

DEPARTMENT OF TRANSPORTATION
DIVISION OF ENGINEERING SERVICES
Transportation Laboratory
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METHOD FOR TESTING EPOXY RESIN ADHESIVES, BINDERS AND SEALANTS

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH**" in Part 10 of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and to determine the applicability of regulatory limitations before any testing is performed.

SCOPE

The procedures used for testing epoxy resin adhesives, binders, and sealants are described in this test method.

This test method is divided into the following parts:

1. Gel Time
2. Bond Strength to Concrete
3. Tensile Adhesion and Cohesion
4. Brookfield Viscosity
5. Slant Shear Strength
6. Sag
7. Tensile Strength and Elongation
8. Infrared Spectra
9. Other Tests
10. Safety and Health

PART 1. GEL TIME

A. APPARATUS

1. Unwaxed paper cups, approximately 8 oz and 2 oz.
2. A wooden tongue depressor, with the ends cut square.

3. A stainless steel spatula with a blade 6 in long x 1 in wide, with the end cut square.
4. A stopwatch accurate to within 1 s or smaller division.

B. PROCEDURE

1. Condition A and B components in air to $25 \pm 1^\circ\text{C}$.
2. Stir the separate components vigorously with the spatula to redisperse any settled material.
3. Using 2 oz cups, measure components A and B in the proportions recommended by the manufacturer into an 8-oz paper cup.
4. Start a stopwatch immediately and mix the components for 60 s using a wooden tongue depressor and taking care to scrape the sides and the bottom of the cup periodically.
5. Probe the mixture once with the tongue depressor every 30 s.
6. The time at which a soft stringy mass forms in the container is the gel time.

PART 2. BOND STRENGTH TO CONCRETE

A. APPARATUS

1. Concrete blocks, 7 in x 3½ in x 2 in, made from the following formula:
 - a. 7.9 kg of Portland cement.
 - b. 3.5 kg of water.
 - c. 17 kg of commercial quality Portland cement concrete (PCC) aggregate, saturated surface-dry, maximum size of ¾ in.
 - d. 17 kg of sand, saturated surface-dry.
2. A suitable test apparatus with a load capacity of 2000-lb force or greater.
3. A sandblasted, 2-in diameter, threaded steel or aluminum rod may be used in place of the pipe cap.
4. A stopwatch accurate to within 1 s or smaller division.

B. PROCEDURE FOR EPOXY ADHESIVES

1. Condition the test equipment, materials, and epoxy components for 24 hr at the testing temperature specified.
2. Stir the separate components vigorously for 30 s.
3. Mix the epoxy in the specified volume or weight ratio for 60 s.
4. Immediately start timing.
5. Place the adhesive on the pipe cap, or rod, and the concrete surface.
6. Press the pipe cap, or rod, firmly in place and remove the excess adhesive.
7. Just before the required test time, insert the hook into the pipe cap, or rod.
8. The sample should be tested at a separation rate of 0.2 in/min.

PART 3. TENSILE ADHESION AND COHESION

A. APPARATUS

1. Ceramic markers and retroreflective pavement markers from the Prequalified and Tested Delineator Materials List, three each per test.
2. A suitable test apparatus with a load capacity of 2000-lb force or greater.
3. A cold box capable of maintaining $-9 \pm 1^{\circ}\text{C}$.
4. An oven capable of maintaining $60 \pm 1^{\circ}\text{C}$.

B. PROCEDURE

1. Stir the separate components vigorously for 30 s.
2. Mix the epoxy in the proper ratio specified by the manufacturer.
3. Prepare test specimens by placing the adhesive on the pipe cap, or rod, and the surface to be tested.
4. Press the pipe cap, or rod, firmly in place and remove the excess adhesive.
5. Cure all specimens for 24 hr at $25 \pm 1^{\circ}\text{C}$.
6. Determine the tensile strength according to Part 2.B of this test method. Test in triplicate.
7. Post cure one ceramic marker test specimen further as follows:
 - a. 48 hr at 60°C .
 - b. Cool to $25 \pm 1^{\circ}\text{C}$, and then place in cold box for 24 hr at $-9 \pm 1^{\circ}\text{C}$.
 - c. Return to $25 \pm 1^{\circ}\text{C}$ and test as in Step No. 6 above.

PART 4. BROOKFIELD VISCOSITY

A. APPARATUS

1. Model RVT Brookfield Syncro-Electric Viscometer, Brookfield Engineering Laboratories, and the appropriate spindles.
2. Round pint-size paint cans.
3. A stainless steel spatula with a blade 6 in long x 1 in wide, with the end cut square.

B. PROCEDURE

1. Condition and mix the epoxy components as described in the Gel
2. Time Procedure in Part 1 of this test method.
3. Fill a pint can within one inch of the top with well-mixed epoxy.
4. Remove entrained air bubbles by vigorous tamping.
5. Insert the proper spindle according to specifications.
6. Measure the viscosity in poise within 10 min of stirring.

