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DIVISION OF ENGINEERING SERVICES
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METHOD OF TESTING COLD APPLIED TWO-COMPONENT POLYSULFIDE POLYMER TYPE JOINT SEALING COMPOUND

A. SCOPE

This method describes the test procedures for the determination of the properties specified for machine extruded or pourable cold applied, two-component, polysulfide polymer type joint-sealing compounds. The base component containing polysulfide polymer shall be referred to as Component B and the component containing the catalyst or accelerator shall be referred to as Component A.

The test method procedure is divided into the following parts:

1. Procedure for Mixing Components
2. Cone Penetration at 25°C and 70°C
3. Cold Flow
4. Resilience at 25°C and 70°C
5. Resilience of Oven-Aged Specimen
6. Bonding to Concrete
7. Non-Volatile Content
8. Viscosity of Components
9. Pot Life of Mixed Joint Sealant

B. REFERENCES

California Test 206
ASTM Designations: C 192, C 719, D 217 and D 5329

C. PROCEDURES

PART 1. PROCEDURE FOR MIXING COMPONENTS

1A. SCOPE

This part of the test method describes the preparation and mixing of the two components of a joint sealant. Incomplete mixing or improper proportioning of the components will not give true test results.

1B. APPARATUS

A laboratory mixer with a propeller-type paddle, 2.5 inches in diameter that can operate at 800 ± 100 rpm.

1C. TEST PROCEDURE

1. It is important that each individual component be uniformly mixed by vigorous stirring before any tests are performed. This is especially important with Component A if it appears in liquid form. In this case, extreme care must be taken to ensure that all settled material is uniformly reincorporated.
2. Weigh a sufficient amount of the two components in the ratio given by the manufacturer to give a volume of about a pint or a quart.
3. Mix the materials with the laboratory mixer for 5 min in a can of approximately twice the volume of the sample. Scrape the sides and bottom of the container occasionally during this mixing period.
4. Mix one batch for the cold flow and volatile tests and a second batch for the penetration, resilience, and bond tests. While pouring the samples, do not scrape the sides or bottom of the container.

PART 2. CONE PENETRATION AT 25°C AND 70°C

2A. SCOPE

This part of the test method is essentially the same as ASTM Designation: D 217. Penetration is expressed as the distance measured to the nearest 1 dmm that the cone penetrates vertically into a sample.

2B. APPARATUS

1. A penetrometer shall be used as specified in ASTM Designation: D 217.
2. Six ounce seamless ointment containers shall have an approximate diameter of 4 in. and approximate height of 1.5 in.
3. A grease cone is required as specified in ASTM Designation: D 217. The total load on the sample, including the penetration cone and penetrometer shaft, shall be 150 g. Handle the cone carefully to avoid bending of the shaft or dulling of the point. If the cone is cleaned with a solvent, be sure the solvent has evaporated completely before use of the cone.
4. A forced air-circulating oven capable of maintaining a temperature of $70\text{ }^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

2C. TEST PROCEDURE, SAMPLES AT 25°C

1. Fill the 6 oz ointment can with the compound; strike off and smooth the surface. Allow the specimens to cure 24 hr or other specified time at room temperature.
2. Condition the sample and cone at $25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 1 hr.
3. Using the penetration cone attachment with the cone set at its highest position and the dial set at zero, lower the indicator assembly until the tip of the cone is in contact with the surface of the specimen. Lock in place.
4. Release the cone shaft locking mechanism for 5 s to permit the cone to penetrate

the surface being tested.

5. Lock the cone shaft, push dial needle shaft down to meet the cone shaft and read the indicating dial.
6. Make three tests for penetration on each sample at points on the surface not less than $\frac{3}{4}$ inch apart and $\frac{3}{4}$ inch from the edge of the container.

2D. TEST PROCEDURE, SAMPLES AT 70°C

1. Prepare the samples and allow them to cure as described in Section C.
2. Condition the sample and cone in an oven at $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 90 min.
3. Remove the sample and cone from the oven. Insert the cone in the penetrometer. Use the same procedure as in Section C to determine the penetration with the exception that the test must be completed within 20 s after removing sample from oven. Return the cone and sample to the oven for 5 min and repeat the determination. Obtain three penetration values in this manner.

PART 3. COLD FLOW

3A. SCOPE

This part of the test method describes the procedure for determining the flow of joint sealing compounds at room temperature.

