INTERNATIONAL STANDARD

ISO 9913-1

First edition
1996-11-01

Optics and optical instruments — Contact lenses —

Part 1: Determination of oxygen permeability and transmissibility by the FATT method

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 9913-1:1996
https://standards.iteh.ai/catalog/standards/sist/536ad330-a061-4dd4-b3ce-184f0050123/iso-9913-1-1996

Optique et instruments d'optique — Lentilles de contact —

Partie 1: Détermination de la perméabilité à l'oxygène et de la transmissibilité de l'oxygène avec la méthode FATT

Reference number
ISO 9913-1:1996(E)

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.

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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

International Standard ISO 9913-1 was prepared by Technical Committee ISO/TC 172, *Optics and optical instruments*, Subcommittee SC 7, *Ophthalmic optics and instruments*.

ISO 9913-1:1996
https://standards.iteh.ai/catalog/standards/sist/3b0ad590-a061-4dd4-b3ce-1847f0050123/iso-9913-1-1996

ISO 9913 consists of the following parts, under the general title *Optics and optical instruments — Contact lenses:*

— *Part 1: Determination of oxygen permeability and transmissibility by the FATT method*

— *Part 2: Determination of oxygen permeability and transmissibility by the coulometric method*

Annex A forms an integral part of this part of ISO 9913. Annexes B and C are for information only.

© ISO 1996

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Optics and optical instruments — Contact lenses —

Part 1:
Determination of oxygen permeability and transmissibility by the FATT method

1 Scope

This part of ISO 9913 describes a polarographic method for the determination of oxygen permeability of contact lens materials and oxygen transmissibility of contact lenses. It specifies the procedures for making the measurements and establishes the conditions under which measurements are made.

It is applicable for determining permeability (Dk) in the range 0 to 75 × 10^{-11} (cm^2/s) [ml O_2/(ml·hPa)].

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 9913-1:1996
https://standards.iteh.ai/catalog/standards/sist/956ad330-a061-4dd4-b3ce-1847f0050123/iso-9913-1-1996

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 9913. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9913 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 8320:1986, *Optics and optical instruments — Contact lenses — Vocabulary and symbols.*

ISO 10339:— [1], *Optics and optical instruments — Contact lenses — Determination of water content of soft lenses.*

ISO 10344:1996, *Optics and optical instruments — Contact lenses — Saline solution for contact lens testing.*

3 Definitions

For the purposes of this part of ISO 9913, the definitions given in ISO 8320 and the following definitions apply.

3.1 dark current (I_d)**:** Current passing through a cell when an impermeable barrier is placed in between the electrodes.

3.2 oxygen permeability (Dk)**:** Rate of oxygen flow under specified conditions through a unit area of contact lens material of unit thickness when subjected to unit pressure difference.

NOTES

1 Oxygen permeability is expressed in units of (cm^2/s) [ml O_2/(ml·hPa)]. An alternative expression for the units is cm^3 O_2·(cm/cm^2)/(s·hPa).

2 Oxygen permeability is a physical property of the material. It is not a function of the shape or thickness of the material sample.

3 To convert the units of Dk for use with mmHg instead of hectopascals (hPa), multiply the numerical value of Dk by 1,333 22.

1) To be published.

3.3 oxygen transmissibility (Dk/*t*): Value for oxygen permeability divided by the thickness (measured in centimetres) of the measured sample under specified conditions.

NOTES

4 Oxygen transmissibility is not an absolute physical property; it is dependent upon the sample thickness.

5 The reciprocal of oxygen transmissibility (Dk/*t*) is called electrical resistivity (*t*/Dk).

3.4 thickness (*t*): Either the thickness at a specified point or the harmonic mean thickness of the specified area of the lens or sample.

4 Principle

The polarographic method directly measures the number of oxygen molecules diffusing through a piece of test material by electrochemically removing the molecules from solution as soon as they pass through the material. For full details of the theoretical background, see annex C.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

5 Reagents

5.1 Oxygen, commercial grade, in a steel cylinder.

ISO 9913-1:1996
https://standards.iteh.ai/catalog/standards/sist/536ad330-a061-4dd4-b3ce-184f8050123/iso-9913-1-1996

5.2 Nitrogen, commercial grade, oxygen free, in a steel cylinder.

6 Apparatus

The test cell shall have the following characteristics.

