TECHNICAL SPECIFICATION

ISO/TS 24348

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2007-07-15

Ophthalmic optics — Spectacle frames — Method for the simulation of wear and detection of nickel release from metal and combination spectacle frames

Optique ophtalmique — Montures de lunettes — Méthode de simulation de l'usure et de détection de la libération du nickel de montures de lunettes en métal et combinées

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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

ISO/TS 24348 was prepared by Technical Committee ISO/TC 172, *Optics and photonics*, Subcommittee SC 7, *Ophthalmic optics and instruments*.

This second edition cancels and replaces the first edition (ISO/TS 24348:2003), which has been technically revised.

Introduction

Adverse skin reaction to nickel has been known for many decades. Nickel is now the most frequent cause of contact allergy, and a significant proportion of the female population is allergic to nickel. Skin absorption of nickel ions, which are released from some nickel-containing materials in direct and prolonged contact with the skin, causes sensitization. Further exposure to soluble nickel salts results in allergic contact dermatitis. It is known that sensitization to nickel requires higher exposure levels than does the elicitation in already sensitized 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. They include this Technical Specification which provides two procedures for testing those parts of metal and combination spectacle frames that come into direct and prolonged contact with the skin.

Clause 4 specifies a method for accelerated wear to simulate two years' use of coated metal and combination spectacle frames. The coatings may include rolled gold covering, electro- and other plating methods, varnish and other organic treatments. Clause 5 attempts to provide 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. It provides a measure of the amount of nickel release from a spectacle frame when immersed for one week in artificial sweat.

Clinical patch-testing of a 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 release rate threshold of 0,5 µg/cm^2/week has been set in the European Parliament and Council Directive 94/27/EC (OJ No. L188 of 1994-07-22). In order to ensure that articles yielding values near this figure are not unnecessarily excluded from European trade as a result of the difficulties inherent in the test method, particularly when applied to intricately-shaped articles, the measured release figures are multiplied by a factor of 0,1. Materials recognised as causing sensitization to nickel would not become acceptable by use of this adjustment. Application of this Technical Specification is confidently expected to significantly reduce the development of allergic contact dermatitis due to nickel.

NOTE Experience of its use and further epidemiological and clinical research may justify changes to test procedure and/or interpretation of the test result.

Ophthalmic optics — Spectacle frames — Method for the simulation of wear and detection of nickel release from metal and combination spectacle frames

1 Scope

This Technical Specification specifies methods for accelerated wear and corrosion, to be used prior to the detection of nickel release from coated metal and combination spectacle frames, and for detecting the release of nickel from those parts of metal and combination spectacle frames, whether coated or not, intended to come into direct and prolonged contact with the skin, in order to determine whether such parts release nickel at a rate greater than 0,5 µg/cm^2/week.

This Technical Specification aims to control those spectacle frames that, if produced with materials and/or surface treatments containing nickel, can be worn by nickel-sensitized persons.

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.

ISO 12870, *Ophthalmic optics — Spectacle frames — Requirements and test methods*

3 Requirement

Those parts of metal and combination spectacle frames that come into direct and prolonged contact with the skin of the wearer shall not have a nickel release greater than 0,5 µg/cm^2/week when tested according to this Technical Specification.

Spectacle frames having a non-nickel coating shall be subject to the corrosion and wear pretreatment in Clause 4 which simulate two years' typical wear.

Spectacle frames that are made of homogeneous alloy or pure metal and are uncoated go directly to the nickel release test procedure in Clause 5.

The parts to be tested shall include:

- the rear surface of rims;
- the rear and lower surface of the bridge, the rear and upper surface of any brace bar and any other nasal-bearing surfaces, including metal nose pads;
- sides, excluding the joints and the zone immediately around the joints, and parts intended to be protected by plastic endcovers (tips).

4 Method for the corrosion and abrasion of coated metal spectacle frames before the determination of nickel release

4.1 Principle

The items to be tested are exposed to a corrosive atmosphere before being placed in a tumbling barrel together with a wear medium of abrasive paste and granules. The barrel is rotated so as to subject the test pieces to wear from the wear medium. The items are then tested for nickel release in accordance with Clause 5.

4.2 Reagents and materials

4.2.1 General

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

4.2.2 Reagents and materials for the corrosion procedure

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

4.2.2.2 **Corrosive medium**, prepared by dissolving 50 g DL-lactic acid, > 85 % purity, and 100 g sodium chloride in 1 000 ml deionized water.

4.2.2.3 **Degreasing solution**, being an appropriately diluted, neutral, commercially available detergent, e.g. a 0,5 % aqueous solution of sodium dodecylbenzene sulfonate.

