Designation: D 4398 – 9502

An American National Standard

Standard Test Method for Determining the Chemical Resistance of Fiberglass-Reinforced Thermosetting Resins by One-Side Panel Exposure[1,2]

This standard is issued under the fixed designation D 4398; 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 test method is intended for use in the evaluation of the chemical resistance of fiberglass-reinforced thermosetting resins that are subjected to one-side panel exposure to specific environments. It takes into consideration the coldwall effects and radiation losses of heat transfer through the laminate wall.

1.2 This test method is supplemental to Practice C 581 and does not supersede it.

NOTE 1—There is no similar or equivalent ISO standard.

1.3 *This standard does not purport to address all of the safety problems, 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:*

C 581 Practice for Determining Chemical Resistance of Thermosetting Resins Used in Glass Fiber Reinforced Structures Intended for Liquid Service[3]

---

[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.23 on Reinforced Plastic Piping Systems and Chemical Equipment.

Current edition approved ~~July 15, 1995.~~ March 10, 2002. Published ~~September 1995.~~ May 2002. Originally published as D 4398 – 84. Last previous edition D 4398 – ~~89~~5.

[2] This revision included the addition of Note 1 and Section 12.

***A Summary of Changes section appears at the end of this standard.**

D 790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials[4]
D 2583 Test Method for Indentation Hardness of Rigid Plastics by Means of a Barcol Impressor[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *glass fiber*—glass filaments for engineering applications, chiefly of a so-called ''E'' type made from a lime-alumina borosilicate glass that is relatively soda free, of high strength-to-weight ratio. Glass fibers are used commercially in the reinforcement of rigid engineering plastic structures.

3.1.2 *thermosetting resins*—linear, relatively low molecular weight thermoplastic polymer chains with ''crosslinks'' which bond the chains together with primary valence bonds. Once cross-linked, such three-dimensional polymers will not soften up appreciably to their decomposition temperature. Typical of such resin systems are the polymers, vinyl esters, and epoxies.

4. Significance and Use

4.1 The results obtained by this test method may serve as a guide in, but not as the sole basis for, predicting the possible performance of the particular glass-fiber-reinforced thermosetting resin laminate in the one-side exposure to the specific environment under evaluation. No attempt has been made to incorporate into the test method all of the factors that may enter into the serviceability of a glass-fiber-reinforced resin structure when subjected to chemical environments.

4.2 This test method provides for the determination of changes in the physical properties of the test panel and test media during and after the one-side exposure in the test media. Determination of changes include: Barcol hardness, appearance of panel, appearance of test media, flexural properties, and thickness.

5. Apparatus

5.1 *Test Cell*—This is a laboratory unit in which only the surface of one side of a test panel is subjected to the corrosive or aggressive environment, which is the normal experience of chemical processing equipment in actual plant operations. It consists of an open-ended glass cylinder, with several ground-glass joint nozzles for insertion of appropriate auxiliaries. The ends of the open glass cylinder are closed off with the fiberglass-reinforced resin panel to be tested, and are tightly sealed with chemical-resistant gaskets, allowing one side of the panel to come in contact with the test media.

5.1.1 Typical ~~of such~~ test cells are ~~the Corrocell[6]~~ shown in Fig. 1 ~~, and the Atlas Test Cell[7] shown in~~and Fig. 2. Other laboratory units for one-side corrosion testing of equal or larger diameter may be used also with correspondingly appropriate panel sizes.

[3] *Annual Book of ASTM Standards*, Vol 08.04.
[4] *Annual Book of ASTM Standards*, Vol 08.01.
[5] *Annual Book of ASTM Standards*, Vol 08.02.

