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BSI Standards Publication

Ophthalmic optics — Reference method for the testing of spectacle frames and sunglasses for nickel release

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National foreword

This British Standard is the UK implementation of EN 16128:2015. It supersedes BS EN 16128:2011 and PD CEN/TS 16677:2014 which are withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CH/172, Ophthalmic optics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Ophthalmic optics - Reference method for the testing of spectacle frames and sunglasses for nickel release

Optique ophtalmique - Méthode d'essai de référence relative à la libération du nickel par les montures de lunettes et les lunettes de soleil

Augenoptik - Referenzverfahren für die Bestimmung der Nickellässigkeit von Brillenfassungen und Sonnenbrillen

This European Standard was approved by CEN on 19 September 2015.

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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 16128:2015) has been prepared by Technical Committee CEN/TC 170 “Ophthalmic optics”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by May 2016, and conflicting national standards shall be withdrawn at the latest by November 2018.

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 supersedes EN 16128:2011 and CEN/TS 16677:2014.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports the harmonising effect of a restriction adopted under Regulation (EC) No 1907/2006 (REACH) of the European Parliament and the Council.

Compared to EN 16128:2011 and CEN/TS 16677:2014, the following changes have been made:

a) Compared to EN 16128:2011, the reference test method has been substantially revised:

   In the method according to EN 16128:2011 the parts to be tested for nickel release are placed in an artificial sweat test solution for one week. The concentration of dissolved nickel in the solution is determined by atomic absorption spectrometry, inductively-coupled plasma spectrometry or other appropriate analytical method.

   The present standard provides, for parts with an organic coating, a coating test based on Electrochemical Impedance Spectroscopy (EIS). The coating test aims at demonstrating that the coating is of sufficient quality to prevent the release of nickel, thereby ensuring that the test sample's nickel release does not exceed the regulatory limit.

   For parts without an organic coating, the present standard specifies a migration test. The migration test makes provision for quantitative testing for the amount of nickel released, to determine whether or not the model's nickel release exceeds the regulatory limit. The migration test comprises two steps: Release of nickel by artificial sweat solution into a test paper and the subsequent quantitative analytical detection of the nickel released into the paper.

   See also the principle described in Clause 4.

b) Compared to CEN/TS 16677:2014 the revisions and refinements made are relatively minor, as follows:

   For the coating test, see Clause 7:

   Amendment of the calculation and presentation of the test result including amendment of the threshold value (see 7.6);

   The dummy or test lenses used in the simulation of wear and corrosion are to be kept in the frame.

   For the migration test, see Clause 8:

Inclusion of the requirement to prepare and analyze a blank sample with every batch of test samples, along with the relevant specifications of sample preparation and procedure (see 8.4.4);

Specification that the incubation shall be made using a climate chamber; the previously permissible alternative to use an oven with a container for insertion of the test samples has been deleted (see 8.4.5);

Inclusion of more detailed specifications as to the permissible and non-permissible combination of the test papers from the various test areas for the analysis;

Inclusion of directions on how to proceed in the case that the design of a model does not allow the application of the test paper at (one of) the specified location(s);

Amendment of the procedure for the application and sealing of the test paper onto the test area using the sealing film; as an alternative to wrapping with the sealing film it is now also permissible to use a folding technique; see the revised Annex B;

Recommendation that the time between the retrieval of the test papers from the test samples and their extraction and analysis does not exceed 3 d (see 8.4.6).

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

This document has been prepared under Mandate M/448 issued by the European Commission in the framework of Regulation (EC) No 1907/2006, REACH, in particular Commission Regulation (EC) No 552/2009 of 22 June 2009 amending regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorization and restriction of Chemicals (REACH) as regards Annex XVII RESTRICTIONS ON THE MANUFACTURE, PLACING ON THE MARKET AND USE OF CERTAIN DANGEROUS SUBSTANCES, PREPARATIONS AND ARTICLES.

The aim of the mandate is the revision of the method of analysis to detect the release of nickel from spectacle frames and sunglasses.

The availability of the new reference method for the determination of the release of nickel will provide the reliable framework to enforce the limit value for nickel release of 0,5 $\mu g/cm^2$/week set forth by European Regulation. It will ensure a uniform application and control of the European legislation in all member states.

Harmonizing the test method for nickel release in all member states is vital with a view to protecting effectively the health of the end consumer, that is, the spectacle wearer. Nickel allergy is still the most frequent contact allergy in Europe and a significant health issue.

1 Scope

This European Standard specifies the reference method for the testing of spectacle frames, ready-to-wear spectacles, sunglasses and other items for eye and face protection for nickel release.

The reference method supports the demonstration of conformity with the limit value for nickel release of 0,5 µg/cm²/week set forth by European Regulation.

The reference method involves the procedural steps shown in Figure 1 and described in Clause 4.

This document applies to those parts of metal spectacle frames and those metal parts of combination spectacle frames that are intended to come into direct and prolonged contact with the skin of the wearer. This document also applies to those relevant metal parts of ready-to-wear spectacles, sunglasses and other items for eye and face protection.

NOTE The reference method for articles apart from spectacle frames, ready-to-wear spectacles, sunglasses and other items for eye and face protection is specified in EN 1811.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

EN ISO 11380, *Optics and optical instruments — Ophthalmic optics — Formers (ISO 11380)*

3 Terms and definitions

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

3.1
model
spectacle frame, ready-to-wear spectacles, sunglass or other item used for eye and face protection produced to a common design, using the same materials and surface treatment, and to which the scope of this document applies

3.2
test sample
spectacle frame, ready-to-wear spectacles, sunglass or other item used for eye and face protection submitted for testing

Note 1 to entry: Fronts or sides may be submitted separately for testing.

3.3
test part
part of a test sample that is intended to come into direct and prolonged contact with the skin and is due to be tested

Note 1 to entry: These parts are defined in 7.3.1 (for the coating test) and in 8.3.1 (for the migration test).

3.4
test paper
piece of laboratory cellulose paper used for testing, at any stage of the procedure after being cut to size for testing

3.5
extraction solution
solution obtained after extraction of nickel ions from the test paper

3.6
appropriate tool
tool enabling the procedure to be performed without causing contamination by nickel or other metal ions, either from the material of the tool or deposits on it

Note 1 to entry: Such tools could be made from plastics, titanium, or stainless steels.

3.7
appropriate equipment
equipment enabling the procedure to be performed without causing contamination by nickel or other metal ions, either from the material of the equipment or deposits on it

4 Principle

Following the simulation of wear and corrosion according to the method specified in EN 12472 (see Clause 6), the reference method comprises the following procedural steps:

1) Coating test, applicable only to test parts with an organic coating, based on Electrochemical Impedance Spectroscopy (EIS) and specified in Clause 7; the coating test aims at demonstrating that the coating of the test sample is of sufficient quality to prevent the release of nickel, thereby ensuring that the test sample's nickel release does not exceed the regulatory limit. The coating test is, however, not sensitive only to nickel ions, so a model can pass the migration test even though it failed the coating test.

2) Migration test for nickel ion release, specified in Clause 8; the migration test makes provision for quantitative testing for the amount of nickel released, to determine whether or not the model's nickel release exceeds the regulatory limit. The migration test comprises two steps: Release of nickel by artificial sweat solution into a test paper and the subsequent quantitative analytical detection of the nickel released into the paper.

For a model that failed the coating test, either new test samples or, subject to the requirements of the person ordering the test, the original test samples may be subjected to the migration test.

Metal frames that are uncoated, i.e. neither organic coating nor metal plating, and made of homogeneous alloys or metals do not require the simulation of wear and corrosion specified in Clause 6 and shall be tested directly in accordance with Clause 8. Unless the manufacturer certifies that a component is homogeneous and uncoated, the component shall be assumed to be coated.

Figure 1 illustrates the procedure.

Requirements for sampling and guidance as to which parts of the test samples shall be subject to testing (the test parts) are given in Clause 5 and in 7.3.1 (for the coating test) and 8.3.1 (for the migration test).

