HIGHWAY RESEARCH REPORT

CURING COMPOUNDS FOR PORTLAND CEMENT CONCRETE

REPORT

68-05

STATE OF CALIFORNIA

TRANSPORTATION AGENCY

DEPARTMENT OF PUBLIC WORKS

DIVISION OF HIGHWAYS

MATERIALS AND RESEARCH DEPARTMENT

RESEARCH REPORT

NO. M & R 635149

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Prepared in Cooperation with the U.S. Department of Transportation, Bureau of Public Roads August, 1968

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STATE OF CALIFORNIA—TRANSPORTATION AGENCY

RONALD REAGAN, Governor

DEPARTMENT OF PUBLIC WORKS
DIVISION OF HIGHWAYS
MATERIALS AND RESEARCH DEPARTMENT
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August, 1968
Interim Report
M&R No. 635149
Subproject No. 39154
Federal No. F-4-13

MR. J. A. LEGARRA
State Highway Engineer

Dear Sir:

Submitted herewith is a research report titled:

CURING COMPOUNDS FOR
PORTLAND CEMENT CONCRETE

Interim Report

Donald L. Spellman
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Co-Investigators

Very truly yours,

JOHN L. BEATON
Materials and Research Engineer

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REFERENCE: Spellman, D. L.; Stoker, J. R.; and Ford, R. W. "Curing Compounds for Portland Cement Concrete" State of California, Department of Public Works, Division of Highways, Materials and Research Department, Research Report M&R No. 635149 Subproject No. 39154, Federal No. F-4-13, August, 1968

ABSTRACT: This report summarizes the work done to date on a research project for developing improved and accelerated methods for acceptance testing of concrete curing compounds. The emphasis has been chiefly on developing (1) modified test methods for the water retention efficiency of curing compounds, and (2) a field method for determining rate of application of curing compounds to concrete pavements.

Water losses at 24 hours from either mortar specimens or from jars of water covered with filter paper treated with curing compound correlated well with water losses obtained in the 72-hour mortar test (AASHO Designation T-155) for curing efficiency. Anomalies in test results when test conditions are varied indicate a need for determining which of the properties of curing compounds measured in the laboratory influence the properties of membrane cured concrete in the field.

KEY WORDS: Curing agents, accelerated testing, test methods, laboratory tests, field tests, concrete curing, concrete properties

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This project was performed in cooperation with the U. S. Department of Transportation, Federal Highway Administration, Bureau of Public Roads, Federal Program No. F-4-13.

The opinions, findings, and conclusions expressed in this report are those of the authors and are not necessarily those held by the Bureau of Public Roads.

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CURING COMPOUNDS FOR PORTLAND CEMENT CONCRETE

INTRODUCTION

This research project was started for the purpose of developing a new or improved test method for evaluating the water retention efficiency of concrete curing compounds. It was hoped that the time and expense required for testing could be reduced.

The test method currently used by the California Division of Highways (AASHO Designation T-155) could be modified in several ways in order to accelerate the test and to reduce materials costs. Among the modifications considered or studied were:

1. Reducing size of mortar specimen
2. Reducing number of mortar specimens used for test
3. Measuring water losses from test specimens after shorter test periods and correlating such losses with losses at 72 hours
4. Accelerating water losses by elevating curing oven temperature
5. Testing curing compounds over less costly substrates than mortar made with Ottawa sand, e.g., mortar containing concrete sand from a good commercial source, or over vapor transmission test jars.

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CONCLUSIONS

Water losses at 24 hours from mortar specimens treated with concrete curing compounds can be used to predict water losses obtained at 72 hours using the method of AASHO Designation T-155.

Materials costs for the water retention test could be reduced by substituting a water vapor permeability test of filter paper treated with curing compound for the mortar test. Such a procedure, however, would overlook the possibility of adverse reaction between the curing seal and portland cement mortar.

Water retention tests are very sensitive to testing conditions and procedures. The present allowable limits for moisture loss are unnecessarily high and were probably set high because of the poor reproducibility of present test methods. Specific test conditions of standard tests are not adequately controlled in that wording is ambiguous and easily misinterpreted.

