INTERNATIONAL STANDARD

ISO 18369-4

Second edition
2017-08

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
ISO 18369-4:2017(E)

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Foreword

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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).

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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.

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 (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 may 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 may 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.

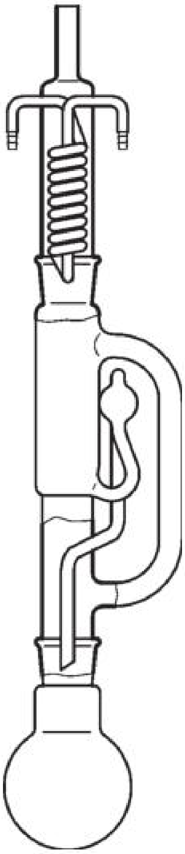


Figure 1 — Extraction apparatus

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 may 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 may 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 ($m_{\text{extracted}}$) in percent of the initial dry mass as shown in Formula (1):

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

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):

$$\%\ \text{extracted} = \frac{m_3}{m_1} \times 100 \tag{2}$$

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 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 may also result from inconsistencies in lens manufacturing method and may 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.

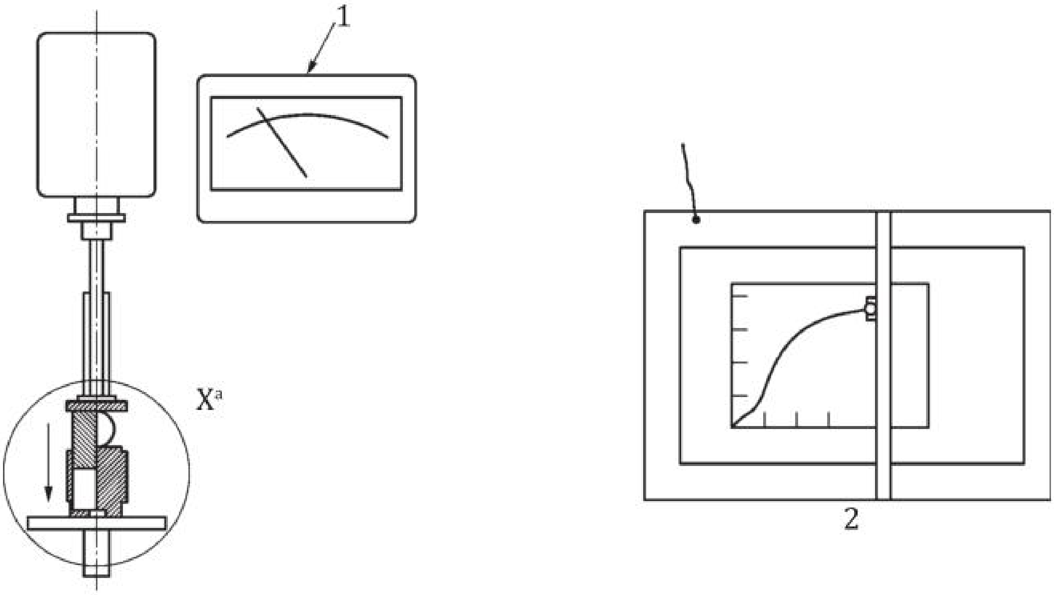
The sample is set at the centre of the upper and lower contact faces so that the whole load is applied in the plane containing the edge.

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.

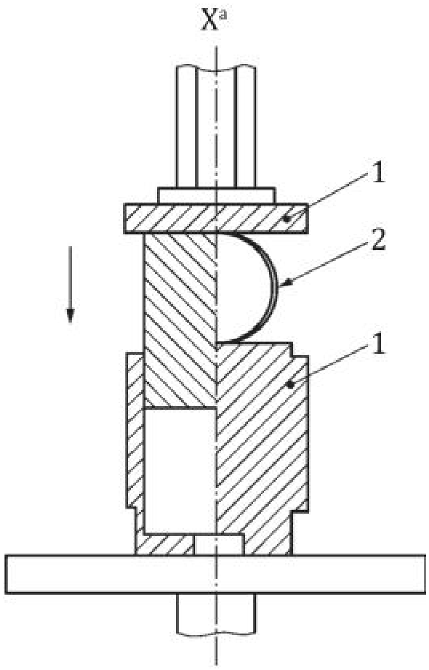
Although it is conventional to use a paper-strip (chart) recorder, other devices may be utilized. If a paper-strip recorder is used, a minimum paper speed of 1 cm/s is recommended.



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

4.3.5 Procedure

Confirm the correct operation and calibration of the apparatus.

Carry out the test at a temperature of 20 °C to 25 °C.

Remove the conditioned sample from the saline solution and dry it carefully.

Measure the back optic zone radius, total diameter, centre thickness and label back vertex power as described in ISO 18369-3. Position the sample in the jig so that the upper and lower edges of the sample lie along the centre line of the upper contact face. Set the velocity of the moving contact face to 20 cm/min (3,33 mm/s) ± 10 %.

The sample and jig may be set horizontally or vertically. If a horizontal system is used, it is necessary to confirm in advance of the test that results do not differ from those obtained using a vertical system.

Start the data recorder and then commence applying the load to the sample. Stop applying the load when the lens ruptures. Record the load in grams at which rupture occurred. Repeat the test with each of the test samples.

4.3.6 Test result

4.3.6.1 General

Use the test results to calculate the arithmetic mean values together with the standard deviation (see note) for flexural deformation strength at rupture (see 4.3.6.2), flexural deformation at rupture (see 4.3.6.3) and flexural deformation strength at 30 % deformation (see 4.3.6.4).

NOTE The estimated standard deviation (σ) is given by Formula (3):

$$\sigma = \sqrt{\frac{\sum\left(x-\bar{x}\right)^2}{\left(n-1\right)}} \tag{3}$$

where

x is the value of a single result;

$\bar{x}$ is the arithmetic mean ($\sum x/n$);

n is the number of measurements/samples in the data set.

4.3.6.2 Flexural deformation strength at rupture

The flexural deformation strength at rupture is the load, in grams, indicated at the moment of rupture during the test.

4.3.6.3 Flexural deformation at rupture

Knowing the time of rupture and the rate of loading at this time, calculate the distance (d) between the contact faces when rupture occurred. Express the flexural deformation as a percentage of the total initial diameter (D_{T}) of the sample as shown in Formula (4):

$$100 \times \left[1 - \frac{d}{D_{\mathrm{T}}}\right] \tag{4}$$

4.3.6.4 Flexural deformation strength at 30 % deformation

Knowing the rate of loading, calculate the time when the total diameter of the sample has decreased by 30 % (see example) and determine the load, in grams, that was being applied at that time. The load can also be derived from the flexural load-deformation curve.

EXAMPLE

Total diameter of the contact lens is 9,6 mm.

Velocity of the moving contact face is 20 cm/min (3,33 mm/s).

30 % deformation = 2,9 mm.

Time taken for the moving contact face to cover 2,9 mm is 0,865 s.

The value needed is the load applied 0,865 s from the start of deformation.

4.3.6.5 Test report

The test report shall conform to that in Clause 5.

4.4 Oxygen permeability

4.4.1 General

Two standardized methods are used for the determination of oxygen permeability of contact lens materials. They are the polarographic method specified in 4.4.3 and the coulometric method is specified in Annex A. Common elements of both methods are detailed in 4.4.2. Calibration of both methods is given in 4.4.4 and the reporting of results is covered in 4.4.5.

