

Moisture Retention Tests and Agitation for Membrane-Forming Curing Compounds for Portland Cement Concrete

MATTHEW D. LOEFFLER, CHRYSSIS G. PAPALEONTIOU, A. H. MEYER, AND D. W. FOWLER

Discussed in this paper are the relative merits of Texas specifications (Tex-219-F) and ASTM specifications (ASTM C 156-80) for the testing of moisture retention by liquid membrane-forming curing compounds. A comparison of the effectiveness of four motorized agitation devices to be used through drum bungholes is also presented. Preliminary work toward the development of a new moisture-retention test to replace Tex-219-F and ASTM C 156-80 is also outlined, in addition to suggestions for the direction of continuing research. Recommendations are presented concerning the 6-month curing compound shelf life in effect at the time of the study and the possibility of extending this shelf life. Research is also reported on the effects of altering application rates and pattern on moisture retention. In addition, the use of optical reflectance as a measure of application rate is examined. Finally, findings on the usefulness of in-line testing samples compared with samples from stored drums are presented.

Curing is recognized as an important process in the manufacture of durable portland cement concrete (PCC). Rapid evaporation of water from the surface of fresh PCC may lead to plastic shrinkage cracking, which becomes critical when the rate of evaporation exceeds $1.0 \text{ kg/m}^2/\text{hr}$. Additionally, inadequate moisture during curing results in lower strength PCC.

Liquid membrane-forming curing compound is one of the materials used for curing PCC pavements. Two general types of compounds are included in the American Society for Testing and Materials (ASTM) and Texas State Department of Highways and Public Transportation (SDHPT) Specifications: (a) Type 1-D, clear or translucent with fugitive dye, and (b) Type 2, white pigmented.

The SDHPT test method currently used is a modification of the ASTM test procedure and was intended to ensure a higher-quality curing membrane for use in the field. Some manufacturers of curing compounds have questioned the suitability of the SDHPT method for evaluating the moisture retention performance of compounds, stating that it is not consistent and not as reliable as the ASTM method.

The SDHPT and ASTM methods for moisture retention are both time consuming and difficult to perform. They involve the proportioning and mixing of mortar, molding and initial curing of specimens, compound application, and final curing. Total test time is more than 72 hr, and two persons are typically required to work at the same time.

Agitation of curing compounds before field application is important in order to achieve a mix of uniform consistency to ensure proper moisture retention and ease of application. Mixing should provide a uniform distribution of pigments and dyes, which makes it possible to detect a nonuniform application by careful visual inspection.

Present SDHPT specifications require liquid membrane-forming curing compound to be storable for 6 months between manufacture and use. This limit has not proven too long to date, but there have also been no experimental data to indicate that a longer or shorter period would be more appropriate.

Recent changes in standard textures used on pavements in Texas have raised questions about both the most suitable compound application rate and the pattern to be used in conjunction with those new textures.

Realizing the need for study in the area of curing compounds, the Texas SDHPT initiated a research study, "Effectiveness of Texas Membrane Curing Compound Quality and Application Requirements," in September 1984. Under the direction of the Materials and Test Division (D-9), and the Center for Transportation Research (CTR), six different curing compounds were selected for laboratory testing. The performance of the SDHPT moisture retention test was compared with the ASTM test, as well as the mixing quality of curing compounds using four different agitation devices. In addition, a study was made of the feasibility of developing a new moisture-retention test that would ensure the same quality of curing compounds and reduce the time and expense of the test.

Tests were also performed on the same six compounds already mentioned on actual shelf life of the compounds. Additionally, seven alternate application patterns and two application rates were checked for moisture retention effectiveness. An attempt was also made to devise an optical reflectance meter to measure compound application rate. Finally, in-line versus drum sample data were analyzed for differences in relevant properties.

MOISTURE RETENTION TESTS

A series of multiple moisture retention tests was conducted to investigate the performance of Texas SDHPT method Tex-219-F in relation to ASTM method C 156-80. Five curing compounds were selected for testing. The manufacturers are identified in this paper as S, P, and M. Designations 1D and 2 next to the letters indicate compound types. The Bituminous Section of the Materials and Test Division (D-9) of the SDHPT performed

M. D. Loeffler and D. W. Fowler, University of Texas at Austin, Austin, Tex. 78712-1076. C. G. Papaleontiou, Center for Transportation Research, University of Texas at Austin, Austin, Tex. 78712-1076. A. H. Meyer, Department of Civil Engineering, University of Texas at Austin, Austin, Tex. 78712-1076.

tests on the five compounds in accordance with the SDHPT method. The Center for Transportation Research at The University of Texas at Austin performed tests in accordance with the ASTM method. Comparison of the two test methods revealed the following procedural differences.

1. The method of calculating moisture loss. ASTM expresses loss in terms of the surface area of the specimen, whereas SDHPT calculates loss in terms of the water present in the specimen at the time of application.

2. The test method for calculating the loss of volatiles from the curing compound. ASTM determines the weight loss by spraying a metal plate with the same quantity of compound used on the specimens and placing it in the chamber with the specimens for 72 hr. The SDHPT calculates the loss in weight of about 2 g of compound placed in a small aluminum dish and heated at a temperature of 220°F for 3 hr.

3. The type of cement used for manufacturing specimens.

ASTM uses Type I cement, whereas SDHPT uses a specific brand of Type III cement.

The moisture loss results on the five compounds are listed in Table 1. For comparison purposes SDHPT results are also shown converted into ASTM units. Table 2 indicates the results expressed as a percentage of the maximum loss allowed by the two methods. It can be seen from Table 1 that both tests exhibited relatively high variability. The ASTM test showed somewhat higher coefficients of variation (CV), however, in four out of the five compounds tested. The *F*-test for variances indicated that at a 5 percent significance level only one ASTM test had a higher variability than the respective SDHPT test. Comparison of the CV of the SDHPT moisture losses and the SDHPT losses expressed in accordance with the ASTM test shows no difference between the values. This indicates that the SDHPT method of calculating moisture loss does not produce any benefit over the ASTM method. The method requires a

TABLE 1 COMPARISON OF MOISTURE LOSS RESULTS IN ACCORDANCE WITH ASTM C 156-80 AND TEX-219-F

72-Hour Moisture Loss	S-1D	S-2	P-1D	P-2	M-1D	Average
Average Moisture Loss (ASTM C 156-80) (kg/m ²)	0.193	0.141	0.190	0.240	0.226	--
Std. Dev. (kg/m ²)	0.092	0.033	0.063	0.075	0.069	0.066
CV (%)	47.7	23.4	33.2	31.3	30.5	33.2
Average Moisture Loss (Tex-219-F) (%)	1.11	2.29	2.60	1.68	0.98	--
Std. Dev. (%)	0.29	0.20	0.30	0.42	0.33	0.31
CV (%)	26.1	8.7	11.5	25.0	33.7	21.0
Average Moisture Loss (Tex-219-F) Converted to ASTM (kg/m ²)	0.178	0.367	0.417	0.271	0.158	--
Std. Dev. (kg/m ²)	0.048	0.031	0.047	0.068	0.052	0.049
CV (%)	27.0	8.4	11.3	25.1	32.9	20.9

