
International Standard



3160/2

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**Watch cases and accessories — Gold alloy coverings —
Part 2: Determination of fineness, thickness and corrosion
resistance**

Boîtes de montres et leurs accessoires — Revêtements d'alliage d'or — Partie 2: Détermination du titre, de l'épaisseur et de la résistance à la corrosion

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Watch cases and accessories — Gold alloy coverings — Part 2: Determination of fineness, thickness and corrosion resistance

0 Introduction

ISO 3160 consists of three parts:

- Part 1: General requirements.
- Part 2: Determination of fineness, thickness and corrosion resistance.
- Part 3: Mechanical properties.¹⁾

1 Scope

This part of ISO 3160 specifies methods to determine fineness, thickness and corrosion resistance for gold alloy coverings on watch cases and horological components.

The tests apply only to significant surfaces.

2 Field of application

This part of ISO 3160 applies to all gold alloy coverings specified in ISO 3160/1.

In this context the term "corrosion" includes tarnishing and oxidation, as well as surface penetration and the effects of the penetration of corrosive agents into gaps in the surface protection.

It is generally required that, except where specified to the contrary, gold alloy covered surfaces should not have suffered any damage after each of the proposed tests. In practice, however, this condition is never strictly fulfilled and certain minute changes are observed especially at the edges of the gold-covered parts. Consequently, interpretation of the results requires a certain amount of common sense and, if necessary, an agreement between supplier and customer. The presence of such almost inevitable faults makes it impossible to sell the tested item as new. In this respect, the tests are therefore to be considered as destructive.

The test methods apply to all gold alloy coverings specified in ISO 3160/1.

3 References

ISO 1463, *Metal and oxide coatings — Measurement of thickness by microscopical examination of cross-section.*

ISO 2177, *Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution.*²⁾

ISO 3160/1, *Watch cases and their accessories — Gold alloy coverings — Part 1 : General requirements.*

ISO 3497, *Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods.*

ISO 3543, *Metallic and non-metallic coatings — Measurement of thickness — Beta backscatter method.*

ISO 3868, *Metallic and other non-organic coatings — Measurement of coating thicknesses — Fizeau multiple-beam interferometry method.*

ISO 4524, *Metallic coatings — Test methods for electroplated coatings of gold and gold alloy.*³⁾

ISO 4538, *Metallic coatings — Thioacetamide corrosion test (TAA test).*

4 Definition

significant surface: That part of the surface which is to receive the gold alloy covering and which is essential to the appearance and serviceability of the component.

In case of no agreement between supplier and customer, significant surface will be any surface which can be touched by a 5 mm diameter ball.

1) In preparation.

2) At present at the stage of draft. (Revision of ISO 2177-1972.)

3) At present at the stage of draft. (Parts 1 to 7.)

7.2.3 Non-significant surfaces

The non-significant surfaces of the object shall be coated with a lacquer or a covering which is sufficiently resistant to prevent any attack on the protected metal throughout the duration of the test.

7.3 Continuity of the covering (porosity test)

7.3.1 Test for a cuprous base-metal with or without nickel, and die-cast zinc based alloy.

7.3.1.1 Test vessel

Use a suitable closed vessel, made of glass or acid-resistant plastic, and expose the sample to the corrosive atmosphere on all sides.

7.3.1.2 Test solution

The solution shall be of the following composition:

- Pure concentrated acetic acid: 25 % (m/m)
- Water: 75 % (m/m)

The vessel shall be filled with this solution to a depth of about 10 mm. The walls of the vessel shall be lined with thick white blotting paper which dips into the liquid.

7.3.1.3 Position of the sample

The sample shall be suspended on a glass hook at a distance of at least 30 mm from the liquid and the walls of the vessel.

7.3.1.4 Test temperature

The test temperature shall be 23 ± 2 °C.

7.3.1.5 Duration of the test

The duration of the test shall be 24 h.

7.3.1.6 Criteria

When observed, the sample shall not reveal to the naked eye either green droplets or accumulations of green deposits anywhere on the significant surface. On zinc die-cast alloys, no white deposit shall appear.

7.3.2 Test for a ferrous base-metal

7.3.2.1 Test vessel

The test shall be carried out in a suitable closed vessel made of glass or acid-resistant plastic, in which the sample is exposed to the corrosive atmosphere on all sides.

7.3.2.2 Test mixture

The supersaturated mixture shall have the following composition:

- Crystallized sodium disulphite $\text{Na}_2\text{S}_2\text{O}_5$: 45 % (m/m)
- Water: 55 % (m/m)

The vessel shall be filled with this solution to a depth of about 10 mm. Its walls shall be lined with thick white blotting paper which dips into the liquid.

7.3.2.3 Position of the sample

The sample shall be suspended on a glass hook at a distance of at least 30 mm from the liquid and the walls of the vessel.

7.3.2.4 Test temperature

The test temperature shall be 23 ± 2 °C.

7.3.2.5 Duration of the test

The duration of the test shall be 24 h.

7.3.2.6 Criteria

When observed, the sample shall not reveal to the naked eye any traces of corrosion anywhere on the significant surface. Slight general tarnishing of low-carat coatings is admissible.

7.3.3 Non-determination of base metal

Where the base metal cannot be determined, use the test described in 7.3.1.