3B. APPARATUS

Two channels, 1 in. wide, 1.5 in. deep and 24 in. long, formed with 24 gauge black iron, galvanized iron or aluminum sheet. Metal plates 1.5 in. \times 1 in. \times $\frac{1}{2}$ in. are inserted near each end of the channels. The metal plates shall be 20 in. apart and held in place with "C" clamps. In order to support the channels in a vertical position from a nail or hook, $\frac{1}{4}$ in. diameter holes are cut in the bottom of the channels, $\frac{1}{2}$ in. from the end.

3C. PREPARATION OF SAMPLES

1. Fill the channels with the sealing compound from the bottom to the top without the entrapment of air.
2. Strike off the excess compound.
3. Remove the plates from one channel at 3 min after filling.
4. Suspend the specimen vertically with the end last filled down.
5. Remove the plates from the second channel 40 min after filling.
6. Suspend the specimen vertically with the end last filled down.
7. Report the total movement of the bottom of the specimen 1 hr after removing the plates, in inches.

PART 4. RESILIENCE AT 25°C AND 70°C

4A. SCOPE

This part of the test method describes the procedure for determining the resilience of joint sealing compounds. It is a measure of the resistance of the compound to infiltration of granular material.

4B. APPARATUS

1. Use the apparatus for penetration as specified in ASTM Designation: D 217, except that the ball penetration tool attachment is used in place of the cone.
2. The ball penetration tool is described in ASTM Designation: D 5329.
3. An oven shall be used as specified in Part 2, Section B.

4C. TEST PROCEDURE, SAMPLES AT 25°C

1. Prepare the samples and condition them in the same manner as for the penetration test at 25°C. See Part 2, Section C of this test method.
2. Determine resilience per ASTM Designation: D 5329.

4D. TEST PROCEDURE, SAMPLES AT 70°C

1. Prepare and cure the samples as for 25°C above.
2. Condition the samples and ball penetration tool for 90 min in an oven at 70°C ± 1°C.
3. Remove the penetration tool and sample from the oven. Insert the tool in the penetrometer and complete the resilience reading within 55 s after removing the sample from the oven.
4. Return the sample and tool to the oven for 10 min before making another reading.
5. Obtain three readings, reporting the average resilience reading.

PART 5. RESILIENCE OF OVEN-AGED SPECIMEN

5A. SCOPE

The resilience of an oven-aged sample of joint sealing compound is intended to measure its resistance to infiltration of mineral matter after the compound has been subjected to an elevated temperature.

5B. APPARATUS

The apparatus used is the same as described in Part 4.

5C. TEST PROCEDURE

1. Place the specimen from the resilience test, of the original sample used in Part 4, in a forced air circulating oven maintained at 70°C ± 1°C.

2. After seven days, remove it from the oven and condition in air for 2 hr at $25^{\circ}\text{C} \pm 1^{\circ}\text{C}$.
3. Then make three tests for resilience as described in Section C of Part 4.

PART 6. BONDING STRENGTH TO CONCRETE

6A. SCOPE

This part of the test method is intended to measure the adhesion of joint sealing compounds to clean dry or moist portland cement concrete with sawed faces when subjected to a temperature of minus 29°C .

6B. APPARATUS

1. Bond extension machine: The apparatus shall be capable of extending the bond specimens as described below from a spacing of $\frac{1}{2}$ in. between blocks to 1 in. at a uniform rate of $\frac{1}{8}$ in. per hour at -29°C . ASTM Designation: C 719 shows a machine that meets this requirement; however, machines of other designs may also be satisfactory.
2. Cooling chamber: A refrigerated enclosure shall maintain the specimens mounted in the bond extension machine at $-29^{\circ}\text{C} \pm 1^{\circ}\text{C}$.
3. Brass spacers are required for molding the specimens: $\frac{1}{2}$ in. \times $\frac{1}{2}$ in. \times 2 in.
4. 3 in. \times 5 in. tin-plated panels.
5. Molds for concrete blocks: The mold shall be of metal and shall be provided with a detachable metal base plate. Means shall be provided for securing the base plate to the mold. The assembled mold and base plate shall be watertight and shall be oiled with mineral oil before use. The inside measurement of the mold shall be 10 in. wide \times 17 in. long \times 3 in. high.
6. A concrete saw having a diamond or silicon carbide cutting edge is required. Blades to be of such size that the saw cut does not exceed $\frac{3}{8}$ in. in width.
7. A drying oven is required that can hold a temperature of $104^{\circ}\text{C} \pm 3^{\circ}\text{C}$.

