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Ophthalmic optics — Contact lenses —

Part 4: Physicochemical properties of contact lens materials

Optique ophtalmique — Lentilles de contact —

Partie 4: Propriétés physicochimiques des matériaux des lentilles de contact

Reference number
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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 on 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 the following URL: www.iso.org/iso/foreword.html.

This document 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 18369-4:2006), which has been technically revised.

A list of all parts in the ISO 18369 series can be found on the ISO website.

This corrected version of ISO 18369-4:2017 incorporates the following corrections.

—"lens" has been replaced by "contact lens" throughout the text.

—"saline" has been replaced by "saline solution" throughout the text.

—In 4.1, "repeatability and reproducibility" has been added before (R&R) to improve clarity.

—In 4.3.2.1, "single vision" has been replaced by "single-vision".

—In 4.3.5, "lens ruptures" has been replaced by "sample ruptures".

—In 4.5.2, "D line" has been replaced by "D-line".

—In 4.5.4.2.2, "4.5.4.1" has been replaced by "4.5.4.1.3", and "4.5.4.2" has been replaced by "4.5.4.2.1".

—In A.7.1, "Figure A.1" has been replaced by "Figure A.2" in two instances.

—In A.7.2, "sample" has been replaced by "test sample" in three instances.

—In A.9.7, "(p_A)" has been replaced by "p_A".

—In Figure A.2, Key 2, "anterior chamber" has been replaced by "anterior environmental chamber".

—In Figure A.2, Key 3, "posterior chamber" has been replaced by "posterior environmental chamber".

—In Annex C, “lens” has been replaced by “hydrogel contact lens”.

—In D.2, “critical angle” has been replaced by “critical angle of incidence”.

—“may” has been replaced by “can” in

- 4.2.1;
- 4.2.2;
- 4.3.1, second sentence, first “may”;
- 4.4.1, third paragraph, last sentence;
- 4.4.3.5.1, second sentence;
- 4.4.3.5.2, NOTE;
- A.9.5, fifth sentence.

—Additional minor editorial changes have been made to improve clarity.

Ophthalmic optics — Contact lenses —

Part 4:
Physicochemical properties of contact lens materials

1 Scope

This document specifies the methods of testing the physicochemical properties of contact lens materials. These are extraction, rigid lens flexure and breakage, oxygen permeability, refractive index and water content.

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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 18369-1:2017, *Ophthalmic optics — Contact lenses — Part 1: Vocabulary, classification system and recommendations for labelling specifications*

ISO 18369-3:2017, *Ophthalmic optics — Contact lenses — Part 3: Measurement methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18369-1 apply.

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

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

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

4 Physicochemical properties of contact lenses

4.1 Repeatability, test methods and units of measure

The physicochemical properties or conditions listed in Table 1 are measurable characteristics of hydrogel and non-hydrogel materials that have been used to produce commercially available contact lenses.

Table 1 — Physicochemical properties: Test methods and units of measure

Property	Units of	Test method	Repeatability
Extractables	mass %	4.2	b
Flexural deformation	g	4.3	b
Oxygen permeability	*Dk* units[a]	4.4	10 %
Refractive index	dimensionless	4.5	0,01
Water content	weight %	4.6	2 % absolute

[a] *Dk* is reported in units of 10^{-11} (cm^2/s) ml O_2/(ml × mmHg) and called "*Dk* units" or barrer.

[b] Repeatability of these test results shall be established in individual laboratories according to ISO 18369-1:2017, 3.1.12.8, 3.1.12.9, 3.1.12.9.1, 3.1.12.9.2 and 3.1.12.9.3.

Clause 4 is applicable to testing laboratories, suppliers and users of contact lens products or services in which measurement results are used to demonstrate compliance to specified requirements.

Alternative test methods and equipment may be used provided the accuracy and precision are equivalent to or more capable than the test methods described.

In developing new test methods, these should be capable of measuring the various parameters with a precision [repeatability and reproducibility (R&R)] of ≤30 % of the allowed tolerance. Resolution greater than 10 % of the tolerance can be used but will affect determination of accuracy, precision, process capability and gauge capability. The number of independent measurements should be chosen for each method to ensure appropriate precision and accuracy.

