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This report discusses 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/or ASTM C 156-80 is also outlined, in addition to suggestions for the direction of continuing research in this direction. Recommendations are presented with regard to the six-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 dealing with the effects of altering application rates and pattern on moisture retention. Finally, the use of optical reflectance as a measure of application rate is examined.

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by

Chryssis G. Papaleontiou
Matthew D. Loeffler
Alvin H. Meyer
David W. Fowler

Research Report Number 427-1F

Effectiveness of Texas Membrane Curing Compound Quality
and Application Requirements

Research Project 3-9-84-427

conducted for

Texas
State Department of Highways and Public Transportation

in cooperation with the
U. S. Department of Transportation
Federal Highway Administration

by the

CENTER FOR TRANSPORTATION RESEARCH
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June 1986

There was no invention or discovery conceived or first actually reduced to practice in the course of or under this contract, including any art, method, process, machine, manufacture, design or composition of matter, or any new and useful improvement thereof, or any variety of plant which is or may be patentable under the patent laws of the United States of America or any foreign country.

PREFACE

This document is a report on the research conducted under Research Project 3-9-84-427, "Effectiveness of Texas Membrane Curing Compound Quality and Application Requirements." This project was conducted by the Center for Transportation Research, The University of Texas at Austin, as part of the Cooperative Highway Research Program sponsored by the State Department of Highways and Public Transportation (SDHPT) and the Federal Highway Administration (FHWA).

The authors would like to express their appreciation to Mr. Frank Hulsey for his invaluable assistance on several aspects of this study. In addition, a great deal or gratitude is due Mr. E. E. Barnes of Celanese Separation Products for his assistance in making certain materials available on a continuing basis.

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Finally, the authors would like to extend a special thanks to Paul Krugler, Dale Foster, and the rest of the Bituminous Section of D-9 of the Texas State Department of Highways and Public Transportation both for their experimental contributions to this study and their advice on the study as a whole.

LIST OF REPORTS

Report No. 427-1F, "Effectiveness of Texas Membrane Curing Compound Quality and Application Requirements," by Chryssis G. Papaleontiou, Matthew D. Loeffler, Alvin H. Meyer, and David W. Fowler, presents results of tests comparing Texas Method Tex-219-F, and ASTM method C 156-80 for evaluation of curing compound moisture retention; several agitation devices and preliminary work on development of a new moisture retention test; investigation of current shelf life specifications; and studies of curing compound application rates and application patterns.

ABSTRACT

This report discusses 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/or ASTM C 156-80 is also outlined, in addition to suggestions for the direction of continuing research in this direction. Recommendations are presented with regard to the six 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 dealing with the effects of altering application rates and pattern on moisture retention. Finally, the use of optical reflectance as a measure of application rate is examined.

SUMMARY

Proper curing of Portland Cement concrete pavement is essential in order to avoid both shrinkage cracking and long-term durability problems. Curing is often accomplished with the aid of liquid membrane curing compounds. There has been some discussion of the possibility that current Texas test methods for compound moisture retention are more strict than ASTM test methods. Tests comparing these two test methods as applied to six different compounds of the types indicated that there was no significant difference between the results of the two methods.

Successful use of curing compound requires that it be properly agitated to uniform consistency prior to use. Four devices were tested as to their accomplishment of this end, and the most effective of these was recommended for use, under certain conditions.

Both the Texas and ASTM test methods for evaluation of curing compound moisture retention are labor and time intensive procedures. Research Project 427 included preliminary investigation of the possibility of treating semi-porous membrane with curing compound and evaluating moisture passage through this treated membrane when it is placed over a volume of water. This method shows great promise in terms of labor, material cost, and time required for testing when compared to the presently employed test procedures.

All types of liquid membrane-forming curing compounds are assigned a shelf life of six months by SDHPT. This term had no experimental basis prior to the research conducted under the auspices of this study. The research conducted during the course of Project 427 indicated that the shelf life of Type 1-D curing compound can be safely extended to one year. Findings also indicated, however, that the shelf life of Type 2 compounds in general could not be reliably extended past the six month value.

Recent changes in standard textures used on pavement in Texas have raised questions about both the proper rate and pattern to be used in conjunction with those new textures. Research included attempts to measure application rate as a function of surface reflectance. The results of this research indicated that the variability of lighting conditions and the opacity of most curing compounds rendered this method ineffective. Along

these lines, different application rates were tested in terms of their moisture retention capabilities. It was found that the current specified rate of 1 gallon per 180 square feet was not significantly less effective in retaining concrete moisture than a rate of 1 gallon per 150 square feet. Further, this latter rate displayed tendency toward pooling, which is a waste of the material.

Finally, Project 427 included an investigation of compound application patterns. Once again, none of the test patterns were found to be superior to the simple single longitudinal pass that is most commonly used at present.

IMPLEMENTATION STATEMENT

Comparison of Tex-219-F and ASTM C 156-80 curing compound test methods indicate that certain aspects of the ASTM method can be applied to the Texas method to simplify its performance without detracting from its reliability.

Tests of agitation devices employed a visual test-tube test described in this report. This test has shown itself to be much more valuable than the use of sample solids content tests, and should prove quite valuable to the Texas State Department of Highways and Public Transportation (SDHPT).

Project testing has also indicated that the shelf life of Type 1-D curing compound can safely be extended to one year.

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CHAPTER 1. INTRODUCTION

BACKGROUND

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 leads to plastic shrinkage cracking which becomes critical when the rate of evaporation exceeds 1.0 $kg/m^2/h$. Additionally the lack of adequate moisture during curing results in lower strengths for PCC.

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

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

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

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

Liquid membrane forming curing compound is presently assumed to possess an effective shelf life of six months. This limit was set somewhat arbitrarily, however, and some manufacturers claim that their material can be restored through agitation up to a year or more after manufacture.

Present specifications require curing compound to be applied at a rate of 1 gallon per 180 square feet of pavement. This rate was based on a pavement texture produced by a burlap drag. The current standard surface texture is produced by an astrograss drag followed by 1/8-inch wide transverse grooves spaced at 1 inch on center. The added exposed area caused by the roughness of this texture, as well as the vertical surfaces and recessed horizontal surfaces produced by the grooving operation may call for changes in the recommended application rate, current application patterns, or both.

Shelf life was evaluated in terms of capacity for reagitation to uniformity and all standard Texas test criteria after three, six, nine, and twelve months of storage. Moisture loss accompanying alternative application patterns were compared to the pattern most commonly employed at present. The application rate was also increased to 1 gallon per 150 sq ft, and the effects on moisture retention and reflectance evaluated.

Test specimens used to confirm product acceptablility are currently taken from the production line at the plant. It has been suggested that it may be more appropriate to draw these samples from a drum of the compound after it has been allowed to sit for some time and then re-agitated. The rationale for this is that such a procedure would provide a sample more closely approximating the material (also delivered in drums) that is actually used in the field.

SCOPE

Realizing the need for study in the area of curing compounds, the Texas State Department of Highways and Public Transportation (SDHPT) initiated Research Study 427, "Effectiveness of Texas Membrane Curing Compound Quality and Application Requirements," in September, 1984. Under the direction of the Bituminous Section of the Materials and Test Division (D-9), and the

Center for Transportation Research, six different curing compounds were selected for laboratory testing. The performance of the Texas moisture retention test was compared to the ASTM test, as well as the mixing quality of curing compounds using four different agitation devices. In addition, the feasibility of developing a new moisture retention test that would insure the same quality of curing compounds and reduce the time and expense of the test was examined. Shelf life was evaluated in terms of capacity for reagitation to uniformity after three, six, nine, and twelve months of storage. SDHPT also conducted full acceptability tests on nine and twelve month samples. Accompanying alternative application patterns were compared to losses using the pattern most commonly employed. The application rate was also increased to 1 gallon per 150 square feet, and the effects on moisture retention and reflectance evaluated. In-line versus storage drum sample properties were compared for significant differences using between two and four batches of each of the six compounds tested during this study. This report summarizes the results of this study.

Chapter 2 describes the moisture retention test procedures. Chapter 3 outlines the agitation tests and the devices used. Chapter 4 describes the new moisture retention test. Chapter 5 describes the shelf life investigation. Chapter 6 presents the procedures for examining alternate application patterns. Chapter 7 describes the procedures involved in the rate of application studies. Chapter 8 describes in-line versus drum sample comparison procedures, Chapter 9 is a presentation and discussion of the experimental tests conducted, and Chapter 10 presents conclusions and recommendations.

CHAPTER 2. MOISTURE RETENTION TESTS

INTRODUCTION

Several concerns have been raised in recent years by some manufacturers of liquid membrane-forming curing compounds about the suitability of Test Method Tex-219-F, "Testing of Concrete Curing Materials." The test method was developed by the Texas State Department of Highways and Public Transportation (SDHPT) and is a modification of ASTM 309, "Liquid Membrane-Forming Compounds for Curing Concrete," and ASTM C 156-80, "Water Retention by Concrete Curing Materials." The test is intended for use in determining the quality and effectiveness of liquid membrane-forming compounds in preventing the evaporation of water from concrete during the early hardening period. Curing compound manufacturers in recent years have questioned the adequacy of the method for evaluating the moisture retention of curing compounds, stating that it is not consistent and not as reliable as the ASTM method, and it is doubtful if it could be translated to higher quality curing in the field.

Late in 1970, Transportation Research Circular 280 had indicated "the trend towards deeply textured pavements requires a larger quantity of curing membrane than pavements textured with a broom or burlap drag." When pavement is grooved, the surface area is not only increased, but the vertical sides of the grooves may not receive the same amount of curing compound as the horizontal surfaces. At that time, Texas specified, and still does specify, a curing compound rate of 180 sq. ft. per gallon. This rate was heavier than the 200 sq. ft. per gallon rate being recommended at the time by others. When Texas specified the transverse texture for pavements, no change was made in the specification for curing compound rate.

A series of moisture retention tests was conducted to investigate the performance of Texas Method Tex-219-F in relation to ASTM C 156-80 method. Six different curing compounds were used for testing. All of them had previously been tested by the Bituminous Section of D-9 of the SDHPT, to determine their conformity with the "Preliminary Tests" of Tex-219-F, which include Vertical Spray Test, Drying Time, and Reflectivity Test. The Center for Transportation Research (CTR) performed moisture retention tests on sixty specimens in accordance with ASTM C 156-80. A similar set of tests on the same compounds and on identical specimens to those tested by CTR were performed by D-9 using the Texas Method Tex-219-F.

In addition to these tests, a third series of moisture retention tests consisting of nine random (blind) samples of curing compounds was tested by both CTR and D-9.

The data from tests performed at D-9 were furnished to CTR for further analysis.

MATERIALS, APPARATUS AND TEST PROCEDURE IN ACCORDANCE WITH ASTM C 156-80

After consultation with the Materials and Test Division of the State Department of Highways and Public Transportation, six liquid membrane-forming curing compounds were selected for laboratory testing. Two types, from each of three manufacturers, as specified by ASTM 309 were tested; these were Type 1-D, clear or translucent with fugitive dye, and Type 2, white pigmented. The manufacturers, for purposes of this report, are identified as S, P, and M. A four gallon sample of each compound was taken as an in-line production sample at the manufacturing plant at the time the barrels of compound, were packaged for delivery to be used in other tests.

The following is a description of the materials used and the test procedure ASTM C 156-80 as it was performed at CTR.

Materials used for mortar were Alamo and Atlas Type I portland cement that conformed to ASTM C 150-80, "Specification for Portland Cement," and graded standard sand that conformed to ASTM C 778 "Specification for Standard Sand." The mortar proportions were determined by adding oven dried sand to a cement paste having a water-cement ratio of 0.40 by weight, to produce a flow of 35 ± 5. The mixing was done at ambient laboratory conditions, approximately 75°F (24°C) and 50 percent relative humidity, in a one-cubic foot mixing machine. The standard mold dimensions were 6 in. by 12 in. (150 mm by 300 mm) at the top, and 5-3/4 in. by 11-3/4 in. (145 mm by 245 mm) at the bottom, and 2 in. inside depth. Metal plates used for determining the loss in weight of volatile matter from the curing compounds were 6 in. by 12 in. (150 mm by 300 mm) with edges raised 1/8 in. (3 mm).

The curing of specimens was accomplished in an environmental chamber at a temperature of 100°F (38°C), and a relative humidity of 32 percent. Due to a lack of humidity control in the chamber, a salt solution of $MgCl_2.H_2O$ was placed in the chamber as described by J. F. Young (Ref 1). A saturated solution of this salt has the ability to maintain a 32 percent relative humidity at a temperature of 100°F.

The mortar specimens were allowed to remain in the environmental chamber during the initial drying for about 2-1/4 to 2-1/2 hours, the time required for surface water to disappear. The application of the curing material was made immediately after the edge sealing and weighing of the uncured specimens. The curing compound was applied at the rate of 1 gal/180 ft^2 ($0.22dm^3/m^2$). The spraying was done using an air operated spray gun.

COMPARISON OF ASTM C 156-80 AND TEX-219-F MOISTURE RETENTION PROCEDURES

The Texas Method Tex-219-F for testing of curing materials is a modification of the ASTM C 156-80 and ASTM C 309 methods. Texas modified the method to improve standard quality with the intent of achieving higher quality concrete curing in the field. Both Texas and ASTM methods specify the same standards and procedures for the reflectance and the drying time of compounds. But in addition, Texas requires a vertical spray test in which a specimen is placed in a vertical position and sprayed with curing compound to examine the effect of running or sagging of the sprayed material.

There are several differences in the moisture retention test procedures of the two methods. The moisture loss in Tex-219-F is expressed as a percentage of the weight of water lost during 72 hours of curing, to the weight of water in specimen at the time compound is applied. ASTM requires calculation of the moisture loss as the loss in weight of water through the curing material in 72 hours, expressed as kilograms lost per square meter of surface. The permissible moisture loss specified should not exceed in 72 hours 4 percent and 0.55 kg/m^2, respectively. In addition, Texas specifies a two percent limit for the 24-hour loss. The correction method for loss in weight of volatile matter from the curing compound is also different in the two tests. Tex-219-F determines the percent solids contained in the compound by calculating the loss in weight of about two grams of compound placed in a small aluminum dish and heated at a temperature of 220°F to 230°F (105°C to 110°C) for three hours. The weight of the solids is determined by multiplying the percent solids with the volume of compound applied on the specimen and the specific gravity as determined from ASTM D 287. ASTM C 156-80 determines the weight loss by spraying a metal plate with the same quantity of compound as used on the specimens and placing it in the chamber with the test specimens.

One other difference in the test procedures is the use by Texas of Alamo Type III (high early strength) cement, instead of Type I (normal) cement used by ASTM. Texas specifies a specific brand to be used in the test in order to reduce variations in moisture loss caused by differences in cement brands. The Type III cement is used to shorten the test time by reducing the initial curing time.

From the above discussion it can be concluded that both methods are basically the same. The introduction by Texas of the vertical spray test gives a simple and good indication of the suitability of compounds on vertical surfaces. The slightly different procedures in the moisture retention tests should not be expected to give different results (if the permissible moisture losses of the two methods are proved to be at the same level), for the following reasons: (a) the weight of moisture loss is obtained using the same procedure in both methods, (b) the expression by Texas of the moisture loss in terms of water in mortar at application should give a better indication than ASTM which expresses loss based on the constant surface area, because loss of moisture is influenced by the amount of moisture present, but specimen moistures do not differ significantly; hence, the effect should be minimal, and (c) the percent solids evaluated by the two methods are not expected to differ considerably. However, the use by

Texas of a certain brand of cement is expected to reduce the variability of the test.

The evaluation of the above variables will be analyzed and discussed in Chapter 8, "Experimental Test Results".

In terms of difficulty and labor intensity in performing the tests, it can be said that both methods are approximately on the same level. Specifically, Tex-219-F requires three more weight recordings and some extra but simple calculations in the moisture retention test. However, the determination of loss in volatile matter during curing is more cumbersome in the ASTM test.

CHAPTER 3. AGITATION TESTS

INTRODUCTION

The purpose of this phase of the study was to evaluate the effectiveness of mechanical agitation on the quality of mixing liquid membrane-forming curing compounds contained in 55-gallon drums.

Both types of curing compounds tested are prone to separation during shipment and storage. In the case of pigmented compounds (Type 2) the separation occurs due to settling of the white pigment. The clear or translucent compounds with fugitive dye (Type 1-D) tend to form a solid crust on the surface of the drum, and in the case of water-emulsion types, water tends to settle at the bottom. Type 2 reflects radiant heat from the sun and results in less of an increase in temperature within the pavement slab throughout the curing period than do the other types. With this type, it is usually possible to detect nonuniform application by careful visual inspection provided the pigments have been uniformly dispersed in the liquid at the time of application. A fugitive dye of a color contrasting well with the concrete in the clear type provides a means of detecting nonuniform application, provided the dye has been uniformly dispersed in the liquid. Therefore, the compounds should be thoroughly mixed before application to secure uniform distribution of pigments and dyes in the liquid. This is of particular importance with the pigmented types because of their tendency to settle rapidly.

The ASTM Standards specify that compounds should be storable for at least six months without deterioration, and that Type 2 should not settle out excessively or cake in the container and should be capable of being mixed to a uniform consistency by moderate stirring.

Laboratory testing included mechanical agitation of compounds stored for three and six months using four different types of blades. Sampling before and after mixing was performed and solids content of samples, as compared to base line content, was correlated with the quality of mixing.

MATERIALS AND AGITATION PROCEDURES

A series of agitation tests was conducted by CTR on six curing compounds, three of Type 1-D, and three of Type 2. These were identical to compounds used for the moisture retention tests. Two types of mechanical agitation were used, one using an electric and one an air-driven motor. The electric motor with its shaft and blade (Type A) is shown in Fig 3.1. The shaft is 27 inches long, and carries only one blade at the bottom (Fig 3.2). Three types of blades were used with the air driven motor. The three shafts with the blades and the motor are shown in Fig 3.3. Figure 3.4 shows the air compressor used to run the motor. Shaft type B is 30 inches long, with blades (Fig 3.5) mounted 3 and 19 inches from bottom, Type C is 24 inches long, with blades (Fig 3.6) 4 inches and 18 inches from bottom, and Type D is 30 inches long with blades (Fig 3.7) 0 inches and 15 inches from bottom. Operating pressures were 55, 60, and 50 psi, respectively. The characteristics of all shafts and their blades are tabulated in Table 3.1. The four agitation devices were selected on the basis of being commercially available and applicable through the 2-inch diameter bunghole of the drums.

Each test had one replication (each shaft was used to mix two drums), hence each type of compound required eight 55-gallon drums. The drums (48 total) were stored in a protected warehouse at the Balcones Research Center to prevent accidental agitation and/or freezing. Three months later a drum was opened and examined for rusting and caking. A 3/8 inch by 1 inch wood stick was inserted in the drum, and the extent and character of portions of the compound that might have separated during storage were determined. Using a syphon pump (Fig 3.8), a sample from the middle third of the drum (approximate midpoint) was taken. The drum was then closed and agitated for five minutes by the appropriate method through the 2-inch diameter bunghole and a sample from the middle third was again taken. The mixing and sampling process was repeated until a total mixing for 30 minutes was reached. The drum head was then removed and top layer and caking that might still be

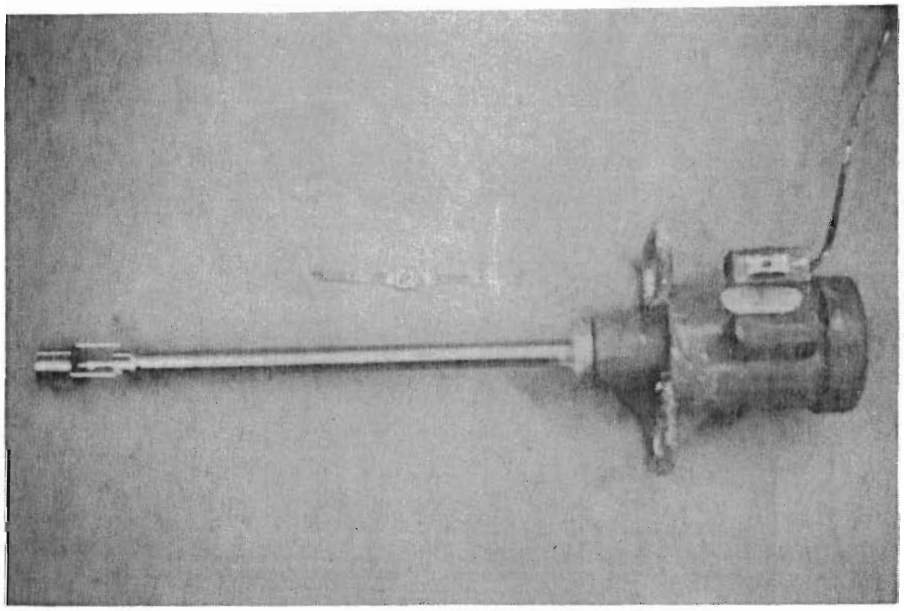


Fig 3.1. Electric motor with shaft (Agitator A).

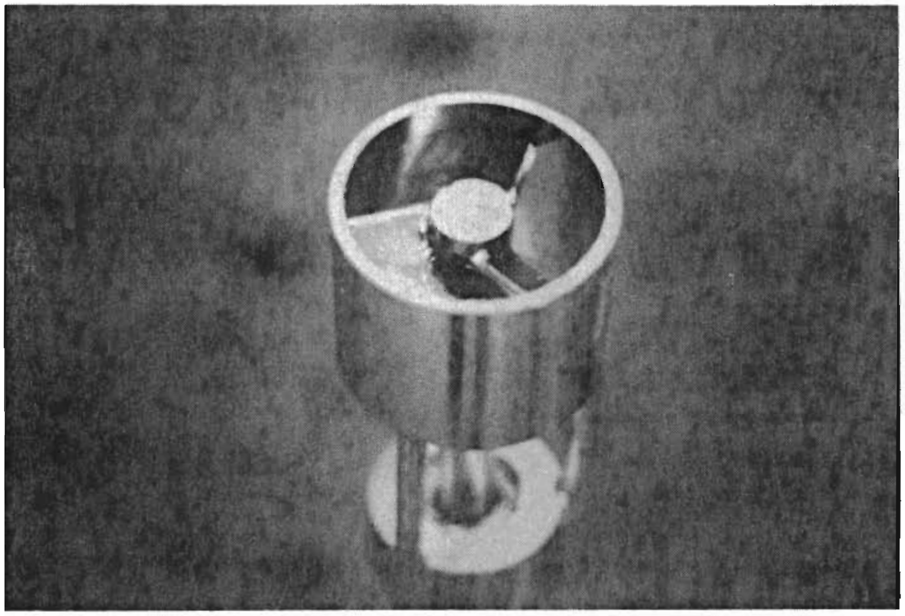


Fig 3.2. Blades of Agitator A.

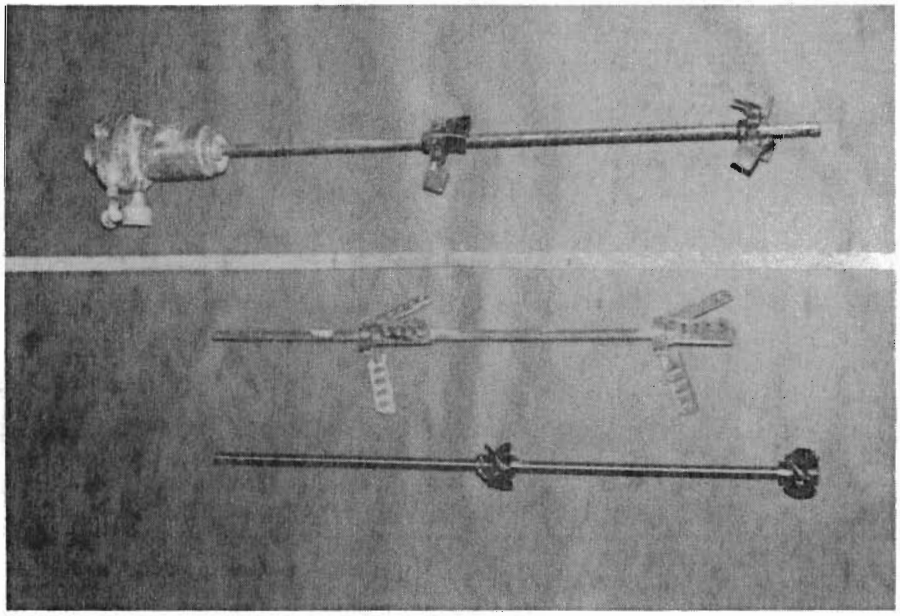


Fig 3.3. Agitators B (with air motor), C, and D.

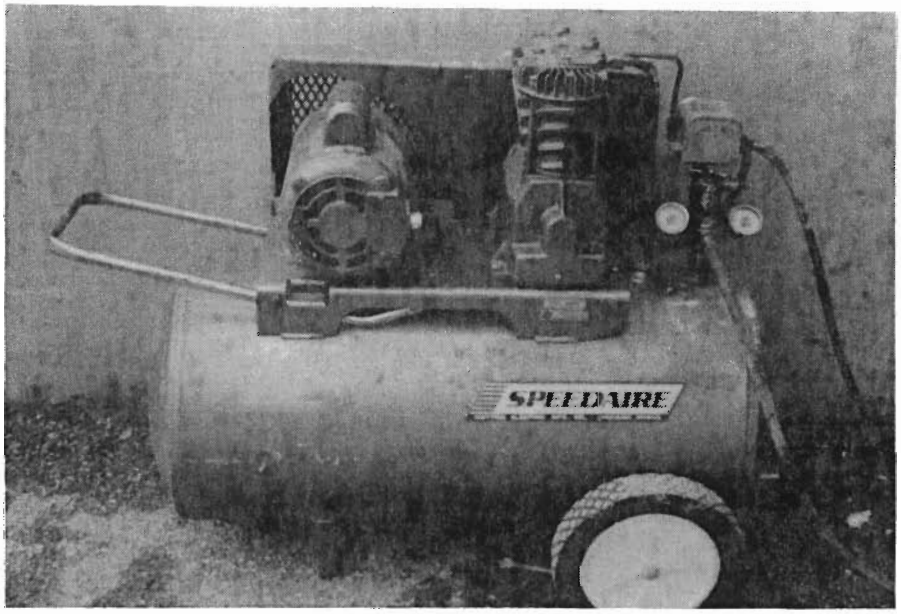


Fig 3.4. Air compressor.

