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Optics and photonics — Optical coatings —

Part 4: Specific test methods: abrasion, adhesion and resistance to water

Optique et photonique — Traitements optiques —

Partie 4: Méthodes d'essai spécifiques: abrasion, adhérence et résistance à l'eau

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 172, *Optics and photonics*, Subcommittee SC 3, *Optical materials and components*.

This fourth edition cancels and replaces the third edition (ISO 9211-4:2012), which has been technically revised.

The main changes are as follows:

— Addition of a new adhesion test method (pull-off test).

A list of all parts in the ISO 9211 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Optics and photonics — Optical coatings —

Part 4: Specific test methods: abrasion, adhesion and resistance to water

1 Scope

ISO 9211 describes surface treatments of components and substrates, excluding ophthalmic optics (spectacles), by the application of optical coatings and gives a standard form for their specification. It defines the general characteristics and the test and measurement methods wherever necessary, but it is not intended to define the process method.

This document describes specific test methods of abrasion, adhesion and resistance to water for coating environmental durability tests that are identified in ISO 9211-3 but not described in other normative references. They are typically performed in sequence with other environmental durability tests, an example is shown in ISO 9211-3:2008, Annex A.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 48-2, *Rubber, vulcanized or thermoplastic — Determination of hardness — Part 2: Hardness between 10 IRHD and 100 IRHD*

ISO 9211-1, *Optics and photonics — Optical coatings — Part 1: Vocabulary*

ISO 14997:2017, *Optics and photonics — Test methods for surface imperfections of optical elements*

ISO 29862, *Self adhesive tapes — Determination of peel adhesion properties*

EN 13144:2018, *Metallic and other inorganic coatings — Method for quantitative measurement of adhesion by tensile test*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 9211-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Preparation prior to testing

Recommended storage time is at least 12 h after the coating process under ambient atmospheric conditions, or as specified between manufacturer and customer.

Before and after subjecting a coated specimen (component or witness sample) to any inspection or test, the specimen shall be properly cleaned using nonresidue cleaning agents only. This is a mutually agreed

method by the manufacturer and customer. Following the cleaning, the coated sample shall be properly dried, as for example, with a lens tissue or soft clean cloth.

5 Abrasion tests: cheesecloth/eraser test (conditioning method 01)

5.1 General

The purpose of these tests is to evaluate to what extent the optical and mechanical properties of optical coatings on components and substrates are affected when subjected to specific abrading conditions at ambient atmospheric conditions.

5.2 Test conditions

5.2.1 General

Abrasion tests shall be conducted using a coating abrasion tester capable of meeting the requirements of 5.2 and 5.3. The length of stroke of the tester shall be approximately 20 mm when the dimensions of the specimen permit. A stroke is defined as one pass in one direction on the surface being tested. The tester shall be operated in a cycling mode. A cycle is defined as one stroke in one direction, followed by a return stroke in the opposite direction. The cycle speed shall be in the range of (30 to 90) cycles per minute. The head of the tester shall be approximately normal to the surface under test during the rubbing operation. The specimen shall be firmly held so that it does not slide during the test.

5.2.2 Moderate abrasion test

The rubbing head of the abrasion tester shall be covered with a pad of cotton cheesecloth in accordance with A.1, approximately 5 mm thick and 10 mm wide. Suggested instructions for preparing the cheesecloth pad, cover and fixture are provided in Annex B.

5.2.3 Severe abrasion test

The rubbing head of the abrasion tester shall be affixed with a standard eraser, in accordance with A.2. The eraser shall be inserted into the holder so that the exposed length does not exceed 3 mm.

It is permissible to clean the eraser with a clean towel but solvents should not be used. Also, the eraser may be conditioned by rubbing it across a clean, smooth or frosted glass surface to wear away some of the rubber if embedded foreign material is suspected.

5.3 Degree of severity: abrasion – cheesecloth/eraser test (conditioning method 01)

The degrees of severity for conditioning method 01 are given in Table 1.

Table 1 — Degrees of severity: abrasion – cheesecloth/eraser test (conditioning method 01)

Degree of severity	Abrader	Number of strokes	Force N
01	Cheesecloth	50	5 ± 1
02	Cheesecloth	100	5 ± 1
03	Eraser	20	10 ± 1
04	Eraser	40	10 ± 1

A requirement for a greater number of strokes shall be indicated by adding a multiplier to the degree of severity. For example, a requirement for 150 cheesecloth rubs would be indicated as “degree of severity 01 × 3”; a requirement for 100 eraser rubs would be indicated as “degree of severity 03 × 5”.

