

# Method for the simulation of wear and corrosion for the detection of nickel release from coated items

The European Standard EN 12472:2005 has the status of a  
British Standard

ICS 39.060

## National foreword

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### Summary of pages

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English Version

## Method for the simulation of wear and corrosion for the detection of nickel release from coated items

Méthode de simulation de l'usure et de la corrosion pour la détermination du nickel libéré par les objets revêtus

Simulierte Abrieb- und Korrosionsprüfung zum Nachweis der Nickelabgabe von mit Auflagen versehenen Gegenständen

This European Standard was approved by CEN on 21 November 2005.

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## Foreword

This European Standard (EN 12472:2005) has been prepared by Technical Committee CEN/TC 283 "Precious metals - Applications in jewellery and associated products", the secretariat of which is held by UNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2006, and conflicting national standards shall be withdrawn at the latest by June 2006.

This European Standard supersedes EN 12472:1998.

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## Introduction

The wear of objects in contact with the skin depends very much on the type and shape of the objects and the activities of the person concerned. This procedure attempts to simulate the wear and corrosion on a coated article during two years of normal use. By its nature this is a pragmatic solution to the problems posed by the evaluation of coated items in contact with the skin, which may be subject to several kinds and varying degrees of wear.

In order to show compliance with Directive 94/27/EC, items should be tested in accordance with the appropriate European harmonised standard(s), the references of which have been published by the European Commission in the Official Journal of the European Communities. Currently, these are EN 1810, EN 1811 and this European Standard.

## 1 Scope

This European Standard specifies a method for accelerated wear and corrosion, to be used prior to the detection of nickel release from coated items that come into direct and prolonged contact with the skin.

## 2 Normative references

The following referenced documents are indispensable for the application of this European Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1811, *Reference test method for release of nickel from products intended to come into direct and prolonged contact with the skin*

## 3 Principle

The items to be tested are exposed to a corrosive atmosphere before being placed into a tumbling barrel together with a wear medium of abrasive paste and granules. The barrel is rotated so as to subject the test pieces to wear from the wear medium. The items are then tested for nickel release in accordance with EN 1811.

## 4 Reagents and materials

Except where indicated, all reagents and materials that can come into contact with samples or reagents shall be demonstrably free of nickel, and all reagents shall be of recognized analytical grade or better.

### 4.1 Corrosion

#### 4.1.1 Container

Container with a lid and a device for suspending the test pieces, all parts made of inert material (e.g. glass or plastic).

**4.1.2 Corrosive medium**

Dissolve 50 g DL-lactic acid, > 85 % purity, and 100 g sodium chloride in 1 000 ml de-ionized water.

**4.1.3 Degreasing solution**

An appropriately diluted, neutral, commercially available detergent shall be used, for example, a 0,5 % aqueous solution of sodium dodecylbenzene sulfonate.

**4.1.4 De-ionized water**

De-ionized water, specific conductivity maximum 1  $\mu$ S/cm.

**4.1.5 Laboratory oven**

Laboratory oven, capable of maintaining a temperature of  $(50 \pm 2)$  °C.

**4.2 Wear**

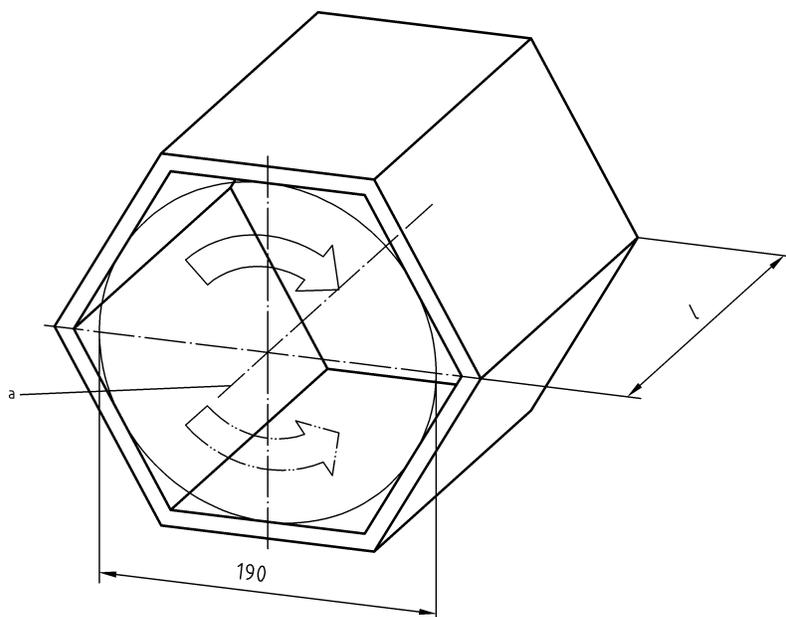
**4.2.1 Tumbling barrel and retaining assembly**

