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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 14 of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and 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. Infrared Curve
3. Bond Strength to Concrete
4. Tensile Adhesion and Cohesion
5. Brookfield Viscosity
6. Shear Ratio
7. Percent of Entrapped Air
8. Slant Shear Strength
9. Sag
10. Storage Stability
11. Tensile Strength and Elongation
12. Compressive Strength
13. Other Tests
14. Safety and Health

PART 1. GEL TIME

A. APPARATUS

1. Unwaxed cups, approximately 250 mL, 50 ± 6 mm at the base.
2. Unwaxed cups, approximately 30 mL.
3. Wooden tongue depressor with ends cut square.
4. Stainless steel spatula with blade 152 by 25 mm , with end cut square.
5. A stopwatch accurate to within 1 s, or smaller division.

B. PROCEDURE

1. Condition A and B components in air to 25 ± 1˚C.
2. Stir the separate components vigorously with the spatula to redisperse any settled material.
3. Using 30-mL cups, measure components A and B in the proportions shown in Table 1 into a 250-mL 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 bottom of the cup periodically.

5. Probe the mixture once with the tongue depressor every 30 s, starting at time shown in Table 1.

6. The time at which a soft stringy mass forms in the container is the gel time.

PART 2. INFRARED CURVE

A. APPARATUS

1. A recording infrared spectrophotometer, having a minimum abscissa range of 40 000 to 6 000 mm^{-1}, and a minimum resolution of 30 mm^{-1} at 30 000 mm^{-1} and 16 mm^{-1} at 10 000 mm^{-1}.

2. A disk holder for a 25-mm diameter disk is required.

3. Two alkali halide crystal disks shall be 25 mm in diameter.

4. Sorvall SS-3 Automatic Superspeed Centrifuge, or a comparable centrifuge, which is able to separate the liquid and solid phases of the epoxy components without previous dilution with solvents.

B. PROCEDURE

1. Place about 15 g of component A into a stainless steel centrifuge tube.

2. Counterbalance with component B in a second centrifuge tube.

3. Centrifuge the two sample components at approximately 1780 rad/s until there is a supernatant liquid layer present in each tube. This takes 20 to 30 min.

4. Place a drop of component A liquid layer on a disk.

5. Place another disk over the drop, rotate, and press down until the liquid has flowed into a uniform layer between the two disks.

6. Place the disks in the holder and run an absorption curve with the infrared spectrophotometer.

7. More or less liquid may be used between the disks so as to produce a maximum absorption of 0.7 to 1.0 for the strongest absorption point on the curve.

8. Clean the disks with toluene and dry.

9. Repeat Step Nos. 4 through 8 with the liquid layer from component B.

10. Compare each curve with the absorption curves for standard materials on file at the Transportation Laboratory. Two materials are considered to be identical if all of the absorption points agree as to wave length and relative magnitude of the peaks in comparison with the other points of absorption.

PART 3. BOND STRENGTH TO CONCRETE

A. APPARATUS

1. Concrete blocks 305 by 305 by 102 mm, prepared with 400 kg/m^3 of concrete and having a tensile strength in excess of 1.7 MPa.

2. Blend equal parts by volume of Ottawa sand which conforms to gradings specified in ASTM Designation: C 190 and ASTM Designation: C 109.

3. Use the same apparatus as specified in California Test 420. Apparatus 1, 2, 3, and 7 or a suitable testing press at a load rate of 2268 kg/min.

4. A sandblasted, 50-mm diameter, threaded steel or aluminum rod may be used in place of the pipe cap.

5. 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 h 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 pipe cap, or rod, firmly in place and remove the excess adhesive.

7. Just before the required test time, insert the hook into the cap (or rod).

8. Proceed according to Part 1, Section A-6 of the California Test 420, or use a suitable testing press to determine the bond strength.

C. PROCEDURE FOR EPOXY MORTARS

1. Clean the 305 by 305-mm surface of the concrete block by sandblasting.

2. Make an approximate 5-mm rim round the concrete block surface using several layers of masking tape.

3. Condition the block, blended Ottawa sand, and the individual epoxy components to 25 ± 1˚C.

4. Stir the separate components vigorously for 30 s.

5. Mix the epoxy components in the specified volume or weight ratio.

6. Mix one part by volume of the mixed epoxy adhesive with three parts by volume of the Ottawa sand blend to make an epoxy mortar.