6.1 The cathode shall be made of 24 carat gold or 99,9 % (*m*/*m*) platinum (min.), shall be located centrally under the test sample, shall have a diameter of 4 mm to 7,2 mm and shall have a polished surface.

6.2 The anode shall be made of silver [99,9 % (*m*/*m*) min.], shall be concentric to the cathode and shall have an area greater than that of the cathode.

6.3 The apparatus shall be capable of maintaining a potential difference between the electrodes of (0,75 ± 0,05) V.

6.4 The electrode face to be used for measuring samples of rigid lens material shall be a smooth spherical surface with a radius of curvature in the range 7,70 mm to 8,30 mm.

6.5 The electrode face to be used for measuring flat samples and hydrogel lenses shall be plane.

6.6 The apparatus shall include a device (see figure B.1) by which the test sample may be pressed against the electrode. The device shall allow oxygen to pass freely into the sample.

6.7 The apparatus shall be capable of maintaining the test sample at 35 °C ± 0,5 °C in an atmosphere with a relative humidity of not less than 95 %.

6.8 If a bath of oxygen saturated water is needed as the oxygen source, then a means of stirring or mixing the water shall be incorporated in the apparatus. The stirrer shall ensure that a minimal but constant stagnant boundary layer is maintained at the sample surface.

6.9 A temperature monitoring device having good thermal contact with the test sample shall be mounted. This device shall be capable of measuring the temperature with an accuracy of 0,5 °C.

NOTE 6 For examples of apparatus, see annex B.

6.10 An ammeter having a range from 0,0 µA to 10,0 µA.

7 Test samples

7.1 The test samples shall have parallel or near-parallel (see note) anterior and posterior surfaces.

NOTE 7 The thickness of the sample used for measurement should be as uniform as possible. In the case of manufactured lenses, the near-parallel condition would correspond to dioptric powers within the range + 0,50 to − 0,50.

7.2 The back optic zone radii of the test samples shall be within the range 7,40 mm to 8,60 mm.

7.3 The surfaces of test samples shall be clean and polished to the quality acceptable in normal lens production for human use.

7.4 In the case of hydrogel material, the samples shall be stored in saline test solution in accordance with ISO 10344 for a minimum of 24 h prior to testing and shall be equilibrated for at least 2 h at test temperature.

8 Test procedure

8.1 Dark current/zero-point correction

Determine the dark current of the cell by placing a barrier between anode and cathode consisting of two Polymethylmethacrylate (PMMA) lenses with an aluminium foil disc between them and measuring the current.

NOTES

8 As this barrier will not transmit oxygen, the equilibrium current is the dark current (I_d) which is the reference for material of zero permeability.

9 Necessary characteristics of the barrier are that it should transmit no oxygen, that it should fit tightly against the anode so that no oxygen can pass around its edge and under it to the cathode and that it should be an electrical non-conductor.

10 If the oxygen-saturated water bath method is used, it will be necessary to carry out the zero-point adjustment (or "correction") using oxygen-free nitrogen gas.

8.2 Measurement of t/Dk

Bring the test equipment to 35 °C ± 0,5 °C and allow the temperature to stabilize. Remove the sample from the saline solution, blot and place the lens on the test cell. If the sample is made of material that does not incorporate water in its molecular structure, place a piece of saturated paper, e.g. cigarette paper, on the surface of the cell and place the sample thereon. Firmly clamp the sample to the surface of the electrode. If the source of oxygen is a water bath, fill the electrode assembly with electrolyte (for example, standard saline solution in accordance with ISO 10344), establish the gas flow, activate the stirring mechanism and ensure that an equilibrium condition has been reached before proceeding.

Allow the current reading to stabilize and record the measurement. Remove the sample from the test cell and measure the centre thickness to an accuracy of ± 0,005 mm.

Repeat the procedure for at least four samples with thicknesses of approximately 0,10 mm, 0,17 mm, 0,24 mm and 0,30 mm.

NOTES

11 Samples should be no thicker than 0,40 mm.

12 Accuracy may be improved by making multiple independent measurements on each sample. Independent measurements are made by removing the sample from the apparatus, re-equilibrating it and repeating the procedure.

9 Expression of results

NOTE 13 For the theoretical background, see annex C.