7.4 Testing with saline and acid agents (synthetic perspiration test)

7.4.1 Test vessel

The test shall be carried out in a closed Pyrex glass (or equivalent) vessel, which can be heated in an oven to 40 °C.

7.4.2 Test solution

The solution used shall have the following composition:

- Sodium chloride: 20 g/l
- Ammonium chloride: 17,5 g/l
- Urea: 5 g/l
- Acetic acid: 2,5 g/l
- Lactic acid: 15 g/l
- Sodium hydroxide: quantity required to bring the pH to 4,7.

The vessel shall be filled with the solution to a depth of about 10 mm.

Annex A

Method of obtaining a sample of gold alloy covering

A.0 Introduction

In order to bring the gold alloy covering to a form suitable for analysis and in order to determine its mass, it is necessary to separate it from the base metal. The gold content is then determined by one of the methods described in 5.1 and 5.2. Since complete mechanical separation is possible only in the minority of cases, this process is carried out by dissolution of the base metal. In order to avoid any attack on the gold alloy covering, the time of exposure to the acid should be kept to a minimum. This may be ensured by one of the following measures:

- a) the corners and edges of the test sample or part of it shall be chamfered by filing, etc., in order to increase the area of attack. Some of the gold alloy covering will inevitably be lost by this process. However, no error will occur since this method is concerned only with the fineness of the gold alloy covering, not with the total amount of gold on the sample.
- b) the base metal is firstly removed as far as possible by mechanical means (by filing, milling, turning, etc.) and the residual base metal dissolved in acid.
- c) a sufficient quantity of the gold alloy covering is removed as far as possible by mechanical means (for example by scraping) and any adhering base metal removed by dissolution in acid.

A.1 Dissolution of the base metal

For copper, nickel, and iron alloys which are not passivated, nitric acid, ρ 1,1 g/ml (1 volume of concentrated nitric acid to 4 volumes of distilled water) is used. For gold alloy coverings of inferior fineness to 700 thousandths a more dilute nitric acid, for example ρ 1,05 g/ml (1 volume of concentrated nitric acid to 9 volumes of distilled water) should be used. The time for complete dissolution will be longer in the more dilute acid. If a problem appears (due to the presence of tin) concerning the solution of the base metal, 2 % of fluorhydric acid or 5 % of fluoboric acid can be added to the solution to avoid this problem (in a polypropylene vessel).

For stainless steel hydrochloric acid, ρ 1,125 g/ml (5 volumes of concentrated hydrochloric acid to 3 volumes of distilled water) is used. Aluminium alloys may be dissolved in 10 % (m/m) sodium hydroxide solution. Generally such samples have undercoats of copper and/or nickel which are not attacked by sodium hydroxide. These undercoats must therefore be dissolved in dilute nitric acid as described above after complete dissolution of the aluminium base metal.

In each case the base metal and undercoats (if any) are dissolved by heating the dissolving medium to a temperature of 90 to 95 °C. After complete dissolution of the base metal the solu-

tion is decanted, the residual coating washed several times first with dilute nitric acid, then with distilled water and dried at 110 °C.

Certain gold alloy coverings will disintegrate completely after dissolution of the base metal. In this case the solution is filtered through a weighed filtering crucible, the residual gold alloy coating is washed and dried as described above.

Special care should be taken with tin-containing base metals as the stannic hydroxide formed clings obstinately to the gold alloy covering. In order to avoid any excessive precipitation of stannic hydroxide, the nitric acid should be renewed several times. Boiling of the solution must be avoided. After complete dissolution of the base metal, the remaining gold alloy covering is washed with distilled water and then treated with hot hydrochloric acid (1 volume of concentrated hydrochloric acid to 9 volumes of distilled water) to ensure complete dissolution of the adhering stannic hydroxide. The remaining gold alloy covering is washed and dried as described above.

A.2 Analysis of the gold alloy covering

The remaining gold alloy covering obtained by dissolution of the base metal and undercoats (if any) is weighed and subjected to analysis by one of the methods described in 5.1 and 5.2. Reference should also be made to ISO 4524 and to standard books on analysis of noble metals, for example, F.E. Beamish: *The Analytical Chemistry of the Noble Metals* (Oxford, 1966).

NOTE — In spite of the precautions taken it will not always be possible to avoid chemical attack on the gold alloy covering during dissolution of the base metal. If this should occur, erroneously high results will be obtained. To avoid this error the following method, which involves the dissolution of the gold alloy covering instead of the base metal, may be used:

The test sample is weighed accurately and covered with a stop-off lacquer on all sides except for the area on which the fineness of the gold alloy covering is to be determined. An area of 0,1 cm² is the minimum that should be taken. The gold alloy covering is then dissolved anodically in a suitable electrolyte which either does not attack the underlying metal or, if so, only at a higher voltage. Commercial electrolytes are available from manufacturers of coulometric plating thickness meters. The dissolution of the gold alloy covering is followed by measuring the cell voltage. The end point is indicated by a sharp rise of cell voltage when the base metal or undercoat is exposed. The electrolysis is then immediately stopped. The resulting solution is subjected to chemical analysis for gold, preferably by atomic absorption spectrometry. The fineness of the gold alloy covering is calculated from the gold content of the solution and the loss in mass of the sample. A high degree of precision in weighing is essential to attain sufficient accuracy of results.

For details of this method, reference should be made to ISO 2177.