INTERNATIONAL STANDARD

ISO 18369-4

First edition
2006-08-15

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 18369-4:2006(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

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

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

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

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

ISO 18369-4 was prepared by Technical Committee ISO/TC 172, *Optics and photonics*, Subcommittee SC 7, *Ophthalmic optics and instruments*.

This first edition cancels and replaces ISO 9913-1:1996, ISO 9913-2:2000, ISO 9914:1995, ISO 10339:1997, ISO 10340:1995 and ISO 11984:1999, which have been technically revised.

ISO 18369 consists of the following parts, under the general title *Ophthalmic optics — Contact lenses*:

— *Part 1: Vocabulary, classification system and recommendations for labelling specifications*

— *Part 2: Tolerances*

— *Part 3: Measurement methods*

— *Part 4: Physicochemical properties of contact lens materials*

Introduction

The ISO 18369 series applies to contact lenses, which are devices worn over the front surface of the eye in contact with the preocular tear film. This part of ISO 18369 covers rigid (hard) corneal and scleral contact lenses, as well as soft contact lenses. Rigid lenses maintain their own shape unsupported and are made of transparent optical-grade plastics, such as polymethylmethacrylate (PMMA), cellulose acetate butyrate (CAB), polyacrylate/siloxane copolymers, rigid polysiloxanes (silicone resins), butylstyrenes, fluoropolymers, and fluorosiloxanes, etc. Soft contact lenses are easily deformable and require support for proper shape. A very large subset of soft contact lenses consists of transparent hydrogels containing water in concentrations greater than 10 %. Soft contact lenses can also be made of non-hydrogel materials, e.g. flexible polysiloxanes (silicone elastomers).

The ISO 18369 series is applicable to determining allowable tolerances of parameters and properties important for proper functioning of contact lenses as optical devices. The ISO 18369 includes tolerances for single vision contact lenses, bifocal lenses, lenses that alter the flux density and/or spectral composition of transmitted visible light (tinted or pigmented contact lenses, such as those with enhancing, handling, and/or opaque tints), and lenses that significantly attenuate ultraviolet radiation (UV-absorbing lenses). The ISO 18369 series of standards covers contact lenses designed with spherical, toric, and aspheric surfaces, and recommended methods for the specification of contact lenses.

Ophthalmic optics — Contact lenses —

Part 4:
Physicochemical properties of contact lens materials

1 Scope

This part of ISO 18369 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 referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 18369-1:2006, *Ophthalmic optics — Contact lenses — Part 1: Vocabulary, classification system and recommendations for labelling specifications*

ISO 18369-3:2006, *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.

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 nonhydrogel materials that have been successfully manufactured into contact lenses. In addition, Table 1 includes repeatability, test methods, and units of measure for these characteristics. If alternative methods are used, they should be so stipulated.

Table 1 — Physiochemical properties: Repeatability, test methods and units of measure

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

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

[b] Repeatability of these test results shall be established in individual laboratories according to the terms and definitions given in ISO 18369-1.

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.

4.2.3 Apparatus

A 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 shall be used. A perforated stainless steel, sintered glass, paper or equivalent extraction thimble fitted with a glass wool plug or other suitable closure shall be used. A vacuum oven or equivalent drying apparatus and an analytical balance capable of weighing to 0,1 mg are required.

4.2.4 Reagents

Distilled or deionized water complying with Grade 3 of ISO 3696:1987 shall be used. The appropriate organic solvent (see Table 2) should be analytical grade or better. Laboratory-grade boiling stones or anti-bumping granules are required along with a suitable active desiccant. Selection of the desiccant will depend upon the characteristics of the test material.

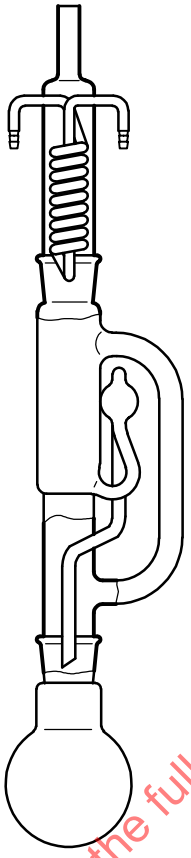


Figure 1 — Extraction apparatus

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

Material	Solvent	Corresponds to
Hydrogels	Water (distilled or deionized)	Mild extraction (simulates in-eye extraction)
	n-Hexane	Mild extraction (non-polar solvent)
	Ethanol or methanol	Extraction of majority of uncrosslinked material (but swells and may degrade material)
	Dichloromethane or chloroform	Extraction of all uncrosslinked material (but swells and is likely to degrade material)
Hard and RGP and silicone elastomers	Water (distilled or deionized)	Mild extraction (simulates in-eye extraction)
	n-Hexane	Mild extraction (non-polar solvent)
	Dichloromethane or chloroform	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 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 to constant mass. Then 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).

4.2.7 Calculation of results

The quantity of extracted material shall be expressed as a mass fraction, $w_{\text{extracted}}$, in percent of the initial dry mass [Equation (1)]:

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

where

m_1 is the mass of lenses prior to extraction;

m_2 is the mass of extracted lenses.

