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Incorporating corrigendum May 2012

BSI Standards Publication

Reference test method for release of nickel from all post assemblies which are inserted into pierced parts of the human body and articles intended to come into direct and prolonged contact with the skin

National foreword

This British Standard is the UK implementation of EN 1811:2011+A1:2015, incorporating corrigendum May 2012. It supersedes BS EN 1811:2011, which is withdrawn.

The start and finish of text introduced or altered by amendment is indicated in the text by tags. Tags indicating changes to CEN text carry the number of the CEN amendment. For example, text altered by CEN amendment A1 is indicated by [A1] ⟨A1⟩.

The UK participation in its preparation was entrusted to Technical Committee STI/53, Specifications and test methods for jewellery and horology.

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This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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31 July 2012	Implementation of CEN corrigendum May 2012: Clauses 5.8 and 5.9 replaced
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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1811:2011+A1

July 2015

ICS 39.060

Supersedes EN 1811:2011

English Version

Reference test method for release of nickel from all post assemblies which are inserted into pierced parts of the human body and articles intended to come into direct and prolonged contact with the skin

Méthode d'essai de référence relative à la libération du nickel par les assemblages de tiges qui sont introduites dans les parties percées du corps humain et les produits destinés à entrer en contact direct et prolongé avec la peau

Referenzprüfverfahren zur Bestimmung der Nickellässigkeit von sämtlichen Stäben, die in durchstochene Körperteile eingeführt werden und Erzeugnissen, die unmittelbar und länger mit der Haut in Berührung kommen

This European Standard was approved by CEN on 5 February 2011 and includes Corrigendum 1 issued by CEN on 30 May 2012 and Amendment 1 approved by CEN on 20 June 2015.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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European foreword

This document (EN 1811:2011+A1:2015) has been prepared by Technical Committee CEN/TC 347 “Methods for analysis of allergens”, the secretariat of which is held by [A1] SNV [A1].

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 January 2016, and conflicting national standards shall be withdrawn at the latest by January 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document includes Corrigendum 1 issued by CEN on 30 May 2012 and Amendment 1 approved by CEN on 20 June 2015.

This document supersedes [A1] EN 1811:2011 [A1].

The start and finish of text introduced or altered by amendment is indicated in the text by tags [A1] [A1].

The modifications of the related CEN Corrigendum have been implemented at the appropriate places in the text and are indicated by the tags [AC] [AC].

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This document supports essential requirements of Commission Regulation (EC) No 1907/2006 (REACH) of the European Parliament and the Council.

[A1] *deleted text* [A1]

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Adverse skin reaction to nickel has been known for many decades. Nickel is the most frequent cause of contact allergy in Europe, and 10 % to 20 % of the patch tested female population and 1 % to 3 % of the patch tested male population are allergic to nickel. Skin absorption of nickel ions, which are released from some nickel-containing materials which are inserted into pierced ears or other pierced parts of the human body or which are in direct and prolonged contact with the skin, causes sensitisation. Further exposure to soluble nickel salts results in allergic contact dermatitis. It is known that sensitisation to nickel requires higher exposure levels than does the elicitation in already sensitised individuals. There is a large variation in the degree of sensitivity to nickel between individuals. This widespread health problem has forced the introduction of a number of measures designed to reduce its prevalence. These measures include the requirements of this standard which provides an *in-vitro* chemical test that correlates as far as possible with the variable human biological reactions that occur when metallic articles containing nickel are in direct and prolonged contact with the skin and pierced parts of the body. The standard provides a measure of the amount of nickel release from an article immersed for one week in artificial sweat. The standard also describes the preparation of a quality control material intended to assist a laboratory in achieving an acceptable precision.

Clinical patch-testing of a small selection of nickel-containing alloys and coatings on nickel-sensitized persons indicates that high and low results achieved with the present analytical method correspond closely with patch-test reactivity. Moreover, a nickel migration limit of 0,5 µg/cm^2/week for articles intended to come into direct and prolonged contact with the skin and a nickel migration limit of less than 0,2 µg/cm^2/week for all post piercing assemblies inserted into pierced ears and other pierced parts of the human body has been set in Commission Regulation (EC) No 1907/2006 of the European Parliament and the Council (in the current version).

1 Scope

This European Standard specifies a method for simulating the release of nickel from all post assemblies which are inserted into pierced ears and other pierced parts of the human body and articles intended to come into direct and prolonged contact with the skin in order to determine whether such articles are in compliance with No. 27 Annex XVII of Regulation (EC) No 1907/2006 of the European Parliament and of the Council (REACH).

Spectacle frames and sunglasses are excluded from the scope of this European Standard.

NOTE Spectacle frames and sunglasses are subject to the requirements of EN 16128:2011 which provides an unchanged re-publication of the technical requirements that had previously been specified in EN 1811:1998, but restricted in scope to apply only to spectacle frames and sunglasses.

2 Normative references

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

EN 12472, *Method for the simulation of wear and corrosion for the detection of nickel release from coated items*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
barrette
component used to secure the watchstrap to the case

3.2
homogeneous
consisting of a single material having a common surface finish

3.3
post assembly
ear stud or body piercing article

3.4
release solution
solution resulting from the release procedure according to 8.2

3.5
representative
best estimate for the effective release rate of all surfaces which are in direct and prolonged contact with the skin or pierced parts of the body under normal conditions of use

NOTE This property is defined with respect to the release rate.

3.6
sample area
a surface(s) that is(are) immersed in the test solution and not covered with a masking agent

3.7
test solution
solution as prepared according to 8.1

3.8
watch crown
winder used to alter the time/date

4 Principle of the procedure

The article to be tested for nickel release is placed in an artificial sweat test solution for one week. The concentration of dissolved nickel in the solution is determined by an appropriate analytical method, for example inductively-coupled plasma spectrometry. The nickel release is expressed in micrograms per square centimetre per week (µg/cm^2/week).