4.2.2.4 **Deionized water**, specific conductivity maximum 1 µS/cm.

4.2.2.5 **Laboratory oven**, capable of maintaining a temperature of (50 ± 2) °C.

4.2.3 Reagents and materials for the wear procedure

4.2.3.1 **Tumbling barrel and retaining assembly**, in accordance with the following description:

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

NOTE Information on sourcing suitable equipment is available from the ISO Central Secretariat.

4.2.3.2 **Rotating system**, capable of imparting to the barrel (4.2.3.1) a constant (30 ± 2) rotations per minute. The rotating system shall be capable of allowing the direction of rotation to be reversed.

NOTE Information on sourcing suitable equipment is available from the ISO Central Secretariat.

4.2.3.3 **Abrasive paste**, produced for dry-tumbling barrels and comprising:

- 6 % to 8 % ester wax of montanic acids – Wax E [CAS No. 73138-45-1];
- 3 % octadecanoic acid (stearic acid) [CAS No. 57-11-4];

- 30 % to 35 % petroleum distillates, hydrotreated light paraffinic [CAS No. 64742-55-8];
- 2 % polyethylene glycol cetyl/oleyl ether [CAS No. 68920-66-1] or triethanolamine [CAS No. 102-71-6];
- 48 % silicon dioxide (quartz) 200 µm mesh size [CAS No. 14808-60-7];
- 6 % to 9 % deionized water.

NOTE Information on sourcing a suitable paste is available from the ISO Central Secretariat.

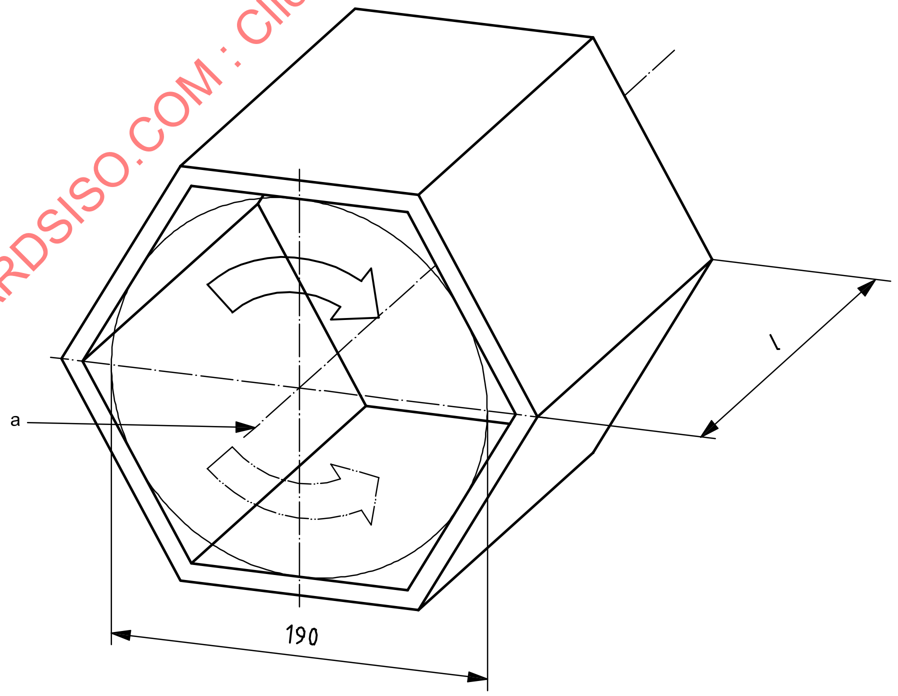
4.2.3.4 Granules, composed of outer shells of coconuts, walnuts, peanuts and almonds, mixed in a ratio 1:1:1:1 by weight, ground and sieved to give a mixture of particles having dimensions between 0,8 mm and 1,3 mm.

NOTE Information on sourcing suitable granules is available from the ISO Central Secretariat.

4.2.3.5 Wear medium, composed of abrasive paste (4.2.3.3) and wear granules (4.2.3.4) which are mixed as indicated in 4.5.1. Before use, the required amount of granulate shall be conditioned in standard laboratory conditions for at least 24 h.