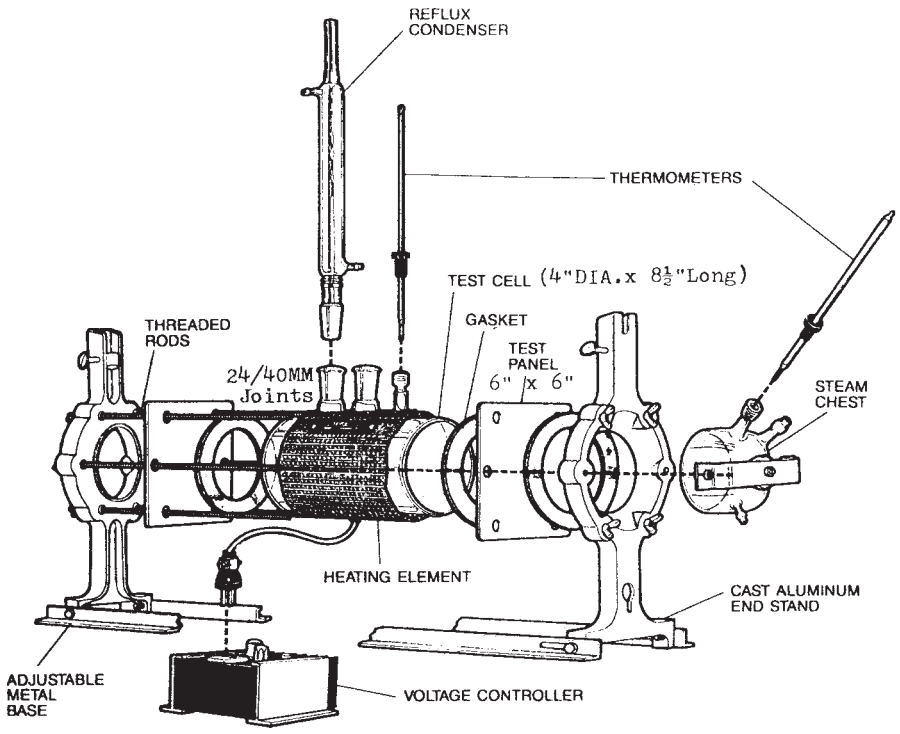


FIG. 1 Test Corrocell Test Unit

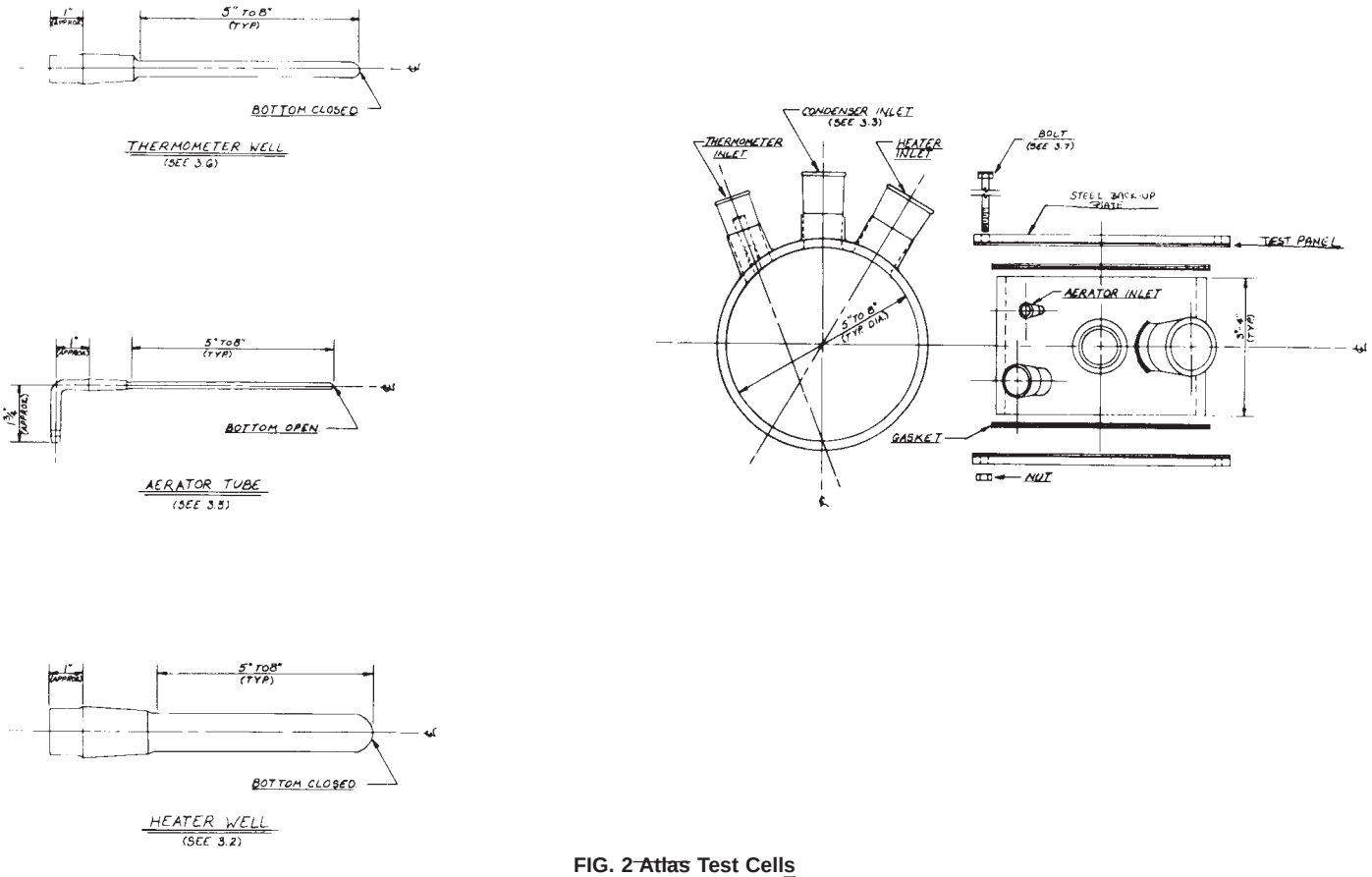


FIG. 2 Atlas Test Cells

5.2 *Reflux Condenser*—To maintain a constant liquid level and constant concentration of the test media.

5.3 *Electrical Heating Mantle*—Wrapped on the exterior to supply heat through the glass cylinder into the liquid, but not

touching the test panel. An alternative source of heat can be provided by an electrical heating element encased in a suitable glass probe.

5.4 *Thermometer*—To register the temperature of the test media under test, as well as that of the vapor phase above the liquid.