TABLE 2 72-HR MOISTURE LOSS RESULTS EXPRESSED AS A PERCENTAGE OF THE MAXIMUM ALLOWABLE LOSS

	Compound Brand/Type				
	S-1D	S-2	P-1D	P-2	M-1D
ASTM C 156-80 0.55 x 100 (%)	35.1	25.6	34.5	43.6	41.1
Standard Deviation (%)	16.7	6.0	11.5	13.6	12.5
Tex-219-F 4% x 100 (%)	27.8	57.3	65.0	42.0	24.5
Standard Deviation (%)	7.3	5.0	7.5	10.5	8.3

TABLE 3 COMPARISON OF ASTM C 156-80 AND TEX-219-F SOLIDS WEIGHT RESULTS IN CURING COMPOUNDS

Compound Brand/Type	ASTM C 156-80 (g)	Tex-219-F (g)
S-1D	2.6	2.6
S-2	5.6	4.5
P-1D	3.2	4.1
P-2	3.9	5.0
M-1D	2.7	2.2
M-2	—	4.7

tighter quality control during the test, however, and also some more weight recordings.

Statistical *t*-tests between ASTM and SDHPT values (Table 1) showed that at a 95 percent confidence level each test method is equally likely to result in higher or lower moisture losses than the other method. In addition, statistical tests on values expressed as a percentage of the maximum loss allowed by each method (Table 2) indicated that each method is equally likely to give stricter values than the other.

The average solids weights contained in curing compounds as determined from the two test methods are shown in Table 3. Differences in weights are insignificant as they correspond to moisture loss of less than 0.02 kg/m².

No conclusion can be drawn from how using one brand of cement affected the variability of the tests because such a study was not attempted. However, the use by SDHPT of Type III,

high early strength cement, reduced the initial curing time of specimens by 30 to 45 min.

In addition to the above tests, a series of tests was conducted on random (blind) samples of curing compounds that were furnished to D-9 for routine testing. A total of nine compounds were tested, all of them being Type 2. D-9 ran the moisture retention tests for 24 hr instead of the specified 72 hr because this is their normal practice in routine tests. The moisture loss results on the nine blind samples are shown in Table 4. It is clear that SDHPT moisture losses were considerably higher than losses obtained using the ASTM method, even though the SDHPT results were reported in 24 hr. Five out of the nine compounds failed the Texas test but none failed the ASTM test.

These results contradict previous results from the five multiple moisture retention tests. Equipment and procedures used in both cases were identical and therefore no viable explanation can be given to the exhibited trend.

Results from multiple moisture retention tests and from blind samples indicate a high variability in both ASTM and SDHPT tests. A relatively easy way to remove this variability is to base the effectiveness of a curing compound on how well it retains moisture when compared with a specimen without curing compound (blank) cast and cured at the same time (1). The moisture retention results from multiple tests based on the moisture loss of blank specimens are shown in Table 5. It is clear that the method reduces the variability considerably. Computed CV ranged between 1.7 and 4.0. In order to determine a minimum acceptable limit of moisture retention in cured specimens, the maximum allowable moisture loss (0.55 kg/in.²) was expressed as a percentage of the average loss of 27 untreated specimens made from two different brands of cement. This value was calculated to be 80 percent.

TABLE 4 COMPARISON OF MOISTURE LOSS RESULTS ON BLIND SAMPLES IN ACCORDANCE WITH ASTM C 156-80 AND TEX-219-F

Blind Sample Code Number	Tex-219-F Moisture Loss in 24 Hours (Percent)	Tex-219-F Moisture Loss in 24 Hours Converted to ASTM C 156-80 (kg/m ²)	ASTM C 156-80 Moisture Loss in 72 Hours (kg/m ²)	Tex-219-F $\frac{\text{Moisture Loss}}{2\%} \times 100$ (Percent)	ASTM C 156-80 $\frac{\text{Moisture Loss}}{0.55} \times 100$ (Percent)
A	4.65	0.748	0.450	233 ^a	82
B	2.67	0.433	0.223	134 ^a	41
C	1.31	0.213	0.208	66	38
D	4.35	0.701	0.552	218 ^a	100
E	2.94	0.470	0.285	147 ^a	52
F	2.64	0.411	0.290	132 ^a	53
G	1.87	0.303	0.223	94	41
H	1.67	0.271	0.373	84	68
I	1.02	0.164	0.189	51	34

^a Indicates curing compound did not pass the test.

TABLE 5 COMPARISON OF THE VARIABILITY BETWEEN ASTM C 156-80 TEST RESULTS AND MOISTURE RETENTION CALCULATED FROM THE MOISTURE LOSS ON BLANK SPECIMENS

Test Number	Moisture Loss Without Compound (kg/m ²) L1	Moisture Loss ASTM C 156-80 (kg/m ²) L2	Moisture Retention (L1 - L2) x 100/L1 (Percent)	Moisture Loss ASTM C 156-80			Moisture Retention		
				Average (kg/m ²)	Std. Dev. (kg/m ²)	CV (Percent)	Average (kg/m ²)	Std. Dev. (kg/m ²)	CV (Percent)
S-1D-1	2.828	0.285	89.9	0.193	0.053	27.5	93.2	3.0	3.2
S-1D-2	2.548	0.143	94.4						
S-1D-3	2.842	0.254	91.1						
S-1D-4	2.570	0.090	96.5						
S-2-1	2.997	0.119	96.0	0.140	0.023	16.4	94.2	1.6	1.7
S-2-2	2.452	0.151	93.8						
S-2-3	2.118	0.150	92.9						
P-1D-1	2.054	0.206	90.0	0.186	0.036	19.4	91.7	2.3	2.5
P-1D-2	2.269	0.130	94.3						
P-1D-3	2.387	0.223	90.7						
P-2-1	2.430	0.169	93.0	0.241	0.043	17.8	89.6	3.5	3.9
P-2-2	2.269	0.318	86.0						
P-2-3	2.344	0.236	89.9						
M-1D-1	2.538	0.168	93.4	0.218	0.040	18.3	90.6	3.6	4.0
M-1D-2	2.376	0.194	91.8						
M-1D-3	2.183	0.293	86.6						

NEW MOISTURE RETENTION TESTS

ASTM and Texas SDHPT standards currently require a time-consuming and labor-intensive test for determining the quality and effectiveness of curing compounds to measure moisture retention. The test requires more than 72 hr to perform and involves mixing of mortar; molding, curing, and sealing of specimens; application of curing compound; and a correction test for compound loss in weight.

A simpler and less time-consuming test was needed that would ensure the same quality and possibly reduce its cost.