PART 5. SLANT SHEAR STRENGTH

A. APPARATUS

1. Ottawa sand, ASTM Designation: C 109.
2. Portland cement, Type II.
3. A suitable mold to make diagonal concrete mortar blocks with a 2-in square base and having one diagonal face 2 in x 4 in, starting about $\frac{3}{4}$ in above the base. The diagonal faces of two such blocks are bonded together, producing a block of dimensions 2 in x 2 in x 5 in.

4. Blocks made from the following composition:

- a. Ottawa sand, ASTM Designation: C 109, 13.65 kg.
- b. Portland cement, Type II, 5.49 kg.
- c. Water, 2.185 L.
- d. Cure the blocks 28 days in a fog room.
- e. Dry and sandblast the diagonal faces.

5. Suitable test press.

B. PROCEDURE

1. Mix the epoxy as described in Part 1 of this test method and apply a coat to each diagonal surface. Press the diagonal surfaces of each block together by hand and remove the excess epoxy adhesive.
2. Align the blocks so that the ends and sides are square and form a block 2 in x 2 in x 5 in. Use blocks of wood or metal against each 2-in square end to keep diagonal faces from slipping until the epoxy hardens.
3. After the specified cure time, apply a suitable capping compound to each of the 2-in square bases and test by applying a compression load with a Universal Test Machine or other suitable testing apparatus at the rate of 5000 lb/min until failure.
4. Report results in psi.
5. For wet shear strength, bond another set of blocks together as described above. Cure 24 hr at $25 \pm 1^\circ\text{C}$, then soak in water for 7 days at $25 \pm 1^\circ\text{C}$ and immediately test as described above in Step No. 3.

PART 6. SAG TEST

A. APPARATUS

1. Unwaxed paper cups, approximately 8 oz and 2 oz.
2. A wooden tongue depressor, with the ends cut square.
3. A stainless steel spatula with a blade 6 in long x 1 in wide, with the end cut square.
4. Leneta chart, Form 2-A Opacity.
5. Metal shims, $\frac{1}{4}$ in thick and at least 7 in long.
6. A metal straightedge, 6 in long.

B. PROCEDURE

1. Place the shims on the glazed face of the Leneta chart to outline an area $2\frac{1}{2}$ in wide x 7 in long.
2. Condition and mix the epoxy components as described in the Gel Time Procedure in Part 1 of this test method.
3. Place the mixed epoxy near one end of the chart, between the shims. Use the metal straightedge to draw down a sheet 7 in long x $2\frac{1}{2}$ in wide x $\frac{1}{4}$ in thick.
4. Remove the shims and immediately hang the chart vertically, with 7-in edges at the top and the bottom, until the epoxy hardens.
5. The epoxy should not sag or flow down the chart. The edges should remain straight.

PART 7. TENSILE STRENGTH AND ELONGATION

A. APPARATUS

1. A leveling table, 12 in x 15 in, about $\frac{1}{2}$ in thick, with the surface milled flat and smooth.
2. Two pieces of Mylar, about 8 in x 12 in each.

3. A milled and polished steel gasket with outside dimensions 8 in x 11 in and inside dimensions $6\frac{1}{2}$ in x $9\frac{1}{2}$ in. The thickness shall be milled to $\frac{1}{8}$ in.
4. Plate glass, $\frac{1}{4}$ in thick x 8 in wide x 11 in long.
5. An air circulation oven capable of maintaining $70 \pm 0.5^\circ\text{C}$.
6. The centrifuge shall be capable of maintaining 2000 rpm, with cups having a capacity of approximately 150 mL each.

B. PROCEDURE

1. Level the table using adjusting screws and a suitable bubble level.
2. Place one piece of the 8 in x 12 in Mylar on the surface of the leveling table and place the steel gasket over the Mylar sheet.
3. Mix the epoxy in the specified volume or weight ratio.
4. Place centrifuge tubes on each pan of a suitable torsion balance. Pour the mixed epoxy into each tube until the tubes are balanced.
5. Immediately place the tubes in the centrifuge and spin at 2000 rpm for 3 min to remove air bubbles.
6. Remove the tubes from the centrifuge and pour the epoxy into the steel gasket, spreading out the epoxy as evenly as possible within the gasket and slightly thicker than the gasket.
7. Roll up the second 8 in x 12 in Mylar sheet and, starting at one edge of the steel gasket, carefully unroll the Mylar sheet over the epoxy, taking care not to trap any air pockets between the epoxy surface and the Mylar sheet.
8. Place the flat plate glass over the Mylar sheet and push down hard to extrude excess epoxy from the edges of the steel gasket.

9. Place a suitable weight on top of the glass plate to keep an even pressure on the epoxy sheet.
10. Cure the specimen for 18 hr at 25°C.
11. Strip the epoxy sheet from the Mylar and the gasket and place in oven for 5 hr at 70°C.
12. Cool to 25°C and cut test specimens with die shown in Figure 1.
13. Proceed as in ASTM Designation: D 638, using a 0.2 in/min press rate and a 1-in gage length.

PART 8. INFRARED SPECTRA

A. APPARATUS

1. Infrared spectrophotometer capable of obtaining transmission spectra in accordance with ASTM Designation: E 1252. Potassium bromide disks with holder attachment for analysis of a capillary film.
2. High-speed centrifuge able to separate the liquid and solid phases of the epoxy components.