9.1 Calculate the value of t/Dk (total) for each test sample using the following equation:

$$t/\mathrm{Dk} = p_{O_2}/F$$

with

$$p_{O_2} = (p_{bar} - p_{H_2O}) \times 0{,}209;$$

$$F = (I - I_d) \times 5{,}804 \times 10^{-2}/A.$$

where

p_{O_2} is the partial pressure of oxygen above the cell in hectopascals (hPa);

F is the oxygen flux in hundreds of metres per cubic centimetre second [m × 10^2/(s × cm^2)];

p_{bar} is the barometric pressure in hectopascals (hPa);

p_{H_2O} is the vapour pressure of water in the atmosphere in hectopascals (hPa);

20,9 is the percentage oxygen in the atmosphere in air at 0 % humidity (if the percentage of oxygen is other than 20,9 %, that value is used);

I is the measured electric current in amperes;

I_d is the dark current of the cell, in amperes, i.e. the current which flows in the absence of the O_2 flux;

A is the area of the central electrode (cathode) in square centimetres.

The area of the cathode when using electrodes with spherical surfaces is given by the equation

$$A = 2\pi r(r - \sqrt{r^2 - D^2/4})$$

where

D is the diameter of cathode, in millimetres;

r is the radius of curvature of the surface, in millimetres.

The area of a cathode with a flat surface is given by the equation

$$A = \pi(D/2)^2$$

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 9913-1:1996
https://standards.iteh.ai/catalog/standards/sist/536ad330-a061-4dd4-b3ce-1847f0050123/iso-9913-1-1996

This procedure is carried out for at least four different sample thicknesses and the resulting values of t/Dk are used to find the rate of change of t/Dk with change of t. This value is equal to 1/Dk.

9.2 Correct each t/Dk value for diffusion "edge effects" by using one of the following equations, as applicable.

For hydrogels when using a cathode with a spherical surface:

$$\left(\frac{t}{\mathrm{Dk}}\right)_{\text{corrected}} = \left(\frac{t}{\mathrm{Dk}}\right)_{\text{measured}} \times \left(1 + \frac{2{,}35t}{D}\right)$$

For hydrogels when using a flat cathode:

$$\left(\frac{t}{\mathrm{Dk}}\right)_{\text{corrected}} = \left(\frac{t}{\mathrm{Dk}}\right)_{\text{measured}} \times \left(1 + \frac{1{,}89t}{D}\right)$$

For non-hydrogels when using a cathode with a spherical surface:

$$\left(\frac{t}{\mathrm{Dk}}\right)_{\text{corrected}} = \left(\frac{t}{\mathrm{Dk}}\right)_{\text{measured}} \times \left[1{,}017\,25 + \frac{4[0{,}587 - 0{,}001\,93(\mathrm{Dk})]t}{D}\right]$$

For non-hydrogels when using a flat cathode:

$$\left(\frac{t}{\mathrm{Dk}}\right)_{\text{corrected}} = \left(\frac{t}{\mathrm{Dk}}\right)_{\text{measured}} \times \left[1{,}015\,75 + \frac{4[0{,}471 - 0{,}001\,93(\mathrm{Dk})]t}{D}\right]$$

where

D is the diameter of the cathode, in millimetres;

t is the thickness of the sample, in millimetres;

Dk is the measured Dk.

NOTE 14 The correction factors for non-hydrogels contain the value of Dk, which at this stage is unknown. The procedure for overcoming this difficulty is to calculate Dk, using uncorrected values of t/Dk in accordance with 9.3. The value described in Dk found in this way is close enough to the true value to use in the corrective formulas. The Dk is calculated again using the corrected values of t/Dk. This value of Dk is the correct one. For convenience of computation, the values of Dk are multiplied by 10^{-11} before use in these functions.

9.3 Calculate the value of Dk using the following regression equation:

$$\mathrm{Dk} = \left[\sum_i t_i^2 - \frac{1}{n}\left(\sum_i t_i\right)^2\right] \Big/ \left\{\left[\sum_i t_i (t/\mathrm{Dk})_i\right] - \frac{1}{n}\left[\sum_i t_i \sum_i (t/\mathrm{Dk})_i\right]\right\}$$

where

$(t/\mathrm{Dk})_i$ is the reciprocal of the transmissibility (electrical resistivity) of the ith sample;

t_i is the centre thickness of the ith test sample, in millimetres;

n is the number of test samples;

Σ is the summation over n different samples.