A test was proposed to meet this need derived from a similar test used in the polymer industry. It consists of placing a known volume of water in a container and sealing it with a membrane covered with curing compound. The container is then placed in a known environment for a period of time. The loss in water represents the evaporation rate through the membrane. The time needed to obtain a moisture loss through the membrane equal to the moisture loss of concrete cured with the same compound in 72 hr represented the appropriate run time of the test. A total of 23 membranes were tested, of which only one indicated some working potential. This membrane is a Celgard® product with a specification number K-442. Curing compound sprayed on the membrane at a rate of 180 ft²/gal partially blocked its pores causing similar evaporation with cured concrete at approximately 6 to 10 hr. All the other membranes were either completely sealed with the compound or were not sealed at all.

Multiple tests were performed by treating the membrane with P-1D and S-2 compounds. The results from these tests are shown in Tables 6 and 7. Values were read directly from plots of moisture loss versus time. As an example, moisture loss with time of six specimens treated with P-1D compound is shown in

Figure 1. The time needed for each specimen to permit a loss of 0.18 kg/m² was read from the graph and tabulated as Test No. 4 in Table 6. Statistical *t*-tests at a 95 percent confidence level suggested a test run time of 6.1 to 16.1 hr for the membrane treated with S-2 compound, and 5.2 to 6.8 hr for the membrane treated with P-1D compound. These results indicate that the

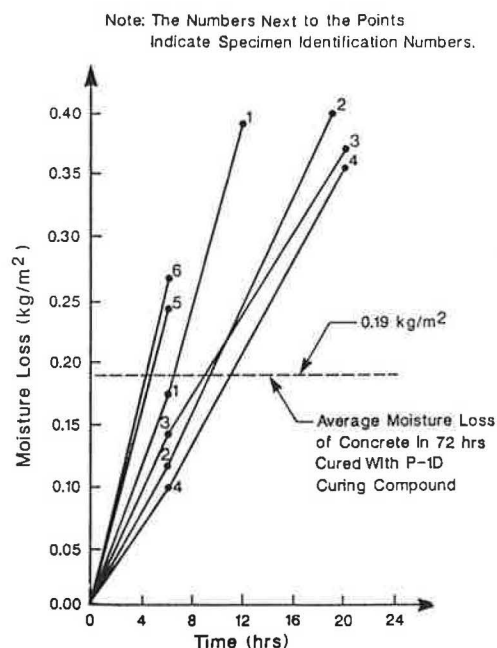


FIGURE 1 Moisture loss through Celanese membrane K-442 treated with P-1D curing compound, Test no. 4.

TABLE 6 MOISTURE LOSS THROUGH CELANESE MEMBRANE K-442 TREATED WITH P-1D CURING COMPOUND

Test Number	Specimen Number	Time for 0.19 kg/m ² Moisture Loss (hr)
1	1	14.7
	2	14.4
	3	9.8
	4	6.2
	5	14.8
2	1	10.8
	2	8.3
	3	17.8
	4	11.4
	5	14.9
3	1	9.8
	2	17.8
	3	12.7
	4	13.0
	5	16.8
4	1	6.7
	2	9.7
	3	9.3
	4	11.0
	5	4.8
	6	4.5
Average		11.1
Std. dev.		4.1
Sample std. dev. of the mean		1.8

TABLE 7 MOISTURE LOSS THROUGH CELANESE MEMBRANE K-442 TREATED WITH S-2 CURING COMPOUND

Test Number	Specimen Number	Time for 0.19 kg/m ² Moisture Loss (Hours)
1	1	4.8
	2	5.7
	3	6.7
	4	4.8
	5	6.6
	6	5.3
2	1	6.3
	2	5.8
	3	6.1
	4	6.3
	5	4.9
3	1	6.9
	2	5.7
	3	6.2
	4	6.5
	5	6.6
	6	6.2
Average		6.0
Std. Dev.		0.7
Sample Std. Dev. of the Mean		0.3

wide time range cannot permit a substitute test for S-2 compound. P-1D compound, however, can give reliable results if tested with the new moisture retention test as the appropriate test time range is very narrow.

The new test is easy to run and requires considerably less time than the 72 hr required by ASTM. A small testing program is required for each curing compound to determine the suitability of the new test and the appropriate run time.

AGITATION

The purpose of this phase of the study was to evaluate the effectiveness of mechanical agitation on the quality of mixing of curing compounds contained in 55-gal drums. Laboratory testing included agitation of six curing compounds, three of Type 1-D, and three of Type 2, stored for 3 and 6 months before mixing. Two types of mechanical agitation were employed, one using an electric, and one an air-driven motor. Table 8 shows the characteristics of the four types of shafts used with the two motors.

The test procedure was as follows. Before agitation took place at 3 months, samples were taken from the top, middle, and bottom thirds of each drum. The drums were then agitated for 5 min and sampled again. Mixing was continued up to 30 min, with sampling every 5 min. The sampling interval was soon changed to 10 min after the first results indicated no change in consistency within 5 min of mixing. Mixing and sampling were continued on the same drums at 6 months.

The solids content of samples was determined from the Tex-219-F test and compared to the base line content to determine the quality of mixing. A perfect mixing would be one that would give the same solids content in samples as in the base line sample. Early solids test results suggested misleading conclusions indicating good mixing where, as observed, re-suspension of separated compound layers had not been achieved. This occurred when separated compound layers had the same solids content. This problem led to the devising of a new test, the visual separation pattern test, in which samples were placed in glass test tubes and settled layers of the various ingredients were compared to base line samples. A perfect mixing would be one that would give equal portions of settled layers of samples taken from the top, middle, and bottom of the drum, and in addition, layer proportions equal to those of the base line sample. This test was found to be difficult to run when compounds were too viscous to be placed in glass tubes, in which case inadequate mixing was obvious, and when compounds did not separate into easily distinguishable layers during storage. This was the case with P-1D compound. Solids content values were uniform throughout the drums before and also after agitation, an indication of no separation of solids during storage. Therefore it was concluded that when the visual test did not show any layer separation the test could still be appropriate to perform.