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.

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. Both 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 back vertex power (F'_v) 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 8,00 mm ± 0,025 mm;

— back surface: single cut, radius of curvature 7,80 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

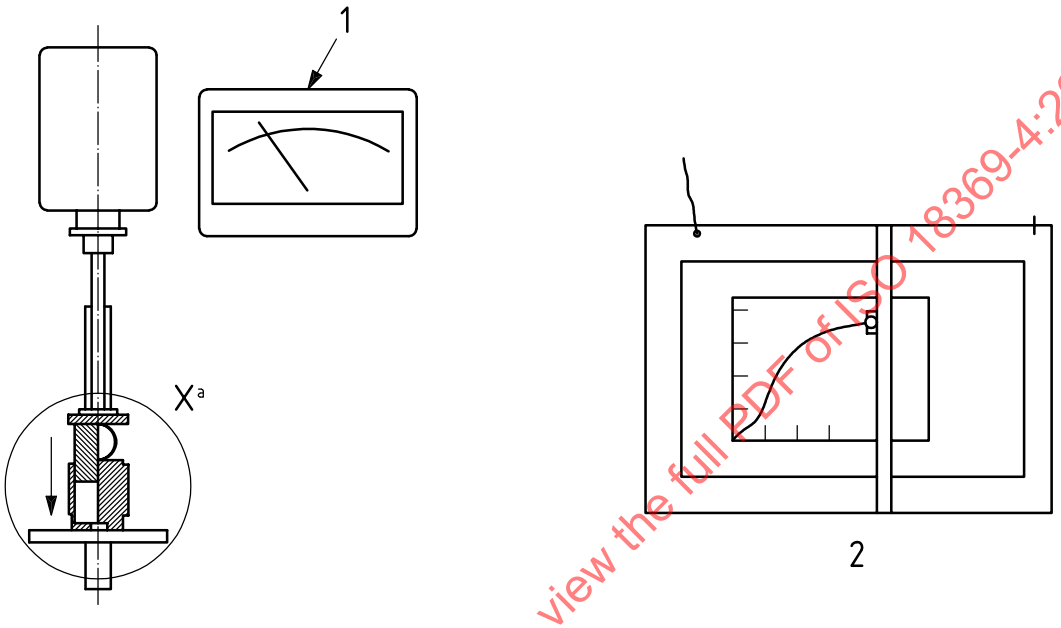
Three contact lenses from each of three different material lots (total of nine contact lenses) shall be tested where a claim is made regarding flexure or strength.

4.3.3 Preparation of samples

Samples shall be stored in standard saline solution conforming to ISO 18369-3:2006, 4.7, for at least 48 h prior to testing. The temperature of this saline solution shall be 20 °C ± 5 °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 4.3.4.2 to 4.3.4.4.



Key

1 load indicator
2 recorder

[a] See Figure 3 for detail X.

Figure 2 — Testing machine

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

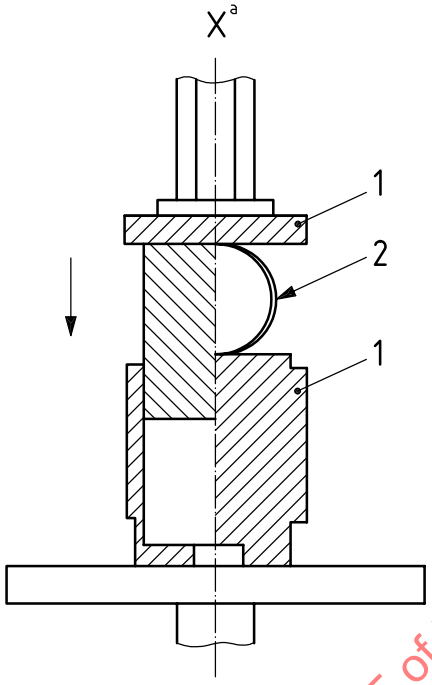
The sample is set at the centre of the upper and lower contact faces so that the whole load is applied in the plane containing the edge.

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

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

4.3.4.4 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 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 an ambient temperature of 20 °C ± 5 °C.

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

Measure the back optic zone radius, total diameter, centre thickness and 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 centreline 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

As the results of the test 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 the expression

$$\sigma = \sqrt{\left(\sum\left[x - \bar{x}\right]^2\right) / (n-1)}$$

where

x is the value of a single result;

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

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 ($\varnothing_T$) of the sample as follows:

$$100 \times \left[1 - \frac{d}{\varnothing_T}\right]$$

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.

4.3.7 Test report

The test report shall conform to that in Clause 5.

4.4 Oxygen permeability

4.4.1 General

There are two standardized methods for the determination of oxygen permeability of contact lens materials. Common elements of both methods are detailed in 4.4.2. They are the polarographic method specified in 4.4.3, which is applicable to all types of materials having an oxygen permeability from 0 to 145, and the coulometric method specified in 4.4.4, which is applicable only to non-hydrogel materials. Calibration of both

methods is given in 4.4.5, and the reporting of results is covered in 4.4.6. 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 calibrated permeability values, as described in ISO 18369-3.