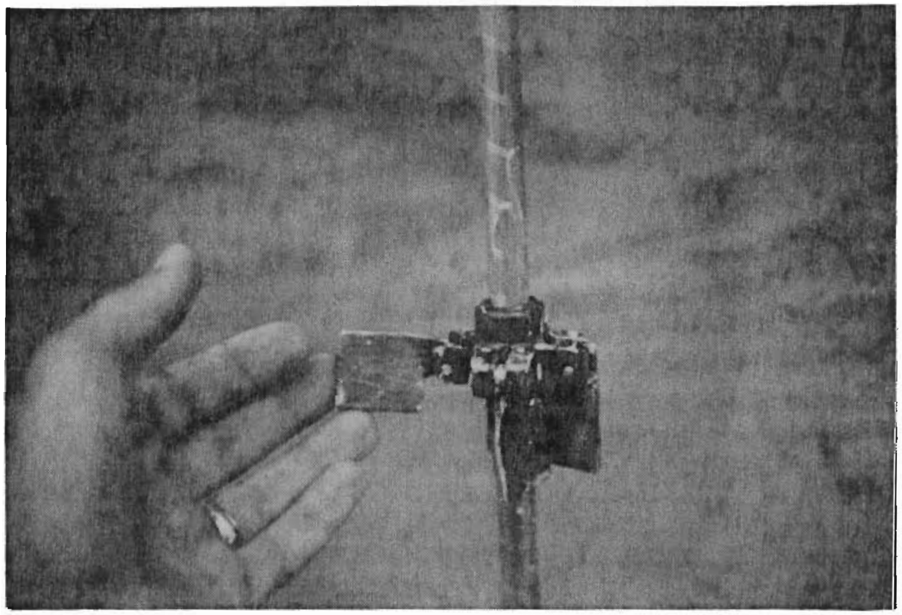


Fig 3.5. Blades of Agitator B.

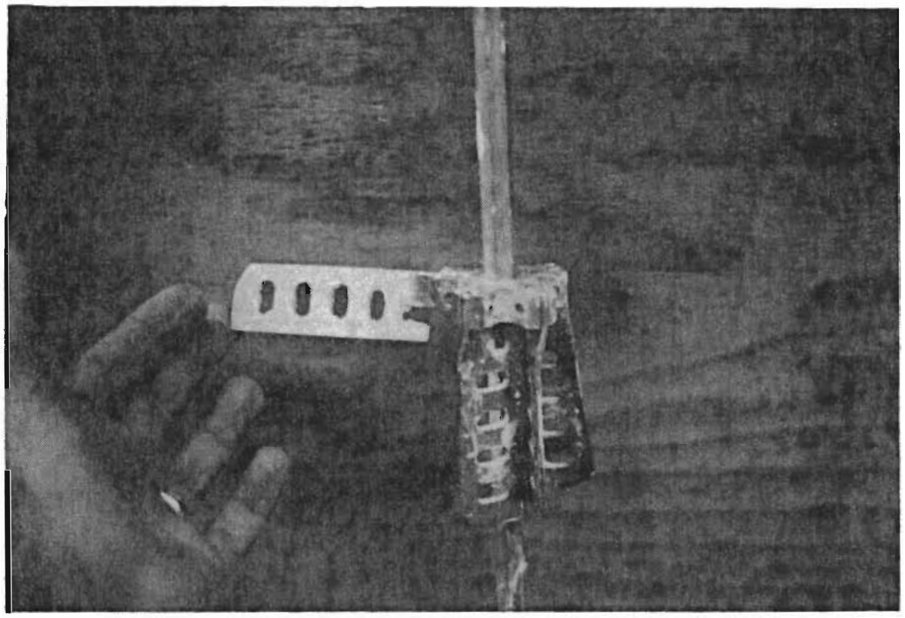


Fig 3.6. Blades of Agitator C.

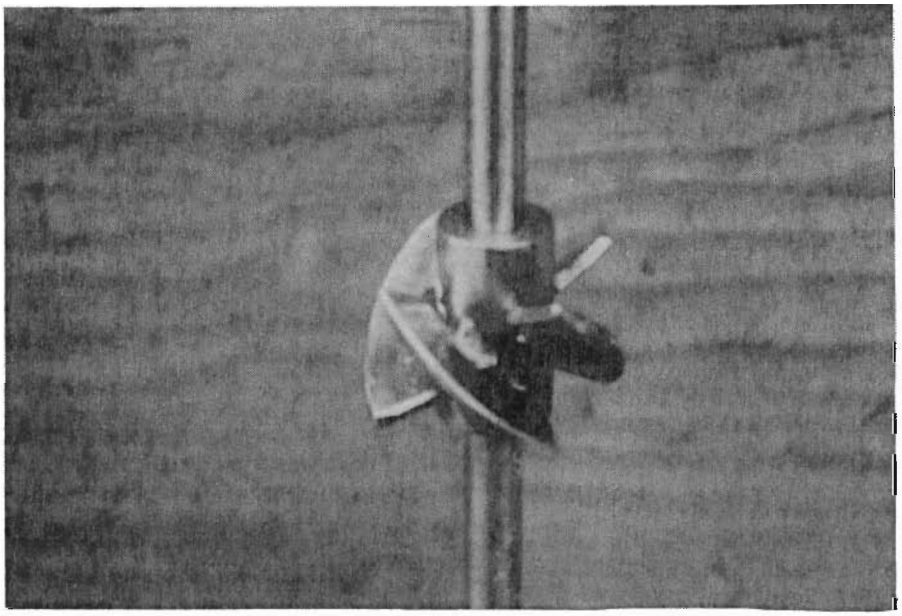


Fig 3.7. Blades of Agitator D.

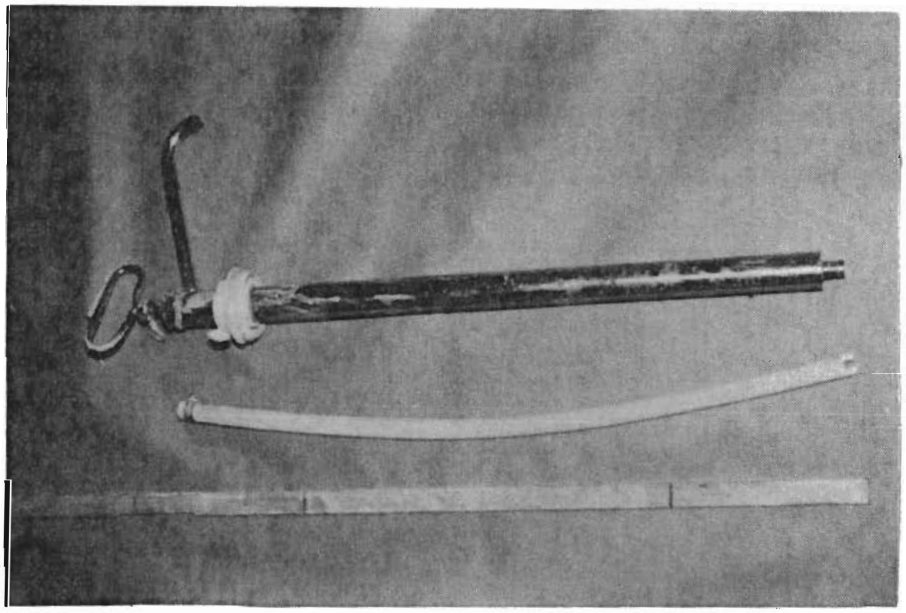


Fig 3.8. Syphon pump for sampling.

TABLE 3.1. 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	Yes	Yes	No

present were measured with the stick. Photographs of the surface of the compound before and after agitation were also taken. The solids content of the samples were determined in accordance with ASTM Method D 1644, "Nonvolatile Content of Varnishes," Method A. These results were compared with solids test results performed by D-9 on in-line samples (base line solids) to determine the mixing quality of each agitation device.

The above sampling procedure was followed for S-1D and S-2 curing compound brands. Mixing and sampling procedures of the other four compounds were revised after some implementation of the initial results had been done. Sampling was changed to 10 minute intervals because the 5 minutes did not give considerable change in solids content. In addition, samples from the top, middle, and bottom of the drum were taken before and after final mixing, while only middle samples were taken at intermittent times. This action was decided upon after solids test results of middle samples had given equal numbers to base line solids even though caking was still present after mixing.

Mixing and sampling of the six curing compounds was repeated at six months, this time obtaining top, middle, and bottom samples only at zero and optimum mixing time as determined from three-month results.

Because it was determined that the solids test results could be misleading, indicating a perfect mixing where resuspension had not been achieved, an additional series of tests was devised 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 a homogeneous consistency. In such a case, top, middle, and bottom samples should show equal portions of settled ingredients in the tubes and in addition, portions should have equal proportions to the base line sample.

CHAPTER 4. NEW MOISTURE RETENTION TEST

INTRODUCTION

Currently ASTM and Texas Standards require a time-consuming and labor intensive test in determining the quality and effectiveness of liquid membrane-forming compounds in particular, to measure moisture retention. The current moisture retention test requires more than 72 hours to perform and it involves proportioning and mixing of mortar, molding, curing, and sealing of specimens, application of curing compound, and a correction procedure for compound loss in weight. Two people are required to work simultaneously for a total of six manhours and the specimens need to occupy the curing cabinet for approximately 74 hours (2 hours initial curing of specimen plus 72 hours curing after compound application).

The need exists to develop a less time-consuming and less difficult moisture retention test that would insure the same quality and perhaps reduce the cost of such tests.

It has been proposed to examine the feasibility of adapting a moisture retention test from the polymer industry for use as a moisture retention test for curing compounds. The polymer industry uses a relatively simple test for moisture retention of polymeric membranes. The test consists of placing a known volume of water in a metal container and sealing the container with the membrane. The container is then placed in a known environment for a period of time and the loss of water is measured by weight.

DESCRIPTION OF A NEW MOISTURE RETENTION TEST

To adopt a new test, the rate of evaporation of normal concrete under curing conditions of the standard moisture retention test (a temperature of 100 ± 2°F, and a relative humidity of 32 ± 2 percent) was determined. Loss of moisture from uncured concrete specimens (blank specimens) cast during the multiple moisture retention tests was determined at two-hour intervals and the resulting curve is shown in Fig 4.1.

Seven identical test cups were manufactured using polymethyl methacrylate (Fig 4.2). Each test cup consists of a cylindrical tube 4 inches (100 mm) in depth and 3 inches (75 mm) in internal diameter, sealed with a flat round cover at the bottom such as to prevent any water leakage. A similar cover with a 3-inch (75 mm) round hole is rigidly clamped at the top of the tube using four bolts. This cover is used to hold the membrane tight on the apparatus. To perform the test, a known volume of water was placed in the tube and a selected membrane was placed on top of the cup and fixed with the cover. Curing compound was sprayed on the membrane at the required rate and cup was placed in a curing cabinet for a period of time. The loss in weight of the cup gave the evaporation rate through the membrane. The loss in weight of curing material for weight loss correction was determined by applying the above procedure without using any water in the apparatus. A test cup with a treated membrane is shown in Fig 4.3.

For purposes of this report, 23 different membranes were selected and their permeabilities were determined in order to choose membranes that have similar permeability to the initial rate of evaporation of uncured concrete. Selected membranes were then cured by applying curing compound at the rate of 180 sf/gal. The results were compared to the results from multiple moisture retention tests described in Chapter 2 for validation.

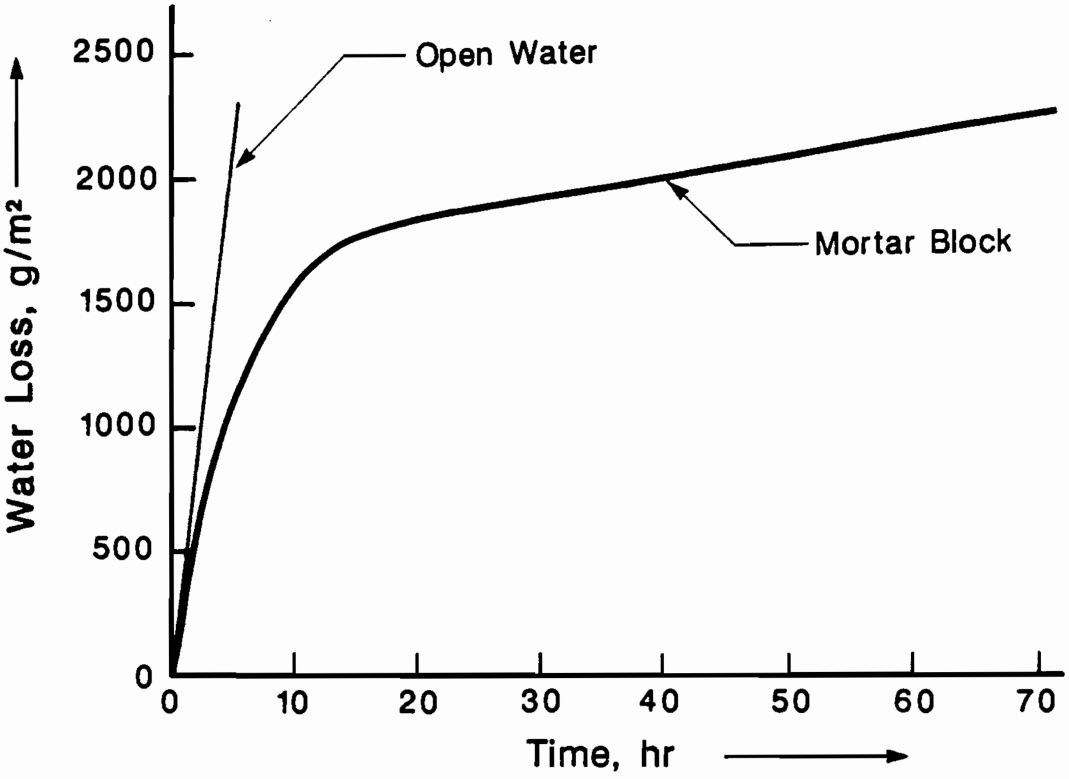
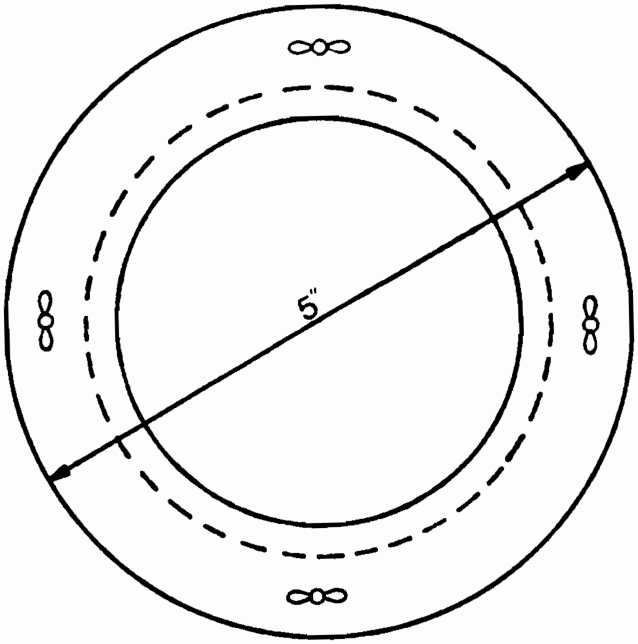
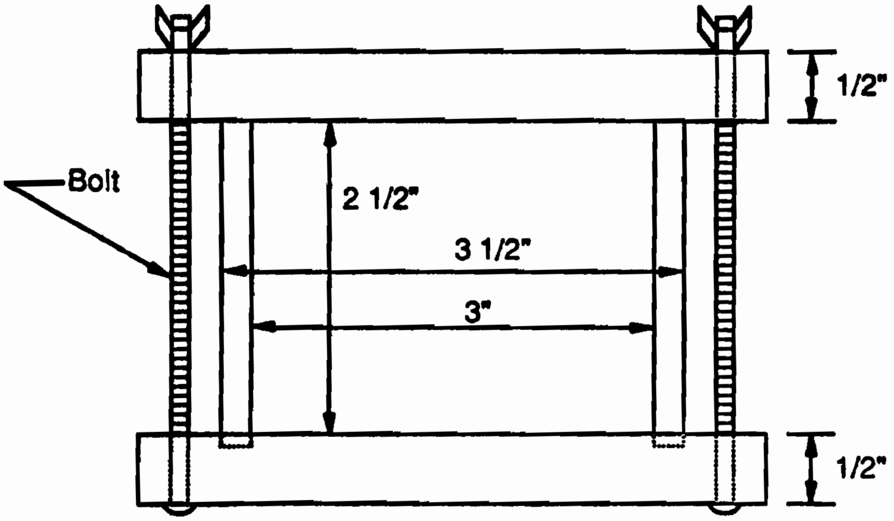


Fig 4.1. Evaporation rates of normal concrete and water.



(a) Plan



(b) Side view

Fig 4.2. Apparatus for new moisture retention test.

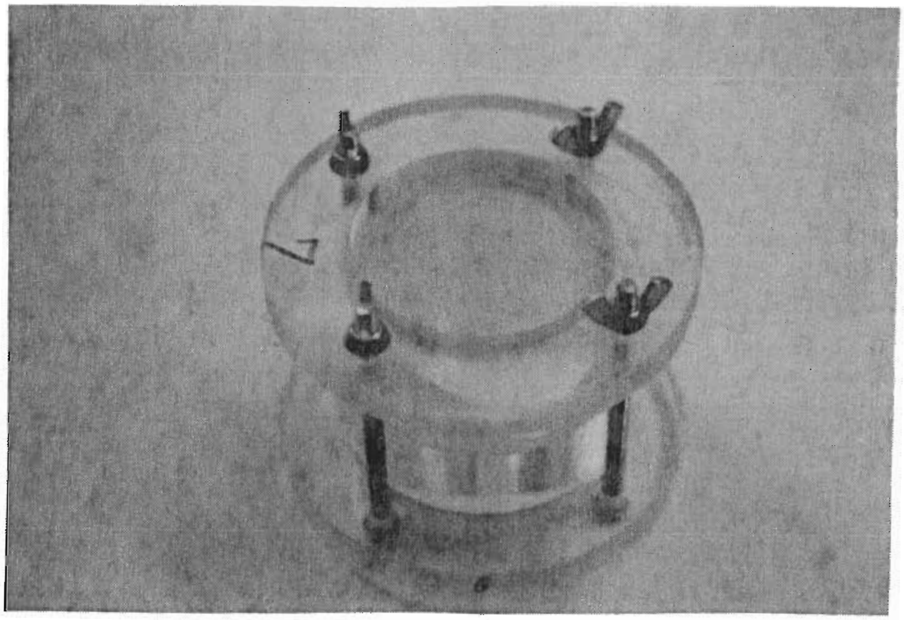


Fig 4.3. New moisture retention test apparatus.

CHAPTER 5. SHELF LIFE

INTRODUCTION

Present SDHPT specifications for the use of liquid membrane-forming curing compounds assume that such compounds perform as intended, given reasonable agitation effort, after a period of undisturbed storage at non-freezing temperatures of up to six months. While this is an accepted practice at present, there is little hard data to support the specification.

The purpose of this phase of the study was to determine the actual period for which various curing compounds could be stored prior to use and still, when agitated, produce the uniform mixture moisture retention characteristics necessary for successful use in the field.

MATERIALS AND TEST PROCEDURE

Six liquid membrane-forming curing compounds, identical to those used in the moisture retention and agitation tests, were tested. Four fifty-five gallon drums of each compound were stored at the same location as those used for the agitation tests. The sampling procedure, solids content test, and preparation of specimens in glass tubes were all identical to those associated with the agitation tests (as described in Chapter 3) except for the following.

First, tube specimens were not prepared for S-1D and S-2 three month samples, since the inadequacy of the solids content test was unknown until after analysis of these samples. Second, these tests also consisted of only middle samples, since this was the original plan of action, modified based on early results. Third, since the goal was not to test agitation devices, but rather to check the shelf life of the materials, agitation was performed using the type "B" air driven agitator shaft, with the drum head removed.

Further, the agitation shaft was typically moved freely within the drum during agitation, including efforts to use the agitation blades to scrape settled solids form the sides and bottom of the drum interior. This agitation procedure was continued until the mixture appeared visually uniform and scraping of the sides and bottom of the drum interior with a 3/8 in by 1 in stick left no appreciable amount of settled solid material adhering to the stick. In most cases, however, total agitation time was limited to 30 minutes, the time having been chosen as a limit to workable field procedure. The agitation procedures described here are not possible with the normal, closed-headed drums supplied to contractors. However, the purpose of the tests was to determine if agitation alone, in any form, could restore curing compounds to a usable state after a certain period of storage. Thus, the normal requirement for the agitation device and method to be applicable through the 2 inch bunghole did not apply to these tests.

CTR Tests

The testing schedule for each type of compound was as follows. The first drum was opened, agitated, sampled, and photographed after three months, six months, nine months, and one year of storage. The second drum was first opened and agitated after six months of undisturbed storage, then again at nine months, and finally, after a year. The third drum was left undisturbed for nine months, opened and agitated at that time, and then examined again after an additional three months. Finally, the fourth drum was allowed to sit for one full year before being opened and agitated. This schedule allowed the effects of various periods of undisturbed storage, as well as of intermediate agitation during storage, to be observed.

Initially, top, middle, and bottom samples were taken from each drum both before and after agitation. The agitation procedure was improved with practice and the visual inspection described earlier proved reliable. Hence, most nine and twelve month samplings, as well as M-1D six month samplings, consisted of top, middle, and bottom samples prior to agitation; but only samples from the middle third of the drum after agitation.

SDHPT Tests

In addition to these tests performed at CTR, Division 9 of SDHPT performed the normal full battery of curing compound tests on nine and twelve month samples of all six curing compounds. These tests included a vertical spray test, drying time, flash temperature, solids content, specific gravity, and standard Tex-219-F moisture retention tests on both types of compound. In addition, Type 2 white pigmented compounds were tested for reflectivity.

CHAPTER 6. APPLICATION PATTERN

INTRODUCTION

For many years the SDHPT, as did most other states, used a longitudinal burlap drag finish on PCC pavements. Some ten years ago a transverse tine texture was adopted that required a 1/8 in wide by 1/8 in deep groove spaced at 1/4 to 1/8 in. Recently a surface finish which combines an astrograss drag longitudinally and a transverse tine groove spaced at 1 inch, plus or minus 1/4 in, was adopted.

The typical pattern for spraying the curing compound is to make a single longitudinal pass with a spray bar having uniformly spaced spray nozzles. The rate of application is judged by the length of pavement covered per barrel of compound.

The purpose of this phase of the study was to determine if any benefits can be derived from changing the direction of travel of the spray bar or applying the compound to the pavement in two coats instead of one, and, additionally, to determine if a higher rate of application (more curing compound) would significantly reduce water retention in the concrete.

MATERIALS AND PROCEDURES

For these tests, it was first necessary to construct a section of a typical field spray bar. Based on information obtained from several contractors and observations made in the field, a five foot long spray bar was constructed and adjusted until a visually uniform spray pattern was achieved. The final form of the spray bar included three nozzles (as shown in Fig 6.1) spaced at 19 inches on center. The bar was passed over the surface to be treated at a height of 34 inches, and the curing compound was supplied, via a pressure pot, under a pressure of 55 psi (Fig 6.2).

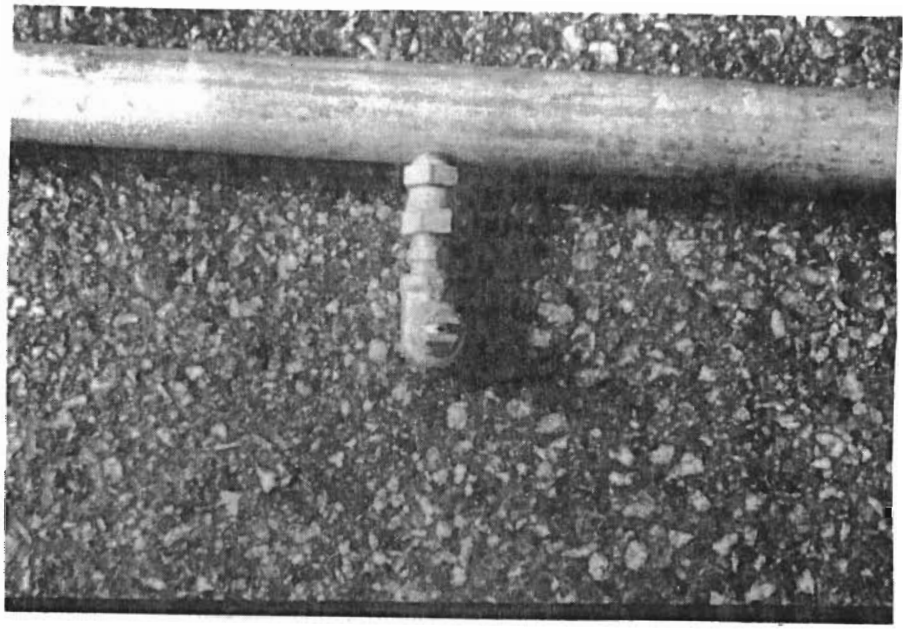


Fig 6.1. One of three nozzles used on spray bar.

