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# Plastics — Determination of refractive index

*Plastiques — Détermination de l'indice de réfraction*



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# Foreword

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

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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 Clause 8, Precision, has been moved to Annex A;

— in Clause 9, 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 Terms and definitions

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/

## 4 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 Table 1 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 | Saturated aqueous solution of zinc chloride made slightly acid, methylene iodide or 1-bromonaphthalene |
| Polyetheretherketone | Methylene iodide |
| 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 Table 2 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 $n_D^{23}$ |
|---|---|
| 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 7.1.3.

## 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.

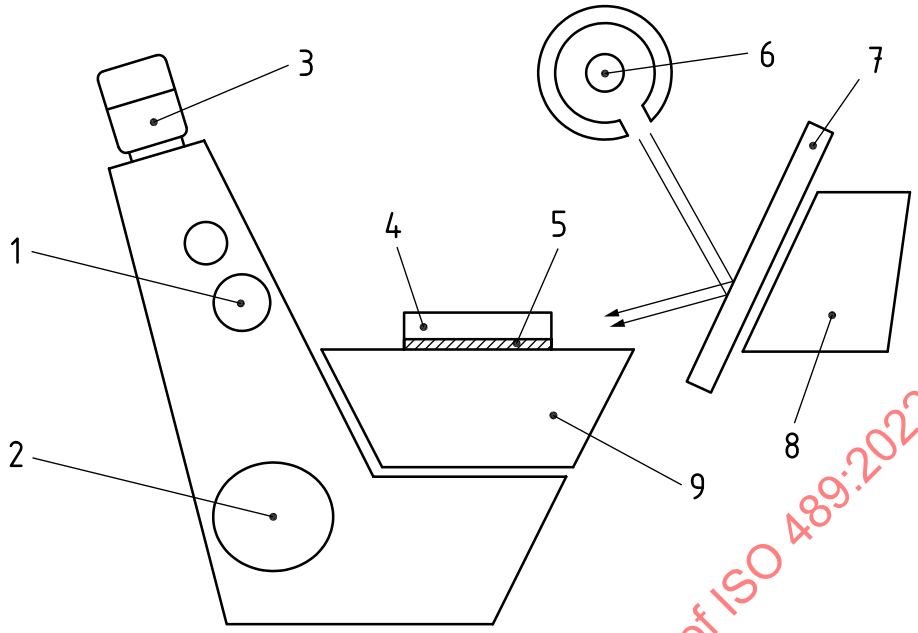
### 7.1.2 Transparent sheet

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.

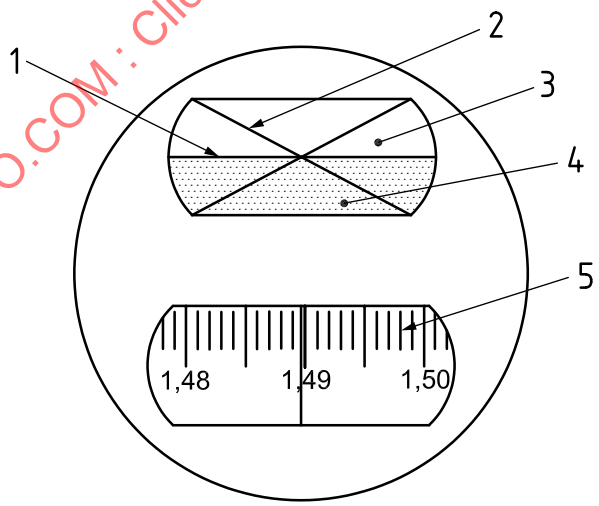
The dispersion, if required, can be found by noting the compensator drum reading and using this, together with the value of the refractive index, to read the dispersion from a chart supplied with the instrument.




**Key**

1 compensator drum
2 adjusting handle
3 eyepiece
4 specimen
5 contacting liquid
6 source of light
7 opal-coloured reflective plate
8 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

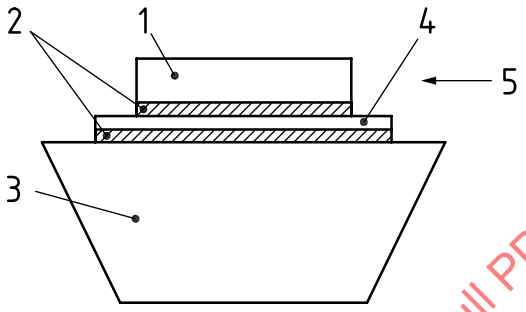
**Figure 2 — Refractometer field of vision**