5.5 *Rheostat Power Source*—A variable resistance for regulating current flow and capable of maintaining the temperature within a range of ±4°F (±2.2°C).

5.6 *Impressor Type Instruments*—As described in Test Method D 2583.

5.7 *Micrometer*—Instrument suitable for measurement to 0.001 in. (0.025 mm).

5.8 *Flexural Properties Testing*—This shall be in accordance with the testing machine described in Test Methods D 790.

6. Test Specimens

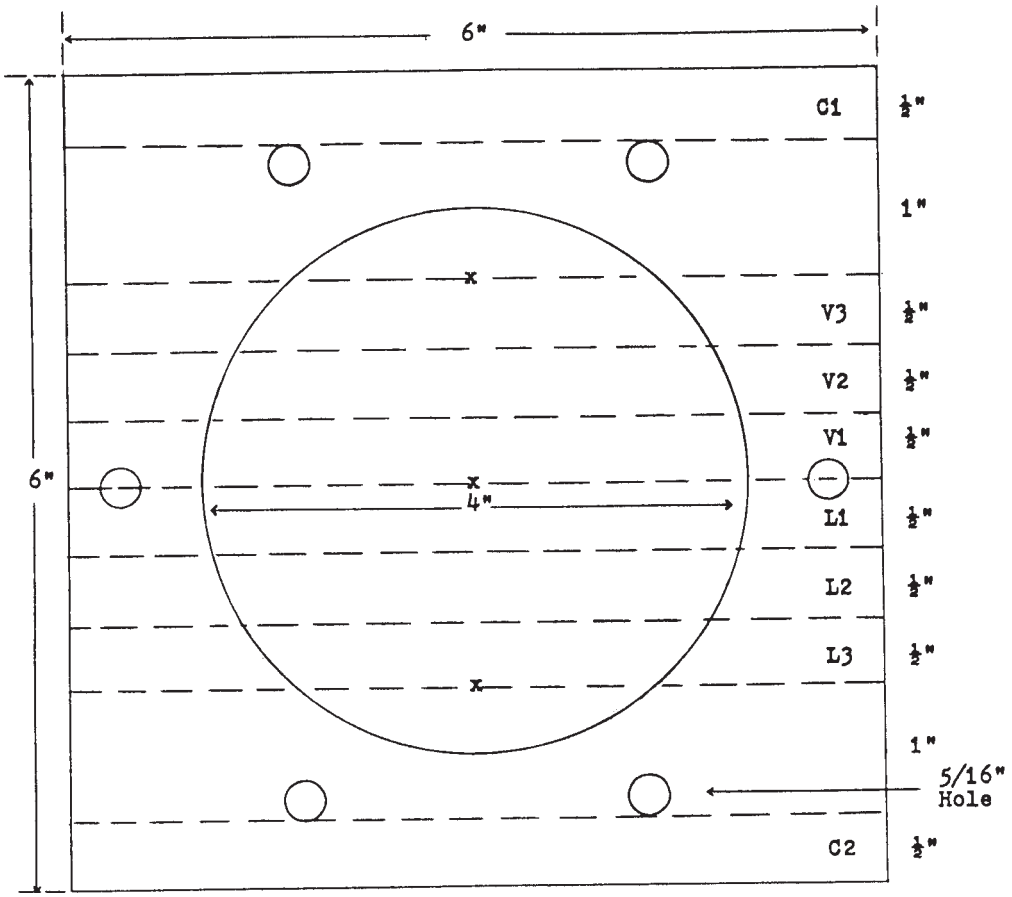
6.1 The fiberglass-reinforced thermosetting resin laminate panels prepared in accordance with Practice C 581, or composites conforming to the construction used in fabrication of that specific structure, may be employed for testing.

