INTERNATIONAL STANDARD

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Jewellery — Determination of silver in silver jewellery alloys — Volumetric (potentiometric) method using sodium chloride or potassium chloride

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

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Foreword

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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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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 13756:1997), which has been technically revised with the following changes:

— addition of an analytical balance in Clause 5;

— change of requirement for sampling in Clause 6;

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

— International 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.

ISO/IEC Directives, Part 2 (sixth edition, 2011), 3.3.1 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."

ISO/IEC Directives, Part 2 (sixth edition, 2011), 3.3.2 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 sodium chloride or potassium chloride

1 Scope

This International Standard specifies a volumetric method for the determination of silver in silver 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.

NOTE   This method is an alternative recommended method to ISO 11427.

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 sodium chloride or potassium chloride 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 (HNO_3), 33 % HNO_3 (mass fraction), with sufficiently low content of halides (check with silver nitrate test).

4.2   Sodium chloride solution, $c(NaCl) = 0,1$ mol/l.

Dissolve 5,84 g of sodium chloride (dried at 105 °C) in water and dilute to 1 000 ml.

4.3   Potassium chloride solution, $c(KCl) = 0,1$ mol/l.

Dissolve 7,44 g of potassium chloride (dried at 105 °C) in water and dilute to 1 000 ml.

4.4   Disodium dimethylglyoxime octahydrate solution.

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

4.5   Silver, minimum purity 999,9 parts per thousand (‰) by mass.

5   Apparatus

5.1   Customary laboratory apparatus.

5.2   Motor-driven plunger or **piston-type burette**, linked to 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 or silver chloride coated silver electrode and Hg/Hg_2SO_4 electrode or other suitable reference electrode.

A silver chloride coating can be obtained by electrolysis, branching a silver electrode as anode in a dilute hydrochloric acid solution $c(HCl) = 0,1$ mol/l. After suitable surface preparation, apply an anodic current density of 1 mA/cm^2 for approximately one hour, until the silver surface is completely covered with silver chloride.

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 sodium chloride or potassium chloride standard solution factor

7.1.1   Preparation of silver standards

Weigh, to the nearest 0,01 mg, three samples, each of 300 mg to 500 mg of silver (4.5), and transfer them to glass beakers. Add 5 ml of nitric acid (4.1) to each, and warm gently to dissolve the silver. Keep the tops of the beakers 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 (5.3). Add the minimum volume of water, within the range of about 20 ml to 60 ml, to satisfy the requirements of the titration apparatus (5.3) with respect to measurement and stirring.

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

7.1.2   Titration of standard silver solution

Add, via the plunger-burette (5.2) under continuous stirring, sufficient sodium chloride standard solution (4.2) or potassium chloride standard solution (4.3) 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 sodium chloride or potassium chloride standard 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 sodium chloride or potassium chloride standard solution factor

The sodium chloride or potassium chloride standard solution factor, F, is calculated using Formula (1)

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

where

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

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

The 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 sodium chloride or potassium chloride standard solution factor shall be determined immediately before analysis of the sample portions.

7.2 Determination

7.2.1 Preparation of the sample solution

Weigh, to the nearest 0,01 mg, two sample portions, each containing between 300 mg and 500 mg of silver, and transfer them to glass beakers. Add 5 ml of nitric acid (4.1) to each and warm gently to dissolve the alloy. Keep the tops of the beakers 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 (5.3). Add water as per 7.1.1.

7.2.2 Elimination of palladium

Palladium, if present, shall be eliminated by adding an aqueous solution of disodium dimethylglyoxime octahydrate (4.4). For each 100 mg of palladium, add 50 ml of this solution before commencing titration.

7.2.3 Titration of the sample solution

Proceed exactly as per the standard solution. It may 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_{Ag} in milligrams of silver in the sample portion is calculated using Formula (2):

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

where

$\overline{F}$ is the sodium chloride or potassium chloride standard solution factor, in milligrams of silver for each millilitre of solution;

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