NOTE Indicative information on the extent of nickel release can be obtained by performing one of the tests specified in CR 12471.

5 Reagents

Except where indicated, all reagents shall be of recognized pro analysis, p.a., grade or better and shall be free of nickel.

5.1 Deionised water according to EN ISO 3696:1995, grade 2.

5.2 Sodium chloride.

5.3 DL-lactic acid, ρ = 1,21 g/ml, > 88 % (m/m).

5.4 Urea.

5.5 Sodium hydroxide in solid tablets, min 98 % pure dehydrate.

5.6 Preparation of 1 M sodium hydroxide solution.

Weigh 4 g ± 0,01 g of sodium hydroxide (5.5) and transfer into a 100 ml beaker and add 50 ml of deionised water (5.1). Stir and cool to room temperature. Transfer the solution to a 100 ml volumetric flask and make up to volume with deionised water (5.1).

5.7 Preparation of 0,1M sodium hydroxide solution

Add 25 ml of 1 M sodium hydroxide (5.6) in a 250 ml volumetric flask and make up to volume with deionised water (5.1).

5.8 ▌AC〉 Hydrochloric acid, ρ = 1,16 g/ml, 32 % m/m. 〈AC▐

5.9 ▌AC〉 Preparation of 0,1 M hydrochloric acid solution

Transfer 1 ml of hydrochloric acid (5.8) into a 100 ml volumetric flask and make up to volume with deionised water (5.1). 〈AC▐

5.10 Nitric acid, ρ = 1,40 g/ml, 65 % (m/m).

5.11 Dilute nitric acid, approximately 5 % (m/m).

Transfer 30 ml of nitric acid (5.10) into a 500-ml beaker containing about 350 ml of deionised water (5.1). Stir and cool to room temperature. Transfer the solution to a 500-ml volumetric flask and make up to volume with deionised water.

5.12 Degreasing solution.

Dissolve 5 g of an anionic surface-active agent such as sodium dodecylbenzene sulfate or sodium alkylaryl sulfate in 1 000 ml deionised water (5.1). An appropriately diluted, neutral, commercially available detergent may be used.

5.13 Wax or lacquer (suitable for electroplating purposes) capable of protecting a surface from nickel release.

The wax or lacquer shall be shown to prevent nickel release from a nickel-releasing surface when one or more coats of the wax or lacquer are applied in the same manner as on a test sample and shall not affect the nickel content of the release solution. The suitability of the wax / lacquer shall be tested.

NOTE Information on sourcing of a suitable wax or lacquer is available from the CEN/CENELEC Management Centre.

6 Apparatus

6.1 A pH-meter, accurate to ± 0,05 pH.

6.2 An analytical instrument capable of detecting a concentration of 0,01 mg nickel per litre in the final release solution.

It is recommended that either an inductively-coupled plasma spectrometer (ICP-OES, optical emission, or ICP-MS, mass spectrometer) or an electro thermal excitation atomic absorption spectrometer (GFAAS) is used.

6.3 Thermostatically controlled water-bath or oven with or without cooling option, capable of maintaining a temperature of (30 ± 2) °C.

6.4 A vessel with lid, both composed of a non-metallic, nickel-free and nitric-acid-resistant material, such as glass and/or polypropylene and/or polytetrafluoroethylene and/or polystyrene.

The sample shall be suspended in the liquid by a holder made from the same materials as listed above, so as to avoid contact of the sample area (7.1.1) with the walls and base of the vessel. The size and shape of vessel and holder shall be chosen so as to minimize the volume of test solution required to completely cover the article to be tested.

In order to remove any trace of nickel, the vessel and holder shall be pre-treated by being stored in a solution of dilute nitric acid (5.11) for at least 4 h. After acid cleaning, rinse the vessel and holder with deionised water and dry.

6.5 Device for length measuring, for example a digital calliper with a minimum resolution of 50 µm or a micrometer with a minimum resolution of 5 µm.

7 Samples

7.1 Sample area

7.1.1 Definition of sample area

Only the surface(s) that come(s) into direct and prolonged contact with the skin and/or that have contact with the pierced parts of the body shall be tested (sample area).

In case of articles which are made of uniform material(s), consideration should be given to testing the whole surface (whether or not it is all in direct and prolonged contact with the skin or with pierced parts of the body) since errors can be introduced by the masking process (see 7.1.3).

The test laboratory shall refer to C.4 in order to determine which surfaces are to be tested.

7.1.2 Determination of sample area

Determination of the sample area (*a*) in square centimetres is achieved by marking the contour of the sample area assuming that the article is worn or used as intended (refer to Annex C) and measuring it by an appropriate measuring device (6.5). In order to achieve the required degree of analytical sensitivity, a minimum sample area of 0,2 cm^2 shall be tested. If necessary, identical articles may be tested together to obtain this minimum area.

The closer the nickel release is to the limits laid down in the regulation, or the smaller the sample area is, the more precise the surface measurement needs to be.

7.1.3 Masking of areas other than sample area

In order to prevent release of nickel from areas other than the sample area, such areas shall be removed or protected from the test solution. This can be achieved after degreasing (refer to 7.2) by the application of one or more coatings of a wax or lacquer (5.13) which has been shown to protect from nickel release.

The test laboratory shall refer to C.4 in order to determine which surfaces are to be tested.

7.2 Sample degreasing before testing

Gently swirl the sample for 2 min in degreasing solution (5.12) at room temperature. Rinse thoroughly with deionised water and dry using an absorbing cloth. After degreasing, articles shall be handled with plastic forceps or clean protective gloves.