7. Coat the cleaned area of the concrete block with a 5-mm thick layer of the epoxy mortar. Trowel mortar to produce smooth, even surface.

8. After the mortar has cured seven days at 25 ± 1˚C, proceed according to Part 1 of California Test 420.

PART 4. TENSILE ADHESION AND COHESION

A. APPARATUS

1. Type A smooth bottom ceramic markers, three each per test.

2. Reflective pavement marker of current State specification, three per test.

3. Use testing apparatus described in Part 3, Item B.

4. Cold box capable of maintaining -9 ± 1˚C.

5. Oven capable of maintaining 60 ± 1˚C.

B. PROCEDURE

1. Stir the separate components vigorously for 30 s.

2. Mix the epoxy in the specified volume or weight ratio.

3. Place 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 excessive adhesive.

5. Cure all specimens for 24 h at 25 ± 1˚C.

6. Proceed according to Part 1, Section A-6 of California Test 420, or use a suitable testing press for the Type A ceramic marker. Test in triplicate.

7. Post cure one Type A ceramic marker further as follows:

   a. 48 h at 60˚C.

b. Return in 25 ± 1˚C, and then place in cold box for 24 h at -9 ± 1˚C.

c. Return to 25 ± 1˚C and test as in Step No. 6 above.

PART 5. BROOKFIELD VISCOSITY

A. APPARATUS

1. Model RVT Brookfield Syncro-Electric Viscometer, Brookfield Engineering Laboratories, Stoughton, Massachusetts with Spindles Nos. 2 and 3.

2. Brookfield Helipath Stand, Model C, with Spindles, TA, TB, TD, TE, and spindle weight.

3. Round, approximately 500-mL paint cans.

4. Stainless steel spatula, having a blade 152 by 25 mm, with the end cut square.

B. PROCEDURE

1. Fill a 500-mL can within 25 mm of the top with well mixed component A.

2. Condition the material to 25 ± 1˚C.

3. Stir the material vigorously for 30 s with a spatula.

4. Remove entrained air bubbles by vigorous tamping.

5. Insert the proper spindle according to specifications.

6. Set the rotation indicator according to specifications.

7. Make viscosity reading within 10 min of stirring.

8. When stable reading is reached, read the 0 to 100 scale. Apply appropriate factor and calculate viscosity, in Pa.s.

9. Repeat for B component.

10. Make additional readings at 0.05 and 0.25 rad/s for use in Part 6, Shear Ratio.

PART 6. SHEAR RATIO

A. APPARATUS

1. Same as Part 5.

B. PROCEDURE

1. Same as Part 5.

2. Calculation

$$\text{Shear Ratio} = V_1 / V_2$$

Where: V_1 = Viscosity at 0.05 rad/s, in Pa·s

and V_2 = Viscosity at 0.25 rad/s, in Pa·s

PART 7. PERCENT OF ENTRAPPED AIR

A. APPARATUS

1. Round, approximately 1-L paint cans.

2. Electric hot plate.

3. Vacuum chamber capable of 0.1 MPa, which is equipped with valves enabling manual control of application and release of vacuum and viewing ports or other means of observing materials under vacuum.

B. PROCEDURE

1. Density, in g/mL, Part 13, must be done before proceeding with this test (W_1).

2. Fill separate, round, 1-L paint cans approximately $^1/_4$ full of each component.

3. Heat cans and contents to 79 to 82˚C.

4. Place the samples into a vacuum oven and evacuate until the samples show signs of frothing over.

5. Vibrate or tamp the samples to facilitate removal of entrapped air.

6. As frothing subsides, increase the vacuum until 0.1 MPa is reached.

7. Cool samples to 25 ± 1˚C after removing from the vacuum oven.

8. Measure the density, in g/mL, as done in Part 13, Item F (W_2).

9. Calculation

$$\% \text{ Air} = [(W_2 \times W_1)/W_1] \times 100$$

PART 8. SLANT SHEAR STRENGTH

A. APPARATUS

1. Ottawa sand, ASTM Designation: C 109

2. Portland cement, Type II

3. Suitable mold to make diagonal concrete mortar blocks with a square base with 50-mm sides and having one diagonal face 50 by 100 mm, starting about 19 mm above the base. The diagonal faces of two such blocks are bonded together, producing a block of dimensions 50 by 50 by 130 mm.