There are other techniques of measuring oxygen permeability and variations upon the standardized methods that may be used if shown to give results after calibration equivalent to those derived from a standardized method.

Oxygen permeability of a material is determined from preliminary measurements of the oxygen transmissibility of several samples of the material in the form of contact lenses. However, there are certain errors in the measurement of transmissibility that can be effectively reduced to insignificance (corrected) when oxygen permeability of the material is derived. It is, therefore, practical and convenient to first cover the derivation of oxygen permeability values corrected for these errors, from preliminary (uncorrected) oxygen transmissibility measurements. The corrected permeability values are then calibrated. Corrected and calibrated oxygen transmissibility values may then be computed from the corrected and normalized permeability values.

Annex C provides information on the calculation of oxygen permeability of hydrogel lenses based on water content.

4.4.2 Common elements of the methods

4.4.2.1 Parameters

Important parameters relevant to the measurement and derivation of oxygen permeability are oxygen flux, oxygen permeability, oxygen transmissibility, thickness (i.e. radial thickness) and mean central thickness. Refer to ISO 18369-1 for the definitions of these terms.

In terms of measurement using the coulometric method, j is equal to the rate of oxygen flow past the coulometric oxygen sensor (µl O_2/s) divided by the area of sample (A) through which the oxygen has passed. With the polarographic method, j is the difference between the measured and dark currents, multiplied by the constant cited in Formula (5) and divided by the central cathode area.

The thickness (t) is the local radial thickness at the point of measurement or the mean central thickness over the measurement area. Having measured the centre thickness and by knowing the refractive index, back surface curvature and refractive power of a particular lens, the mean central thickness can also be calculated. Unless otherwise indicated, t should be stated in centimetres (cm).

In terms of measurement using the coulometric method, Dk is equal to the measured oxygen transmissibility (Dk/t) multiplied by the mean central thickness (t). With the polarographic method, oxygen permeability is corrected by adjustment of the value taken for the area exposed to oxygen flow, and by taking the slope of a line $1/Dk$ derived from plotting measured oxygen resistance t/Dk against thickness (t). Oxygen permeability is a physical property of the material and is not a function of the shape or thickness of the material sample.

In terms of measurement using the coulometric method, Dk/t is equal to the oxygen flux (j) divided by the difference in oxygen tension (partial pressure of oxygen) between atmospheres at the two exposed surfaces of the sample contact lens. With the polarographic method, oxygen transmissibility is the oxygen permeability corrected for edge and barrier layer effects (Dk) divided by thickness (t). Oxygen transmissibility is a property of the lens material and lens thickness and, therefore, depends on the design of the contact lens.

4.4.2.2 Test samples

The oxygen permeability of hydrogel and non-hydrogel flexible materials in the form of finished contact lenses incorporating various powers and designs can be determined using this document. The oxygen permeability of hydrogel or non-hydrogel materials in the form of standardized test samples can also be determined. Test samples with opposing surfaces that are nearly parallel are preferred (see 4.4.3.7.1).

If the aim of investigation is to determine oxygen permeability through the measurement of preliminary transmissibilities of finished contact lenses, the mean thickness within the central area of a contact lens exposed to oxygen flow (see 4.4.2.1) should be included in the test report (see 4.4.5). This thickness, however, is not a factor in the derivation of preliminary oxygen transmissibility (Dk/t) from oxygen flux measurements [see Formula (5) and Formula (10)].

The back optic zone radii shall be known. The back and front optic zone diameters shall be greater than the chord diameter ($2h$) of the central lens area tested for gas exchange. Test samples shall be clean and polished to the quality acceptable in normal contact lens production for human use.

In the case of hydrogel materials, the test specimens should be stored in standard saline solution (see ISO 18369-3:2017, 4.9) and should be equilibrated (see ISO 18369-1:2017, 3.1.1.21, for definition) at eye temperature (35 °C ± 1,0 °C). The conditions of equilibration are described in the test report.

4.4.3 Polarographic method

4.4.3.1 General

The determination of oxygen permeability of hydrogel and non-hydrogel, rigid and flexible contact lens materials, using a polarographic oxygen sensor is described. The procedure specifies how measurements are taken and establishes the conditions under which measurements are made.

The polarographic method is applicable to the determination of the corrected oxygen permeability (Dk) of rigid, hydrogel and non-hydrogel flexible materials in the form of contact lenses, incorporating various refractive powers and rotationally symmetric lens geometries, and corrected oxygen permeability (Dk) of hydrogel, non-hydrogel flexible materials, and rigid contact lens materials in the form of standardized test samples.

4.4.3.2 Principle

The polarographic method directly measures the number of oxygen molecules diffusing through a test material by electrochemically removing the molecules from solution after they pass through the material. After a molecule of oxygen emerges from the sample material, it contacts the centre electrode (cathode) of the oxygen sensor, placed against the back surface of the sample, and is instantaneously involved in a chemical reaction that yields four hydroxyl ions. This production of ions constitutes the electric current which is quantified by the apparatus, and which is proportional to the number of molecules removed. The measured current is used to calculate the preliminary (uncorrected) oxygen transmissibility, $(Dk/t)_{\text{preliminary}}$, expressed as ml O_2/(A·s), through the material as in Formula (5):

$$\left(\frac{Dk}{t}\right)_{\text{preliminary}} = \frac{(I - I_{\text{d}})}{p_{\text{A}} \times A} \times 5{,}804 \times 10^{-2} \tag{5}$$

where

p_{A} is the barometric pressure less the water vapour pressure, expressed in mmHg, multiplied by the oxygen fraction in the oxygenated gas (e.g. 0,209 for air);

I is the steady state current, in amperes, from the oxygen sensor (100 % conversion efficiency is assumed);

A is the area, in cm^2, of the cathode face in the oxygen sensor;

I_{d} is the "dark current", in amperes, of the oxygen sensor (i.e. the current that flows in the absence of oxygen flux);

$5{,}804 \times 10^{-2}$ is the product of the volume of one kilogram mole at standard conditions of temperature and pressure (STP) divided by Faraday's constant divided by the number of charges per molecule of oxygen reduced, assuming four charges per molecule.

In order to derive the oxygen permeability of lenses made of a particular material, correction shall be made for edge effects (see 4.4.3.3) and boundary-layer effects (see 4.4.3.4). Only then may the corrected oxygen transmissibility of a contact lens made from the material be calculated.

4.4.3.3 Correction for edge effects

An artifact common to diffusion-type methods, termed the "edge effect," shall be accounted for. This effect occurs whenever the front and back diffusion areas through which oxygen passes are not equal and aligned. In the polarographic method, the oxygen that eventually contacts the cathode at the back of the lens, funnels to the cathode from a larger frontal area of the lens sample than represented by the cathode area at the back of the sample. In effect, A in Formula (5) has been underestimated and oxygen flux is no longer a simple linear function of cathode area as noted in the formula. However, if the cathode diameter is at least ×10 larger than the sample thickness, a comparatively simple numerical procedure applied to the preliminary Dk/t values will correct for the edge effect, within the accuracy necessary for this document.

As it will be necessary to have the reciprocal of transmissibility in 4.4.3.4, the edge effect correction will be applied to the preliminary reciprocal (t/Dk, or resistance) values. Each preliminary t/Dk value shall be corrected by using the appropriate formula given below. In Formula (6) to Formula (9), t and the cathode diameter (D_{cathode}) are expressed in millimetres, and for convenience, Dk values are stated in "Dk units", i.e. in units of 10^{-11} multiplied by 10^{11} before use in the formulae.

