of potassium bromide (dried at 105 °C) in water and dilute to 1 000 ml.

4.3 Disodium dimethylglyoxime octahydrate solution.

Dissolve 10 g of disodium dimethylglyoxime octahydrate in 1 000 ml of water.

4.4 Pure silver, minimum purity 999,9 parts by mass per thousand (‰).

5 Apparatus

5.1 Customary laboratory apparatus.

5.2 Motor-driven plunger or piston-type burette, linked to a potentiometer or automatic titrator and capable of delivering increments of 0,05 ml at the equivalence point.

5.3 Titration apparatus, with combination silver electrode coated with silver bromide and Hg/Hg_2SO_4 or other suitable reference electrode.

5.4 Analytical balance, with a reading accuracy of 0,01 mg.

6 Sampling

The sampling procedure for silver and silver alloys shall be performed in accordance with ISO 11596.

7 Procedure

WARNING — Suitable health and safety procedures should be followed.

7.1 Determination of potassium bromide factor

7.1.1 Preparation of silver standards

Weigh three samples of silver (4.4); each of 300 mg to 500 mg, accurately to the nearest 0,01 mg, and transfer them into three glass beakers. Add 5 ml of nitric acid (4.1) to each beaker, and warm gently to dissolve the silver. Tops of the beakers can be covered with watch glasses. Heat until evolution of nitrogen oxides ceases. Allow to cool. Rinse the watch glasses into beakers. Add the minimum volume of water required to satisfy the requirements of the titration apparatus (5.3) in respect of measurement and stirring. Put the beaker in the titration apparatus (5.3).

The mass of the standard silver samples should lie within 20 mg of the mass of silver in the sample portion (8.1).

7.1.2 Titration of the standard silver solution

Add, via the plunger-burette (5.2) under continuous stirring, sufficient potassium bromide solution (4.2) to precipitate about 95 % of the silver in the solution. Titrate the remaining silver in such a manner that the equivalence point can be interpolated from 0,05 ml additions of the potassium bromide solution.

NOTE This split titration approach can be effected automatically, using an automatic titrator with so-called dynamic volume dosing based on the measured potential difference across the electrodes in the titration apparatus (5.3).

7.1.3 Calculation of the potassium bromide standard solution factor

The potassium bromide standard solution factor, F, is calculated using Formula (1):

$$F = \frac{m_{\mathrm{AgF}}}{V_{\mathrm{AgF}}} \tag{1}$$

where

m_{AgF} is the mass of silver, in milligrams;

V_{AgF} is the volume, in millilitres, of potassium bromide solution at equivalence point.

The successive values of factor determinations shall not differ from each other by more than 0,05 % relative value. The mean value, $\overline{F}$, shall be used in subsequent calculations for maximum accuracy. The potassium bromide factor shall be determined immediately before analysis of the sample portions.

7.2 Determination

7.2.1 Preparation of the sample solution

Weigh between 300 mg and 500 mg of sample to the nearest 0,01 mg, and transfer to a glass beaker. Add 5 ml nitric acid (4.1) and warm gently to dissolve the alloy. Tops of the beakers can be covered with watch glasses. Heat until evolution of nitrogen oxides ceases. Allow to cool. Rinse the watch glasses into beakers. Transfer to the titration apparatus (5.2). Add the minimum volume of water to satisfy the requirements of the titration apparatus (5.3) in respect of measurement and stirring.

7.2.2 Elimination of palladium

Palladium, if present, shall be eliminated by addition of an aqueous solution of disodium-dimethylglyoxim-octahydrate (4.3). For each 100 mg of palladium, add 50 ml of this solution before commencing the titration.

7.2.3 Titration of the sample solution

Proceed exactly as for the standard solution. It can be necessary to carry out a pilot determination to obtain an approximate value of the silver content.

8 Calculation and expression of results

8.1 Calculation

The mass, m_{Ags}, in milligrams of silver in the sample portion is calculated using Formula (2):

$$m_{\mathrm{Ags}} = \overline{F} \cdot V_{\mathrm{Ags}} \tag{2}$$

where

$\overline{F}$ is the mean value of the potassium bromide standard solution factor expressed in milligrams of silver for each millilitre of solution;

V_{Ags} is the volume, in millilitres, of potassium bromide at equivalence point.

The silver content of the sample, W_{Ag}, in parts per thousand (‰) by mass is calculated using Formula (3):

$$W_{\mathrm{Ag}} = \frac{m_{\mathrm{Ags}}}{m_{\mathrm{s}}} \cdot 10^3 \tag{3}$$

where

m_{s} is the mass, in milligrams, of the sample. (7.2.1).

8.2 Repeatability

The results of duplicate determinations shall agree to better than 1 part per thousand (‰) by mass of silver. If the variation is greater than this, the assay 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 (ISO 11427);

d) silver content of the sample, in parts per thousand (‰) by mass, 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 laboratory carrying out this analysis;

i) signature of laboratory manager and operator.

Bibliography

[1] ISO 9202, *Jewellery — Fineness of precious metal alloys*

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