4. Blocks made from the following composition:

   a. Ottawa sand, ASTM Designation: C 109, 1365 g

   b. Portland cement, Type II, 549 g

   c. Water, 2185 mL

   d. Cure block 28 days in a fog room.

   e. Dry and sandblast diagonal faces.

5. Suitable test press.

C. PROCEDURE

1. Mix the epoxy, as described in Part 3, Bond Strength, Item C, and apply a coat to each diagonal surface. Press diagonal surfaces of each block together by hand and remove excess epoxy adhesive.

2. Align the blocks so that the ends and sides are square and form a block 50 by 50 by 130 mm. Use blocks of wood or metal against each 50 by 50-mm end to keep diagonal faces from slipping until epoxy hardens.

3. After the specified cure time, apply a suitable capping compound to each of the 50 by 50-mm base and test by applying a compression load with a Universal Test Machine or other suitable testing apparatus at the rate of 2270 kg/min until failure.

4. Report results in Pa.

5. For wet shear strength, bond another set of blocks together as described above. Cure 24 h at 25 $\pm$ 1˚C; then soak in water for seven days at 25 ± 1˚C, and immediately test as described above in Item C3.

PART 9. SAG TEST

A. APPARATUS

1. Unwaxed cups, approximately 250 mL, 50 ± 6-mm diameter base.

2. Unwaxed cups, approximately 60 mL.

3. Wooden tongue depressor with ends cut square.

4. Stainless steel spatula with blade, 150 by 25 mm, and with the end cut square.

5. Leneta chart, Form 2-A opacity.

6. Metal shims, 6-mm thick, and at least 175 mm in length.

7. Metal straightedge, 150 mm in length.

B. PROCEDURE

1. Place the shims on the glazed face of Leneta chart to outline an area 65-mm wide by 180-mm long.

2. Condition and mix epoxy components as in the Gel Time procedure (Part 1).

3. Place mixed epoxy near one end of the chart, between the shims. Use metal straigh 180-mm long by 6-mm wide by 6-mm thick.

4. Remove the shims and immediately hang chart vertically, with 180-mm edges at top and bottom, until the epoxy hardens.

5. Epoxy should not sag or flow down the chart. The edges should remain straight.

PART 10. STORAGE STABILITY

A. APPARATUS

1. Same as Part 5.

2. Oven capable of maintaining 46 ± 2˚C.

B. PROCEDURE

1. Determine the viscosity and shear ratio for each epoxy component, Parts 5 and 6.

2. Store components in closed containers in oven maintained at 46 ± 2˚C for two weeks.

3. Remove containers from oven. Cool and redetermine viscosity and shear ratio at same temperature and using the same spindle and apparatus as in original test.

4. Viscosity and / or shear ratio changes in excess of 15 % is cause for rejection of the material.

PART 11.TENSILE STRENGTH AND ELONGATION

A. APPARATUS

1. Leveling table, 305 by 380 by 13 mm, with the surface milled flat and smooth.

2. Cut two pieces of Mylar about 200 by 300-mm each.

3. Milled and polished steel gasket with outside dimensions 205 by 280 mm, and inside dimensions 165 by 241 mm. The thickness shall be milled to 3.2 mm.

4. Plate glass: 6-mm thick, 205-mm wide and 280-mm long.

5. Air circulation oven capable of maintaining 68.3 ± 0.5˚C.

6. The centrifuge shall be capable of maintaining 210 rad / s, 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. Cut two pieces of Mylar about 200 by 300-mm each.

3. Place one piece of Mylar on surface of the leveling table and place steel gasket over the Mylar sheet.

4. Mix the epoxy in the specified volume or weight ratio.

5. Place centrifuge tubes on each pan of a suitable torsion balance. Pour mixed epoxy into each tube until tubes are balanced.