### 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**

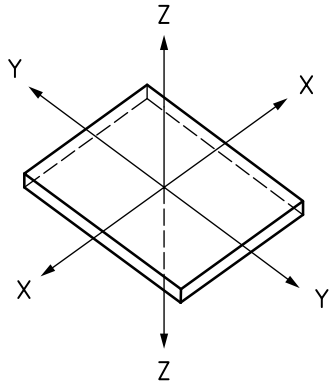
| | | | |
|---|---|---|---|
| 1 | glass plate | 4 | film specimen |
| 2 | contacting liquid | 5 | light |
| 3 | main prism | | |

**Figure 3 — Method for measuring refractive index of film**

### 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.




**Key**

X at right angle to the machine direction

Y machine direction

Z thickness

**Figure 4 — Film with multiple refractive indices**

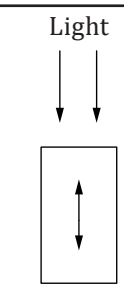
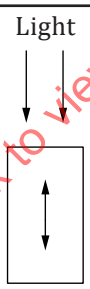
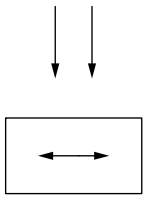
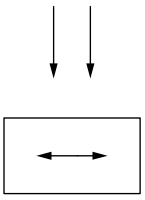
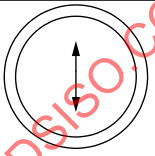
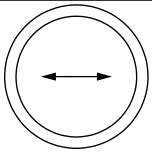
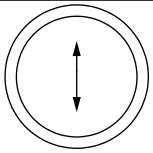
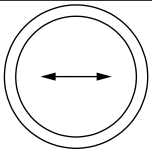
| **Measurement direction** | **Thickness** (Z axis) | **At right angle to machine direction** (X axis) | **Thickness** (Z axis) | **Machine direction** (Y axis) |
|---|---|---|---|---|
| Positioning of specimen with multiple refractive indices (machine direction indicated by arrows) | Light  | Light  | Light  | Light  |
| Polarizing direction (indicated by arrows) |  |  |  |  |

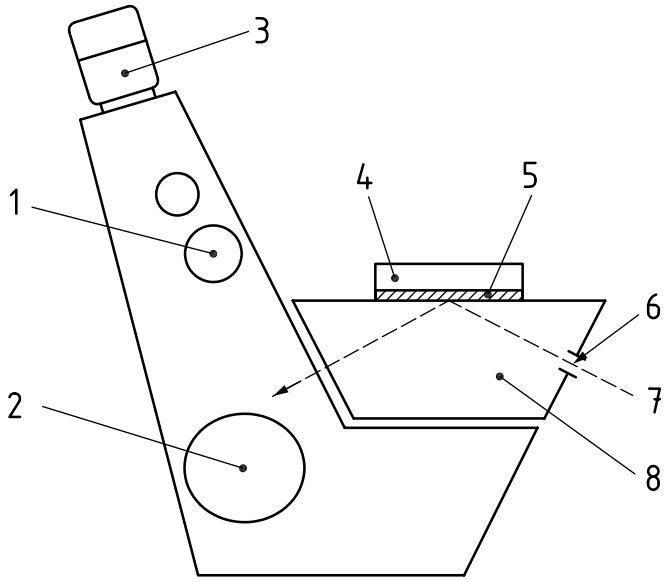
**Figure 5 — Combination of specimen-positioning direction and polarizing-filter direction**



### 7.1.5 Translucent, coloured and opaque material

For translucent, coloured and opaque materials, the reflection mode of the Abbe refractometer shall be used. In this mode, the light enters the prism through an upper window and reflects at the interface between the prism and the specimen as shown in Figure 6.

NOTE In cases of translucent, coloured or opaque material, it is difficult to measure the refractive index by the transmission method because of a lack of reflected light. In such cases, it is possible to measure refractive indices by the reflection mode. When the reflection mode is used, the bright field and the dark field are inverted, and their contrast becomes poor.




**Key**

| | | | |
|---|---|---|---|
| 1 | compensator drum | 5 | contacting liquid |
| 2 | adjusting handle | 6 | window for lighting |
| 3 | eyepiece | 7 | light |
| 4 | specimen | 8 | main prism |

**Figure 6 — Method for measuring refractive index of translucent, coloured and opaque materials**



## 7.2 Method B

Carry out the following determination at (23 ± 0,5) °C.

Place a small amount of an immersion liquid (4.2.3) on a slide. The immersion liquid shall be of a known refractive index (see Table 2) which is close to that of the material under test.