Table 9 shows the quality of mixing of the six compounds mixed with the four agitators, as determined from the solids and visual tests. The mixing time column represents the optimum time used with the best agitator in each compound. Type 1-D curing compounds were easily remixed to a homogeneous consistency with Type D agitator after either 3 or 6 months of storage. Agitators B and C performed about the same, failing

TABLE 8 SHAFT AND VANE CHARACTERISTICS OF AGITATORS

	A	B	C	D
Driving Power	Electricity	Air	Air	Air
Operating Pressure (psi)	--	55	60	50
RPM	--	990	590	1390
Shaft Length (Inches)	27	30	24	30
Number of Vanes	1	2	2	2
Distance of Vanes from Shaft Bottom (Inches)	0	3, 19	4, 18	0, 15
Type of Vane	Non-Expandable	Expandable	Expandable	Non-Expandable
Vane Opening (Inches)	2	4 - 3/4	8	2
Holes on Blades	No	No	Yes	No

TABLE 9 COMPARISON OF THE MIXING QUALITY OF COMPOUNDS USING FOUR AGITATION DEVICES

Curing Compound	Storage Time (Months)	Mixing Time (Minutes)	Mixing Quality Agitation Devices			
			A	B	C	D
S-1D	3	30	Bad	Good	Good	Perfect
	6		Bad	Good	Good	Perfect
S-2	3	30	Bad	Good	Fair	Fair
	6		Bad	Good	Fair	Fair
P-1D	3	10	Perfect	Perfect	Perfect	Perfect
	6		Perfect	Perfect	Perfect	Perfect
P-2	3	30	Bad	Good	Fair	Fair
	6		Bad	Good	Fair	Fair
M-1D	3	10	Perfect	Perfect	Perfect	Perfect
	6		Perfect	Perfect	Perfect	Perfect
M-2	3	30	Fair	Fair	Good	Fair
	6		Bad	Fair	Bad	Bad

only to redisperse the top layer in S-1D compound. Agitator A performed badly in compound S-1D but mixed perfectly in P-1D and M-1D. None of the agitators could mix any of the Type 2 compounds perfectly, either at 3 or 6 months, the major problem being the resuspension of the settled white pigment. In general, agitator B was the best of the four devices in Type 2 compounds, while C and D were less efficient. Agitator A did not mix the compounds at all.

SHELF-LIFE STUDY

To investigate this issue, four drums of each of the six curing compounds were set aside for shelf-life testing. Over the course of a year, one of these four drums was opened, agitated, and sampled four times; at 3, 6, 9, and 12 months of age. A second drum was opened and sampled on the same schedule, but the procedure at the 3 month test data stage was deleted. A third

drum was tested at only 9 and 12 months of age. Finally, the fourth drum was left unagitated for the full year, and only opened and tested after that full period had elapsed. In this way, effects of undisturbed storage periods of various durations could be examined, as well the effects of agitation efforts between manufacture and the final agitation just before use.

In all cases, complete and thorough agitation was assured by removal of the drum head, followed by the use of agitator D, as described earlier, in combination with manual movement of the agitation while in operation through the material and against the bottom and sides of the drum. This was done to assure resuspension of settled solids, especially relevant in the case of Type 2 (white pigmented) curing compound.

The test procedure for each drum was as follows. Before agitation, the drum was photographed and samples taken from the top, middle, and bottom thirds of the drum. The agitation was then performed until the compound took on a uniform appearance and consistency. After agitation, a final sample was taken from the middle third of the drum.

The examination of these test specimens had essentially four aspects. First, qualitative observations about the appearance of the compound in the drum were made. Second, all samples taken were subjected to the solids content test, and those values were compared to the known baseline solids content of the material. Third, when possible, part of each sample was transferred to a visual test specimen tube, again for comparison with the properties of a baseline sample. Finally, SDHPT performed its full battery of curing compound quality control tests on all post-agitation curing-compound samples.

As an example, the set of shelf-life separation patterns for the drum of Type S-1D curing compound that was left unagitated for the full 12 month duration of the test is shown in Figure 2.

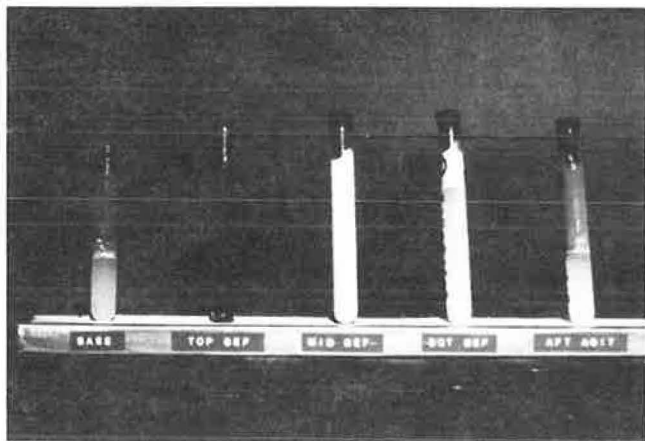


FIGURE 2 Shelf life separation pattern of Type S-1D curing compound after 12 months.

The results of these tests on the six curing compounds tested showed a high incidence of unpredictable and anomalous behavior. On some occasions, delays in testing or other factors may have caused these incidences, but quite often the behavior was inexplicable. Fortunately, these cases were exceptions, and most tests proved helpful in assessing the actual shelf life of a

given material. The conclusions reached on the basis of these tests were as follows.

S-1D Curing Compound

The solids content and visual separation pattern tests conducted by CTR indicated acceptable uniformity following agitation and close similarity to base line material after one year of storage. The quality control tests performed by SDHPT indicated some alterations in material properties after this period of storage, but these alterations were not drastic, and were not even consistently detrimental in nature. These observations led to the conclusion that the shelf life of Type S-1D curing compound could safely be extended to 12 months.

S-2 Curing Compound

CTR solids content and visual separation pattern tests indicated no serious deviations from the behavior of the base line material after the full 12-month term of the investigation. SDHPT tests, however, showed a severe thickening of the compound after 9 months of storage. This was evident, to a more severe degree, at 12 months. There was a delay of 4 to 5 months between sampling and SDHPT testing of the 12-month sample. This may have contributed to the thickening problem in those samples, but the delay associated with the 9-month samples was much shorter and, as mentioned before, the thickening problem was evident in these samples as well. The thickening of the material was severe enough to interfere with proper testing of the curing compound. Because of this, it was believed that a shelf life of more than 6 months could not be recommended for Type S-2 curing compound.

P-1D Curing Compound

The P-1D curing compound was the most unusual of the six tested. By all appearances, this compound exhibited no separation except in two of four sets of samples taken during the course of the study. Solids content tests never indicated a layering of the material, and the layering that was indicated by the separation pattern test and by simple observation was eliminated in the first few minutes of agitation. In any case, no material degradation was indicated by the CTR test findings. If anything, SDHPT quality control tests indicated a slight improvement of material properties with time.

In addition to this retention of acceptable properties, the P-1D compound was qualitatively judged by the experimenters to be the best of the six tested. It provided moisture retention abilities on a par with all five of the other materials, was by far the least susceptible to settling and caking, and was the easiest to reagituate.

The Type P-1D compound did, however, have one disadvantage over the other compounds. The compound remained essentially unchanged provided it was not agitated until just before use. However, if the compound was agitated, and then allowed to sit for some time before use, a waxy solid film formed over the top of the compound. Although agitation would break up the film, the particles would not re-dissolve and so constitute a threat to the spraying apparatus. The crust and suspended particles following agitation are shown in Figure 3.



FIGURE 3 Crust development over Type P-1D.

The shelf life recommendation for this compound is thus 12 months, provided there is no agitation effort until just before use.

