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Plastics — Determination of refractive index

Plastiques — Détermination de l'indice de réfraction

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Foreword

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

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

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

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 489:1999), which has been technically revised.

The main changes compared to the previous edition are as follows:

- in the Scope, the description about the precision of the explanation of the method A and method B has been deleted;
- normative references have been updated;
- the definition of the temperature control device of method A has been changed;
- the text of <u>Clause 8</u>, Precision, has been moved to <u>Annex A</u>;
- in <u>Clause 9</u>, the type of the immersing liquid used in method B has been added;
- the document has been editorial revised.

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

Plastics — Determination of refractive index

1 Scope

This document specifies two test methods for determining the refractive index of plastics, namely:

- Method A: a refractometric method for measuring the refractive index of moulded parts, cast or extruded sheet or film, by means of a refractometer. It is applicable not only to isotropic transparent, translucent, coloured or opaque materials but also to anisotropic materials.
- Method B: an immersion method (making use of the Becke line phenomenon) for determining the refractive index of powdered or granulated transparent materials by means of a microscope. Monochromatic light, in general, is used to avoid dispersion effects.

NOTE The refractive index is a fundamental property which can be used for checking purity and composition, for the identification of materials and for the design of optical parts. The change in refractive index with temperature can give an indication of transition points of materials.

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 291, Plastics — Standard atmospheres for conditioning and testing

3 hTerms and definitions/standards/sist/1883043a-f6ae-40c6-b2a4-64c7c176b012/iso-

No terms and definitions are listed in this document.

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

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

Apparatus and materials

4.1 Method A

- **4.1.1 Abbe refractometer,** or any other refractometer that can be shown to give the same results, accurate to 0,001 and capable of measuring the refractive index in the range from 1,300 to 1,700. A temperature-controlling device (4.1.4) shall be provided for the specimens and prisms.
- **4.1.2 White or sodium lamp,** used as a source of light.

4.1.3 Contacting liquid.

WARNING — The contacting liquid may present an environmental hazard during handling, storage and disposal. It is the responsibility of the user of this document to verify its toxicity and establish national and regional regulations for safe handling and disposal.

The contacting liquid shall have a refractive index higher than that of the material to be examined and shall not soften, attack or dissolve the plastic material. The liquids listed in <u>Table 1</u> may be used for the respective plastic materials, but other liquids meeting these requirements may also be used.

Table 1 — Contacting liquids

Plastic material	Contacting liquid
Cellulose derivatives	Aniseed oil or 1-bromonaphthalene
Fluorine-containing polymers	1-Bromonaphthalene
Urea-formaldehyde	Aniseed oil or 1-bromonaphthalene
Phenol-formaldehyde	1-Bromonaphthalene
Polyethylenes	1-Bromonaphthalene
Polyamides	1-Bromonaphthalene
Unsaturated polyester	1-Bromonaphthalene
Polyisobutylene	Saturated aqueous solution of zinc chloride made slightly acid
Poly(methyl methacrylate)	Saturated aqueous solution of zinc chloride made slightly acid or 1-bromonaphthalene
Polystyrene	Saturated potassium mercury(II) iodide solution
Styrene-acrylonitrile copolymers	1-Bromonaphthalene
Vinyl resins (vinyl chloride copolymer or plasticized PVC)	1-Bromonaphthalene
Poly(vinyl chloride)	1-Bromonaphthalene
Poly(ethylene terephthalate)	Methylene iodide
Polycarbonate	Methylene iodide
Diethylene glycol bis(allyl carbonate) (CR 39)	Methyl salicylate, aniseed oil or 1-bromonaphthalene
Polyarylate https://standards.iteh.ai/catalog	Saturated aqueous solution of zinc chloride made slightly acid, methylene iodide or 1-bromonaphthalene
Polyetheretherketone	Methylene iodide 022
Polypropylene	1-Bromonaphthalene

4.1.4 Temperature control system, capable of maintaining the temperature of the main prism, subprism and specimen at (23 ± 0.5) °C.

4.2 Method B

- **4.2.1 Microscope**, having a magnifying power of at least 200x, an objective giving approximately 20x of primary magnification and a substage condenser fitted with a centering illuminating-aperture diaphragm capable of being stopped down to give a very narrow axial beam.
- **4.2.2 Monochromatic light**, usually the sodium D line, having a wavelength of 589 nm, is used as the light source for the microscope.
- **4.2.3 Immersion liquids**, with different refractive indices.

WARNING — The contacting liquid may present an environmental hazard during handling, storage and disposal. It is the responsibility of the user of this document to verify its toxicity and establish national and regional regulations for safe handling and disposal.

The immersion liquids listed in <u>Table 2</u> with known refractive indices can be used separately and also as mixtures when different increments of accuracy are needed. The immersion liquids shall not soften, attack, dissolve or swell the surface of the particles.

Table 2 — Immersion liquids

Immersion liquid	Refractive index at 23 °C	
	$n_{ m D}^{23}$	
n-Butyl carbonate	1,410	
Tri-n-butyl citrate	1,444	
n-Butyl phthalate	1,491	
1-Bromonaphthalene	1,657	
Diiodomethane (methylene iodide)	1,747	
Aqueous solution of potassium mercury(II) iodide	1,419 to 1,733 ^a	
Silicone oils	1,37 to 1,56 ^a	
^a Useful range for the purpose of the test.		

5 Preparation of test specimens

5.1 Method A

Cut, from the sample, specimens of such a size as to fit on the face of the fixed half of the refractometer prisms.

The following dimensions are recommended for sheet specimens:

thickness: 3 mm to 5 mm.