6. Immediately place tubes in centrifuge and spin at 210 rad / s for 3 min.

7. Remove tubes from centrifuge and pour epoxy into steel gasket, spreading out the epoxy as evenly as possible within the gasket and slightly thicker than the gasket.

8. Roll up the second piece of Mylar sheet, and starting at one edge of the steel gasket, carefully unroll the Mylar sheet over the epoxy, taking care not to entrap any air pockets between the epoxy surface and the Mylar sheet.

9. Place flat plate glass over the Mylar sheet and push down hard to extrude excess epoxy from edges of steel gasket.

10. Place a suitable weight on top of glass plate to keep an even pressure on the epoxy sheet.

11. Cure the specimen for 18 h at 25˚C.

12. Strip epoxy sheet from Mylar and gasket and place in oven for 5 h at 70˚C.

13. Cool to 25˚C and cut test specimens with die shown in Figure 1.

14. Proceed as in ASTM Designation: D 638, using a 5-mm/min press rate and a 25-mm gage length.

PART 12. COMPRESSIVE STRENGTH

A. APPARATUS

1. Brass cylinder, 3.2-mm thick, 108 mm in height, and exactly 50.8 mm for the internal diameter. The cylinder is split lengthwise 1.6 mm in width, such that when clamped tight, the split is drawn together and the internal diameter is exactly 50.8 mm.

2. Two screw-type hose clamps for each brass cylinder, maximum opening 64 mm.

3. Hardwood dowel, 38 mm in diameter and 205 mm in length.

4. Steel spatula.

5. Mylar sheet, 50 by 50 mm.

6. A blend of equal parts by volume of Ottawa sand, which each conforms to the gradings specified in ASTM Designation: C 190.

7. A release agent, aerosol type preferred.

B. PROCEDURE

1. Position two screw clamps around the brass cylinder at equidistant positions and tighten clamps so that the slot is drawn together.

2. Coat inside of cylinder with release agent.

3. Place Mylar sheet on flat surface of table and place brass cylinder over Mylar sheet.

4. Mix the epoxy in the specified volume or weight ratio and place in a suitable container, and mix thoroughly for at least 2 min or until no streaks are apparent.

5. For each unit volume of mixed epoxy, add 4 volumes of Ottawa sand and mix thoroughly with a steel spatula.

6. Fill cylinder to the halfway point and use the hardwood dowel to stamp the mortar into the cylinder with 25 brisk strokes. Fill the remainder of the cylinder with mortar and tamp again with 25 strokes of the hardwood dowel. Strike off the surface flush and flat with the steel spatula.

7. Allow specimen to cure for one week at 25˚C.

8. Remove screw clamps and Mylar sheet from bottom of cylinder. Remove mortar cylinder from brass cylinder.

9. Cap top and bottom of mortar cylinder with suitable capping compound.

10. Load to failure at 4550 kg/min with a test press. Report compressive strength in pascals.

PART 13. OTHER TESTS

A. WEIGHT PER EPOXY EQUIVALENT

1. Use procedures specified in ASTM Designation: D 1652.

B. AMINE VALUE

1. Use exactly same procedures and reagents as specified in ASTM Designation: D 1652. Calculate the amine value in mg equivalent KOH g of resin.

   Amine No. = (mL HBr x Normality HBr x 56.1) / mass of sample.

C. ASH CONTENT

1. Use the procedure as specified in ASTM Designation: D 482.

D. DUROMETER HARDNESS AT 25˚C, TYPE D

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

E. PERCENT WEIGHT GAIN IN WATER AT 25˚C

1. Use procedure specified in ASTM Designation: D 570.

F. DENSITY (g/mL)

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

G. ALKALINITY

1. Use the test procedure as specified in ASTM Designation: D 664.

2. Alkalinity, equivalent / 100 g = [mL HCl x Normality HCl] / (10 x sample in g)

H. WATER

1. Use the test procedure as specified in ASTM Designation: D 95.

I. RESISTANCE TO CALCIUM CHLORIDE

1. Use the test procedure as specified in ASTM Designation: D 543.

J. LOW TEMPERATURE CREEP

1. Use the test procedure as specified in California Test 419.

PART 14.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 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.