For hydrogels, tested with a spherical cathode, the corrected t/Dk, $(t/Dk)_{\text{corrected}}$, is given by Formula (6):

$$\left(\frac{t}{Dk}\right)_{\text{corrected}}=\left(\frac{t}{Dk}\right)_{\text{preliminary}}\times\left[1+\frac{(2{,}35\times t)}{D_{\text{cathode}}}\right] \tag{6}$$

For hydrogels, tested with a flat cathode, the corrected t/Dk, $(t/Dk)_{\text{corrected}}$, is given by Formula (7):

$$\left(\frac{t}{Dk}\right)_{\text{corrected}}=\left(\frac{t}{Dk}\right)_{\text{preliminary}}\times\left[1+\frac{(1{,}89\times t)}{D_{\text{cathode}}}\right] \tag{7}$$

For non-hydrogels, tested with spherical cathode the corrected t/Dk, $(t/Dk)_{\text{corrected}}$, is given by Formula (8):

$$\left(\frac{t}{Dk}\right)_{\text{corrected}}=\left(\frac{t}{Dk}\right)_{\text{preliminary}}\times\left[1{,}017\,25+\frac{\left[0{,}587-0{,}001\,93(Dk)\right]\times 4t}{D_{\text{cathode}}}\right] \tag{8}$$

For non-hydrogels, tested with flat cathode, the corrected t/Dk, $(t/Dk)_{\text{corrected}}$, is given by Formula (9):

$$\left(\frac{t}{Dk}\right)_{\text{corrected}}=\left(\frac{t}{Dk}\right)_{\text{preliminary}}\times\left[1{,}015\,75+\frac{\left[0{,}471-0{,}001\,93(Dk)\right]\times 4t}{D_{\text{cathode}}}\right] \tag{9}$$

The correction factors for non-hydrogels contain the value of Dk, which at this stage is unknown. The procedure to overcome this difficulty is to calculate Dk using the values of t/Dk (uncorrected for boundary-layer effect) in such a way as to find values close enough to the true values to use in the corrective formulas for edge effect. This can be done by use of the following regression formula, Formula (10), or by employing suitable statistical software:

$$Dk=\frac{\left(i\sum t_i^{\,2}\right)-\dfrac{\left(i\sum t_i\right)^2}{n}}{\left\{i\sum t_i\,(t/Dk)_i-\dfrac{\left(\sum t_i\right)\left[i\sum\left(\dfrac{t}{Dk}\right)_i\right]}{n}\right\}} \tag{10}$$

where $i\sum$ is a summation over the n different measurements, each measurement having a value of thickness (t_i) and value of resistance $(t/Dk)_i$.

The value of Dk found by using Formula (5) is close enough to be used in the corrective formulae for the edge effect, using Formula (6) to Formula (9). Then, Dk is calculated again in 4.4.3.4 using the values of t/Dk corrected for the edge effect. This value of Dk will be the final outcome corrected for edge and boundary-layer effects.

4.4.3.4 Correction for boundary layer effects

For each material tested, preliminary Dk/t values are determined for at least four lenses substantially differing with respect to thickness. The different thickness values may be obtained by stacking parallel or near-parallel lenses.

Stacking lenses to simulate thicker samples is not ideal. It should only be used when monolithic samples are not available. An acknowledgement of the use of stacked lenses to determine the permeability should be included with the results.

When the reciprocal transmissibilities (t/Dk) are plotted against thickness (t), the slope of the least squares regression line is the inverse of oxygen permeability ($1/Dk$) corrected for boundary-layer effects[7].

This somewhat involved procedure is necessary to eliminate experimental artifacts due to stagnant boundary layers of liquid present at the front and/or rear contact lens surfaces, as well as for the damp filter paper "aqueous bridge" which separates a non-hydrogel lens from the cathode of the oxygen sensor. These layers act as constant and significant barriers to the flow of oxygen in addition to that of the test sample. The resistance of these films adds to the resistance of the sample to result in the preliminary Dk/t value calculated from the sensor's current and corrected for the edge effect. However, if the apparatus is correctly made and the proper procedure is followed, these boundary layer resistances have a zero rate of change with sample thickness. Thus, their effects on the calculated permeability value, Dk, are eliminated by the statistical reciprocal slope technique noted above.

4.4.3.5 Reagents and materials

4.4.3.5.1 Oxygenated gas, consisting of a mixture of oxygen gas and nitrogen or other gases. Air from the ambient environment can be used. The gas shall be humidified to the point that it is water vapour-saturated when it contacts the front surface of a test specimen. Other gas mixtures can be used with appropriate rationale and the partial pressure in Formula (5) adjusted accordingly.

4.4.3.5.2 Filter paper, saturated in standard saline is necessary as an "aqueous bridge" between the back surface of non-hydrogel test specimens and the front surface of the oxygen sensor. The layer of filter paper shall be of the same type and thickness between measurements and shall be consistently saturated. Typically, a suitable form of cigarette paper has been used but other forms of filter paper should also suffice.

NOTE Lenses that produce atypical equilibration responses may require an aqueous bridge.

4.4.3.6 Apparatus

An oxygen transmission apparatus, equivalent to the Rehder single-chamber system[1] diagrammed in Figure 5 and Figure 6, consists of a clamping device, retaining O-ring, nylon mesh, filter paper, heated box, thermistor, polarographic cell, ohmmeter and micro-ammeter. The test specimen is firmly held against the sensor cathode by the clamping device, which presses the nylon mesh in firm contact with the front surface of the test specimen. The retaining O-ring holds the nylon mesh in place. So oriented when clamped, a hydrogel test specimen is sandwiched between the polarographic cell and the nylon mesh (see Figure 4 and Figure 5).

The polarographic cell body, which holds the electrodes and thermistor, is made of an oxygen impermeable polymer. The polarographic sensor's cathode is platinum or 24-carat gold and 4 mm in diameter. The silver anode (W 99,8 % purity) is annular, surrounding the cathode, and is separated from the cathode by the oxygen impermeable polymer. A small hole is drilled in the anode in which the thermistor is embedded. The face of the sensor is lathed and polished using processes identical to that used for contact lens surfaces. The sensor face shall be spherical and convex (typically having a radius of 7,5 mm to 9,0 mm to match the curvature of the sample lenses) when used for testing of rigid, hydrogel and non-hydrogel contact lenses. A sensor of similar design, but having a flat face, may be used for testing of flat samples and hydrogel contact lenses.

The polarographic cell assembly is removable, allowing for substitution of alternate cathodes having flat or convex faces. The cathode assembly is made of non-gas permeable plastic with the cathode embedded at the centre line. The sample is held against the cathode face by a nylon mesh retained by the O-ring.

The electronics associated with the apparatus consist of a digital voltmeter to read the thermistor, a micro-ammeter to measure the oxygen flux current and the necessary circuitry to maintain a constant voltage between the cathode and anode of the oxygen sensor.

