Standard Practice for
Determining Chemical Resistance of Thermosetting Resins Used in Glass-Fiber-Reinforced Structures Intended for Liquid Service[1]

This standard is issued under the fixed designation C581; 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.

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

1. Scope

1.1 This practice is designed to evaluate, in an unstressed state, the chemical resistance of thermosetting resins used in the fabrication of reinforced thermosetting plastic (RTP) laminates. This practice provides for the determination of changes in the properties, described as follows, of the test specimens and test reagent after exposure of the specimens to the reagent: hardness of specimens, weight change thickness, appearance of specimens, appearance of immersion media, and flexural strength and modulus.

1.1.1 This practice is also useful to evaluate other factors, such as surfacing veils and the effect of resin additives, on the chemical resistance of the resin.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

NOTE 1—There is no known ISO equivalent to this standard.

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:*[2]

D790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials

D883 Terminology Relating to Plastics

D2563 Practice for Classifying Visual Defects in Glass-Reinforced Plastic Laminate Parts

D2583 Test Method for Indentation Hardness of Rigid Plastics by Means of a Barcol Impressor

D2584 Test Method for Ignition Loss of Cured Reinforced Resins

3. Terminology

3.1 *Definitions of Terms:*

3.1.1 For definitions of terms associated with plastic materials, see Terminology D883.

4. Significance and Use

4.1 The results obtained by this practice shall serve as a guide in, but not as the sole basis for, selection of a thermosetting resin used in an RTP structure. No attempt has been made to incorporate into the practice all the various factors that will potentially affect the serviceability of an RTP structure when subjected to chemical environments. These factors will potentially include stress, different resin-to-glass ratios, and multiple veils.

5. Apparatus

5.1 *Hardness Testing Instrument*—This shall be as described in Test Method D2583.

5.2 *Flexural Properties Testing Apparatus,* in accordance with Test Methods D790.

5.3 *Thickness Measurement*—A micrometer suitable for measurement to 0.001 in. (0.025 mm).

5.4 *Containers,* of sufficient size, capacity, and inertness to allow total immersion of reinforced thermosetting plastic specimens in the specific corrosives chosen for testing. These containers shall, when necessary, be capable of maintaining liquid levels of volatile solutions, that is, solvents. This can be accomplished by the use of reflux condensers.

5.5 *Heating Apparatus*—A constant temperature oven, heating mantle, or liquid bath capable of maintaining temperature

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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.

within range of ±4.0°F (±2.2°C). Take proper precautions if the corrosives selected are flammable liquids.

5.6 *Analytical Balance,* suitable for accurate weighing to 0.001 g.

6. Reagents

6.1 The test media shall consist of the reagents or solutions to which the RTP laminates are to be exposed.

7. Test Specimens

7.1 *Standard Laminates*—Prepare standard fiber-reinforced laminates using identical reinforcement in all of the laminates. The laminates shall be constructed of the following materials:

7.1.1 *Surfacing Mat (Veil)*—A thin mat of fine fibers used primarily to produce a smooth, resin-rich surface on a reinforced plastic. The surfacing veil helps determine the thickness of the resin-rich layer, reduces microcracking and provides a non-wicking chemically–resistant layer. The surfacing veil shall be compatible with the resin, and manufactured with uniform fiber distribution and non-bundled fibers. The dry veil layer(s) shall be a minimum 10 mils in thickness and produce a 10 to 15 mil resin-saturated veil layer per 10 mils of dry veil. To eliminate the surfacing veil as a variable in corrosion tests, prepare each laminate within a test group with the same surfacing veil.

7.1.2 *Chopped Strand Mat*—Type E glass fiber with sizing and binder compatible with the resin. It is acceptable to use other glass fiber compositions, but using such alternate compositions shall be considered as variables for comparison to the standard.

7.1.2.1 Note that this practice applies to E-CR glass fiber, which is a type of E glass fiber. If the E glass fiber material is of the E-CR type that information shall be identified in the test report.

7.1.3 *Resin*—Catalyzed and promoted in accordance with the resin manufacturer's recommendation.