5.4 Recovery

Subsequent to the rubbing operation, the specimen shall be cleaned as described in Clause 4.

5.5 Evaluation

The film on the specimen shall be visually examined in reflected and/or transmitted light, with the unaided eye, for evidence of physical damage to the coating. The examination shall be performed using either the methods specified in Annex C, in ISO 14997:2017, A.3, or as agreed between the supplier and the user. The method used shall be stated. The coating shall not show any evidence of damage, such as abrasion or coating removal. If slight sleeking or scratching is visible and the cheesecloth or eraser and/or coating is suspected of having foreign material embedded in it, another area of the surface shall be retested using a fresh cheesecloth pad or eraser.

6 Adhesion test: tape test (conditioning method 02)

6.1 General

The purpose of these tests is to evaluate to what extent the mechanical properties of optical coatings on components and substrates are affected when subjected to peel, i.e. combined tensile and shear stress conditions at ambient atmospheric conditions.

6.2 Test conditions

6.2.1 The adhesive tape used for this test shall be clear in colour with an adhesive strength on steel of at least 9,8 N per 25 mm width when tested in accordance with ISO 29862, Method 1, 180° angle of peel. It shall be 12 mm to 13 mm wide. It shall show no evidence of deterioration and shall be capable of being unwound from the roll at a normal rate of speed without showing evidence of adhesive offsetting, adhesive splitting, or stringing out of adhesive, nor breakage or splitting of the tape backing. The tape shall be free of bare spots or foreign particles or any defect that may affect serviceability or appearance.

6.2.2 Apply approximately 25 mm of tape to the coated surface when the dimensions of the specimen permit, with sufficient tape remaining to securely grasp with a thumb and finger.

6.2.3 Press the tape firmly onto the coated surface. Rub the non-adhesive surface of the tape with a finger to ensure firm contact with the specimen and to work out any air bubbles that may be present.

6.2.4 Unless otherwise required in the relevant specification, do not apply the tape within 2 mm of any rim of the specimen.

6.2.5 Hold the specimen firmly in one hand, and in the other the end of the tape that protrudes beyond the edge of the test specimen.

6.2.6 Remove the tape at an angle perpendicular to the coated surface at one of the rates indicated in Table 2.

6.3 Degree of severity: adhesion – tape test (conditioning method 02)

The degrees of severity for conditioning method 02 are given in Table 2.

Table 2 — Degrees of severity: adhesion – tape test (conditioning method 02)

Degree of severity	Rate of tape removal
01	Slow (approximately 2 s to 3 s per 25 mm)
02	Quick (approximately 1 s per 25 mm)
03	Snap (much less than 1 s per 25 mm)
NOTE The "snap" rate of removal refers to a snapping action of the wrist and fingers.	

6.4 Recovery

Do not clean the specimen prior to evaluation.

6.5 Evaluation

The film on the specimen shall be visually examined in reflected and/or transmitted light, with the unaided eye, for evidence of coating removal. The examination shall be performed using either the methods specified in Annex C, in ISO 14997:2017, A.3, or as agreed between the supplier and the user. The method used shall be stated. The coating shall not show any evidence of coating removal.

Unless otherwise required in the relevant specification, visual discolourations of the coating such as stains, smears, streaks or cloudiness shall be acceptable if the specimen conforms to the optical and other environmental durability requirements of the relevant specification.

7 Adhesion test: crosshatch test (conditioning method 03)

7.1 General

The purpose of this test is to evaluate to what extent the adhesion properties of optical coatings on components and substrates are affected after cutting the coating (distorting the stress and influencing the adhesion) at ambient atmospheric conditions.

7.2 Test conditions

7.2.1 Use a cutting device [e.g. a razor blade (suitable for soft substrates) or a diamond scribe] to make six parallel cuts in the coating, 1,5 mm ± 0,5 mm apart and approximately 15 mm to 20 mm in length, when the dimensions of the specimen permit. Cut through the coating completely but do not cut too deep into the substrate. Rotate the sample approximately 90° and in the same way make six parallel cuts perpendicular to the first set of cuts. Check the cut pattern for flakes or other irregularities and record the results.

7.2.2 Apply adhesive tape as specified in 6.2.1 over the crosshatch pattern in accordance with 6.2.2 to 6.2.5.

7.3 Degree of severity

There is no degree of severity necessary for conditioning method 03 and the degree of severity notation should be ignored in this instance.