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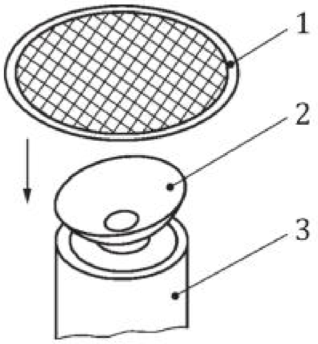
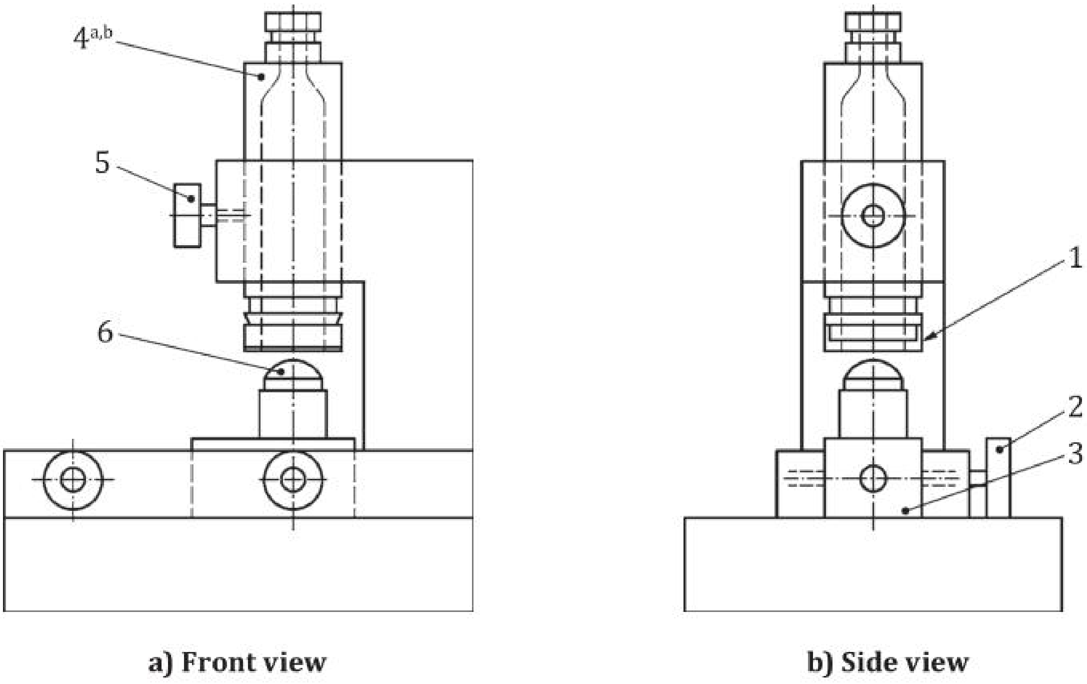
[1] The Rehder single-chamber system is the trade name of a product supplied by Crea Tech/Rehder Development Co., 48454 Harbor Drive, New Baltimore, Michigan 48047, USA. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used, if they can be shown to lead to the same results.



Key

1 ring with mesh
2 hydrogel lens
3 flat polarographic cell

Figure 4 — Hydrogel sample between sensor and retaining mesh



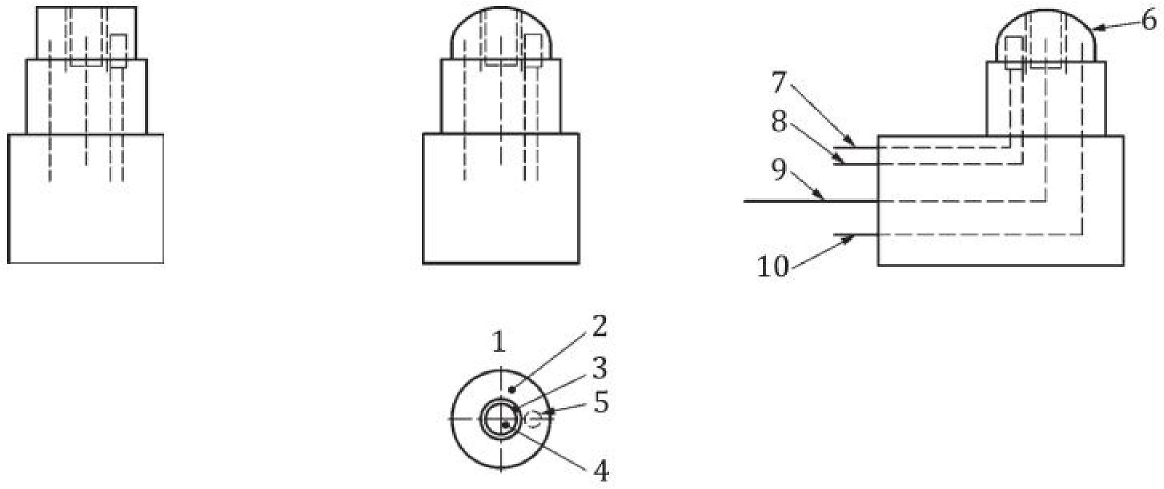
a) Front view

b) Side view

Key

1 nylon mesh stretched over end of sleeve to hold sample onto cell
2 polarographic cell clamp screws
3 cell fits between rails
4 movable sleeve
5 sleeve clamp screw
6 polarographic cell
[a] Small end of movable sleeve for holding flat samples.
[b] Large end of movable sleeve for holding curved samples.

Figure 5 — Clamping mechanism for polarographic measurement



a) Front view; flat cell **b) Front view; radius cell** **c) Side view**

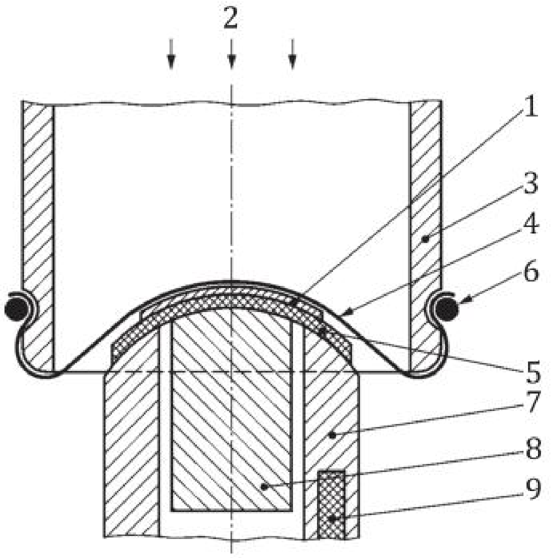
Key

1	top view radius or flat cell
2	silver anode
3	insulation
4	gold or platinum cathode
5	temperature sensor
6	curvature to match base curve of sample
7	connection to temperature readout
8	temperature sensor
9	cathode
10	anode

Figure 6 — Polarographic cell body

The entire clamped sensor with sample system is then placed in a heated box and held at 35 °C ± 1 °C. Within the box is a reservoir of water that maintains a water vapour-saturated atmosphere at the exposed surface of the test specimen.

In the case of a non-hydrogel rigid test specimen, the back surface of the polymer shall closely match the curvature of the face of the sensor and cathode. A layer of saline-saturated paper (thin filter paper or cigarette paper) is added to the system prior to clamping. The filter paper is placed between the back surface of the non-hydrogel test specimen and the oxygen sensor, so as to provide the previously described "aqueous bridge," necessary for proper function of the polarographic sensor (see Figure 7).



Key

1	contact lens
2	oxygen flow
3	movable sleeve
4	nylon mesh to hold sample onto cell
5	saline saturated filter paper
6	O-ring
7	silver anode
8	gold or platinum cathode
9	thermistor

Figure 7 — Polarographic cell showing non-hydrogel sample between sensor and retaining mesh

4.4.3.7 Procedure

4.4.3.7.1 Thickness of samples

Measure the thickness of parallel or near-parallel samples according to ISO 18369-3:2017, 4.5. In general, commercial lenses with optical power between −1,00 D and plano can be considered to be near-parallel surface samples.