The tumbling barrel and retaining assembly shall be in accordance with the following description:

- barrel of hexagonal cross-section and internal diameter of 19 cm perpendicular distance between opposite sides designed to rotate around its axis which is orientated horizontally (see Figure 1);
- retaining assembly, suitable for attaching the test items so that they do not come into contact with each other during tumbling;
- retaining assembly, with items attached, shall be inserted into the barrel for tumbling.

NOTE Examples are given in Annex A of retaining assemblies suitable for attaching typical items.

Dimensions in millimetres

**Key**

- l length of barrel axis, as required
- a axis of rotation

**Figure 1 — View of tumbling barrel****4.2.2 Wear test apparatus**

A device capable of imparting to the barrel (4.2.1) a constant ( $30 \pm 2$ ) rotations per minute. The device shall be capable of allowing the direction of rotation to be reversed.

NOTE Information on sourcing suitable equipment is available from the CEN Management Centre.

**4.2.3 Abrasive paste**

Abrasive paste produced for dry-tumbling barrels. The abrasive paste shall comprise:

- 6 % to 8 % Ester wax of montanic acids-Wax E [CAS No. 73138-45-1];
- 3 % Octadecanoic acid (stearic acid) [CAS No. 57-11-4];
- 30 % to 35 % Petroleum distillates, hydrotreated light paraffinic [CAS No. 64742-55-8];
- 2 % Polyethylene glycol cetyl/oleyl ether [CAS No. 68920-66-1] or Triethanolamine [CAS No. 102-71-6];
- 48 % Silicon dioxide (quartz) 200  $\mu\text{m}$  mesh size [CAS No. 14808-60-7];
- 6 % to 9 % De-ionized water.

NOTE Information on sourcing a suitable paste is available from the CEN Management Centre.

#### **4.2.4 Granules**

This material shall have the following composition:

- outer shells of coconuts, walnuts, peanuts and almonds, mixed in a ratio 1:1:1:1 by weight, ground and sieved to give a mixture of particles having dimensions between 0,8 mm and 1,3 mm.

NOTE Information on sourcing suitable granules is available from the CEN Management Centre.

#### **4.2.5 Wear medium**

The medium is composed of abrasive paste (4.2.3) and granules (4.2.4) which are mixed as indicated in 5.3.1.

### **5 Procedure**

#### **5.1 Sample preparation**

Before being subjected to the corrosion (5.2) and wear (5.3) procedures, different parts of some items may need to be separated from each other.

NOTE 1 Parts of items which are not intended to come into prolonged contact with the skin may be removed before being subjected to corrosion and/or wear.

Gently swirl the sample(s) for 2 min in a degreasing solution (4.1.3) at room temperature. Rinse thoroughly with de-ionized water (4.1.4) and gently dry with absorbent paper. After degreasing, samples should be handled with plastic forceps or clean protective gloves.

NOTE 2 This cleaning stage is intended to remove extraneous grease and skin secretions due to handling, but not any protective coatings.

#### **5.2 Corrosion procedure**

If applicable, the items to be tested shall first be opened to reveal critical surfaces. The items are suspended a few centimetres above the corrosive medium (4.1.2) in a closed container (4.1.1) and placed in the laboratory oven (4.1.5) for 2 h at 50 °C. Remove the container from the oven and carefully open it under a fume hood. Rinse the items with de-ionized water (4.1.4). Place on absorbent paper and allow to dry at room temperature for about 1 h, then perform the wear procedure (5.3) without delay.

NOTE This stage is intended to affect metallic coatings, as well as lacquers and plastic coatings.

#### **5.3 Wear procedure**

##### **5.3.1 Preparation of wear medium**

Weigh a sufficient quantity of granules (4.2.4) in order to fill the tumbling barrel (4.2.1) to half its depth. Add 7,5 g of abrasive paste (4.2.3) for every kg of granules and homogenize by rotating in the barrel for 5 h. If the wear medium has not been used within 1 week, re-homogenise by rotating it in the barrel for 1 h.