Prior to handling testing or disposing of any materials, testers are required to read the Caltrans Laboratory Safety Manual. Users of this method do so at their own risk.

REFERENCES:

California Tests 419 and 420
ASTM Designations: C 109, C 190, D 95, D 482, D 543, D 570, D 638, D 664, D 1475, D 1652 and D 2240

End of Test (California Test 434 contains 10 pages)

TABLE 1
TEST PARAMETERS FOR GEL TIME TEST

Specification	Type Epoxy	A Component (in mL)	B Component (in mL)	Elapsed Time After Mixing Before First Probe (in min)
8040-xx-01	Fast setting, low viscosity	120	60	12
8040-xx-02	Injection grouting PCC pavements	60	60	2
8040-03	Concrete binder, low viscosity	120	60	12
8040-06	Inductive loop, sealant	60	60	5
8040-07	Adhesive, rapid set for pavement markers	60	60	2
8040-08	Adhesive for bonding new to old concrete	120	60	20
8040-09	Adhesive, standard set for pavement markers	60	60	2

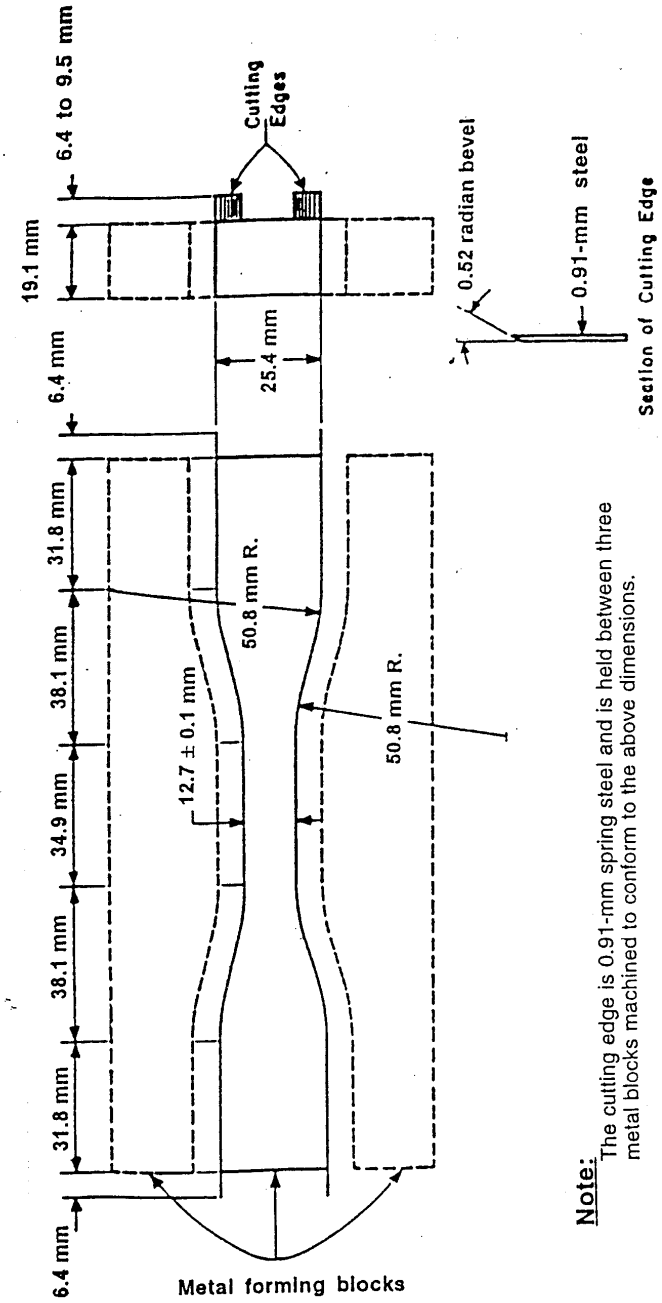

FIGURE 1 - CUTTING DIE FOR TENSILE TESTING