4.2.3.6 Retaining assembly, consisting of a threaded rod which carries three metal hexagonal plates (see Figures 2 and 3). The end plate, A, is drilled part way through with holes of nominal diameter 1,5 mm, or as appropriate, positioned 10 mm to 15 mm from the edge of the plate, to take the ends of the tips of the sides. The next plate, B, is perforated with holes of nominal diameter 5,0 mm, or as appropriate, positioned 10 mm to 15 mm from the edge of the plate, to take the joint ends of the sides, together with an aperture of 40 mm nominal diameter to act as a filling hole for the abrasive mixture. A silicone rubber sheet with small holes matching the position of those in plate B holds the sides to prevent them from rotating in the assembly. The final plate, C, is undrilled apart from the hole for the threaded rod. A threaded nut on the inside of the last two plates holds them the required distance from plate A, while a second nut on the outside clamps the assembly together. The volume between plates A and B is approximately 5 l ± 0,5 l, but will vary depending upon the length of the sides or width of the spectacle fronts to be tested.

Dimensions in millimetres



Key

l length of barrel, as required

[a] Axis of rotation.

Figure 1 — View of tumbling barrel

Dimensions in millimetres

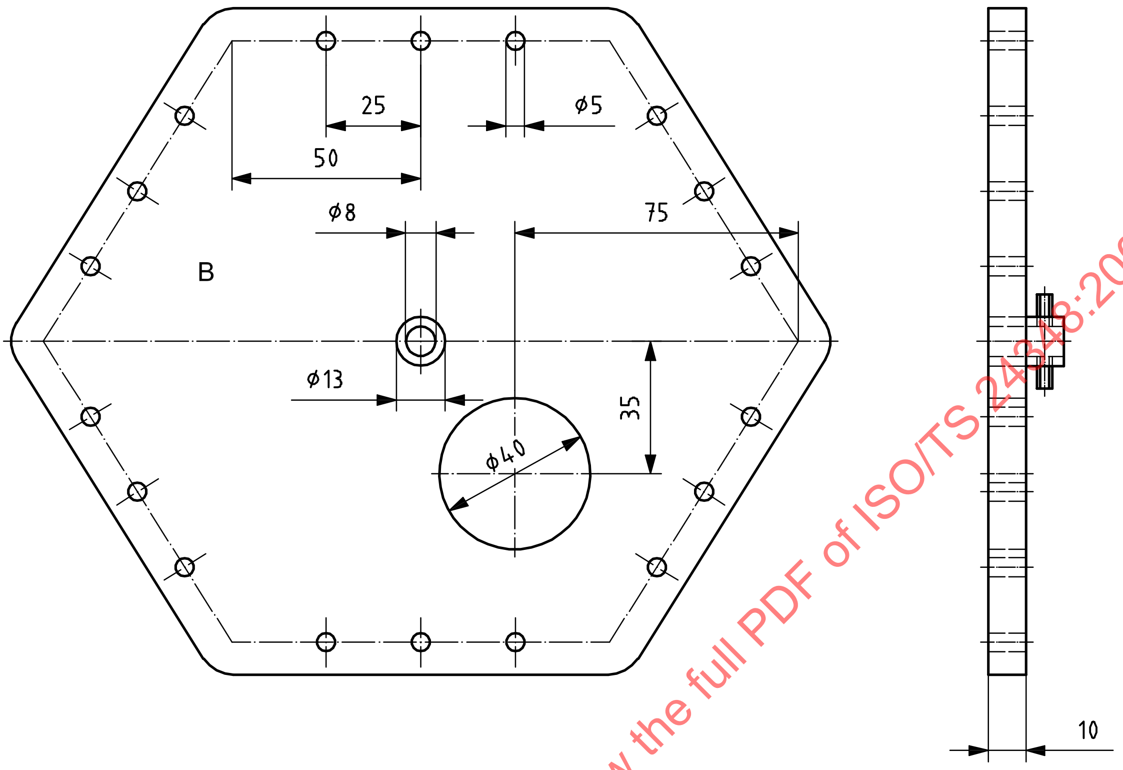
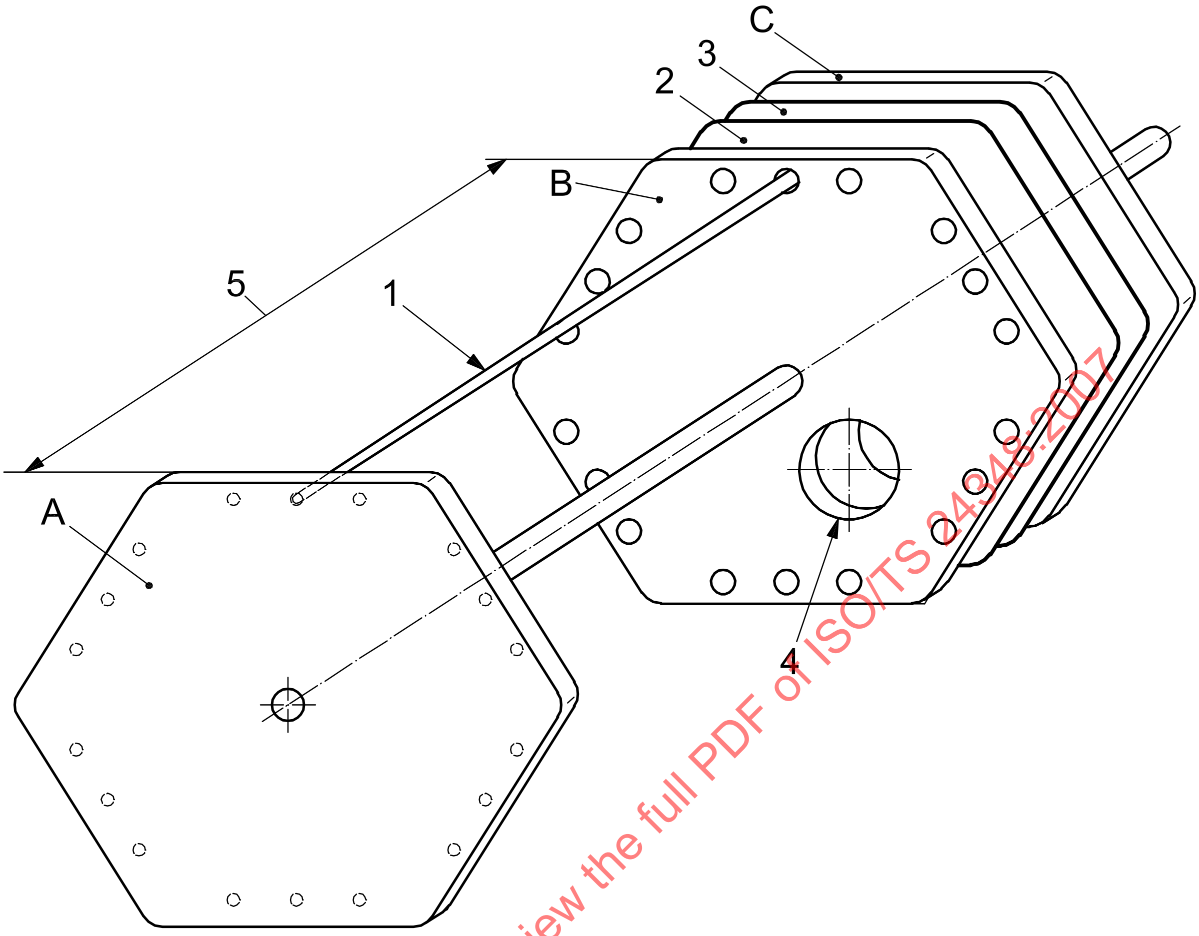


