INTERNATIONAL STANDARD

ISO 489

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

International Standard ISO 489 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 489:1983), of which it constitutes a technical revision.

Plastics — Determination of refractive index

1 Scope

This International Standard 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. The method is recommended when great accuracy is required. It is not applicable to powdered or granulated material.

— 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 should, in general, be used to avoid dispersion effects. The accuracy of this method is about the same as that of method A. It is applicable to isotropic translucent, coloured materials but is not applicable to opaque materials nor to anisotropic materials.

NOTE 1 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 may give an indication of transition points of materials.

NOTE 2 The accuracy of method B is approximately the same as that of method A when an experienced operator uses the method with extreme care (see clause 7).

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method.*

3 Apparatus and materials

3.1 Method A

3.1.1 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 (3.1.4) shall be provided for the specimens and prisms.

3.1.2 White or sodium lamp, used as a source of light.

3.1.3 Contacting liquid.

WARNING — The contacting liquid may present an environmental hazard during handling, storage and disposal. Verify its toxicity and follow 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

3.1.4 Temperature-controlled water bath, capable of maintaining the temperature at (23 ± 0,5) °C for the main prism, sub-prism and the specimen.

NOTE The circulating water should be distilled water.

3.2 Method B

3.2.1 Microscope, having a magnifying power of at least × 200, an objective giving approximately × 20 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.

3.2.2 Monochromatic light, usually the sodium D line, having a wavelength of 589 nm, is used as the light source for the microscope.

3.2.3 Immersion liquids, with different refractive indices.

WARNING — The contacting liquid may present an environmental hazard during handling, storage and disposal. Verify its toxicity and follow 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 (for example, a difference of 0,002 to within ± 0,001). 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.	

4 Preparation of test specimens

4.1 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:

width: 8 mm

length: 20 mm

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:

width: 8 mm

length: 20 mm

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

For anisotropic material, see 6.1.3.

4.2 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.

4.3 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.

5 Conditioning

5.1 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.

5.2 Set up the test apparatus in an atmosphere maintained at (23 ± 2) °C and (50 ± 5) % relative humidity.

6 Procedure

6.1 Method A

If an Abbe refractometer (3.1.1) 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.

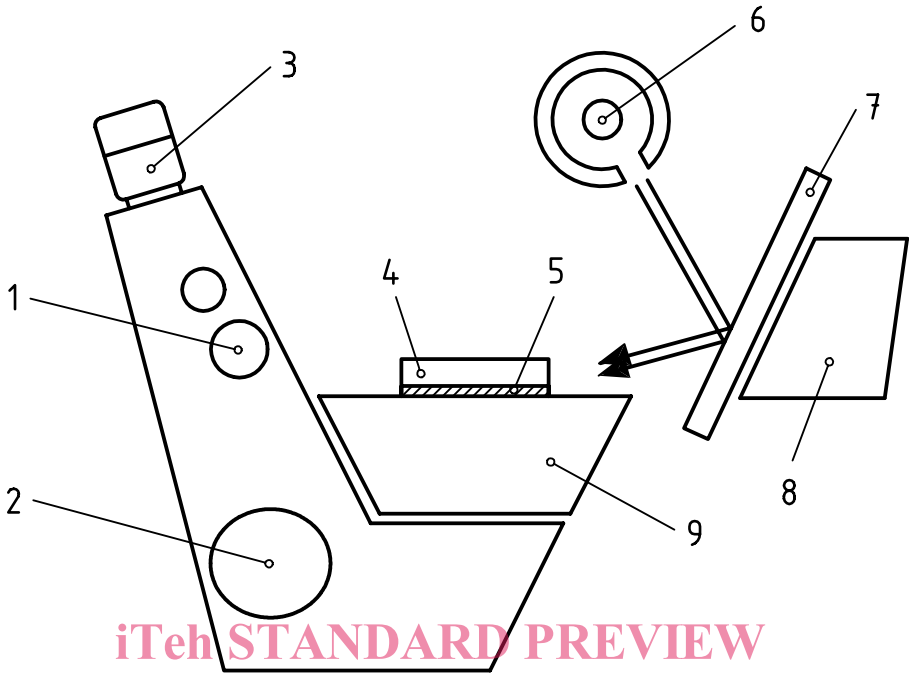
6.1.1 Transparent sheet

Place a small drop of the contacting liquid (3.1.3) 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 index arm 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 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 as shown in Figure 2.

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

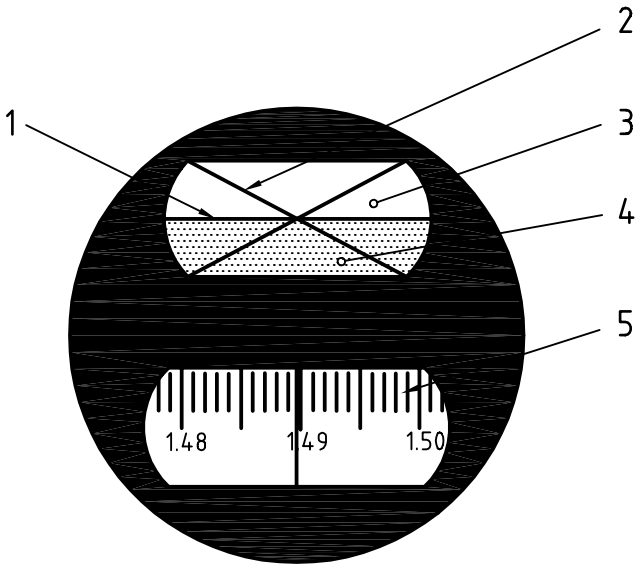
The dispersion, if required, can be found by noting the compensator drum reading and using this, together with the value of the refractive index, to read the dispersion from a chart supplied with the instrument.



Key

1	Compensator drum	4	Specimen	7	Opal-coloured reflective plate
2	Index arm	5	Contacting liquid	8	Sub-prism
3	Eyepiece	6	Source of light	9	Main prism

Figure 1 — Method for measuring refractive index of transparent sheet



Key

1	Boundary line	4	Dark half of eyepiece field
2	Cross-hair lines	5	Scale for refractive index
3	Light half of eyepiece field		

Figure 2 — Refractometer field of vision