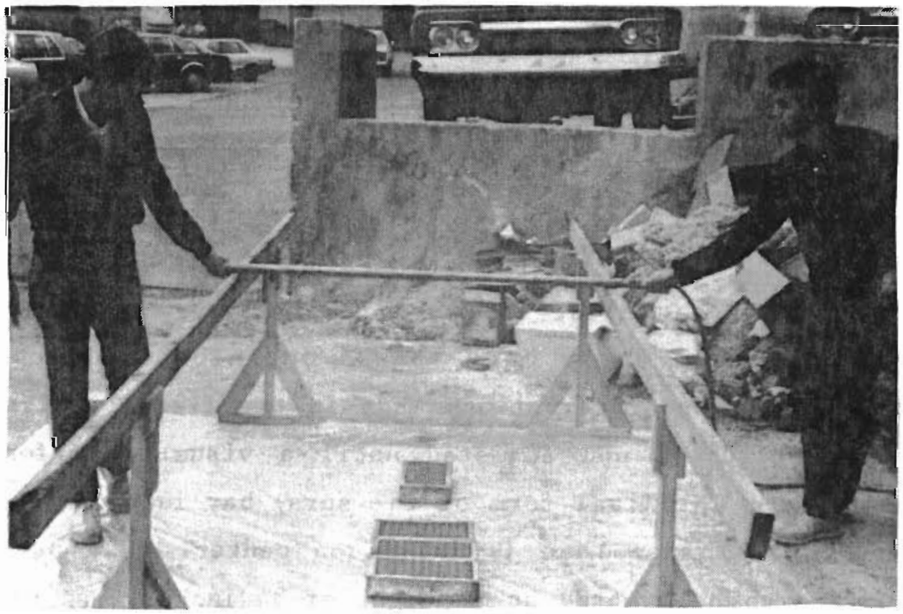


Fig 6.2. Spray bar being used for a "parallel to grooves" application pattern test.

For reasons of consistency and ease of handling, all application pattern tests were performed using type P-ID curing compound. Seven different application patterns were tested:

(1) spray bar passed over the concrete surface once, in a direction perpendicular to the tine grooves
(2) spray bar passed over the concrete surface once, in a direction parallel to the tine grooves
(3) spray bar passed over the concrete surface twice in the same direction, perpendicular to the tine grooves
(4) spray bar passed over the concrete surface twice in the same direction, parallel to the tine grooves
(5) spray bar passed over the concrete surface twice in opposite directions, perpendicular to the tine grooves
(6) spray bar passed over the concrete surface twice in opposite directions, parallel to the tine grooves
(7) spray bar passed over the concrete surface twice, once parallel to the tine grooves, once perpendicular to the tine grooves.

In all cases, the total final application rate was the present SDHPT standard of 1 gallon per 180 square feet.

The experimental procedure for each of the seven cases was as follows. Four standard ASTM C 156-80 mortar blocks were prepared. The C 156-80 moisture retention test was performed on each of these, with the following procedural changes. First, instead of the standard smooth texture, three of the blocks were textured with the newly adopted texture including both the grooves at 3/4-inch spacing and the astrograss drag. The fourth block was textured, as a standard, using the first grooved texture (with grooves at 1/2-inch spacing).

For each application pattern, the three blocks with the new SDHPT texture were treated with curing compound in the specific pattern being tested. The standard block, with the old grooved texture, was always treated with curing compound using a single pass of the spray bar, moving the bar perpendicular to the grooves.

MOISTURE RETENTION CAPABILITY

A final set of four ASTM C 156 type mortar blocks was prepared. Using the optimum application pattern, this set of blocks was treated exactly as the earlier application pattern block sets, except that curing compound was applied at a rate of 1 gallon per 150 sq ft. The results of this test were then combined with the corresponding earlier tests, using an application rate of 1 gallon per 180 sq ft, to suggest the application rate necessary to reduce moisture loss to the degree necessary to eliminate the cracking problem noted by SDHPT.

CHAPTER 7. APPLICATION RATE

INTRODUCTION

One problem that every inspector for PCC pavement faces is how to determine if the membrane being applied is uniform. In many cases the decision is obvious because with visual inspection it can be determined that the compound is collecting in the grooves or is not covering the sides of the grooves or is streaked due to differences in the nozzles. However, it is difficult to judge whether or not the lighter covered portion has an adequate membrane.

It seems reasonable that a device that measured reflectivity could be calibrated against some standard and be used to estimate membrane thickness. This assumes that a thicker membrane would contain more white pigment or fugitive dye and, hence, have a higher reflectivity.

REFLECTANCE STUDIES

The proposal was made to develop a device and methodology to compare reflectance ratios for various application rates of various curing compounds. The device built consisted of two Cadmium Sulfide photo cells in a wheatstone bridge circuit. The circuit as built is diagrammed in Fig 7.1. Figures 7.2 and 7.3 show the prototype adjusted to read 0 at 18 percent reflectance (grey cards) and taking a reading of -250 mV from a known 90 percent reflectant surface (white card). The device was calibrated by reading light reflections from some standard surface with both the photo cells, and adjusting the other resistances in the circuit until there is no measured potential drop across the circuit.

Using several other surfaces of known reflectance under constant light conditions, one of the photo cells is placed on these surfaces, one at a time, while the other photo cell remains over the original surface. Reading

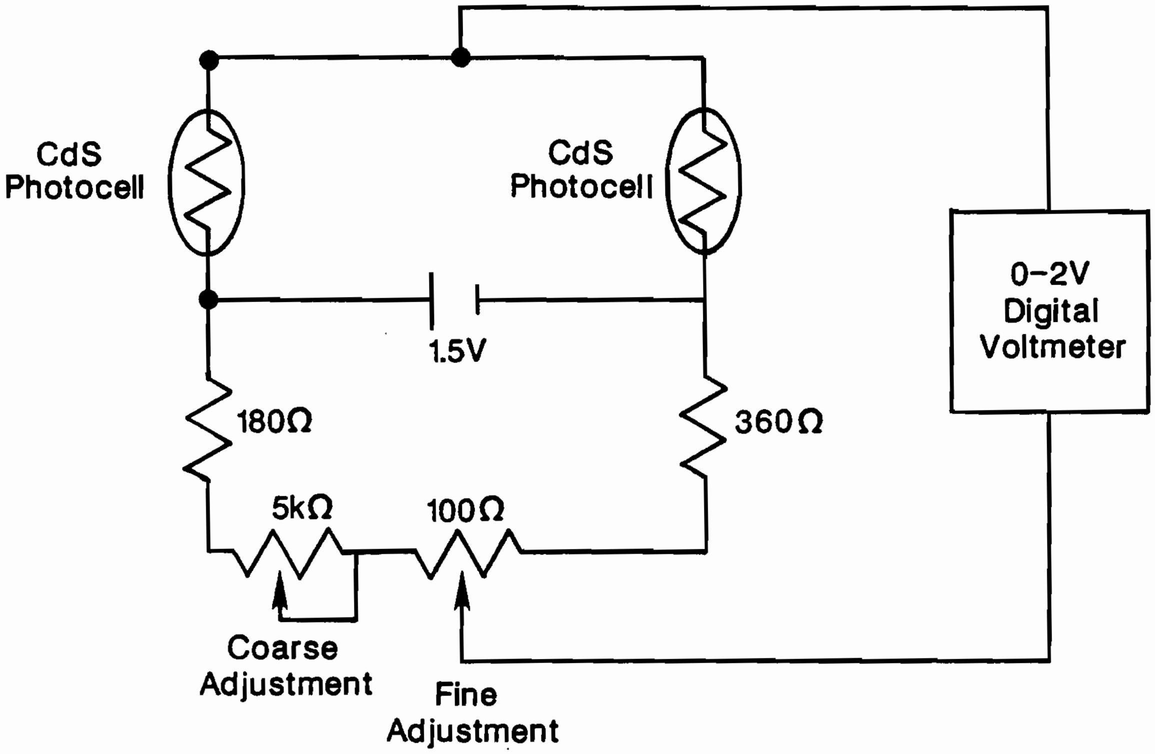


Fig 7.1. Circuit diagram of reflectance meter used in rate of application studies.

Fig 7.2. Reflectance meter standardized to 18 percent reflectance.

Fig 7.3. Reflectance meter reading -250 mV from 90 percent reflectant surface while standardized to 18 percent reflectance.

a potential drop for each of the known surfaces, a curve relating potential drop and reflectance of the surface is generated. This curve, along with the reflectance meter, can be used to determine the reflectances of concrete surfaces coated with various amounts of curing compound. In theory, the inspector would merely have to measure surface reflectance to verify that the correct coverage rate had been achieved.

In order to investigate the correlation between reflectance and rate of coverage, type P-2 curing compound was tested in the following manner. The reflectance meter was standardized to a surface of a known reflectance of 18 percent. Three fresh concrete surfaces were then treated with curing compound at rates of 1 gallon per 150 sq ft, 1 gallon per 180 sq ft, and 1 gallon per 200 sq ft. Reflectance readings for each of these coverage rates were recorded and examined for significance. This procedure was then repeated, with the reflectance meter standardized to a 90 percent reflectance.

CHAPTER 8. IN-LINE VERSUS DRUM SAMPLING

INTRODUCTION

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 prior to sampling, as field material has been.

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 re-agitation, some time after manufacture.

PROCEDURE

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 the "in-line" sample had been.

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 out and examined. These observations are presented in Chapter 9 of this report.

CHAPTER 9. EXPERIMENTAL TEST RESULTS

INTRODUCTION

The experimental tests outlined in the previous chapters were performed to investigate the adequacy of the current Texas State Department of Highways and Public Transportation specification related to membrane curing compounds with regard to moisture retention and agitation quality. In addition the feasibility of adapting a new moisture retention test was examined for the purpose of providing an easier and less time-consuming test.

Six curing compounds were selected for multiple moisture retention tests. For purposes of this study the manufacturers are identified as S, P, and M. Designations 1D and 2 next to the letters indicate curing compounds Types 1D and 2, respectively. A series of moisture retention tests were conducted by CTR, using the ASTM method, and by the Bituminous Section of D-9, using the Texas method on identical curing compound samples, to investigate the performance of the two test methods. Agitation tests were performed using four different agitation devices. The curing compounds were delivered and stored in 55-gallon open-headed drums. Mixing was performed after three and six months of storage. The mixing quality was determined by performing solids and visual inspection tests on agitated samples. For the new moisture retention test a total of 23 different membranes were selected. Permeability of cured membranes was evaluated and results were compared with multiple moisture retention tests for validation.

Experimental results from the three series of tests along with discussion and comments are included herein.

MOISTURE RETENTION TESTS

Multiple Moisture Retention Tests in Accordance with ASTM C 156-80

The moisture retention ability of five curing compounds was evaluated by the Center for Transportation Research (CTR) in accordance with ASTM C 156-80 and ASTM C 309. The sixth curing compound was not tested because at the time of testing tiny grains had developed in the material during storage, making spraying impossible. Three tests were performed on each compound, two consisting of three specimens each and one of four specimens, except for the first compound, from which four tests consisting of three specimens each were performed. Thus, a total of 52 specimens were manufactured and tested.

The results of these tests are tabulated separately for each type of compound in Tables 9.1, 9.2, and 9.3. These results indicated that the average moisture loss of all the specimens in 72 hours is 0.193 kg/m^2 for Compound S- , 0.141 kg/m^2 for Compound S-2, 0.190 kg/m^2 for Compound P-1D, 0.240 kg/m^2 for Compound P-2, and 0.226 kg/m^2 for Compound M-1D. All these values are considerably less than the maximum permissible value of 0.55 kg/m^2, ranging from 26 to 44 percent of that value. Standard deviations for moisture loss range from 0.033 to 0.092 kg/m^2, with corresponding coefficients of variations of 23.4 to 47.7 percent. Calculating the standard deviations by averaging for each compound the moisture losses in each test, the standard deviation range changes to 0.023 to 0.053 kg/m^2, with corresponding coefficients of variations of 16.4 to 27.5 percent. The average coefficients of variation of the two averaging procedures are 33.2 and 19.9 percent, respectively.

Multiple Moisture Retention Tests in Accordance with Tex-219-F

The moisture retention ability of six curing compounds was evaluated by the Bituminous Section of D-9 of the SDHPT on samples identical to those furnished to CTR. The tests were performed in accordance with Texas Method Tex 219-F. D-9 did perform the tests on the compound that CTR could not complete tests for, because the tests were performed shortly after the delivery of the material and before the development of grains in the material. A total of sixty specimens, ten for each compound, were tested. For each compound, five tests were performed, with each consisting of two specimens.

The results of the tests are tabulated separately for each compound in Tables 9.4, 9.5, and 9.6. Columns three and four in each table give the

TABLE 9.1. MOISTURE RETENTION TEST RESULTS IN ACCORDANCE WITH ASTM C 156-80

(A) S-1D

Test Number	Specimen Number	72-Hour Moisture Loss (kg/m^2)	Average (kg/m^2)
1	1 2 3	0.314 0.217 0.325	0.285
2	4 5 6	0.082 0.178 0.168	0.143
3	7 8 9	0.207 0.282 0.272	0.254
4	10 11 12	0.094 0.104 0.072	0.090
Average (kg/m^2)		0.193	0.193
Std. Dev. (kg/m^2)		0.092	0.053
C. V. (%)		47.7	27.5

(continued)

TABLE 9.1. (CONTINUED)

(B) S-2

Test Number	Specimen Number	72-Hour Moisture Loss (kg/m^2)	Average (kg/m^2)
	1	0.133	
1	2	0.155	0.119
	3	0.069	
	4	0.133	
2	5	0.187	0.151
	6	0.133	
	7	0.156	
3	8	0.144	0.150
	9	0.119	
	10	0.181	
Average (kg/m^2)		0.141	0.140
Std. Dev. (kg/m^2)		0.033	0.023
C. V. (%)		23.4	16.4

TABLE 9.2. MOISTURE RETENTION TEST RESULTS IN ACCORDANCE WITH ASTM C 156-80

(A) P-1D

Test Number	Specimen Number	72-Hour Moisture Loss (kg/m^2)	Average (kg/m^2)
1	1	0.144	0.206
	2	0.233	
	3	0.241	
2	4	0.102	0.130
	5	0.166	
	6	0.123	
3	7	0.284	0.223
	8	0.252	
	9	0.220	
	10	0.134	
Average (kg/m^2)		0.190	0.186
Std. Dev. (kg/m^2)		0.063	0.036
C. V. (%)		33.2	19.4

(continued)

TABLE 9.2. (CONTINUED)

(B) P-2

Test Number	Specimen Number	72-Hour Moisture Loss (kg/m^2)	Average (kg/m^2)
1	1	0.197	0.169
	2	0.100	
	3	0.211	
2	4	0.361	0.318
	5	0.275	
	6	0.318	
3	7	0.305	0.236
	8	0.219	
	9	0.209	
	10	0.209	
Average (kg/m^2)		0.240	0.241
Std. Dev. (kg/m^2)		0.075	0.043
C. V. (%)		31.3	17.8

TABLE 9.3. MOISTURE RETENTION TESTS RESULTS IN ACCORDANCE WITH ASTM C 156-80: M-1D

Test Number	Specimen Number	72-Hour Moisture Loss (kg/m^2)	Average (kg/m^2)
	1	0.218	
1	2	0.132	0.168
	3	0.154	
	4	0.172	
2	5	0.237	0.194
	6	0.172	
	7	0.301	
3	8	0.237	0.293
	9	0.323	
	10	0.312	
Average (kg/m^2)		0.226	0.218
Std. Dev. (kg/m^2)		0.069	0.040
C. V. (%)		30.5	18.3

TABLE 9.4. MOISTURE RETENTION TEST RESULTS IN ACCORDANCE WITH TEX-219-F CONVERTED TO ASTM C 156-80

(A) S-1D

		Tex-219-F Moisture Loss (Percent)			ASTM C 156-80 Converted Moisture Loss (kg/m^2)	
Test Number	Specimen Number	24 hours	72 hours	Average	72 hours	Average
1	1	0.62	1.29	1.54	0.206	0.249
	2	0.87	1.80		0.292	
2	3	0.48	0.89	0.89	0.142	0.142
	4	0.41	0.89		0.142	
3	5	0.55	1.30	1.16	0.206	0.185
	6	0.55	1.02		0.163	
4	7	0.75	1.16	1.09	0.185	0.174
	8	0.61	1.02		0.163	
5	9	0.62	0.89	0.88	0.142	0.141
	10	0.61	0.87		0.140	
Average (kg/m^2)		0.61	1.11	1.11	0.178	0.178
Std. Dev. (kg/m^2)			0.29	0.21	0.048	0.034
C.V. (%)			26.1	18.9	27.0	19.1

(continued)

TABLE 9.4. (CONTINUED)

(B) S-2

		Tex-219-F Moisture Loss (Percent)			ASTM C 156-80 Converted Moisture Loss (kg/m^2)	
Test Number	Specimen Number	24 hours	72 hours	Average	72 hours	Average
1	1	1.33	2.00	2.07	0.323	0.334
	2	1.46	2.13		0.344	
2	3	1.29	2.37	2.31	0.376	0.366
	4	1.43	2.25		0.355	
3	5	1.55	2.36	2.29	0.376	0.366
	6	1.41	2.21		0.355	
4	7	1.25	2.37	2.54	0.387	0.409
	8	1.42	2.70		0.430	
5	9	1.41	2.08	2.24	0.333	0.360
	10	1.40	2.40		0.387	
Average (kg/m^2)		1.40	2.29	2.29	0.367	0.367
Std. Dev. (kg/m^2)			0.20	0.14	0.031	0.022
C.V. (%)			8.7	6.1	8.4	6.0

TABLE 9.5. MOISTURE RETENTION TEST RESULTS IN ACCORDANCE WITH TEX-219-F CONVERTED TO ASTM C 156-80

(A) P-1D

		Tex-219-F Moisture Loss (Percent)			ASTM C 156-80 Converted Moisture Loss (kg/m²)	
Test Number	Specimen Number	24 hours	72 hours	Average	72 hours	Average
1	1	1.38	2.26	2.66	0.357	0.422
	2	1.50	3.06		0.486	
2	3	1.48	2.94	2.81	0.475	0.454
	4	1.48	2.68		0.432	
3	5	1.36	2.97	2.78	0.475	0.443
	6	1.23	2.59		0.411	
4	7	1.23	2.30	2.36	0.368	0.379
	8	1.35	2.42		0.389	
5	9	1.35	2.41	2.41	0.389	0.389
	10	1.21	2.40		0.389	
Average (kg/m²)		1.36	2.60	2.60	0.417	0.417
Std. Dev. (kg/m²)			0.30	0.21	0.047	0.033
C.V. (%)			11.5	8.1	11.3	7.9

(continued)

TABLE 9.5. (CONTINUED)

(B) P-2

Test Number	Specimen Number	Tex-219-F Moisture Loss (Percent) 24 hours	72 hours	Average	ASTM C 156-80 Converted Moisture Loss (kg/m^2) 72 hours	Average
1	1	1.21	2.09	2.23	0.333	0.335
	2	1.21	2.36		0.376	
2	3	0.55	1.09	1.08	0.172	0.172
	4	0.54	1.07		0.172	
3	5	0.94	1.74	1.93	0.280	0.307
	6	1.09	2.11		0.333	
4	7	0.82	1.50	1.57	0.237	0.248
	8	0.82	1.63		0.258	
5	9	0.82	1.64	1.60	0.301	0.274
	10	0.67	1.55		0.247	
Average (kg/m^2)		0.87	1.68	1.68	0.271	0.271
Std. Dev. (kg/m^2)			0.42	0.30	0.068	0.048
C.V. (%)			25.0	17.9	25.1	17.7

TABLE 9.6. MOISTURE RETENTION TEST RESULTS IN ACCORDANCE WITH TEX-219-F CONVERTED TO ASTM C 156-80

(A) M-1D

		Tex-219-F Moisture Loss (Percent)			ASTM C 156-80 Converted Moisture Loss (kg/m^2)	
Test Number	Specimen Number	24 hours	72 hours	Average	72 hours	Average
1	1	0.29	1.38	1.51	0.219	0.241
	2	0.29	1.64		0.262	
2	3	0.30	0.90	0.73	0.144	0.117
	4	0.22	0.56		0.090	
3	5	0.23	0.69	0.69	0.112	0.112
	6	0.23	0.69		0.112	
4	7	0.36	0.97	0.97	0.155	0.155
	8	0.23	0.96		0.155	
5	9	0.23	0.96	1.03	0.155	0.166
	10	0.23	1.09		0.176	
Average (kg/m^2)		0.26	0.98	0.98	0.158	0.158
Std. Dev. (kg/m^2)			0.33	0.23	0.052	0.037
C.V. (%)			33.7	23.5	32.9	23.4

(continued)

TABLE 9.6. (CONTINUED)

(B) M-2

Test Number	Specimen Number	Tex-219-F Moisture Loss (Percent) 24 hours	72 hours	Average	ASTM C 156-80 Converted Moisture Loss (kg/m^2) 72 hours	Average
1	1	0.70	2.03	2.16	0.327	0.343
	2	0.98	2.28		0.359	
2	3	0.92	2.02	1.81	0.316	0.284
	4	0.91	1.59		0.252	
3	5	0.92	1.88	1.87	0.295	0.295
	6	1.04	1.85		0.295	
4	7	1.18	2.02	2.03	0.320	0.324
	8	1.10	2.04		0.327	
5	9	0.98	1.79	1.77	0.284	0.279
	10	0.92	1.74		0.273	
Average (kg/m^2)		0.97	1.92	1.92	0.305	0.305
Std. Dev. (kg/m^2)			0.19	0.13	0.031	0.022
C.V. (%)			9.9	6.8	10.2	7.2

moisture loss results of the Texas procedure in 24 and 72 hours. For purposes of comparison with ASTM, these results, which have percentage units, were converted into units of kg/m^2 as specified in ASTM C 156-80. The conversion was done using the weights obtained in the Texas Method and the formula for moisture loss used in ASTM. Only the 72-hour results were converted because ASTM specifies moisture loss in 72 hours only. The results indicate that the average moisture loss of all the specimens in 72 hours is 1.11 percent for Compound S-1D, 2.29 percent for Compound S-2, 2.60 percent for Compound P-1D, 1.68 percent for Compound P-2, 0.98 percent for Compound M-1D, and 1.92 percent for Compound M-2. These values range from 25 to 65 percent of the maximum allowable loss of 4 percent. Standard deviations for moisture loss range from 0.19 to 0.42 percent with corresponding coefficients of variation of 9.9 to 33.7 percent. Calculating the standard deviation of the converted values using for each compound the average losses in five tests, the range changes to 0.13 to 0.30 percent with corresponding coefficients of variation of 6.8 to 17.9 percent. The average coefficients of variation of the two averaging procedures are 21.0 and 14.9, respectively.

Multiple Moisture Retention Tests on Blind Samples

In addition to multiple moisture retention tests on six compounds, a series of tests was conducted on random (blind) samples of curing compounds from samples that were furnished to D-9 for routine testing. Nine different compounds were tested, all of them being Type 2. CTR performed tests according to ASTM Method on 27 specimens, three for each compound. The Bituminous Section of D-9 performed tests according to Tex-219-F on 18 specimens or 2 per compound. Tables 9.7 and 9.8 show the tabulized results of CTR and D-9, respectively. CTR moisture loss results were determined in 72 hours, whereas D-9 results were determined in 24 hours. D-9 stopped the tests after 24 hours because this is their normal practice in routine tests. From their long experience, if a test passes the requirement at 24 hours, then it will in all likelihood pass the 72-hour requirement.

TABLE 9.7. MOISTURE RETENTION TEST RESULTS ON BLIND SAMPLES IN ACCORDANCE WITH ASTM C 156-80.

Code Number	Specimen Number	Compound Brand Type	ASTM C 156-80 Moisture Loss in 72 Hours (kg/m^2)	Average
A	1	S-2	0.422	0.450
	2		0.400	
	3		0.529	
B	1	S-2	0.284	0.223
	2		0.198	
	3		0.187	
C	1	P-2	0.244	0.208
	2		0.180	
	3		0.201	
D	1	S-2	0.523	0.552
	2		0.609	
	3		0.523	
E	1	S-2	0.278	0.285
	2		0.278	
	3		0.300	
F	1	S-2	0.286	0.290
	2		0.286	
	3		0.297	
G	1	S-2	0.195	0.223
	2		0.270	
	3		0.205	
H	1	S-2	0.369	0.373
	2		0.369	
	3		0.380	
I	1	S-2	0.175	0.189
	2		0.218	
	3		0.175	

TABLE 9.8. MOISTURE RETENTION TEST RESULTS ON BLIND SAMPLES IN ACCORDANCE WITH TEX-219-F CONVERTED TO ASTM C 156-80

			Tex-219-F		ASTM C 156-80	
Code Number	Specimen Number	Compound Brand/ Type	Moisture Loss in 24 Hours (Percent)	Average	Converted Moisture Loss in 24 Hours (kg/m^2)	Average
A	1	S-2	4.97	4.65	0.802	0.748
	2		4.33		0.695	
B	1	S-2	2.30	2.67	0.374	0.433
	2		3.03		0.492	
C	1	P-2	1.26	1.31	0.202	0.213
	2		1.36		0.224	
D	1	S-2	4.44	4.35	0.712	0.701
	2		4.26		0.690	
E	1	S-2	3.10	2.94	0.497	0.470
	2		2.78		0.443	
F	1	S-2	2.50	2.64	0.400	0.411
	2		2.78		0.422	
G	1	S-2	1.93	1.87	0.314	0.303
	2		1.80		0.292	
H	1	S-2	1.79	1.67	0.292	0.271
	2		1.55		0.249	
I	1	S-2	1.14	1.02	0.185	0.164
	2		0.90		0.142	

Comparison Between ASTM C 156-80 and Tex-219-F Moisture Retention Test Results

The results from multiple moisture retention tests on six curing compounds using test methods ASTM C 156-80 and Tex-219-F are summarized in Table 9.9. Because CTR did not perform tests on curing compound M-2, comparisons do not include this type of compound. Both methods exhibited high variability. For the worst case measured using ASTM (S-1D) if normality is assumed, then 95 percent of the values could be within X - 1.96 Std Dev and X + 1.96 Std Dev (X signifies the mean value and Std Dev the standard deviation). This translates to values as low as $0.193 - 1.96 \times 0.092 = 0.013$ kg/m^2 and as high as $0.193 + 1.96 \times 0.092 = 0.373$ kg/m^2 could be obtained from this particular test. Similar results apply for the Texas method.

Texas results converted into ASTM units showed an average standard deviation of 0.049 kg/m^2, while ASTM results showed a value of 0.066 kg/m^2. In fact, Texas standard deviations were lower than ASTM values in all the tests. With respect to the amount of variability relative to the mean, Texas tests showed lower coefficients of variation (C. V.) in all compounds except in one test that exhibited almost equal coefficients in the two methods. The average Texas and ASTM C.V. from the five tests were 20.9 percent and 33.2 percent, respectively.