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 harmonic 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, 1, 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 Equation (2) and divided by the central cathode area.

The thickness, t, is the radial thickness at the point of measurement or the harmonic 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 harmonic mean central thickness may 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 sample 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 part of ISO 18369. The oxygen permeability of hydrogel or non-hydrogel materials in the form of standardized test samples can also be determined.

If the aim of investigation is to determine oxygen permeability through the measurement of preliminary transmissibilities of finished contact lenses, the harmonic 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 4.4.6). This thickness, however, is not a factor in the derivation of preliminary oxygen transmissibility (Dk/t) from oxygen flux measurements [Equation (2) or (10)].

The back optic zone radii of test samples may be infinite, as in the case of a flat sample, or may vary within the range 7,00 mm to 9,00 mm, as in the case of contact lenses. The back and front optic zone diameters shall be greater than the chord diameter ($2h$) of the central lens area tested for gas exchange. The surfaces of

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 shall be stored in standard saline solution (see ISO 18369-3:2006, 4.7) for at least 24 h at room temperture (20 °C ± 2,0 °C) prior to testing and shall be equilibrated at eye temperature (35 °C ± 0,5 °C) for at least 2 h prior to measurement.

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 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 and non-hydrogel, rigid and flexible contact lens materials in the form of standardized test samples.

4.4.3.2 Overview of principle

The polarographic method directly measures the number of oxygen molecules diffusing through a test material by electrochemically removing the molecules from solution as soon as they pass through the material. As 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 converted to 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 (preliminary), expressed as ml O_2/(A·s), through the material as in Equation (2):

$$Dk/t \text{ (preliminary)} = \frac{(I - I_d)}{p_A \times A} \times 5{,}804 \times 10^{-2} \quad (2)$$

where

p_A is the (barometric pressure less the vapour pressure), expressed in hPa, multiplied by 0,209 which is the oxygen fraction in oxygenated gas;

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

I is the current, in amperes, from 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 as in ISO 18369-2.

4.4.3.3 Correction for edge effects

An artifact common to all diffusion-type methods, termed the "edge effect", shall be accounted for. This effect occurs whenever the cross section of material through which oxygen passes is not constant from one surface of the sample, through the thickness of material, to the back surface. 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 Equation (2) has been underestimated, and oxygen flux is no longer a simple linear function of cathode area as noted in the equation. However, if the ratio between the cathode area and the sample thickness is maintained in the correct range, as set in the specifications for the apparatus, a comparatively simple numerical procedure applied to the preliminary Dk/t values will correct for the edge effect, within the accuracy necessary for this part of ISO 18369.

As it will be necessary to have the reciprocal of transmissibility in 4.4.3.4, the edge effect correction will here 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 these next four equations, Equations (3) to (6), 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 equations:

For hydrogels, tested with a spherical cathode, the corrected t/Dk, t/Dk (corrected), is given by Equation (3):

$$t/Dk\ \text{(corrected)} = t/Dk\ \text{(preliminary)} \times \left[1 + \frac{(2{,}35 \times t)}{D_{\text{cathode}}}\right] \tag{3}$$

For hydrogels, tested with a flat cathode, the corrected t/Dk, t/Dk (corrected), is given by Equation (4):

$$t/Dk\ \text{(corrected)} = t/Dk\ \text{(preliminary)} \times \left[1 + \frac{(1{,}89 \times t)}{D_{\text{cathode}}}\right] \tag{4}$$

For non-hydrogels, tested with spherical cathode the corrected t/Dk, t/Dk (corrected), is given by Equation (5):

$$t/Dk\ \text{(corrected)} = t/Dk\ \text{(preliminary)} \times \left\{1{,}017\ 25 + \frac{\left[0{,}587 - 0{,}001\,93\left(Dk\right)\right] \times 4t}{D_{\text{cathode}}}\right\} \tag{5}$$

For non-hydrogels, tested with flat cathode, the corrected t/Dk, t/Dk (corrected), is given by Equation (6):

$$t/Dk\ \text{(corrected)} = t/Dk\ \text{(preliminary)} \times \left\{1{,}015\ 75 + \frac{\left[0{,}471 - 0{,}001\ 93\left(Dk\right)\right] \times 4t}{D_{\text{cathode}}}\right\} \tag{6}$$

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, Equation (7):

$$Dk = \frac{\left[{}_i\sum t_i{}^2 - \frac{\left({}_i\sum t_i\right)^2}{n}\right]}{\left\{{}_i\sum t_i\left(t/Dk\right)_i - \left(\sum t_i\right)\left[{}_i\sum\left(t/Dk\right)_i\right]/n\right\}} \tag{7}$$

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 Equation (7) is close enough to be used in the corrective formulas for the edge effect, Equations (3) to (6). 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. 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.

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 Repeatability of measurement

A single polarographic determination of corrected oxygen permeability (Dk) using at least four lenses of one material differing by thickness shall have a repeatability of ± 10 % or less.

4.4.3.6 Reagents and materials

4.4.3.6.1 Oxygenated gas, consisting of a mixture of oxygen gas (20,9 %) and nitrogen or other gases (79,1 %). Air from the ambient environment can be used as it fits this specification. The gas shall be humidified to the point that it is water vapor-saturated when it contacts the front surface of a test specimen.