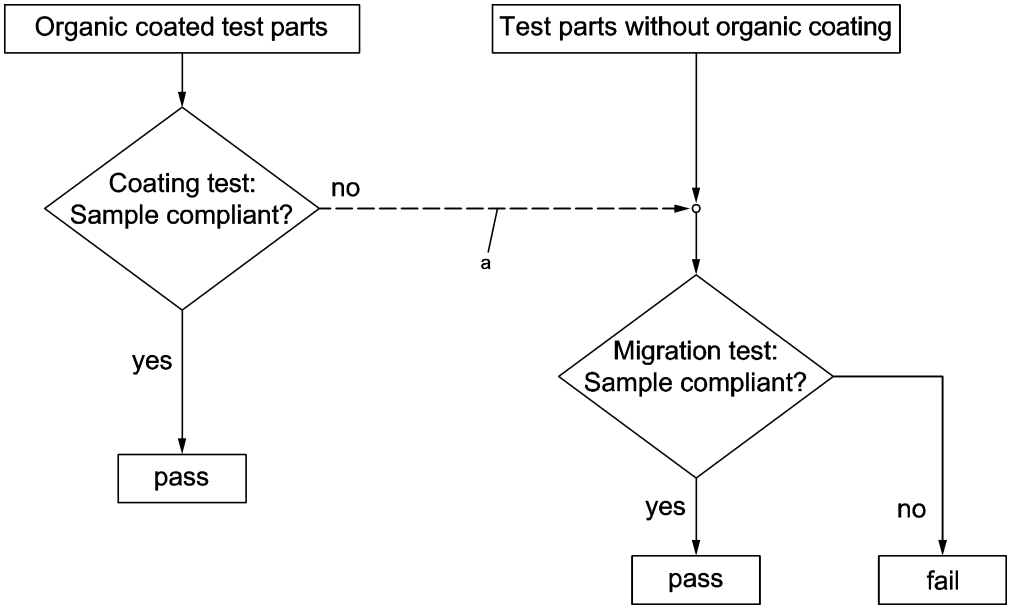
5 Selection of test samples

Two specimens of each model to be tested shall be selected at random for either the coating or the migration test.

The selected specimens shall be identified.

Test samples used for the coating test may be tested in a subsequent migration test, but not vice versa.

If a test sample is likely to be subjected to the migration test after the coating test, it shall be washed thoroughly in deionized water immediately after the coating test and allowed to dry to avoid corrosion from sodium chloride. It is preferable that new test samples are used for the migration test.



Key

[a] The coating test is not sensitive only to nickel ions, so a model can pass the migration test even though it failed the coating test.

Figure 1 — Diagrammatic overview of the reference test method

6 Simulation of wear and corrosion

6.1 Preparation of test samples

For spectacle frames, if not already fitted with dummy or demonstration lenses, the test samples shall be fitted with a pair of suitable organic lenses within the range of −1,00 D to +1,00 D and with an edge thickness of between 1,5 mm and 2,5 mm. These test lenses shall be edged either in accordance with the manufacturer's electronic instructions or with a digitally controlled edging machine that uses the tracing made of the individual test sample or, where appropriate, using a mechanical former in accordance with EN ISO 11380. The bevel angle of the edged lens shall be 120° −2° +3° for spectacle frames featuring a rim with a groove.

For all test samples, sides and fronts shall be separated from each other. Removing end covers (side tips) from sides is optional. Unless they have a metal-bearing surface, nose pads shall be removed before the wear phase. Sides shall be dismantled from fronts, either by unscrewing the dowel (hinge) screw or by cutting the joint across the charniers.

WARNING — Care shall be taken not to damage the coating on areas that are subsequently tested, particularly the coating near any cut.

Ensure that all the separate test parts remain identified throughout all steps of the overall procedure.

6.2 Procedure

Perform the simulation of wear and corrosion according to EN 12472.

When the simulation is completed, remove the test samples. Gently swirl them for 2 min in degreasing solution (see 7.2.4) at room temperature. Rinse thoroughly with deionized water. Gently dry in a clean air stream or allow to dry on absorbent paper.

After degreasing, handle the test samples with appropriate tools or clean laboratory gloves.

Disassemble three-piece rimless fronts. Disassemble fronts of combination frames and remove any plastics parts.

Then subject the test parts to the selected test: coating test, see Clause 7, and/or migration test, see Clause 8.

7 Coating test

7.1 General

The purpose of the coating test is to verify if the surface treatment of a model is able to limit the release of metal ions (hence including nickel), in order to identify good quality coatings. Test samples that are not identified as “pass” may be subjected to the migration test, see Clauses 4 and 5.

The parts of spectacle frames needing consideration are only those intended to come into direct and prolonged contact with the skin of the wearer, see 7.3.1.

7.2 Apparatus and consumables

7.2.1 Masking agent, suitable for electroplating purposes and capable of electrically insulating the test part from the saline solution. Application of more than one coat is acceptable, and may be preferable.

Test the masking agent to verify that it is suitable by using it to coat a metal strip without an organic coating and show that the adhesion is good and impedance results are high (greater than $5{,}0 \cdot 10^6\ \Omega \cdot \mathrm{cm}^2$).

NOTE 1 A metal strip with approximate dimensions of 2 mm thick, 6 mm wide, and 100 mm long is suitable.

It is useful if the masking agent is coloured or fluorescent, to make the masked areas more visible.

NOTE 2 Lacomit is the trade name of a suitable product.[1)]

7.2.2 Deionized water, according to EN ISO 3696, grade 3 or to European Pharmacopaeia, for rinsing and preparation of the saline solution (7.4).

7.2.3 Sodium chloride of recognized pro analysis, p.a., grade or better, for preparation of the saline solution (7.4).

7.2.4 Degreasing solution, Sodium Dodecyl Sulfate (SDS) at a concentration of 0,5 % in deionized water, to clean the test samples after cutting, etc. and before testing. An appropriately diluted, neutral, commercially available detergent may also be used.

7.2.5 Apparatus for preparation of 1 % saline solution in deionized water.

1) Lacomit is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

7.2.6 Electro-chemical cell, made of glass, suitable for mounting firstly a Standard Calomel (SCE) or Ag/AgCl Reference Electrode, secondly a Graphite or Platinum Counter Electrode and thirdly, the test part.

NOTE The recommended counter electrode is a high density pure graphite rod, approximately 6 mm in diameter.

7.2.7 Laboratory clamps, suitable for holding the test part, with the selected area immersed in the electrolyte in the electro-chemical cell, but with the electrical contact area kept dry.

7.2.8 Potentiostat, having the ability to perform A.C. Electrochemical Impedance Spectroscopy (EIS) at 1 Hz and having a current sensitivity of better than 1 pA.

NOTE An example of suitable apparatus is Gamry apparatus series 600 with EIS 300 software package.[2)]

7.2.9 Electrical clamps, e.g. small crocodile clips, capable of providing secure electrical contact with the test part, reference and counter electrodes.

7.2.10 Calibration (dummy) cell, usually supplied by the potentiostat manufacturer together with the instrument, appropriate to test the potentiostat in the impedance range expected.

7.2.11 Appropriate tools, needed to perform the procedure. See 3.6 for the definition of “appropriate tools". To prevent possible contamination by nickel or other metal ions, clean all tools well before use.

7.2.12 Laboratory gloves, e.g. latex or PVC, but not cotton.

7.2.13 Faraday cage; either an earthed Faraday cage, earthed aluminium foil or earthed conductive cloth to house the electro-chemical cell during measurement.

7.3 Preparation of test samples for the coating test

7.3.1 Parts to be tested

See Clause 5 for sampling requirements.

For testing of models with the coating test, the parts to be tested are:

a) the front, comprising the rims, the bridge and the brace bar (if applicable) but excluding the pad arms and pad boxes, lugs and, if temporal, closing block joints;

b) sides (temples), including metal collets, but excluding the joints, a zone (ideally 10 mm) around the joints, and areas intended to be protected by plastic end covers (tips).

For each of the two test samples, the front and the two sides shall be tested separately; all three parts of both test samples shall pass in order for the model to pass.

7.3.2 Dismantling and/or cutting and/or masking

7.3.2.1 General

Prior to submission to the coating test, the test sample shall have been subject to the method for simulation of wear and corrosion according to EN 12472. See Clause 6.

2) Gamry apparatus series 600 with EIS 300 software package is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product.

After completion of the simulation of wear and corrosion, select and separate those parts (or areas) which are subject to the coating test from those that are not. This can be achieved by one or more of the following:

a) dismantling (see 7.3.2.2);

b) cutting (see 7.3.2.3 and Annex A);

c) masking (see 7.3.2.4 and Annex A).

Consider the need for preparing the electrical contact area (see 7.3.4) and for determination of surface area (see 7.3.3) when deciding where to cut or which areas to mask. Masking any "complicated" details of the test sample which would not come into direct and prolonged contact with the skin could simplify the determination of surface area and/or enhance its precision.

Cutting of the test samples except across the charniers (see 6.1), to separate the parts (or areas) to be tested from those not to be tested, is permissible, but should be avoided if at all possible.

NOTE Cutting can be avoided by masking all the test sample except those parts (or areas) to be subjected to the test.

CAUTION — If cutting is undertaken, great care shall be taken to avoid contamination by metal ions e.g.

- **from the base material;**
- **from metal particles getting underneath the masking;**
- **by damaging the organic coating that is near the cut and subsequently tested;**
- **from metal on tools or fingers or the work area.**

Ensure that all the separate parts of the test samples remain identified, e.g. assign identification numbers or codes, while not physically touching or modifying them.