P-2 Curing Compound

Throughout the CTR tests on P-2 compound, both solids content and visual separation patterns were similar to those of the base line samples up to 1 year after manufacture. The same was true of the results of the SDHPT curing compound quality control tests performed on the Type P-2 compound samples.

Unlike the P-1D compound, the P-2 compound was the most susceptible to settling and caking and was the most difficult to agitate to uniformity. As stated earlier, after the necessary agitation, the compound did retain its original properties throughout the term of the tests. It was suggested, therefore, that the recommended shelf life for Type P-2 curing compound be extended to 12 months.

M-1D Curing Compound

All CTR tests on Type M-1D curing compound showed that this material could be agitated to uniformity with relative ease

after up to 1 year of storage. Solids content data, and required agitation times at different stages of the study, suggested that intermittent agitation could reduce the time necessary for agitation just before use. However, such a practice would not reliably reduce ultimate total agitation time, and would require set up of the agitation device on several occasions instead of just one. As a result, intermittent agitation was not recommended. It was recommended, however, that the shelf life of this material be extended to 1 year.

M-2 Curing Compound

Type M-2 curing compound was the least satisfactory according to the findings of CTR tests. At both 9 and 12 months, agitation did not regenerate separation patterns similar to the base line sample. More important, large hard particles were discovered in the compound when it was first tested at 3 months of age and at all times thereafter. These particles were of sufficient size to clog commonly used spray application equipment.

Despite these particles, SDHPT was able to perform its battery of quality control tests on the M-2 compound samples. The results of these tests disclosed no material degradation over the term of the study. Nevertheless, the effect of the particles on field procedures was seen as sufficient reason not to extend the recommended shelf life of this material to any duration greater than 6 months.

APPLICATION PATTERN STUDY

SDHPT currently specifies that curing compound be applied in a single sprayed-on coat at a rate of 180 ft²/gal. As this is the easiest procedure, the compound is typically applied by a spray bar traveling parallel to the direction of travel (perpendicular to the tined grooves in the new pavement textures).

It was suspected that changing the number of coats (while retaining the same total rate of coverage) and the direction of travel of the spray bars could alter the effectiveness of the membrane. To investigate this potential benefit, the following test procedure was performed for seven different application patterns. Four standard ASTM C 156-80 moisture-retention test specimens were prepared and tested in accordance with the ASTM procedure, except that three of the specimens were textured with the new combined texture produced by an Astro-grass drag followed by tines at 3/4 in. on center. These specimens were then treated with curing compound in the pattern being investigated. The fourth specimen was textured with the old tined-only texture with grooves at 1/2 in. on center. This specimen was used as a standard to compare results of the different application patterns. The seven patterns examined were

1. The present standard of a single pass perpendicular to the texture grooves.
2. One pass parallel to the texture grooves.
3. Two passes in the same direction perpendicular to the texture grooves.
4. Two passes in the same direction parallel to the texture grooves.

TABLE 10 MOISTURE LOSS (kg/m^2) FOR DIFFERENT APPLICATION PATTERNS

Specimen	Pattern 1	Pattern 2	Pattern 3	Pattern 4	Pattern 5	Pattern 6	Pattern 7
Standard	0.603	1.108	1.269	1.356	1.227	1.312	1.936
1	1.04	0.721	1.388	1.732	1.216	1.076	1.753
2	0.775	1.012	1.818	0.753	1.055	1.420	1.399
3	0.979	0.818	1.463	1.076	1.248	1.237	1.194
Standard Deviation	0.140	0.148	0.230	0.499	0.103	0.172	0.283
Mean	0.933	0.850	1.556	1.187	1.173	1.244	1.449
CV (Percent)	15.0	17.4	14.8	42.0	8.8	13.8	19.5

5. Two passes in opposite directions perpendicular to the texture grooves.

6. Two passes in opposite directions parallel to the texture grooves.

7. One pass parallel to the texture grooves, combined with one pass perpendicular to the texture grooves.

The results obtained from these tests are presented in Table 10. These results were analyzed both in the raw form and after normalization to the average loss from the standard specimen. The analysis consisted of *F*- and *t*-tests for significant differences in means between the moisture loss accompanying each of the six alternate application patterns (Numbers 2 through 7) and the standard pattern, Number 1.

The *F*-tests showed no significant differences between moisture losses using any of the six alternative application patterns and the standard pattern. This was true of both the raw and normalized data. The *t*-tests suggested significant differences between Patterns 3, 5, 6, and 7 and the standard pattern. The level of confidence for these differences was 90 percent or greater, but the direction of the differences indicated inferior performance in the alternative patterns. As a result, it was recommended that the current pattern (Number 1) be retained as the standard.

APPLICATION RATE STUDY

A more direct approach to increasing the effectiveness of the curing compound membrane is to increase its thickness by increasing the rate of compound application. The first step in this part of the study was to construct a device that could correlate rate of coverage to surface reflectance. The device constructed consisted of two cadmium sulfide photo cells in a Wheatstone bridge circuit (see Figures 4 and 5). The device would be calibrated by placing both photo cells over one surface and adjusting the circuit resistance until no potential drop was read. One of the cells would then be moved over the surface to be examined and the new potential drop read.

Curves were developed using surfaces of different known reflectance for standardization to 90 and 18 percent reflectant

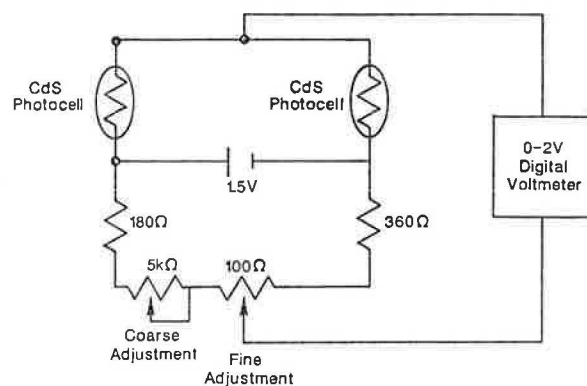


FIGURE 4 Circuit diagram of reflectance meter used in rate of application studies.

surfaces. These curves are presented in Figure 6. Unfortunately, as indicated by the dashed lines in Figure 5, the variation in the readings was too great to provide a reliable correlation between voltage drop in the circuit and surface reflectance. In addition, surface treated with Type P-2 curing compound at rates of 150 ft^2/gal , 180 ft^2/gal , and 200 ft^2/gal produced virtually useless patterns of reflectance versus application rate behavior (see Figures 7 and 8).

This indirect approach having failed, the attempt was made to directly measure the change in moisture loss accompanying an increase in application rate from 180 ft^2/gal to 150 ft^2/gal . The results are shown in Table 11. Interestingly, the higher application rate was actually accompanied by a reduced moisture-retention ability. Though the *F*- and *t*-tests confirmed the significance of this finding from a purely statistical point of view, problems during testing and a small sample size call this conclusion into question. It is important to note, however, that the specimens treated at the higher application rate began to show signs of compound pooling in depressed areas and inadequate membrane development within the surface grooves. Both these observations would explain the increased moisture loss rates that accompanied the higher application rate. Overall, there seemed to be no significant benefit to be derived from increasing the curing compound application rate above 180 ft^2/gal .

