AST INTERNATIONAL

**Designation:** D 542 - 9500

An American National Standard

# Standard Test Method for Index of Refraction of Transparent Organic Plastics<sup>1</sup>

This standard is issued under the fixed designation D 542; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

# 1. Scope\*

- 1.1 This test method covers a procedure for measuring the index of refraction of transparent organic plastic materials.
- 1.2 A refractometer method is presented. This procedure will satisfactorily cover the range of refractive indices found for such materials. Refractive index measurements require optically homogeneous specimens of uniform refractive index.

Note 1—This test method and ISO 489 are technically equivalent.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

# 2. Referenced Documents

2.1 ASTM Standards:

D 618 Practice for Conditioning Plastics

C 162 Terminology of Glass and Electrical Insulating Materials for Testing Glass Products<sup>2</sup>

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D<sup>2</sup>0 on Plastics and is the direct responsibility of Subcommittee D<sup>2</sup>0.40 on Optical Properties. Current edition approved—Sept. Nov. 10,—1995. 2000. Published—November 1995. January 2001. Originally published as D 542 – 39. Last previous edition—D 542 – 90. A five-year review of this test method revealed that no technical changes are needed. Changes include (a) the addition of an ISO statement, (b) addition of Keywords section, (c) deletion of all references to non SI units, and (d) editorial changes for improved clarity. D 542 – 95.



D-883 Terminology Relating to 618 Practice for Conditioning Plastics for Testing<sup>3</sup>

D 883 Terminology Relating to Plastics<sup>3</sup>

D 1898 Practice for Sampling of Plastics<sup>3</sup>

E 284 Terminology of Appearance<sup>4</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>5</sup>

2.2 ISO Standard:

ISO 489-1983 Determination of the Refractive Index of Transparent Plastics—Part A<sup>6</sup>

# 3. Terminology

- 3.1 Definitions:
- 3.1.1 For definions of terms used in this test method, see Terminologies D 883 and E 284.
- 3.1.2 dispersion—variation of refractive index with wave length of light.

C 162, C14

3.1.23 index of refraction, n—the numerical expression of the ratio of the velocity of light in a vacuum to the velocity of light in a substance at a specified wavelength.

E 284, E12

#### 4. Significance and Use

4.1 This test method measures a fundamental property of matter which is useful for the control of purity and composition for simple identification purposes, and for optical parts design. This test method is capable of readability to four figures to the right of the decimal point.

# 5. Apparatus

- 5.1 The apparatus for this test method shall consist of an Abbe' refractometer (Note 2), a suitable source of white light, and a small quantity of a suitable contacting liquid (Note 2 and Note 3).
  - Note 2—Other suitable refractometers can be used with appropriate modification to this procedure as described in Section 7.
- Note 3—A satisfactory contacting liquid is one which will not soften or otherwise attack the surface of the plastic within a period of 2 h of contact. The index of refraction of the liquid must be higher, but not less than one unit in the second decimal place, than the index of the plastic being measured; for example,  $n_d$  of the sample = 1.500,  $n_d$  of the contacting liquid  $\geq$ 1.510.

#### 6. Sampling

- 6.1 Samples shall be in accordance with the pertinent considerations outlined in Practice D 1898.
- 6.2 Samples may be drawn from any materials presentation (for example, pellets, film, sheet, fabricated articles, etc.) which permits preparation of a satisfactory specimen as described herein.

# 7. Test Specimens

- 7.1 The test specimen shall be of a size that will conveniently fit on the face of the fixed half of the refractometer prisms (Note 4). A specimen measuring 6.3 by 12.7 mm on one face is usually satisfactory.
- Note 4—For maximum accuracy in the refractometer method, the surface contacting the prism must be flat. This surface can be judged for flatness, provided the specimen has been satisfactorily polished, by observing the sharpness of the dividing line between the light and dark field as viewed in the ocular. A sharply defined straight dividing line indicates satisfactory contact between the specimen and prism surfaces.
- 7.2 The surface to be used in contact with the prism shall be flat and shall have a good polish. A second edge surface perpendicular to the first and on one end of the specimen shall be prepared with a fair polish (Note 5). The polished surfaces shall intersect without a beveled or rounded edge.
- Note 5—It has been found possible to prepare a satisfactorily polished surface by hand polishing small specimens on an abrasive material backed by a piece of plate glass. Fine emery paper (for example, No. 000 Behr-Manning polishing paper) followed by a polishing rouge compound suspended in water on a piece of parchment paper has successfully been used as the abrasive to produce a polished surface.
  - 7.3 A minimum of three specimens should be prepared and measured.