A statistical procedure using inferences for variances was used to determine the confidence level of variability between the two methods. Performing the "F" test for variances using ASTM and converted Texas values, it was determined that at a significance level of 5 percent one test showed a higher variability in the ASTM method and the other four showed no difference. When the significance level was increased to 10 percent, results were the same.

These results indicate that the ASTM method exhibited somewhat higher variability than the Texas method. Despite this, both methods showed a low level of repeatability, which could result in inaccuracies in moisture loss determination.

In order to detect any difference in the average moisture losses obtained from the two methods, or otherwise, if any of the methods resulted

TABLE 9.9. COMPARISON OF MOISTURE LOSS RESULTS IN ACCORDANCE WITH ASTM C 156-80 AND TEX-219-F.

72-Hour Moisture Loss	Compound Brand/Type						
	S-1D	S-2	P-1D	P-2	M-1D	M-2	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
C. V. (%)	47.7	23.4	33.2	31.3	30.5		33.2
Average Moisture Loss (Tex-219-F) Converted to ASTM (kg/m^2)	0.178	0.367	0.417	0.271	0.158	0.305	--
Std. Dev. (kg/m^2)	0.048	0.031	0.047	0.068	0.052	0.031	0.049
C.V. (%)	27.0	8.4	11.3	25.1	32.9	10.2	20.9
Average Moisture Loss (Tex-219-F) (%)	1.11	2.29	2.60	1.68	0.98	1.92	--
Std. Dev. (%)	0.29	0.20	0.30	0.42	0.33	0.19	0.31
C. V. (%)	26.1	8.7	11.5	25.0	33.7	9.9	21.0

Note: The average values do not include the results from M-2 curing compound.

in higher values, statistical tests of hypotheses and significance were employed. At a confidence level of 95 percent, it was determined that

(a) both methods gave equal values in two tests,
(b) the Texas method gave higher values in two tests, and
(c) the ASTM method gave a higher value in one test.

When the confidence level was increased to 99 percent, the results were the same.

Even though these results indicate that the Texas method produced somewhat higher values of moisture loss when converted to ASTM, they do not necessarily mean that the Texas method is stricter than ASTM in accepting a curing compound. To determine this, moisture losses from the two tests were expressed as a percentage of the maximum loss allowed by the two methods (Table 9.10). The percentage values allowed the comparison of the two methods without converting Texas results into ASTM units. Statistical analysis of these data determined that at a confidence level of 95 percent

(a) both methods gave equal values in two tests,
(b) the Texas method gave higher values in two tests, and
(c) the ASTM method gave higher values in one test.

When the level was increased to 99 percent, the results were the same.

It is evident that comparison of the losses expressed as a percentage of the maximum allowable did not change the results. It is therefore concluded that each method is equally likely to produce higher values than the other. Therefore, no general conclusion can be made on which method is stricter in accepting a curing compound.

The above results are based on average moisture losses and standard deviations calculated by considering all specimens tested in each compound as being a part of one test only. Therefore, the assumption was made that all specimens had been tested under identical conditions even though tests had been performed at different times. Such an assumption gave the ability to examine the variability among the various specimens without normalizing the results in each test, which decreases variability. However, the Texas and

TABLE 9.10. 72-HOUR 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	M-2
$\frac{\text{ASTM C 156-80}}{0.55} \times 100$ (%)	35.1	25.6	34.5	43.6	41.1	--
Standard Deviation (%)	16.7	6.0	11.5	13.6	12.5	--
$\frac{\text{Tex-219-F}}{4\%} \times 100$ (%)	27.8	57.3	65.0	42.0	24.5	48.0
Standard Deviation (%)	7.3	5.0	7.5	10.5	8.3	4.8

ASTM methods specify that a test should consist of a set of three or more specimens of a given curing material with the moisture loss being their average. Thus, using the central limit theory, average and standard deviation values were recomputed and the results were tabulated in Tables 9.1 through 9.6. Table 9.11 shows the comparison of the moisture loss results of the average test values using the two methods. Table 9.12 shows the losses obtained from the two methods expressed as a percentage of the maximum allowable.

It is evident from Table 9.11 that the ASTM test had a higher C.V. than the Texas test in four out of five tests. Statistical tests for variances cannot be employed in this case because of small sample sizes.

To determine which of the two test methods gave higher moisture losses, tests of hypotheses and significance for differences of means were employed which determined that at a confidence level of 95 percent

(a) both test methods gave equal values in three tests, and
(b) the Texas method gave higher values in two tests.

When the confidence level was increased to 99 percent the results did not change. Therefore, using average test values, the ASTM method did not give higher values than Texas as previously.

Moisture losses were expressed as before as a percentage of the maximum allowed by each test to determine which method was stricter in accepting a curing compound. Statistical tests for differences of means indicated that at a confidence level of 95 percent

(a) both methods gave equal values in two tests,
(b) the Texas method gave higher values in two tests, and
(c) the ASTM method gave higher values in one test.

The results were also the same at a confidence level of 99 percent.

It is evident that the averaging procedure of central limit theory gave about the same results as previously; either test method could give lower or higher moisture losses. Therefore, using one or the other averaging

TABLE 9.11. COMPARISON OF MOISTURE LOSS RESULTS (AVERAGE TEST VALUES) IN ACCORDANCE WITH ASTM C 156-80 AND TEX-219-F.

	Compound Brand/Type						
	S-1D	S-2	P-1D	P-2	M-1D	M-2	Average
Average Moisture Loss (ASTM C 156-80) (kg/m^2)	0.193	0.140	0.186	0.241	0.218	--	
Std. Dev. (kg/m^2)	0.053	0.023	0.036	0.043	0.040	--	0.039
C. V. (Percent)	27.5	16.4	19.4	17.8	18.3	--	19.9
Average Moisture Loss (Tex-219-F)	0.178	0.367	0.417	0.271	0.158	0.305	
Converted to ASTM (kg/m^2)	0.034	0.022	0.033	0.048	0.037	0.022	0.035
C. V. (Percent)	19.1	6.0	7.9	17.7	23.4	7.2	14.8
Average Moisture Loss (Tex-219-F)(percent)	1.11	2.29	2.60	1.68	0.98	1.92	
Std. Dev. (Percent)	0.21	0.14	0.21	0.30	0.23	0.13	0.22
C. V. (Percent)	18.9	6.1	8.1	17.9	23.5	6.8	14.9

Note: The average values do not include the results form M-2 curing compound.

TABLE 9.12. MOISTURE LOSS RESULTS (AVERAGE TEST VALUES) EXPRESSED AS A PERCENTAGE OF THE MAXIMUM ALLOWABLE LOSS.

	Compound Brand/Type					
	S-1D	S-2	P-1D	P-2	M-1D	M-2
$\frac{\text{ASTM C 156-80}}{0.55} \times 100$ (%)	35.1	25.5	33.8	43.8	39.6	--
Std. Dev. (Percent)	5.6	2.4	3.8	4.5	4.2	--
$\frac{\text{Tex 219-F}}{4\%} \times 100$ (%)	27.8	57.3	65.0	42.0	24.5	48.0
Std. Dev. (Percent)	3.7	2.5	3.7	5.3	4.1	2.3

procedure produces no definite trends and no conclusion can be made as to which test gives higher or stricter results. It was stated in Chapter 2 that the two test methods should not give any differences in moisture loss values because their two major differences, the percent solids determination and the expression of moisture loss, were not significant. However, the use of Type III cement was expected to reduce the variability in the test.

Tables 9.9 and 9.11 show a comparison of Tex-219-F moisture loss values converted into ASTM units of kg/m^2. The corresponding coefficients of variations for each compound are shown to be almost the same in all five compounds. This shows that expressing moisture loss in terms of the water present in a specimen at the time of application, which considers and corrects the effect of variability in the amount of water, does not produce any change in the results. This occurs for two reasons. First, the amount of water lost in 72 hours compared with the water present in a specimen at application is very small (normally their difference is less than 4 percent), and any change in water present in the specimen will cause a small effect; and second, the difference in the weight of water present in various specimens (which occurs due to different initial drying periods and/or different mortar weights) is not significant. Therefore, expressing moisture loss in terms of the variable water present instead of the fixed surface area does not produce any advantage.

The second major difference between the two test methods is the determination of the weight of solids contained in curing compounds, used in the correction for moisture loss. Table 9.13 shows a comparison of the weights obtained from the two procedures. Differences in weights ranged from zero to 1.1 grams, which correspond to ASTM moisture loss of up to 0.02 kg/m^2. This value compared to the maximum allowable loss of 0.55 kg/m^2 is insignificant.

No conclusion can be made as far as the effect of using one brand of cement on the variability of the tests, because no data were collected. However, using a Type III cement reduced the test run time because ASTM specimens, which were made of Type I cements, had to be left in the curing cabinet for initial curing for about 2 1/4 to 2 1/2 hours, which is 30 to 45 minutes more than the time specified by the Texas method.

TABLE 9.13. COMPARISON OF ASTM C 156-80 AND TEX-219-F SOLIDS WEIGHT RESULTS IN CURING COMPOUNDS

Compound Brand/Type	ASTM C 156-80 (gr.)	Tex-219-F (gr.)
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

The above results indicate that, of the three modifications of ASTM introduced by Tex-219-F, only two produced positive effects. The use of the water present in the specimen at the time of application for moisture loss determination does not appear to improve the test and it requires more weight recordings and calculations during the test which make the test more complicated and difficult to run. The other modification, the use of aluminum pans into which the compound is poured and not sprayed for solids determination, proved to be an accurate substitution of the ASTM procedure making the test less complicated and easier to perform.

Comparison Between ASTM C 156-80 and Tex 219-F Moisture Retention Test Results on Blind Samples

Moisture retention test results on blind samples in accordance with the ASTM and Texas methods are summarized in Table 9.14. The curing compound application rate was 180 sf/gal for both tests. Texas moisture losses which were obtained in 24 hours only, were converted as before into ASTM units. For comparison purposes, results from both methods were expressed as a percentage of their allowable losses by dividing Texas values by 2 percent (the allowable loss in 24 hours) and ASTM by 0.55 kg/m^2.

It is evident that moisture losses obtained using the Texas method were considerably higher than losses obtained using ASTM, even though the Texas results were reported in 24 hours. Five curing compounds of the nine tested failed the Texas test, two of them exceeding the allowable limit by more than 100 percent. None of the compounds, except one which was at limit, failed the ASTM test. Converted 24-hour Texas losses were higher in seven cases and lower in two than the 72-hour ASTM losses. However, the Texas results exhibited higher losses in all the tests when losses were expressed as a percentage of the maximum allowable.

Considering only the results on blind samples it can be concluded that the Texas test would reject more samples than the ASTM test in evaluating a curing compound. Results showed the 24-hour Texas moisture losses were higher than the 72-hour ASTM losses in all the tests. This contradicts the results from multiple moisture retention tests, which indicated that either

TABLE 9.14. 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)	$\frac{\text{Tex-219-F}}{2\%} \times 100$ (Percent)	$\frac{\text{ASTM C 156-80}}{0.55} \times 100$ (Percent)
A	4.65	0.748	0.450	233*	82
B	2.67	0.433	0.223	134*	41
C	1.31	0.213	0.208	66	38
D	4.35	0.701	0.552	218*	100
E	2.94	0.470	0.285	147*	52
F	2.64	0.411	0.290	132*	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

*Indicates curing compound did not pass the test.

method is equally likely to produce higher values than the other method. Equipment and procedures used in both multiple and blind sample tests were identical and therefore no reasonable explanation can be given to the exhibited trend.

Moisture Loss Results on Blank Specimens

The moisture loss of blank specimens (specimens with no curing compound) was investigated at CTR. A blank specimen was cast for every test run, each run consisting of three or four cured and one uncured specimen. Therefore, moisture losses of cured and blank specimens were determined under the same conditions. Two different brands of Type I cement, namely Atlas and Alamo, were used. Eleven blank specimens were made from Atlas cement and fourteen (including blank specimens for blind samples) were made from Alamo cement.

Table 9.15 shows the moisture loss results on blank mortar specimens using the two cement brands. Atlas cement showed an average loss of 2.500 kg/m^2 and a standard deviation of 0.299 kg/m^2, while the Alamo values were 2.239 kg/m^2 and 0.178 kg/m^2, respectively. Both cement brands showed a wide variability in moisture loss potential even though each mix had essentially the same flow characteristics and the same curing conditions. Looking at Atlas cement, moisture loss varied from 2.054 kg/m^2 to 2.997 kg/m^2, for a range of 0.943 kg/m^2 or 1.7 times the maximum allowable loss by the test. Alamo cement had moisture loss values of from 2.011 kg/m^2 to 2.538 kg/m^2, for a range of 0.527, almost equal to the maximum allowable loss.

Using tests for hypothesis and significance, it was determined that Atlas values had a higher variability than Alamo values at a 5 percent significance level.

These results indicate, as first suggested in Ref 4, that the variable moisture loss potential of the mortar specimens is a major contributor to the variability of this test. Clearly, when it is possible for two different mortar batches having the same mix proportions, same brand of cement, same flow, and same curing condition to have a difference in moisture loss of 0.943 kg/m^2 in 72 hours, the effect of the moisture retention capability of the same curing compound on these two mixes could be significantly different.

TABLE 9.15. MOISTURE LOSS RESULTS ON MORTAR SPECIMENS WITHOUT CURING COMPOUND (BLANK SPECIMENS)

72-Hour Moisture Loss (kg/m^2)			
Test Number	Atlas Cement	Test Number	Alamo Cement
S-1D-1	2.828	P-2-2	2.269
S-1D-2	2.548	P-2-3	2.344
S-1D-3	2.842	M-1D-1	2.538
S-1D-4	2.570	M-1D-2	2.376
S-2-1	2.997	M-1D-3	2.183
S-2-2	2.452	A	2.538
S-2-3	2.118	B	2.387
P-1D-1	2.054	C	2.237
P-1D-2	2.269	D	2.194
P-1D-3	2.387	E	2.022
P-2-1	2.430	F	2.065
		G	2.054
		H	2.129
		I	2.011
Average	2.500		2.239
Std. Dev.	0.299		0.178
C.V. (%)	12.0		7.9

Furthermore, it was shown that the level of moisture loss and the variability of the loss within a test is highly dependent on the cement brand. Table 9.16 shows a comparison of moisture loss on cured specimens cast with the two cement brands. Atlas specimens showed a higher C.V. as expected, because Atlas blank specimens had a higher variability in moisture loss potential.

However, in order to detect the exact effect of different brands of cement on moisture loss level and variability, moisture retention tests should be run on the same compound using the same test (either Texas or ASTM) and different cement brands.

One relatively easy way to remove much of the variability involved in different moisture losses between different specimen sets as suggested in Ref 4, is to base the effectiveness of a curing compound on how well it retains moisture when compared with the moisture loss on a blank set of specimens cast at the same time. Table 9.17 shows the moisture retention results for each set of specimens, based upon the loss of moisture on blank specimens. Table 9.18 shows the results on blind samples.

A comparison of the moisture loss results according to ASTM C 156-80 and the corresponding moisture retention results is shown in Table 9.19. Moisture loss results exhibited a coefficient of variation ranging from 16.4 percent to 27.5 percent, while the moisture retention C.V. ranged from 1.7 percent to 4.0 percent. The very low C.V. of the suggested procedure indicates that the method gives an effective way of removing the variability in the test. Of course, this means that blank specimens must be cast each time, but this does not add much to the required work because these specimens need not be treated with curing compound.

In order to find a minimum acceptable limit of the moisture retained in cured specimens for the suggested new test, the following procedure was employed. The mean 72-hour moisture losses of mortar specimen without curing compound made from Atlas and Alamo cement (see Table 9.15) was expressed at a 95 percent confidence level. The values obtained were 3.04 and 2.55 kg/m^2, respectively. Using the maximum allowable moisture loss of cured specimen in 72 hours (0.55 kg/m^2), the moisture retention was computed to be 82 and 78 percent, respectively (the procedure is the same as used in Table 9.15). The

TABLE 9.16. COMPARISON OF MOISTURE LOSS RESULTS ON MORTAR SPECIMENS OF ATLAS VERSUS ALAMO CEMENT

72-Hour Moisture Loss (ASTM C 156-80)							
Atlas Cement				Alamo Cement			
Test Number	Average (kg/m^2)	Std. Dev. (kg/m^2)	C. V. (%)	Test Number	Average (kg/m^2)	Std. Dev. (kg/m^2)	C. V. (%)
S-1D-1	0.285	0.059	20.7	P-2-2	0.318	0.043	13.5
S-1D-2	0.143	0.053	37.1	P-2-3	0.236	0.047	19.9
S-1D-3	0.254	0.041	16.1	M-1D-1	0.168	0.045	26.8
S-1D-4	0.090	0.016	17.8	M-1D-2	0.194	0.038	19.6
S-2-1	0.119	0.045	37.8	M-1D-3	0.293	0.039	13.3
S-2-2	0.151	0.031	20.5	A	0.450	0.069	15.3
S-2-3	0.150	0.026	17.3	B	0.223	0.053	23.8
P-1D-1	0.206	0.054	26.2	C	0.208	0.033	15.9
P-1D-2	0.130	0.033	25.4	D	0.552	0.050	9.1
P-1D-3	0.223	0.065	29.1	E	0.285	0.013	4.6
P-2-1	0.169	0.060	35.5	F	0.290	0.006	2.1
				G	0.223	0.041	18.4
				H	0.373	0.006	1.6
				I	0.189	0.025	13.2
Average C.V.			25.8				14.1

TABLE 9.17. MOISTURE RETAINED BY APPLICATION OF CURING COMPOUND (72-HOUR DATA)

Test Number	Moisture Loss Without Compound (kg/m^2) L1	ASTM C 156-80 Moisture Loss With Compound (kg/m^2) L2	Moisture Retention (L1 - L2) * 100/L1 (%)
S-1D-1	2.828	0.285	89.9
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
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
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
P-2-2	2.269	0.130	94.3
P-2-3	2.344	0.236	89.9
M-1D-1	2.538	0.168	93.4
M-1D-2	2.376	0.194	91.8
M-1D-3	2.183	0.293	86.6

TABLE 9.18. MOISTURE RETAINED BY APPLICATION OF CURING COMPOUND BLIND SAMPELS (72-HOUR DATA)

Sample No.	Moisture Loss Without Compound (kg/m^2) L1	ASTM C 156-80 Moisture Loss With Compound (kg/m^2) L2	Moisture Retention (L1 - L2) * 100/L1 (Percent)
A1	2.538	0.422	83.4
A2		0.400	84.2
A3		0.529	79.2
B1	2.403	0.284	88.2
B2		0.198	91.8
B3		0.187	92.2
C1	2.237	0.244	89.1
C2		0.180	92.0
C3		0.201	91.0
D1	2.194	0.523	76.2
D2		0.609	72.2
D3		0.523	76.2
E1	2.022	0.278	86.3
E2		0.278	86.3
E3		0.300	85.2
F1	2.065	0.286	86.2
F2		0.286	86.2
F3		0.297	85.6
G1	2.054	0.195	90.5
G2		0.270	86.9
G3		0.205	90.0
H1	2.129	0.369	82.7
H2		0.369	82.7
H3		0.380	82.2
I1	2.011	0.175	91.3
I2		0.218	89.2
I3		0.175	91.3

TABLE 9.19. COMPARISON OF THE VARIABILITY BETWEEN ASTM C 156-80 TEST RESULTS AND MOISTURE RETENTION CALCULATED FORM THE MOISTURE LOSS ON BLANK SPECIMENS.

			Moisture Loss ASTM C 156-80			Moisture Retention		
Test Number	Moisture Loss ASTM C 156-80 (kg/m^2)	Moisture Retention (Percent)	Average (kg/m^2)	Std. Dev. (kg/m^2)	C. V. (Percent)	Average (Percent)	Std. Dev. (Percent)	C. V. (Percent)
S-1D-1	0.285	89.9	0.193	0.053	27.5	93.2	3.0	3.2
S-1D-2	0.143	94.4						
S-1D-3	0.254	91.1						
S-1D-4	0.090	96.5						
S-2-1	0.119	96.0	0.140	0.023	16.4	94.2	1.6	1.7
S-2-2	0.151	93.8						
S-2-3	0.150	92.9						
P-1D-1	0.206	90.0	0.186	0.036	19.4	91.7	2.3	2.5
P-1D-2	0.130	94.3						
P-1D-3	0.223	90.7						
P-2-1	0.169	93.0	0.241	0.043	17.8	89.6	3.5	3.9
P-2-2	0.318	86.0						
P-2-3	0.236	89.9						
M-1D-1	0.168	93.4	0.218	0.040	18.3	90.6	3.6	4.0
M-1D-2	0.194	91.8						
M-1D-3	0.293	86.6						

minimum acceptable moisture retention is taken as the average of the two cement types, which is 80 percent.

THREE AND SIX-MONTH AGITATION TESTS

S-1D Curing Compound

Table 9.20 shows a 3-month solids test result of middle samples taken every 5 minutes of agitation. Results before mixing indicate that separation of the ingredients occurred because all the samples exhibited solids contents considerably less than the base line content. Separation was also detected with the stick, which showed a 9 to 10 inch thick brownish top layer and 8 inch thick cake at the bottom. After 5 minutes of mixing the solids content of the air-driven agitated drums increased considerably, but the electrically agitated drums did not change in solids content. As mixing time increased using the Type B agitator, the solids content increased and reached base line levels at 30 minutes. Drum samples from agitators C and D exceeded base line solids content at low mixing time and then decreased in solids content as mixing proceeded. These samples reached the base line values at 30 minutes, also. Electrically mixed drums did not change consistency and stayed at low solids content, averaging about 15 percent for a base line of 26 percent. Photographs of the surface of the drum's contents showed that the air driven agitators had thoroughly mixed the compound causing the brownish top layer to turn to pink, while the electrically driven agitators had not redistributed the top layer.

These results indicated that agitators B, C, and D produced a compound of homogeneous consistency at 30 minutes. Agitator A failed to reincorporate the separated top layer into the mix.

Solids test results for 6-month agitation are shown in Table 9.21. Top, middle, and bottom samples were obtained before mixing and at 30 and 40 minutes of mixing. Solids ranged from 55 to 57 percent in top, 16 to 21 percent in middle, and 6 to 16 percent in bottom samples. It is evident that the top layer contained most of the solids that seal the concrete surface and prevent water from evaporating. Therefore, it is the most critical

TABLE 9.20. 3-MONTH SOLIDS TEST RESULTS -- S-1D (PERCENT)

Drum No.	Agitation Method	Sample	Mixing Time (Minutes) 0	5	10	15	20	25	30
5	A	M	16.18	17.02	18.25	18.78	18.22	19.23	14.14
6	A	M	19.07	16.39	15.57	16.08	15.80	16.12	16.35
7	B	M	16.87	18.34	19.24	20.12	21.41	20.63	22.62
8	B	M	17.35	22.37	23.01	23.12	25.56	24.65	25.56
9	C	M	16.67	28.50	28.00	27.65	27.43	26.96	26.56
10	C	M	16.01	28.44	28.12	27.54	27.80	27.65	26.43
11	D	M	17.87	25.83	24.22	24.95	25.53	26.08	26.06
12	D	M	13.94	27.49	27.12	27.75	25.57	26.14	26.01

Note: Base Line Solids = 25.88 Percent

TABLE 9.21. 6-MONTH SOLIDS TEST RESULTS -- S-1D (PERCENT)

		0 Minutes Mixing Time			30 Minutes Mixing Time			40 Minutes Mixing Time		
Drum No.	Agitation Method	Sample T	Sample M	Sample B	Sample T	Sample M	Sample B	Sample T	Sample M	Sample B
5	A	57.01	15.75	10.20	55.95	14.95	13.97	56.47	14.90	14.71
6	A	--	16.31	12.72	57.17	14.64	13.12	54.11	16.02	15.44
7	B	55.93	19.69	12.93	25.15	25.80	24.13	25.80	25.82	24.30
8	B	--	21.24	8.33	26.04	25.80	23.90	24.70	25.62	24.95
9	C	--	18.66	6.40	26.30	26.72	17.37	25.43	26.50	12.13
10	C	--	18.68	10.05	26.93	27.28	11.81	26.31	26.77	20.43
11	D	--	19.31	12.54		25.82	25.98	25.32	25.51	25.59
12	D	--	17.01	15.82	26.11	26.02	26.33	26.13	26.23	25.91

Note: Base Line Solids = 25.88 Percent

ingredient, and its redispersion is essential to the curing quality. After mixing for 40 minutes, agitators B and D produced a uniform consistency in the three layers. Their solids content leveled to the base line content. Agitator C mixed thoroughly top and middle layers, but failed to reincorporate into the mix the bottom layer as evidenced by the solids content, which stayed at a low level. Agitator A did not mix any of the three levels and after 40 minutes of mixing, there was no change from the original condition of the compound.

Figure 9.1 shows 6-month visual test results. Three portions had separated during storage; a red top layer, a pink middle layer, and a bottom layer of water. Agitator A failed to remix any of the top layer which contained most of the solid particles. Agitators B and C produced uniform mixing. The samples of the compound taken from the middle and bottom layers were almost the same as the base line values, but samples from the top layer contained more pink portion than appropriate. Agitator D performed the best, producing a homogeneous consistency with all compound portions equal to base line proportions.

The results indicated that S-1D compound after 3 or 6 months storage could be remixed to a homogeneous consistency using air driven agitators. Both solids and visual tests showed that agitator D gave the best results. Solids test indicated that Agitators B and C gave a perfect mixing at the top, while the visual test showed that the top layer did not have the same compound proportions as the base line. In addition, the solids test showed considerably less solids in the bottom of drums no. 9 and 10 stirred with agitator C, while the visual test showed perfect mixing. Despite this, agitators B and C could be considered to give a good mixing. Both tests indicated that agitator A did not cause any mixing in drums no. 5 and 6.

S-2 Curing Compound

Table 9.22 shows 3-month solids test results of middle samples obtained every five minutes of mixing. Solids contents before mixing ranged from 37 to 40 percent, the base line being 39 percent. Mixing up to 30 minutes had the effect of changing the contents in each drum by less that 1.5 percent.