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RECOMMENDATIONS

Inasmuch as variations in the laboratory conditions appear to cause considerable variation in water retention test results, correlation with field evaluations of curing compounds is desirable for assessing laboratory results and for setting quality standards. The effectiveness of various materials as curing compounds should be evaluated using other criteria, e.g., the influence on strength, abrasion resistance or other properties of portland cement concrete, in addition to water retention test results.

It is recommended that this project be continued with a broadened scope. The purpose of the extended project will be to define realistic water retention efficiency limits and to determine which laboratory test method is most suitable for measuring the water retention capabilities of curing compounds. Work to be performed under this proposal should be coordinated with other research performed under the project "Concrete Test Methods and Specifications", Federal Program No. F-4-13, and another project "Research of Highway Concrete Problems", Federal Program No. D-3-17.

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REPORT OF PROGRESS

Under this project, a number of testing procedures have been attempted for evaluating concrete curing compounds.

1. Mortar Methods

These methods are modifications of AASHO Designation T-155.

a. The "Small Pan" Method

In this method, the mortar specimens have approximately one-fourth the surface area of those used in AASHO Designation T-155. Less material and less oven space is required for this method than for the standard method. It was found inadvisable to reduce the testing time to less than 72 hours with the smaller specimens because of the low losses. To control the rate of application of compound and to determine the water loss properly by this method requires a balance capable of weighing 1600 to 1700 grams rapidly to the nearest 0.1 gram.

b. Accelerated Mortar Method

It was found that water losses at 24 hours could be used to predict the 72-hour loss of tests conducted in accordance with AASHO Designation T-155. Of 197 pans tested at 24 hours, 187 (about 95%) indicated good correlation with the results at 72 hours. This is considered sufficient justification for changes in AASHO T-155 to accelerate testing until an improved method can be developed. In Test Method No. Calif. 534-A (Appendix A), the number of test specimens has been reduced from three pans to two pans per test.

2. Water Vapor Permeability Tests

In these procedures, a membrane formed by applying curing compound to a porous substrate, e.g., filter paper, is used to seal a container partially filled with water. Water loss through the membrane is determined by weighing the container before and after storage in an oven at temperatures up to 155°F.

Several types of containers and methods of sealing the membranes were attempted. Losses were roughly proportional to the area of membrane exposed to the air, and losses were

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considerably greater than those from similar areas of mortar. A preliminary draft has been prepared for a test method in which water loss after 24 hours in an oven operated at approximately 137°F is measured, using wide-mouth, screw-top, pint jars partially filled with water and sealed with filter paper coated with curing compound. (Appendix B.)

3. Other Methods

An attempt was made to equate the visual appearance under the microscope of certain types of curing compounds with their water retention performance. Under this procedure, a film of curing compound .010-inch thick is drawn by means of an adjustable blade over a glass slide. The slide is dried for two hours and then examined under a microscope. Wax based material appears soapy when it is good, but lumpy when it is poor. Wax and resin combination material appears smooth when good, but sandy or gritty when poor. Borderline material is hard to detect, and formulation changes might confuse interpretation. This method is useful for studying and comparing the films formed by various compounds, but is not suitable for acceptance testing.

A few tests of laboratory prepared mortar and concrete specimens for flexural strength and abrasion resistance indicate that these properties are significantly affected by curing methods, but so far are inconclusive in comparing the effectiveness of cure of various membrane curing compounds. Such tests, while having merit because they measure actual curing effectiveness, take a long time to perform and are relatively expensive.

In developing the accelerated test methods and correlating their results with those obtained using AASHO Designation T-155, certain anomalies were discovered which caused laboratory testing of water retention to appear to be somewhat unrealistic for predicting field performance:

1. Occasionally, samples shown to be acceptable by a mortar test method failed when tested using the filter paper method, and vice versa.

2. When the filter paper method was prolonged for 72 hours, the water lost during the third 24-hour period was double that of each of the first two 24-hour periods for all curing compounds tested except one compound based on chlorinated rubber. The chlorinated rubber compound permitted the loss of equal amounts of water in each 24-hour period. The cause for the change in rate of moisture loss is unknown.

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3. Water losses were much greater from mortar specimens when tested at 110°F than when cured at 100°F - significantly more than expected on the basis of the ratio of water vapor pressure at 110°F to the vapor pressure at 100°F. Some types of curing compounds were found to be more sensitive to testing at elevated temperatures than others. Since pavements often reach temperatures of 120°F or more during the first few days after placement, this factor should be further examined.

