INTERNATIONAL STANDARD

ISO 11427

First edition
1993-05-01

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

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

Reference number
ISO 11427:1993(E)

Foreword

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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 11427 was prepared by Technical Committee ISO/TC 174, *Jewellery*.

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International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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

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

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 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 % (*m/m*), ρ_{20} = 1,2 g/cm^3, free of halide.

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

Dissolve 11,90 g of potassium bromide (dried at 105 °C) in water and dilute to 1 litre.

4.3 Disodium dimethylglyoxime octahydrate, solution.

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

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

5 Apparatus

Ordinary laboratory apparatus and

5.1 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.

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

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

7.2 Determination of the potassium bromide factor

7.2.1 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. Heat until the evolution of nitrogen oxides ceases. Cool, by dilution to about 100 ml with water, and transfer to the titration apparatus (5.2).

NOTE 2 The mass of the standard silver samples should lie within 20 mg of the mass of silver in the test portions (7.1).

7.2.2 Titration of the standard silver solution

Add, via the plunger-burette (5.1) and with continuous stirring, sufficient potassium bromide solution (4.2) to precipitate about 95 parts by mass per hundred (%) 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 3 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 the potassium bromide factor

The potassium bromide 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 potassium bromide solution at the equivalence point.

The factor determinations should not differ from each other by more than 0,05 parts by mass per hundred (%) relative value. The mean value, F, shall be used in subsequent calculations for maximum accuracy. The potassium bromide factor shall be determined immediately before analysis of the test portions.

7.3 Determination

7.3.1 Preparation of the test solution

Weigh between 300 mg and 500 mg of the test portion (7.1), to the nearest 0,01 mg, and transfer to a glass beaker. Add 5 ml of nitric acid (4.1) and warm gently to dissolve the alloy. Heat to drive off nitrogen oxides.

7.3.2 Elimination of palladium

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

7.3.3 Titration of the 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. The volume of potassium bromide solution at the equivalence point is interpolated from these measurements.

8 Expression of results

8.1 Method of calculation

8.1.1 Since the potassium bromide 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

$$m_{Ag} = FV_s$$

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.

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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.

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