NOTE This procedure coats the granules with the abrasive paste, forming the wear medium that is used to simulate wear.

Keep the wear medium in a closed container until use and between uses.

After 2 wear procedures, add 7,5 g more abrasive paste for every kg of granules. Re-homogenise the wear medium by rotating it in the barrel for a further 5 h.

After a total of 4 wear procedures, discard the wear medium and prepare fresh material.

### 5.3.2 Attachment of test items

Items shall be attached inside the retaining assembly so that they cannot come into contact with each other, or collide with the barrel walls or other parts that could cause damage during tumbling.

NOTE Annex A gives examples of how typical items are attached.

### 5.3.3 Tumbling

Place the retaining assembly with the mounted items into the empty tumbling barrel, then fill the barrel to half its depth with the wear medium (4.2.5). Close the tumbling barrel and place it horizontally on the rotating system (4.2.2).

Rotate the tumbling barrel at a speed of  $(30 \pm 2)$  rotations per minute for a total of  $5 \text{ h} \pm 5 \text{ min}$ . The direction of the rotation shall be reversed after  $2,5 \text{ h} \pm 5 \text{ min}$ .

NOTE It is permissible to leave the items in the barrel overnight.

## 5.4 Determination of nickel release

Remove the retaining assembly from the barrel and detach the items. Gently wipe off any remaining wear medium using a soft cloth or paper tissues.

Check the items for unexpected damage, e.g. stone(s) missing from broach. If necessary and justified, rectify damage or exclude item(s) from further testing. Test the items for nickel release in accordance with EN 1811.

NOTE If only indicative information on the extent of nickel release is required, such information can be obtained by performing one of the tests specified in CR 12471.

## 6 Test report

In addition to the information required by EN 1811, the test report shall contain the following particulars:

- a) reference to this European Standard, i.e. EN 12472:2005;
- b) if appropriate, a description of removed or opened parts and method of mounting;
- c) details of any deviations from this standard method, if relevant;
- d) any unusual feature(s) observed during the test.

## Annex A (informative)

### Detailed examples of how to attach different types of items

#### A.1 General

A retaining assembly is used to mount the test items. This is inserted into the hexagonal drum.

Materials which have been found suitable for fixing test items in the retaining assembly are silicone rubber sheet, silicone rubber straps, and nylon lines of different dimensions.

#### A.2 Attachment of items with only one side in contact with the skin

Items having one surface that can come into prolonged contact with the skin, and another surface that is less likely to come into prolonged contact with the skin, should be mounted so that they are fixed and cannot move inside the barrel. Examples are spectacle frames and some watch straps. The test samples should be fixed so that they are positioned with the surface that can come into contact with the skin turned towards the rotation axis, which is the centre of the barrel, and with the other surface turned towards the wall of the barrel. If the items are relatively long, for example, spectacles or watch straps, then their long axis should, if possible, be fitted parallel to the barrel axis. Items for wear should be positioned between 10 mm and 30 mm from the barrel walls.