4. While some curing compounds have nearly the same water retention efficiency when applied at 500 sq.ft./gal. as when applied at 200 sq.ft./gal., other compounds are very sensitive to rate of application. Some of the "poorer" compounds can provide a satisfactory cure if they are applied thicker.

5. Surface texture of mortar and the times at which the finishing and curing operations are performed influence water retention test results. Laboratory mortar specimens for the water retention test do not simulate the types of textures produced on bridge decks and pavements. Therefore, correlations between mortar and concrete surface textures, water retention efficiencies as measured in the laboratory, and the rates of application of curing compounds required in the field for effective curing need to be determined.

In order to compare the effectiveness of field applications of various curing compounds, the amount of material actually applied to the concrete should be determined. This is essential for research work. Test Method No. Calif. 535-A (Appendix C) was developed under this project as a field method for measuring the rate at which curing compound is applied to portland cement concrete pavements. It should prove to be a valuable tool for field inspectors as well.

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MATERIALS AND RESEARCH DEPARTMENT

State of California
Department of Public Works
Division of Highways

Test Method No. Calif. 534-A
October 7, 1968
(4 pages)

METHOD OF TEST FOR WATER RETENTION EFFICIENCY OF LIQUID MEMBRANE-FORMING CONCRETE CURING COMPOUNDS

Scope

This test method, which is a modification of AASHO Designation: T-155 (ASTM Designation: C-156), describes the laboratory procedure for determining the efficiency of liquid membrane-forming curing compounds in preventing moisture loss from concrete during the early hardening period.

Procedure

A. Apparatus

1. Molds - Molds shall be rigid water-tight pans 6x12-inches at the top, 5-3/4 x 11-3/4 inches at the bottom, within ± 1/4-inch, and 2 ± 1/8-inch in depth on the inside. Molds shall have a flat rim at the top on all sides, approximately 1/4-inch in width. It is recommended that this rim be reinforced by welding a 1/4-inch rod around the outside perimeter of the pan, just beneath the rim.

2. Metal Plates - Metal plates shall be 6x12-inch with edges raised slightly to retain sprayed compound.

3. Curing Cabinet - The cabinet for curing the specimens shall maintain a temperature of 100 ± 3F (37.8 ± 1.7C) and a relative humidity of 30 ± 4%. Air flow shall be sufficient to remove the solvent vapors quickly, but no detectable air current shall strike directly on the surface of any test specimen stored in the cabinet.

B. Materials

1. Graded Ottawa sand conforming to the requirements of ASTM Designation: C-109.

2. Portland Cement, Type II. <u>DO NOT USE CONCRETE ADMIXTURES OR AIR-ENTRAINING CEMENT IN THIS TEST.</u>

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C. Preparation of Specimen Molds

Thoroughly clean the molds before each use and apply a thin coating of a suitable mold release compound.

D. Preparation of Mortar Test Specimens

Machine mix all batches of mortar at room temperature (73 ± 3F) following the schedule given for mixing mortars in ASTM Designation: C-305.

1. Number of Test Specimens

a. The test shall consist of two specimens. Prepare each specimen from a separate batch of mortar.

2. Proportioning and Mixing Mortar

a. Trial Batch. Whenever a new lot of cement or sand is to be used, prepare a trial batch of mortar having a water-cement ratio of 0.4 with sufficient sand to produce a flow of 35 ± 5 as measured in ASTM Designation: C-87. Use these proportions in preparing subsequent test batches.

b. Mortar for Test Specimens. Using the mix proportion determined by the trial batch, prepare sufficient mortar to fill a 12x6x2-inch pan.

3. Molding Specimens

Place the mortar in the mold in two approximately equal layers; consolidate each layer by tamping or with a mechanical vibrator. Avoid overvibration. Strike off the excess mortar and finish the surface with a wooden screed having a flat 2-inch wide screeding surface. Advance the screed along the long axis of the specimen using a circular motion. Do not work the surface more than necessary to produce a reasonably even finish.

4. Storage of Specimens

a. After molding, place the specimens in the curing cabinet in a level position. Space the specimens uniformly on the shelf with a clear space of from two to seven inches on all sides of each specimen. Use dummy specimens to replace test specimens when a shelf is not filled with test specimens.