Figure 2 — Plan view of the upper part of the tumbling barrel — Component B



Key

1 sample side

2 silicone sheet, perforated

3 silicone sheet, plain

4 fill hole

5 adjust as required

Figure 3 — Exploded diagram of retaining assembly for spectacle sides

4.3 Sample preparation

Before being subjected to the corrosion procedure (see 4.4) and the wear procedure (see 4.5), spectacle sides and fronts shall be separated from each other, and endcovers (side tips) removed from sides and nose pads from fronts where appropriate. If the spectacle frame is to be subject to the accelerated wear procedure given in this Technical Specification, then it shall be fitted with test lenses as specified in ISO 12870 before being subjected to the accelerated wear procedure.

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

Gently swirl the sample(s) for 2 min in degreasing solution (4.2.2.3) at room temperature. Rinse thoroughly with deionized water (4.2.2.4) and gently dry with absorbent paper. After degreasing, samples should be handled with plastic forceps or clean protective gloves.

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

4.4 Corrosion procedure

Suspend the items to be tested a few centimetres above the corrosive medium (4.2.2.2) in a closed container (4.2.2.1) placed in the laboratory oven (4.2.2.5) for 2 h at 50 °C. Remove the container from the oven and carefully open it under a fume hood. Rinse the items with deionized water (4.2.2.4). Place on absorbent paper and allow to dry at room temperature for about 1 h, then perform the wear procedure specified in 4.5 without delay.

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

4.5 Wear procedure

4.5.1 Preparation of wear medium

Weigh a sufficient quantity of wear granules (4.2.3.4) in order to fill the tumbling barrel (4.2.3.1) to half its depth. Add 7,5 g of abrasive paste (4.2.3.3) for every kilogram of wear granules and homogenize by rotating in the barrel for 5 h. If the wear medium has not been used within 1 week, re-homogenize by rotating it in the barrel for 1 h.