4.4.3.6.2 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.

4.4.3.7 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 (Figure 4 and Figure 5).

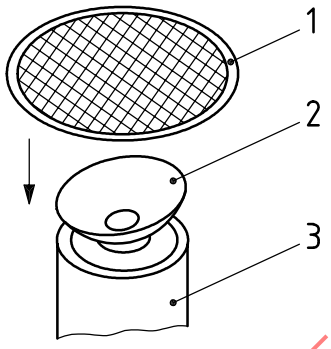
The polarographic cell body, which holds the electrodes and thermistor, is made of an oxygen impermeable polymer. The polarographic sensor's cathode is platinum or 24 carat gold and 4 mm in diameter. The silver anode (≥ 99,8 % purity) is annular, surrounding the cathode, and is separated from the cathode by the oxygen impermeable polymer. A small hole is drilled in the anode in which is potted the thermistor. 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) when used for testing of

1) The Rehder single-chamber system is the trade name of a product supplied by Rehder Development Company, Castro Valley, California, 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.

rigid 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 centreline. The sample is held against the cathode face by a nylon mesh retained by the O-ring.

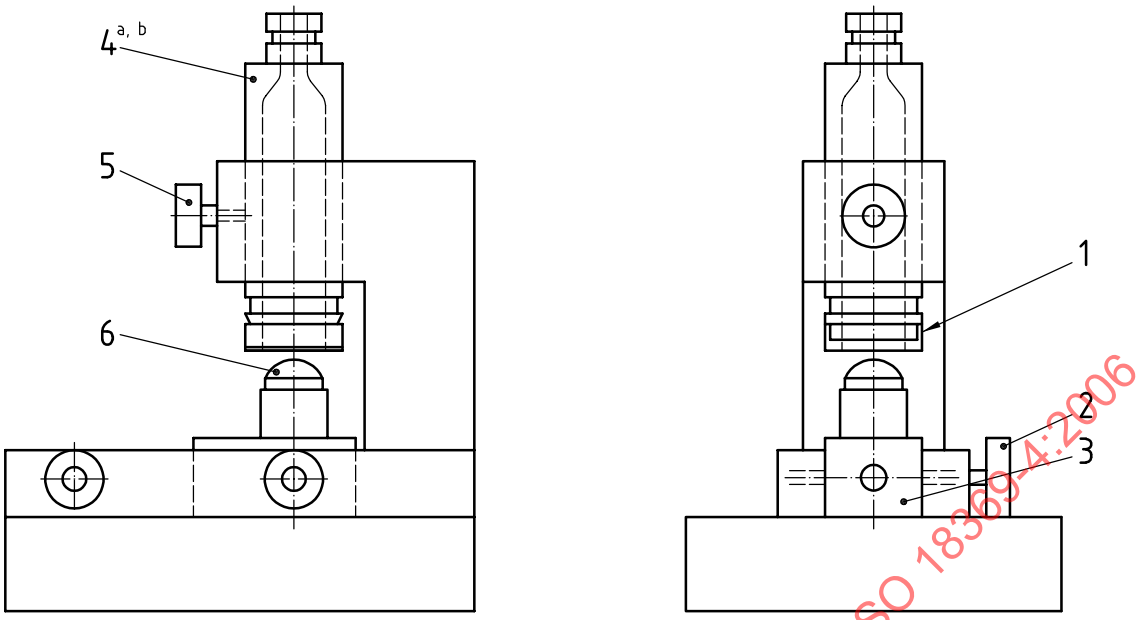
The electronics associated with the apparatus consist of a digital voltmeter to read the thermistor, a micro-ammeter to measure the oxygen flux current, and the necessary circuitry to maintain a constant voltage between the cathode and anode of the oxygen sensor.



Key

1 ring with mesh
2 hydrogel lens
3 flat polarographic cell

Figure 4 — Hydrogel sample between sensor and retaining mesh



a) Front view

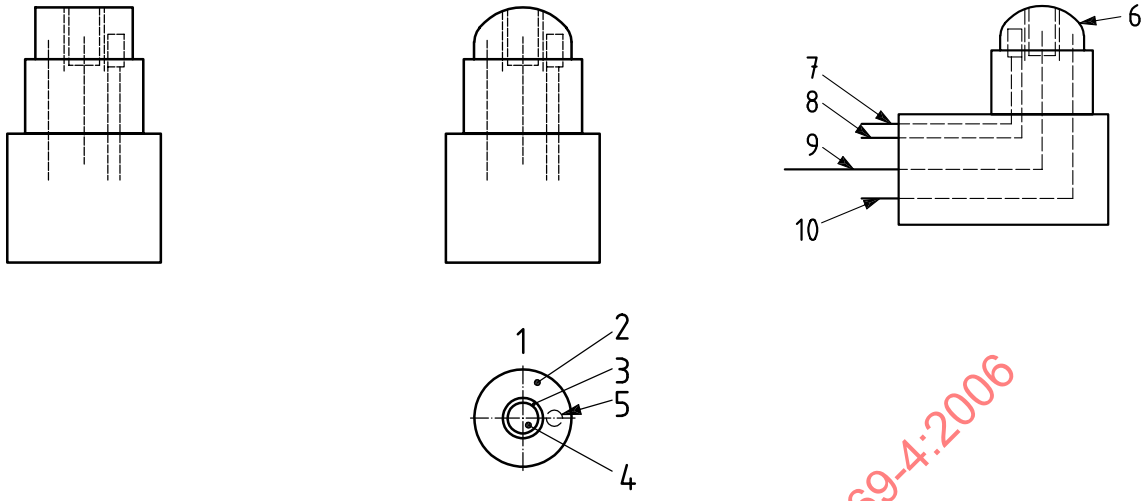
b) Side view

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 radiused samples.