TABLE 9.22. 3-MONTH SOLIDS TEST RESULTS -- S-2 (PERCENT)

			Mixing Time (Minutes)						
Drum No.	Agitation Method	Sample	0	5	10	15	20	25	30
5	A	M	39.97	38.07	37.79	37.18	37.70	37.51	39.58
6	A	M	37.74	38.52	37.46	37.60	37.81	37.15	37.19
7	B	M	38.01	37.66	37.65	37.52	37.58	37.59	37.81
8	B	M	38.56	37.70	38.07	38.70	37.91	38.02	37.93
9	C	M	36.56	36.61	36.57	36.36	36.45	36.34	36.56
10	C	M	37.74	37.43	37.94	37.74	38.03	38.03	38.15
11	D	M	38.36	38.51	38.40	38.35	38.62	38.80	38.73
12	D	M	38.05	38.94	39.43	38.81	39.20	39.41	39.23

Note: Base Line Solids = 39.27 Percent

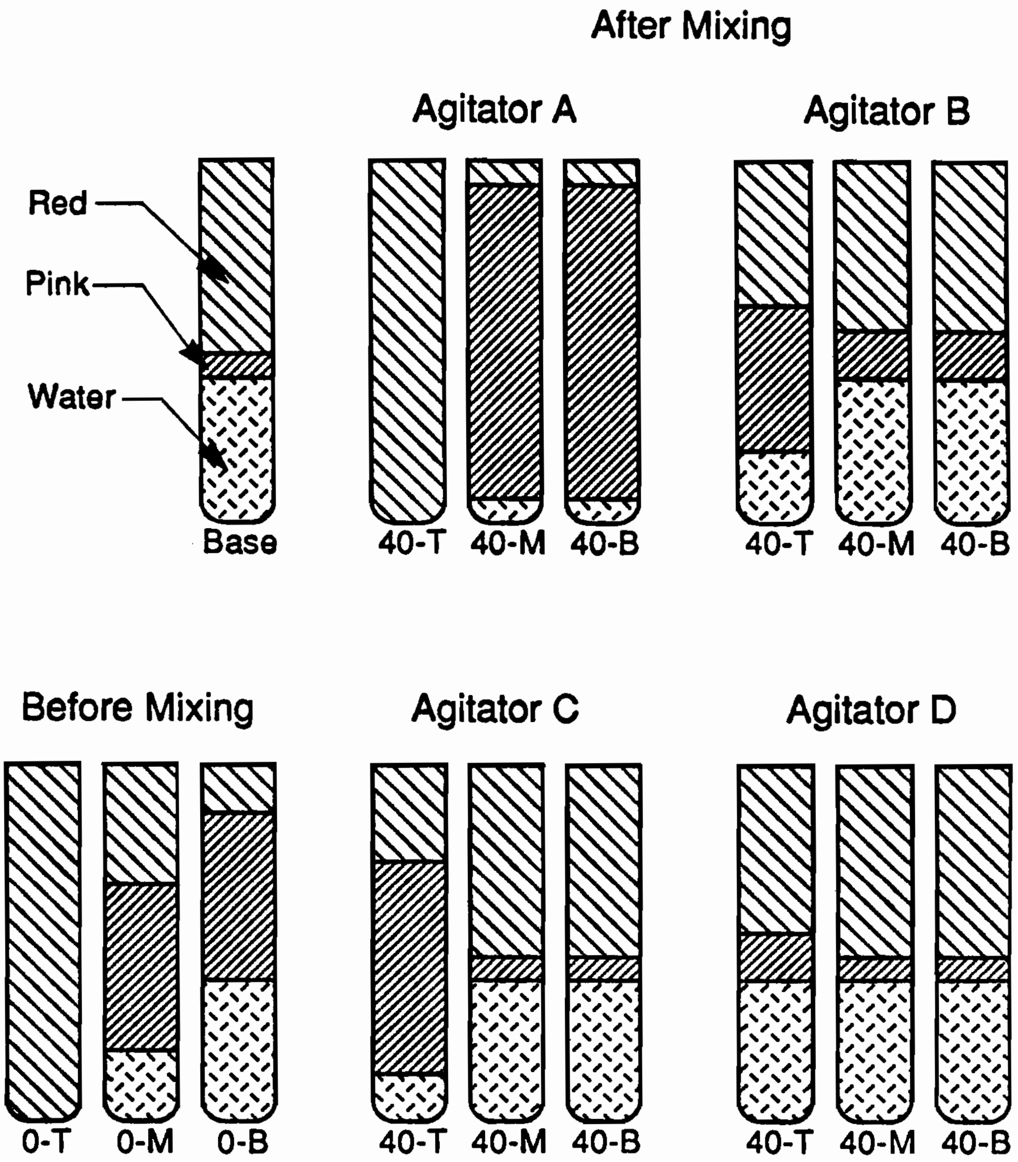


Fig 9.1. 6-Month visual test (S-1D).

Of course, these results show the mixing condition only at the middle of the drums and nothing can be said about the top and bottom positions. Comparing base line solids with contents before and after mixing, it can be concluded that a 3-month storage did not cause any separation, and thus mixing did not have any effect. Even though, examination of the ingredients with the stick showed that an 8 to 10 inch top layer of yellow resin had separated from the white pigment. After mixing, this layer disappeared completely only in drums no. 11 and 12 where type D agitator was used.

It was after 3-month tests on S-1D and S-2 curing compounds that a visual test was devised and photographs of drums before and after agitation were taken to obtain a more clear understanding of the mixing condition.

Table 9.23 shows mixing results on solids content after 6 months of drum storage. Mixing was performed for 20 minutes, and top, middle, and bottom samples were obtained before and after agitation. The top sample before mixing consisted only of yellow resin and exhibited very high solids content ranging from 54 to 58 percent. Middle samples ranged from 37 to 40 percent and were at the same solids level with 3-month results. Corresponding 3- and 6-month drums exhibited less than 1 percent solids difference. Bottom samples had 2 to 12 percent less solids than the middle samples from the same drum. After agitation drums No. 5 and 6 had the same solids at the three levels as before agitation, indicating that pigment and resin had not been reincorporated in the mix at all. The same happened with drums no. 9 and 10 mixed with agitator type C, except with bottom samples that showed considerably less solid after agitation. Agitator type D in drums no. 11 and 12 performed a fairly good mixing at the top and middle levels, but resuspension of settled pigment had not been achieved. Agitator type B mixed thoroughly the top layer lowering its solids content to the base line level and increasing the middle content from 1 percent below base line to 2 percent above. This occurred because the high solid content resin had been incorporated in the middle layer causing a rise in its content. The bottom layer solids content changed from 31 to 35 percent.

Photographic data before and after agitation showed that the top brown color disappeared when the drums were mixed with the air driven blades. An

TABLE 9.23. 6-MONTH SOLIDS TEST RESULTS -- S-2 (PERCENT)

Drum No.	Agitation Method	0 Minutes Mixing Time			20 Minutes Mixing Time		
		Sample T	Sample M	Sample B	Sample T	Sample M	Sample B
5	A	--	37.01	35.58	57.95	36.82	35.82
6	A	--	37.50	35.48	57.32	37.10	35.39
7	B	--	38.32	26.89	40.91	41.32	34.92
8	B	--	38.10	35.01	41.08	40.83	36.05
9	C	53.85	36.77	29.97	53.81	36.73	6.03
10	C	58.40	38.16	32.77	56.87	37.80	25.38
11	D	--	39.03	33.78	48.45	40.28	30.17
12	D	--	40.27	38.32	43.92	38.79	27.02

Note: Base Line Solids = 39.27 Percent

examination of portions of the compound that might have separated during storage showed a visual depth of 6 to 9 inches, which decreased by 1 to 2 inches in drums mixed with agitators A and C and disappeared completely with agitators B and D.

Visual inspection results are shown in Fig 9.2. Resin, pigment, and water were the major ingredients of the top, middle, and bottom levels before mixing, respectively. Agitators A and C performed the same (they showed almost no change from the condition before mixing), but C was able to reincorporate very small amounts of resin and pigment in middle and top layers, respectively. Agitator B mixed top and middle levels thoroughly and base line proportion were achieved. Bottom water was almost perfectly remixed, but no resin was incorporated. Agitator type D remixed to some extent top and middle layers (its performance was much better than A and C) but failed to change original condition in bottom.

Results from solids and visual tests are mostly compatible but in some cases there were some differences. Both showed that agitators A and C had not mixed compound at all and that agitators B and D performed a better mixing, with type B being the best. The tests indicated different bottom conditions on the same drums. Solids tests in some cases showed a very small difference in contents of bottom samples taken before and after agitation, while visual inspection showed a thorough mixing of the bottom layer. For example, a remarkable difference existed between bottom samples from drum no. 8 before and after mixing. Despite this, the solids test showed only a 1 percent difference between two samples. In addition, solids contents of middle and bottom samples differed slightly, even though the middle layer consisted almost completely of pigment and bottom layer of water. In such cases, solids tests results could be misleading and could indicate good mixing where there was no mixing at all. It is evident that sampling is required from the three levels in the give false results (like 3-month tests) if decision is based only upon these samples.

The main problem with this brand of compound was resuspension of the bottom cake. Only agitator B mixed the bottom layer to some extent and probably a longer agitation time could produce a homogeneous mix.

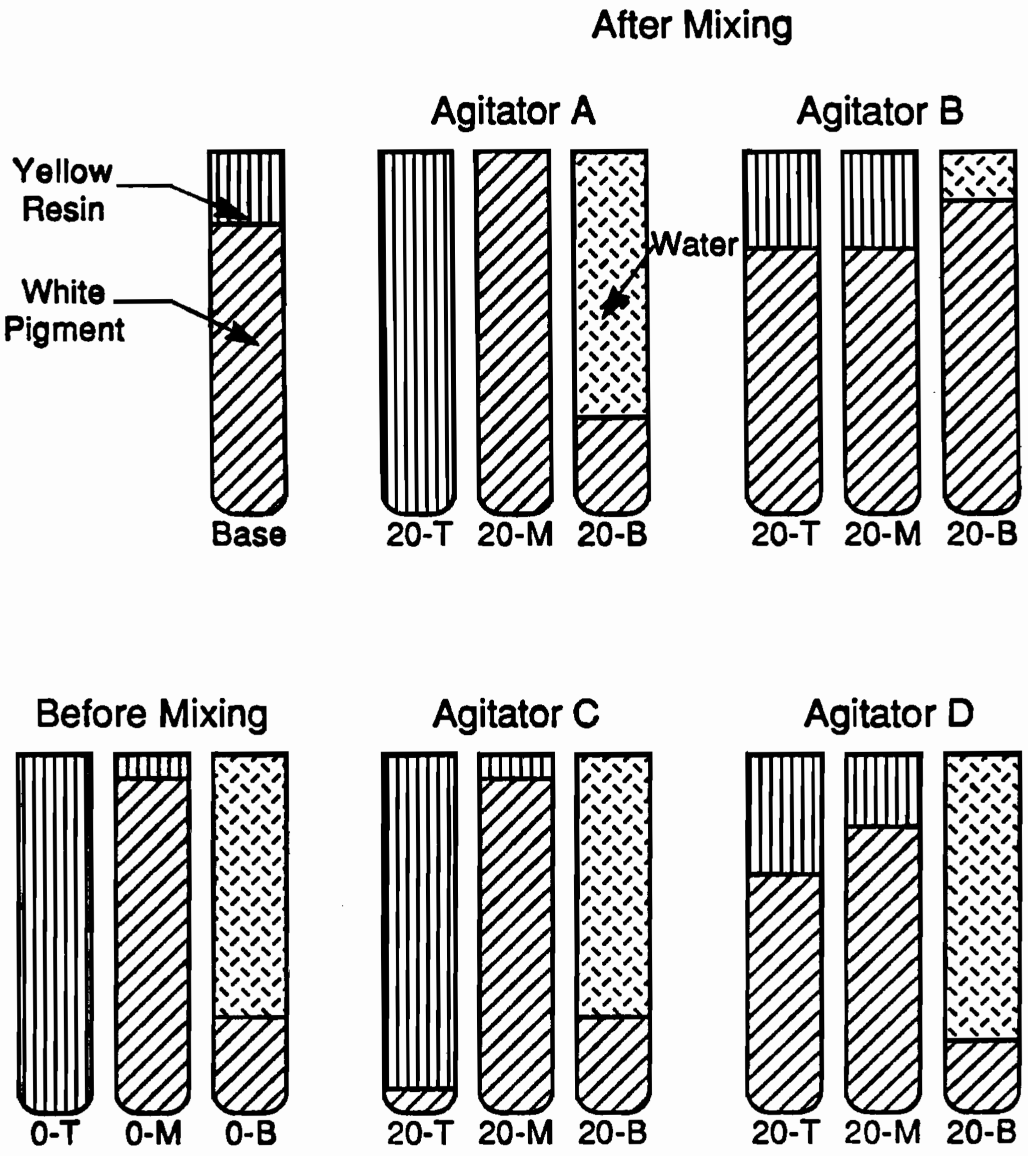


Fig 9.2. 6-Month visual test (S-2).

P-1D Curing Compound

Table 9.24 shows 3-month solids test results on Preston Pacific Type 1-D compound. Samples were taken every 10 minutes for a total mixing time of 40 minutes. All samples before agitation had solids contents ranging from 39.5 to 43.0 percent. Bottom samples from drum no. 12 was an exception, its content being 2.4 percent. The base line of the compounds was 43.77, therefore, all samples before mixing contained 0.5 to 3.5 less solids than base lines. Mixing up to 40 minutes did not cause any considerable change in compound consistency. The solids content of the bottom layer of drum no. 12 stayed at the same level as before mixing. These results had indicated that 40 minutes of mixing using any of the four methods did not cause any change in the solids content which stayed constantly below base line.

Examination of portions of compound ingredients showed that a 1/2 inch top layer had separated out during storage, but it remixed after agitation.

The visual test at 3 months showed no separation of portions or color differences and samples before or after mixing looked identical. Only bottom samples from drum no. 12 before and after agitation which contained water and traces of yellow pigment were different. This verified the low solids content indicated in the solids test.

Six-month solids test results are shown in Table 9.25. Mixing was performed only at 10 minutes because the above results indicated no agitation beyond this point. All contents before agitation were higher than the corresponding 3-month contents and ranged very close to base line. Mixing for 10 minutes changed the contents slightly, but changes did not follow a definite pattern. Top, middle, and bottom solids contents in each drum were about the same except from drum no. 12 which had again a very low content.

From 3- and 6-month tests it can be concluded that the four agitation devices gave the same quality of mixing.

Inspection of the drums before mixing indicated that a 1/4-inch crust had been developed in all of them. The crust remained unchanged when drums were mixed with types A, C, and D agitators, but it was chopped without complete dissolving when mixed with agitator B. This condition made the compound unusable after a storage period of 6 months.

TABLE 9.24. 3-MONTH SOLIDS TEST RESULTS -- P-1D (PERCENT)

		Mixing Time (Minutes)			Mixing Time (Minutes)			Mixing Time (Minutes)		
		0			10	20	30	40		
Drum No.	Agitation Method	Sample T	Sample M	Sample B	Sample M	Sample M	Sample M	Sample T	Sample M	Sample B
5	A	41.38	40.87	40.91	41.17	41.27	40.25	39.48	41.27	41.09
6	A	42.06	40.96	41.26	40.32	40.52	41.12	39.88	40.27	40.74
7	B	40.94	39.70	39.47	39.80	41.87	42.25	40.01	41.28	39.44
8	8	41.35	41.15	40.13	40.41	41.01	41.83	39.66	40.67	40.25
9	C	40.27	41.71	40.79	41.01	42.34	41.30	40.73	41.50	41.18
10	C	40.74	41.05	42.90	41.43	42.22	40.19	40.73	40.97	42.43
11	D	41.23	41.61	41.79	40.22	40.42	41.88	43.79	40.58	43.00
12	D	40.73	40.71	2.41	39.48	39.41	40.00	41.19	39.27	0.67

Note: Base Line Solids = 43.67 Percent

TABLE 9.25. 6-MONTH SOLIDS TEST RESULTS -- P-1D (PERCENT)

Drum No.	Agitation Method	0 Minutes Mixing Time			10 Minutes Mixing Time		
		Sample T	Sample M	Sample B	Sample T	Sample M	Sample B
5	A	42.82	42.81	43.13	42.17	43.63	41.80
6	A	43.60	43.28	42.17	44.02	46.84	43.19
7	B	44.63	42.98	43.33	43.75	44.51	42.51
8	B	44.29	45.57	43.50	41.68	42.14	43.29
9	C	44.26	42.19	41.31	46.45	44.92	43.08
10	C	43.40	41.72	42.44	45.05	44.70	42.86
11	D	42.72	44.46	42.42	43.63	44.02	42.62
12	D	44.53	39.00	7.04	44.04	41.03	8.56

Note: Base Line Solids = 43.67 Percent

Visual tests at 6 months again did not show any separation or color differences among samples, except from drum no. 12 in which the bottom consisted of water.

The results indicated that for this type of compound mixing quality is independent of the type of agitation. In addition, based on 3-month solids test results, it can be concluded that mixing was not required because it did not cause any change in solids level. The development of a solid crust at six months made the compound unusable.

P-2 Curing Compound

Table 9.26 shows a 3-month solids test results on brand P-2 curing compound. The top layer contained yellow resin in all drums, therefore for simplicity only one top sample was obtained. This had a very low solids content (22.5 percent), less than half the base line (46.5 percent). Middle and bottom layers had solids ranging from 45 to 60 percent. Mixing changed mainly the top and middle layer contents. The Type A agitator did not mix the compound well because the solids content of the top layer increased by only 2-3 percent and middle decreased by 7 percent. The solids content of the bottom layer decreased by 4 percent. The Type B agitator distributed the solids in the three layers to around 40 percent, except from drum no. 8, where the solids content of the bottom layer remained the same as before agitation. The Type C agitator changed top and middle solids to 34 percent, but bottom layer remained unchanged. Finally, the type D agitator levelled solids to 29 percent, but without changing the bottom layer solids content. It can be said that the air-driven agitators were able to mix the top and middle layers to a homogeneous consistency but one with less solids than the base line. The electric agitator did not produce the same homogeneous mixture. None of the four agitators, except type B in drum no. 7, could resuspend the cake at the bottom.

Examination of the extent of separation of compound layers indicated that a 9-inch yellow resin layer had been formed at the top during the 3-month storage period. This was completely incorporated in the compound when air driven agitators were used. The electric mixer was not able to dissolve

TABLE 9.26. 3-MONTH SOLIDS TEST RESULTS -- P-2 (PERCENT)

		Mixing Time (Minutes)			Mixing Time (Minutes)		Mixing Time (Minutes)		
		0			10	20	30		
Drum No.	Agitation Method	Sample T	Sample M	Sample B	Sample M	Sample M	Sample T	Sample M	Sample B
5	A	22.56	52.64	52.37	53.90	51.87	24.62	45.41	50.71
6	A	--	55.10	59.96	47.79	48.70	25.47	48.44	53.19
7	B	--	49.51	50.29	34.06	38.81	40.74	40.77	41.40
8	B	--	45.32	49.53	27.06	38.29	39.57	40.22	50.29
9	C	--	11.83	55.22	32.76	34.84	34.50	34.50	52.55
10	C	--	55.30	54.62	34.60	32.71	33.49	33.71	54.91
11	D	--	53.50	53.08	27.67	28.49	29.25	29.10	52.29
12	D	--	51.49	52.44	27.09	27.66	28.10	28.15	56.65

Note: Base Line Solids = 46.48 Percent

this layer. These results were verified by photographs taken before and after agitation. Furthermore, a 19-inch thick cake indicated separation of pigment at the bottom. This remained unchanged when agitators A, C, and D were used, but it decreased considerably when type B was used.

Results from the 3-month visual test are shown in Fig 9.3. The homogeneous consistency of top and middle layers produced by agitator types B, C, and D as shown in the solids test is also indicated by this test; top and middle layers had exactly the same amounts of resin and pigment. Uniformity was not achieved as shown with type A agitator, and this again verified solids test. The differences in pigment portions among results from the four agitators are clearly represented by the solids content. Pigment portions in samples were decreased in the order B, C, and D, and the same pattern was followed in the solids content.

The above results indicated that both solids and visual tests are good methods in determining mixing quality. Agitators A, D, C, and B gave progressively better mixing. All agitators failed to mix the bottom cake which could bring settled solids into upper layers and distribute the solids content in the three layers.

Six-month solids test results are shown in Table 9.27. Optimum mixing time was 30 minutes. Mixing again produced a homogeneous consistency only in the top and middle layers. Compared to 3-month results, the solids level in these two layers was slightly decreased and in the bottom layer was increased. This indicated further settling of solids. The wooden stick when it was removed from the bottom had 13 to 19 inches of cake, which meant that the cake was too firm to be reincorporated with the liquid during mixing.

Fig 9.4 shows 6-month visual test. Top and middle samples are almost identical to 3-month results, except that all bottom samples have no resin incorporated. In fact, bottom samples were too viscous to pour in the glass tubes.

Three-and six- month results indicated that for this type of compound, agitator B gave the best mixing results, with C, D, and A following, but none of the agitators was able to reincorporate with the liquid settled cake at the bottom, either at 3 or 6 months. It has also been shown that the mixing level of the four agitators after 3 or 6 months of storage was the same. In

TABLE 9.27. 6-MONTH SOLIDS TEST RESULTS -- P-2 (PERCENT)

Drum No.	Agitation Method	0 Minutes Mixing Time			30 Minutes Mixing Time		
		Sample T	Sample M	Sample B	Sample T	Sample M	Sample B
5	A	--	52.10	58.31	23.99	51.73	52.99
6	A	20.68	37.43	59.79	20.79	52.72	59.91
7	B	--	49.54	58.21	31.11	31.30	48.96
8	B	20.83	44.18	61.69	38.09	37.86	63.13
9	C	--	43.14	58.17	33.45	33.42	57.83
10	C	--	44.02	59.94	33.19	33.08	57.09
11	D	20.58	--	63.68	28.93	28.85	55.57
12	D	--	35.10	58.69	28.24	28.02	37.32

Note: Base Line Solids = 46.48 Percent

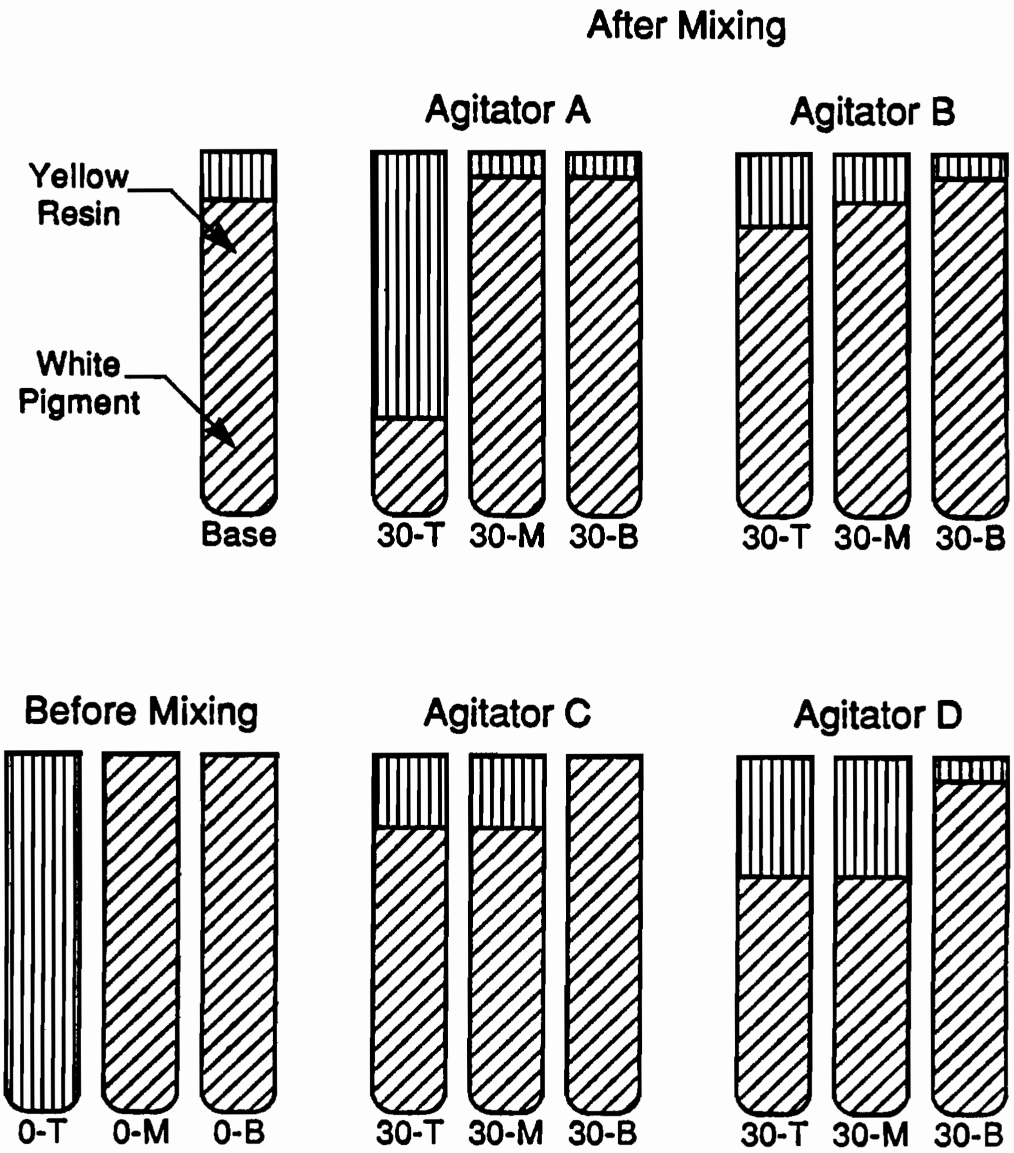


Fig 9.3. 3-Month visual test (P-2).