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

7.3 Quality control samples

As a quality control check, the nickel release from a quality control sample shall be determined (refer to Annex B) on a frequent basis.

The quality control sample shall be degreased in the same way as the sample according to 7.2 and shall be used only once.

8 Procedure

8.1 Preparation of test solution

The test solution consists of deionised water (5.1) containing:

— 0,5 % (m/m) sodium chloride (5.2);

— 0,1 % (m/m) lactic acid (5.3);

— 0,1 % (m/m) urea (5.4); and

— 1 M (5.6) and 0,1 M (5.7) sodium hydroxide solution.

The test solution shall be prepared as follows:

Pour 900 ml of freshly prepared deionised water (5.1) to a 1 000 ml beaker. Add 1,00 ± 0,01 g of urea (5.4), 5,00 ± 0,05 g of sodium chloride (5.2) and 1,00 ± 0,01 g of lactic acid (5.3), and stir until dissolved.

Calibrate a pH meter in accordance with the manufacturer's instructions using freshly prepared buffer solutions.

Immerse the pH electrode into the test solution and measure the pH. Slowly and gently, add drop by drop a volume of 1 M sodium hydroxide (5.6) until a pH of 5,5 ± 0,05 is reached and subsequently with continuous stirring, add slowly and gently drop by drop a volume of 0,1 M sodium hydroxide (5.7) until a pH 6,5 ± 0,05 is reached and remains stable.

Measure the pH after 10 min from the last addition of 0,1 M sodium hydroxide to ensure that the pH is in the range 6,5 ± 0,05.

Transfer the solution to a 1 000 ml volumetric flask and make up to volume with deionised water. Before use, ensure that the pH of the test solution is in the range of pH 6,5 ± 0,05.

If it is necessary to reduce the pH of the solution to 6,5 ± 0,05 before testing, this shall be done by adding slowly and gently with continuous stirring drop by drop a volume of 0,1 M hydrochloric acid (5.9).

The test solution shall be prepared daily.

8.2 Release procedure

NOTE In the following text the term "test solution" represents the solution as prepared according to 8.1, the "release solution" is the solution resulting from the release procedure. See also definitions.

Place the sample, suspended by its holder, in the test vessel (6.4). Add an amount of test solution corresponding to approximately 1 ml per cm^2 sample area. The suspended sample area shall be totally immersed. It is not necessary to immerse areas which are completely protected by wax or lacquer (see 7.1.3). The minimum volume of test solution shall be 0,5 ml irrespective of the surface area. Note the sample area and the amount of the test solution used. Close the vessel with a tight lid in order to prevent evaporation of the test solution. Leave the vessel undisturbed in a thermostatically controlled water-bath or oven (6.3) at (30 ± 2)°C for (168 ± 2) h without agitation.

The quality control sample (7.3) shall be determined and suspended in an appropriate volume of test solution. It shall be treated in the same manner as a sample.

After (168 ± 2) h, slowly remove the sample from the release solution. To collect solution contained in cavities of the sample, the sample shall be turned appropriately. The sample shall not be rinsed.

Quantitatively transfer the release solution to an appropriately sized volumetric flask washed with dilute nitric acid (5.11). In order to prevent redeposition of dissolved nickel, add dilute nitric acid (5.11), to achieve a concentration of about 1 % nitric acid, when the flask is made up to volume (V in ml) using the test solution.

The choice of volumetric flask size shall take into account the sensitivity of the instrumentation used for the nickel determination (refer to 6.2). The minimum final volume to which the release solution may be diluted is 2 ml.

Filtering should be avoided if possible as the expected concentration change and the possibility of contamination can influence the result.

Test solution shall be stored in a refrigerator below 10 °C if used in the preparation of calibration solutions.

8.3 Determination of nickel

8.3.1 General

Determine the nickel content of the release solution using an analytical spectrometer (refer to 6.2).

For the determination of nickel using an analytical spectrometer the following procedures shall be applied.

8.3.2 Calibration solutions

The calibration solutions used for the nickel determination shall match the matrix of the test solution plus any added nitric acid and adequately cover the concentration range of nickel in the release solutions.

8.3.3 Detection limit and quantification limit

For the determination of the limit of detection and quantification, it is recommended to apply an established method such as the IUPAC standard as described in [4] and to report concentration values close to the detection limit and limit of quantification in terms of release rate. If it is required to dilute the release solution, the nickel concentration of the diluted release solution shall exceed the limit of quantification.

8.3.4 Number of test samples

A minimum of three test samples of the same batch shall be tested wherever possible.

8.3.5 Number of replicate measurements

At least two replicate measurements of each release solution shall be carried out.

8.4 Blank tests

For each sample, duplicate blank tests shall be carried out at the same time as the testing of the sample. Identical vessels and holders shall be used and the test procedure is identical except that no sample is placed in the vessels. Identical amounts of test solution, and dilute nitric acid shall be used.

9 Calculations

9.1 Nickel release

The nickel release of a sample, *d*, expressed in micrograms per square centimetre per week (µg/cm^2/week), is given by the equation:

$$d = \frac{V \times (C_1 - C_2)}{1\,000 \times a} \quad (1)$$

where

a is the sample area (3.6) of the test article, in square centimetres (cm^2);

V is the release solution volume after dilution, in millilitres (ml);

C_1 is the mean nickel concentration in the diluted release solution after 1 week, in micrograms per litre (µg/l);

C_2 is the mean value of the nickel concentration in the blank solutions after 1 week, in micrograms per litre (µg/l).

9.2 Interpretation of results

9.2.1 General

An inter-laboratory comparison of this test method was undertaken in 2008 according to ISO 5725 (all parts). Fifteen laboratories determined the nickel release from three nickel containing materials of a predefined dimension.