6C. MATERIALS USED IN CONCRETE BLOCKS

1. Aggregate: The aggregate shall conform to the requirements in Section 90 of the Caltrans Standard Specifications and the following detailed requirements:

The coarse aggregate shall consist of crushed limestone, have an absorption of not more than 1.5 %, as determined by California Test 206, and shall conform to the following grading:

Sieve Size	Percent Passing
$\frac{3}{4}$ in.	98.5 ± 1.5
$\frac{1}{2}$ in.	66 ± 3
$\frac{3}{8}$ in.	33 ± 3
No. 4	1.5 ± 1.5

The fine aggregate shall consist of natural siliceous sand and shall conform to the following grading:

Sieve Size	Percent Passing
No. 4	100
No. 8	85 ± 3
No. 16	65 ± 5
No. 30	45 ± 5
No. 50	21 ± 5
No. 100	7 ± 2

2. Portland Cement: The portland cement shall conform to the requirements in Section 90 in Caltrans Standard Specifications.

6D. DESIGN OF CONCRETE MIX

1. The concrete shall have an approximate water-cement ratio of 0.48 that produces concrete with a slump of 2.5 in. ± 0.5 in.
2. The ratio of fine aggregate to total aggregate shall be approximately 40 %, by solid volume.
3. The cement content shall be 6 ± 0.5 bags per cubic yard of concrete.
4. The air content shall be 5.0 % ± 0.5 % and shall be obtained by the addition to the batch as mixed of a sufficient quantity of an air-entraining admixture.

6E. FABRICATION OF TEST BLOCKS

1. Mix the concrete, fill the mold and moist cure the block in accordance with ASTM Designation: C 192.
2. After moist curing the specimens for not less than 28 days, saw the concrete block into 1 in. × 2 in. × 3 in. test blocks in the following manner:
 - a. First cut the 10 in. × 17.5 in. × 3 in. block in half by making a cut down the long axis through the center. This yields two slabs.
 - b. Cut each into two 5 in. × 17.5 in. × 3 in. slabs by making cuts at a distance of 2 inches from the sawed faces.
 - c. Then saw each of the four 2 in. × 17.5 in. × 3 in. slabs obtained as described above, into twelve 1 in. × 2 in. × 3 in. blocks.
 - d. Cut a ½ in. portion from the 2 in. × 3 in. face that was in contact with the mold face. Then cut twelve 1 in. × 2 in. × 3 in. blocks from the slab.
 - e. Each 10 by 17.5 in. × 3 in. slab yields forty-eight 1 in. × 2 in. × 3 in. test blocks. These dimensions permit sawing by blades up ⅜ in. wide.
3. After sawing and while still wet from the sawing operations, lightly scrub the surfaces of the blocks with a stiff-bristled brush while holding under a stream of running water.

4. Then store the blocks in the laboratory air in such a manner as to preclude dust accumulation on the surfaces.

6F. PREPARATION OF TEST SPECIMENS

1. Prepare six test specimens – three for the dry-bond and three for the wet-bond tests. Two blocks are required for each specimen.
2. Dry six blocks for the dry-bond test to a constant weight in an oven maintained at $104^{\circ}\text{C} \pm 3^{\circ}\text{C}$. Then brush the blocks with a stiff-bristled brush and place the blocks in a desiccator until used.
3. Soak six blocks in water for 24 hr. Remove the blocks from water and drain off the excess moisture before assembling them for testing, as described below.
4. Prepare each of the three dry-bond specimens by placing two dry concrete blocks on a base plate with the 1 in. \times 3 in. faces in contact with the base plate.
5. Space the concrete blocks $\frac{1}{2}$ in. apart by means of blocks $\frac{1}{2}$ in. \times $\frac{1}{2}$ in. \times 2 in. standing on their $\frac{1}{2}$ in. \times $\frac{1}{2}$ in. ends and forming an opening between them $\frac{1}{2}$ in. \times 2 in. long. Use clamps, tape or other suitable means to hold the blocks in position.
6. Prepare the six wet-bond blocks using the same procedure as with the dry-bond blocks. Put the wet-bond blocks into water until immediately before filling the spaces between them.
7. Fill the spaces between the dry-bond blocks with the freshly mixed compound to be tested from the bottom to the top to exclude air pockets. Strike off excess overflow with a spatula.
8. Immediately before filling each wet-bond specimen, remove from water and drain. Wipe off excess moisture from all surfaces of the hole to be filled, using a dry absorbent swab. Fill in the same manner as for the dry-bond specimens.