NOTE 2—It is acceptable to add fillers, such as antimony trioxide for improved fire performance or thixotropes for viscosity control, but it is possible that this will detract from the corrosion resistance of the test laminate.

7.2 *Dimensions and General Properties*—The laminates shall conform to the required dimensions and general properties of 7.2 and be fabricated in accordance with 7.3.

7.2.1 *Laminate Size*—A suitable laminate size has been found to be 26 by 33 in. (660 by 838 mm) after trimming. This laminate size is not restrictive and other dimensions are acceptable.

7.2.2 *Thickness*—The thickness of the cured standard laminate shall be between 0.120 and 0.140 in. (3.05 and 3.56 mm).

7.2.3 *Reinforcement Content*—The glass fiber and binder shall be 4.73 ± 0.47 oz/ft^2 (three layers of 1.5 oz/ft^2 chopped strand mat 4.5 oz/ft^2 having a nominal binder content of 3.5 % and two layers of 10 mil surfacing mat 0.23 oz/ft^2 having a nominal binder content of 7 %)—determined by preweighing the materials prior to construction of the laminate. This is equivalent to 23.6 weight % (12.5 volume %) glass fiber when using a resin having a cured specific gravity of 1.15. Such a laminate will have a thickness of 0.125 in. (3.18 mm). The use of resins having different specific gravities will result in different weight percentages of glass fiber, but the volume percentage of glass fiber will remain the same. When using synthetic organic fiber surfacing veil, the glass content shall be 4.50 ± 0.45 oz/ft^2 (three layers of 1.5oz/ft^2 chopped strand mat having a nominal binder content of 3.5 %).

7.2.4 *Hardness*—The hardness shall be at least 90 % of that of a fully-cured clear casting of the resin, or of a similarly constructed laminate as defined by the resin manufacturer. Hardness shall be determined in accordance with s5.1. Note that the use of synthetic veil will result in significantly lower hardness values. The hardness value will vary with the type of resin and number of plies of synthetic veil. The resin manufacturer needs to be contacted for the allowable Barcol hardness value of a laminate containing synthetic veils with the specific resin.

7.2.5 *Laminate Condition*—The laminate shall meet Acceptance Level I of Table I of Practice D2563.

7.3 *Fabrication of Standard Laminate*—The sequence of lay-up shall be as follows:

7.3.1 Apply catalyzed resin and a 10-mil (0.25-mm) surfacing mat on a flat surface covered with plastic release film or treated with a suitable release agent and roll to distribute resin.

NOTE 3—Use the following formula as a guide to determine the total weight of resin to be used. This is equivalent to 12.5 volume % glass fiber in the laminate. Grams resin equals grams glass fiber material per 7.2.3 times 2.82 *G*. Where *G* equals specific gravity of cured resin. It is acceptable to use excess resin due to loss by adhering to mixing containers, rollers, and other factors. A suggested amount of excess resin is 10 to 15 % by weight.

7.3.2 Follow with three plies of 1.5 oz/ft^2 chopped strand mat and resin. Roll after each ply to distribute and wet-out the chopped strand mat. Rolling with a serrated roller is acceptable after each ply to remove entrapped air but it shall be done in accordance with 7.3.4. The mat weight shall be within ±5 % of 1.5 oz/ft^2 upon weighing the full 26 by 33-in. cut (660 by 838-mm) piece, (or other full dimension used, 7.2.1.).

NOTE 4—Cut chopped strand mat so that the 26-in. dimension is across the width of the roll and the 33-in. dimension is along the machine direction of the mat. Mat weight variation will most commonly occur across the width of the mat. If a wide roll of mat, 52 in. (1320 mm) or greater, is used, place the two plies of mat in the laminate such that the center cut of one ply is placed over the outside edge of the second ply. If narrower width mat is used, reverse the second ply 180° in the machine direction and lay it on top of the first ply to minimize weight variations.