The performance characteristic $u_{t,r}$ arising from this trial defines the total relative uncertainty in the calculated nickel release (9.1). It is a combination of the relative reproducibilities of the surface area determination $s_{R,rel}$ (area), and the analytical determination of the nickel release $s_{R,rel}$ (Ni). The measurement uncertainty arising from this trial according to ISO 5725 (all parts) is 46 %.

For the calculation of the range for compliance assessment, the resulting expanded measurement uncertainty is used.

[A1]

9.2.2 Conformity assessment [A1]

9.2.2.1 General

It is an essential requirement that all laboratories interpret their results using the same procedure. The following rules for deciding upon compliance of articles with respect to their migration limits are to be applied.

This subclause is only applicable if the measurement uncertainty as taken from 9.2.1 is applied.

9.2.2.2 Articles with a migration limit of 0,5 µg/cm²/week

[A1] Because of the combined measurement uncertainty of 46 %, an article is non-compliant only when the nickel release is greater than or equal to 0,88 µg/cm^2/week. Hence an article shall be accepted and be permitted to be placed on the market if the measured value is less than 0,88 µg/cm^2/week.

NOTE For further explanation, see Annex A. [A1]

9.2.2.3 Articles with a migration limit of 0,2 µg/cm²/week

[A1] Because of the combined measurement uncertainty of 46 %, an article is non-compliant only when the nickel release is greater than or equal to 0,35 µg/cm^2/week. Hence an article shall be accepted and be permitted to be placed on the market if the measured value is less than 0,35 µg/cm^2/week.

NOTE For further explanation, see Annex A. ⟨A1⟩

9.2.3 Uncertainty budget

Laboratories may use for decision-making, estimates of $u_{t,r}$ smaller than the above mentioned value. In these cases it is required to detail the reasons and provide an uncertainty budget detailing the sources of uncertainty, accompanied by appropriate experimental evidence. This information shall be included in the test report.

10 Test report

The test report for each determination shall include at least the following information:

a) identification of the sample including source, date of receipt, description;

b) a reference to this standard, i.e. ⟨A1⟩ EN 1811:2011+A1:2015 ⟨A1⟩;

c) sample preparation;

d) description of the sample area including the size of the sample area expressed in square centimetres;

e) if relevant, specification of the use of wax or lacquer for protection of areas during immersion in the test solution;

f) the volume of test solution used;

g) for each replicate measurement, the nickel release value;

h) explanation of any use of a performance characteristic other than the value as determined by the interlaboratory trial (refer to Annex A);

i) the limit of quantification of the method;

j) a statement on compliance or non-compliance of the sample to its respective limiting value;

k) any unusual features observed during the determination;

l) starting and completion dates of test;

m) identification of laboratory carrying out the analysis;

n) signature of laboratory manager and operator.

Annex A
(informative)

A1 Expanded measurement uncertainty of the test procedure and compliance assessment

The results of an inter-laboratory comparison undertaken in 2008 according to ISO 5725 gave as an estimate for the combined expanded measurement uncertainty a value of $u_{t,r}$ = 46 %. The performance characteristic $u_{t,r}$ is related to the relative reproducibilities of the surface area determination $s^2_{R,r}(area)$ and the analytical determination of the nickel release $s^2_{R,r}(Ni)$ by the following formula:

$$u^2_{t,r} = s^2_{R,r}(area) + s^2_{R,r}(Ni) \quad \text{(A.1)}$$

In order to determine whether a tested article is non-compliant with its respective limit, a t-test is applied. This test decides whether a determined nickel release value significantly exceeds its limit. This is the case when the lower confidence interval boundary of the measured release exceeds the legally prescribed limit. This may be re-formulated in a way that a value significantly exceeds the limit if it is larger than the limit plus one half of the width of the confidence interval of the measured value. The confidence interval of the measured value is assessed as the expanded uncertainty of the value, i.e. the combined uncertainty times the coverage factor *k(α)*. The test is one-sided since the legal limit does not have an uncertainty. Experience has shown that the distributions of experimental data follow a log-normal rather than a normal distribution, in particular in the region of the lower legal limit. Thus, the estimate for the expanded uncertainty is added in a multiplicative way.

Taking into account the above, the decision limit for compliance is defined as

$$\overline{d}_{meas} \le d_{\lim} \cdot (1 + k(\alpha) \cdot u_{t,r}) \quad \text{(A.2)}$$

where

$k(\alpha)$ is the coverage factor for the chosen significance level (0,05) and the one sided t-test which gives a corresponding value of 1,65, assuming a large number of degrees of freedom for the combined uncertainty;

$d_{\lim}$ is the legal 0,2 µg/cm^2 /week or 0,5 µg/cm^2/week limit, respectively;

$\overline{d}_{meas}$ is the nickel release value (value *d* in Formula(1)). Note that $\overline{d}_{meas}$ is determined with a mean of replicate concentration measurements;

$u_{t,r}$ is the combined expanded measurement uncertainty.

NOTE 1 For guidelines to the evaluation of uncertainty in measurement it is advised to refer to ISO/IEC Guide 98-3: “Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM 1995)”, and more specifically for analytical measurements to the EURACHEM/CITAC Guide 2012 “Quantifying Uncertainty in Analytical Measurement“. Guidance on the relationship between analytical results, the expanded measurement uncertainty and limit values are given in the EURACHEM/CITAC Guide 2007 “Use of uncertainty information in compliance assessment”.

Using this decision criterion for articles that shall show compliance with the migration limit of 0,5 µg/cm^2/week, an article will be deemed to be non-compliant when the nickel release value *d* (9.1) is greater than or equal to 0,88 µg/cm^2/week. For articles that shall show compliance with the migration limit of 0,2 µg/cm^2/week, an

article will be deemed to be non-compliant when the nickel release value *d* (9.1) is greater than or equal to 0,35 µg/cm^2/week.