7.3.2.2 Dismantling

The dummy or test lenses needed for the simulation of wear and corrosion (see 6.1) shall be left in the front. If not already removed for the simulation of wear and corrosion according to EN 12472, however, dismantle nose pads from fronts.

7.3.2.3 Cutting

Cut the frame by hand-sawing or using cutting pliers.

Cut edges and any other areas of the parts to be tested that are not covered by an organic coating, other than those intended for use as electrical contact areas (see 7.3.4), will require masking. See 7.3.2.4 for requirements regarding masking.

Ensure that cut edges are rounded in order to help get a good uniform coating of masking agent.

7.3.2.4 Masking

Gently swirl the test sample(s) for 2 min in degreasing solution (7.2.4) at room temperature. Rinse thoroughly and carefully with deionized water (7.2.2) and gently dry in a clean air stream or allow to dry on absorbent paper.

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

After degreasing, handle the test samples with appropriate tools or clean laboratory gloves.

Masking can be done by dipping or painting/brushing. Whichever method is used, ensure that the resulting masking film is much thicker than the original coating of the test sample or the part thereof. Ensure that cut edges are covered. Dip or paint/brush twice, allowing approximately 30 min between the applications. After allowing the masking agent to dry for a minimum of 15 min, inspect the quality of masking visually with a low-powered (e.g. 2x to 5x) magnifier to ensure complete coverage of the appropriate areas. If required, apply an additional layer(s) of masking agent. See Annex A for details.

Pad arms and pad boxes, and, if temporal, closing blocks including their screws shall be masked, if intended to be immersed.

The grooves of rims do not need masking, but they may be masked in areas adjacent to pad arms and lugs during the masking process.

Other parts to be masked may depend upon the choice of electrical contact area and whether or not the test sample is cut.

Allow the masking agent to cure completely. In the case of doubt, follow the masking agent manufacturer's instructions. When allowing the masking agent to dry and cure, support the test sample so that the masked areas are not in contact with a surface.

If the test parts will not be tested immediately after the masking agent has dried and cured, then protect them from potential damage by wrapping with paper tissue or placing individually in a polythene bag.

7.3.3 Determination of test area

The measured value with the coating test method is always in impedance per area, expressed in units of $\Omega \cdot cm^2$. Therefore, the method involves a need for determination of the test area, which is the immersed but unmasked surface area of the test sample.

Determine the test area to the nearest cm^2. Direct measurement of surface area may be used; CAD data from the frame manufacturer and/or classical picture analyser software may be helpful.

7.3.4 Preparation of electrical contact area

The following serve as the electrical contact areas:

— for sides: either the end furthest from the joint, or the joint;

— for fronts: the joint end of the lug that is not masked.

Abrade the electrical contact area e.g. with a hand-held rotary model-making tool, file, or emery or silicon carbide paper, so that the organic coating is completely removed. Take care not to damage the coating outside the contact area during this process.

NOTE Wear suitable protective equipment, if appropriate.

7.4 Preparation of saline solution

Prepare a solution of saline by dissolving 10 g ± 0,1 g of Sodium Chloride (7.2.3) in 1 l of deionized water (7.2.2). Ensure that the saline solution is at 25 °C ± 5 °C. Prepare a fresh saline solution after 7 d.

7.5 Procedure

7.5.1 Preparation of the electro-chemical cell

Wash the electro-chemical cell with deionized water (7.2.2) or with saline solution (7.4).

Fill the electro-chemical cell with saline solution (7.4). Change the solution at least once on each day of testing.

Ensure that the reference and counter electrodes (e.g. calomel) have been immersed for a sufficient time before use. If stored in a dry state they will need to be (if appropriate, filled with potassium chloride and) immersed in deionized water or saline solution for a minimum of 8 h. The equipment therefore cannot be used immediately after assembly.

7.5.2 Insertion and connection of the test part in the electro-chemical cell

IMPORTANT — The technique requires a very good electrical connection between the electrodes, including the test part, and the potentiostat, and a Faraday cage or other method to shield the electro-chemical cell from stray electromagnetic radiation.

Immediately before the EIS measurement, verify that the electrical contact area on the test part is clean and free from masking agent, and is not tarnished. If necessary, apply further abrasion to achieve this, while taking care not to damage the coating in the test area.

Suspend the test part in the saline solution so that it is not resting on the bottom and is at a minimum distance of 10 mm from the sides of the cell, ensuring that the electrical contact area is above the level of the saline solution. Ensure that any area of the test part that does not require testing is either masked or is above the level of the saline solution, while the counter and reference electrodes are appropriately immersed according to the equipment manufacturer's instructions. Because the distance between the test parts and the electrodes may affect the resistance in the solution, the test part, counter and reference electrodes shall be arranged in a triangular arrangement with a separation of approximately 50 mm between each.

NOTE 1 A convenient way to do this is for the counter and reference electrodes to be mounted at the 50 mm separation on a piece of insulating material resting on the rim of the electro-chemical cell so that they remain permanently in the saline solution while testing. A second piece of insulating material can then be used to mount the test part at the appropriate distance.

Ensure that the electrical connections on the abraded contact area of the test part and the electrodes are tight.

NOTE 2 One method is to use a screwed terminal block to clamp firmly on the test part, with a braided copper flex in the other end of the terminal block to make good contact with the crocodile clip on the potentiostat lead.

If a lid is used, bring the electrode leads through the lid to enable connection to the potentiostat (7.2.8).

Immerse the test part in the saline solution in the electrochemical cell for 3 min before measuring the EIS response. Use either a Faraday cage or wrap the electrochemical cell in a conducting material, e.g. earthed aluminium foil or conductive cloth.

7.5.3 Determination of open circuit potential and measurement of electrochemical impedance of test samples

After immersing the test part in the saline solution as specified in 7.5.2, set the instrument to measure the impedance of the test part at the fixed frequency of 1 Hz and with the amplitude of the applied signal at 10 mV rms. Most instruments will automatically set the DC potential at the Open Circuit Potential (OCP) found at the beginning of the measurement. Take the first measurement of impedance as soon as possible after the test part has been immersed in the saline solution for 3 min, and take the two subsequent measurements in as close a succession as possible. Carry out the measurement at room temperature.

If the instrument does not automatically set the DC potential to the OCP of the test part, this will have to be done manually.

Verify that the ratio:

$$\frac{(\text{highest impedance reading} - \text{lowest impedance reading})}{\text{lowest impedance reading}}$$

for the individual test part is no greater than 0,3 (30 %); if it is, remove the test part, wash with deionized water, allow to dry and wait at least 24 h. Then re-measure the test part, taking care with the electrical connections and electromagnetic shielding. If the ratio on re-testing is again greater than 0,3 (30 %), a new test sample shall be tested.

NOTE A different immersion time might change the test part's electrical properties.

7.5.4 Calibration and verification of the equipment

Verify the calibration of the potentiostat daily by running the test on the calibration (dummy) cell (7.2.10). Perform any other checks, e.g. on the electrodes, according to the instrument's manufacturer's instructions.

7.6 Calculation of results

7.6.1 General

Calculate the average of the three values obtained and report this value as the impedance (|Z|) at 1 Hz in $\Omega \cdot cm^2$, in scientific notation to two significant figures.

Some models of potentiostat allow the operator to enter the surface area of the sample into the software before starting the measurement. This type of equipment is likely to report the impedance in $\Omega \cdot cm^2$. If the equipment reports the impedance in Ω, the value in $\Omega \cdot cm^2$ is obtained by multiplying the impedance in Ω by the immersed surface area in cm^2. In the latter case, multiplication is necessary because this is effectively a calculation for resistances in parallel, not series.

7.6.2 Criteria for pass or fail of the test sample

Classify the test part as pass or fail. A pass is considered to be an impedance greater or equal to the threshold limit of $3{,}0 \cdot 10^5\ \Omega \cdot cm^2$, while a fail is considered to be an impedance lower than the threshold limit. For each of the two test samples, each of the two sides and the front shall be tested separately, and each test part shall pass in order for the model to pass.

NOTE 1 This threshold limit was established by round robin tests, in which the impedance was compared with the nickel release. The threshold limit has been chosen both to allow a margin of safety and to include an allowance for uncertainty of measurement.

NOTE 2 A pass result indicates compliance with the migration limits specified in paragraph 1(b) and (c) of entry 27 of Annex XVII to the REACH regulation. Because it is a coating test, Electrochemical Impedance Spectroscopy gives a result that includes all metal ions, not just nickel ions. Therefore, a failed model could demonstrate conformity with the regulatory limit by submission to the migration test.