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b. Remove test specimen from the cabinet 1-1/4 to 1-3/4 hours after molding. Use a putty knife or paint scraper to form a "V" shape groove approximately 1/8-inch deep and not over 1/16-inch wide between the edge of the mortar and the mold. (Mortar should be dry enough that the groove will not collapse or fill with water.) Fill the groove with a sealant which will remain pliable at 100F and which is resistant to the solvents in the curing compound. Return the specimen to the cabinet for approximately 1/2 hour.

c. Test the mortar surface condition by rubbing a small area with the finger tip. The specimen shall be ready for brushing when rubbing produces only a few fine bubbles. Brush the entire surface of the mortar lightly in the longitudinal direction with a 2-inch flat sash tool, Morck 791-2 or equivalent. (Brushing shall be just sufficient to remove the sheen from the surface.)

E. Application of Curing Compound

1. Mortar Specimens

Immediately after brushing, weigh the mortar specimen to the nearest gram. With a suitable spray gun, and, unless otherwise specified, apply ten (10) grams of curing compound in a uniform coating on the mortar specimen. Keep overspray to a minimum. Wipe off any drops of compound which may adhere to the under surface of the rim but do not attempt to remove compound from upper surface of the rim of the pan. Determine the actual rate of application by reweighing the mortar specimen immediately after applying the curing compound.

2. Metal Plate

Weigh the metal plate to the nearest gram. Apply ten (10) grams of curing compound in the same manner used for the mortar specimen. Determine the actual rate of application by reweighing the plate immediately after applying the curing compound.

F. Testing of Treated Specimens

After applying the compound and reweighing, place the mortar specimens and the metal plate in the curing cabinet. Cure for 24 hours at 100 $\pm$ 3F and 30 $\pm$ 4% relative humidity. Then remove from the curing cabinet and weigh to the nearest gram.

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G. Calculation

1. Calculate the total weight lost by each mortar specimen as the weight of the specimen immediately after applying the curing compound less the weight of the specimen after the 24-hour curing period.

2. Calculate the volatile loss of the curing compound in grams as the weight of the metal plate immediately after applying the compound less the weight of the plate after 24 hours in the curing cabinet.

3. Calculate the water loss in grams from each mortar specimen as the total weight lost by the specimen less the volatile loss of the curing compound.

Reporting of Results

Record all weights and calculations on Form HMR T-577. Report the results of this test on Form T-584.

References

AASHO Designation: T-155
ASTM Designations: C-87, C-109, C-156, and C-305

End of Text on Calif. 534-A

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MATERIALS AND RESEARCH DEPARTMENT

State of California
Department of Public Works
Division of Highways

Test Method No. Calif. 535-A
October 7, 1968
(3 pages)

METHOD OF TEST FOR DETERMINING APPLICATION RATE OF CONCRETE CURING COMPOUND IN THE FIELD

Scope

This test method, an adaptation from Test Method No. Calif. 339, describes the procedure for determining the rate at which concrete curing compound is applied to portland cement concrete pavements.

Procedure

A. Apparatus

1. Balance, capacity 100 grams, sensitive to 0.1 gram
2. Suitable weighing box or windshield for balance
3. Plastic sheet, .040" thick by 4"x8". This may be cut from sheet plastic template material, cellulose acetate, Service and Supply No. 17246.

B. Materials

1. Jute stock paper, 150# Riegles, 8-1/2" x 11"
2. Cotton pads, 4" x 8" (Bauer & Black No. 540 or equal)
3. Double coated pressure-sensitive tape, 1/4" width
4. Double coated pressure-sensitive tape, 1" width

C. Preparation of Test Plates

1. Attach cotton pads to plastic sheets with 1/4" double coated pressure-sensitive tape.
2. Weigh the entire plate to establish tare weight
3. Attach the plate to 1/2 sheet (5-1/2" x 8-1/2") of jute stock with 1" double coated pressure-sensitive tape as shown in Figure 1.

D. Sampling and Weighing

1. Longitudinal Distribution

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Place 5 test plates along the pavement approximately 3 feet from the edge at random intervals (7-13') over a 50-foot length ahead of the spray rig.

(See Figures II and III.)