NOTE 2 For further information on articles made from composite materials refer to Annex D. A1

Annex B
(normative)

Requirements for quality control material

The quality control material has been shown to have a nickel release rate of (0,31 ± 0,06) µg/cm^2/week. This value is based on the accepted results (laboratory means) of 11 laboratories participating in an interlaboratory comparison ILC conducted in June 2008.

The relative method reproducibility demonstrated in this ILC was 33,3%.

In order to be compliant with the performance requirements of this standard, values determined for this quality control material by individual laboratories should be allocated within an interval of [0,19; 0,49] µg/cm^2/week, with the left value indicating the lower, and the right value the upper bound of acceptable laboratory results.

NOTE 1 [A1⟩ *deleted text* ⟨A1]. The variability of surface area determination for the rather uncomplicated shape of the quality control material is part of the reproducibility estimate given above.

NOTE 2 Information on sourcing of a suitable quality control sample is available from the CEN Management Centre.

The following alloy composition has shown to be suitable:

For precise manufacture of the quality control material a minimum mass of 1 kg has to be alloyed. Gold (99,99 %), copper (99,9 %), nickel (99,9 %) and zinc (99,9 %) are weighed to an accuracy of ± 0,1 g so as to achieve the following composition:

Table B.1

Element	Content % (*m/m*)
Au	76,0
Cu	16,0
Ni	6,0
Zn	2,0

Alternatively, it is possible to use a pre-alloy with the following composition:

Table B.2

Element	Content % (*m/m*)
Cu	66,7
Ni	25,0
Zn	8,3

and to weigh 24 % (*m/m*) of this pre-alloy together with 76 % (*m/m*) of gold.

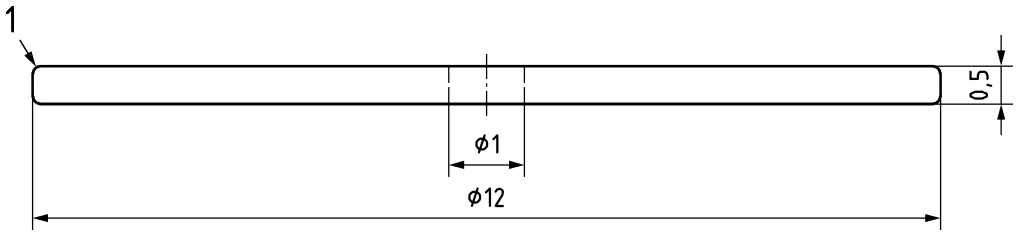
Tolerances in the final composition should not exceed ± 0,1 % of gold and ± 0,2 % of copper, nickel and zinc, respectively. The quality control alloy should have a Vickers Hardness of (190 ± 5) HV.

The quality control sample discs shall be made of the material specified above, and have the following dimensions:

— Diameter: (12,0 ± 1,0) mm;

— Thickness: (0,5 ± 0,1) mm;

— Centre hole diameter: (1,0 ± 0,2) mm.



Key

1 smooth edges

Figure B.1 — Dimensions of quality control sample

WARNING — All operations should be carried out so as to avoid contamination of the surface of the material with nickel.

Annex C
(normative)

Requirements for preparation of all post assemblies which are inserted into pierced parts of the human body and articles intended to come into direct and prolonged contact with the skin prior to nickel testing

C.1 General

This annex provides requirements to testing laboratories on the preparation of articles, prior to nickel release testing.

Further information on articles made from composite materials can be found in Annex D.

C.2 Requirements and principle

The article to be tested for nickel release is examined firstly to determine what method of nickel release testing is required followed by selecting the surfaces that are most likely to come into direct and prolonged contact with the skin or pierced parts of the body. The final stages involve the surface area determination of these surfaces and choosing appropriate apparatus for the article prior to nickel release testing.

C.3 Determination of the nickel release test method

Non-coated articles and articles with nickel containing outer coatings that come into direct and prolonged contact with the skin and all post assemblies which are inserted into pierced parts of the body shall be tested according to this standard.

Articles with non-nickel containing surface coatings that come into direct and prolonged contact with the skin shall be tested according to EN 12472, followed by this standard.

The method of testing and the respective tested surfaces and all other details pertaining to the preparation of the article prior to nickel release testing shall be included in the test report. X-Ray fluorescence spectrometry, microscopic techniques or other suitable methods should be used in deciding whether an article has a non-nickel containing coating.

C.4 Determination of surfaces coming into direct and prolonged contact with the skin or pierced parts of the body

C.4.1 Procedures for homogeneous and inhomogeneous articles

C.4.1.1 General

The basic requirement of this standard is that the release rate is representative of the article under test. Surfaces which are not in direct and prolonged contact with the skin or pierced parts of the body may be excluded from further consideration; all other surfaces shall be treated depending on whether the parts are homogeneous or inhomogeneous. This shall be determined by visual inspection.

While the total surface area is well understood, the terms "surface in direct and prolonged contact with the skin" and "surface in contact with pierced parts of the body" can lead to misinterpretations. Guidance is given in this clause.

C.4.1.2 Homogeneous articles and all post assemblies

For homogeneous articles the release from the complete surface is fully representative regardless of which part of the surface is in contact with the skin or pierced parts of the body. The total surface area shall be calculated and the release determined from the whole article since errors can be introduced by the masking process (see 7.1.3).

C.4.1.3 Procedure for inhomogeneous articles

C.4.1.3.1 General

For the case of inhomogeneous articles surfaces shall be representative. Therefore it is required to distinguish between parts which are not in direct and prolonged contact with the skin (under normal conditions of use), and parts which are in contact. The following situations can occur.