7.4 Conditioning

Remove the tape according to Table 2, degree of severity 03.

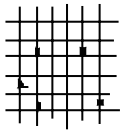
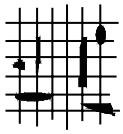
7.5 Recovery

Do not clean the specimen prior to evaluation.

7.6 Evaluation

Rate the crosshatch results according to Table 3. Magnification may be used.

Table 3 — Rating system for adhesion – crosshatch test (conditioning method 03)

Classification	Picture	Description
0	—	The edges of the cuts are completely unchanged; none of the squares are detached.
1		Small flakes of the coating are detached at the intersections; less than 5 % of the total area is affected.
2		Small flakes of the coating are detached along the cuts and the intersections; the area affected is 5 % to 15 % of the total area.
3		Some parts of the squares are detached; the area affected is 15 % to 35 % of the total area.
4		Whole squares are detached; the area affected is 35 % to 65 % of the total area.
5	—	Flaking and detachment worse than classification 4.

8 Resistance to water: exposure to water (conditioning method 04)

8.1 General

The purpose of these tests is to evaluate to what extent the optical and mechanical performance characteristics of optical coatings on components and substrates are affected after immersion in distilled or deionized water or a saltwater solution.

8.2 Test conditions

8.2.1 The test container shall be made of non-reactive material, such as suitable glass or ceramic, with a volume adequate to submerge the test part(s) completely.

8.2.2 The specimen(s) shall be held in the test container using a specimen holder made of non-reactive material, such as polytetrafluoroethylene (PTFE) or acetal polymer.

8.2.3 The water used for tests shall be distilled or deionized. Its resistivity shall be greater than or equal to 0,2 MΩ·cm, at a temperature of 23 °C ± 2 °C.

8.2.4 The pH of the water or salt solution shall be between 6,5 and 7,2, measured at a temperature of 23 °C ± 2 °C. Only diluted, chemically pure hydrochloric acid or chemically pure sodium hydroxide solution shall be used to adjust the pH. The pH shall be measured either electrometrically by means of a glass electrode, or colourmetrically using bromothymol blue as an indicator.

8.2.5 The saltwater solution shall be prepared by dissolving sodium chloride in distilled or deionized water at room temperature to obtain a concentration of 45 g/l. The sodium chloride shall not contain more than 1 % impurities in total.

8.2.6 A cyclic test consists of boiling the specimen for 2 min in either distilled or salt water and cooling it for 1 min at room temperature in distilled water. This cyclic test applies only to degree of severity 12.

8.3 Degree of severity: resistance to water – exposure to water (conditioning method 04)

The degrees of severity for conditioning method 04 are given in Table 4.

Table 4 — Degrees of severity: resistance to water – exposure to water (conditioning method 04)

Degree of severity	Exposure time	Solution
01	6 h	Distilled or deionized water
02	24 h	Distilled or deionized water
03	96 h	Distilled or deionized water
04	6 h	Salt water
05	24 h	Salt water
06	96 h	Salt water
07	5 min	Boiling distilled or deionized water
08	15 min	Boiling distilled or deionized water
09	5 min	Boiling salt water
10	15 min	Boiling salt water
11	60 min	Boiling salt water
12	2 min + 1 min	Boiling distilled or salt water[a] followed by distilled water at room temperature

[a] The user shall specify whether distilled or salt water is to be used and the number of cycles.

8.4 Recovery

After immersion, specimens subjected to the water solubility test shall be dried with a soft clean cloth. Specimens subjected to the salt solution shall be gently washed in distilled or deionized water not warmer than 38 °C to remove salt deposits. Specimens then shall be dried with a soft clean cloth or with filtered dry nitrogen gas.

8.5 Evaluation

The film on the specimen shall be visually examined in reflected and/or transmitted light, with the unaided eye, for evidence of flaking, peeling, cracking or blistering. The examination shall be performed using either the method specified in Annex C or in ISO 14997:2017, A.3, or as agreed between the supplier and the user. The method used shall be stated. The coating shall not show any evidence of physical deterioration.

Unless otherwise required in the relevant specification, visual discolourations of the coating, such as stains, smears, streaks or cloudiness, shall be acceptable if the specimen conforms to the optical and other environmental durability requirements of the relevant specification.

9 Adhesion test: pull-off test (conditioning method 05)

9.1 General

This adhesion test has a much higher degree of severity than the adhesion tests according to conditioning method 02 – tape test and conditioning method 03 – crosshatch test. Whereas conditioning method 02 test mainly refers to the peel resistance of the optical coating as deposited (including coating stress) and conditioning method 03 to the peel resistance of the coating as cut (distorting coating stress) conditioning method 05 refers to the adhesive strength of the optical coating as deposited (including coating stress).