B. PROCEDURE

Analyze each component as shown in the following steps.

1. Place about 15 g of a single component in a centrifuge tube.
2. Centrifuge the component at approximately 15,000 rpm until there is a supernatant liquid layer present in the tube. This may take about 20 min.
3. Place a drop of the liquid supernatant on the disk for analysis.
4. Obtain a transmission spectra in absorbance units as described in ASTM Designation: E 1252.
5. Compare the spectra to those on file at the Transportation Laboratory.

PART 9. OTHER TESTS

A. DUROMETER HARDNESS AT 25°C, TYPE D

Use the procedure as specified in ASTM Designation: D 2240.

B. DENSITY (g/mL)

Use the procedure as specified in ASTM Designation: D 1475.

PART 10. SAFETY AND HEALTH

This method may involve hazardous materials, operations, and equipment. This method does not purport to address all the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

Epoxy resins contain irritants, especially to the skin, eyes, and respiratory system. Persons handling these materials shall use appropriate protective clothing, including rubber or plastic gloves. If any epoxy resin should contact the skin, it shall be removed immediately with a dry cloth or paper towel, and the area of contact washed thoroughly with soap and water. Solvents shall *not* be used, because they carry the irritant into the skin. Cured epoxy resins are innocuous.

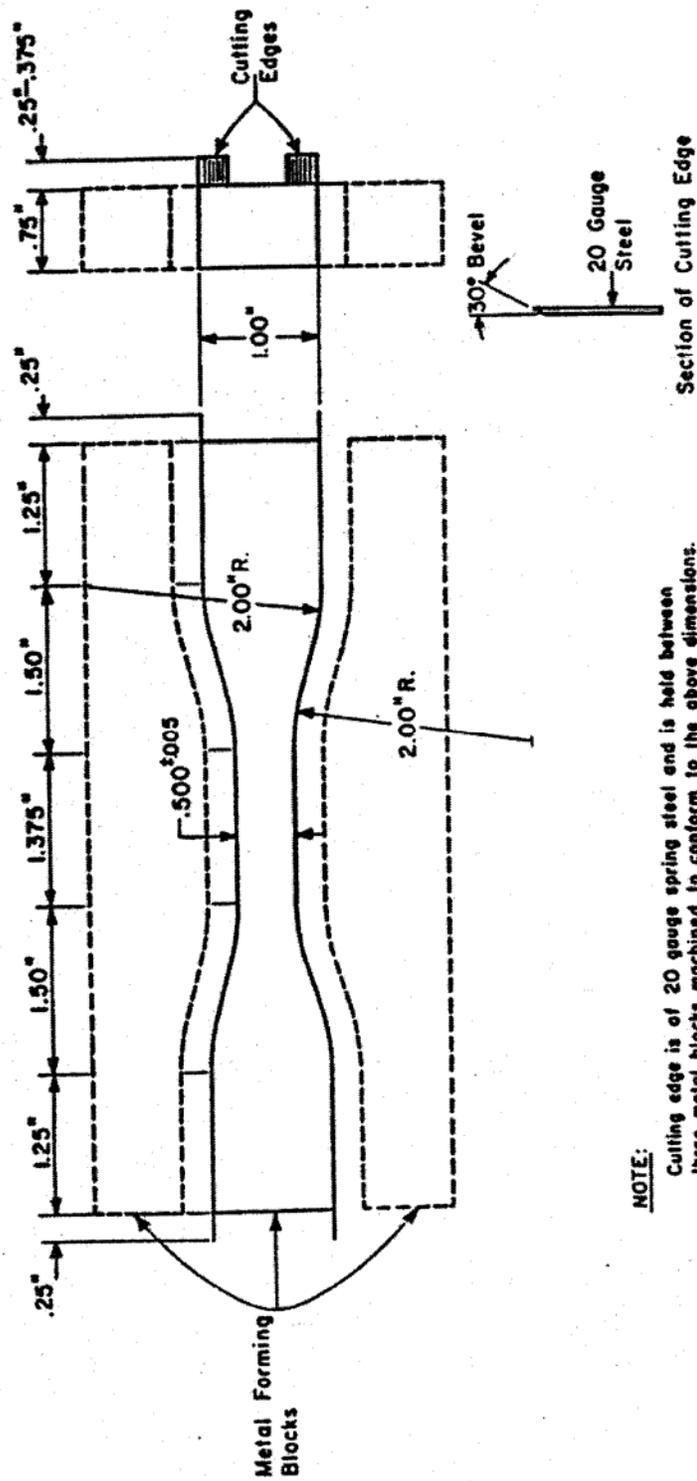
Users of this method do so at their own risk.

Prior to handling, testing, or disposing of any materials, testers are required to read the California Department of Transportation *Laboratory Safety Manual*.

REFERENCES

**ASTM Designations: C 109, D 638, D 1475,
D 2240 and E 1252**

**End of Text
(California Test 434 contains 6 pages)**



CUTTING DIE FOR TENSILE TEST

FIGURE 1. CUTTING DIE FOR TENSILE TESTING