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

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

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

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

4.5.2 Attachment of test items

Attach the items inside the retaining assembly so that they cannot come into contact with each other or collide with the barrel walls or other parts that could cause damage during tumbling.

Fix the test samples into the two hexagonal flanges of the barrel, positioning them with the inside surface of sides or the back surface of fronts turned towards the rotation axis. Fit the samples so that they do not move. Fill any vacant positions with waste samples.

Sides shall be fitted into the holes in plates A and B, the silicone rubber sheets holding the sides firmly to prevent them from rotating in the assembly.

Spectacle fronts shall be mounted so that one of the lugs is inserted into the holes in plate B. It may be necessary to straighten the lug or twist the rim immediately either side of the lug so that the lug fits into the hole. The opposite lug should be firmly secured to plate A using adhesive tape. Alternatively, the spectacle fronts may be held to the plates using adhesive tape at both ends. Whatever method is used to fix spectacle fronts (e.g. using only adhesive tape), it is important to ensure that parts of the sample to be tested are not covered or protected.

4.5.3 Tumbling

After fitting the test samples between plates A and B, place the retaining assembly into the empty tumbling barrel (4.2.3.1), then fill the tumbling barrel to half its depth with the wear medium (4.2.3.5). Then fit the second silicone rubber sheet and plate C together with its retaining nut. Close the tumbling barrel and place it horizontally on the rotating system (4.2.3.2).

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

NOTE After tumbling, it is permissible to leave the items in the barrel overnight.

4.6 Determination of nickel release

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

Check the items for unexpected damage, e.g. test lenses fallen out of the fronts. If necessary and justified, exclude item(s) from further testing. Test the items for nickel release in accordance with Clause 5.

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

5 Method for the determination of nickel release

5.1 Principle

The parts of the spectacle frame 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 nickel release is expressed in micrograms per square centimetre per week (µg/cm^2/week).

The value of the measured nickel release multiplied by an adjusting factor of 0,1 shall be no greater than 0,5 µg/cm^2/week after the test sample's treatments and evaluations, according to the procedure specified in 5.5.

5.2 Materials and reagents

Except where indicated, all reagents shall be of recognised pro analysis, per analytical grade or better, and shall be free of nickel.

5.2.1 Deionized and aerated water: fill a tall-form 2 l beaker with deionized water, specific conductivity $\leqslant$ 1 µS/cm. Saturate with air by attaching a gas distribution tube (porosity 1) to a cork and positioning the lower end of the tube on the bottom of the beaker. Allow grease-free air to flow at a rate of at least 150 ml/min. for 30 min.

5.2.2 Sodium chloride.

5.2.3 DL-lactic acid, $\rho = 1{,}21$ g/ml, $>$ 85 % by mass.

5.2.4 Urea.

5.2.5 Ammonia solution, $\rho = 0{,}91$ g/ml, 25 % by mass.

5.2.6 Dilute ammonia solution, (1 % by mass); transfer 10 ml of ammonia solution (5.2.5) into a 250 ml beaker containing 100 ml of deionized water. Stir and cool to room temperature. Transfer the solution to a 250 ml volumetric flask and make up to volume with deionized water.

5.2.7 Nitric acid.

5.2.8 Dilute nitric acid, approximately 5 % by mass; transfer 30 ml of nitric acid (5.2.7) into a 500 ml beaker containing 350 ml of deionized water (5.2.1). Stir and cool to room temperature. Transfer the solution to a 500 ml volumetric flask and make up to volume with deionized water.

5.2.9 Degreasing solution: dissolve 5 g of an anionic surface-active agent such as sodium dodecylbenzene sulfonate or sodium alkylaryl sulfonate in 1 000 ml water. An appropriately-diluted, neutral, commercially-available detergent may be used.

5.2.10 Wax or lacquer, (suitable for electroplating purposes) capable of protecting a surface from nickel release, and 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 tested for nickel release according to 5.5 (see Annex A).

5.3 Apparatus

5.3.1 pH-meter, accurate to ± 0,02 pH.

5.3.2 Analytical spectrometer, capable of detecting a concentration of 0,01 mg nickel per litre and, after optimization, meeting the performance criteria given under a) and b):

a) **minimum precision:** the standard deviation of ten measurements of the absorption of a full matrix calibration solution containing 0,05 mg/l of nickel shall not exceed 10 %;

b) **limit of detection:** considered as twice the standard deviation of ten measurements of the absorbance of a full matrix solution containing nickel at a concentration level selected to give an absorbance just above that of the zero calibration solution. The limit of detection of nickel in a matrix similar to the final test solution shall be better than 0,01 mg/l.