NOTE 1 Lens powers outside this range are likely to give a reduction in measurement accuracy.

Determine the harmonic mean central thickness (t_{HM}) of powered samples.

For a rotationally symmetric contact lens, this can be calculated from a series of (h + 1) radial thickness measurements at intervals of equal annular area from the centre point (point 0) to the edge point (point h) of the circular zone by Formula (11):

$$t_{\mathrm{HM}} = \frac{h+1}{\frac{1}{t_0}+\frac{1}{t_1}+\frac{1}{t_2}+\frac{1}{t_3}+\ldots+\frac{1}{t_h}} \qquad (11)$$

where

h	is a series of concentric circles indicating zones of equal surface area from the lens geometric centre to the edge of the exposed sample area;
t_{HM}	is the harmonic mean thickness of a radially symmetric test sample;
$t_{0\text{ to }h}$	are the radial thicknesses measured at intervals of equal area from the centre (t_0) to the edge (t_h) of the exposed sample area.

NOTE 2 The number of zones is equal to $h + 1$.

When the appropriate geometric and optical properties of the lens are known, these properties can be used to calculate mean central thickness, in millimetres, as defined in Formula (12)[2]:

$$\frac{1}{t_{\mathrm{HM}}}=\frac{-4{,}606(n-1)}{d^2F\times10^{-3}}\log_{10}\left[1-\left(d^2F\times10^{-3}\right)/2(n-1)t_{\mathrm{C}}\right] \quad (12)$$

where

n	is the refractive index;
F	is the lens power, in dioptres;
$2d$	is the diameter of the central zone, in millimetres;
t_{C}	is the centre thickness, in millimetres.

Alternately, the average central thickness can be determined by appropriately placed measurements of the local thickness across the central zone.

4.4.3.7.2 Determination of the dark current, I_{d}

This procedure establishes the reference current for a material of zero oxygen permeability. Allow the ammeter to measure the equilibrium current after a barrier to oxygen has been used instead of a test sample. The barrier may be the combination of two PMMA contact lenses with an aluminium foil disc between them. The necessary characteristics of the barrier are that it shall

- transport no oxygen,
- fit tightly against the oxygen sensor so that oxygen cannot pass around its edge and under it to the cathode, and
- be an electrical non-conductor. As the cathode receives no input of oxygen molecules through or around this barrier, the small equilibrium current realized is the dark current.

An alternative procedure for measuring the dark current is to place a hydrogel lens on the sensor and condition the lens in a water-saturated, oxygen-free atmosphere until an equilibrium current is established.

4.4.3.7.3 Measurement of equilibrium current

Bring the test equipment to a stable temperature of 35 °C ± 1 °C and remove the test sample from its storage condition. If the sample is made of a material that does not incorporate water into its molecular structure, place a piece of saturated filter paper on the surface of the lower cell and place the sample on top of it. If the material incorporates water into its structure, the filter paper is not necessary. Firmly clamp the sample to the surface of the cathode, trapping the sample between the cathode and the nylon mesh retained by the O-ring.

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[2] Fatt and Ruben, 1994. See Reference [9].

An equilibrium condition shall be reached before proceeding. Next, allow the current reading to stabilize at 35 °C and record the measurement. This is the equilibrium current to be used in the calculation of preliminary Dk/t.

Remove the sample and repeat the equilibrium current measurement for a total of at least four samples of each test material differing in terms of thickness from as low as, perhaps, 0,04 mm, to as high as, perhaps, 0,40 mm. Accuracy can be improved by making at least four independent measurements on each test sample and by increasing the number of test samples. Make independent measurements by removing a sample from the apparatus, re-equilibrating the sample in saline and repeating the measurement of equilibrium current.

NOTE Equilibrium is typically reached in 30 min or less but can potentially take up to 2 h.

4.4.3.8 Analysis of data

The preliminary Dk/t for each test sample is calculated as noted in 4.4.3.2. The area of the cathode in the case of a flat cathode face is given by Formula (13):

$$A = \pi h^2 \tag{13}$$

and of a spherical cathode face, by Formula (14):

$$A = 2\pi r\left[r - \sqrt{\left(r^2 - h^2\right)}\right] \tag{14}$$

where

$2h$ is the chord diameter of cathode;

r is the radius of curvature of the cathode face;

A is the area of the cathode face.

The reciprocal of the preliminary transmissibility for each test sample (t/Dk, or oxygen resistance) is obtained and the appropriate edge correction formula from 4.4.3.3 is applied. Using the edge-corrected t/Dk values for samples made from the same contact lens material, the boundary-layer effects are corrected as shown in 4.4.3.4. The plot of mean t/Dk against t shall approximate a linear relationship in order that a valid Dk value be determined. Multiple measurements per material thickness may increase the ability to obtain the required linear relationship[3][6][7] attainable up to a Dk at least 190×10^{-11} (cm^2/s) [ml O_2/(ml · mmHg)]. The corrected oxygen permeability (Dk) is the result for each test material that shall finally be normalized as noted in 4.4.4.

4.4.4 Normalization of the corrected oxygen permeability using reference lenses

Normalization shall be carried out using at least four contact lenses of each of the selected reference materials, in various thicknesses, having known permeability. The reference lenses shall be designed in a manner identical to that of standardized test samples detailed in 4.4.2.2. Oxygen transmissibilities (Dk/t) of the reference lenses and the oxygen permeabilities (Dk) of the reference materials shall be spaced so as to determine the measuring accuracy over an appropriate range of measurement. The reference Dk of the materials shall be known to ±5 %.

The preliminary Dk/t of each reference lens shall be measured at least four times and the arithmetical mean for each lens shall be calculated. Corrections for the edge and boundary-layer effects shall be applied, when appropriate. The t/Dk against t plot shall approximate a linear relationship. The relationship between measured corrected Dk and the reference Dk of the reference materials shall be used to construct a final standard curve, if applicable. Examples of normalization are shown in References [3], [6] and [7].

The reproducibility of normalized, corrected *Dk* and *Dk*/*t* values using the polarographic, coulometric or other methods should be 10 % or less.

NOTE A series of seven rigid non-hydrogel contact lens reference materials can be obtained from the custodian of the Oxygen Permeability Reference Material Repository, Dr. William J. Benjamin, 937 Tulip Poplar Lane, Hoover, Alabama, USA, 35244. The materials are available in the form of contact lens buttons from single lots, and can be lathed and polished by the user to the appropriate specifications as reference samples for the polarographic and coulometric methods. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named.

4.4.5 Test report

In addition to those items noted in Clause 5, the following items need to be specified in the test report:

a) an identification of the test samples, to include their back optic zone radii and diameters, refractive powers, thicknesses within the exposed central area, and chord diameter (2*h*) of the cathode (polarographic method) or circular area of sample exposed to oxygen flow during testing (coulometric method);

b) the barometric pressure at the time and location of testing and the percentage of oxygen used in the oxygenated test gas;

c) the mean oxygen permeability (*Dk*) of the sample material and the mean oxygen transmissibility (*Dk*/*t*) of the test sample, as corrected by use of the final calibration curve noted in 4.4.4 (at least four sequences shall be performed per sample);

d) the number of test sequences performed with the test sample to establish the mean current (polarographic method) or mean oxygen flow rate (coulometric method) upon which the calculation of *Dk* and *Dk*/*t* and standard deviation depend. At least four sequences shall be performed per sample and the R-squared term from the regression analysis should be included;

e) the method used (polarographic or coulometric) and the method of calibration;

f) the date of test and any deviation, by agreement or otherwise, from the test procedures specified in this document;

g) the institution, agency and/or person conducting the procedure and responsible for accuracy and reliability of the data.