4.2 Extractables

4.2.1 General

Soxhlet extraction with different solvents is a standard method for quantitative determination of substances extractable from contact lenses. The contact lenses are dried to constant mass and the difference between the original dry mass of the lenses and the extracted dry mass determines the quantity of extractable substances (extractables).

Knowledge of the quantity and identity of extractable substances is helpful in evaluating new contact lens materials and in determining the subsequent pre-clinical examination programme. The material extracted from the contact lenses can be examined by appropriate chromatographic, spectrophotometric and wet analytical methods to identify residual monomers, cross-linking agents, catalysts, etc. that were employed in the polymerization process.

4.2.2 Principle

This method uses a normal Soxhlet extraction apparatus. Water and at least one suitable organic solvent are used for extraction. In selecting the organic solvent(s) to be used, consideration should be given to the effect of the solvent upon the matrix of the material. Ideally, a solvent should not swell or degrade the contact lens material. However, in the development of new contact lens materials, a solvent that causes reversible swelling can give valuable information relating to the possibility for extraction over extended periods of time. Choice of a solvent that degrades the polymer network during extraction is not recommended, as it will remove both uncrosslinked and crosslinked material, resulting in inaccurate measurement of extractables.

4.2.3 Apparatus

4.2.3.1 Standard borosilicate glass Soxhlet extraction apparatus (see Figure 1), consisting of the Soxhlet extractor (30 ml suggested), condenser, round bottom flask (100 ml suggested) and a heating mantle.

4.2.3.2 Perforated stainless steel, sintered glass, paper or equivalent extraction thimble fitted with a glass wool plug or other suitable closure.

4.2.3.3 Vacuum oven or equivalent drying apparatus and an analytical balance capable of weighing to 0,1 mg.

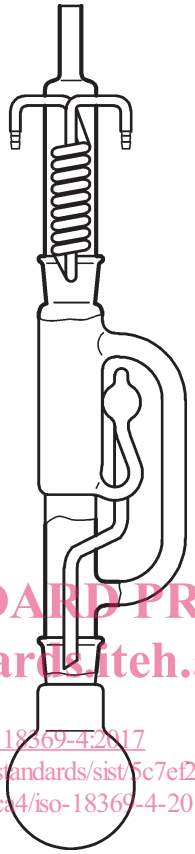


Figure 1 — Extraction apparatus

4.2.4 Reagents

4.2.4.1 Distilled or deionized water complying with ISO 3696:1987, Grade 3.

4.2.4.2 Appropriate organic solvent (see Table 2) of analytical grade or better.

4.2.4.3 Laboratory-grade boiling stones or anti-bumping granules, along with a suitable active desiccant. Selection of the desiccant will depend upon the characteristics of the test material.

Table 2 — Guide to the selection of solvents for use in extraction of contact lenses

Material	Suggested solvents	Corresponds to
Hydrogels (including silicone hydrogels)	Water (distilled or deionized)	Mild extraction (simulates in-eye extraction)
	n-Hexane, or Organic alcohol (e.g. ethanol, iso-propanol or methanol)	Mild extraction (non-polar solvent) Extraction of majority of uncrosslinked material (but swells and might degrade material)
Rigid gas permeable and silicone elastomers	Water (distilled or deionized)	Mild extraction (simulates in-eye extraction)
	n-Hexane, or Dichloromethane or chloroform	Mild extraction (non-polar solvent) Extraction of all uncrosslinked material (but swells and is likely to degrade material)

4.2.5 Test samples

Test samples shall be representative of the finished product and shall be in finished contact lens form. The method of preparing and finishing the lenses shall reflect, as far as possible, the normal production processes including sterilization. A sufficient number of lenses shall be used so that the total dry mass before extraction shall be no less than 200 mg.

Hydrophilic lenses are usually packaged in a solution containing inorganic salts. When using water as the extracting solution, an adjustment in the calculation should be made for the contribution of the inorganic salt of the packaging solution. The water content of the lenses will be required in order to accurately calculate the contribution of the inorganic salt to the extractables. Alternatively, the lenses may be equilibrated in at least two changes of water each for 24 h at room temperature prior to beginning the test.

4.2.6 Test procedure

Dry the lenses, preferably under vacuum, at 60 °C ± 5 °C or other appropriate temperature to constant mass.

NOTE 1 Drying to constant mass is achieved when two consecutive weighings between drying do not differ by more than 0,5 mg per gram of lens weight.