7.7 Test report

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

a) identification of the model or the test sample(s), respectively, including source, date of receipt, description, and, optionally, the assigned identification number(s) or code(s);

b) a reference to this document, i.e. EN 16128:2015;

c) documentation (verbal description and, optionally, photographs) of the parts of the test sample(s) and their preparation, including whether or not the test samples were subjected to the simulation of wear and corrosion according to EN 12472, and including the size of the test areas, expressed in cm^2;

d) test result and a statement on compliance or non-compliance of the model to the threshold value;

e) any unusual features observed during the determination;

f) starting and completion dates of test;

g) the date the instrument was last calibrated;

h) identification of laboratory carrying out the test;

i) signature of person responsible for the validation of the test report.

EXAMPLE The numerical values of the test result could be presented in the form of the table below:

Test sample	**Tested part**	**Identification number or code**	**Test area** [cm^2]	**Impedance per unit area** [$\Omega \cdot cm^2$]	**Result**[a] (pass or fail)
First test sample	right side				
	left side				
	front				
Second test sample	right side				
	left side				
	front				
Overall test result[b] **(pass or fail):**					

a Defined by whether the impedance per unit area is greater than or equal to, or less than the threshold given in 7.6.

b Defined by: Pass – all test parts pass, Fail – any (or all) test part(s) fails.

8 Release of nickel and its quantitative analytical detection (migration test)

8.1 General

The analysis for nickel released from a test sample into a test paper is based on extraction followed by ICP-OES [3], or ICP-MS [4] or GFAAS [5].

The parts of models needing consideration are only those intended to come into direct and prolonged contact with the skin of the wearer, see 8.3.1.

Test papers impregnated with a known amount of nickel are used as control samples to monitor the extraction and the analysis procedure.

A blank sample shall be tested with every batch of test samples. The blank sample shall consist of a piece of test paper impregnated with artificial sweat solution, which would then be extracted and analysed for nickel.

8.2 Apparatus and consumables

8.2.1 Laboratory cellulose paper, of highly-absorbing type, with one side covered with polyethylene (PE), capable of being cut into fine strips of width 0,15 cm without fraying, and for which the specific weight (grammage) can be measured within an uncertainty of ± 0,5 %.

3) ICP-OES: inductively-coupled plasma optical emission spectrometer.

4) ICP-MS: inductively-coupled plasma mass spectrometer.

5) GFAAS: graphite furnace atomic absorption spectrometer.

NOTE A suitable product is LabSorb Ultra from Sartorius-Stedim Biotech. [6)]

8.2.2 Appropriate tools including

— **cutting device**, being sufficiently sharp to cut the laboratory cellulose paper without tearing or fraying, e.g. a craft knife with replaceable blades or ones that can be snapped off to leave a new cutting edge

— **straight edge**

— **ruler** or equivalent that can be read, with interpolation, to 0,25 mm or smaller

— **scissors**, and

— **tweezers**.

See 3.6 for the definition of "appropriate tools".

To prevent possible contamination by nickel, clean all tools well before use.

8.2.3 Laboratory balance, that is accurate to at least 0,1 mg.

8.2.4 Appropriate equipment, such as vessels and containers needed to perform the procedure.

See 3.7 for the definition of "appropriate equipment".

If using reusable equipment, clean thoroughly before use.

8.2.5 Degreasing solution, to clean the test samples before testing. Dissolve 5 g of an anionic surface-active agent such as sodium dodecylbenzene sulfate or sodium alkylaryl sulfate in 1 000 ml deionized water (8.2.6). An appropriately diluted, neutral, commercially available detergent may be used.

8.2.6 Deionized water, of EN ISO 3696, grade 2 or better for rinsing.

8.2.7 Reagents and apparatus for preparation of artificial sweat solution, as specified in 8.4.2.2 and 8.4.2.3.

8.2.8 Nitric acid, ρ = 1,40 g/ml, 65 % (mass fraction).

8.2.9 Dilute nitric acid, approximately 2 % (mass fraction). Transfer 15 ml of nitric acid (8.2.8), into a 500-ml beaker containing about 350 ml of deionized water (8.2.6). Stir and cool to room temperature. Transfer the solution to a 500-ml volumetric flask and make up to volume with deionized water.

8.2.10 Pasteur pipette or micropipette, for saturating the test paper with artificial sweat solution.

8.2.11 Absorbing paper, to put underneath the test paper when saturating with artificial sweat solution.

8.2.12 Stretchable sealing film, of laboratory quality, 5 cm wide and capable of being stretched easily in one direction only.

6) LabSorb Ultra from Sartorius-Stedim Biotech is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

NOTE A suitable product is Stretchable Parafilm ® M Sealing Film.[7)]

8.2.13 Climate chamber for incubation of the test samples, capable of maintaining a temperature of 30 °C ± 2 °C and relative humidity of at least 95 % and equipped with racks or shelves to support the test samples. The racks or shelves may be designed to hold several test samples stacked side by side, horizontally to avoid the risk of condensation running down the test part and under the sealing film, and without contact with each other to avoid cross-contamination.

8.2.14 Ultrasonic bath to perform the extraction, fitted with supports for the extraction flask(s).

8.2.15 Laboratory gloves, e.g. latex or PVC, but not cotton.

8.3 Preparation of test samples for the migration test

8.3.1 Parts to be tested

See Clause 5 for sampling requirements.

The parts to be tested shall include:

— the outer surface of rims;

— the rear surface of the bridge except when a non-metallic insert bridge has been fitted, the rear surface of any brace bar and any other nasal bearing surfaces, including metal nose pads;

— sides, including metal collets, but excluding the joints and a zone (ideally 10 mm) around the joints, and areas intended to be protected by plastic end covers (tips);

— metal decorative trims, if fitted, on the inside of plastic sides and plastic end covers.

For each of the two test samples, the above parts shall be tested in the combinations shown in Table 1, and each combination of parts shall pass in order for the model to pass.

Table 1 — Combinations of the test parts in the migration test

	First test sample	**Second test sample**
Front	Right and left rim, together	Right and left rim, together
Bridge	Bridges of the two test samples together	
Side	Right and left side, together	Right and left side, together
Trims	Trims of the two test samples together	
The results of each of these combinations, as applicable, shall pass for the model to pass.		

8.3.2 Guidance on selection of test areas on the parts to be tested

The shape and size of the test paper or pieces of paper shall be chosen so that the paper best simulates the area intended to come into direct and prolonged contact with the skin.

If the construction does not allow the appropriate application of the test paper (e.g. fronts with a sheet front to which the eye rim is mounted behind) then it is allowed to place the test paper on another comparable position (i.e. front side of the sheet front in this example), if there is not sufficient area on the lower rim(s).

7) Stretchable Parafilm ® M is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

IMPORTANT — The minimum size of the test paper and the maximum volume of extraction solution will be governed by the detection limit of the analytical equipment for nickel determination.

See normative Annex B for sketches of frames and areas typically needing testing, the shape of the test paper and its application and wrapping with sealing film.

8.3.3 Dismantling and degreasing

Prior to submission to the migration test, the test sample shall have been subject to the method for simulation of wear and corrosion according to EN 12472. See Clause 6.

If not already removed for the simulation of wear and corrosion, optionally dismantle sides and nose pads from fronts. Remove dummy or test lenses from fronts.

Gently swirl the test samples for 2 min in degreasing solution (8.2.5). Rinse thoroughly with deionized water (8.2.6). Gently dry in a clean air stream or allow to dry on absorbent paper.

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

Ensure that all the separate parts of the test samples are labelled with their assigned identification number or code.

IMPORTANT — Starting from this point in the procedure, always handle samples with clean laboratory gloves (8.2.15) and use appropriate tools (3.6) to avoid contamination.

8.4 Procedure

8.4.1 Preparation of test paper including determination of its area

8.4.1.1 Make sure that performing the steps specified in 8.4.1.2 to 8.4.1.4 will not contaminate the test paper with nickel or other impurities. Wear laboratory gloves (8.2.15) for these steps.

8.4.1.2 Determine the specific weight (grammage) of the laboratory cellulose paper (8.2.1) by cutting a square sample 5 cm x 5 cm from the sheet, and measure its dimensions to 0,25 mm precision using a ruler or equivalent (8.2.2). Weigh the square sample precisely by use of the laboratory balance (8.2.3), and calculate the specific weight (mg per cm^2). Retain this test square in the same packet as the remaining laboratory cellulose paper for future use.

The determination of specific weight (grammage) using the square sample shall be done each time immediately before a batch of paper strips are (cut and) weighed from that sheet, because the grammage is dependent on the relative humidity at the time of cutting and weighing.

NOTE This is based on methods given in EN ISO 536.

To cut a square of paper in order to determine its grammage, it is recommended to draw a square 5 cm x 5 cm on a piece of paper using drawing instruments and a black pen. This can then be placed under the laboratory cellulose paper to act as a guide when cutting.