7.3.3 Follow with a 10-mil (0.25-mm) surfacing mat as in 7.3.1.

7.3.4 Remove the air by rolling over the surface with a serrated metal or plastic roller. Take care not to expel enough resin to raise the glass content above the permissible maximum. The laminate is considered within the range of allowable levels of resin and glass if the thickness of the laminate is within 0.120 and 0.140 in. (3.05 and 3.56 mm), as described in 7.2.2.

7.3.5 After the lay-up is completed, cover the laminate with a plastic release film to prevent air inhibition or to provide a uniform smooth glossy surface, or both. Carefully smooth down to remove entrapped air.

NOTE 5—It is acceptable to use any convenient method for the application of the release film. Regardless of how it is applied, it is critical that any entrapped air between the film and the laminate be entirely removed. One method of application is done by previously wrapping the film around a metal rod. Starting at one edge of the laminate, slowly unroll the film from the rod, keeping a bead of resin ahead of the rod as you cross the laminate. Any entrapped air remaining can be removed by rubbing a tongue depressor across the release film surface. Carefully pull the film taut and fasten at the edges to prevent wrinkling of the film. Placing stops (neoprene has been found to be suitable) around the edges of the laminate and passing a heavy metal roller over the laminate helps to insure uniform controlled thickness.

7.3.6 Cure as recommended by the resin manufacturer. The cure schedule shall be reported.

7.3.7 Trim edges as required.

7.4 *Record of Standard Laminate Construction*—Record the properties of the standard laminate as follows:

7.4.1 *Hardness*—Determine Barcol hardness on the strip as described in 7.2.4 in accordance with Test Method D2583.

7.4.2 *Laminate Conditions*—Visually examine the laminate. The laminate shall meet Acceptance Level I of Table 1 of Practice D2563.

7.4.3 If the laminate meets the requirements of this specification, retain the laminate sections for preparation of test specimens.

NOTE 6—The major criteria for accepting a laminate is thickness and not glass content. If glass content is desired, cut eight 1 by 1 in. specimens from the center of the laminate and test in accordance with Test Method D2584.

7.5 *Individual Test Specimens:*

7.5.1 Specimens for immersion in test solutions shall be approximately 4 by 5 in. (101.6 by 127 mm), cut from the standard laminate.

7.5.2 Identity of specimens shall be maintained by suitable means.

7.5.3 Cut edges and drilled holes, if used for suspension, shall be sanded smooth and coated with paraffinated resin.

7.5.4 The number of specimens required is dependent on the number of test solutions to be employed, the number of different temperatures at which testing is performed, and the number of test intervals. In addition, at least two 4 by 5 in. (101.6 by 127 mm) specimens shall be available for test (see 8.4) following the curing period, prior to immersion.

8. Procedure

8.1 *Measurement of Specimens*—Immediately following the curing period, measure the thickness of the specimens to the nearest 0.001 in. (0.025 mm) at the geometric center of each of the intended 1 by 3 in. (25.4 by 76.2 mm) specimens that will be cut for flexural tests after the completed exposures. Measure the weight of the specimens to the nearest 0.01 g. These thickness and weight measurements shall also be used for comparison against thickness and weight measurements after the completed exposures.

8.2 *Exposure*—Following the curing period, as specified in 7.3.6, prior to immersion, record a brief description of the color and surface appearance of the coupons and the color and the clarity of the test solution. The total number of coupons per container is not limited except by the ability of the container to hold the coupons without touching each other or the container. The coupons must always be completely immersed. Coupons shall be vertical, parallel, and spaced a minimum of 0.25 in. (6.35 mm) apart. There shall be a minimum of 0.50 in. (12.7 mm) between coupon edges and the container or the liquid surface. Place the closed container in a constant temperature oven adjusted to the required temperature or in a suitably adjusted liquid bath. Examine the coupons after 30, 90, 180 days, and one year of immersion or other time intervals as required to determine the rate of attack.

8.2.1 Discard the test solution and replace it with fresh solution as often as necessary to maintain original composition and concentration. As a minimum, solutions known to be stable need to be replaced at the end of each test period.

8.3 *Cleaning and Examination After Exposure*—Clean the coupon and dry by blotting with a paper towel. Cold tap water is normally used for specimen cleaning. If other cleaning agents are used, verify that they do not attack the resin being tested.