The purpose of these tests is to evaluate to what extent the mechanical properties of optical coatings on components and substrates are affected when subjected to tensile (pull-off) stress conditions at ambient atmospheric conditions.

9.2 Test conditions

9.2.1 The test is carried out under tensile pull-off conditions by using either a tensile testing machine (single-sample test) or an analytical centrifuge (multiple-sample test). As specified in EN 13144:2018, 4.4, special attention shall be given to the selection and application of adhesives for use in the test. The selection of an adhesive class (e.g. cyanoacrylates, epoxides, polyurethanes and others) and a specific adhesive type have to consider the materials of both the coating to adhesive and the test block or test stamp to adhesive interface. All surfaces shall be clean and dry before applying the adhesive.

The adhesives shall be prepared and applied in accordance with the manufacturer's instructions and in a way that no shear forces are introduced in the assembly. The minimum quantity of adhesive necessary to obtain a uniform adhesive film thickness between the constituents of the test assembly shall be used; any excess adhesive shall be removed. Pot life, setting and curing conditions shall be in agreement with the specification of the adhesive and provided in the test report.

The adhesion of the adhesive to the test block or test stamp should be improved by slightly abrading the adherend of the test block or the test stamp prior to applying the adhesive onto it.

9.2.2 To carry out the test, a test stamp with an adherend of a diameter of 10 mm (resulting in an adhesive area of 78,5 mm^2) is adhesively bonded on the optical coating/substrate system.

9.2.3 The adherend of the test stamp is made of stainless steel. The surface of stainless steel is usually covered by a native self-healing oxide. Most of the optical coatings are also oxides. Hence, the adhesive used for bonding the test stamp on the optical coating/substrate system must be suitable for oxides. In other cases (e.g. optical semiconductors or fluoride coatings on top of the optical layer stack) an appropriate adhesive is selected.

9.2.4 After curing/setting of the bonded assembly (test stamp/adhesive/optical coating/substrate system) according to the specification of the adhesive supplier or self-defined, the assembly is ready for the pull-off test.

9.2.5 The pull-off test is performed with linear load increase of 10 N/s until rupture. From this, the rupture force can be determined. The resulting failure pattern (using test stamp and coating/substrate side for evaluation) is evaluated according to ISO 10365 and the designation of the resulting failure mode is derived.

9.3 Degree of severity: adhesion – pull-of test (conditioning method 05)

Environmental aspects such as storage (after deposition and prior to testing) under harsh environments may further significantly increase the degree of severity.

The test parameter load rate (force increase per time) may also affect the degree of severity. Typical load rates may vary from 1 N/s to 100 N/s with a default value of 10 N/s, however this may also depend on the substrate thickness and the selected adhesive for test stamp bonding.

9.4 Evaluation

In case the failure mode is identified as DF (delamination failure according to ISO 10365) or is at least close to it, i.e. partial delamination failure, the measured rupture force per 78,5 mm^2 corresponds to the adhesive strength of the optical coating or is close to it. In case the failure occurs within the adhesive (cohesive failure) or at one of the bonded interfaces (adhesive failure), i.e. at the surface of the optical coating or the test stamp, or in case even the substrate is damaged (substrate failure) the adhesive strength is larger than the measured rupture force per 78,5 mm^2. The same applies in case of mixed failure modes if no delamination failure is observed.

10 Environmental test code

The code for coating environmental durability tests according to Figure 1 can be shown on an optics drawing in the surface coating box with the other coating requirements, see ISO 10110-9 for more information.

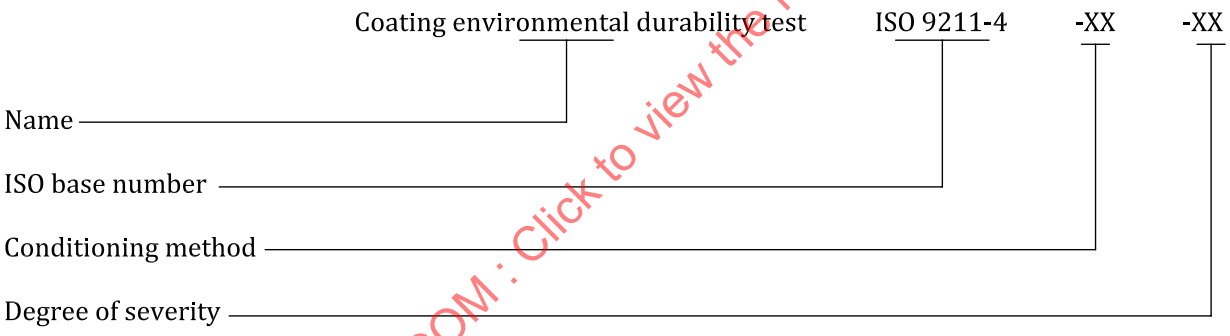


Figure 1 — Code for coating environmental durability tests

Annex A
(normative)