Figure 5 — Clamping mechanism for polarographic measurement



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

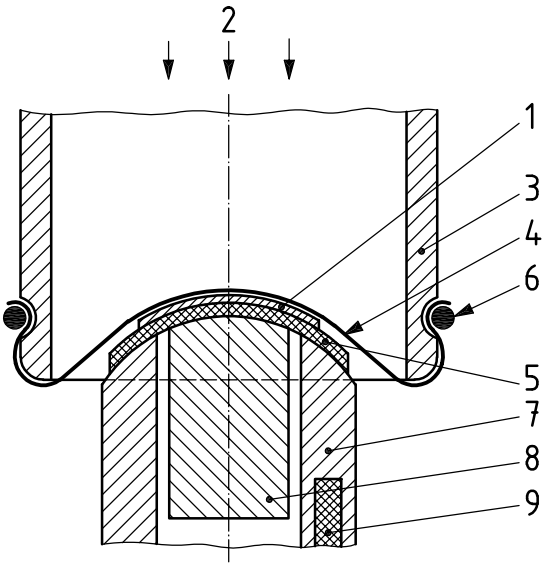
Key

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 ± 0,5 °C. Within the box is a reservoir of water that maintains a water vapor-saturated atmosphere of not less than 98 % relative humidity 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 water-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

4.4.3.8 Procedure

4.4.3.8.1 Thickness of samples

Measure the thickness of parallel or near-parallel samples according to ISO 18369-3. Determine the harmonic mean central thickness of powered samples as required in 4.4.2.1 and as defined in ISO 18369-1:2006, 2.1.2.4.3.

4.4.3.8.2 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. Necessary characteristics of the barrier are that it shall transport no oxygen, that it shall fit tightly against the oxygen sensor so that oxygen may not pass around its edge and under it to the cathode, and that it 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.

4.4.3.8.3 Measurement of equilibrium current

Bring the test equipment to a stable temperature of 35 °C ± 0,5 °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.

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 4 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.

4.4.3.9 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 Equation (8):

$$A = \pi h^2 \tag{8}$$

and of a spherical cathode face, by Equation (9):

$$A = 2\pi r\left[r - \left(r^2 - h^2\right)^{1/2}\right] \tag{9}$$

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[1] [4] [5] attainable up to a Dk at least 145×10^{-11} (cm^2/s) [ml O_2/(ml·hPa)]. The corrected oxygen permeability, Dk, is the result for each test material that shall finally be calibrated as noted in 4.4.5.

4.4.4 Coulometric method

4.4.4.1 General

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

The coulometric method is applicable for determining oxygen transmissibility of rigid and non-hydrogel soft contact lenses, incorporating various refractive powers and radially symmetric lens geometries. It is also applicable to contact lens materials in the form of standardized test samples. It may be especially useful for determination of permeability values above 145×10^{-11} (cm^2/s) [ml O_2/(ml·hPa)], which fall above the established range of the polarographic method of measurement, but is not applicable to hydrogel materials or hydrogel contact lenses.

4.4.4.2 Principle

A rigid or non-hydrogel flexible contact lens is placed in an oxygen gas transmission apparatus with exposed front and rear lens surface areas in contact with gas mixtures at eye temperature (35 °C). The gaseous environments at the anterior and posterior lens surfaces are separated by the contact lens, which acts as a barrier to the net flow of oxygen from the anterior environment to the posterior environment. The two environments and the contact lens can be purged of all detectable oxygen gas. Once purged, an oxygen-containing gas is allowed to fill the anterior environmental chamber and to diffuse through the contact lens. An inert carrier gas, initially oxygen-free, is allowed to flow across the posterior environmental chamber at the posterior lens surface and to remove oxygen molecules that have crossed the contact lens barrier. The carrier gas, now containing a small concentration of oxygen, is directed to a coulometric sensor that creates a current proportional to the concentration of oxygen flowing past the detector. With appropriate instrument calibration, such that the concentration of oxygen at the detector is precisely known, the rate of oxygen flow (μl O_2 /s) past the detector can be determined and recorded.