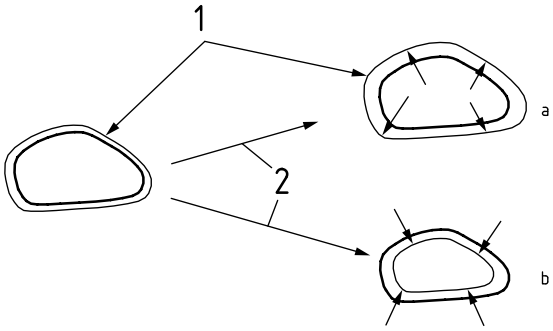
If the refractive index of the material to be examined is unknown, it is recommended that an immersion liquid with a refractive index of about 1,56 be used.

Place some particles of the material to be examined into the liquid on the slide and add a cover slip. Align the condenser and stop it down to give a narrow beam of axial illumination.

Place the sample preparation on the microscope stage and focus on the particles. Slightly defocus by increasing the separation between the microscope objective and the sample preparation. The Becke line, seen as a bright halo around or within the particle, will move towards the medium having the higher refractive index as shown in Figure 7.

Repeat the test with preparations in other immersion liquids with a known refractive index and particles of the material to be examined until a match is found, or until the index of the test sample is found to be between two known indices in the series of liquid standards. The Becke line phenomenon does not appear when the microscope objective is raised or lowered if the refractive index of the material to be examined is equal to the refractive index of the immersion liquid used for the determination test.

Any bubbles which appear during the preparation are useful for checking the focus when the match of the sample and the immersion liquid is close.




**Key**

1 becke line

2 lift the lens-barrel of the microscope to defocus slightly

a Where the immersion liquid has a higher refractive index than that of the sample, the Becke line is seen moving towards the immersion liquid.

b In the opposite case, the Becke line is seen moving towards the particle.

**Figure 7 — Becke line and its movement**

## 8 Precision

The precision data is shown in Annex A.

## 9 Test report

The test report shall include the following information:

a) a reference to this document (including its year of publication), i.e. ISO 489:2022;

b) all details necessary for the complete identification of the material under test;

c) the method used (A or B);

d) the type of light source and the wavelength used;

e) the position in the original sample from which the specimen was cut (for method A only);

f) the dispersion, if measured (for method A only);

g) the type of immersion liquid used (for method B only);

h) the refractive index to the nearest significant figure warranted by the precision of the measurements (to more than three significant figures if the refractive index is expressed using method A);

i) any deviation, by agreement or otherwise, from the method specified;

j) the date of measurement.

# Annex A
(informative)

# Precision data

**A.1** An international trial involving eight laboratories was conducted in 1996 to determine the precision of the two methods. Measurements were made on eleven samples for method A and six samples for method B, among which three samples were of the same materials. The results are shown in Table A.1. No outliers were detected by Grubb's test.

The anisotropic specimens were carefully selected and their orientation direction was identified for the series of measurements performed using method A.

Reproducibility means closeness of agreement between individual results obtained with the same method on identical test material but under different conditions (different operators, different apparatus, different laboratories and/or at different times).

Repeatability means closeness of agreement between successive results obtained with the same method on identical test material, under the same conditions (same operator, same apparatus, same laboratory and at short intervals of time).

NOTE 1 In method A, PMMA cast sheet and fully shrunk and annealed PMMA cast sheet, which would be expected to have little orientation and optical stress, give a reproducibility standard deviation of less than 0,000 4. Others range from 0,000 7 to 0,008 depending on the scattering of the data due to local differences within the samples. In the case of the fully shrunk and annealed PMMA cast sheet, the reproducibility standard deviation is slightly smaller than that calculated from the data from the within-laboratory tests. A possible interpretation is that the values of reproducibility were inverted for this particular material because $s_R$ can only be greater than or equal to $s_{R_w}$. Nonetheless, the precision of this method is very high for this material and the difference between $s_R$ and $s_{R_w}$ is only 0,000 1 which corresponds to the detection limit of the method.

NOTE 2 In general, method B for granules gives worse reproducibility than method A. This is clearly shown by the data generated for sheet and granulated samples from the same material: for PMMA (2, 15) and PS (6, 17).

**Table A.1 — Interlaboratory trial data**

| No. | Method | Plastic material | Average refractive index $n_D^{23}$ | Within-laboratory reproducibility standard deviation $s_{R_w}$ | Reproducibility standard deviation $s_R$ |
|---|---|---|---|---|---|
| 1 | A | PMMA cast sheet | 1,491 1 | 0,000 36 | 0,000 41 |
| 2 | A | PMMA shrunk and annealed cast sheet | 1,491 4 | 0,000 58 | 0,000 46 |
| 3 | A | PC extruded sheet (machine direction) | 1,583 9 | 0,000 40 | 0,000 82 |
| 4 | A | PC extruded sheet (transversal direction) | 1,583 7 | 0,000 65 | 0,000 67 |