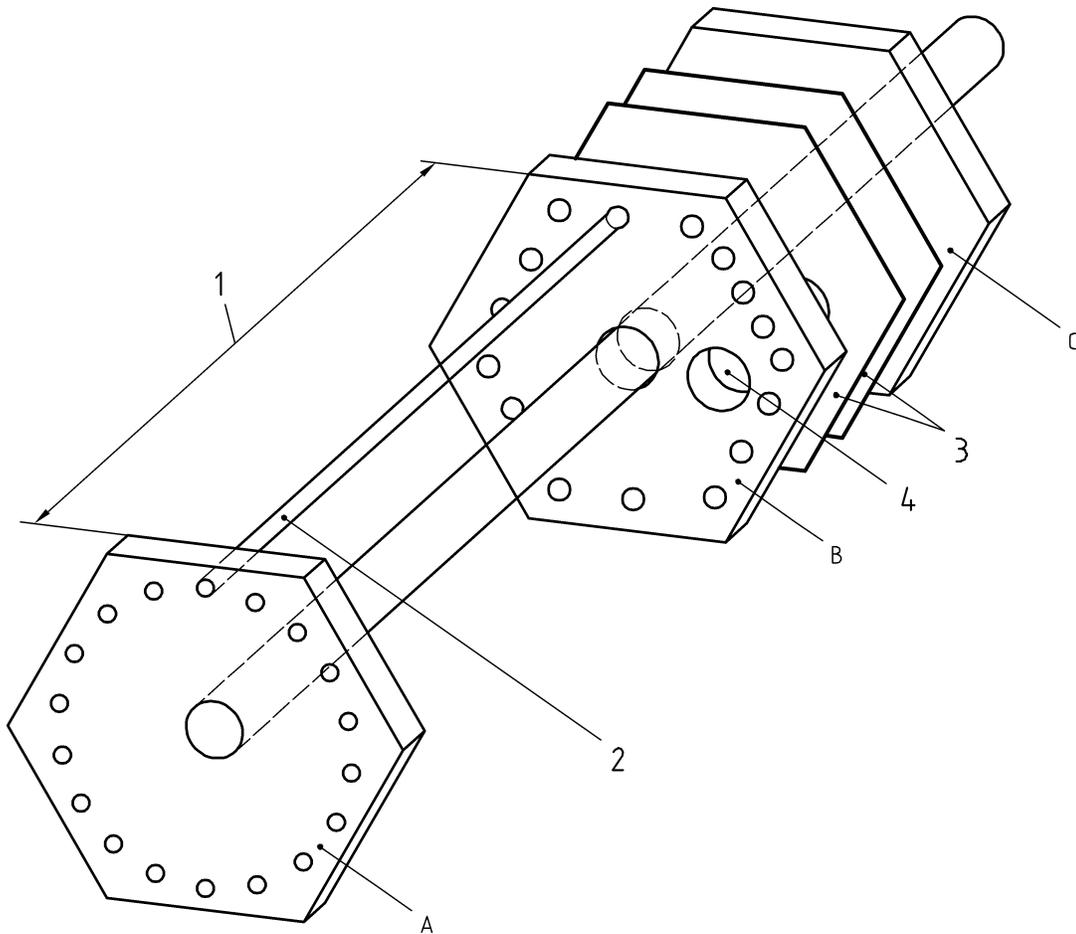
#### A.3 Spectacle frames

EN ISO 12870 gives details on which parts of spectacle frames should be tested.

The assembly consists of a threaded rod which carries three metal hexagonal plates - see Figure A.1. The end plate (A) is drilled part way through with holes of nominal diameter 1,5 mm, or as appropriate, to take the ends of the tips of the sides. The next plate (B - see Figure A.2) is perforated with holes of nominal diameter 5,0 mm, or as appropriate, to take the joint ends of the sides, together with an aperture of 40 mm nominal diameter to act as a filling hole for the abrasive mixture. A silicone rubber sheet with small holes matching the position of those in plate (B) holds the sides firmly to prevent them from rotating in the assembly. A second, un-perforated, silicone sheet presses the sides into plate (A). The final plate (C) is un-drilled apart from the hole for the threaded rod. A threaded nut on the inside of the last two plates holds them the required distance from plate A, while a second nut on the outside clamps the assembly together. The sides should be mounted with the inside surface towards the axis of the assembly.

Spectacle fronts should be mounted so that one of the lugs is inserted into the holes in plate (B). It may be necessary to straighten the lug or to twist the rim immediately either side of the lug so that the lug will fit into the hole. The opposite lug should be firmly secured to plate (A) with adhesive tape. The back surface of the front should be mounted towards the axis of the assembly. Whatever method is used to fix spectacle fronts (e.g. using only adhesive tape), it is important to ensure that parts of the sample to be tested are not covered or protected.

After fitting the test samples between plates (A) and (B), the retaining assembly should be inserted into the barrel and the wear medium added. The second silicone rubber sheet and plate (C) together with its retaining nut should then be fitted, and the drum closed.

**Key**

- 1 adjust as required
- 2 sample side
- 3 silicone sheets (plain and perforated)
- 4 fill hole
- A, B, C Metal plates