For maximum accuracy, the surface of the test specimen in contact with the prism (the measurement face) shall be optically flat and well-polished. Eliminate any burs formed by cutting or any contamination attached to the specimen.

Satisfactory contact between the specimen and the prism is indicated when the dividing line between the light and dark halves of the eyepiece field appears sharp and straight.

Ensure that the edge of the specimen (perpendicular to the first) is also optically flat and fairly well-polished. The two polished surfaces shall intersect along a sharp line without a bevelled or rounded edge.

The following dimensions are recommended for film specimens:

— thickness: the actual film thickness, but not less than 2 μm.

For anisotropic material, see <u>7.1.3</u>.

5.2 Method B

The test sample consists of particles of the material to be examined, for example powder, granules or chips. The particles shall have dimensions sufficiently small and be so distributed as to permit simultaneous observation of approximately equal areas of the sample and the surrounding area in the field of view.

Ensure that the thickness of the test sample is significantly lower than the working distance of the microscope objective.

5.3 Required number of specimens or measurements

For sheets or films, five specimens are required. In the case of powders, pellets and granules, a quantity of sample sufficient to make five measurements is required.

6 Conditioning

- **6.1** Condition the specimens in accordance with ISO 291 at (23 ± 2) °C and at (50 ± 5) % relative humidity for not less than 88 h prior to the test if no other period of conditioning is stated in the relevant material specification.
- **6.2** Set up the test apparatus in an atmosphere maintained at (23 ± 2) °C and (50 ± 5) % relative humidity.

7 Procedure

7.1 Method A

7.1.1 General

If an Abbe refractometer (4.1.1) is used, carry out the following procedure. For other refractometers, modify the procedure in accordance with the manufacturer's recommendations, if necessary.

Carry out the determination at (23 ± 0.5) °C.

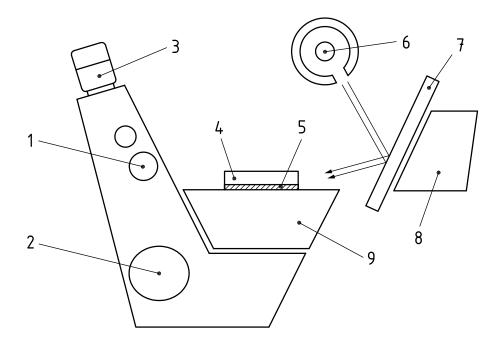
7.1.2 Transparent sheet

Place a small drop of the contacting liquid (4.1.3) on the well-polished surface of the transparent sheet specimen (the measurement face) and place it in firm contact with the surface of the prism with the polished edge of the specimen towards the light source as shown in Figure 1. Adjust the adjusting handle of the refractometer until half of the eyepiece field is dark.

Adjust the compensator (Amici prisms) drum until all colours have been removed from the field. Then adjust the adjusting handle by means of the vernier until the dividing line between the light and dark portions of the field coincides exactly with the point of intersection of the eyepiece cross-hairs as shown in Figure 2.

Read the refractive index of the material from the instrument scale.

The dispersion, if required, can be found by noting the compensator drum reading and using this, together with the value of the refractive index, to read the dispersion from a chart supplied with the instrument.



Key

- 1 compensator drum
- 2 adjusting handle
- 3 eyepiece
- 4 specimen
- 5 contacting liquid

- 6 source of light
- 7 opal-coloured reflective plate
 - sub-prism
- 9 main prism
- Figure 1 Method for measuring refractive index of transparent sheet

Key

- 1 boundary line
- 2 cross-hair lines
- 3 light half of eyepiece field

- 4 dark half of eyepiece field
- 5 scale for refractive index

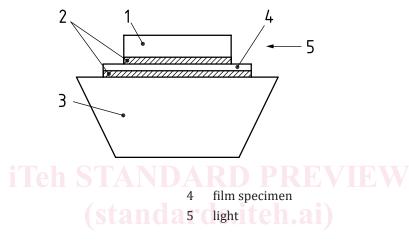
Figure 2 — Refractometer field of vision

7.1.3 Film

Place a drop of the contacting liquid (4.1.3) on the main prism followed by a film specimen. Place another drop of contacting liquid on the top of the film and then place the glass plate on the specimen as shown in Figure 3. The refractive index of the glass plate shall be greater than that of the film specimen.

Use the sodium lamp illuminator for the measurements of anistropic film such as oriented ones so as to obtain a beam of steady incident light and to avoid any dispersion effects. As shown in Figure 1, open the sub-prism and place the opal-coloured reflective plate against it to reflect the light of the sodium lamp onto the edge of the glass plate.

NOTE It is difficult to measure the refractive index of film specimens because films are very thin and this results in a limited amount of incident light passing through the edge of the specimen. To compensate for this, a glass plate can be placed on top of the specimen.



Key

- 1 glass plate
- 2 contacting liquid
- 3 main prism

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Figure 3 — Method for measuring refractive index of film

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7.1.4 Anisotropic material

In the case of anisotropic material such as injection or extrusion mouldings, different values of the refractive index may be found when measurements are made in different parts of the specimen (see Figure 4). In such cases, different specimens are prepared with their polished edges parallel or perpendicular to the machine direction.

By attaching a polarizing filter to the eyepiece of the Abbe refractometer, the measurement of specimens with multiple refractive indices can be made. By using different combinations of the specimen-positioning direction (illuminated surface facing the machine direction or at 90° from the machine direction) and of the polarizing-filter direction (rotating the filter 90° between two positions) as shown in Figure 5, the refractive index can be measured in any particular direction.