6.2 Test panels shall be of appropriate dimensions to provide sufficient surface area to seal the open ends of the test cell, as well as to provide sufficiently exposed surface to enable coupons to be cut from it for determination of physical properties. As an example, Fig. 3 shows such pertinent dimensional data for the panels used with the Corrocell. If test panels are used with larger-diameter test units, the mounting arrangement may be adjusted accordingly, or C-clamps may be used.

7. Test Environment and Conditioning

7.1 The test media shall consist of the reagents, solutions, slurries, gases, or products that actually constitute the specific environment to which the fiberglass-reinforced thermosetting resin system will be exposed.

7.2 The standard reagents for basic evaluation of resin-glass systems, as detailed in the Appendix of Practice C 581, can serve as test solutions when comparing the relative corrosion resistance of specific fiberglass-reinforced resin laminates.



NOTE 1—This example is for the CORROCELL Test Cell, the dimensions for the ATLAS Test Cell, and any other similar cells will be different.

FIG. 3 Dimensions for One-Side Panel

7.3 The test media and conditioning shall simulate the anticipated service conditions as closely as possible.

7.4 Mechanical agitation, or mixing through aeration, may be employed to eliminate stratification of liquids, or to prevent sedimentation or settling out of the slurry.

8. Procedure

8.1 *Measurement of Panels*—Measure the thickness of the test panels to the nearest 0.001 in. (0.025 mm) at the geometric center of the panel and 1.50 in. (38.1 mm) directly above and below that center point, for reference points in the later determination of any thickness changes.

8.2 *Preliminary Inspection of Panels*—Prior to affixing the panels to the test cylinder, record a brief description of the color and surface appearance of the panels. Make ten readings of the Barcol hardness around the periphery of the square panel at a distance no greater than 0.75 in. (19 mm) from the edge, recording the individual values. Do not take any Barcol hardness readings within the inner portion of the test panel that will be exposed to the specific test media. Record the color and clarity of the test media prior to exposure of the panels. Save a portion of the initial test media for future comparison with the exposed test media on completion of the test period. Retain a section of the original unexposed panel for use in evaluating any apparent changes to the exposed panel.

8.3 *Operation*:

8.3.1 Place the panels on the ends of the test cell and fill the vessel with the test media until it has reached the geometric center of the panels. Elevate the temperature of the test media to the desired operating temperature. Make certain that the reflux condenser is performing properly to maintain constant volume in the cell.

8.3.2 Examine the surfaces of the panels as desired after 30, 60, 90, 180 days, and after 1 year of exposure, or at any other interval of exposure, as indicated by their performance. Record any changes in surface appearance of panels and changes in the test media. Terminate the test if the panels show excessive chemical attack.

NOTE 2—Although one test cell can be employed to complete the 30, 60, 90, 180 days, and 1 year (360 days) exposure study, generally two test cells are used for duplicate or comparative testing and expediency. The panel replacement procedure commonly scheduled in the use of two test cells follows along those suggested in 8.3.3.

8.3.3 Typical panel exposure schedule:

	Cell 1		Cell 2	
	Side One	Side Two	Side One	Side Two
Initial setup	30 days	360 days	30 days	360 days
Replacement 1	60 days	None	60 days	None
Replacement 2	90 days	None	90 days	None
Replacement 3	180 days	None	180 days	None

8.3.4 At the end of the prescribed exposure period remove the panels from the ends of the glass test cell, rinse with water, dry, and determine any changes in thickness. Examine the exposed surfaces of the panels to determine any significant surface attack or changes in color. Record any changes in color or sedimentation of the test solutions.