8.3.1 Note any indication of surface attack on a coupon, any discoloration of the test solution, and the formation of any sediment.

8.3.2 After final blotting, immediately measure the coupon thickness to the nearest 0.001 in. (0.025 mm) in the geometric center of each intended 1 by 3 in. (25 by 76.2 mm) specimen. Measure the coupon to the nearest 0.01g. The Barcol hardness can then be checked, taking an average of ten readings on each coupon, a minimum of 0.50 in. (12.7 mm) from the edge.

8.3.3 After washing and measuring thickness, weight, and Barcol hardness, place the coupons in an air-tight polyethylene bag for conditioning or shipping as described in 8.4.1.

8.4 *Flexural Testing*—Determine the flexural strength and modulus for: (*1*) two sets of three specimens immediately following the curing period, and (*2*) one set of three specimens after each inspection, for each solution, and each test temperature. Calculation of flexural strength and modulus after exposure shall use the coupon thickness determined at the time of flexural testing as measured in 8.3.2. The two pretested sets shall be taken from the center of the laminate as described in 7.2.1. The flexural strengths for these two sets shall be averaged together for use in calculating the retained flexural strength in 9.2. The flexural modulus values shall also be averaged for use in 9.2.

8.4.1 Flexural tests shall be conducted in accordance with Procedure A of Test Methods D790, except for the conditioning parameters specified in this document. Coupons being tested at the exposure location shall be placed in the conditioning environment for a minimum of 2 h immediately following the "cleaning and examination" described in 8.3. The coupon shall be tested during the same day after removing the coupon from the test environment. For testing at a different location, the clean, dry coupons shall be placed in a vapor tight bag for shipment.

NOTE 7—In cases of volatile chemical exposure, special methods of specimen handling will probably be required.

8.4.2 Three 1 by 3 in. (25 by 76.2 mm) (see Fig. 1) are cut from each 4 by 5 in. coupon. After cutting, the specimen edges

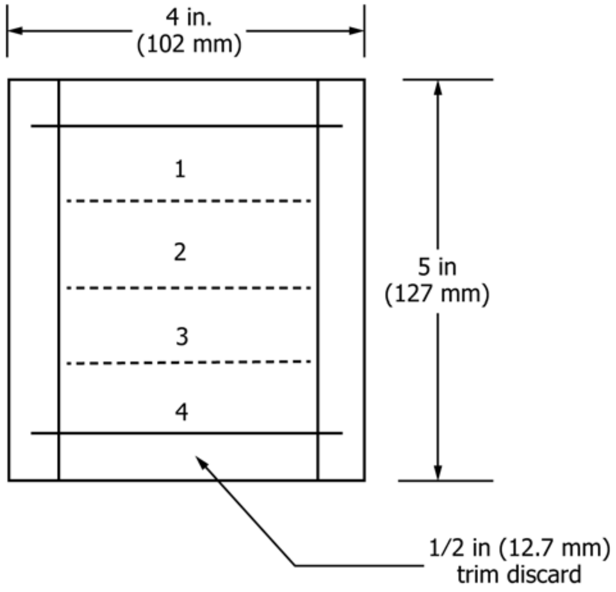

FIG. 1 Specimen Cutting Guide

shall be routed or sanded to provide a nick-free edge. Test specimens shall be the full thickness of the exposure coupon.

9. Calculation

9.1 *Barcol Hardness Change*—Tabulate or construct a graph showing the actual hardness readings of the specimens exposed at a given temperature, and the test period, in days.

9.2 *Retained Flexural Strength and Modulus*—Calculate to the nearest 1.0 %, the percentage retention of flexural strength and flexural modulus of the specimen during immersion for each examination period, taking the flexural strength and flexural modulus after curing as 100 %:

$$\text{Retained flexural strength, \%} = \left[S_2/S_1\right] \times 100 \tag{1}$$

where:

S_1 = flexural strength of specimen after curing period, and
S_2 = flexural strength of specimen after test period.

$$\text{Retained flexural modulus, \%} = \left[E_2/E_1\right] \times 100 \tag{2}$$

where:

E_1 = flexural modulus of specimen after curing period, and
E_2 = flexural modulus of specimen after test period.