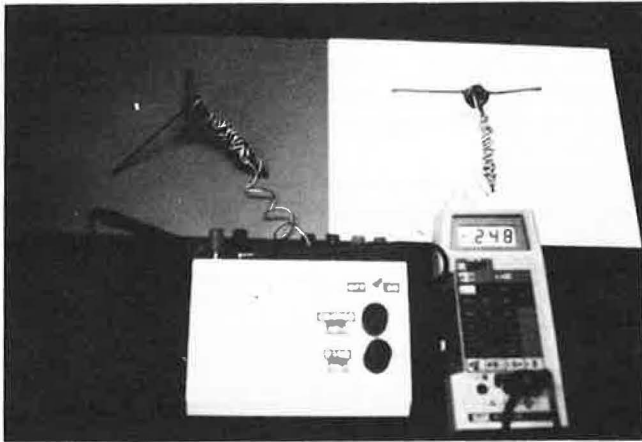


FIGURE 5 Prototype reflectance meter reading -248 mV from 90 percent reflectant surface when standardized to 18 percent reflectance.

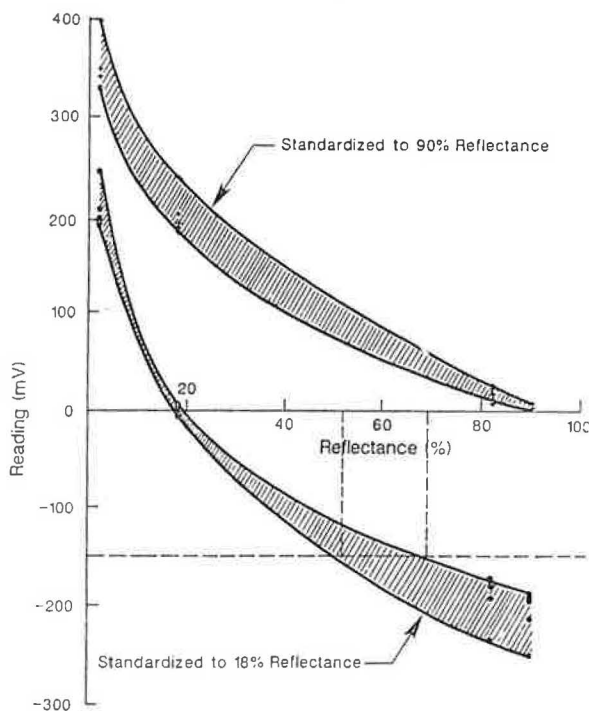


FIGURE 6 Standard reflectance curves for prototype reflectance meter.

gal. For this reason, it was recommended that the current rate of 180 ft²/gal be retained.

IN-LINE VERSUS DRUM SAMPLING

Samples of curing compound are typically taken directly from the production line to be subjected to SDHPT tests for product acceptability. It has been suggested that these so-called in-line samples may not accurately reflect the actual properties of a compound that has been packaged in a drum and allowed to settle before sampling, as field material has been.

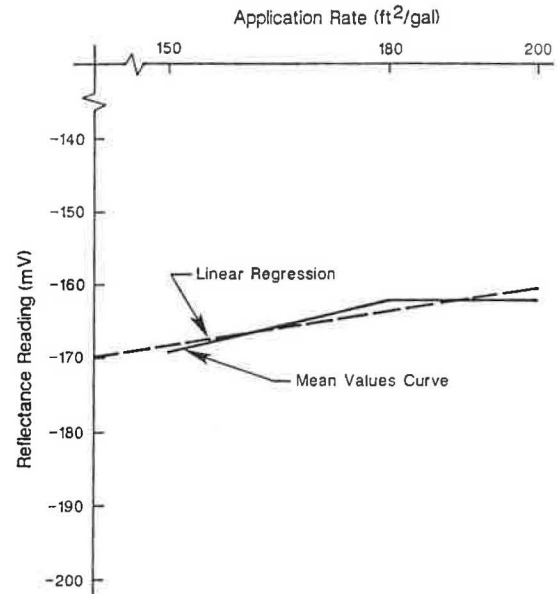


FIGURE 7 Correlation of reflectance readings and P-2 compound application rates with meter standardized to 18 percent reflectance.

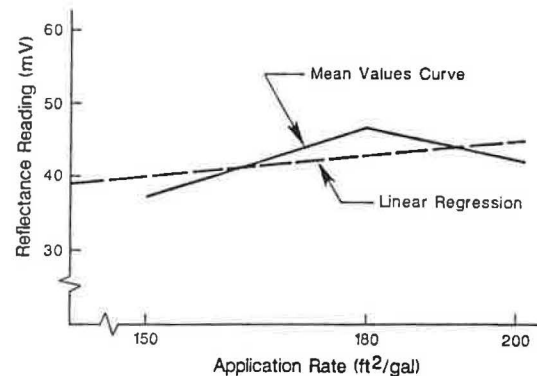


FIGURE 8 Correlation of reflectance readings and P-2 compound application rates with meter standardized to 90 percent reflectance.

TABLE 11 MOISTURE LOSS RATES (kg/m²) USING APPLICATION RATES OF ONE GALLON PER 180 FEET² AND ONE GALLON PER 150 FEET²

Specimen No.	Application Rate	
	1 gal/180 ft ²	1 gal/150 ft ²
1 ^a	1.6136	1.719
2	0.904	1.171
3	1.033	1.139
Average	1.183	1.343
CV (Percent)	31.95	24.27
Average neglecting specimen No. 1	0.969	1.155
CV Neglecting specimen No. 1	9.41	1.96

^aNozzle on spray bar passing over these specimens was operating improperly.

A reasonable check on this concern should be to compare several in-line samples of curing compound to drum samples of the same compound taken after reagitation, some time after manufacture.

In-line samples of between two and four different batches of each of the six curing compounds used during this study were taken at the manufacturing plant and subjected to full SDHPT acceptance tests. Seven days after the in-line sampling, a drum of each batch previously sampled was agitated and sampled. This drum sample was then subjected to the same battery of SDHPT tests as was the in-line sample.

Because only one pair of samples was tested from each batch, rigid statistical analysis could not be performed. However, general trends in behavioral differences were sought and examined. Based on the results presented in Tables 12 through 14, the following conclusions were derived:

1. Solids content and moisture-retention data from tests of Type S-1D compound suggest consistently lower solids content and higher moisture loss in drum samples compared with in-line samples. This suggests that the drum samples are preferable.