Materials for abrasion testing of optical coatings

A.1 Cheesecloth material

A.1.1 Yarn

The yarn shall be made from cotton, free from waste and loading materials, carded, drawn and spun into single yarns.

A.1.2 Warp

The warp of the cloth shall have 41 yarns to 47 yarns per 25 mm and the filling shall have 33 yarns to 39 yarns per 25 mm. The total number of yarns in a 25 mm by 25 mm square shall be 76 to 84. The mass shall be 45 g/m^2 to 54 g/m^2.

A.1.3 Type

The type of cheesecloth shall be bleached. Prior to use, it shall be laundered to remove completely the sizing agent and then dried.

A.2 Eraser (rubber-pumice) material

A.2.1 General

The eraser[1)] shall be a uniform mixture of rubber and abrasive, formed by an extrusion process. It shall be composed of no less than 15 % by mass of pumice. All abrasive shall be fine ground, such that 100 % will pass through a sieve with opening 45 µm. The formulation shall not contain any ingredient which might leave a residue on the surface under test that would lubricate subsequent strokes during the test procedure.

A.2.2 Hardness

The finished eraser shall have an international rubber hardness degree (IRHD) of 75 ± 5 on both ends, in accordance with ISO 48-2.

A.2.3 Accelerated aging

The eraser shall show a hardness change of not more than 10 points after being placed in an air oven for seven days at a temperature of 70 °C ± 2 °C.

A.2.4 Shape and size

The diameter of the eraser shall be from 6,5 mm to 7 mm, so that it fits tightly and is held by friction in an abrasion test fixture, such as that shown in Figure A.1. It shall be of sufficient length to be held securely in the abrasion test fixture with not more than 3 mm exposed.

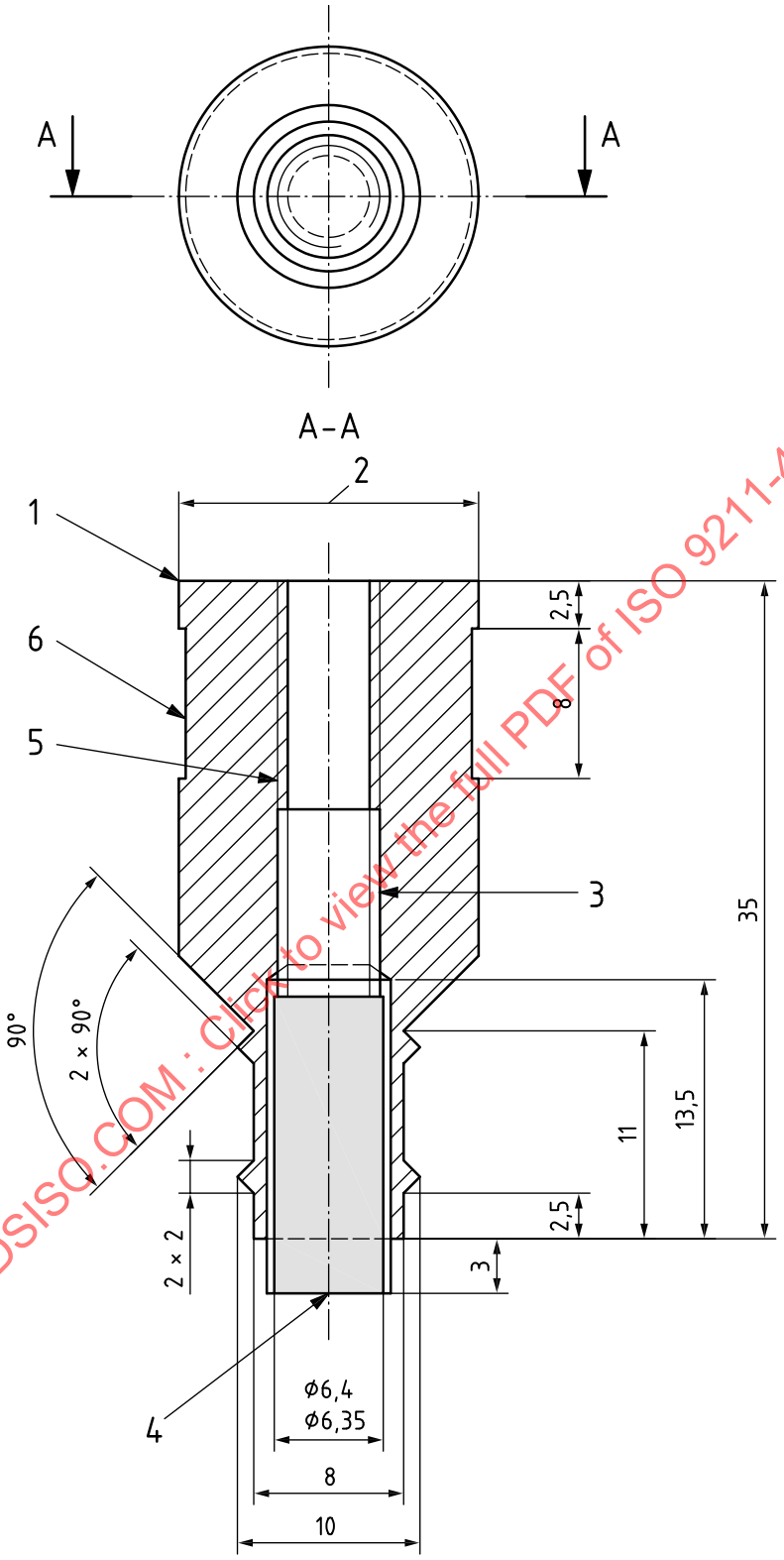
1) Erasers manufactured to these requirements are available from Summers Optical, P.O. Box 162, Fort Washington, PA 19034, USA. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this source of supply. Equivalent erasers may be available from other sources, and may be used subject to agreement between supplier and user.

