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Standard Specification for Liquid Optical Adhesive[1]

This standard is issued under the fixed designation D2851; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers liquid optical adhesive for use in bonding glass to glass or other transparent adherends.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are provided for information purposes only.

1.3 The following precautionary caveat pertains only to the test method portion, Section 6, of this specification: *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.* Specific precautionary statements are given in 6.1.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D542 Test Method for Index of Refraction of Transparent Organic Plastics

D897 Test Method for Tensile Properties of Adhesive Bonds

D904 Practice for Exposure of Adhesive Specimens to Artificial Light

D907 Terminology of Adhesives

D1084 Test Methods for Viscosity of Adhesives

E308 Practice for Computing the Colors of Objects by Using the CIE System

3. Terminology

3.1 *Definitions*—Many terms in this specification are defined in Terminology D907.

4. Significance and Use

4.1 The bond strength tests provide reasonably accurate information with regard to the bond strength of the adhesives. Bond strength data may be suitable for specification acceptance, service evaluation, manufacturing control, research, and development. Bond strength tests are not considered significant for applications differing widely from the test in rate and type of loading.

5. General Requirements

5.1 The adhesive shall be in liquid form and free of solvent in order to avoid bubble formation in the adhesive layer. Each component shall be completely reactive (without residual volatile products). The adhesive may be heat-, catalyst-, or radiation-cured.

5.1.1 *Volatility*—Volatile content of the adhesive shall not exceed 0.5 %, unless otherwise agreed upon between the manufacturer and the purchaser.

5.1.2 *Viscosity*—The viscosity of the adhesive shall be within a well-defined range as agreed upon between the manufacturer and the purchaser.

5.1.3 *Color*—The color of the adhesive shall not exceed the color of a platinum-cobalt standard solution No. 300.

5.1.4 *Cleanliness*—The number and size of foreign particles found in 100 mL of the adhesive shall not exceed 5 particles in the size range from 10 to 100 µm (0.1 mm), and none larger than 100 µm. The number and size of foreign particles in the catalyst required to cure 100 mL of the adhesive shall not exceed 2 particles in the size range from 10 to 100 µm, and none larger than 100 µm.

5.1.5 *Refractive Index*—The refractive index of the cured adhesive shall be within a well-defined range as agreed upon between the manufacturer and the purchaser.

5.1.6 *Stability*—The liquid adhesive shall not change in viscosity by greater than 20 % of its original viscosity nor show any formation of solids, when tested as described in 6.6.

5.1.7 *Light Transmission*—Visible light transmission through a bonded glass doublet (two glass disks bonded as described in 6.8) shall not be less than 98.5 % of the total light transmitted through a single glass disk. There shall be no change in light transmission of a glass doublet after the environmental test (see 6.9).

5.1.8 *Environmental Test*—Separation within the adhesive layer or from either glass surface of the glass doublet shall not exceed 1.0 mm in depth nor extend beyond 180° of the periphery of the adhesive layer (see 6.9).

[1] This specification is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.60 on Adhesive Material Classification System.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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5.1.9 *Bond Strength*—The tensile strength of a bonded glass doublet after being subjected to the environmental test (see 6.9) shall be greater than 200 psi (1.38 MPa) or any value agreed upon between the manufacturer and the purchaser.

6. Test Methods

6.1 *Volatility*:

6.1.1 Thoroughly clean three petri dish bottoms, 95-mm outside diameter, by washing with detergent and a scrub brush. Rinse with warm running tap water. Place petri dishes in an air-circulating oven for 30 min at 65 ± 2°C (150 ± 4°F). Raise temperature to 110 ± 2°C (230 ± 4°F) for 1 h. Remove petri dishes from oven and place in a desiccator. After petri dishes have cooled to room temperature, approximately 2 h, weigh each dish to the nearest 0.1 mg. (**Warning**—In addition to other precautions, do not handle petri dishes with bare hands. Use tongs.)

6.1.2 Replace each petri dish in the oven at 110 ± 2°C (230 ± 4°F) for 1 h. Repeat cool-down procedure. Reweigh each petri dish to the nearest 0.1 mg. If weight agrees to within ±0.2 mg of initial weight, proceed to 6.1.3. If weight exceeds this ±0.2-mg range, repeat process until a constant weight for each petri dish is obtained.