4.5 Refractive index

4.5.1 General

The refractive index of contact lens material shall be measured at 589 nm (sodium D-line) or 546,1 nm (mercury e-line). Several measurement techniques can be used depending on the shape and measurement condition (e.g. dry, blotted or immersed) of the contact lens.

4.5.2 Abbe refractometer

The refractive index is determined by measuring the critical angle of incidence for total internal reflection of light of wavelength 546,1 nm (mercury e-line) or 589 nm (sodium D line) using a calibrated Abbe refractometer or an equivalent refractometer at room temperature. Light passes from the prism surface of the refractometer into the contact lens material. The critical angle is related by Snell's Law to the refractive index of the flat specimen tested and of the transparent flat reference surface of the refractometer, upon which the test specimen is placed during measurement and is calculated using Formula (15):

$$n = \frac{n' \sin(\alpha)}{\sin(90^\circ)} \tag{15}$$

where

n is the refractive index of the test specimen;

n' is the refractive index of the reference surface;

α is the critical angle of incidence upon the reference surface.

It is necessary to use a contacting fluid between the contact lens material and the refractometer prism for all materials except hydrogels. The contacting fluid shall have a refractive index greater than that of the test specimen and shall not be harmful to the test material or to the refractometer prism assembly. The refractometer shall have a precision of at least 0,000 5. The repeatability of the method is ±0,001 units of refractive index.

As the refractive index of contact lens materials generally does not exceed 1,55, a contacting fluid with a refractive index of at least 1,55 is sufficient. Organic solvents should not be used. Aqueous solutions, such as saturated zinc bromide (n_d = 1,564, density ρ = 2,510 g/ml), have been found to be suitable.

The use of a prism coupling device for measuring refractive index can provide enhanced flexibility of contact lens geometry and measurement condition. See Annex D.

4.5.3 Test samples

Test samples shall be flat or capable of being flattened against the reference surface of the refractometer during measurement. The test surface placed against the reference surface shall be polished to a smooth finish. Hydrogel specimens shall be equilibrated (see ISO 18369-1:2017, 3.1.1.21, for definition) at a temperature of 20,0 °C ± 0,5 °C in standard saline solution (see ISO 18369-3:2017, 4.9) or packaging solution , if used, to support labelling.

The hydrogel test specimens may be taken out of the saline and placed directly onto the prism without blotting. The shadow line of the saline and the shadow line of the hydrogel will be observed.

4.5.4 Procedure

4.5.4.1 Preparation of test specimen

4.5.4.1.1 General

With tinted materials, the light transmitted might be insufficient to enable a sharp image to be formed. If this occurs and if the material was not tinted before polymerization, the determination should be carried out with a specimen of the same material which has not been tinted.

4.5.4.1.2 Rigid and non-hydrogel materials

Prepare a test specimen of such a size as will fit on the face of the fixed half of the refractometer prism, having one surface optically flat and polished (the measurement face). If it is necessary to bring light into the system through an edge of the specimen, ensure that this edge is optically flat and is polished perpendicular to the measurement face.

NOTE 1 Specimens of soft non-hydrogel materials, such as silicones, can consist of actual contact lenses.

NOTE 2 A convenient shape for the test specimen is a rectangle of thickness 0,5 mm to 3,0 mm, which is slightly smaller than the face of the refractometer prism.

4.5.4.1.3 Hydrogel materials

Check that the surface of the test specimen in contact with the prism (the measurement face) is smooth and that the thickness of the specimen is approximately constant. Stabilize the state of hydration of the specimen by equilibrating (see ISO 18369-1:2017, 3.1.1.21, for definition) it at 20,0 °C ± 0,5 °C in standard saline solution (see ISO 18369-3:2017, 4.9).

NOTE Contact lenses of approximately constant thickness provide the most convenient form of test specimen for hydrogel materials.

4.5.4.2 Measurement

4.5.4.2.1 Rigid and non-hydrogel materials

Ensure that both the specimen and refractometer are at a temperature of 20 °C to 25 °C. Place a drop of contacting fluid on the fixed refractometer prism and firmly press the specimen against the prism with the polished edge, if used, towards the light source. The temperature of the prism assembly used to measure the refractive index shall be 20,0 °C ± 0,5 °C.

NOTE Satisfactory contact between the test specimen and the prism is indicated by a sharp and straight dividing line appearing between the light and dark portions of the field of view.

For direct-reading non-compensated refractometers, read the refractive index (or solids content) directly at the dividing line between the light and dark field. For refractometers with an external scale, read the refractive index after adjusting the index arm of the refractometer, so that the dividing line between the light and dark fields is coincident with the eyepiece hairline. If the refractometer has chromatic compensating prisms, adjust these to remove all colour from the field before the refractive index is read.

4.5.4.2.2 Hydrogel materials

With the refractometer at a temperature of 20 °C to 25 °C, remove the specimen from its hydrating solution, prepare as described in 4.5.4.1 and immediately press the measurement face firmly against the refractometer fixed prism. Measure the refractive index as described in 4.5.4.2. The temperature of the prism assembly used to measure the refractive index of the hydrogel shall be 20,0 °C ± 0,5 °C.

NOTE Satisfactory contact between the test specimen and the prism is indicated by a sharp and straight dividing line appearing between the light and dark portions of the field of view. If the specimen is not pressed sufficiently firmly against the prism, a faint secondary line may be seen somewhat removed from the sharp line. This is caused by the hydrating fluid and will give a refractive index which can be verified by measuring the hydrating fluid independently.

4.5.4.2.3 Anisotropic materials

If the specimen is known to have different refractive indices in different areas or if it is desired to check for this possibility, mask the measurement surface of the specimen so that the refractive index can be measured in selected areas.

4.5.5 Expression of test results

Take a minimum of three independent measurements. The test result shall be given as the average, standard deviation and sample size. If the refractometer scale is marked in percent solids or if the refractometer is calibrated at a wavelength other than the chosen reference wavelength, correct the reading to give the refractive index at the reference wavelength.

4.5.6 Test report

The test report shall conform to that in Clause 5.

4.6 Water content

4.6.1 General

4.6.2 specifies a gravimetric method for determination of water content and also a refractive index method (see Annex B).

Water content is the amount of water, expressed as a mass fraction in percent (w_{water}) present in a hydrated material that has been fully equilibrated in standard saline at room temperature as given by Formula (16):

$$W_{\text{water}} = \frac{m_{\text{water, hydrated material}}}{m_{\text{hydrated material}}} \times 100 \tag{16}$$

where

$m_{\text{water, hydrated material}}$ is the mass of water in the hydrated material;

$m_{\text{hydrated material}}$ is the mass of the hydrated material.

In this context, dissolved solutes such as sodium chloride and buffers contribute to the mass of the hydrated material.

4.6.2 Gravimetric determination of water content/absorption by loss on drying using an oven

4.6.2.1 General

Using gravimetric method, water content (ω_{water}) is calculated after measurement of the dry mass and hydrated mass of hydrogel and non-hydrogel test specimens at room temperature using Formula (17):

$$W_{\text{water}} = \frac{m_{\text{hydrated}} - m_{\text{dry}}}{m_{\text{hydrated}}} \times 100 \tag{17}$$

where

m_{hydrated} is the mass of the hydrated hydrogel and non-hydrogel test specimens;

m_{dry} is the mass of the dry hydrogel and non-hydrogel test specimens.