9.4 If the value of Dk is to be normalized (see note), carry out the following procedure.

Measure samples of the standard material specified in annex A. Normalize the calculated values of permeability [Dk (calc)] as follows:

Dk (normalized) = Dk (calc) × Dk (standardized value of standard material)/Dk (measured value of standard material).

NOTE 15 This normalization procedure is necessary to make results obtained with different apparatus directly comparable. As the oxygen source concentration is more difficult to verify when using the oxygen-saturated water bath method, it is recommended that the normalization procedure be used whenever this method is employed.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 9913-1:1996
https://standards.iteh.ai/catalog/standards/sist/936ad350-a661-4dd4-b5ec-1847f0050123/iso-9913-1-1996

10 Test report

The test report shall include the following information:

a) the type of apparatus used (i.e. water saturated with oxygen or oxygen saturated with water);

b) the type of material tested;

c) the thickness of the samples, the number of samples at each thickness and the number of independent measurements on each sample;

d) the value of permeability found and a statement of whether or not the value was normalized (see 9.4) to a standard material;

e) if a standard material was used for normalization, the above information on this standard material and, if it is a hydrogel, its water content.

Annex A
(normative)

Standard material for normalization

A.1 General

This annex specifies a material for use in the standardization of permeability measurements and to give permeability values under standardized conditions.

A.2 PolyHEMA, nominally 38 % (*m/m*)

The standard material shall comprise untinted polyhydroxyethylmethacrylate (polyHEMA) with no added UV-blocking agent and at a nominal moisture content of 38 % (*m/m*).

When the water content (see A.3) of this standard material, measured at (35 ± 0,5) °C, is 38 %, the Dk at 35 °C shall be taken to be 9,5 × 10^{-11}. For samples of this standard material whose water content is close to but not equal to 38 %, the Dk shall be calculated using the equation

$$Dk_{35\,°C} = 2{,}0 \times 10^{-11}\, e^{(0{,}041\,1 \times W)}$$

where W is the water content expressed as a percentage (*m/m*).

A.3 Water content

The water content shall be determined for the samples, using the procedure described in ISO 10339, immediately after the permeability has been measured.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 9913-1:1996
https://standards.iteh.ai/catalog/standards/sist/536ad330-a061-4dd4-b3ce-1847f0050123/iso-9913-1-1996

Annex B
(informative)
Examples of apparatus

B.1 Water-saturated air/oxygen source

B.1.1 Figures B.2 and B.3 show an apparatus employing a water-saturated air/oxygen source which satisfies the requirements of clause 6.

B.1.2 Figure B.2 shows the cell which holds the electrodes and the thermistor. The cell body is made of polymethylmethacrylate. The 24 carat gold cathode (a) is 4,24 mm in diameter and 6,0 mm long. The silver anode (b) is a hollow cylinder 7,0 mm long with an outer diameter of 7,0 mm and inner diameter of 5,0 mm. A small hole is drilled in the anode in which is secured the thermistor (c).

Using a contact lens lathe, the electrode surface is cut and polished to give a spherical surface with a radius of curvature of 7,8 mm. The spherical electrode shown in figure B.2 is designed for use with rigid contact lenses. For flat samples and for hydrogel lenses, an electrode of similar design but with a flat surface is used.

B.1.3 Figure B.3 shows the holding and clamping device in which the cell (C) is secured by thumbscrew (E) and incorporates the hold-down mechanism for the test lens. The lens is firmly pressed against the cell by a ring (R). To ensure that the sample or lens surface is firmly pressed against the surface of the cathode when a flat cathode is used, a nylon mesh is attached in the fashion of a drum head on ring (R). To get a tight fit, the mesh is boiled in water to soften the nylon just prior to it being fastened to the ring. For example, to place a hydrogel lens in the cell, the lens is placed on the cathode with its concave side up. The ring with the mesh is placed over it and pressed down using the cylinder (B). A thumb screw (D) locks the cylinder (B) in position.

B.1.4 The entire assembly is enclosed in a heated box and held at 35 °C. The box contains a reservoir of water which maintains a saturated atmosphere.

B.1.5 The electronics associated with the apparatus consist of a digital voltmeter to read the thermistor, a microammeter to measure the oxygen flux current and the necessary circuitry to maintain the correct voltage between the electrodes.

B.2 Oxygen-saturated water bath

B.2.1 Figures B.4 and B.5 illustrate an alternative apparatus employing an oxygen-saturated water bath which satisfies the requirements of clause 6.