8.3.5 Cut three ½-in. wide horizontal strips from the test panels in the vapor phase, that portion above the surface of the liquid, and three similar ½-in. wide horizontal strips from the submerged liquid phase. Cut suitable control strips from the top and bottom edges of the panel for comparison as unexposed sections. Identify vapor phase strips as V-1, V-2, and V-3, and those from the liquid phase as L-1, L-2, and L-3, with control strips marked C-1 and C-2 (see Fig. 3). With test units of different dimensions employing larger size panels, a similar group of test strips should be cut from the vapor and liquid exposed portions, using the geometric center of the larger panel as the common reference point.

8.3.6 *Flexural Tests*—Run flexural tests in accordance with Procedure A, Method 1, midpoint, Test Methods D 790 upon all of the strips identified in 8.3.5. The strips should be placed with the exposed surface facing upwards, with that surface under compression at break or failure. Test panels are forwarded to testing laboratories in a polyethylene airtight bag containing some of the test media, or in a glass wide-mouth jar if polyethylene is incompatible. The elapsed time between the removal of the panel from the test media and the flexural tests should be uniform for all panels and as short as possible.

8.3.7 *Barcol Hardness Measurements*—Barcol hardness tests (Test Method D 2583) are to be run upon the cut strips after determination of the residual physical properties, as well as on the exposed surface above V-3 and below L-3.

9. Interpretation of Results

9.1 *Physical Properties*—Changes in the flexural strength or flexural modulus of the exposed test panels are significant criteria in determining the chemical resistance and acceptability of the fiberglass-reinforced resin laminate in the specific exposure. Flexural strength and flexural modulus changes are those most frequently reported in the literature in evaluating the test results.

9.2 *Appearance of Specimens*—Visual inspection of the test panels exposed surfaces should be carried out to determine any cracks, loss of gloss, etching, pitting, softening, delamination, changes in thickness, blistering, severe discoloration or charring, leaching, fiber blossoming, or dissolution of the resin. All of these changes indicate some degree of degradation. An unexposed panel should be retained for comparison.

9.3 *Appearance of the Test Media*—Discoloration of the test media and the formation of sediment or precipitates are significant points to look for in the exposed test media. An initial discoloration may indicate extraction of a soluble component. Continuation

of the test using fresh solution may indicate whether the attack is progressive. A sample of the original test media should be retained for comparison.

10. Report

10.1 The report shall contain the following:
10.1.1 Company and individual reporting the data.
10.1.2 Complete identification of material tested, including resin, accelerator, catalyst, curing schedule, and reinforcement.
10.1.3 Conditioning procedure.
10.1.4 Hardness, flexural strength, and flexural modulus.
10.1.5 Color and surface appearance of test panels before test.
10.1.6 Test conditions, test media, and temperature.
10.1.7 Total duration of tests, examination periods, and changes of immersion medium.
10.2 For final examination report:
10.2.1 Barcol hardness. (See 8.3.7.)
10.2.2 Appearance of exposed surfaces of test panels. (See 9.2.)
10.2.3 Appearance of residual test media. (See 9.3.)
10.2.4 Flexural strength and flexural modulus percent retention values. (See 8.3.6.)
10.2.5 Changes in thickness. (See 8.1.)
10.2.6 Ambient temperature outside of test cell.

11. Precision and Bias

11.1 No general statement of precision can be made because of lack of sufficient data at this time.
11.2 No statement of bias can be prepared for this test method since there is no absolute method for use as comparative basis.

NOTE 3—The wide compositional differences in the numerous environmental test conditions, as well as the specific variables between the laminated panels, arising from choices of resin formulations and glass fiber reinforcements, make it difficult to provide meaningful data that will establish satisfactory precision and bias statements.

12. Keywords

12.1 contact molded; corrosion resistant equipment; glass-fiber reinforced; laminate; reinforced thermosetting plastic (RTP); thermoset epoxy resin; thermoset polyester resin; thermoset vinyl ester resin

SUMMARY OF CHANGES

Committee D20 has identified the location of the following changes to this test method since the last issue, D 4398–95, that may impact the use of this standard.

(1) All references to equipment manufacturers were omitted.