C.4.1.3.2 Situation 1

C.4.1.3.2.1 General

Only one type of material with one type of surface finish is in direct and prolonged contact with the skin.

C.4.1.3.2.2 Procedure 1

The article shall be dismantled and/or cut to obtain those homogeneous sub-parts which are in direct and prolonged contact with the skin.

In all cases where dismantling or cutting of inhomogeneous articles into homogeneous parts is not possible, masking shall be applied, and the release determined from the non-masked surface.

C.4.1.3.2.3 Result

Calculations are carried out with respect to the non-masked surface which shall resemble the most probable surfaces in direct and prolonged contact with the skin.

C.4.1.3.3 Situation 2

C.4.1.3.3.1 General

More than one type of material is in direct and prolonged contact with the skin, due to the construction of the article.

C.4.1.3.3.2 Procedure 2

The article shall be dismantled and/or cut to obtain those homogeneous sub-parts which are in direct and prolonged contact with the skin, then follow the procedure C.4.1.2 for each of the sub-parts. Every sub-part shall comply with this standard.

In all cases where dismantling or cutting of inhomogeneous articles into homogeneous parts is not possible, selective masking shall be applied, and the release from the non-masked surface of each of the different sub-parts assessed.

C.4.1.3.3.3 Result

Calculate the weighted-by-area-fraction mean release rate from all the different sub-parts which are in direct and prolonged contact with the skin. Every sub-part shall comply with this standard.

Calculations are carried out with respect to the non-masked surface which shall resemble the most probable surfaces in direct and prolonged contact with the skin.

C.4.1.3.4 Situation 3

C.4.1.3.4.1 General

Articles where it is not feasible to dismantle/cut and mask

C.4.1.3.4.2 Procedure 3

In all cases where neither situations 1 or 2 apply, and/or both dismantling/cutting and masking is not possible, follow C.4.1.2 and test the whole article. This procedure provides a release rate averaged over the different materials of the article. Since there is no other opportunity, this average release rate is the best estimate. This also applies to situations where parts which are not in direct and prolonged contact with the skin or pierced parts of the body cannot be excluded due to mechanical or chemical considerations.

C.4.2 Jewellery products

C.4.2.1 General

Jewellery articles shall be tested in component form.

Removal will be done in such a way as to minimize exposure of the substrate. Any substrate that is exposed shall be masked off. The testing laboratory, after subjective examination of the article, can decide that to disassemble component parts could result in unacceptable damage to the surface to be tested. In such cases the article shall be tested as a complete article. If this is the case, the test report shall include suitable reference to this fact.

If articles require cutting, they shall be hand cut using suitable tools in order to eliminate any adverse influence on the rate of nickel released from the expose edge. Laser cutting shall not be used as it has been shown to result in a heat-treated edge, which can markedly influence nickel release rates.

C.4.2.2 Post assemblies and associated parts

C.4.2.2.1 Parts coming into direct and prolonged contact with the skin and/or pierced parts of the body

Post assemblies (ear studs and body piercing articles) have close contact with the wound channel, the pierced parts of the body and the skin. For types of typically used post assemblies refer to Figures C.1 and C.2.

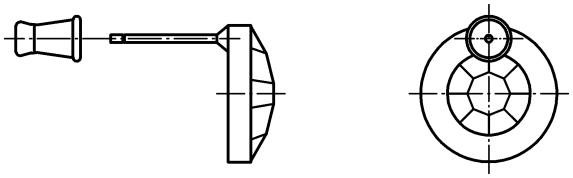


Figure C.1 — Example for post assembly with close body contact

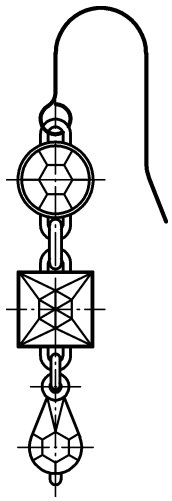
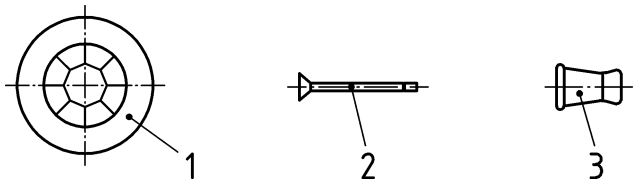


Figure C.2 — Example for fish hook post assembly with decorative element

A post assembly generally comprises of two to three main parts.



Key

1, 3 parts that hold the assembly in place against one or both sides of the post assembly (usually a scroll or clip for an earring and a ball for a body piercing article)

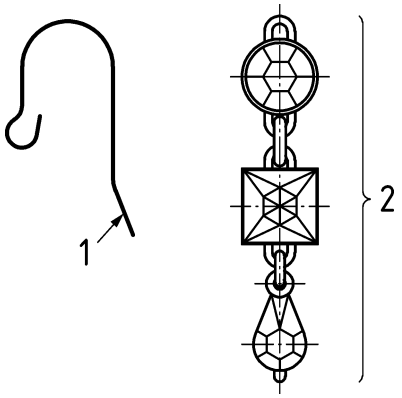
2 part that penetrates the pierced part of the body, usually a post or a stud

Figure C.3 — Parts comprising a post assembly

Parts that penetrate the body and the skin-facing surfaces of the parts that hold the assembly in place have direct and prolonged contact with pierced part of the body and the skin.

C.4.2.2.2 Decorative attachments of post assemblies

Any additional decorative element(s) attached to the post assembly shall be disassembled and tested in component form (refer to Figure C.4), provided that to do so would not cause unacceptable surface damage. In the case that after subjective examination of the article unacceptable damage would occur to the test surface, it is recommended to test such surfaces as composite sections. Any surfaces that constitute the post assembly shall be masked, as shall any surfaces exposed as a result of removing the post assembly by cutting.