9.2.1 Calculate flexural strength and modulus properties in accordance with Section 12 of Test Methods D790.

9.2.2 Construct graphs showing the average percentage of retained flexural strength and the average flexural modulus of the specimens broken at a given examination period after immersion in a particular test solution at a given temperature, plotting the percentage of retained flexural strength and flexural modulus as the vertical axis, and the test period, in days, as the horizontal axis.

9.3 *Percent Weight and Thickness Change*—Calculate to the nearest 0.01 % the percent weight and thickness change of the specimen during immersion for each examination period.

9.4 Calculate the percent weight and thickness change, and tabulate or graph these values as a function of the test period, in days.

10. Interpretation of Results

10.1 *Mechanical Properties of the Specimen*—Because of the chemical nature of certain types of plastic materials, the rate of change with time is of more significance than the actual value at any one time. A plot of the test results will indicate whether a particular specimen will approach constant flexural strength, flexural modulus, or hardness with time or will continue to change as the test progresses.

10.2 *Appearance of Specimen*—Visual inspection of the exposed specimen for surface cracks, loss of gloss, etching, blistering, pitting, softening, changes in thickness, or other irregularities, is very important, for these conditions indicate some degradation of the laminate by a chemical environment.

10.3 *Appearance of Immersion Medium*—It is possible that discoloration of the test solution and the formation of sediment will be significant factors. It is possible that an initial discoloration will indicate extraction of soluble components.

10.4 *Weight and Thickness of Specimen*—Weight and thickness changes can indicate the extent of chemical degradation or absorption of the test solution.

NOTE 8—Carry out all test exposures for the longest practical time to assure valid results. It is particularly important to obtain 6 and 12-month results in order to determine whether the properties are stable over a period of time. Short-term results (less than six months) can be unreliable when evaluating resins.

11. Report

11.1 Report the following information:

11.1.1 Company and individual preparing standard laminates.

11.1.2 Complete identification of material tested including resin, nonvolatile content, accelerator, catalyst, reinforcement, surfacing mat, and filler, such as fire-retardant additive or thixotropes.

11.1.2.1 As this practice applies to E-CR glass fiber, a type of E glass fiber, if the E glass fiber material is of the E-CR type that information shall be identified in the test report.

11.1.3 Cure cycle including room temperature gel time, time at room temperature before testing or before post-cure if required, post-cure time, and temperature. Any special post-curing techniques such as boiling water or steam for FDA-type applications shall also be reported.

11.1.4 Glass content of standard laminate, if run in accordance with Note 6.

11.1.5 Hardness, flexural strength of control coupons.

11.1.6 Color and surface appearance of specimens before testing.

11.1.7 Test conditions; immersion medium, temperature, and the like.

11.1.8 Total duration of test in days, and examination periods, in days. For each examination period, the data listed in 11.1.8.11 through 11.1.8.6 are required.

11.1.8.1 Pretesting samples conditioning (if different than standard).

11.1.8.2 Appearance of specimens after immersion (surface cracks, loss of gloss, etching, pitting, softening, and the like).

11.1.8.3 Appearance of immersion medium (discoloration, sediment, and the like).

11.1.8.4 Barcol hardness of the specimens before and after exposure.

11.1.8.5 Weight and thickness before and after exposure.

11.1.8.6 Flexural strength and flexural modulus of coupons and percent retention of flexural strength and flexural modulus.

11.1.9 Graph showing percent retention of flexural strength and flexural modulus plotted against test periods.

12. Precision and Bias

12.1 No precision statement can be made for this practice, since controlled round-robin test programs have not been run. The test results of this practice are obtained to assign bias statements to the subjective results since there are no standards. The bias of the quantitative results are covered by Test Methods D790.

13. Keywords

13.1 chemical resistance; glass-fiber-reinforced; glass-reinforced plastic (GRP); laminate; liquid service; reinforced thermosetting plastic (RTP); reinforced thermosetting resins; thermosetting resins

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