International Standard 489

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Plastics — Determination of the refractive index of transparent plastics

Plastiques — Détermination de l'indice de réfraction des plastiques transparents

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 489 was developed by Technical Committee ISO/TC 61, *Plastics.*

It was submitted directly to the ISO Council, in accordance with clause 6.11.2 of part 1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 489-1966, which had been approved by the member bodies of the following countries:

Australia	India	Spain
Austria	Italy	Sweden
Belgium	Japan	Switzerland
Chile	Netherlands	United Kingdom
Czechoslovakia	New Zealand	USA
Finland	Poland	USSR
France	Portugal	Yugoslavia
Germany, F.R.	Romania	
Hungary	South Africa, Rep. of	

No member body had expressed disapproval of the document.

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Plastics — Determination of the refractive index of transparent plastics

1 Scope and field of application

1.1 This International Standard specifies two methods of test for determining the refractive index of transparent plastics, namely

Method A: Refractometric method for measuring the refractive index of transparent cast, moulded or sheet materials by means of a refractometer. The method is recommended when great accuracy is required. It is not applicable to powdered or granulated transparent materials.

Method B: Immersion method (Becke line phenomenon) for determining the refractive index of powdered or granulated transparent materials by means of a microscope and making use of the Becke line phenomenon. Monochromatic light should, in general, be used to avoid dispersion effects. The accuracy of this method is about the same as that of method A.

1.2 Refractive index is a fundamental property which can be used for the control of purity and composition, for the identification of materials and for the design of optical parts.

The change of refractive index with temperature may give an indication of transition points of materials.

2 Apparatus and materials

2.1 Method A

2.1.1 Abbe refractometer or any other refractometer that can be shown to give the same results.

2.1.2 Source of white light.

2.1.3 Contacting liquid (see 5.1).

2.2 Method B

2.2.1 Microscope, having a magnifying power of at least X 200, an 8 mm objective of good quality, and a centring substage condenser capable of being stopped down to a very narrow axial beam.

2.2.2 Immersion liquids, with different refractive indices (see 5.2).

3 Preparation of test specimens

3.1 Method A

The test specimens cut from the sample should be of such a size as will fit on the face of the fixed half of the refractometer prisms.

The following dimensions are convenient:

width	6 mm
length	12 mm
thickness	3 mm

For maximum accuracy the surface of the test specimen in contact with the prism should be quite flat and well polished. Satisfactory contact between the test specimen and the prism is indicated by the dividing line between the light and dark halves of the eyepiece field appearing sharp and straight. A second surface with a fair polish is prepared perpendicular to the first and on one end of the test specimen.

These two polished surfaces should intersect along a sharp line without a bevelled or rounded edge.

For anisotropic materials, see 5.3.

3.2 Method B

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

The thickness of the particles should be significantly less than the working distance of the microscope objective.

4 Procedure

4.1 Method A

The procedure for the Abbe refractometer is described here. For other refractometers the procedure may be modified, if necessary.

Carry out the determination at 20 ± 0,5 °C.

Place a small drop of the contacting liquid on the polished surface of the test specimen and place it in firm contact with the surface of the prism with the polished edge of the specimen towards the source of light. Adjust the index arm of the refractometer until half of the eyepiece field is dark. Adjust the compensator (Amici prisms) drum until all colour is removed from the field, after which adjust the index arm 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.

Read the refractive index of the material for the sodium D line from the instrument scale.

Precision: about 0,001.

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

4.2 Method B

Carry out the determination at 20 ± 0,5 °C.

Place a small amount of an immersion liquid of known refractive index (see 5.2) close to that of the material under test on a slide. (If the approximate 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.) Bring some particles of the material to be examined into the liquid on the slide and add a cover slip. The condenser should be aligned and stopped down to give a narrow beam of axial illumination.

Place the preparation on the stage of the microscope. After focusing the microscope on the centre of the preparation and then moving upwards until the upper part of the preparation is focused, the Becke line (a narrow luminous circle surrounding the material in the liquid) is seen moving towards the medium having the higher refractive index.

If the focus is lowered, the Becke line moves towards the medium having the lower refractive index.

Repeat the test with preparations of other immersion liquids with 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. When the Becke line phenomenon does not appear, the refractive index of the material to be examined is equal to the refractive index of the immersion liquid used for the determination test. The Becke line phenomenon does not set in when the focus is raised or lowered.

A few bubbles in the preparation are a useful check on focusing when the match of sample and liquid is close.

Precision: about 0,001 when experienced technicians use the method with extreme care; for thicknesses of test specimens less than 0,030 mm, the precision is greater than 0,001.

5 Notes on procedure

5.1 The contacting liquid should have a refractive index that is higher than that of the material to be examined and should not soften, attack or dissolve the plastics material.

The liquids listed in table 1 may be used for the respective plastics materials.

Table 1

Plastics material	Contacting liquid
Cellulose derivatives	Monobromonaphthalene or aniseed oil
Fluorine-containing polymers	Monobromonaphthalene
Urea resins	Monobromonaphthalene or aniseed oil
Phenolic resins	Monobromonaphthalene
Acrylic resins	Saturated aqueous solution of zinc chloride made slightly acid
Polyethylenes	Monobromonaphthalene
Polyamides	Monobromonaphthalene
Polyesters	Monobromonaphthalene
Polyisobutylene	Saturated aqueous solution of zinc chloride made slightly acid or saturated potassium mercury(II) iodide solution
Polymethyl methacrylate	Saturated aqueous solution of zinc chloride made slightly acid
Polystyrene	Saturated potassium mercury(II) iodide solution
Vinyl resins (in general)	Monobromonaphthalene
Polyvinyl chloride	Saturated potassium mercury(II) iodide solution or in some cases monobromonaphthalene

5.2 The immersion liquids listed in table 2 with known refractive index can be used separately and can give mixtures differing by increments of the desired accuracy, for example a difference of 0,002 to within ± 0,001 accuracy.

Table 2

Immersion liquid	Refractive index at 20 °C, n_D^{20}
n-Butyl carbonate	1,411
Tri-*n*-butyl citrate	1,445
n-Butyl phthalate	1,492
Monobromonaphthalene	1,658
Di-iodomethane (methylene iodide)	1,742
Aqueous solutions of mercury(II) potassium iodate	1,419 to 1,733*
Silicone oils	1,37 to 1,56*

* Useful range for the purpose of the test.

5.3 In the case of anisotropic samples such as injection or extrusion mouldings, different values of refractive index may be found when measurements are made in different parts of the sample. In such cases, test specimens are made with their polished surfaces parallel and perpendicular to the direction of orientation and the measurements are made in different parts of the test specimen by moving it into different positions on the surface of the prism.

A range should only be reported if it exceeds the precision of the measurements.

6 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) identification of the material under test;

c) the method used (A or B);

d) the position on the original sample from which the test specimen was cut (for method A only);

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

f) the refractive index to the nearest significant figure warranted by the precision of the measurements (if with method A the refractive index is expressed to more than three significant figures, the wavelength of light for which the measurement was made shall also be reported);

g) any deviation, by agreement or otherwise, from the procedure specified.

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