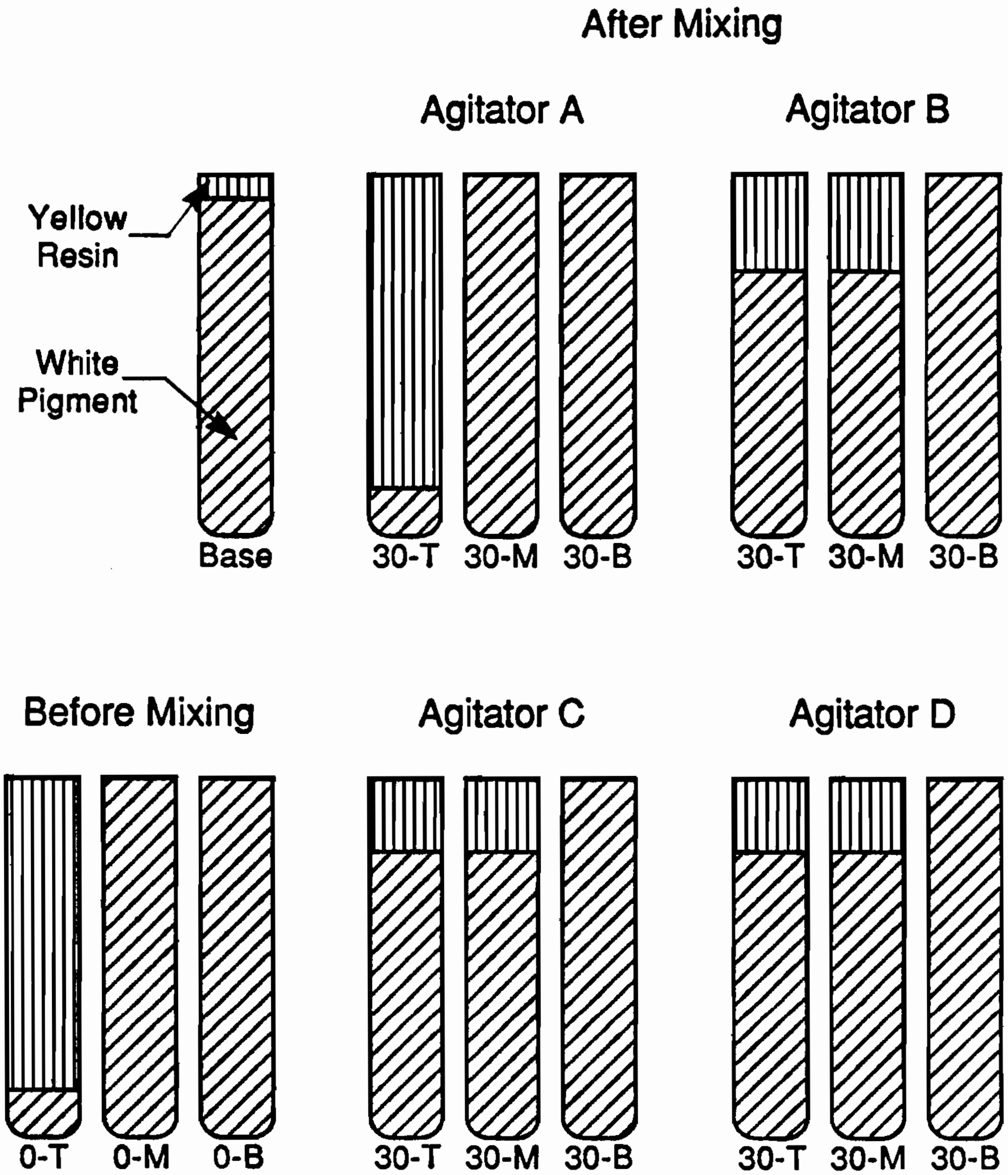


Fig 9.4. 6-Month visual test (P-2).

addition, both solids and visual tests can be good indicators of the mixing quality.

M-1D Curing Compound

Table 9.28 shows 3-month solids test results for top, middle, and bottom samples at zero and 40 minutes of agitation and middle samples at 10, 20, and 30 minutes. Top samples from all the drums had solids contents before mixing of more than 30 percent, while the bottom samples were clear water and had less than 1 percent solids. Middle samples averaged up to 22 percent of the base line content. Ten minutes of agitation using any of the four mixing devices was adequate to mix the top 1/2 to 1 inch of red resin and give exactly 22 percent solids in all the samples. Further mixing did not produce any change in content, and 40 minute samples from the three levels showed the same solids. The visual test was performed, but since all samples looked the same (except bottom samples before mixing which consisted only of water) results are not shown.

Because mixing for 10 minutes was sufficient, 6-month test was run for 10 minutes only. However, mixing using type A agitator was extended to 20 minutes. Table 9.29 shows 6-month results. All agitating methods gave perfect resuspension, even though separation occurred. Six month visual tests showed that in drums no. 6, 8, 11, and 12, water had not settled at the bottom, which indicated that 3-month mixing prevented settlement. Interestingly enough, agitator type C, which had earlier shown a good mixing performance, did not mix the water at the bottom of drum no. 7.

The above results indicated that 10 minutes of mixing using any of the four agitation devices was sufficient to produce a compound with homogeneous consistency. Perfect resuspension was achieved with drums stored for three and six months as well. The solids content and visual inspection tests gave exactly the same results, therefore they are not complementary, and any one can be adequate for evaluating the degree of settling and mixing.

TABLE 9.28. 3-MONTH SOLIDS TEST RESULTS -- M-1D (PERCENT)

		Mixing Time (Minutes)			Mixing Time (Minutes)			Mixing Time (Minutes)		
		0			10	20	30	40		
Drum No.	Agitation Method	Sample T	Sample M	Sample B	Sample M	Sample M	Sample M	Sample T	Sample M	Sample B
5	A	31.88	22.84	0.47	20.68	22.40	22.53	22.37	22.02	22.34
6	A	31.15	22.01	0.53	22.55	22.44	21.81	21.95	22.25	22.12
7	B	31.98	22.85	0.48	23.07	22.51	22.06	22.37	22.64	21.95
8	B	35.65	22.39	0.62	22.24	22.33	22.44	22.47	23.03	22.39
9	C	36.17	24.98	0.55	22.51	22.58	22.35	22.47	22.46	22.54
10	C	30.87	29.65	0.41	22.63	22.40	22.62	22.41	28.27	17.91
11	D	24.07	22.56	14.62	22.09	22.44	22.08	22.32	22.08	22.57
12	D	31.99	29.48	0.67	22.35	22.42	21.76	22.40	22.32	22.24

Note: Base Line Solids = 22.39 Percent

TABLE 9.29. 6-MONTH SOLIDS TEST RESULTS -- M-1D (PERCENT)

		0 Minutes Mixing Time			10 Minutes Mixing Time		
Drum No.	Agitation Method	Sample T	Sample M	Sample B	Sample T	Sample M	Sample B
5	A	22.22	22.39	20.81	22.49*	22.77*	22.54*
6	A	24.36	23.50	21.49	22.58*	22.75*	22.45*
7	B	37.08	31.31	0.51	25.12	25.22	0.43
8	B	39.31	22.72	19.54	22.66	22.59	22.04
9	C	33.69	32.14	0.46	22.59	22.78	22.77
10	C	36.28	31.31	0.76	22.31	22.79	23.00
11	D	36.48	22.64	20.33	22.61	22.72	22.61
12	D	33.68	22.56	20.46	22.47	22.59	22.69

Note: Base Line Solids = 22.39 Percent

* (indicates 20 minutes mixing time)

M-2 Curing Compound

Three-month solids test results on brand M-2 curing compound are shown in Table 9.30. Samples were obtained from the three levels at zero and 30 minutes of mixing, and from the middle at 10 and 20 minutes. Top samples before mixing were not obtained because the top layer was extended to the middle of the drum and samples from top and middle positions would be the same. Solids content of middle samples ranged between 43 and 47 percent. The base line solids content was 41.5 percent. Bottom samples exhibited a wider range and varied between 39 and 50 percent. While all middle samples showed higher solids contents than the base line, half of the bottom samples had higher and half had lower solids than base line sample. After mixing for 10 minutes, the solids content dropped very close to base line. Mixing for an additional 10 minutes did not produce any changes. Samples from top, middle, and bottom positions at 30 minutes of mixing did not show high variations and ranges around base line. Different agitators did not show a definite solids pattern and all of them gave approximately the same solids contents.

Photographs before and after mixing showed that the top dark brown color in drums no. 5 and 6 turned into light brown indicating that some of the white pigment mixed with top layer. In all the other drums, the top color changed to white, indicating complete remixing of top layer. These results are verified with the stick, which showed disappearance of top layer in the same drum.

Results from 3-month visual tests are shown in Fig 9.5. The top layer before mixing consisted completely of yellow resin, the middle layer of white pigment and some resin, and the bottom layer of white pigment and water. Agitator type A could only reincorporate part of the pigment in the top layer, but it mixed thoroughly the middle of the drum. Water at the bottom dissolved but resin was not resuspended. Agitators B and D performed in a similar manner. They gave a fairly good mixing at top and middle positions but failed to agitate the bottom pigment. Agitator type C in drums no. 9 and 10 performed the best agitation; it remixed more resin than the other agitators in top and middle positions and some in the bottom.

TABLE 9.30. 3-MONTH SOLIDS TEST RESULTS -- M-2 (PERCENT)

Drum No.	Agitation Method	Mixing Time (Minutes)			Mixing Time (Minutes)		Mixing Time (Minutes)		
		0			10	20	30		
		Sample T	Sample M	Sample B	Sample M	Sample M	Sample T	Sample M	Sample B
5	A	N/A	42.55	43.28	37.19	42.13	41.81	46.14	43.79
6	A	N/A	44.04	40.66	41.37	48.07	43.09	46.06	43.00
7	B	N/A	46.97	50.33	41.24	40.71	40.30	42.53	48.74
8	B	N/A	46.47	40.23	41.07	42.91	42.60	40.93	41.55
9	C	N/A	45.19	38.62	40.97	41.03	40.25	41.73	42.24
10	C	N/A	42.77	44.65	42.55	40.56	41.06	43.50	43.29
11	D	N/A	44.79	[illegible].43	40.53	41.23	40.30	40.39	40.31
12	D	N/A	44.06	38.94	38.76	39.75	39.52	38.98	40.24

Note: Base Line Solids = 41.51 Percent

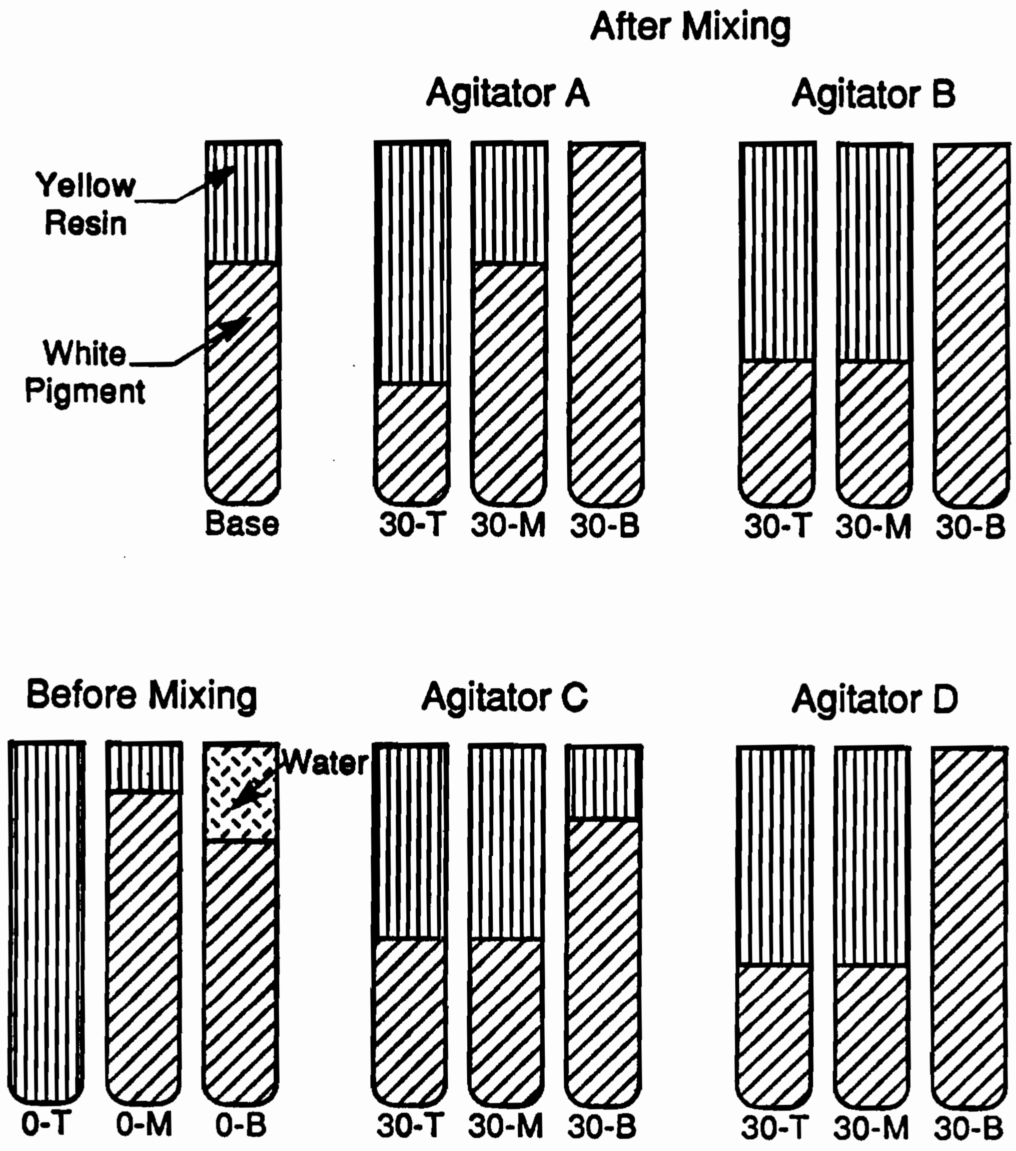


Fig 9.5. 3-Month visual test (M-2).

Table 9.31 shows 6-month solids test results. Optimum agitation time was set at 20 minutes because 3-month test showed no further change in solids content after this mixing time.

Solids content of top samples at zero time were all equal to the base line content. Middle samples exhibited somewhat higher values. Seven drums had 1.5 to 3.0 percent more solids than base line, but two had considerably less. Solids were at the same level with the corresponding three-month results except from the two drums. Bottom samples exhibited very high variation in solids content, ranging from 32 to 62 percent. Mixing did not change considerably the solids content of the top layer, but it lowered the middle layer to base line level. Bottom samples in all drums was reduced by 2 to 16 percent, except from no. 5 and 6, where it remained the same.

Comparing 3- and 6-month solids results, very slight differences could be seen between top and middle samples from corresponding drums. But all of the 6-month bottom samples had lower solids contents than the 3-month samples.

Three-month agitation did not produce any difference in solids content among the four agitation methods. Therefore, it can be concluded that all agitation methods produced the same mixing. There is an exception in the middle samples from drums no. 5 and 6, which produced higher solids contents than the other drums. Top and middle contents of 6-month agitation were the same for all agitators, but bottom contents of agitators C and D were much less than the other two. From these results it can be concluded that agitator type B gave the best mixing with types A, D, and C following.

Examination of the extent of separation of the compound showed that the 16-inch top layer only in drums no. 5 and 6 did not dissolve after mixing. Also, the settled pigment in drums 7 through 12 reduced from about 19 inches to 13 inches, but in drums 5 and 6 it remained exactly the same. This shows that agitator type A did not mix the layers at all, which contradicts solids results that indicated agitator A as being the second best. Figure 9.6 shows 6-month visual test results. Top and middle samples before mixing had the same proportions as the 3-month samples, but the bottom sample had much more water than the corresponding 3-month sample. This indicated that the 3-month mixing did not prevent any further separation. Agitator A resuspended

TABLE 9.31 6-MONTH SOLIDS TEST RESULTS -- M-2 (PERCENT)

Drum No.	Agitation Method	0 Minutes Mixing Time			20 Minutes Mixing Time		
		Sample T	Sample M	Sample B	Sample T	Sample M	Sample B
5	A	42.33	43.17	40.11	42.08	42.19	40.92
6	A	42.84	44.77	31.73	43.53	40.68	32.59
7	B	41.24	44.64	62.30	41.24	41.48	46.17
8	B	41.74	31.77	45.85	42.78	41.80	41.16
9	C	41.79	43.57	27.77	42.03	41.86	25.98
10	C	41.43	44.94	32.00	41.85	41.23	28.52
11	D	41.83	38.85	44.16	42.36	42.48	36.59
12	D	40.71	42.18	35.34	39.49	36.89	30.62

Note: Base Line Solids = 41.51 Percent

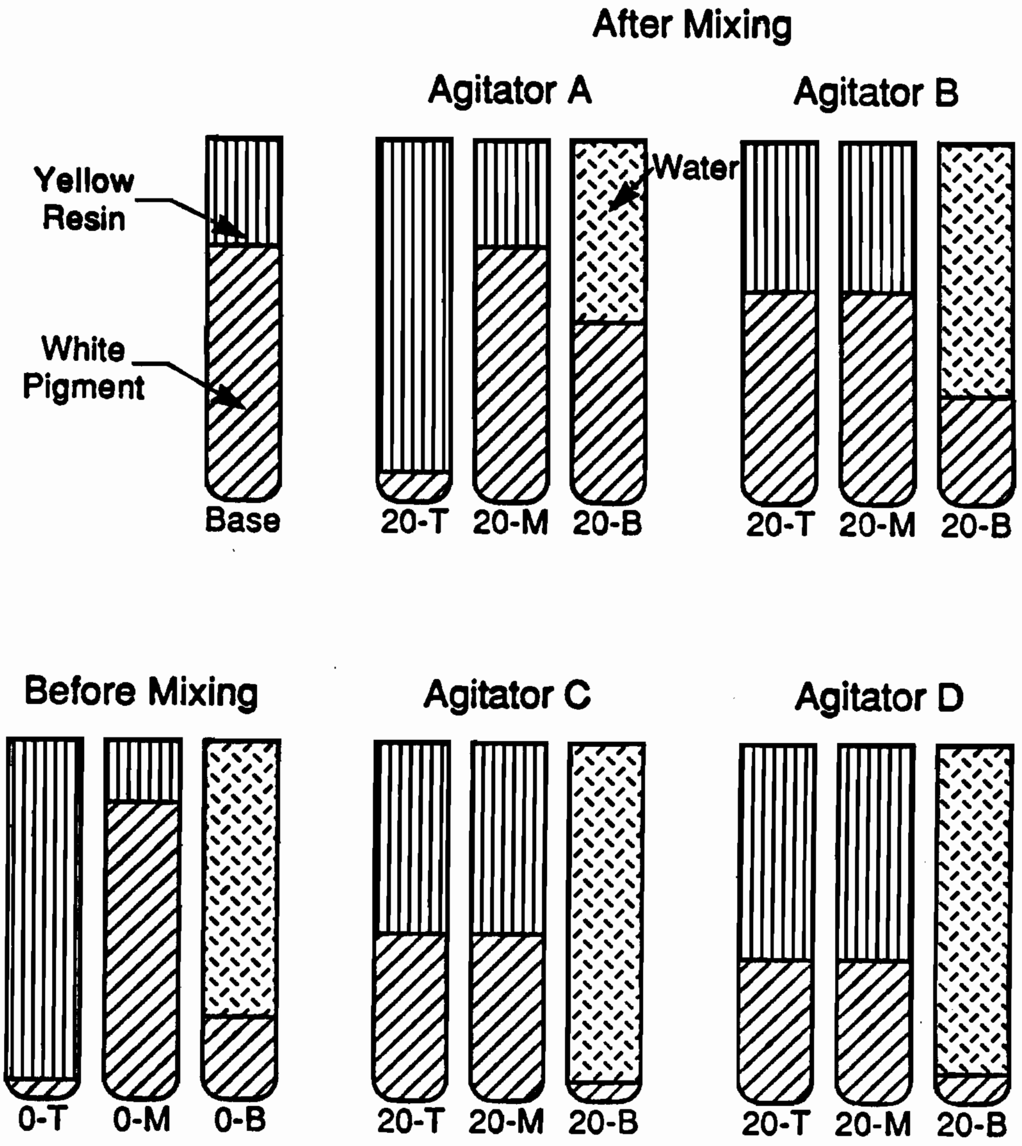


Fig 9.6. 6-Month visual test (M-2).

only a small fraction of pigment in the top layer and a relatively larger portion of resin in the middle layer, but it could not distribute the water in the bottom layer. These results compared to the 3-month results shows that agitator A could not redissolve portions that separated further after 3-month mixing. The same happened with agitator B, which produced less agitation than in the 3-month test. Portions of pigment and resin were almost the same in the top and middle samples at 3- and 6-month tests when mixed with agitators C and D, but bottom samples consisted almost of water.

The above results indicated that a greater separation of compound portions occurred after 3-months of storage and that mixing quality at six months was lower for all the agitator types. The best mixing was achieved by agitator B. Types C and D gave almost the same results. Type A did not produce any mixing. This contradicts solids test results which indicated agitator A as the second best. In addition, solids test indicated perfect resuspension of top and middle positions for all the agitators because results gave solids contents equal to base line. But as the visual test showed, this was not true because all samples had higher portions of resin and less pigment. The reason that the solids test gave misleading results might be the fact that resin and pigment contained the same amounts of solids. In such cases, conclusions derived from solids can only give false results.

Comparison of Moisture Retention Ability of Curing Compounds and Solids Content

Table 9.32 shows a comparison of moisture loss of treated concrete and solids content of curing compounds. As indicated, there is no relationship between the two. For example, curing compounds S-1D and P-1D had about the same moisture loss according to the ASTM test, but the solids content of P-1D was twice that of S-1D, even though solids content of P-1D was much higher.

TABLE 9.32. COMPARISON OF MOISTURE RETENTION ABILITY OF CURING COMPOUNDS WITH SOLIDS CONTENT.

	72-Hour Moisture Loss		
Compound Brand/ Type	ASTM C 156-80 (kg/m^2)	Tex 219-F (Percent)	Solids Content (Percent)
S-1D	0.193	1.11	25.6
P-1D	0.190	2.60	43.7
M-1D	0.226	0.98	22.4
S-2	0.141	2.29	39.3
P-2	0.240	1.68	46.5
M-2	---	1.92	41.5

NEW MOISTURE RETENTION TEST

The feasibility of developing a moisture retention test using a permeable membrane instead of PCC to evaluate a curing compound was examined in this study. The key concept was to obtain a membrane with a similar permeability to the initial rate of evaporation of normal concrete. A total of 23 membranes (12 fabric cloths, 10 polymeric membranes and one paper towel) were tested and their permeabilities were determined. Table 9.33 lists the fabrics and membranes used. Figures 9.7 to 9.9 show the permeability rates of fabrics 1 through 12. For comparison purposes, the evaporation rates of normal concrete and a pan of water were also plotted. All the fabrics exhibited near similar permeability rates and lower than the initial rate of evaporation of concrete. As expected, the rate of water lost through the membranes was constant. Figures 9.10 to 9.12 show the permeability rates of Celanese membrane films along with the evaporation rates of normal concrete and water. These membranes also exhibited lower permeability rates than the initial evaporation of concrete.

Since none of the membranes exhibited a permeability similar to the initial rate of evaporation of concrete, preliminary tests were performed using fabrics 2, 3, and 12 cured with P-1D curing compound at a rate of 180 sf/gal, to examine their behavior. As shown in Fig 9.13, the treated fabrics exhibited the same permeability as the untreated ones, which indicates that the curing compound did not seal the voids in the cloths, but it accumulated on the fibers. In addition, Fig 9.13 shows a treated and untreated common paper towel. The treated paper exhibited about the same permeability as cured concrete in ten to twelve hours. The ten Celanese membranes were also treated with P-1D curing compound. The compound sealed completely the surface of nine membranes and let no water evaporate through. However, the microporous film K-442 showed some working potential.

To examine the working potential of membrane K-442, a scale of very high precision (0.01 gr) was employed to perform further testing. Figures 9.14 to 9.17 show the results from four multiple moisture retention tests using the K-442 film treated with P-1D curing compound. The moisture loss of concrete in 72 hours treated with P-1D curing compound tested according to ASTM C 156-

TABLE 9.33. MEMBRANES USED FOR THE NEW MOISTURE RETENTION TEST

(a) Fabric Cloths

Material No.	Material Type	Specification No.
1	100% Polyester Craftfelt	1-64 SAN
2	100% Cotton Muslin (Bleached)	4-47 KR #406
3	50% Polyester, 50% Muslin	3/VL 1-82 SSP
4	100% Cotton Canvas	4-EV 273 SPE
5	100% Cotton Canvas	4/DVU 1-50 SKR
6	100% Cotton Canvas	4/DVU 1-50 SEE
7	100% Cotton Canvas	3/DEO 1-18 PNE
8	100% Cotton 6 oz. Denim	152 PLR #10400
9	100% Cotton Denim	4-DTU 71-SAN
10	65% Polyester, 35% Cotton Broadcloth	4-32 SPI
11	100% Dacron Polyester	676 SSP
12	100% Polyester	7-14 RK

(b) Celanese Membranes (Celgard Products)

Material No.	Specification Number
1	2400
2	2402
3	3500
4	3401
5	K-442
6	K-443
7	4400
8	4510
9	5511
10	5550

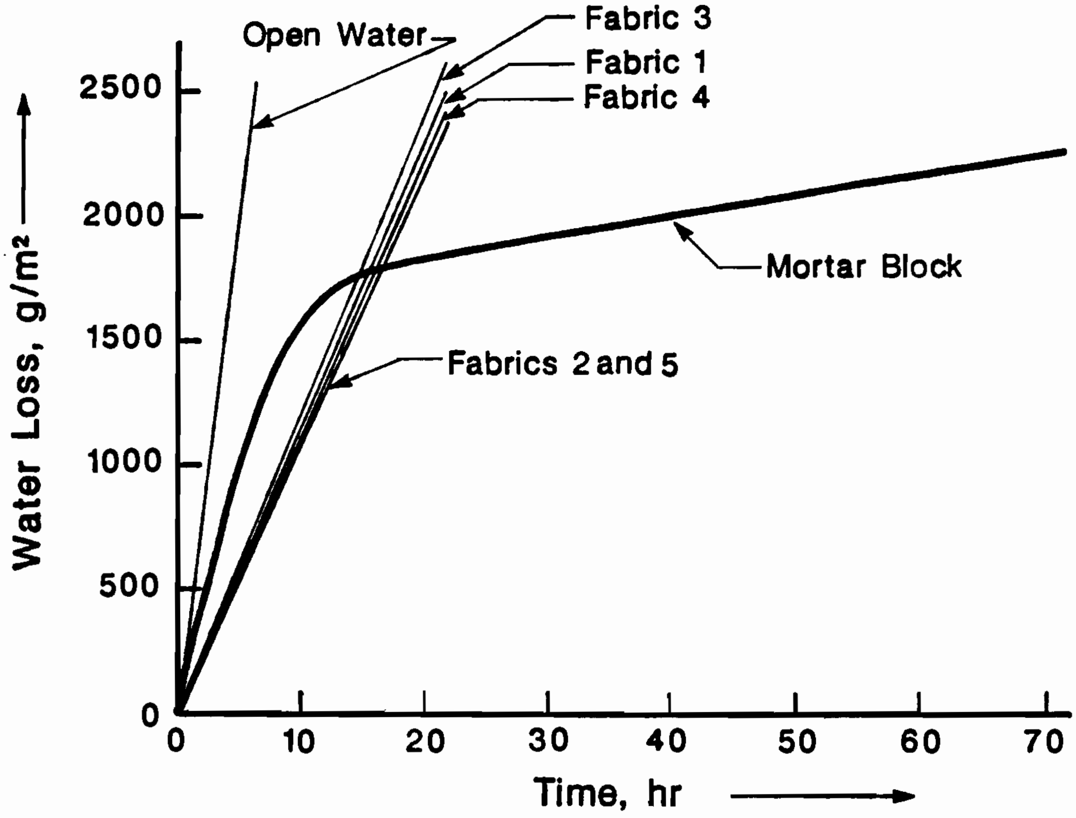


Fig.9.7. Permeability of fabric cloths Nos. 1 through 5.