4.4.4.3 Calculated values

Oxygen flux (q_{O_2}), oxygen transmissibility (Dk/t), and oxygen permeability (Dk) can be calculated using Equation (10), knowing the area of the contact lens through which oxygen should have passed (A, in cm^2), the thickness of the lens or sample (t), the calculated oxygen flux (q_{O_2}) or recorded rate of oxygen volume flow past the detector (μl O_2 / s), and the oxygen tension difference between anterior (p_A) and posterior (p_P) environmental chambers during measurement ($p_A - p_P$, in hPa, where p_A is approximately 207 hPa and p_P is assumed to be zero).

$$Dk = \frac{t \times q_{O_2}}{p_A \times A} \times \frac{1}{10^3} \tag{10}$$

where

Dk is the oxygen permeability of test sample, in Dk units {10^{-11} (cm^2 / s) [ml O_2 / (ml·hPa)]};

p_A is the (barometric pressure less the vapour pressure), expressed in hPa, multiplied by 0,209 which is the oxygen fraction in oxygenated gas;

t is the radial thickness or mean harmonic central thickness, in cm, of the test sample (measured);

A is the exposed area, in cm^2, of test sample (measured);

q_{O_2} is the rate of oxygen flow, in μl O_2/s, past the detector (measured);

$\frac{1}{10^3}$ is the conversion factor from μl to ml.

4.4.4.4 Edge effect

When there is a difference between the front surface area exposed to flow of oxygen and the back surface area through which oxygen flows to be detected, the so-called “edge effect” may influence the measured oxygen transmissibility and permeability of test samples. These anterior and posterior surface areas are nearly identical when using the coulometric method, so that the “edge effect” is negligible.

4.4.4.5 Boundary layer effect

As aqueous solution does not contact either surface of the rigid or non-hydrogel flexible sample being tested, boundary layers are not present. Therefore, the coulometric method for non-hydrogel samples does not entail the use of correction factors for the boundary layer effect.

4.4.4.6 Repeatability of measurement

A single coulometric determination of oxygen transmissiblity (Dk/t) and/or oxygen permeability (Dk) shall have a repeatability of ± 5 % or less.

4.4.4.7 Reagents and materials

4.4.4.7.1 Oxygen-free "carrier gas", consisting of a mixture of nitrogen gas (97 % to 99,5 %) and hydrogen gas (0,5 % to 3 %), dry and containing not more than 100 µl/l [100 ppm[2)]] of oxygen. An oxygen trap and a moisture trap are used to ensure that the carrier gas is essentially oxygen-free and dry prior to reaching the diffusion cell. Thus, vapor pressure in Equation (10) is assumed to be zero.

4.4.4.7.2 Oxygenated "test gas", consisting of a mixture of oxygen gas (20,9 %) and nitrogen gas (79,1 %). A moisture trap dries the test gas prior to introduction into the anterior environmental chamber of the diffusion cell.

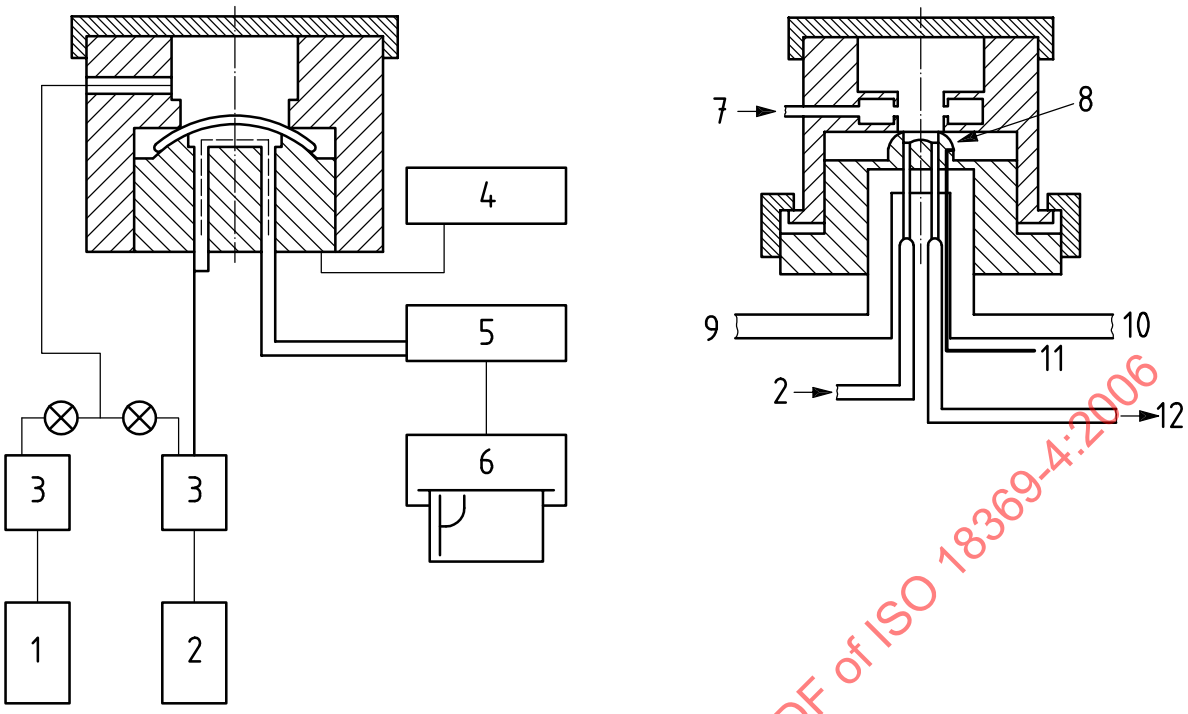
4.4.4.7.3 Sealing grease for sealing the contact lens test sample to the two halves of the diffusion cell. The grease is a high-viscosity non-silicone stopcock grease or a high-vacuum grease that is nearly impermeable to oxygen.