It is recommended that either an inductively-coupled plasma-optical emission spectrometer or an electrothermal excitation atomic absorption spectrometer is used.

5.3.3 Thermostatically controlled water-bath or oven, capable of maintaining a temperature of (30 ± 2) °C.

5.3.4 Container, either:

a) **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 minimize contact of the sample area (see 5.4.1.1) 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 frame component to be tested.

Or, for testing spectacle sides:

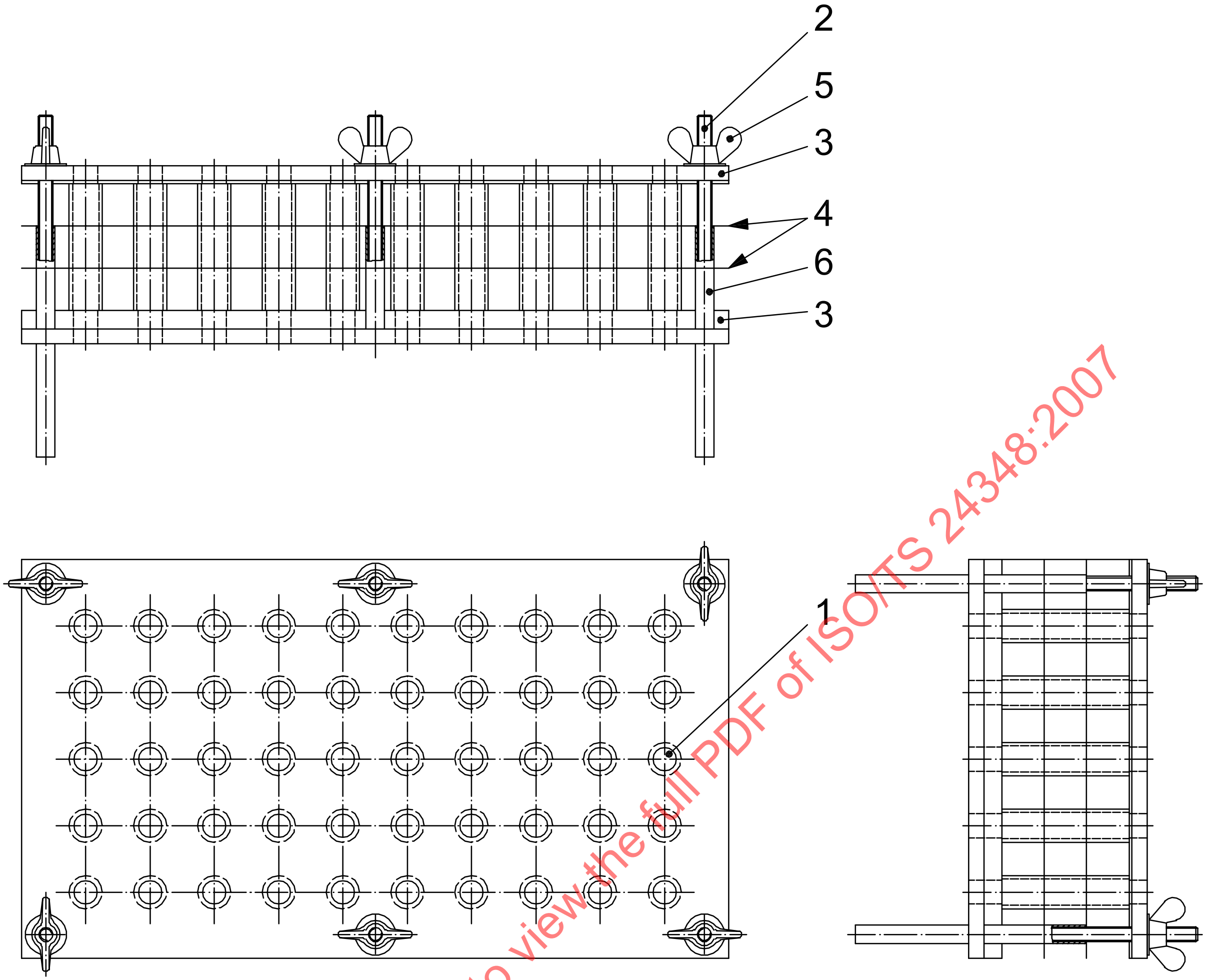
b) **test-tubes**, having suitable dimensions considering the appropriate ratio between the exposed surface of the sides and the volume of the test solution (0,5 ml/cm^2), and closed at the ends by two silicone rubber sheets. The test samples shall pass easily through the silicone rubber sheets and the sheets shall simultaneously seal the test-tubes. It is therefore always possible to place the sides with their ends outside the tube, while the parts to be tested for nickel release lie within the solution.