2. Transverse Distribution

Where fixed nozzles on a distributor bar are used, it is desirable to determine transverse distribution. Determine the transverse distribution either at the time of starting or at the time of completing the application of compound by placing 5 test plates across the slab. Space the test plates at random, or under nozzles which appear to be delivering at abnormal rates. Make arrangements to place the test plates on the pavement and to remove them without stepping on newly placed concrete. Have the spray rig travel at its normal rate at the time it passes over the test plates.

3. As soon as the spray rig has passed, remove the test plates from the pavement. Remove and discard the jute paper backing.

4. Immediately weigh the plates to the nearest 0.10 gram and record its weight.

E. Calculations

1. Calculate the total weight of curing compound applied to each test plate as the final weight less the tare weight. Read the approximate application rate in square feet per gallon from Table 1. Calculate the corrected rate of application at each test plate by multiplying the approximate rate by the specific gravity of a well-mixed representative sample of the curing compound. (If possible, this sample should be taken from a feed line to the spray nozzle. The specific gravity shall be determined by means of a suitable pycnometer or hydrometer.)

2. Calculate the average application rate in square feet per gallon as the sum for the test plates divided by five.

F. Notes

1. Weigh the wet test plates as soon as possible to prevent loss of volatiles. Under extreme drying conditions, include a plastic bag with the tare weight. Immediately after the spray rig passes, place the wet pad inside the bag and weigh.

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2. Time the rate of advance of the spray rig by means of a stop watch over several fifty-foot sections to establish the average time of travel for fifty feet. Then check the time taken to spray the test section by the same method to determine if the spray rig operator maintains the usual forward speed. If the time varies more than 10% from the average, consider the test invalid and repeat the test.

3. Shield test pads placed near the edge of the pavement slab from overspray from nozzles applying compound to the exposed edge of slipformed pavement.

REFERENCES

Test Method No. Calif. 339

End of Text on Test Method No. Calif. 535-A

Table 1 attached
Figures I, II, III, and IV attached

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TABLE 1

CONVERSION TABLE
SPRAY RATE, SQUARE FEET PER GALLON
FROM NET WEIGHT OF CURING COMPOUND ON 4"x8"
TEST PANELS

Grams	.0	.1	.2	.3	.4	.5	.6	.7	.8	.9
1	840	764	700	646	600	560	525	494	467	442
2	420	400	382	365	350	336	323	310	300	290
3	280	271	262	255	247	240	233	227	221	215
4	210	205	200	195	191	187	182	179	175	171
5	168	165	162	158	155	153	150	147	145	142
6	140	138	135	133	131	129	127	125	124	122
7	120	118	117	115	114	112	110	109	108	106
8	105	103	102	101	100	99	98	97	96	94
9	93	92	91	90	90	88	88	87	86	85
10	84	83	82	82	81	80	79	79	78	77
11	76	76	75	74	74	73	72	72	71	71

Example: 5.6 gms. = 150 sq.ft. per gallon

Approximate rate of application - sq.ft./gallon based on a specific gravity of 1.00

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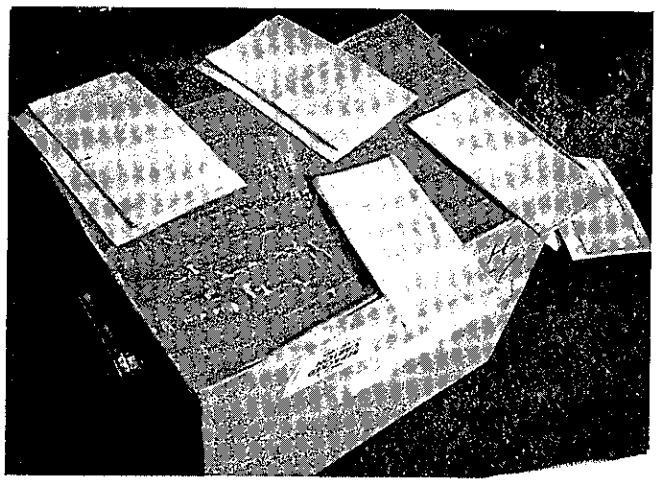