8.4.1.3 After determining the specific weight, cut the laboratory cellulose paper (8.2.1) into strips, using a zone near to where the square sample was cut. The laboratory cellulose paper shall be cut with the paper surface (matte surface) uppermost, the polyethylene film down. Using a plastic straight edge or ruler as a guide, the cutting device shall be used to cut strips of width from 0,15 cm upwards. Scissors may also be used for this step. The long paper strip shall then be cut into appropriate lengths (about 0,8 cm to 3 cm) using scissors, to give the test paper, i.e. the piece of paper used for testing the test sample.

Irregular shapes may be used since their area is determined from their weight. It will be appropriate to specify/use more than one dimension since various parts of test samples differ in size, and test samples differ in dimensions and design.

NOTE The area of the applied test paper will affect the precision of the final result: the larger the area of the applied test paper, the better will be the precision of the migration test. On the other hand, too long a piece of paper may make wrapping difficult.

8.4.1.4 Immediately after weighing the square sample (8.4.1.2), determine the surface area of the pieces of dry test paper. Weigh each piece precisely (0,1 mg or better) by use of the laboratory balance (8.2.3), and calculate its surface area from its weight and the specific weight of the laboratory cellulose paper.

NOTE This can be done before testing for a large number of paper pieces.

8.4.1.5 Put each set of test papers into a separate labelled tube and record the total surface area of each set together with the tube's identification number or code.

The sets are constituted by the combinations shown in Table 1, i.e. right and left rim, right and left side together for each of the two test samples and bridges or brace bars of the two test samples together: five combinations and, hence, five sets (tubes) in total for the two test samples.

8.4.2 Preparation of artificial sweat solution

8.4.2.1 General

The composition and preparation of the artificial sweat solution shall be in accordance with 8.4.2.2 to 8.4.2.4.

The artificial sweat solution shall be prepared on the day of use.

8.4.2.2 Reagents

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

8.4.2.2.1 Deionized water according to EN ISO 3696, grade 2.

8.4.2.2.2 Sodium chloride

8.4.2.2.3 DL-Lactic acid, ρ = 1,21 g/ml, > 88 % (mass fraction).

8.4.2.2.4 Urea

8.4.2.2.5 Sodium Hydroxide in solid tablets, min 98 % pure dehydrate.

8.4.2.2.6 Preparation of 1 M sodium hydroxide solution: Weigh 4 g ± 0,5 g of sodium hydroxide (8.4.2.2.5) and transfer into a 100 ml beaker and add 50 ml of deionized water (8.4.2.2.1). Stir and cool to room temperature. Transfer the solution to a 100 ml volumetric flask and make up to volume with deionized water (8.4.2.2.1).

8.4.2.2.7 Preparation of 0,1 M sodium hydroxide solution: Add 5,0 ml of 1 M sodium hydroxide (8.4.2.2.6) in a 50 ml volumetric flask and make up to volume with deionized water (8.4.2.2.1).

8.4.2.2.8 Hydrochloric acid, ρ = 1,16 g/ml, 32 % (mass fraction).

8.4.2.2.9 Preparation of 0,1 M hydrochloric acid solution: Add 50 ml of deionized water into a 100 ml volumetric flask, then add 1 ml of hydrochloric acid (8.4.2.2.8) and make up to volume with deionized water (8.4.2.2.1).

8.4.2.3 Apparatus

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

8.4.2.4 Preparation of artificial sweat solution

The artificial sweat solution consists of deionized water (8.4.2.2.1) containing:

— 0,5 % (mass fraction) sodium chloride (8.4.2.2.2);

— 0,1 % (mass fraction) lactic acid (8.4.2.2.3);

— 0,1 % (mass fraction) urea (8.4.2.2.4); and

— 1 M (8.4.2.2.6) and 0,1 M (8.4.2.2.7) sodium hydroxide solution.

The artificial sweat solution shall be prepared as follows:

Pour 900 ml of freshly prepared deionized water (8.4.2.2.1) into a 1000 ml beaker. Add 1,00 g ± 0,01 g of urea (8.4.2.2.4), 5,00 g ± 0,05 g of sodium chloride (8.4.2.2.2) and 1,00 g ± 0,01 g of lactic acid (8.4.2.2.3), 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 artificial sweat solution and measure the pH. Slowly and gently, while continuing stirring, add drop by drop a volume of 1 M sodium hydroxide (8.4.2.2.6) until a pH of 5,50 ± 0,05 is reached and subsequently with continuous stirring, add slowly and gently drop by drop a volume of 0,1 M sodium hydroxide (8.4.2.2.7) until a pH 6,50 ± 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 6,50 ± 0,05.

Transfer the solution to a 1000 ml volumetric flask and make up to volume with deionized water. Before use, ensure that the pH of the artificial sweat solution is in the range of pH 6,50 ± 0,05.

If it is necessary to reduce the pH of the solution to 6,50 ± 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 (8.4.2.2.9).

8.4.3 Applying artificial sweat solution to the test paper and attaching it to the test sample

8.4.3.1 Take the test papers out of the tube, record the set identification number or code together with the test part's assigned identification number or code.

8.4.3.2 Identify the bare paper surface and place on the absorbing paper (8.2.11) with this side up. Then, using the Pasteur pipette or micropipette (8.2.10), saturate with artificial sweat solution (8.4.2). Add sufficient artificial sweat solution to overflow so that the absorbing paper underneath the test paper takes up the excessive solution. Check by visual observation for a change in colour of the absorbing paper to indicate that saturation has been reached.

8.4.3.3 Then immediately place the test paper on the required part of the test sample, using appropriate tweezers (8.2.2).

8.4.3.4 Seal with sealing film of sufficient size so that the test paper is securely held in place and the evaporation of artificial sweat solution will be prevented.

See normative Annex B for further details on where the test paper shall be applied and on wrapping or folding.

WARNING — The positioning of the test paper and its sealing are critical steps of the overall procedure. The operator will require training to perform these steps adequately while ensuring that the test paper does not move during the wrapping or folding procedure.

8.4.4 Blank sample

A blank sample shall be prepared and analysed with every batch of test samples.

Impregnate a piece of test paper of minimum area 0,6 cm^2 according to 8.4.3.2.

Extract the blank sample paper according to 8.4.7.2 using 3 ml of 2 % nitric acid.

Analyse the resulting solution for nickel according to 8.4.7.3.

8.4.5 Incubation of test sample with test paper attached (release of nickel into paper)

Within 30 min of sealing, place the test samples in the climate chamber at a temperature of 30 °C ± 2 °C and relative humidity of at least 95 %. Incubate for 168 h ± 2 h (i.e. 7 d), then remove the test samples. The test samples shall be placed horizontally to avoid the risk of condensation running down the test part and under the sealing film, and without contact with each other to avoid cross-contamination.

8.4.6 Retrieval of the test paper from the test samples

Immediately after removing the test samples from the climate chamber, open (if folded) or cut (if wrapped) the sealing film and remove the test paper with appropriate tweezers (8.2.2), then put the test papers of each combination into a graduated polyethylene tube and record the assigned identification numbers or codes. Then proceed to analysis (see 8.4.7).

WARNING — On removal of the test paper from the sample, it may break into pieces. Take care to retrieve all pieces including any remaining on the sealing film.

If the analysis is not made immediately, put the test papers into a labelled graduated polyethylene tube.

Once the test papers are in the closed tube, the time until the analysis is made is not critical, and they may therefore be left, preferably by no more than 3 d, until the nickel analysis is undertaken.

8.4.7 Analysis of the test papers for nickel

8.4.7.1 General

The analysis of the test papers for nickel is based on extraction in 2 % nitric acid followed by ICP-OES, ICP-MS or GFAAS.

Alternative methods, e.g. methods involving solid sampling, may be used if shown to give equivalent results.

To have a sufficient amount of nickel for analysis by ICP-OES, the absolute minimum test area to use is 0,6 cm^2. If, however, the dimensions of the test sample allow, an area larger than 0,6 cm^2 shall be used.

NOTE See 8.3.1 and Annex B.

The volume of 2 % nitric acid to use will be governed by the area of the test paper used.

For ICP-OES, the volume of 2 % nitric acid to surface area ratio shall not exceed 3 ml per 0,6 cm^2.

Other equipment may be more sensitive and allow a different ratio, e.g. ICP-MS.

8.4.7.2 Extraction procedure

Add a quantity between 3 ml and 5 ml of 2 % nitric acid depending on the requirements of the instrument that will be used for the analysis into the graduated polyethylene tube with the test papers.

Place the tube in an ultrasonic bath at room temperature; subject the tube to ultrasound for 15 min without heating.

NOTE The test paper will not dissolve.

Carefully remove the tube from the ultrasonic bath.

Then proceed to analysis of the extraction solution (see 8.4.7.3), preferably on the same day as the extraction.

