BS 6670-4: 1986 ISO 4524-4: 1985

Methods of test for

# Electroplated gold and gold alloy coatings —

Part 4: Determination of gold content

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

UDC 669.21.058:620.1



## Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Surface Coatings (other than Paints) Standards Committee (SRC/-) to Technical Committee SRC/5, upon which the following bodies were represented:

Assay Offices Committee of Great Britain

British Jewellers' Association

British Metal Finishing Suppliers' Association

British Telecommunications plc

Cutlery and Silverware Association

Design and Research Centre for the Gold, Silver and Jewellery Industries

**Electronic Engineering Association** 

Institute of Metal Finishing

Institution of Corrosion Science and Technology

Metal Finishing Assn.

Ministry of Defence

National Association of Goldsmiths of Great Britain and Ireland

This British Standard, having been prepared under the direction of the Surface Coatings (other than Paints) Standards Committee, was published under the authority of the Board of BSI and comes into effect on 30 May 1986

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The following BSI references relate to the work on this standard: Committee reference SRC/5 Draft for comment 81/50885 DC

#### ISBN 0 580 15120 4

#### Amendments issued since publication

Amd. No.	Date of issue	Comments

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#### National foreword

This Part of BS 6670 has been prepared under the direction of the Surface Coatings (other than Paints) Standards Committee. It is identical with ISO 4524-4:1985 "Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 4: Determination of gold content" prepared by Sub-committee 3, Electrodeposited coatings and related finishes of Technical Committee 107, Metallic and other non-organic coatings, of the International Organization for Standardization (ISO) with the active participation and approval of the UK.

A number of the Parts of BS 6670 are intended to replace certain of the appendices of BS 4292:1968, "*Electroplated coatings of gold and gold alloy*". Now that the test methods required for this purpose have been published, BS 4292:1968 is being revised. Upon completion of the revision it will no longer contain the texts of test methods and BS 4292:1968 will be withdrawn.

At present, the following Parts of BS 6670 have been published.

- Part 1: Determination of coating thickness;
- Part 2: Environmental tests;
- Part 3: Electrographic tests for porosity;
- Part 4: Determination of gold content;
- Part 5: Adhesion tests.

Further Parts of this standard will be prepared, if required.

**Terminology and conventions.** The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

In British Standards it is current practice to use the symbol "L" for litre rather than "l".

Wherever the words "this part of ISO 4524" appear, referring to this standard, they should be read as "this Part of BS 6670".

#### Cross-reference

International Standard	Corresponding British Standard
ISO 4524-1:1985	BS 6670 Methods of test for electroplated gold and gold alloy coatings Part 1:1986 Determination of coating thickness (Identical)

This Part describes methods of test only and should not be used or quoted as a specification. Reference to this Part should state that the method of test used is in accordance with BS 6670-4.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

#### Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 and 2, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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#### 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  $\bf 5$  and  $\bf 6$  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 (Q = 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.

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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

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

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.

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## Publications referred to

See national foreword.

BS 6670-4: 1986 ISO 4524-4: 1985

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