# 8. Conditioning

8.1 Conditioning—Condition the test specimens at 23  $\pm$  2°C and 50  $\pm$  5 % relative humidity for not less than 40 h prior to the test in accordance with Procedure A of Practice D 618. In cases of disagreement, the tolerances should be  $\pm$ 1°C and  $\pm$ 2 % relative humidity.

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 08.01. 15.02.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol-14.02. 08.01.

<sup>&</sup>lt;sup>4</sup> Available from American National

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036., Vol 06.01.

<sup>&</sup>lt;sup>5</sup> Supporting data are available from

<sup>&</sup>lt;sup>5</sup> Annual Book of ASTM-Headquarters. Request Research Report RR: D20 - H54. Standards, Vol 14.02

<sup>&</sup>lt;sup>6</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

8.2 Test Conditions—Conduct tests at  $23 \pm 2^{\circ}$ C and  $50 \pm 5^{\circ}$ % relative humidity, unless otherwise directed in a pertinent specification. In cases of disagreement, the tolerances shall be  $\pm 1^{\circ}$ C and  $\pm 2^{\circ}$ % relative humidity. If the index of refraction of the material is found to be highly temperature dependent, then the temperature shall be accurately controlled to  $23 \pm 0.2^{\circ}$ C.

#### 9. Procedure

9.1 Remove the hinged illuminating prism from the refractometer, if necessary. Place a source of diffuse polychromatic light so that even illumination is obtained along the plane of the surface of contact between the specimen and the refractometer prism. Place a small drop of the suitable contacting liquid on the polished surface of the specimen and then place the specimen in firm contact with the surface of the prism with the polished side of the specimen toward the specified light source. Determine the index of refraction in the same manner as specified for liquids. This shall be done by moving the index arm of the refractometer until the field seen through the eyepiece is one-half dark. Adjust the compensator (Amici prisms) drum to remove all color from the field of the ocular. Adjust the index arm using the vernier until the dividing line between the light and dark portions of the field exactly coincides with the intersection of the cross hairs as seen in the eyepiece. Read the value of the index of refraction for the Sodium D Line (see Note 6). Determine the dispersion by reading the compensator drum and applying this figure, along with the index of refraction, to a chart or table supplied with the instrument.

Note 6—Sodium light from a sodium burner or discharge lamp is of use in increasing the precision of this test method as well as making the reading of the refractometer easier.

#### 10. Report

- 10.1 Report the following information:
- 10.1.1 The index of refraction to the nearest significant figure warranted by the accuracy and duplicability of the measurement. If the index is specified to more than three significant figures, the wavelength of light for which the measurement was made shall be specified.

Note 7—In the case of nonisotropic materials, for example, injection- and compression-molded materials, the index observed will be the average value for a thin layer of small area at a point of contact near the center of the refractometer prism. For a complete and accurate determination of the variation of the index throughout the test specimen, it is necessary to make the measurement at more than one point on the surface and within the body of the material. This can be done by preparing a contacting surface both perpendicular and parallel to the molding pressure or flow. After the test specimen is contacted to the prism it may be translated carefully for short distances along the prism surface in the direction of the light source while the variation of index is followed with the refractometer. This procedure should be repeated a sufficient number of times and for a sufficient number of specimens to determine the range of indices involved. The average value and the range of the index readings obtained from these specimens shall be reported if the range exceeds the accuracy of the measurement. If the variations in index are systematic with the orientation of the test specimen, and if these variations exceed those found between specimens of the same material, the nature of these variations shall be reported with the average value.

Note 8—Care should be taken to work rapidly to avoid changes in the refractive index of the plastic due to its absorption of the contacting liquid.

- 10.1.2 The temperature in degrees Celsius at which the index was measured.
- 10.1.3 If available, the dispersion shall be reported along with the index of refraction.

# 11. Precision and Bias <sup>7</sup>

- 11.1 *Precision*—A limited round robin was conducted in 1978, involving seven materials (four polymers and three glass standards) tested by four laboratories. An individual determination is a test result. Each laboratory obtained six test results for each material (three determinations on two different days). Statistical tests indicated there is no significant difference in the averages or variances from Day 1 to Day 2 so that both days' data were combined. The data in Table 1 and Table 2 are based on this round robin.
- 11.1.1 Because of the limited number of laboratories participating in this round robin, interpretation of  $S_R$  and  $I_R$  is not recommended.