TABLE 12 IN-LINE SAMPLE VERSUS SAMPLE SDHPT TEST RESULTS FOR S-1D AND S-2 CURING COMPOUNDS

Compound Type	Batch Number	Sample ^a	Vertical Spray ^b	Drying Time ^b	Reflectivity (Percent)	Flash Temperature (°F)	Solids Content (Percent)	Specific Gravity	24-Hour Moisture Loss (Percent)
S-1D	1	I	S	S	--	102	24.15	0.955	0.30
	1	D	S	S	--	114	18.00	0.959	1.17
	2	I	S	S	--	117	25.40	0.957	0.42
	2	D	S	S	--	115	16.21	0.965	1.91
	3	I	S	S	--	114	25.77	0.945	0.38
	3	D	S	S	--	115	16.91	0.955	1.89
S-2	1	I	S	S	63.6	109	40.79	1.091	1.31
	1	D	S	S	68.5	98	41.44	1.106	1.03
	2	I	S	S	63.9	115	40.11	1.093	0.68
	2	D	S	S	68.4	96	39.68	1.087	1.46
	3	I	S	S	63.2	112	40.20	1.108	1.06
	3	D	S	S	65.1	116	38.83	1.076	1.43

^a I implies in-line same, D implies drum sample.

^b Reported as S = satisfactory, or U = unsatisfactory.

TABLE 13 IN-LINE SAMPLE VERSUS SAMPLE SDHPT TEST RESULTS FOR P-1D AND P-2 CURING COMPOUNDS

Compound Type	Batch Number	Sample ^a	Vertical Spray ^b	Drying Time ^b	Reflectivity (Percent)	Flash Temperature (°F)	Solids Content (Percent)	Specific Gravity	24-Hour Moisture Loss (Percent)
P-2	1	I	S	S	61.4	100	47.61	1.015	1.71
	1	D	S	S	61.7	112	45.97	1.018	1.41
	2	I	S	S	60.2	113	43.23	0.970	0.99
	2	D	S	S	61.4	115	47.37	0.926	1.28
	3	I	S	S	63.9	113	47.57	1.004	1.40
	3	D	S	S	61.1	116	48.75	1.020	0.90
	4	I	S	S	60.0	110	46.67	1.008	1.71
	4	D	S	S	61.4	113	47.38	0.999	1.54
P-1D	1	I	S	S	--	97	47.30	0.895	0.70
	1	D	S	S	--	90	51.63	0.901	0.59
	2	I	S	S	--	96	48.79	0.903	0.63
	2	D	S	S	--	80	50.45	0.899	0.65

^a I implies in-line same, D implies drum sample.

^b Reported as S = satisfactory, or U = unsatisfactory.

TABLE 14 IN-LINE SAMPLE VERSUS SAMPLE SDHPT TEST RESULTS FOR M-1D AND M-2 CURING COMPOUNDS

Compound Type	Batch Number	Sample ^a	Vertical Spray ^b	Drying Time ^b	Reflectivity (Percent)	Flash Temperature (°F)	Solids Content (Percent)	Specific Gravity	24-Hour Moisture Loss (Percent)
M-1D	1	I	S	S	--	115	26.41	0.929	0.19
	1	D	S	S	--	109	26.06	0.933	0.21
	2	I	S	S	--	122	26.33	0.932	0.35
	2	D	S	S	--	110	30.06	0.929	0.80
	3	I	S	S	--	110	25.09	0.934	0.27
	3	D	S	S	--	108	24.95	0.859	0.21
M-2	1	I	S	S	74.8	113	47.66	1.075	0.25
	1	D	S	S	64.5	111	47.63	1.073	0.62
	2	I	S	S	76.7	113	44.35	1.079	0.19
	2	D	S	S	67.1	113	43.20	1.073	0.35

^a I implies in-line same, D implies drum sample.

^b Reported as S = satisfactory, or U = unsatisfactory.

2. Flash temperature data from Type P-1D compound tests suggested lower drum-sample flash temperatures than those found in in-line samples. Again, this suggests that the use of drum samples is preferable.

3. Flash temperature data from Type M-1D compound tests also suggested lower drum-sample values. This reaffirmed Conclusion 2.

4. Drum samples of Type M-2 curing compound were found to be inferior to corresponding in-line samples in terms of reflectivity and moisture retention capability, again reaffirming Conclusion 2.

5. No consistent and significant trends in drum versus in-line test results were noted in Types S-2 or P-2 curing compounds.

CONCLUSIONS

Moisture Retention Tests

1. Expressing moisture loss in terms of the water present in the specimen at the time of application (Tex-219-F) instead of the surface area of the specimen (ASTM C 156-80) does not reduce the variability of the test. The CV of Texas moisture losses was identical to the CV of the losses modified according to the ASTM procedure in all five compounds tested.

2. The SDHPT method applies a faster, simpler, and equally accurate test compared with the ASTM method for the determination of loss in volatile matter from compounds during curing of specimens.

3. The Type III (high early strength) cement used by the SDHPT test for the manufacture of test specimens reduces the initial curing time by 30 to 45 min. However, no conclusions can be made about the effect of using only one brand of cement on the variability of the tests because no data were collected.

4. Both tests exhibited the same high variability.

5. Each test is equally likely to result in higher or stricter values than the other.

6. Moisture retention tests on nine curing compounds furnished to D-9 for routine testing indicated that SDHPT results were higher in all the compounds despite the fact that SDHPT losses were reported in 24 hr and ASTM in 72 hr. Of the nine compounds tested, five failed the SDHPT test; none failed the ASTM test. These results contradict Conclusion 5, derived from the five multiple moisture retention tests, but no viable explanation can be offered.

7. The variability of moisture-retention tests performed in accordance with ASTM decreased when moisture loss of specimens was expressed as a function of the moisture loss of a specimen without curing cast and cured at the same time. Based on this, it appears that it would be beneficial to express the SDHPT moisture loss in the same way.

Agitation Tests

1. Both solids content and visual-inspection tests were found to be good indicators of the extent of mixing of curing compounds. Results of solids tests were misleading only when separated compound portions had the same contents of solids.

2. To determine the quality of agitation, samples should be obtained from the top, middle, and bottom thirds of the drums.

3. Type 1-D curing compounds stored up to 6 months were easily remixed to a homogeneous consistency using Type D agitator. Mixing times were 30 min for S-1D and 10 min for P-1D and M-1D.

4. Type 2 curing compounds could not be remixed to a homogeneous consistency after either 3 or 6 months of storage. None of the agitators could completely reincorporate into the mix the settled white pigment at the bottom and the resin formed at the top of the drums.

New Moisture Retention Tests

Celanese microporous film K-442 was found to exhibit potential in the development of a new moisture-retention test. The test is easy to run and requires considerably less time than the 72 hr required by ASTM.

Shelf-Life Tests

1. All three Type 1-D curing compounds could be safely stored for 12 months before use. Therefore, a 12-month shelf life was recommended for Type 1-D curing compounds in general.

2. Though the Type P-2 compound was satisfactory after 1 year of storage, the other two brands of Type 2 compound were not. For this reason, it was recommended that the present 6 month shelf life be retained for Type 2 white pigmented compounds.