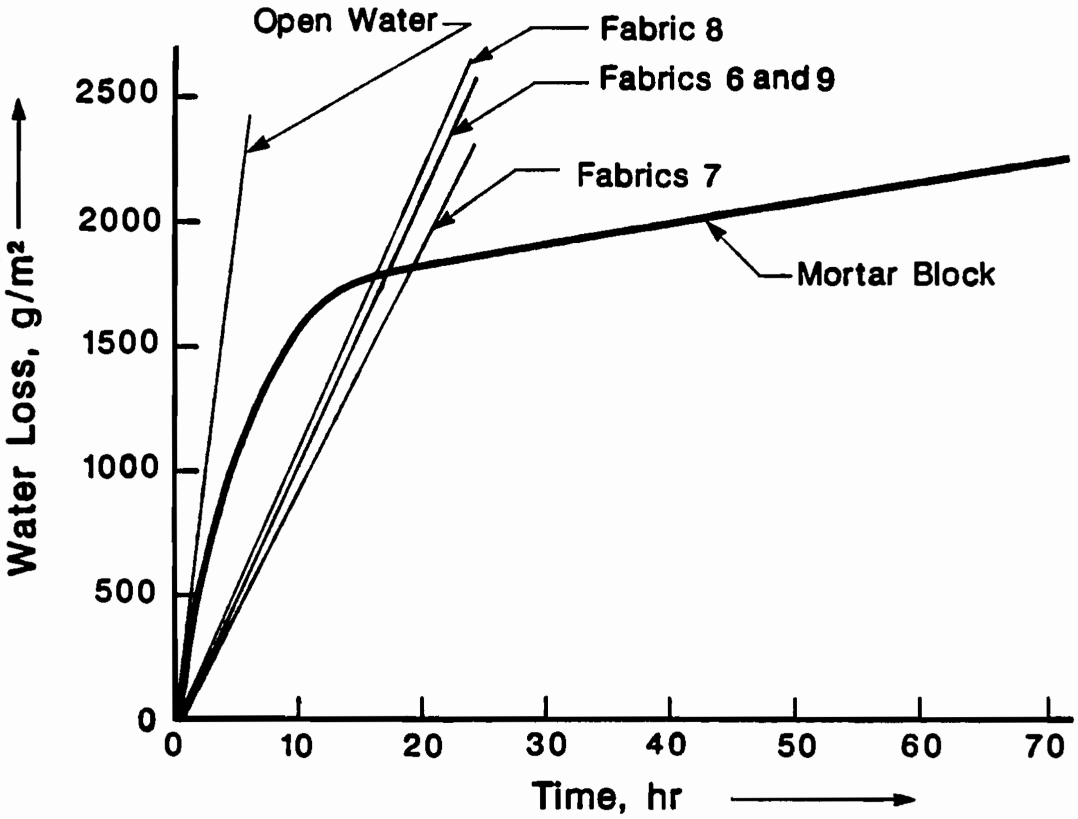


Fig 9.8. Permeability rates of fabric cloths Nos. 6 through 9.

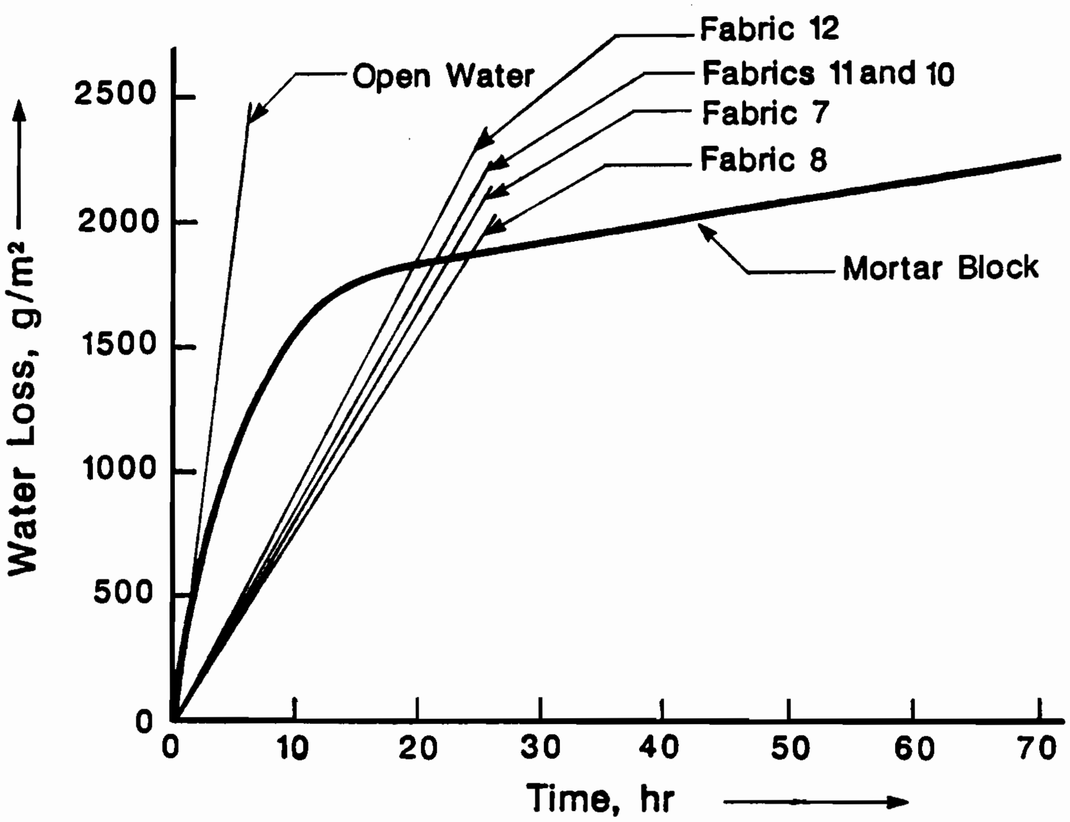


Fig 9.9. Permeability of fabric cloths Nos. 7 through 12.

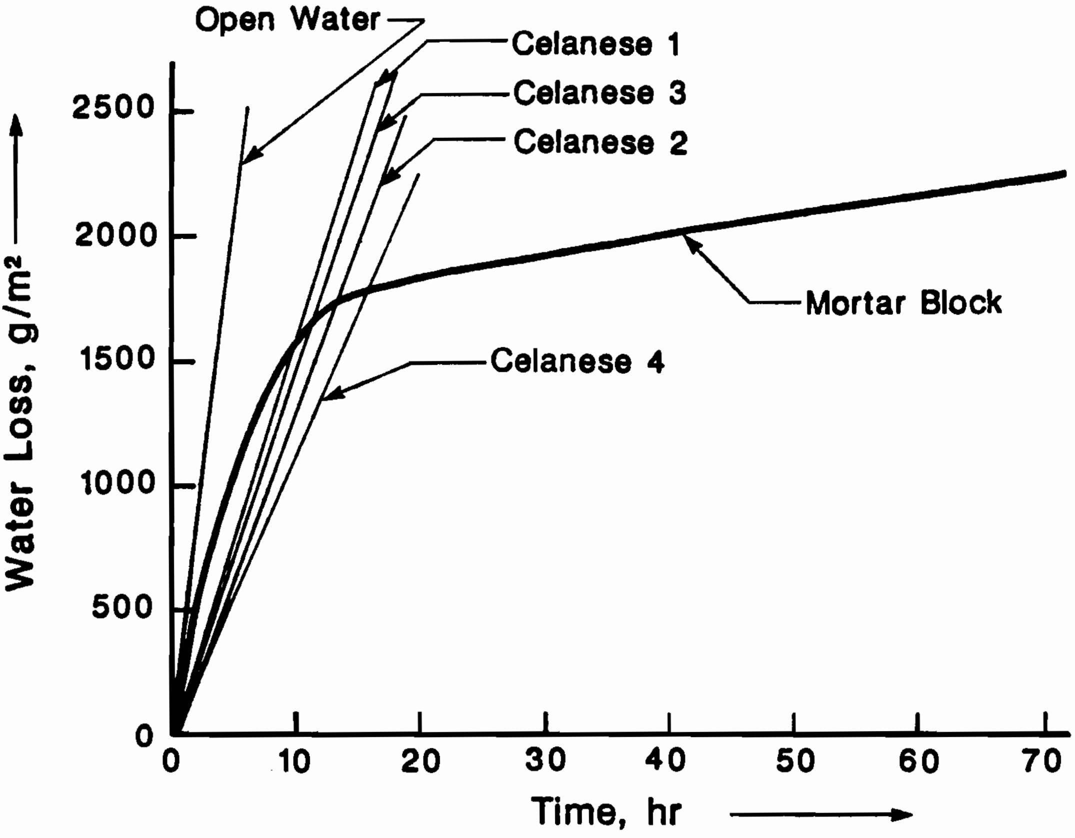


Fig 9.10. Permeability rates of Celanese membranes Nos. 1 through 4.

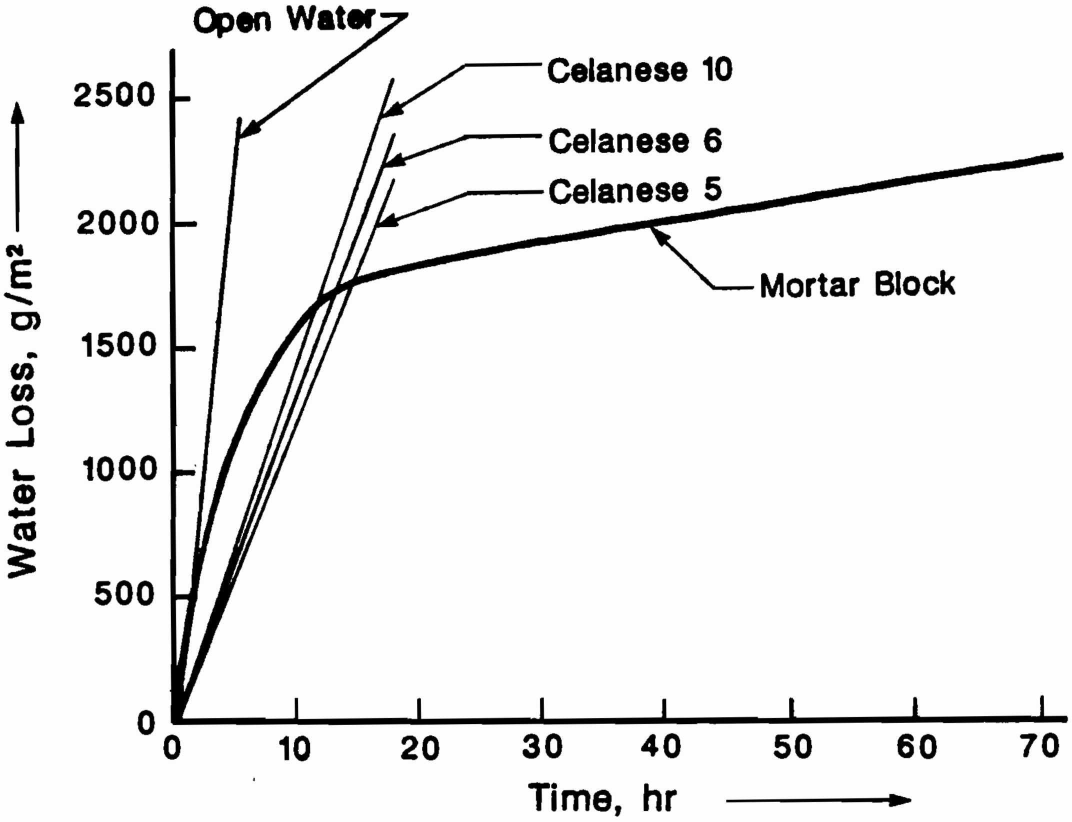


Fig 9.11. Permeability rates of Celanese membranes Nos. 5, 6, and 7.

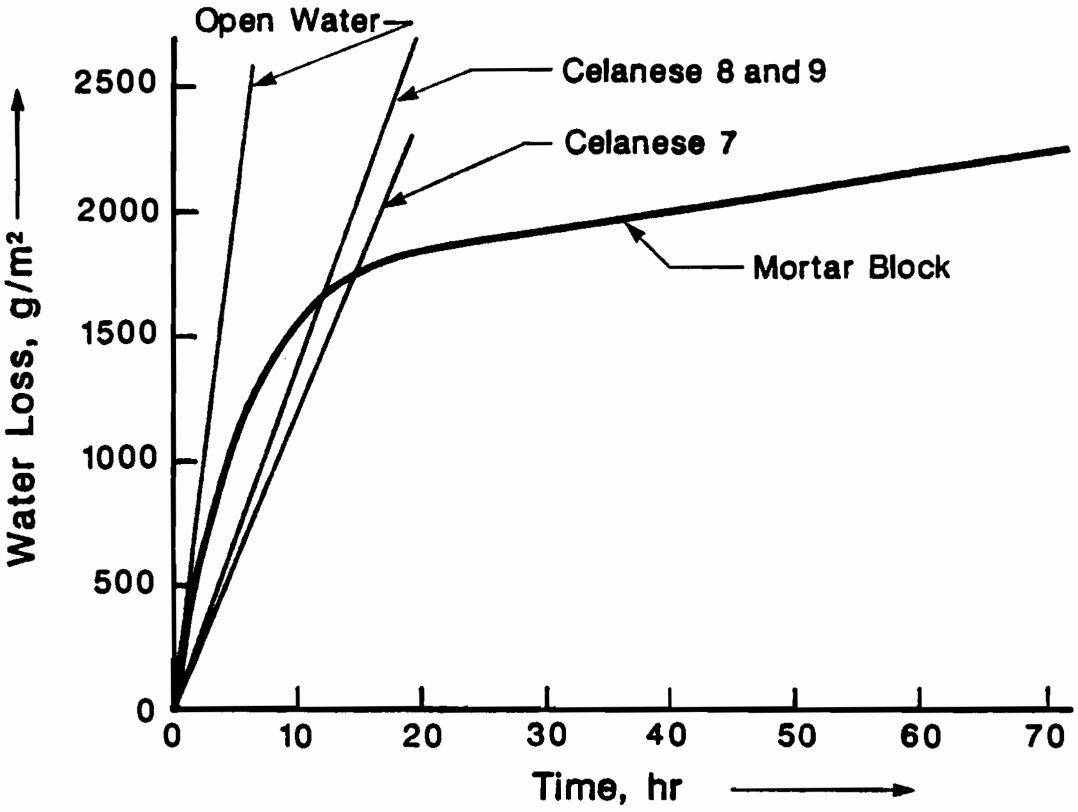


Fig 9.12. Permeability rates of Celanese membranes Nos. 7 through 9.

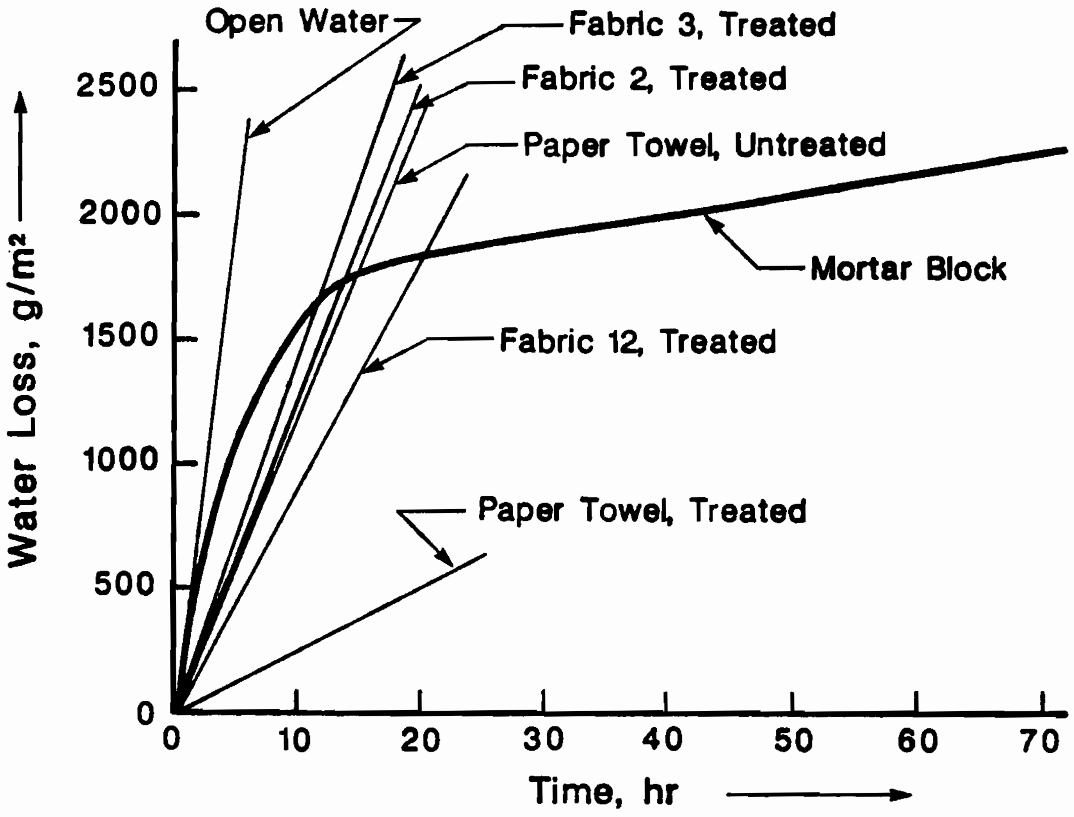


Fig 9.13. Permeability rates of treated fabric cloths Nos. 2, 3, and 12, and treated and untreated paper towel.

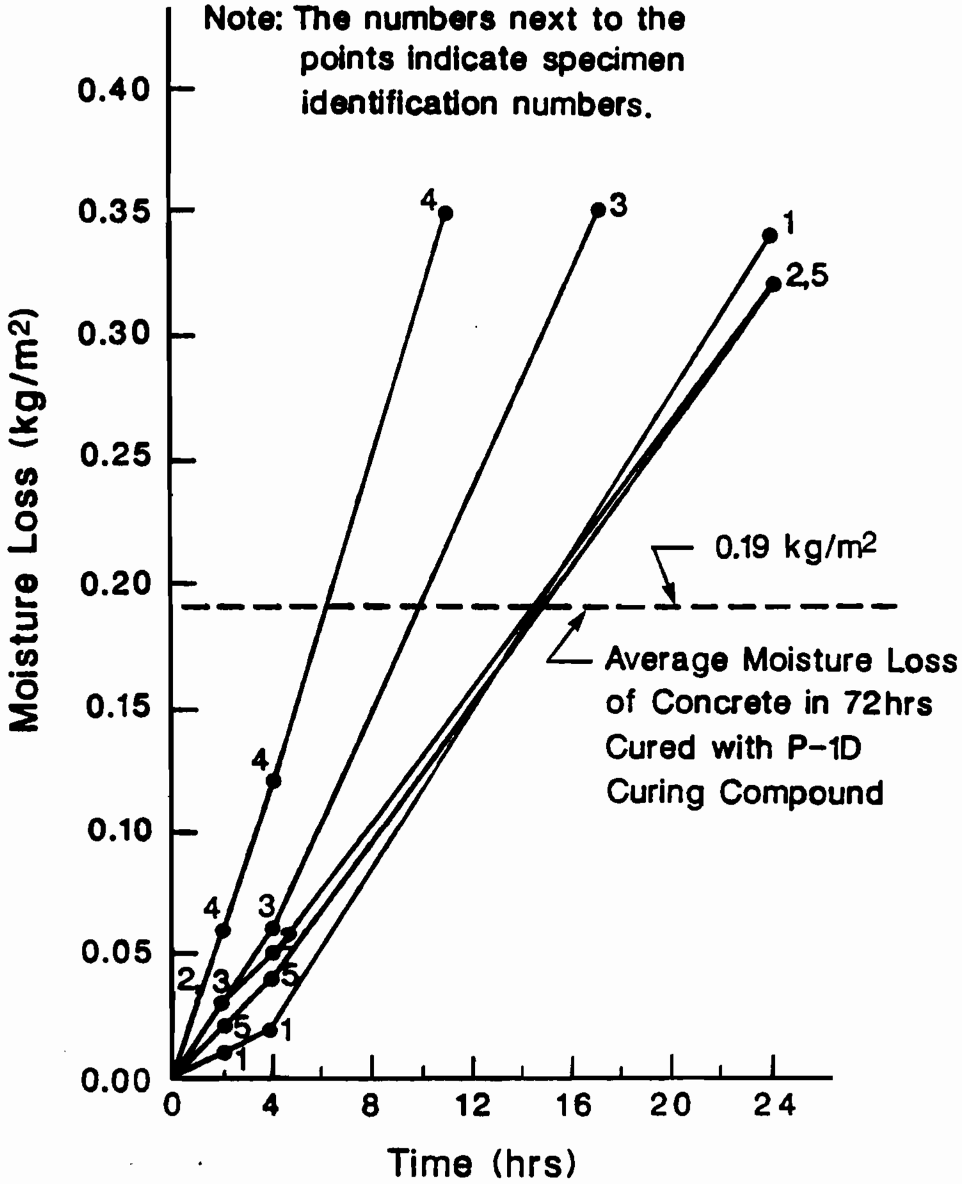


Fig 9.14. Moisture loss through Celanese membrane K-442 treated with P-1D curing compound -- Test No. 1.

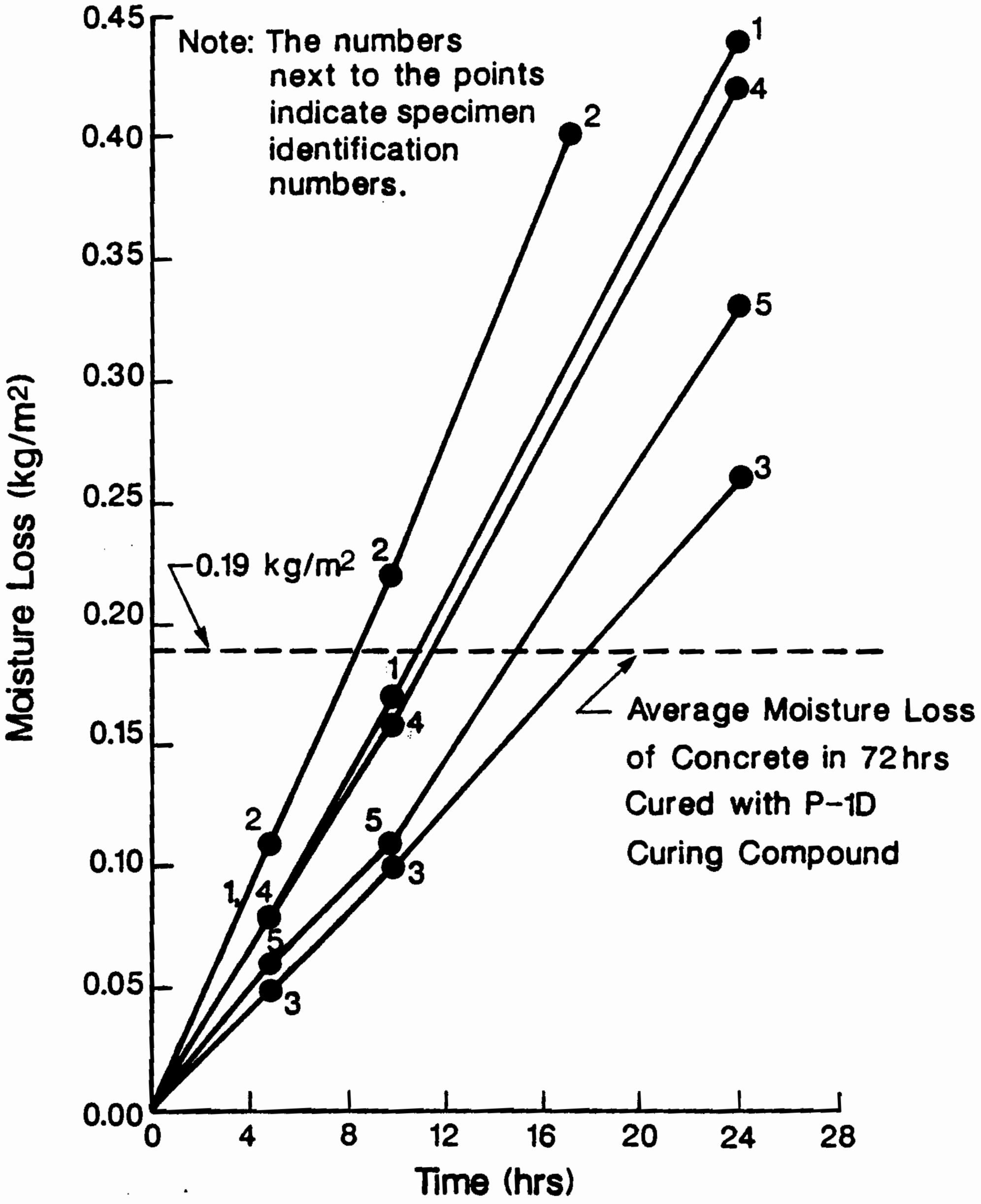


Fig 9.15. Moisture loss through Celanese membrane K442 treated with P-1D curing compound -- Test No. 2.