4.4.4.8 Apparatus

4.4.4.8.1 Oxygen gas transmission apparatus, equivalent to the Ox-Tran 100A[3)] diagrammed in Figure 8, consisting of a diffusion cell, O-ring, heating unit, flowmeter, coulometric oxygen sensor, load resistor and recorder. Suitable oxygen-impermeable ports, valves, and tubing allow purging of oxygen from each of the two environmental chambers inside the diffusion cell, introduction of oxygenated test gas into the anterior environmental chamber (the upper chamber, in Figure 8), and flow of carrier gas through the posterior environmental chamber (the lower chamber, in Figure 8) to the coulometric oxygen sensor. An oxygen trap ensures that carrier gas is free of oxygen before entering the posterior environmental chamber. Moisture traps ensure that gases are dry.

2) Parts per million (ppm) is a deprecated unit. Note that 1µl/l = 1 ppm.

3) Ox-Tran 100A is the trade name of a product supplied by Modern Controls, Inc., Minneapolis, Minnesota, 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.



a) **Device schematic** b) **Lens fixture assay**

Key

1	air	5	coulometric detector	9	thermal control loop
2	carrier gas	6	chart recorder output	10	to heat loop
3	gas dryer	7	test gas	11	thermocouple feed-back loop
4	temperature controller	8	contact lens	12	carrier gas to detector

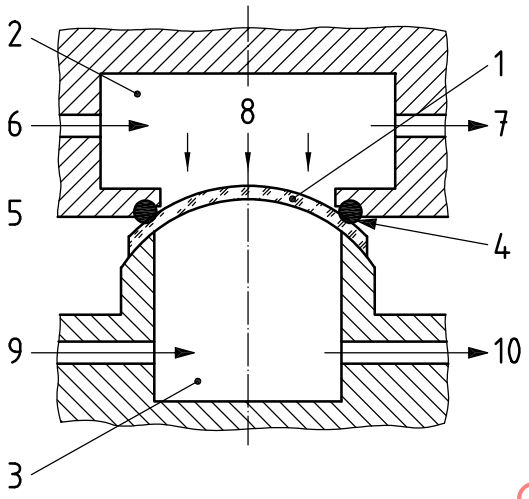
Figure 8 — Coulometric apparatus

4.4.4.8.2 Diffusion cell (see Figure 9), impermeable to oxygen and in two halves. When closed around a test sample, the cell houses two environmental chambers separated by the sample. The specific volumes enclosed by each cell half, when clamped, are not critical. These volumes should be small enough, however, to allow for rapid gas exchange, but not so small that an unsupported test sample, which happens to sag or bulge, could contact the top or bottom of the cell. The cell shall incorporate a thermistor to verify temperature and shall be fitted with a heating unit to maintain the chambers and sample at 35 °C ± 0,5 °C. An appropriately sized annular groove in the anterior half of the diffusion cell (the half that delivers oxygenated test gas to the anterior surface of the sample) retains a neoprene O-ring against the anterior test sample surface. The surface of the posterior cell half shall have a smooth annular mounting rim in contact with the posterior surface of the sample. The mounting rim shall be free of radial scratches.

4.4.4.8.3 Neoprene O-ring, for defining the area of test sample exposed to oxygen flow, by which pressing against the anterior test sample surface when the two halves of the diffusion cell are closed. Prior addition of the sealing grease to each annular area of contact with the specimen will ensure an oxygen-free central area of test sample when the diffusion cell is purged of oxygen. Therefore, an annular zone of compression sandwiches the test sample between the pre-greased O-ring contained in the anterior half of the diffusion cell and the pre-greased annular smooth surface of the posterior half of the diffusion cell.

The area (A) of the test sample exposed to flow of oxygen is determined after measurement of oxygen flow rate by examination of the annular imprint left on the front or back surface of the test sample after it has been removed from the diffusion cell. The inner circular diameter ($2\,h$) of the imprint specifies the outer limit of sample area through which oxygen passed during measurement. $2\,h$ is the chord diameter across the exposed back surface of a contact lens sample, and should be less than the back central optic zone diameter. For a flat test sample, the area (A) of sample exposed to oxygen flow is πh^2. The area (A) of a spherically

curved sample (i.e, a spherical contact lens) exposed to oxygen flow can be determined from Equation (9), when 2 h has been measured and the back central optic radius is known.



Key

1	rigid contact lens sample	6	inflow of test gas
2	anterior chamber	7	outflow
3	posterior chamber	8	oxygen flow
4	O-ring	9	inflow of carrier gas
5	surface coated with sealing grease	10	outflow to coulometric oxygen sensor

Figure 9 — Diffusion cell for coulometric method

4.4.4.8.4 Heating system and oxygen trap, consisting 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 flowmeter having an operating range of 5 ml/min to 100 ml/min shall monitor the flow rate of carrier and test gases.