6G. TESTING THE SPECIMENS

1. After conditioning the specimen blocks for 24 hr at room temperature, place them in the holders of the bond extension machine for further conditioning at $-29^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 1 hr.
2. Place slight tension on the specimens.
3. Start the bond extension machine.
4. Operate the machine for the time required to obtain the specified extension (2 hr for 50 % or 4 hr for 100 % extension).
5. Stop the bond extension machine and release the tension on the concrete block specimens.
6. Remove the specimens from the machine, expose them to room temperature for 30 min, and then examine them immediately for defects described in Section H.

7. Repeat the above procedure twice, which comprises three cycles or one complete test.

6H. INTERPRETATION OF TEST RESULTS

A bond block shall be considered a failure if a separation in the sealing compound or a separation between the sealing compound and concrete block occurs which, when measured perpendicularly to the face of the sealant, is in excess of the following amount:

- a. Dry-bond blocks, 1/8 in.
- b. Wet-bond blocks, 1/4 in.

PART 7. NONVOLATILE CONTENT

7A. SCOPE

This part of the test describes the procedure for determining the non-volatile content of joint sealant at 70°C.

7B. TEST PROCEDURE

1. Transfer 3 g to 5 g of freshly mixed material to a tared dish, approximately 2.5 inches in diameter. Spread the material with a 1 in. wide square ended spatula to cover the bottom of the dish with a uniform thickness of about 1/32 in. Prepare samples in duplicate
2. Weigh to the nearest 1 mg and immediately place the sample in a forced air-circulating oven for 24 hr at 70°C ± 1°C.
3. Cool the sample to room temperature and reweigh to nearest 1 mg.
4. Calculate the nonvolatile content, to the nearest 1 %, as follows:

$$\text{Nonvolatile} = \frac{(\text{Final mass of the sample} \times 100)}{\text{Initial mass of the sample}}$$

PART 8. VISCOSITY OF COMPONENTS

8A. SCOPE

This part of the test describes the procedure for determining the original viscosity of the two components used to make machine extruded joint sealants.

8B. APPARATUS

Brookfield viscometer: This device shall be capable of operating at 5 rpm. It shall be equipped with a No. 5 spindle.

8C. PREPARING AND TESTING SAMPLES

1. Mix each component until it is uniform. See precautions in Part 1.

2. Fill a pint or quart can approximately 75 % full with the component to be tested. Adjust the temperature of the sample to 25°C.
3. Determine the viscosity using the No. 5 spindle on the Brookfield viscometer set at 5 rpm.

PART 9. POT LIFE OF MIXED JOINT SEALANT

9A. SCOPE

This part of the test describes the procedure for determining the viscosity of pourable joint sealants 5 min after mixing and also for determining the application time or pot life.

9B. APPARATUS

In addition to the Brookfield viscometer described in Part 8, use of a No. 6 spindle is required.

9C. PREPARING AND TESTING SAMPLES

1. Adjust the two components to 25°C.
2. Mix the sample as described in Part 1 of this test method.
3. Fill a pint or quart can approximately 75 % full.
4. When 5 min has elapsed after the end of the mixing period, determine the viscosity using the No. 5 spindle at 5 rpm.
5. Remove the No. 5 spindle from the viscometer. Clean the spindle at once. Replace it with a No. 6 spindle. Adjust the mixture to 25°C.
6. At 5-min intervals, determine the viscosity using the No. 6 spindle at 5 rpm.

9D. TEST REPORT

Report application time as the time at which the viscosity of the sealant is first equal to or greater than that specified for application time.

D. HEALTH AND SAFETY

It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Prior to handling, testing or disposing of any materials, testers must be knowledgeable about safe laboratory practices, hazards and exposure, chemical procurement and storage, and personal protective apparel and equipment.

Caltrans Laboratory Safety Manual is available at:

http://www.dot.ca.gov/hq/esc/ctms/pdf/lab_safety_manual.pdf

Users of this method do so at their own risk

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(California Test 413 contains 9 pages)**