Allow the lenses to cool to room temperature under vacuum or in a closed container over active desiccant before weighing. Then, weigh the dry lenses to ±0,1 mg (m_1). Next, place the lenses into the extraction thimble, place boiling stones in the flask, if necessary, and fill the flask to approximately 70 % of its capacity with the appropriate solvent (see Table 2). Place the round-bottom flask in the heating mantle. Place the extraction thimble into the Soxhlet apparatus. Then, attach the Soxhlet apparatus to the flask. Place a condenser on top of the extraction apparatus. When using a volatile or flammable solvent, the extraction apparatus should be placed in a fume hood.

Turn on heat and water and extract the lenses for at least 4 h. Allow the solvent to cool to room temperature before removing the lenses from the extraction thimble. Dry the lenses to constant mass as described above and weigh to the nearest 0,1 mg (m_2). Calculate results as per Formula (1).

NOTE 2 If the dried lenses are fragile and fragmentation might have occurred leading to inaccuracies in measurement, the extraction solvent can be quantitatively dried down to constant mass and the resultant extractables residue weighed to the nearest 0,1 mg (m_3). In this case, calculate results as per Formula (2).

4.2.7 Calculation of results

The quantity of extracted material shall be expressed as a mass fraction ($m_{extracted}$) in percent of the initial dry mass as shown in Formula (1):

$$\% \text{ extracted} = \frac{(m_1 - m_2)}{m_1} \times 100 \quad (1)$$

where

m_1 is the mass of lenses prior to extraction;

m_2 is the mass of extracted lenses.

Alternatively, the extraction solvent can be quantitatively dried down to constant mass and the resultant extractables residue weighed to the nearest 0,1 mg (m_3) and used to calculate the quantity of extracted material as shown in Formula (2):

$$\% \text{ extracted} = \frac{m_3}{m_1} \times 100 \quad (2)$$

4.2.8 Test report

The test report for extractables shall conform to that in Clause 5 and contain the following information for hydrophilic material:

a) the composition of the initial hydrating solution;

b) a statement as to whether the percentage of extractable substances has been adjusted for the salt content of the hydrating solution;

c) if the contact lenses were equilibrated in water before the beginning of the test;

d) the method used to calculate quantity of extracted material, e.g. whether Formula (1) or Formula (2) was used for the calculation.

4.3 Rigid lens flexural deformation and rupture

4.3.1 Principle

The test, which is a destructive test, applies an increasing load at the edge of a rigid contact lens across the total diameter until, ultimately, the test sample fractures. The test is carried out in an apparatus which allows the load and flexural deformation to be monitored continuously. Both the flexural deformation strength and flexural deformation at rupture are determined, as well as flexural deformation strength at 30 % deformation. The latter is derived from the flexural load-deformation curve. Either normal production or specially constructed rigid contact lenses can be tested.

It should be noted that variability in the test results can also result from inconsistencies in lens manufacturing method and might not necessarily be indicative of the material itself.

4.3.2 Sampling

4.3.2.1 General samples

In order to demonstrate the degree of resistance to breakage by the material, general samples for testing shall be normal, commercially available rigid, single-vision contact lenses and shall not have been specially treated or adjusted.

Contact lenses which have toroidal zones or truncations shall not be used.

The specified label back vertex power (F'_L) shall be the same for all samples and shall be between +0,50 D and −0,50 D.

The specified back optic zone radius (r_0), or radius of the vertex sphere, shall be the same for all samples and shall be between 7,75 mm and 7,85 mm.

4.3.2.2 Samples for material comparison

When special samples are prepared in order to compare materials, the contact lenses shall have the following specifications:

— front surface: single cut, radius of curvature 8,000 mm ± 0,025 mm;

— back surface: single cut, radius of curvature 7,800 mm ± 0,025 mm;

— total diameter: 9,5 mm ± 0,1 mm;

— centre thickness: 0,20 mm ± 0,01 mm;

— edge thickness: 0,24 mm ± 0,01 mm;

— edge form: rounded;

— maximum prismatic error: 0,5 cm/m.

The method of manufacture shall be stated in the test report.

4.3.2.3 Quantity

Three contact lenses from each of three different material lots (total of nine contact lenses) shall be tested where a claim is made regarding flexure or strength.