8.4.7.3 Determination of nickel release

The determination of nickel in the extraction solution shall be done in accordance with a standard procedure.

NOTE Standard procedures are, e.g. EN ISO 11885 (ICP-OES) and EN ISO 17294-2 (ICP-MS).

The following principal points shall be observed:

a) Detection limit:

   1) If the measured concentration of nickel release is below the limit of detection of the analytical spectrometer, the result shall be reported as a release rate of e.g. < *x* µg/cm²/week, where *x* is the limit of detection in µg.

   2) If it is necessary to dilute the extraction solution for measurement purposes, the nickel concentration of the diluted extraction solution shall exceed the limit of quantification of the analytical spectrometer.

   3) The limit of detection of the analytical spectrometer shall be in the order of 10 µg/l.

b) Calibration: The calibration solutions used for the nickel determination shall match the matrix of the extraction solution and, if required, the diluted extraction solution, plus any added 2 % nitric acid. The concentration range of the calibration solutions shall be chosen so that they can accurately determine the concentration of nickel in the extraction solution and if required the diluted extraction solution.

c) Control the calibration with a certified standard solution prepared by a different supplier from the one used to prepare the calibration solutions.

d) Ensure that the materials and solutions used are not contaminated with nickel by performing a blank sample (extract an unused test paper) each day on which tests are performed.

Make three replicate measurements of the nickel concentration on the same day from each extraction solution, and calculate the mean value.

8.4.7.4 Quality control solution

8.4.7.4.1 Preliminary considerations

The quality control solution will allow the monitoring of the extraction procedure and the determination of the nickel release in test papers. Six test paper samples, two sets of three, shall be impregnated with one of two different concentrations of the quality control solution. The test papers

impregnated with the quality control solutions shall be analysed for nickel at the same time as the test papers from nickel release.

8.4.7.4.2 Preparation of the control solutions

Quantitatively transfer the required volume of the nickel ICP Standard Solution to an appropriately-sized volumetric flask and dilute to volume using the artificial sweat solution to obtain two control solutions of the following concentrations:

— 2,5 mg/l;

— 5,0 mg/l.

8.4.7.4.3 Procedure

Cut six pieces of test paper of area approximately 1,0 cm^2 ± 0,2 cm^2. Using a micropipette impregnate each of the pieces of paper with 100 µl of one of the concentrations of the quality control solutions. Three pieces of test paper shall be used for each concentration. The papers in each set will therefore have been impregnated with 0,25 µg or 0,50 µg of nickel, respectively.

Perform the extraction as described in 8.4.7.2, and determine the nickel in the test paper (as described in 8.4.7.3).

8.4.7.4.4 Calculation and report

For each set of impregnated test papers (0,25 µg and 0,50 µg), calculate the amount of nickel that has been recovered from the test paper.

Report the mass of nickel (in µg) evaluated by the spectrophotometer and hence calculate the percentage recovered. If the recovery is not above 90 % the laboratory will have to check its measurement procedure.

8.5 Calculation of migration test results

Calculate and present the result as the amount of nickel released in µg/cm^2/week for each combination of test parts.

Give an estimate of the uncertainty of the result.

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

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

where

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

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

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

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

8.6 Interpretation of migration test results

8.6.1 General

An inter-laboratory comparison of this test method was undertaken in 2013.

The performance characteristic *u* arising from this trial defines the relative uncertainty in the calculated nickel release (see 8.5). The measurement uncertainty from this trial was 31 %.

For the calculation of compliance assessment the expanded measurement uncertainty is used.

8.6.2 Assessment of compliance

It is an essential requirement that all laboratories interpret their results using the same procedure. In order to determine whether a test part is non-compliant a statistical test is applied. This test decides whether a determined nickel release value significantly exceeds the 0,5 µg/cm²/week limit.

$$\overline{r}_{\text{measured}} \leq r_{\text{limit}} \cdot \left(1 + \left(k\left(\alpha\right) \cdot u\right)\right) \quad (2)$$

where

$k(\alpha)$ is the coverage factor for the chosen significance level of 0,05 (95 %) and the one sided *t*-test which gives a corresponding value of 1,65, assuming a large number of degrees of freedom for the uncertainty;

u is the measurement uncertainty of 0,31 (31 %) arising from the inter-laboratory comparison performed in 2013;

r_{limit} is the legal 0,5 µg/cm²/week limit;

$\overline{r}_{\text{measured}}$ is the mean of the replicates of the nickel release determinations [value *d* in Formula (1)].

A combination of test parts will be deemed to be non-compliant when the nickel release value *d* (see 8.5) is greater than or equal to 0,76 µg/cm²/week according to Formula (1).

This 0,76 µg/cm²/week result is based on the measurement uncertainty being 31 %. The laboratory shall therefore ensure that their own measurement uncertainty is not greater.

8.7 Test report

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

a) identification of the model and test sample(s), respectively, including source, date of receipt, description, and, optionally, the assigned identification number(s) or code(s);

b) a reference to this document, i.e. EN 16128:2015;

c) documentation (verbal description and, optionally, photographs) of the parts of the test sample with paper attached, and including the size of the test area, expressed in cm²;

d) the volume of 2 % nitric acid used;

e) for each replicate measurement, the nickel release value; the laboratory's measurement uncertainty shall be available on request;

f) the limits of quantification and detection of the instrument;

g) the test result for each combination of test parts, and a statement on compliance or non-compliance of each combination of test parts, and therefore the model, in accordance with 8.6.2;

h) any unusual features observed during the determination;

i) starting and completion dates of test;

j) identification of laboratory carrying out the test;

k) signature of person responsible for the validation of the test report.

EXAMPLE The numerical values of the test result for each model could be presented in the form of the table below.

Test sample	**Test part**	**Area of each paper**[a] [cm^2]	**Total area**[c] [cm^2]	**Volume of extraction solution**[d] [cm^3]	**Analysed nickel concentration**[e] [mg/l]	**Nickel release** [$\mu g/cm^2$/week]
First test sample (optionally give the assigned identification number or code)	right rim					
		b				
	left rim					
		b				
	right side					
	left side					
	bridge					
Second test sample (optionally give the assigned identification number or code)	bridge					
	right rim					
		b				
	left rim					
		b				
	right side					
	left side					
Overall test result[f] **(pass or fail):**						

a Surface area of each test paper used for testing the single part of a test sample.

b This second cell is given for convenience should it be necessary to use two pieces of paper to test the rim.

c Total surface area of the test papers used for testing of the combination of parts of a test sample.

d Volume of the extraction solution used for testing of the combination of parts of a test sample.

e Result of analysis for the combination of parts of a test sample.

f Defined by: Pass – all combinations of test parts pass, Fail – any (or all) combination(s) fails.

Annex A
(informative)

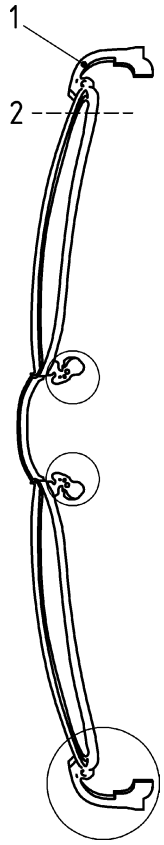
Cutting and masking of test samples (Coating test)

A.1 Fronts

The front can be suspended by one lug, say the left, keeping the joint and closing block above the saline solution. In this case, the pad arms and boxes, right lug, joint and closing block will all need masking. See Figure A.1.

The lug area can be masked by dipping, as shown in Figure A.2.

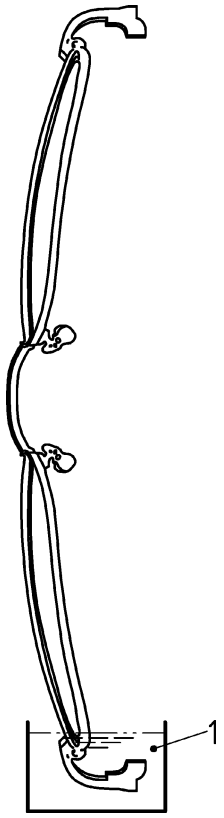
To define the test area more precisely, it may be better to mask both lug areas, and then remove the masking agent on the electrical contact area on one of the lugs. If the electrical contact lug area is not partly masked, a water-proof marker pen should be used to identify the level to which the front shall be immersed.