In order to remove any trace of nickel, the container and holder [a) or b)] shall be pre-treated by being stored in a solution of dilute nitric acid (5.2.8) for at least 4 h. After acid treatment, rinse the container and holder with deionized water and dry.

5.3.5 Test-tube holder: The test-tubes [5.3.4 b)] are inserted into a support constructed of two middle-thickness plates drilled with holes of diameter slightly smaller than the internal diameter of the test tubes, two thin plates (always drilled in register with the two load-bearing plates, holes just larger than the outer diameter of the tubes) and two silicone rubber sheets (see example in Figure 4). The size of the rubber sheets is the same as that of the stainless steel plates, while the thickness of the outer plates is nominally 8 mm and of the inner plates is nominally 2 mm.

The two rubber sheets shall form a perfect watertight seal with the ends of the tubes to avoid the solution leaking from the test-tubes. The tubes are clamped between the plates by six threaded rods that cross the whole support and are secured by the upper knobs, thus creating a compact structure. The distance between the plates is ensured by appropriate short-spacing tubes placed on the rods, while the whole structure is supported by four feet of convenient height.

The silicone rubber shall be for chemical purposes and shall demonstrably be free from releasing or absorbing nickel from the solution.



Key

1 glass test-tubes
2 threaded rods
3 drilled thick plates
4 drilled thin plates
5 plate locking nuts
6 spacing thin tubes

Figure 4 — Example of holder for the test-tubes for testing spectacle sides

5.4 Samples

5.4.1 Sample area

5.4.1.1 General

Only the surface(s) of the frame defined in Clause 3 shall be analysed. In this Technical Specification such surfaces are referred to as “sample areas”.

For convenience, manufacturers may test the bridge, rims and sides of frames without masking the inside and front surfaces of rims, the front and upper surfaces of the bridge and the front and lower surfaces of any brace bar; if the item thus tested passes the nickel release requirement, then it shall be deemed that the surfaces specified for testing in ISO 12870 would pass.

5.4.1.2 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 item is worn or used as intended. 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 items may be treated together to obtain this minimum area.

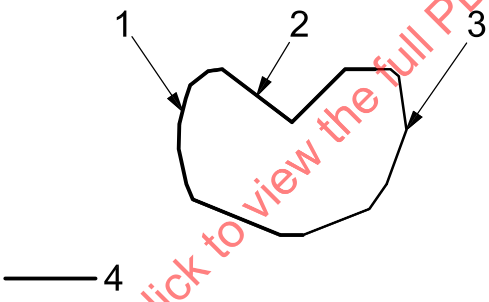
NOTE If an item is being tested to ascertain its conformity with Directive 94/27/EC, the accuracy with which the sample area of the item has to be determined is dependent on the nickel release of this item. The closer the nickel release is to 0,5 µg/cm^2/week, the limit laid down in the directive, the more accurately the surface area must be determined.

5.4.1.3 Areas other than sample areas

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 may be achieved after degreasing (see 5.4.2) by, for example, the application of one or more coatings of a wax or lacquer (5.2.10) which has been shown to protect from nickel release. Annex A gives guidance on the coating of protected areas prior to testing.

NOTE 1 Parts typically needing masking are the pad arms, joints and the inside and front surfaces of rims (see Figure 5 and paragraph two of 5.4.1.1).

NOTE 2 ISO 12870 specifies positions for cutting a front so that, for example, pad arms and closing block joints are excluded from testing.



Key

1 front surface

2 inside surface

3 rear surface

4 mask

Figure 5 — Cross-section of rim showing those parts of the rim which may be masked

5.4.2 Sample preparation

Remove test lenses from fronts.

Gently swirl the sample for 2 min in degreasing solution (5.2.9) at room temperature. Rinse thoroughly with deionized water and dry. After degreasing, the spectacle frame should 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. However, it will also substantially remove any nickel contamination that might be present on the surface of the item. If there is a requirement to determine this nickel, the cleaning stage can be omitted. However, note that omission of this cleaning stage might itself affect the nickel release from the item.

5.5 Procedure

5.5.1 Preparation of the test solution

5.5.1.1 The test solution representing the artificial sweat consists of deionized and aerated water (5.2.1) containing:

- 0,5 % by mass sodium chloride (5.2.2);
- 0,1 % by mass DL-lactic acid (5.2.3);
- 0,1 % by mass urea (5.2.4);
- ammonia solution, 1 % (5.2.6).