A.2.5 Workmanship

The eraser shall be free from any excessive holes, cracks, splits or foreign particles which might adversely affect its use.

A.2.6 Storage

It is recommended that supplies of erasers are stored in a dark and dry environment (<60 % relative humidity) at a nominal temperature between 2 °C and 10 °C.



Key

1	abrasion test fixture	4	eraser conforming to Annex A
2	size to fit abrasion tester	5	thread for set screw
3	set screw	6	place for set screw

NOTE All dimensions except hole for the eraser are nominal and are not critical.

Figure A.1 — Abrasion test fixture with eraser installed

Annex B
(informative)

Cheesecloth pad, pad cover and fixture preparation for moderate abrasion testing of optical coatings

B.1 Cheesecloth pad preparation

B.1.1 Put on finger cots or latex, vinyl or nitrile gloves.

B.1.2 Place a clean cotton towel or clean tissue paper on the work surface.

B.1.3 Clean all tools (scissors, callipers and 11 mm diameter punch) with isopropyl alcohol (IPA) before use.

B.1.4 Cut and arrange into a stack 32 layers of cheesecloth material that complies with A.1. The length and width of the stack should be sufficient to punch from it the number of 11 mm diameter pads that are needed. When compressed, the stack should be approximately 6 mm thick.

B.1.5 Fold a clean piece of paper in half.

B.1.6 Place the stacked layers of cheesecloth inside the folded piece of paper.

B.1.7 Staple through the paper and layers at each of the four corners of the stack of cheesecloth, in order to keep them from moving out of alignment during the punching operation.

B.1.8 Place the paper-covered cheesecloth stack on a piece of polytetrafluoroethylene or similar material and place the polytetrafluoroethylene on the workspace of a punch press.

B.1.9 Place an 11 mm diameter hole punch on top of the paper-covered cheesecloth stack.

B.1.10 Punch through the stack.

B.1.11 Carefully remove the hole punch from the stack of cheesecloth.

B.1.12 Remove the completed cheesecloth pad from the inside of the hole punch by pushing it out of the punch with a dowel or similar device.

B.1.13 Carefully place the completed cheesecloth pad inside a clean container.

B.1.14 Repeat steps B.1.9 to B.1.13 to cut additional cheesecloth pads as required.

B.2 Cheesecloth pad cover and fixture preparation

B.2.1 Cut five 50 mm by 50 mm squares of cheesecloth. Two will be used as a permanent pad cover and three will be used as replaceable pad covers.