Key

1	electrical contact area
2	surface of saline solution in electro-chemical (potentiostat) cell
circles	masked areas

Figure A.1 — Method for testing fronts



Key

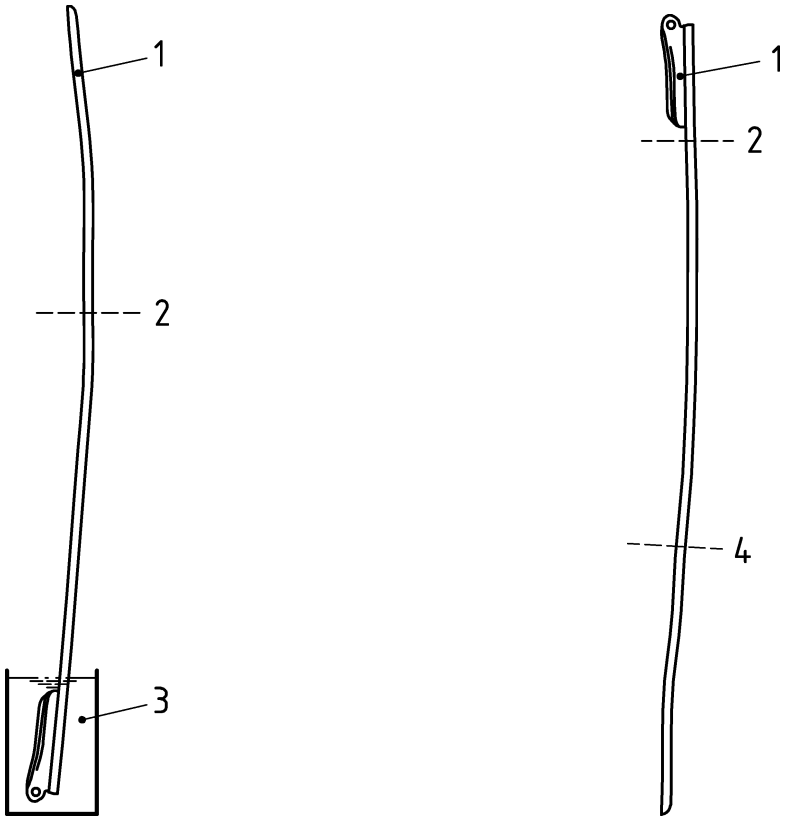
1	container of masking agent

Figure A.2 — Method for masking lugs

A.2 Sides (temples)

Electrical contact can be made on the part of the side that is normally covered by the plastics end cover (tip), see Figure A.3 a). The joint will need masking, and the saline solution level will need to be just below where the side tapers to take the plastics end cover (tip). To define the test area more precisely, it may be better to mask both ends of the side, and then remove the masking agent on the electrical contact area on the tip end by wiping with a tissue or cloth before it has dried. If the electrical contact end is not partly masked, a water-proof marker pen should be used to identify the level to which the side shall be immersed.

Alternatively, the electrical contact can be made on the joint, the tip of the side that is normally covered by the plastics end cover (tip) cut off and masked, perhaps by dipping. See Figure A.3 b). Also mask the zone between (1) and (2), using, for example, a brush.



Key

1 electrical contact area

2 surface of saline solution in electro-chemical (potentiostat) cell

3 container of masking agent

4 cut and mask

Figure A.3 — Examples of masking and testing sides

Annex B
(normative)

Selection of test areas and application of the test paper (Migration test)

B.1 General

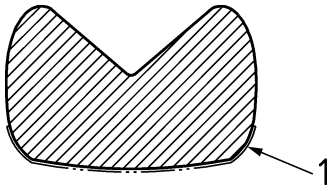
The simulation consists of applying absorbing laboratory cellulose paper (the test paper) saturated with artificial sweat solution onto the surfaces that are intended to come into direct and prolonged contact with the skin of the wearer, to cover the test paper with sealing film to avoid evaporation and allowing the migration process to take place, followed by removal of the test paper, extraction and analysis. This Annex provides guidance on, firstly, selecting the areas where the test papers shall be applied, and secondly, on how they shall be applied.

To hold the test paper securely in place, the stretchable sealing film can be wrapped or folded over the test part and test paper. Wrapping is recommended only for wide surfaces. Folding is much more suitable for fine parts such as the rim. Fold the sealing film over the test part and test paper so that the top and bottom of the sealing film meet, and press them firmly together to seal all the film; verify that no air bubbles are trapped between the two layers. The temperature in the climate chamber will improve the adhesion of the sealing film to the test part; folding rather than wrapping makes the retrieval of the test paper from the test samples faster because the sealing film can be unpeeled and does not need to be cut.

NOTE This technique requires practice before consistent results are obtained.

B.2 Rims

The test paper shall be applied on the outer surface of the rim, as indicated by (1) in the cross-section shown in Figure B.1.



Key

1 test area

Figure B.1 — Illustration of a spectacle rim in cross-section, showing the area to be tested

The possible areas to be tested are indicated by the letter T or T1 in Figure B.2. Of these, the lower rims should preferably be tested (T), and the two test papers from each test sample (right and left rim) combined together for the analysis (see 8.3.1).

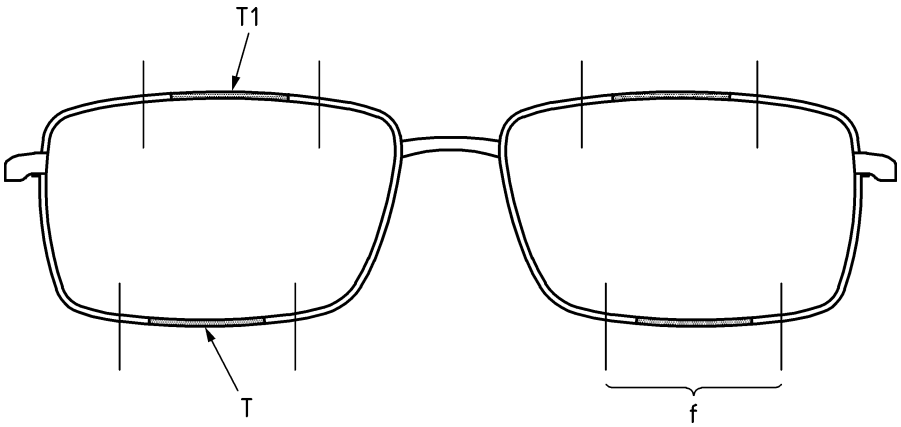
Avoid applying the test paper to the groove and the steeply curved edges adjacent to the groove.

Cut a length of sealing film that exceeds the width of the test paper and stretch the middle across what was the width when on the roll.

Prepare the test paper as indicated in 8.4.3. Using appropriate tweezers, apply the saturated test paper to the rim with the paper side against the test part, lay the stretched sealing film on the test paper and then keep the test paper and sealing film in position with a finger without applying excessive pressure

which could squeeze some of the artificial sweat solution into areas not being tested or move the test paper from its required position; then fold the sealing film.

In the rare cases where two pieces of test paper do not give a sufficient area (i.e. at least 0,6 cm^2), then the upper rims of each test sample should also be selected (T1 in Figure B.2), i.e. four pieces of test paper may be combined for the analysis.



Key

T area preferably to be tested

T1 area to be added if strips at T do not give sufficient area

f strip of sealing film

Figure B.2 — Illustration of a spectacle front, showing area of the rims to be tested (T, and if necessary, T1) **and where the strip of sealing film shall be applied**

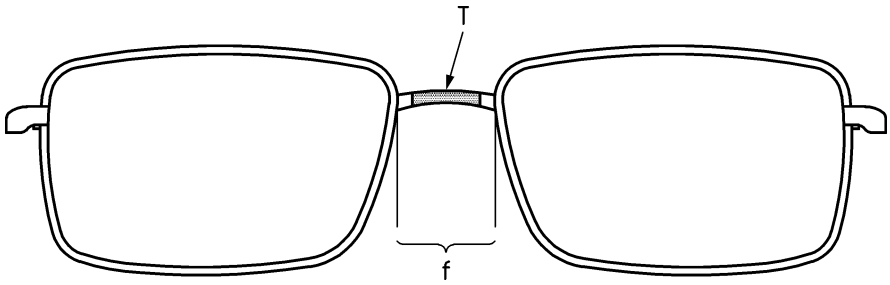
B.3 Bridge

The area to be tested is indicated by the letter T in Figure B.3.

If the frame has only a bridge, the bridges of both test samples shall be tested and the two test papers combined for the analysis (see 8.3.1). The test papers shall be shorter than the width of the bridge.

Cut a length of sealing film of the same width as the bridge and stretch it in the middle.

Prepare the test paper as specified in 8.4.3. Using appropriate tweezers, apply the saturated test paper to the rear surface of the bridge with the paper side against the test part, lay the stretched sealing film on the test paper and then keep the test paper and sealing film in position with a finger without applying excessive pressure which could squeeze some of the artificial sweat solution into areas not being tested or move the test paper from its required position; then fold the sealing film.



