INTERNATIONAL STANDARD

ISO
13756

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

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

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.

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.

International Standard ISO 13756 was prepared by Technical Committee ISO/TC 174, *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 method to ISO 11427[1] which has been identified as the reference method.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 9202:1991, *Jewellery — Fineness of precious metal alloys*

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 recognised analytical grade and only distilled water or water of equivalent purity.

4.1 Nitric acid, 33 % (*m/m*), (ρ_{20} = 1,2 g/ml), with a halide content of less than 5 ppm.

---

[1] ISO 11427:1993, *Determination of silver in silver jewellery alloys — Volumetric (potentiometric) method using potassium bromide.*

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 1000 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 1000 ml.

4.4　Disodium dimethylglyoxime octahydrate solution.

Dissolve 10 g of disodium dimethylglyoxime octahydrate in 1000 ml of water.

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

5　Apparatus

Ordinary laboratory apparatus and the following.

5.1　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.

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

NOTE — 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.

6　Sampling

The sampling procedure for silver and silver alloys shall be agreed upon until a standard method has been published.

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

7　Procedure

7.1　Test portion

The mass of sample used for the titration shall contain between 300 mg and 500 mg of silver and shall be weighed to an accuracy of 0,01 mg.

7.2　Determination of sodium chloride or potassium chloride standard solution factor

7.2.1　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 (5.2). Add the minimum volume of water, within the range of about 20 ml to 60 ml, to satisfy the requirements of the titration apparatus (5.2) with respect to measurement and stirring.

NOTE — The mass of the standard silver samples should lie within 20 mg of the mass of silver in the test samples.

7.2.2 Titration of standard silver solution

Add, via the plunger-burette (5.1) and with continuous stirring, sufficient sodium chloride standard solution (4.2) or potassium chloride standard solution (4.4) to precipitate about 95 (m/m) % 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 may 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 vessel (5.2).

7.2.3 Calculation of sodium chloride or potassium chloride standard solution factor

The sodium chloride or potassium chloride standard solution factor, F, is calculated using the formula

$$F = \frac{m_{Ag}}{V_s}$$

where

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

V_s is the volume, in millilitres, of sodium chloride or potassium chloride standard solution at the equivalence point.

The factor determinations shall not differ from each other by more than 0,05 parts per hundred (%) by mass, relative value. The mean value, 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 test portions.

7.3 Determination

7.3.1 Preparation of test solutions

Weigh, to the nearest 0,01 mg, two test 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 (5.2). Add water as for 7.2.1.

7.3.2 Elimination of palladium

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

7.3.3 Titration of test solution

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

8 Expression of results

8.1 Method of calculation

8.1.1 Since the sodium chloride or potassium chloride factor, F (7.2.3), is expressed in milligrams of silver for each millilitre of solution, the mass m_{Ag}, in milligrams, of silver in the test portion is calculated using the formula

$$m_{Ag} = F \cdot V_s$$

8.1.2 Calculate the silver content of the sample, w_{Ag}, in parts by mass per thousand (‰), using the formula

$$w_{Ag} = \frac{m_{Ag}}{m_s} \times 10^3$$

where m_s is the mass, in milligrams, of the test portion (7.1).

8.2 Repeatability

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

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

i) signature of the laboratory manager and operator.