4.4.4.8.5 Coulometric sensor, for measuring the humidity of gases. The extended use of the coulometric sensor with dry gases may result in decreased output and increased response time. Should the coulometric sensor become too dry, the situation can be corrected by injection of approximately 2 ml of deionized water into the sensor at its inlet connection.

It is important that saline not come in contact with the coulometric sensor, for the detector would then require replacement. Thus, only deionized water (not saline) should be injected into the sensor if it becomes dry.

4.4.4.8.6 Coulometric sensor and recording system, for determining the concentration of oxygen in the carrier gas. An oxygen-sensitive coulometric sensor operating at an essentially constant efficiency (95 % or above) shall convert the concentration of oxygen in the carrier gas, after appropriate calibration, into rate of oxygen flow past the detector (µl/s). The sensor shall be a porous nickel-cadmium electrode block used in conjunction with a graphite block, soaked in potassium hydroxide electrolyte, or be an equivalent sensor. 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.

4.4.4.9 Conditioning of test samples

Conditioning of test samples is required to out-gas all oxygen from the material prior to measurement. In the case of highly oxygen-permeable materials, such as silicone, silicone-acrylate, fluoropolymers, etc., the purging of oxygen from the diffusion cell prior to measurement will suffice. However, for non-hydrogel poor oxygen transmitters, such as samples made of polymethylmethacrylate (PMMA), test samples should be conditioned in a desiccator over calcium chloride for at least 48 h prior to enclosure and subsequent purging in the diffusion cell.

4.4.4.10 Procedure

4.4.4.10.1 Thickness of samples

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

4.4.4.10.2 Preparation of the apparatus

If preceding tests have exposed the apparatus to high moisture levels, it may be necessary to out-gas the system in order to remove residual moisture. This is achieved by purging the system overnight with carrier gas dried by moisture traps, with the coulometric sensor bypassed. Heating the apparatus will speed the drying and out-gassing process.

4.4.4.10.3 Insertion of test sample

Remove the test sample from its conditioning environment and place its peripheral posterior surface on the greased 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 greased 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.

4.4.4.10.4 Purging the system of oxygen

With the coulometric sensor bypassed, start the flow of carrier gas into the anterior and posterior environmental chambers of the diffusion cell at a rate of 50 ml/min to 60 ml/min. After 3 min to 4 min, reduce the flow rate to 5 ml/min to 15 ml/min and maintain for 10 min to 30 min. Next, divert carrier gas, which has passed through both environmental chambers, to the coulometric sensor. The sensor output will most likely increase abruptly at this point, indicating that the carrier gas is not oxygen-free. Assuming there are no leaks in the system, this oxygen is most likely a result of continued out-gassing of the test sample. The out-gassing will continue until the sensor output stabilizes at a constant low value, the zero voltage output (V_0). It is best to periodically divert carrier gas to the sensor for monitoring of oxygenation rather than to continually subject the sensor to the flow of gas for long periods. Thick test samples and/or materials having very low oxygen transmissibility may require purges of several hours, or even overnight, before a steady low voltage output is achieved. Test samples of high oxygen transmissibility may require only 10 min to 20 min for V_0 to be reached.

4.4.4.10.5 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 as occurred during the purge. Initiate a similar flow of oxygenated test gas through the anterior environmental chamber, substituted for the oxygen-free carrier gas used there during the purge. 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 sensor will require a relatively long 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 which end output stability may be reached in 5 min to 10 min. For this reason, test samples of comparable oxygen transmission qualities should be tested together.

If there is some question about the stability of the sensor reading before determination of the end output (V_E), the sensor can be bypassed overnight (6 h to 8 h) and then the flow can be re-established and V_E re-measured. If the two V_E estimates are the same, the correct value is obtained originally. If the second V_E is higher, then steady-state conditions are attained initially.

4.4.4.10.6 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):

$$q_{O_2} = \frac{K\left(V_E - V_0\right)}{R_L} \tag{11}$$

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.

4.4.4.10.7 Approximate calibration

Determine V_0 and V_E in the manner described in 4.4.4.10.4 and 4.4.4.10.5 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 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 Equation (10), 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 Equation (10)]. Next calculate the calibration constant (K) with the use of Equation (11), 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.

4.4.4.10.8 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;

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 is 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.

4.4.5 Calibration of instrumentation

Calibration 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 $\pm$ 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 calibration curve, if applicable. Examples of calibration are shown in References [1], [4] and [5] of the Bibliography.

The reproducibility of calibrated, 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 (University of Alabama at Birmingham, School of Optometry, Birmingham AL, USA 35294-0010). 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 part of ISO 18369 and does not constitute an endorsement by ISO of the product named.

4.4.6 Test report

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

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

b) 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 depend, and the standard deviation.

4.5 Refractive index

4.5.1 General

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 587,6 nm (helium 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 Equation (12):

$$n = \frac{n' \sin(\alpha)}{\sin(90°)} \tag{12}$$

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.