B.2.2 Figure B.4 shows the double bath arrangement. The inner beaker is filled with standard saline solution into which are placed the electrode apparatus, the temperature control sensor, the gas infusion system and the stirrer. This beaker is placed in the outer beaker which is filled with water into which the temperature-controlling heater is placed. The outer beaker rests on the magnetic stirrer drive unit.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 9913-1:1996
https://standards.iteh.ai/catalog/standards/sist/536ad330-a061-4dd4-b3ce-1847f0050123/iso-9913-1-1996

The magnetic stirrer is used to ensure uniform and controlled mixing. The stirrer itself is 3,0 cm in diameter and 0,8 cm thick and is rotated at a constant speed of 5 Hz. The sample is held (30 ± 0,1) mm above the top of the stirrer.

NOTE 16 Although the stirrer mixes fairly thoroughly, it is impossible to prevent stagnation and hence a concentration gradient will exist near any stationary oxygen sink such as the sample. It is important that the stirring speed and stirrer-to-sample distance be very accurately duplicated for any set of test samples, otherwise the boundary film will vary from measurement to measurement and the correction method given in this part of ISO 9913 will not be effective.

Multiple independent measurements of the same sample can be used to verify that the test conditions are well duplicated by verifying that the data are essentially the same from trial to trial.

B.2.3 The oxygen-containing gas mixture is introduced via a diffuser located at a height midway between the sample and the stirrer and 30 mm to the side of the electrode centreline.

NOTE 17 It is important that the oxygen concentration in the solution at the sample surface be constant for all samples of a specific material under test. If this condition is not met, an unknown variable is introduced into the calculation and the procedure given in this part of ISO 9913 for removing the effect of boundary layers will not work adequately.

The critical oxygen concentration may be controlled in the following ways.

a) If the gas introduced at the diffuser has the same partial pressure as the atmosphere, an equilibrium will be established between the oxygen concentration in the solution and the atmospheric oxygen in contact with the free surface.

b) If the oxygen partial pressure of the gas entering the solution is different from the partial pressure of oxygen in the atmosphere, a gradient will develop in the solution at the equilibrium condition. This condition will only be stable if the gas pressure and flow rate entering the diffuser are tightly controlled and constant.

It is best to use both of the above strategies, i.e. to supply a gas mixture with an oxygen concentration equal to atmospheric and supply this gas at a constant flow rate.

B.2.4 Figure B.5 shows the electrode configuration. This design has a removable cathode, allowing either a flat or convex cathode to be installed, so that either flat samples or lenses can be measured. The cathode assembly is made of non-gas permeable plastic with the gold or platinum electrode embedded in it on the centre line. The sample is held against the cathode by a retainer made of non-gas-permeable plastic. This retainer is either flat or curved to match the cathode in use.

The retainer has a circular aperture in its centre through which oxygen passes to the sample and which defines the area of the sample through which oxygen enters. The retainer, and hence the sample, is held onto the electrode assembly by a threaded stainless steel or plastic (non-gas-permeable) sleeve which screws into the electrode assembly.

An O-ring seal is provided between the sleeve and the retainer. This acts as a fluid seal to prevent the internal electrolyte from leaking into the saline solution bath. This electrode design uses a 0,5 solution of N-KCl as an electrolyte connecting the cathode with the anode.

The anode is a silver sleeve forming the surface of the upper portion of the electrode. It is surrounded by the stainless steel body of the electrode assembly, leaving a small space in between. Into this space the electrolyte is poured after the sample is in place, and it forms a seal at the lower end of the chamber.

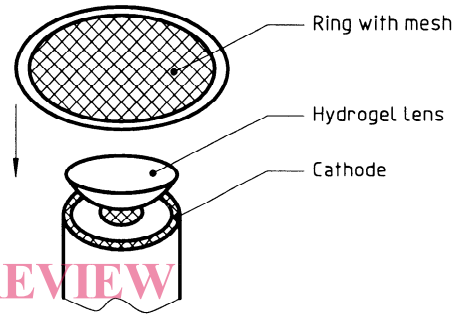

Figure B.1 — Example of a device used for pressing the test sample against the electrode

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 9913-1:1996
https://standards.iteh.ai/catalog/standards/sist/536ad330-a061-4dd4-b3ce-1847f0050123/iso-9913-1-1996