4.3.3 Preparation of samples

Samples shall be stored in standard saline solution conforming to ISO 18369-3:2017, 4.9, for at least 48 h prior to testing. The temperature of this saline solution shall be 20 °C to 25 °C.

4.3.4 Apparatus

4.3.4.1 Testing machine (see Figure 2), applying a load to the sample at a fixed rate in either the horizontal or vertical plane and composed of the units described in 4.3.4.1 to 4.3.4.3.

Sample holding jig (see Figure 3), applying the load to the edge of the sample.

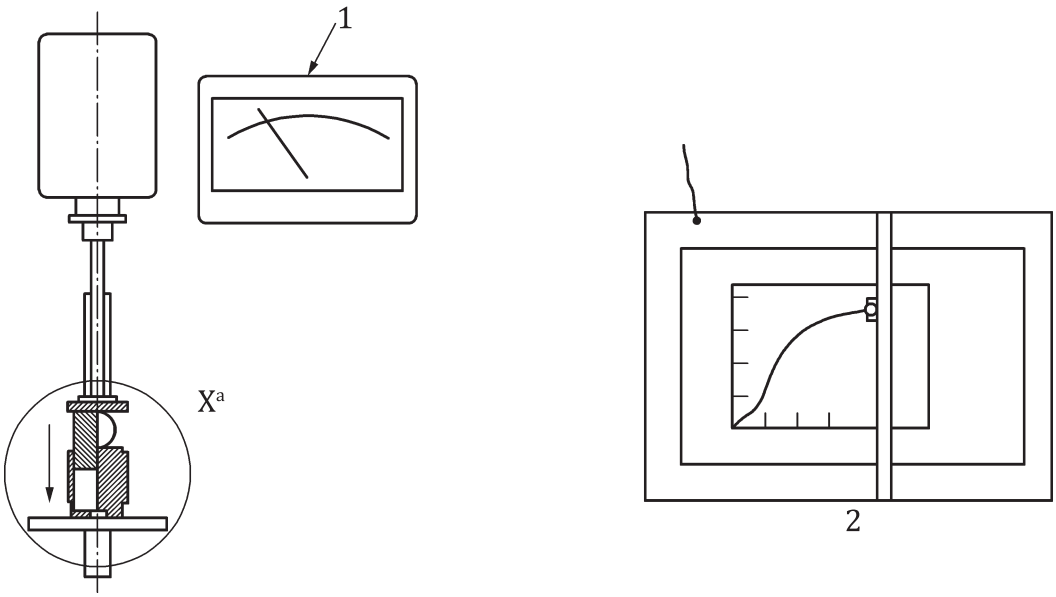
The sample is set at the centre of the upper and lower contact faces so that the whole load is applied in the plane containing the edge.

NOTE The contact faces are constructed so that the load is the only force applied to the sample.

4.3.4.2 Load indicator, capable of indicating the total load applied to the sample.

4.3.4.3 Data recorder, to which the testing machine is connected, and which, after commencement of application of the load to the sample, provides a recording of the total load applied to the sample as a function of time.

Although it is conventional to use a paper-strip (chart) recorder, other devices may be utilized. If a paper-strip recorder is used, a minimum paper speed of 1 cm/s is recommended.



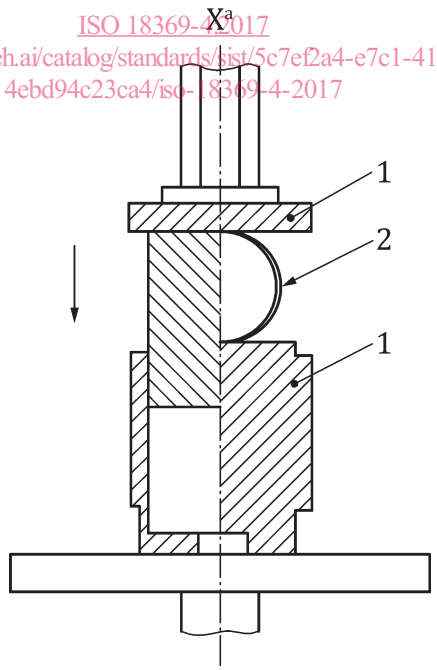
Key

1 load indicator

2 recorder

a See Figure 3 for detail X.

Figure 2 — Testing machine



Key

1 test specimen setting jig

2 test specimen

a Detail of Figure 2.

Figure 3 — Test specimen setting jig