5.5.1.2 Transfer (1,00 ± 0,01) g of urea, (5,00 ± 0,01) g of sodium chloride and (940 ± 20) µl of lactic acid to a 1 000 ml beaker. Add 900 ml of freshly prepared deionized and aerated water and stir until all the added reagents are completely dissolved. Calibrate the pH-meter (5.3.1) in accordance with the manufacturer's instructions using freshly prepared buffer solutions. Immerse the pH electrode in the test solution. Stir gently and carefully add dilute ammonia solution until a stable value of (6,5 ± 0,1) pH is reached. Transfer the solution to a 1 000 ml volumetric flask and make up to volume with deionized and aerated water. Before use, ensure that the pH of the test solution is in the range 6,4 to 6,6. Use the test solution within 3 h of preparation.

5.5.2 Release procedure

5.5.2.1 Place the sample, suspended by its holder, in the test vessel [5.3.4 a)]. Add an amount of test solution corresponding to approximately 1 ml per cm^2 sample area. The suspended sample shall be totally immersed. However, it is not essential to immerse areas which are completely protected by wax or lacquer. The minimum volume of test solution added shall be 0,5 ml irrespective of the surface area. Close the vessel with a tight lid in order to prevent evaporation of the test solution.

When testing spectacle sides, place the test-tubes [5.3.4 b)] in the tube holder (5.3.5) and ensure that the two silicone rubber sheets perfectly adhere to the test-tubes ends in order to avoid a possible leak of solution. Then insert the test samples to be analysed into the test-tubes, pushing them through the silicone rubber until there is only the test sample area inside the tubes. Fill each tube with the freshly prepared artificial sweat solution using a syringe or a similar technique.

Note the sample area and the amount of test solution used.

Leave the vessel undisturbed in a thermostatically-controlled water-bath or oven (5.3.3) at (30 ± 2) °C for 168 h without agitation.

5.5.2.2 After one week, take the containers from the water-bath or oven, remove the samples from the test solution and rinse them with a small quantity of deionized water, adding the rinsings to the test solution. Quantitatively transfer the test solution to an appropriately sized volumetric flask (see NOTE 1) washed with acid. In order to prevent redeposition of dissolved nickel, add dilute nitric acid (5.2.8) and deionized water to the test solution to achieve a concentration of about 1 % nitric acid when the flask is made up to volume, V. The minimum final volume to which the test solution may be diluted is 2 ml.

NOTE 1 The choice of volumetric flask size should take into account the sensitivity of the instrumentation used for the nickel determination (see 5.3.2).

For sides, insert a piece of plastic tube (length: about 1 cm and diameter 1 mm to 2 mm) cut in half along its length through the rubber into each test-tube.

NOTE 2 This makes the insertion of the small aspiration tubes of the atomizer of the analytical spectrophotometer easier.

5.5.3 Determination of nickel

Determine the nickel content of the test solution using the analytical spectrometer (5.3.2).

5.5.4 Number of replicates

Whenever possible, the determination shall be carried out on at least two identical samples.

NOTE Ideally, the determination should be carried out on at least nine nominally identical samples. In this case the release value shall be associated with the mean measurement.

5.5.5 Blank tests

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, rinsing water and dilute nitric acid shall be used.

5.6 Calculations

5.6.1 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

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

where

V is the dilution volume of the sample test solution, in millilitres;

C_1 is the nickel concentration in the diluted test solution after one week, in micrograms per litre;

C_2 is the mean value of the nickel concentration in the blank solutions after one week, in micrograms per litre;

A is the sample area of the test object, in square centimetres.

5.6.2 Interpretation of results

Multiply the result, d, established in 5.6.1 by a factor of 0,1 to obtain an adjusted analytical figure.

A sample shall be deemed to have a nickel release of more than 0,5 µg/cm^2/week if the adjusted figure is greater than 0,5 µg/cm^2/week.

NOTE Due to the imprecision of the method specified in this Technical Specification, a multiplication factor is required to adjust the analytical result to take into consideration the factors detailed in Annex B, including the performance characteristics obtained from an inter-laboratory trial.

6 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 and form;

b) sampling procedure;

c) a reference of this Technical Specification, i.e. ISO/TS 24348;

d) whether the accelerated wear procedure in Clause 4 was used;

e) a description of the sample area including the size of the sample area, expressed in square centimetres;

f) the volume of test solution used;

g) for each replicate the nickel release and its adjusted figure;

h) if relevant, details of any deviations from this standard method;

i) any unusual features observed during the determination;

j) starting and completion dates of test;

k) identification of the laboratory carrying out the analysis;

l) signature of laboratory manager and operator.