Application Pattern

1. *F*-tests for significance in differences of means showed no differences between moisture losses using any of the six alternative application patterns and the standard pattern. This was true of both the data in its raw state and normalized to the mean loss from the control specimens.

2. Significant differences in results were indicated by *t*-tests using application Pattern 3 (two unidirectional passes perpendicular to grooves), Pattern 5 (two bidirectional passes perpendicular to grooves), Pattern 6 (two bidirectional passes parallel to grooves), and Pattern 7 (one pass parallel to grooves and one pass perpendicular to grooves). These differences were detrimental with moisture loss rates, however, and so were not the desired results. Again, these findings applied to both the raw and normalized data.

Application Rate

1. Using the prototype reflectance meter developed for this study, readings from the same surface were found to vary considerably under different lighting conditions.

2. Readings from surfaces treated with Type 2 white-pigmented curing compound at 150 ft²/gal to 200 ft²/gal varied by significantly less than the typical same-surface variation noted in 1.

3. Regression on the reflectance readings from surfaces treated at three different application rates showed a very low correlation between application rate and reflectance reading.

4. Moisture loss study comparing application rates of 180 ft²/gal and 150 ft²/gal indicated significant differences in near-moisture losses using these two rates. However, small sample size and the illogical nature of the results (higher moisture loss with higher application rates) call these results seriously into question.

5. Qualitative visual observations made during the test, described in application rate 4, suggested that compound pooling in surface depressions had begun with use of the 150 ft²/gal application rate.

Drum Versus In-Line Sample Comparison

Solids content and moisture retention data from most of the compounds tested suggest that drum samples are stricter than in-line samples, and are therefore preferable.

RECOMMENDATIONS

Based on the results and conclusion of this study the following recommendations are made:

1. Moisture loss of cured specimens should be expressed as a function of the moisture loss of a specimen without curing compound cast at the same time. The moisture retained should be equal to or greater than 80 percent.

2. Speed and blade diameter of agitators used for Type 1-D curing compounds should be 1,400 rpm and 2 in., respectively. Mixing should be continued until a homogeneous consistency at the top, middle, and bottom of the drum is accomplished, as determined by the solids content or the visual inspection tests.

3. Before a decision is made concerning the implementation of the new moisture-retention test as a substitute for the ASTM test C 156-80, a testing program should be conducted for each curing compound to determine the suitability of Celgard® membrane K-442 and the appropriate run time.

4. As it is not practical to assign a separate shelf life to each type of material made by every manufacturer, it is suggested that shelf life be maintained at 6 months for Type 2 compounds, but extended to 12 months for Type 1-D compounds.

5. A new standard application pattern is not recommended because of lack of evidence supporting the change.

6. It is recommended that visual inspection be continued as the standard procedure because the human eye appears to be more sensitive and able to survey larger areas than the reflectance meter.

7. No significant increase in moisture retention was noted with the increase in application rate to 150 ft²/gal. Therefore, it is recommended that the current standard rate of 180 ft²/gal be continued.

8. All observed trends indicate that drum samples are preferable to in-line samples for testing purposes. Though this could not be confirmed by strict statistical analysis, the trends observed, combined with the realization that a drum sample has a history more similar to field material than does an in-line sample, suggest recommendation of drum samples for use in SDHPT curing compound acceptability tests.

ACKNOWLEDGMENT

The authors are pleased to acknowledge the combined support of the Center for Transportation Research at The University of Texas at Austin and the Texas State Department of Highways and Public Transportation, in cooperation with the U.S. Department of Transportation, Federal Highway Administration. Special acknowledgment is made to the staff of the Center for Transportation Research of the University of Texas at Austin for their assistance in preparing the drafts and figures in this paper.

REFERENCES

1. W. B. Ledbetter. *Moisture Retention Performance of White-Pigmented Curing Compound*. Texas Transportation Institute, Texas A&M University, College Station, 1982.
2. *Transportation Research Circular 208: Curing of Concrete Pavements*. TRB, National Research Council, Washington, D.C., 1979.
3. S. M. S. Shariat and P. D. Pant. Curing and Moisture Loss of Grooved Concrete Surfaces. In *Transportation Research Record 986*, TRB, National Research Council, Washington, D.C., 1984, pp. 4-8.
4. American Society for Testing and Materials. *American Society for Testing and Materials Standards*, Philadelphia, Pa., 1984.
5. Texas State Department of Highways and Public Transportation.

Standard Specifications for Construction of Highways, Streets, and Bridges, Sept. 1, 1982.

The contents of this paper reflect the views of the authors, who are responsible for the facts and validity of the developments presented herein. The contents do not necessarily reflect the official views or policies of the Federal Highway Administration. This paper does not constitute a standard, specification or regulation.

Material brand names that may appear in the text are solely for illustrative or clarity purposes and should not be construed to mean endorsement of any kind.

Publication of this paper sponsored by Committee on Chemical Additions and Admixtures for Concrete.

A New Process for the Laying of Monolithic Composite Continuously Reinforced Concrete Pavements

Y. CHARONNAT, J. P. AUGOYARD, AND L. PONSART

Technical as well as financial developments have led to a new approach to rigid pavement techniques in France for heavy-traffic roads. Continuously reinforced concrete with the use of local aggregates is currently preferred. A monolithic composite structure made up of two layers of concrete of different composition is increasingly used. New techniques, involving continuous reinforcing and double-layer rigid pavement, have called for the development of specific equipment in order to produce good functional characteristics while allowing for construction performance and quality equivalent to that found at more traditional construction sites. Analysis of the results obtained on the highway construction site discussed in this paper reveals that the aims set by the designers have been fully met.

The use of cement concrete pavements on the French national highway network has been intermittent for the past 20 years. After a period of uncertainty, new market conditions have boosted the use of hydraulic concrete in conjunction with the use of continuous reinforcing. New equipment had to be designed to adapt to the functions required. The construction of the highway section described in this paper allows an assessment to be made of the progress.

Y. Charonnat, Laboratoire Central des Ponts et Chaussées, 58 Boulevard Lefebvre, 75732 Paris, Cedex 15, France. J. P. Augoyard and L. Ponsart, GTM International, B.P. 326-92003 Nanterre, Cedex, France.

REQUIREMENTS FOR ROAD CONSTRUCTION IN FRANCE

In 1985, some 850,000 m² (1 million yd²) of concrete pavement were built on heavy-traffic roads, including new lanes and overlays. This work, considered a substantial amount for France, accounted for the efforts made by companies to develop high-performance equipment.

Opening of the Market

Although there were other factors involved, this renewal of construction techniques was the result of three factors:

1. Highway administrators' taking into account maintenance costs and disruption to users by the performance of such maintenance;
2. Officials of the Direction des Routes (Public Roads Authority) wanting to apply appropriate techniques to specific cases; and
3. Prime contractors and users becoming more familiar with such techniques as a result of increasing small-scale road networks made of concrete.