6.1.3 To each petri dish add approximately 10 g of adhesive, and mix with catalyst or any component part required for curing. Weigh each dish to the nearest 0.1 mg. Cure the adhesive by following the procedure described by the manufacturer. Then place each sample in an oven at 105 ± 2°C (220 ± 4°F) to a constant weight (same procedure as in 5.1.1 but at 105°C). Reweigh each sample to the nearest 0.1 mg and calculate the percent volatile matter as follows.

$$\text{Volatile matter, \%} = [(A - B)/A] \times 100 \quad (1)$$

where:

A = weight of original sample, and
B = weight of cured adhesive taken to constant weight.

6.2 *Viscosity*—Measure the viscosity of the adhesive in accordance with Test Methods D1084. (Brookfield, Method B, or any other suitable viscometer may be used.)

6.3 *Color*—Determine the color of the adhesive by comparing it with platinum-cobalt (Pt-Co) standards. Place 100 mL of adhesive in a 100-mL Nessler tube. Make color observations by placing the adhesive-filled Nessler tube vertically over a sheet of white paper while looking down and through the filled tube. Compare the color observed with a standard Nessler tube, containing 100 mL of the Pt-Co standard held in the same manner.

6.3.1 Prepare the Pt-Co standard solutions by dissolving 1.246 g of potassium platinic chloride and 1000 g of cp crystalline cobaltous chloride hexahydrate in a solution of 300 mL of distilled water and 100 mL of concentrated hydrochloric acid. Dilute to 1000 mL in a volumetric flash using distilled water. This shall be the concentrated stock solution that has a Pt-Co number of 500. Dilute the concentrated stock solution with distilled water as shown below to obtain the desired Pt-Co standard.

Pt-Co Number	Stock Solution of Pt-Co 500, mL	Distilled H_2O to Be Added, mL
500	100	0
450	90	10
400	80	20
350	70	30
300	60	40
250	50	50
200	40	60
150	30	70
100	20	80
75	15	85
50	10	90
0	0	100

Store the stock solutions in clean glass bottles and stopper them to prevent evaporation loss.

6.4 *Foreign Particle Content*—Inspect a 100-mL sample of the liquid adhesive and a sample of its catalyst for cleanliness by filtering each through a low-form Gooch crucible fitted with a filter membrane of 5.0 to 10.0-µm pore size and a color suitable to easily detect and examine foreign particles. Measure the size of the particles by means of a calibrated traversing microscope (10×) or other equivalent means. If the viscosity of the sample is too high, dilute it with any clean and suitable solvent to permit easy flow of the liquid through the filter membrane.

6.5 *Refractive Index*—Prepare a solid adhesive specimen by measuring approximately 6.3 by 12.7 by 12.7 mm (0.25 by 0.5 by 0.5 in.). Make two adjacent surfaces measuring 6.3 by 12.7 mm flat (0.25 by 0.5 in.), smooth, and perpendicular to each other. Polish by means of a rotating (500 r/min) felt-covered polishing wheel wetted with a solution of cerium oxide (polishing grade) in water. Use the polished surface in contact with the prism. Determine the index of refraction in accordance with the refractometric method described in Test Method D542. The contacting fluid between the prism and the adhesive surface may be bromonaphthalene. A fluid with a closer matching refractive index can be prepared by diluting the bromonaphthalene with refined mineral oil (medicinal grade).

6.6 *Stability*—Place the adhesive in an air-circulating oven at 57 ± 1.1°C (135 ± 2°F) for 168 h or at any temperature or time agreed upon between the manufacturer and the user.

6.7 *Light Transmission*—Determine the visible light transmission in accordance with Practice E308. The specimen shall consist of a bonded glass doublet prepared as described in 6.8. Make a comparison with the light transmission through one glass disk similar to the one used for preparing the doublet. Clean the glass disk as described in preparing the disk for bonding. All three doublets shall pass. Retain for environmental test (see 6.9).

6.8 *Glass Doublet Preparation*—The flat and polished plate glass (silvering grade) disks shall be 31.75± 0.8 mm (1.25 ± 0.03 in.) in diameter and 6.35 mm (0.25 in.) thick, with edges lightly ground to remove their sharpness. Clean with a camel's hair brush that is wet with ethanol. Rinse the flat surface to be bonded in a stream of clean ethanol and then allow it to dry in a desiccator for 1 h. Apply the adhesive, prepared in accordance with manufacturer's instructions, dropwise to the center of one of the clean and dry disks. Place a second disk over the drop of adhesive with a rotary motion so that the adhesive

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