**TABLE 1 Precision** 

Material	Average	$S_r^A$	$S_R^{\ B}$	$I_r^C$	$I_R^D$
Glass Standard No. 1	1.356	0.001	0.002	0.003	0.006
PTFE	1.366	0.001	0.002	0.003	0.006
PVCA	1.477	0.002	0.002	0.006	0.006
Glass Standard No. 2	1.490	0.002	0.003	0.006	0.008
PA66	0.537	0.002	0.003	0.006	0.008
Glass Standard No. 3	1.567	0.001	0.002	0.002	0.006
PF	1.614	0.004	0.004	0.011	0.011

 $<sup>^{</sup>A}$  S <sub>r</sub> = within-laboratory standard deviation.

 $<sup>\</sup>frac{^{7}\,\text{Supporting data are available from ASTM Headquarters. Request Research Report RR: D20-1154.}$ 

 $<sup>^{</sup>B}S_{R}$  = between-laboratories standard deviation.

 $<sup>^{</sup>C}I_{r} = 2.83 S_{r}$ 

 $<sup>^{</sup>D}I_{R}^{r} = 2.83 S_{R}.$ 

#### **TABLE 2 Bias**

Material	Certified Value	Test Average	Bias	Statistically Significant <sup>A</sup>	
Glass Standard No. 1	1.3554	1.3556	+0.0002	No	
Glass Standard No. 2	1.4903	1.4897	-0.0006	No	
Glass Standard No. 3	1.5684	1.5673	-0.0011	Yes	

 $<sup>^{</sup>A}$ "t"-test (α = 0.05) = bias/ $S_{r}$ / $\sqrt{n}$ ).

- 11.1.2 Anyone wishing to participate in the development of precision and bias data for this test method should contact the Chairman, Subcommittee D20.40, through ASTM Headquarters.
- Note 9—The following explanations of  $I_r$  and  $I_R(11.3-11.3.3)$  are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 and Table 2 should not be rigorously applied to acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories.
- 11.2 Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 11.3-11.3.3 would then be valid for such data.
- 11.3 Concept of  $I_r$  and  $I_R$ —If  $S_r$  and  $S_R$  have been calculated from a large enough body of data, and for test results that were from testing one specimen:
- 11.3.1  $I_r$ : Repeatability—Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the  $I_r$  value for that material
- 11.3.2  $I_R$ : Reproducibility—Comparing two test results for the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the  $I_R$  value for that material.
  - 11.3.3 Any judgment in accordance with 11.3.1 and 11.3.2 would have an approximate 95 % (0.95) probability of being correct.
  - 11.4 Bias—Bias is the systematic error which contributes to the difference between a test result and a true (or reference) value.
  - 11.4.1 The data for bias was determined from the three certified glass standards and is reported in Table 2.

# 12. Keywords

12.1 dispersion; index of refraction

# **APPENDIX**

(Nonmandatory Information)

#### X1. REFRACTIVE INDEX ROUND ROBIN

#### **SUMMARY OF CHANGES**

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also contain descriptions of the changes or reasons for the changes, or both.

#### D 542–00:

TABLE X1.1 Test of Variance Between Days

Material	$S_r(1)^A$	$S_r(2)^A$	F <sup>B</sup>	$S_R(1)^A$	$S_R(2)^A$	F <sup>B</sup>
Standard Glass No. 1	0.011	0.007	2.47	0.0016	0.0017	1.13
PTFE	0.0015	0.0016	1.14	0.0017	0.0016	1.13
PVAC	0.0023	0.0016	2.07	0.0024	0.0016	2.25
Standard Glass No. 2	0.0023	0.0021	1.20	0.0029	0.0026	1.24
PA66	0.0021	0.0025	1.42	0.0032	0.0025	1.64
Standard Glass No. 3	0.0008	0.0007	1.31	0.0019	0.0021	1.22
PF	0.0050	0.0051	1.04	0.0050	0.0051	1.04

A(1)/(2) - (1) = day 1 data, (2) = day 2 data.

 $<sup>^{</sup>B}$  F 0.95 (2,2) = 19.00.

# TABLE X1.2 "t" Test Between Days

Material	X (1) <sup>A</sup>	X (2) <sup>A</sup>	t <sup>B</sup>
Standard Glass No. 1	1.3557	1.3554	0.230
PTFE	1.3658	1.3656	0.091
PVCA	1.4760	1.4777	0.607
Standard Glass No. 2	1.4893	1.4902	0.289
PA66	1.5369	1.5377	0.245
Standard Glass No. 3	1.5669	1.5677	0.752
PF	1.6145	1.6137	0.112

<sup>&</sup>lt;sup>A</sup> There is no significant difference in averages or variances within or between laboratories from Day 1 to Day 2.

# (1) Revised Section 3, Terminology.

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<sup>&</sup>lt;sup>B</sup> t = 0.95 (4) = 2.776