International Standard 4524/4

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 4 : Determination of gold content

Revêtements métalliques — Méthodes d'essai des dépôts électrolytiques d'or et d'alliages d'or — Partie 4 : Détermination de la teneur en or

First edition — 1985-04-01

UDC 669.218.7 : 543 : 546.59 **Ref. No. ISO 4524/4-1985 (E)**

Descriptors : coating, metal coatings, electrodeposited coatings, gold plating, decorative coatings, protective coatings, tests, determination of content.

Price based on 2 pages

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4524/4 was prepared by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*.

Printed in Switzerland

Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 4 : Determination of gold content

1 Scope and field of application

This part of ISO 4524 specifies methods for determining the gold content of electrodeposited gold and gold alloy coatings for engineering, and decorative and protective purposes.

2 Reference

ISO 4524/1, *Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 1 : Determination of coating thickness.*

3 General

With certain gold alloy coatings having gold contents of less than about 90 %, there is a possibility that some of the alloying element may be dissolved in the nitric acid used during removal of the coating from the basis metal (see clause 4). If this should occur, erroneously high results may be obtained. In such cases, samples of gold coatings shall be supplied which can be mechanically stripped from the basis metal.

A high degree of precision in weighing is essential to attain sufficient accuracy of results, and relatively large samples may be required. If necessary, special thickly plated samples may be prepared for the purpose. However, test samples need not be limited to selected areas of articles and they can consist of one or more entire small articles from a plated batch, or samples may be taken from the more heavily plated areas (including outer and edge areas) of large articles.

If a gold content greater than 99 % has been agreed, special arrangements should be made between purchaser and supplier to ensure this purity. Such arrangements should be spectrographic analysis of trace contaminants using specially thickly plated test samples or agreement about controlling the gold plating solutions employed, to ensure that contamination by basis metal impurities does not exceed certain maxima.

The methods given in clauses 5 and 6 are recommended for determining the gold content of gold coatings containing less than 99 % gold, but other methods, for example the electron microprobe method, may be used.

4 Removal of gold coating from basis metal and undercoats (if any)

Cut or otherwise remove a suitable piece or pieces from the sample or, if necessary, take an entire article or articles and cut into suitably sized pieces. Remove as much as possible of the base material from the test specimen by mechanical means before stripping in order to minimize potential attack on the gold coating. Place in a small beaker and add a quantity of dilute nitric acid [1 volume of concentrated acid (ϱ = 1,42 g/ml) to 3 volumes of distilled water] at approximately 20 °C; this acid may, however, dissolve some of the alloying elements in the case of some alloy coatings. Allow the basis metal and undercoat (if any) to dissolve completely. Special care should be taken when the basis metal contains tin as the tin(IV) hydroxide formed clings tenaciously to the gold alloy covering. The precipitation of tin(IV) hydroxide can be prevented by adding 2 % (V/V) hydrofluoric acid or 5 % (V/V) fluoroboric acid to the diluted nitric acid. Decant, wash the residual coating several times with distilled water, and dry it at about 100 °C.

5 Fire assay

The determination can be carried out on 5 mg of stripped coating but, if possible, it is preferable to take a larger mass to obtain greater accuracy.

5.1 Procedure

Weigh the stripped coating on an assay balance to the nearest 0,01 mg and wrap in a sheet of lead foil (assay grade) together with a quantity of pure silver and a small piece of pure copper. The mass of the silver shall be 2 to 2,5 times that of the gold present and the mass of the copper shall be approximately 0,1 times that of the gold present. The mass of the lead foil shall be approximately 30 times the mass of the sample piece, with a minimum of 1 g.

Cupel in a muffle furnace designed for gold assaying at a temperature of 1 100 to 1 150 °C; flatten the resulting bead and anneal at a temperature of about 700 °C for about 1 min. Roll into a thin strip and reanneal. "Part" the annealed strip in nitric acid [$\varrho \approx$ 1,2 g/ml, about 25 % (m/m)] followed by nitric acid ($\varrho \approx$ 1,3 g/ml). In both cases the acid should be heated to boiling and kept just boiling throughout. Anneal at a temperature of about 700 °C for about 5 min. and weigh the resulting gold "cornet" to the nearest 0,01 mg.

One or more "proof" assays should be carried out alongside the sample assay, taking a known mass of pure gold corresponding approximately to that present in the sample piece and appropriate amounts of alloying elements.

5.2 Calculation

The gold content of the coating, w_{Au}, expressed as a percentage by mass, is given by the equation

$$w_{Au} = \frac{100\ m_1}{m_0}$$

where

m_0 is the mass, in milligrams, of the stripped coating;

m_1 is the mass, in milligrams, of the "cornet" from the fire assay of the sample piece after applying any correction from the proof assays.

6 Spectrophotometric and atomic absorption spectrometric methods

Use the chemical methods specified in ISO 4524/1, weighing the washed and dried coating before dissolution in the hot aqua regia.

7 Test report

The test report shall include at least the following information :

a) a reference to this part of ISO 4524, including an identification of the specific method used;

b) the result(s) of the test(s) carried out and the form in which these are expressed;

c) any unusual features noticed during the determination;

d) any operation not included in this part of ISO 4524 or in the part of ISO 4524 to which reference is made;

e) any other relevant information requested by the purchaser.