Key

1 fish hook part that penetrates the pierced part of the body

2 decorative attachment

Figure C.4 — Disassembled fish hook post assembly with decorative attachment

C.4.2.3 Necklaces, bracelets, chains and anklets

Bolt ring and end rings are to be tested separately, if the end rings can be easily removed. If not, the end rings are to remain attached to the ends of the chain, and tested together with the bolt ring.

Lobster claw clasp and end rings are to be tested separately, if the end rings can be easily removed. If not, the end rings are to remain attached to the ends of the chain, and tested together with the lobster claw clasp.

Box fastener and end rings are to be tested separately, if the end rings can be easily removed. If not, the end rings are to remain attached to the ends of the chain, and tested together with the box fastener.

Pendant or other decorations are to be tested separately, where possible, depending on expected level of surface damage that can result from disassembly.

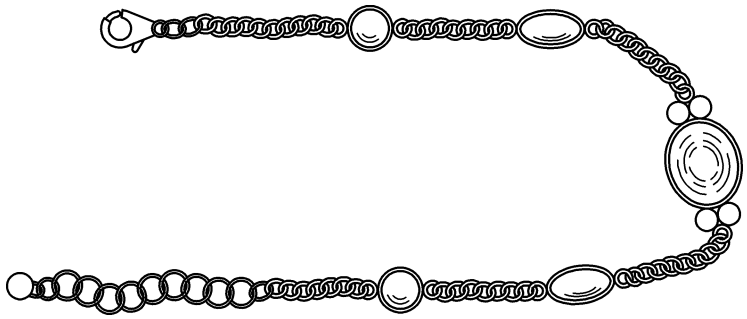
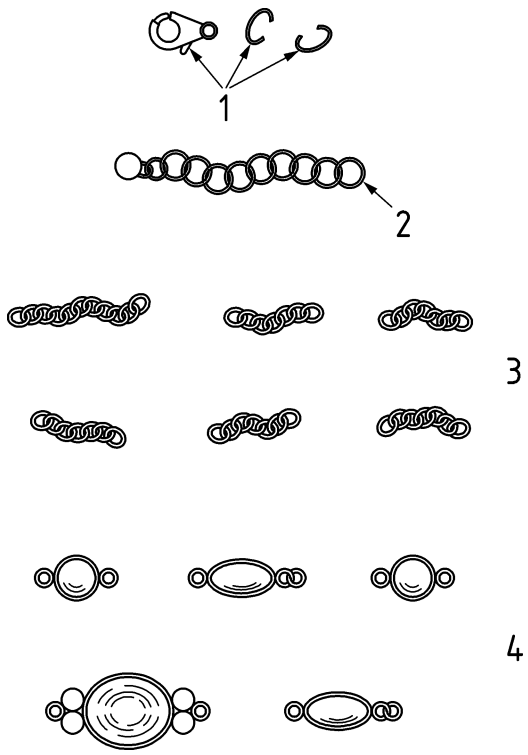


Figure C.5 — Example for chain before dismantling



Key

1 lobster claw

2 end rings

3 chain

4 pendant

Figure C.6 — Example for chain parts after dismantling

C.4.2.4 Bangles

The ‘tongue’ part of a box fastener shall be removed. The exposed surface shall be ‘masked off’.

C.4.2.5 Rings

No parts of the ring (refer to Figure C.7) shall be removed prior to testing.

No parts of the ring shall be ‘masked off’.

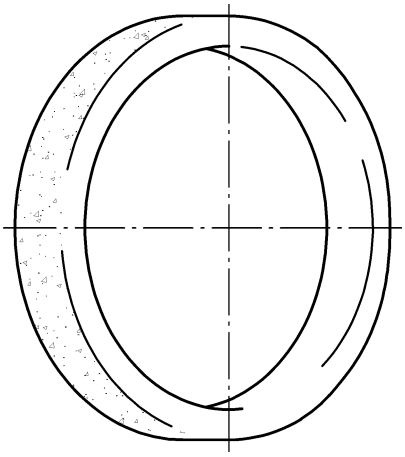


Figure C.7 — Ring example

C.4.2.6 Watches

C.4.2.6.1 General

Watches shall be tested in component form.

C.4.2.6.2 Parts to be tested

The following watch components (refer to Figure C.8) shall be tested:

1) watch case;

2) push-button(s) and/or crown (if they can be easily removed without causing undue damage); if they cannot, they shall remain in place and be tested along with the case);

NOTE Push-buttons are usually part of a digital watch, crowns are usually part of a mechanical watch. Some watches have both, buttons and crowns.

3) watch case back;

4) bracelet or buckle parts.

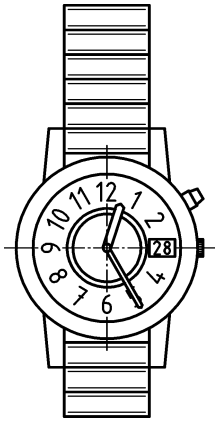


Figure C.8 — Example for watch

C.4.2.6.3 Parts to be removed from watch before testing

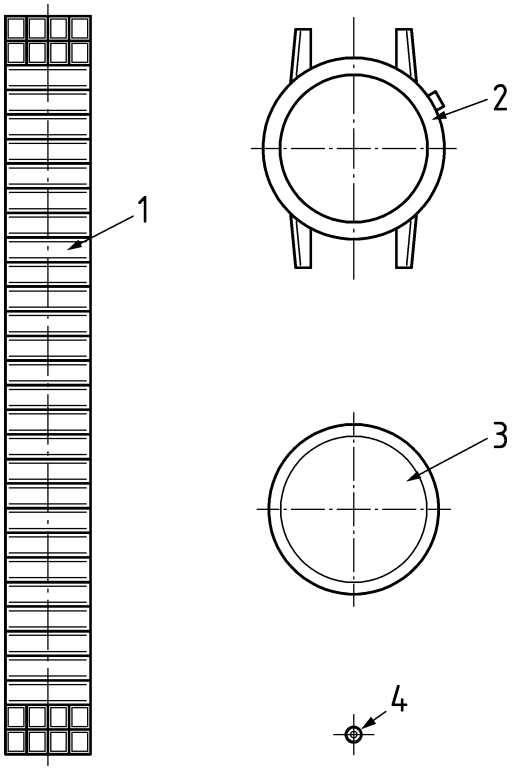
Watch parts which shall be removed and not tested (refer to Figure C.9) are the following:

— non-metallic parts;

— barrettes;

— internal workings, including battery, if available.

Watch glasses shall be removed only if possible without causing damage.



Key

1 bracelet

2 watch case

3 watch case back

4 push-button and/or crown

Figure C.9 — Example for watch after dismantling

C.4.3 Other articles such as textiles, footwear, garments, leather goods and mobile phones

Rivet buttons, tightener rivets, zip metalmarks, shoe buckles, belt buckles contained or intended to be used in garment, footwear or leather goods and parts of mobile phones or other articles can be composed of metallic materials that can release nickel.

These articles shall be tested in component form.

If the metallic article is composed of several metals, only the surface that is in contact with the skin shall be tested. Other surfaces shall be masked.

If the metallic article consists of one metallic material, the whole surface shall be tested.

The volume of the test solution (8.1) is given in Table C.1.

C.5 Methods of determining the surface areas

C.5.1 Surface area measurements

Each selected surface area shall be examined to determine its geometrical form and hence to decide upon the necessary mathematical formulae to calculate the surface area. The measurement of surface areas shall be undertaken where appropriate using a digital calliper or digital micrometer. For intricate surface areas a microscope profile projector or other microscopic techniques may assist in the surface area calculation.

Laboratories shall be aware that the resolution of their measuring device influences the accuracy of their surface area measurements, particularly for small articles.

When calculating the surface area, account should be taken of the elasticity of the skin and of the manner in which articles come into contact with the skin. Parts have an outer (non-contact) and inner (in-contact) surface such that the outer surface has to be masked (see 7.1.3). Doubts may arise as to whether part of the circumference area is in contact with the skin due to the "pillow" effect. A pragmatic approach is to leave the circumference unmasked, and account for the error committed in form of an additional uncertainty. An estimate for this additional uncertainty in absolute terms is one half of the circumference area, in relative terms the same value divided by the full unmasked area of the part under investigation.

The surface area of articles made essentially from sheet material, such as watch-cases, some medallions and lockets, can be assumed to be that area projected by all parts within 2 mm of the uncompressed skin-contact surface.

C.5.2 Minimum surface area

In order to achieve the required degree of analytical sensitivity, a minimum sample area of 0,2 cm² shall be tested. If necessary, identical articles may be treated together to obtain this minimum area.

C.5.3 Simplification of surface area determination using common shapes of consumer products

Wherever possible, common geometrical shapes of consumer products should be used for the calculation of the surface area. Examples for geometrical shapes are:

a) rectangular solids,

b) prisms,

c) cylinders,

d) cones and

e) spheres.

C.6 Testing apparatus prior to nickel release testing

When choosing a testing apparatus for an article it shall be chosen as far as practically possible to achieve a ratio of test solution (ml) to surface area (cm^2) close to 1. For articles where due to their size or shape it is not possible to achieve a 1 to 1 ratio, suggested ratios are given in Table C.1.

Table C.1 — Suggested ratio of test solution to surface area

Surface area cm^2	Ratio of test solution (ml) to surface area (cm^2)
0 to 5	1 to 1
5 to 10	10
10 to 25	25
25 to 50	50
> 50	100

For information concerning the testing apparatus, refer to 6.4.

NOTE For information about testing apparatus for coated articles, refer to EN 12472.

Annex D
(informative)

Articles made from composite materials

Where the sample area of an article is composed of homogeneous materials using the same surface finish, it can be assumed that the nickel release from the sample area of the article is the same as that of the homogeneous finish. However, there are instances in which the nickel release from composite material can exceed the value of 0,2 or 0,5 µg/cm^2/week. It is therefore incumbent on the manufacturer to be aware of the situations in which this can occur. These include:

a) the occurrence of bimetallic corrosion when a nickel-containing alloy is in electrochemical contact with a more noble metal/alloy in the sample area; examples are:

   1) contact of a stainless steel of low corrosion resistance, due to low chromium content or a high sulphur content, with a more noble metal/alloy such as gold, platinum or a higher alloyed stainless steel;

   2) brazing of a stainless steel with a silver-based alloy;

   3) contact of silver with a nickel underlayer depending upon the thickness of the silver top layer;

   4) contact with a nickel underlayer depending upon the thickness of the chromium top layer;

   5) plating of white gold alloys with rhodium or other precious metals depending upon the thickness of the precious metal coating;

   6) soldering of nickel alloys using phosphorus containing solders.

b) organic coatings;

c) protective or decorative organic coatings over nickel plated or nickel containing alloys depending upon the thickness of the organic coating;

d) surface condition, examples are:

   1) as a result of welding, brazing, soldering or other heat treatment; or the presence of nickel in the plating solution used to plate the surface layer; or damage to the surface in the course of assembly;

   2) any degreasing, grinding or polishing operation that modifies the surface of the article.

Where coated samples representative of the materials used in the production of finished articles are to be tested, they should be prepared at the same time as the articles that are to be placed on the market, using the same coating conditions, technique and solutions.

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