The method is straightforward in principle. Test specimens in their hydrated state, with excess surface water removed, are weighed on an analytical balance. The test specimens are then dried in a conventional or vacuum oven and weighed again. The difference is the mass of water lost by evaporation.

4.6.2.2 Removal of excess surface saline

4.6.2.2.1 General

The accuracy of the method is limited by the difficulty of reliably blotting the test specimen to remove excess saline from the surfaces before determination of the hydrated mass. Care shall be taken to remove all surface water; however, the specimens shall not be over-blotted so as to remove water from within the material. Blotting the test specimen shall be performed as quickly as possible to avoid loss of water from the test specimen by evaporation.

To minimize the errors caused by variation in removal of excess surface saline, the hydrated test specimens should weigh in the range of 100 mg to 300 mg. A ring test has shown that the determination of thick polymer discs gave a reproducibility of ±0,4 % water content, and a single lens a reproducibility of ±1,0 % water content. If the mass of the lens is less than 100 mg, more samples are required to obtain this reproducibility.

NOTE Moulded lenses can have a significantly lower weight than 100 mg. The manufacturing of significantly thicker samples might be impractical and might not represent the final lens product. Lenses that weigh less than the 100 mg value are more sensitive to blotting errors due to the increased surface area to volume ratio.

4.6.2.2.2 Dry blotting

Place the specimen on a dry, clean, lint-free, absorbent cotton or linen cloth. The cloth is folded over the specimen and the specimen is blotted lightly three times with a fingertip. The probability of over-blotting is increased with the dry blotting technique, which can lead to an underestimation of water content.

4.6.2.2.3 Wet blotting

Place the specimen on a clean, lint-free absorbent cloth which has been dampened with an appropriate amount of saline. The upper and lower surfaces of the specimen are in contact with the damp cloth and a suitable amount of pressure applied. The probability of leaving surface water on the lens is increased with the wet blotting technique, which can lead to an over-estimation of water content.

4.6.2.3 Measurement of hydrated test specimen mass

Place the hydrated test specimen on a previously dried and weighed glass slide (or suitable weighing dish) immediately after blotting. Then, weigh the slide and specimen to the nearest 0,1 mg and subtract the slide mass to obtain the hydrated specimen mass. Weighing shall be performed as quickly as possible to avoid loss of water from the test specimen by evaporation.

4.6.2.4 Oven drying of test specimens

Dry the glass slide and hydrated test specimen at 100 °C to 110 °C in a conventional oven to a constant mass (typically 16 h to 18 h). Then place the slide and dry specimen in a specimen jar half-filled with active desiccant (e.g. anhydrous calcium sulphate) and allow to cool inside the closed jar to room temperature for 30 min.

NOTE 1 Drying to constant mass is achieved when two consecutive weighings between drying do not differ by more than 0,1 mg to 0,3 mg (assuming a 100 mg to 300 mg sample).

NOTE 2 If the test material is found to degrade at the temperature of 100 °C to 110 °C, the material can be dried according to 4.2.6.

Repeat drying and weighing steps with new supplies of active desiccant until the sample reaches constant mass.

Alternatively, the lenses are dried, preferably under vacuum, at 60 °C ± 5 °C or other appropriate temperature to constant mass. The lenses are then allowed to cool to room temperature under vacuum, in a dry atmosphere or in a closed container over active desiccant before weighing.

4.6.2.5 Measurement of dry test specimen mass

Weigh the dry test specimen and previously weighed glass slide or suitable weighing dish to the nearest 0,1 mg and subtract the slide or dish weight to obtain the dry specimen mass. Weighing shall be performed as quickly as possible to avoid absorption of water from the air by the material.

4.6.2.6 Calculation of water content

Having determined the dry and hydrated masses, Formula (17) is used to calculate water content in percent.

4.6.3 Test report

The test report shall conform to that in Clause 5.

5 Test report

When any test method has been carried out in accordance with the specification detailed in this document, a test report shall be prepared and shall contain at least the following information:

a) the name of the laboratory carrying out the test;

b) all necessary details for the identification of the contact lens tested;

c) a reference to this document (i.e. ISO 18369-4:2017) and relevant subclause;

d) any deviations from the specified method;

e) the test result, including, where possible, an estimation of the error;

f) the date of test and the name of the responsible person.

A.7.4 Heating system and oxygen trap

This consists of a simple resistive heating unit, or an equivalent unit, elevating the temperature of the diffusion cell and test sample to 35 °C. A thermistor and appropriate electronic control feedback loop shall serve to monitor and maintain temperature at a constant value (±0,5 °C). An "oxygen trap" containing 3 g to 5 g of 0,5 % platinum or palladium on alumina, or its equivalent, shall essentially remove all residual oxygen from the carrier gas. The oxygen trap catalyses the elimination of oxygen molecules (O_2) by reaction with hydrogen molecules (H_2) present in the carrier gas. Such traps have specific capacities and should be replaced as capacity is met. A flow meter having an operating range of 5 ml/min to 100 ml/min shall monitor the flow rate of carrier and test gases.

A.7.5 Coulometric sensor

The coulometric instrument and sensor should be maintained as recommended by the manufacturer.

A.7.6 Coulometric sensor and recording system

An oxygen-sensitive coulometric sensor operating at an essentially constant efficiency (95 % or above) shall convert the mass of oxygen in the carrier gas, after appropriate calibration, into rate of oxygen flow past the detector (µl/s). Care shall be taken so that saline, free chlorine, oxidizing agents and carbon dioxide do not come in contact with the sensor.

A load resistor is selected, typically of 5,3 Ω to 53 Ω, across which the output voltage is measured on a voltage recorder. The recorder should be capable of reading voltages from 0,100 mV to 50 mV and have a resolution of at least 10 µV. An input impedance of 5 000 Ω or higher is acceptable. The output voltage and load resistance are used to determine the current produced from the oxygen.

A.8 Conditioning of test samples

Conditioning of test samples may be the same as 4.4.2.2 unless otherwise noted.

A.9 Procedure

A.9.1 Thickness of samples

Measure the thickness of parallel or near-parallel samples according to 4.4.2. Determine the mean central thickness of other samples with a series of such measurements as detailed in 4.4.2.

A.9.2 Preparation of the apparatus

The instrument should be set up and operated as described by the manufacturer.

A.9.3 Insertion of test sample

Remove the test sample from its conditioning environment and place its peripheral posterior surface on the annular smooth surface of the mounting rim of the posterior environmental chamber. Make the sample free of wrinkles and creases. Next, place the anterior half of the diffusion cell, such that the O-ring contacts the front surface of the test sample directly over the annular area of the sample already in contact with the posterior mounting rim. Then, clamp the two halves of the diffusion cell snugly together, enclosing the test sample within them.

A.9.4 Purging the system of oxygen

The baseline (V_0) voltage output may be measured by placing an oxygen-impermeable barrier over the lens holder and allow the system to reach an equilibrium voltage. Alternately, a shunt that allows the carrier gas to bypass the environmental chambers and flow directly to the sensor may be used to establish the baseline voltage.