FIGURE I
Test Plates

FIGURE II
Placing Test Plates on Pavement

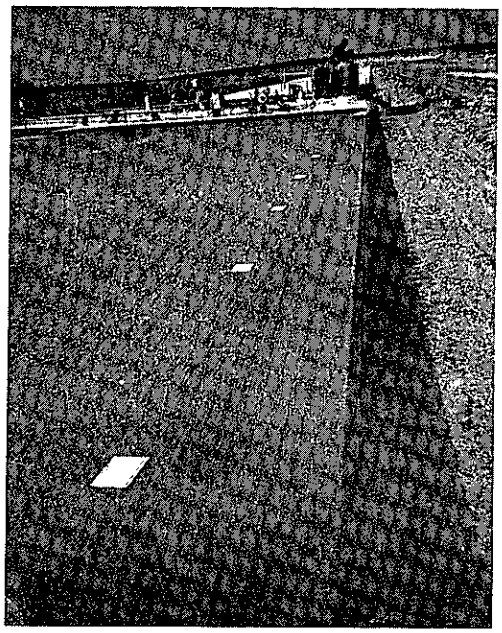

FIGURE III
Test Plates in Position for Test

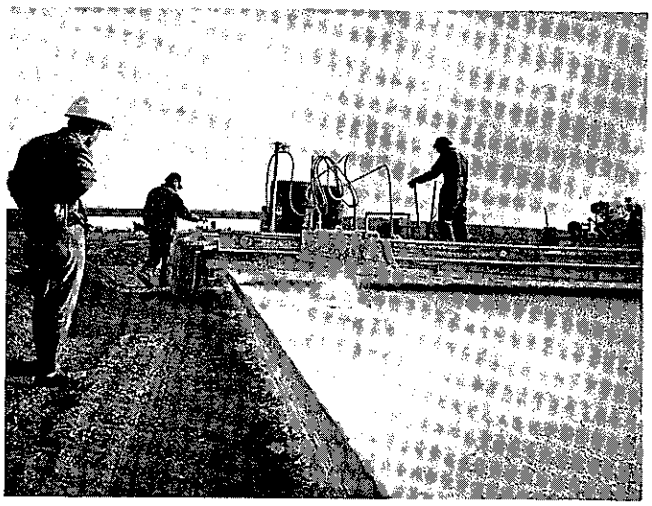

FIGURE IV
Spray Rig Passing over Test Plates

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TENTATIVE METHOD OF TEST FOR WATER VAPOR TRANSMISSION THROUGH CONCRETE CURING COMPOUNDS

Scope

This test method is intended for laboratory use in determining the efficiency of liquid membrane-forming compounds as measured by their ability to prevent water vapor transmission.

Procedure

A. Apparatus

1. Balance sensitive to 0.1 g. with a capacity of at least 600 g.
2. Suitable shield for balance.
3. Electrically heated forced ventilation oven with a wind velocity of 3-5 mph. capable of maintaining a temperature of 137°F±3°F. In addition, it shall contain a rotating shelf with minimum dimensions of those described in ASTM Designation D-6-67.
4. One pint widemouth screw cap glass jars with lid rings.
5. External mix spray gun.
6. 11 cm. shark skin filter papers.
7. Graduated cylinder.

B. Test Record Form

No specific form is provided for reporting results.

C. Test Procedure

1. Place balance shield over balance.
2. Weigh several individual filter papers and determine average tare weight.
3. Place a filter paper on the balance, then thoroughly mix the curing compound sample and spray one gram of the curing compound evenly over the entire surface of the paper. Prepare four filter papers for each compound to be tested. The spraying shall be done within a maximum of 2 minutes. (This is to

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prevent the rapid loss of volatiles which could adversely affect the coverage.)

4. After spraying, let the filter papers dry for 30 to 45 minutes.

5. A few minutes before the above drying period ends fill 4 pint jars for each test with 300 ml of distilled water. Included with the 4 jars for each compound tested shall be at least one control jar for each shelf in the oven. The control shall consist of a filter paper which has no curing compound sprayed on it.

6. Wipe the rim of the jars dry, then center the filter papers on the jars. Place a jar ring over each of the filter papers and tighten. When tightening, care should be taken not to wrinkle the filter paper.

7. Weigh the jars and record the initial weight and time. Then place the specimens on a rotating shelf in the oven.

8. After a 24-hour period, remove the specimens from the oven, reweigh and calculate the loss.

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