Key

T area to be tested

f strip of sealing film

Figure B.3 — Illustration of a spectacle front, showing the bridge and its area to be tested (T) **and the width of the strip of sealing film** (f)

B.4 Brace bar

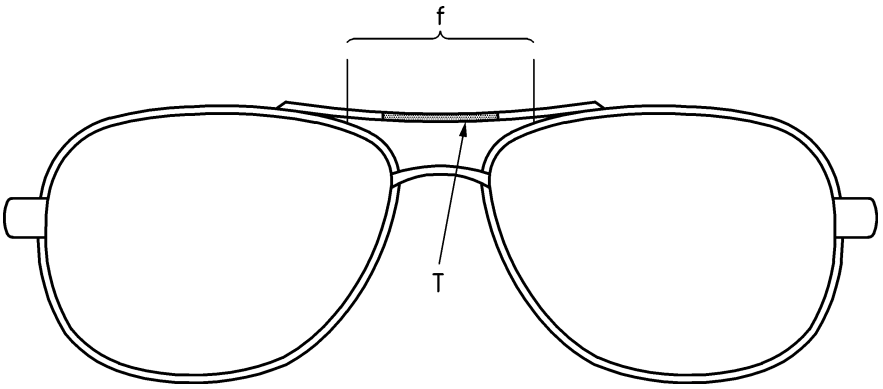
If the frame has a brace bar or both bridge and brace bar, the brace bars of both test samples shall be tested and the two test papers combined for the analysis (see 8.3.1).

The area to be tested is indicated by the letter T in Figure B.4.

The test paper shall be shorter than the distance between the soldered joints at the ends of the brace bar, while the sealing film should be cut to a length equalling or just shorter than this distance. Stretch the film in the middle.

Prepare the absorbing paper as specified in 8.4.3. Using appropriate tweezers, apply the saturated test paper to the rear surface of the brace bar with the paper side against the test part, lay the stretched sealing film on the test paper and then keep the test paper and sealing film fixed in position with a finger without applying excessive pressure which could squeeze some of the artificial sweat solution into areas not being tested or move the test paper from its required position. Then fold the sealing film.

The bridges and brace bars should be tested separately, but if the two strips of test paper from either the bridges or the brace bars do not give sufficient area (i.e. at least 0,6 cm^2), then the test papers from the bridges (see B.3) and brace bars should be combined for the analysis, i.e. four pieces of test paper in total.



Key

T area to be tested

f strip of sealing film

Figure B.4 — Illustration of a spectacle front, showing the brace bar and its area to be tested (T) **and the width of the strip of sealing film** (f)

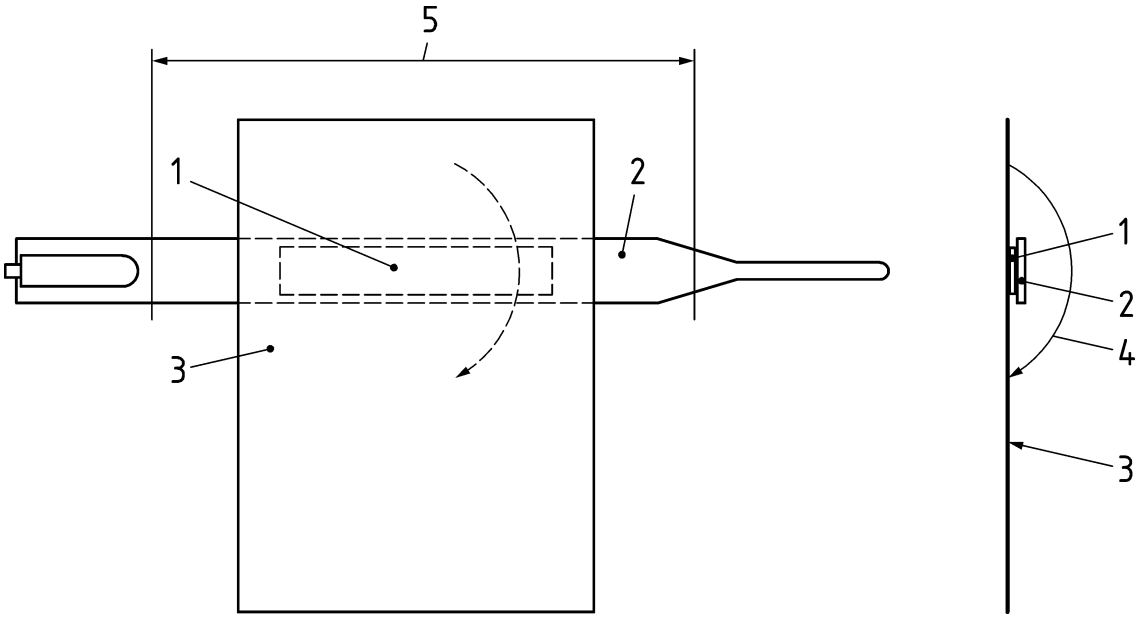
B.5 Sides

The areas to be tested for both test samples are the inside surfaces between the joint and where the end cover (temple tip), when fitted, covers the side; the test paper should be applied closer to the tip than to the joint, as shown in Figures B.5 and B.6. The two test papers (right and left side) from each test sample will be combined together for the analysis (see 8.3.1).

Cut a length of sealing film that exceeds the width of the test paper and stretch it in the middle.

Prepare the absorbing laboratory cellulose paper as specified in 8.4.3. Using appropriate tweezers, apply the saturated paper on the area to be tested with the paper side against the test part, lay the stretched sealing film on the test paper and then keep the test paper and sealing film in position with a finger without applying excessive pressure which could squeeze some of the artificial sweat solution into areas not being tested, or move the test paper from its required position. Then fold the sealing film.

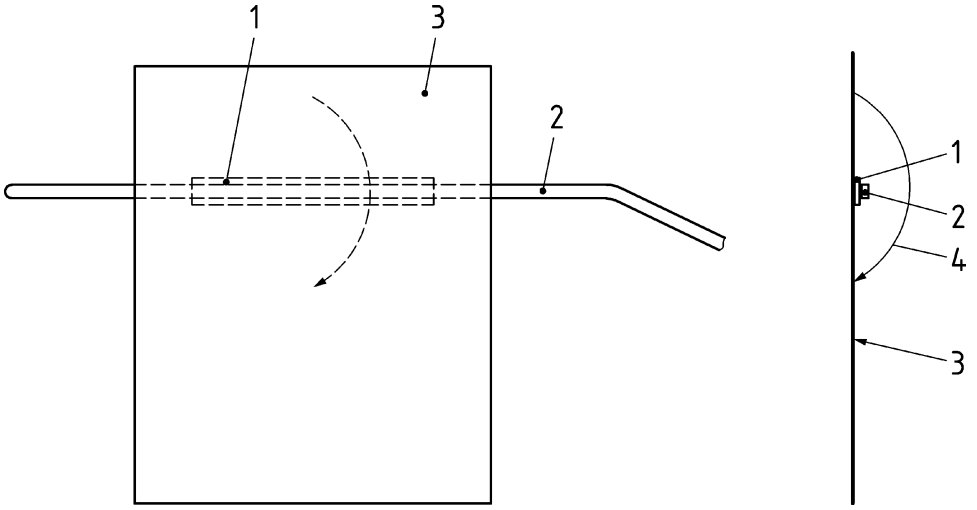
If the side is relatively large in area, as shown in Figure B.5, then the film can be wrapped securely around the area to be tested in order to seal the test paper onto the test part. Check again that the test paper has not moved. The sealing film will gradually shrink to seal the test paper to the test sample.



Key

1 test paper
2 side (temple)
3 sealing film
4 fold under
5 possible test area

Figure B.5 — Example of a broad side



Key

1 test paper
2 side (temple)
3 sealing film
4 fold under

Figure B.6 — Example of a fine side

B.6 Trims

If the sides are fitted with trims, the trims of both sides, if applicable, of both test samples shall be tested and the test papers combined for the analysis (see 8.3.1). The test papers shall be smaller than the size of the trim.

Cut a length of sealing film that exceeds the size of the test paper and stretch it in the middle.

Prepare the test paper as specified in 8.4.3. Using appropriate tweezers, apply the saturated test paper to the trim with the paper side against the trim, lay the stretched sealing film on the test paper and then keep the test paper and sealing film in position with a finger without applying excessive pressure which could squeeze some of the artificial sweat solution into areas not being tested or move the test paper from its required position; then fold the sealing film.

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[7] Regulation (EC) No 1907/2006 of the European Parliament and of the Council of 18 December 2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH), establishing a European Chemicals Agency, amending Directive 1999/45/EC and repealing Council Regulation (EEC) No 793/93 and Commission Regulation (EC) No 1488/94 as well as Council Directive 76/769/EEC and Commission Directives 91/155/EEC, 93/67/EEC, 93/105/EC and 2000/21/EC

[8] Commission Regulation (EC) No 552/2009 of 22 June 2009 amending regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorization and restriction of Chemicals (REACH)

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