**Figure A.1 — Retaining assembly specifically designed for mounting spectacle frames (Not to scale)**

Dimensions in millimetres

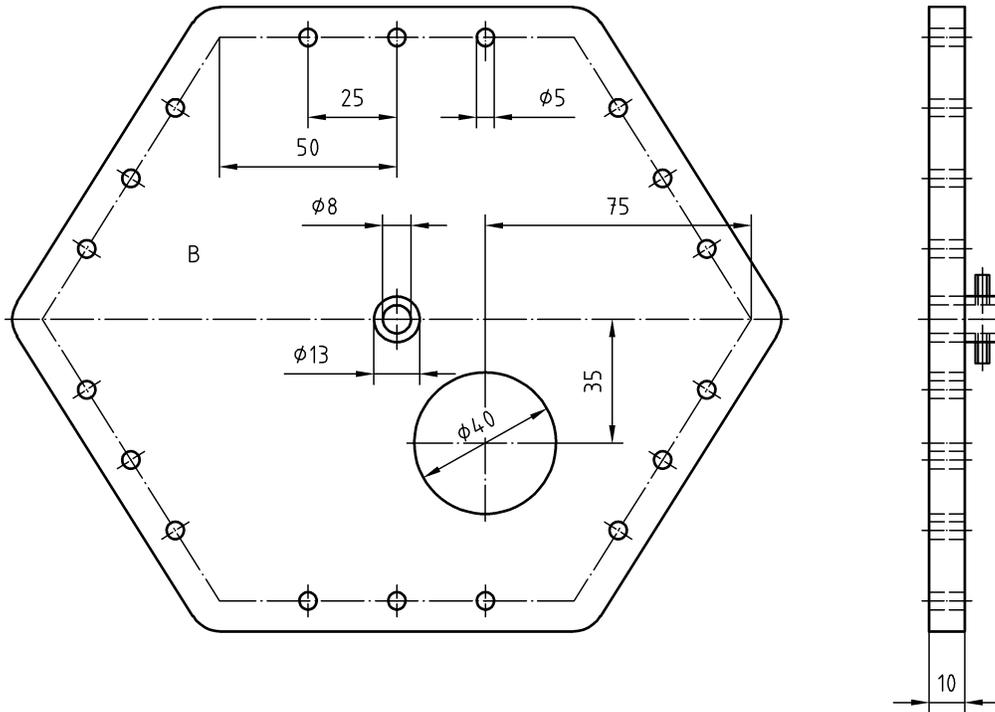


Figure A.2 — Diagram of the middle plate (B) and the spacing nut

#### A.4 Chains, watches, bracelets, rings etc.

Test items shall be attached to the retention assembly so that, where it is possible, the surfaces that come into direct and prolonged contact with the skin, are mounted parallel to the barrel wall and face towards the centre of the barrel. When attaching the items to the retaining assembly, they need to be sufficiently secure so that they cannot come into contact with each other, or collide with the barrel walls or other parts that could cause damage during tumbling.

Small items such as rings, buttons, brooch etc. may be attached to the retaining assembly through the holes in the plastics strips, using nylon thread or other suitable materials. For items where it is not possible, to use the plastic strips, due either to their size or shape, then they can be directly attached to the retaining assembly again using nylon thread or other suitable materials. Examples are shown in Figure A.3.

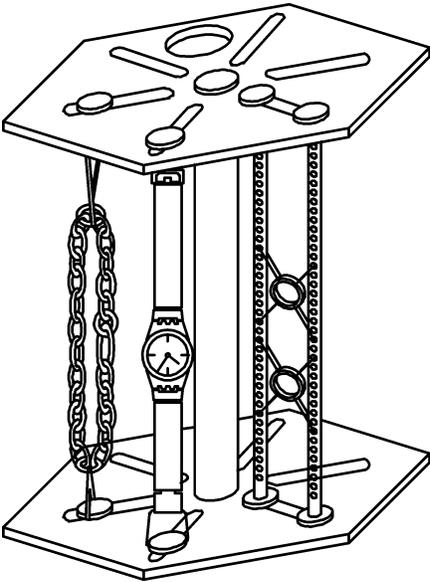


Figure A.3 — Examples of how to fix different types of items in retaining assembly

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- [1] European Parliament and Council Directive 94/27/EC of 30 June 1994 amending for the 12<sup>th</sup> time Directive 76/769/EEC on the approximation of the laws, regulations and administrative provisions of the Member States relating to restrictions on the marketing and use of certain dangerous substances and preparations, Official Journal of the European Communities (OJ) No. L 188, 22.7.1994, p. 1
- [2] EN 1810:1998, *Body-piercing post assemblies — Reference test method for determination of nickel content by flame atomic absorption spectrometry*
- [3] CEN/CR 12471:2002, *Screening tests for nickel release from alloys and coatings in items that come into direct and prolonged contact with the skin*
- [4] EN ISO 12870, *Ophthalmic optics — Spectacle frames — Requirements and test methods (ISO 12870:2004)*



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