A.9.5 Determination of the end voltage output

Once V_0 has been established, maintain the flow of carrier gas through the posterior environmental chamber of the diffusion cell at the same rate. Mount the lens, if it is not already in place, and initiate a similar flow of oxygenated test gas through the anterior environmental chamber. As oxygen is transmitted through the contact lens test sample and is taken to the coulometric sensor via the carrier gas, the output of the detector will increase gradually. Ultimately, the output will stabilize at a constant value, the end voltage output (V_E). The output should stabilize within approximately 15 min but may require a relatively longer time (1 h to 2 h) to stabilize to a low-end output voltage, characteristic of a contact lens of low oxygen transmissibility (thick, low *Dk*). This will be especially true immediately after having stabilized to a lens with high oxygen transmissibility. For this reason, test samples of comparable oxygen transmission qualities should be tested together.

A.9.6 Calculation of oxygen flow rate (μl O_2/s)

The rate of oxygen flow past the coulometric sensor is the critical value from which oxygen transmissibility and oxygen permeability are derived. The oxygen flow rate, q_{O_2}, can be calculated knowing the zero and end output voltages (V_0 and V_E), the resistance of the load resistor (R_L), and the calibration constant (K):

$$q_{O_2} = \frac{K\left(V_E - V_0\right)}{R_L} \tag{A.2}$$

where

K is the calibration constant;

V_E is the end voltage output;

V_0 is the zero voltage output;

R_L is the load resistance.

This calculation can be internally performed by the software of modern automated instruments. Follow the manufacturer's procedures in this case.

A.9.7 Determination of the calibration constant (*K*) using reference films

Determine V_0 and V_E in the manner described in A.9.4 and A.9.5 for a non-hydrogel standard reference material (SRM) of known oxygen transmissibility (*Dk*/*t*). Typically, SRM 1470 has been used, as it is available from the National Institute of Standards and Technology (NIST) in the USA. *Dk*/*t* for SRM 1470 is 0,072 ± 0,004 5 *Dk*/*t* units. The rate of oxygen flow (μl O_2/s) can be derived from Formula (A.1), knowing the exposed area (A) of the reference sample and the partial pressure of oxygen in the anterior environmental chamber. (p_A) is approximately 207 hPa (155 mmHg) but depends on barometric pressure at the time and location of measurement [see Formula (A.1)]. Next, calculate the calibration constant (K) with the use of Formula (A.2), knowing R_L and having measured V_0 and V_E.

It is best to calibrate the apparatus several times with different samples of the standard reference material so as to establish a tight confidence interval for the calibration constant (K). A significant drift in the value of K over time could be indicative of a reduction in efficiency of the coulometric sensor.

This determination can be internally performed by the software of modern automated instruments using specifically designed, NIST traceable test specimens. Follow the manufacturer's procedures in this case.

A.9.8 Shutdown procedures

At the conclusion of testing, but when it is expected that other tests will be performed soon, place the instrument in standby condition by

a) bypassing the coulometric sensor,

b) shutting off the test gas and rerouting carrier gas to the anterior diffusion cell chamber, as well as continuing carrier gas flow to the posterior chamber, and

c) reducing carrier gas flow rate to less than 5 ml/min.

It is important to avoid back diffusion of air into the system. Therefore, care should be taken to ensure a positive flow of carrier gas through the system at all times. This flow can be low (<5 ml/min) when the instrument is not in use. When it is anticipated that the apparatus will not be used for a long time, electrical power to the heating unit may be turned off.

A.10 Test report

In addition to those items noted in Clause 5, the following items need to be specified:

a) an identification of the test samples, to include their back optic zone radii and diameters, refractive powers, thicknesses within the exposed central area and chord diameter (2*h*) of the cathode (polarographic method) or circular area of sample exposed to oxygen flow during testing (coulometric method);

b) the barometric pressure at the time and location of testing and the percentage of oxygen used in the oxygenated test gas;

c) the mean oxygen permeability (*Dk*) of the sample material and the mean oxygen transmissibility (*Dk*/*t*) of the test sample, as corrected by use of the final standard curve noted in 4.4.5 (at least four sequences shall be performed per sample);

d) the number of test sequences performed with the test sample to establish the mean current (polarographic method) or mean oxygen flow rate (coulometric method) upon which the calculation of *Dk* and *Dk*/*t* and the standard deviation depend. At least four sequences shall be performed per sample and the R-squared term from the regression analysis should be included;

e) the method used (polarographic or coulometric) and the method of calibration;

f) the date of test and any deviation, by agreement or otherwise, from the test procedures specified in this document;

g) the institution, agency and/or person conducting the procedure and responsible for accuracy and reliability of the data.

Annex B
(informative)

Determination of water content by refractive index

The refractive index of a hydrogel contact lens material is a function of refractive indices of its water and solid components, relative to the proportion of the material occupied by those components according to the Principle of Gladstone and Dale. Thus, the volume fraction of water (ϕ_{water}) expressed in percent, of hydrogels can be calculated from the refractive indices of the hydrated material (n_{wet}), the dry material (n_{dry}), and standard saline solution (see ISO 18369-3:2017, 4.9) in which the material was equilibrated (n_{std}):

$$\phi_{\text{water}} = \frac{n_{\text{dry}} - n_{\text{wet}}}{n_{\text{dry}} - n_{\text{std}}} \times 100 \qquad \text{(B.1)}$$

Measure the refractive index of the hydrated hydrogel test specimen as described in 4.5. If the refractive index of the dry material is known and the index of standard saline solution is measured, the water content can be calculated using Formula (B.1). Otherwise, the refractive index of the dry material and of standard saline solution shall also be measured as in 4.5 before substitution into these formulae. The volume fraction of water (ϕ_{water}) expressed in percent, can be changed to units of mass fraction (w_{water}) expressed in percent, knowing the specific gravity of the test material.

Should the refractive index of the dry material be estimated rather than measured, the water content is also an estimate. Water content is an estimate as well, should it be calculated by mass using an estimated specific gravity for the test material. See References [4] and [7].

Annex C
(informative)

Calculation of oxygen permeability of hydrogel lenses based on water content

In a conventional (non-silicone) hydrogel contact lens (water content above 36 %), the solid phase is essentially impermeable to oxygen. Oxygen is effectively only transported through the water phase. Consequently, the oxygen permeability of a conventional hydrogel contact lens material can be predicted from its water content[4].

$$Dk = 1{,}67e^{0{,}0397 \times WC} \tag{C.1}$$

where

WC is the water content of the lens expressed in percent.

Formula (C.1) should not be used if the solid phase is expected to transmit an appreciable amount of oxygen.

n is the refractive index of the test specimen;

n' is the refractive index of the reference surface;

α is the critical angle of incidence upon the reference surface.

Once the angle of incidence (θ) equals the critical angle (α), the laser light is absorbed into the bulk material and the detector signal drops (as shown in Figure D.1). Since the prism index of refraction (n') is known, Formula (17) can be used to accurately calculate the refractive index of the bulk material. It is important that the bulk material be in intimate contact with the prism base.

D.3 Method

The temperature of the prism assembly used to measure the refractive index of the hydrogel shall be 20 °C ± 0,5 °C.

Take a minimum of three independent measurements.

D.4 Results

The test result shall be given as the average, standard deviation and sample size. If the refractometer scale is marked in percent solids or if the refractometer is calibrated at a wavelength other than the chosen reference wavelength, correct the reading to give the refractive index at the reference wavelength.

D.5 Test report

The test report shall conform to that in Clause 5.

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ICS 11.040.70

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