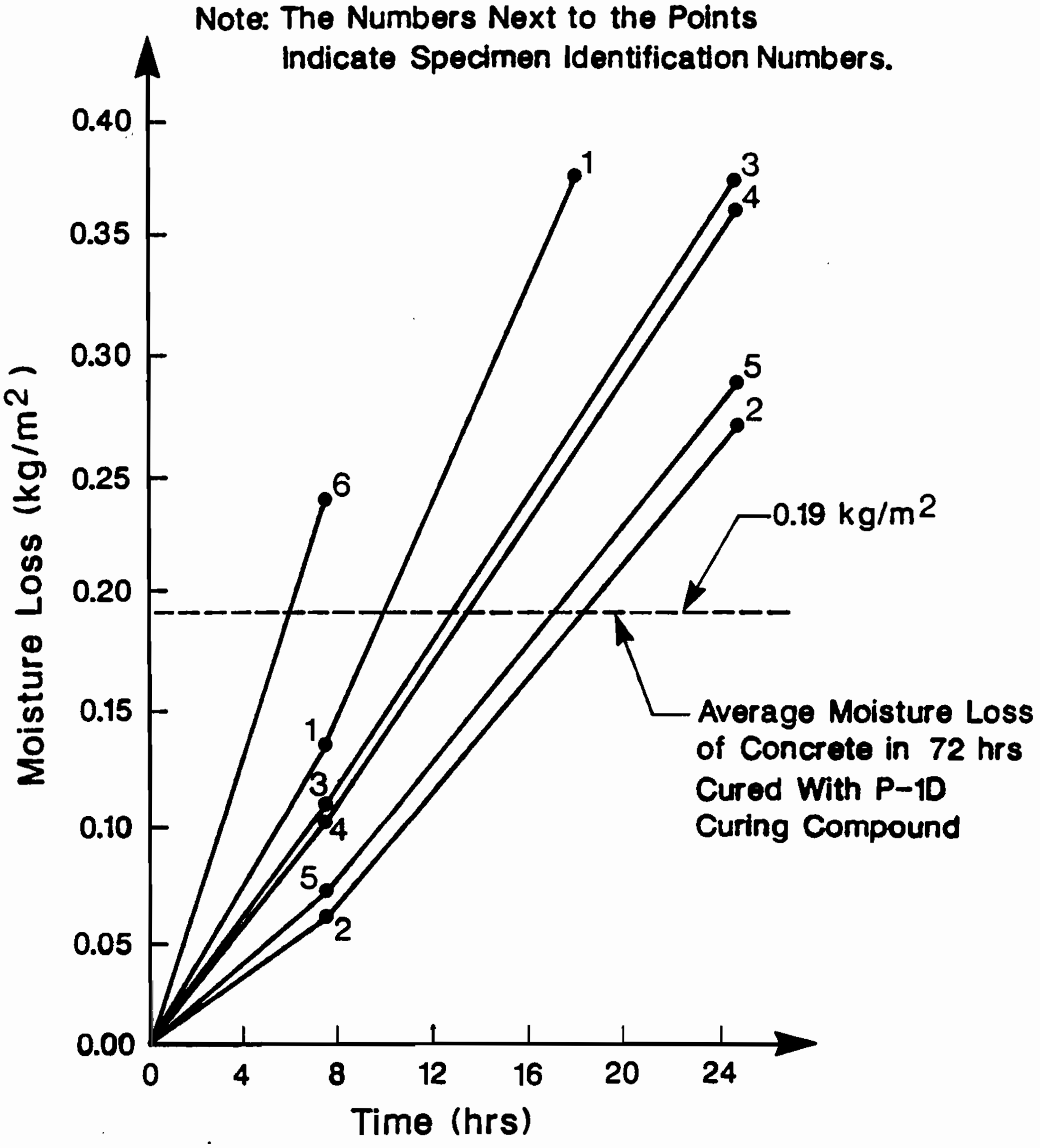


Fig 9.16. Moisture loss through Celanese membrane K-442 treated with P-1D curing compound -- Test No. 3.

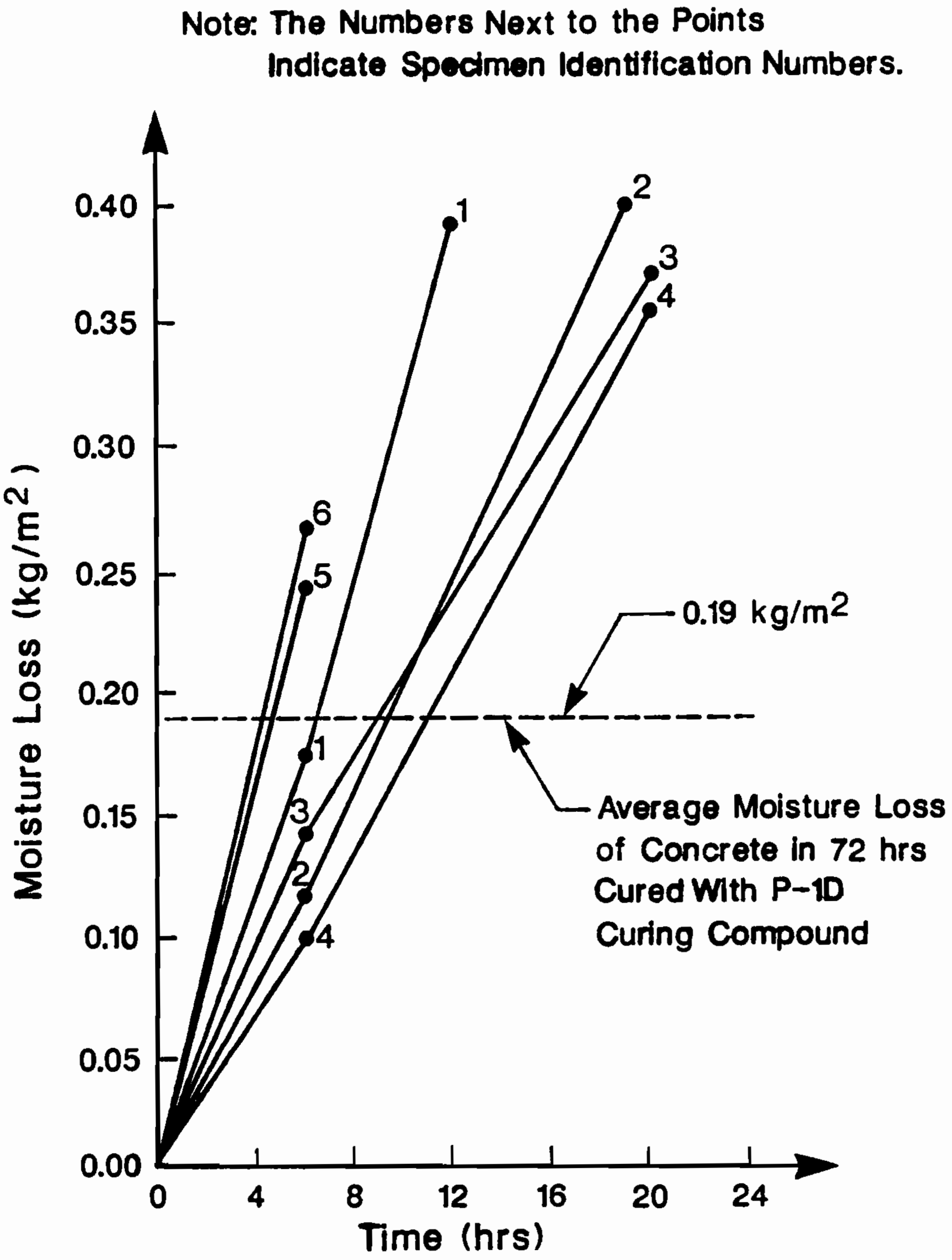


Fig 9.17. Moisture loss through Celanese membrane K-442 treated with P-1D curing compound -- Test No. 4.

80 was found to be 0.19 kg/m^2 (see Table 9.2). The time required to obtain the same moisture loss in the new test varied from 5 to 18 hours at an average of 11 hours. The results from the four tests as obtained from Figs 9.14 to 9.17 are tabulated in Table 9.34. Using the "t" statistic at a 95 percent confidence level, it was found that the time required in the new test to achieve the moisture loss of 0.19 kg/m^2 can vary between 6.1 and 16.1 hours. The range of 10 hours is very high and may lead to serious inaccuracies which indicates that the new test cannot satisfactorily substitute for the ASTM test when this type of curing compound is used.

Figures 9.18 to 9.20 show the moisture loss through the K-442 film treated with S-2 curing compound. The moisture loss of concrete treated with S-2 compound and tested according to the ASTM test was found to be 0.367 kg/m^2 (see Table 9.1). The time needed in the new test to obtain the same loss varied from 4.8 to 6.9 hours with an average of 6 hours. The results from the three tests are tabulated in Table 9.35. Using the "t" statistic at a 95 percent confidence level, it was found that the time required in the new test to obtain the same loss with the ASTM test can vary between 5.2 and 6.8 hours. The range is considerably low and therefore the new test may be a satisfactory substitute for the ASTM test.

Figure 9.21 shows the moisture loss through paper towel treated with P-1D curing compound. The time required to obtain the same loss with the ASTM test varied from 13 to 26 hours. No further testing was performed with the paper towel because of the high variability in the results and the relatively long running time required. However, other brands of curing compound may give more satisfactory results.

The above results indicated that when a Type 1-D curing compound was tested, the new moisture retention test gave unsatisfactory results. However, the Type 2 curing compound gave very satisfactory results.

The new test is very easy to run and requires considerably less test time than the 72 hours 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.

TABLE 9.34. MOISTURE LOSS THROUGH CELANESE MEMBRANE K-442 TREATED WITH P-1D CURING COMPOUND

Test No.	Specimen No.	Time for 0.19 kg/m^2 Moisture Loss (Hours)
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 9.35. MOISTURE LOSS THROUGH CELANESE MEMBRANE K-442 TREATED WITH S-2 CURING COMPOUND

Test No.	Specimen No.	Time for 0.367 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

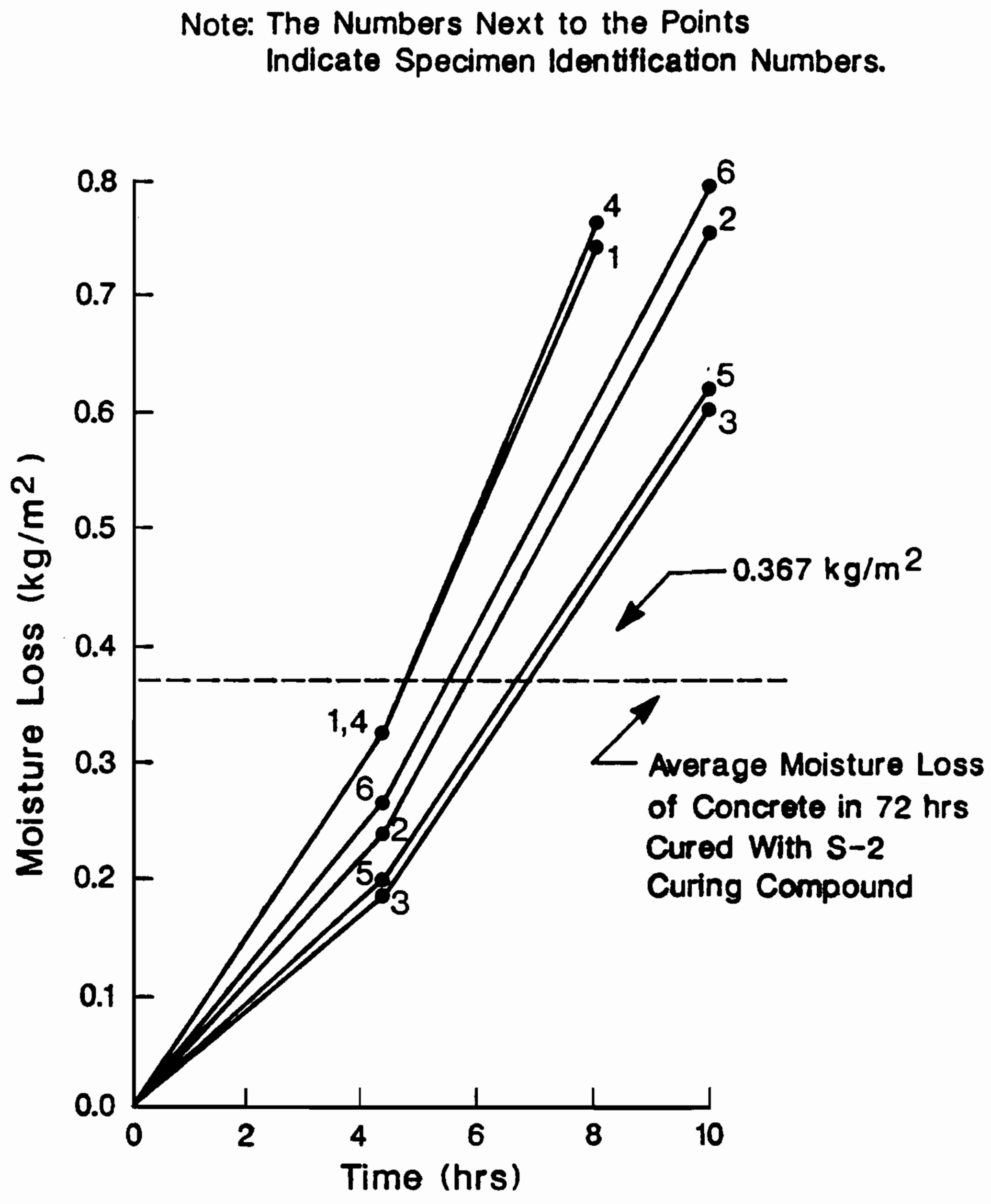


Fig 9.18. Moisture loss through Celanese membrane K-442 treated with S-2 curing compound -- Test No. 1.

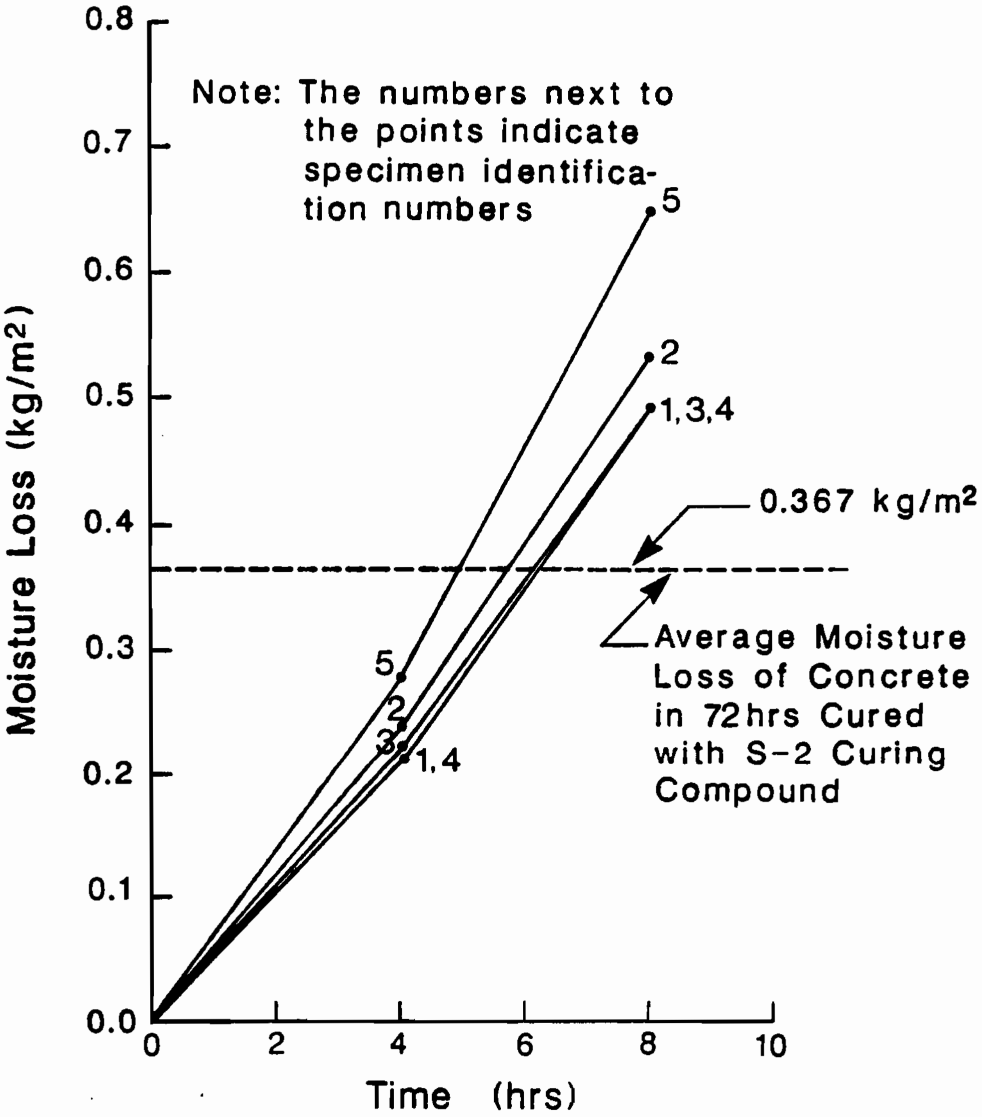


Fig 9.19. Moisture loss through Celanese membrane K-442 treated with S-2 curing compound -- Test No. 2.

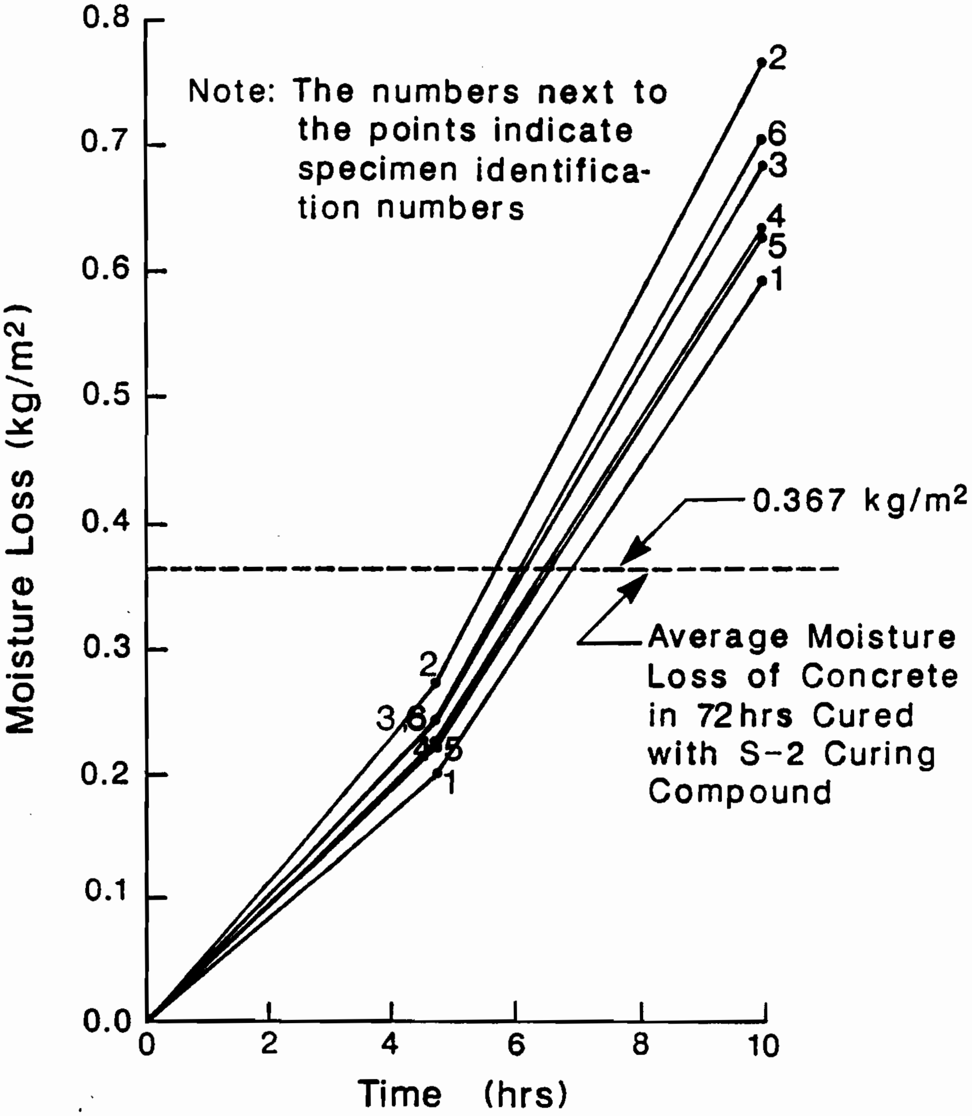


Fig 9.20. Moisture loss through Celanese membrane K-442 treated with S-2 curing compound -- Test No. 3.

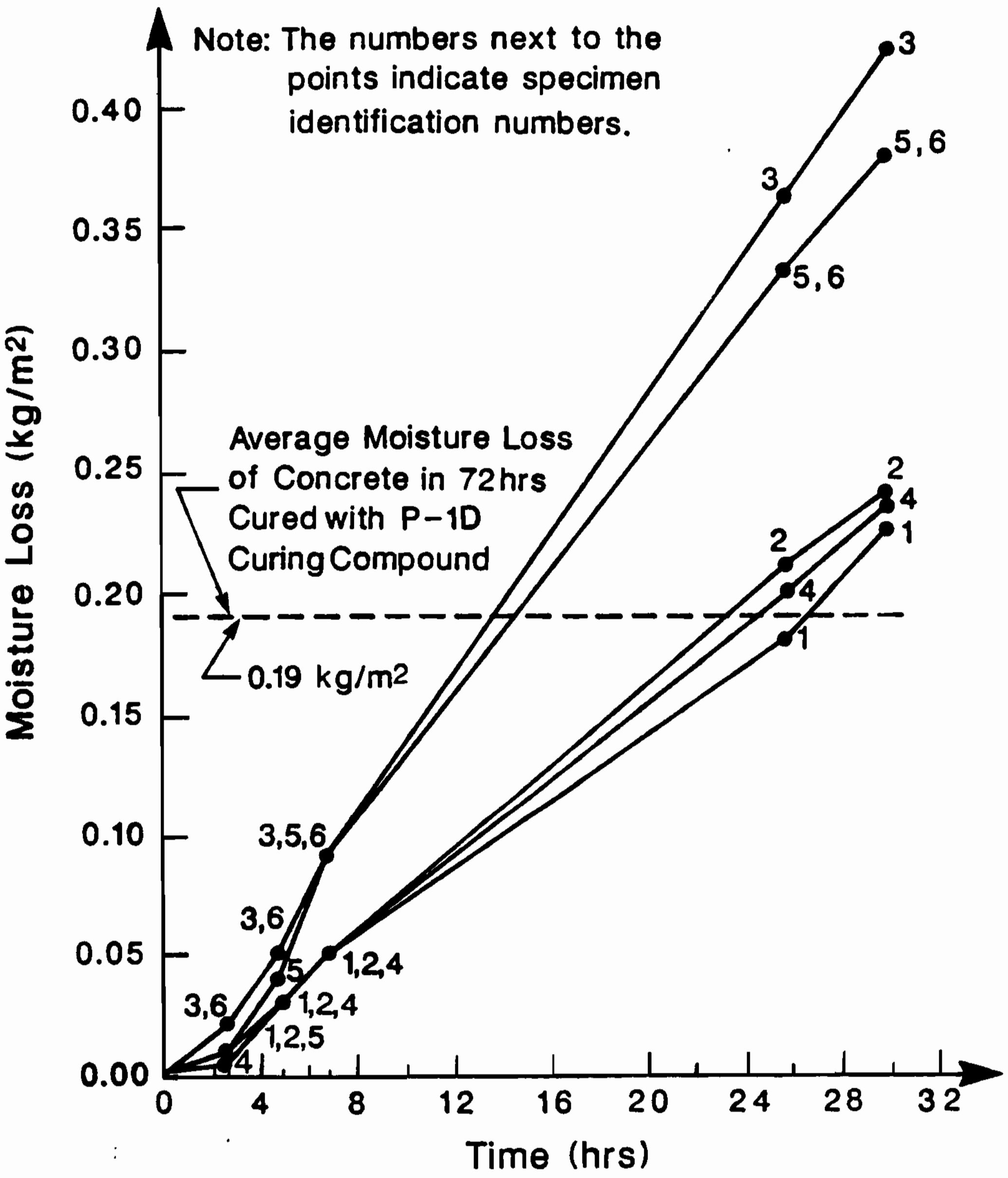


Fig 9.21. Moisture loss through paper towel treated with P-1D curing compound.

SHELF LIFE TEST

S-1D Curing Compound

Table 9.36 contains the results of the solids content tests performed on the shelf life samples of type S-1D curing compound. Figures 9.22, 9.23, and 9.24 represent, respectively, the results of the visual tests performed on this compound at six, nine, and twelve months.

The available data at three months consisted only of middle sample solids content values before and after agitation. These data give some indication of progress toward proper solids content at the middle of the drum, but there was no basis for assuming complete or uniform mixing action. As described earlier in this report, it was this problem that lead to the practice of drawing top and bottom samples in addition to middle samples from each drum, and to the development of the visual uniformity test.

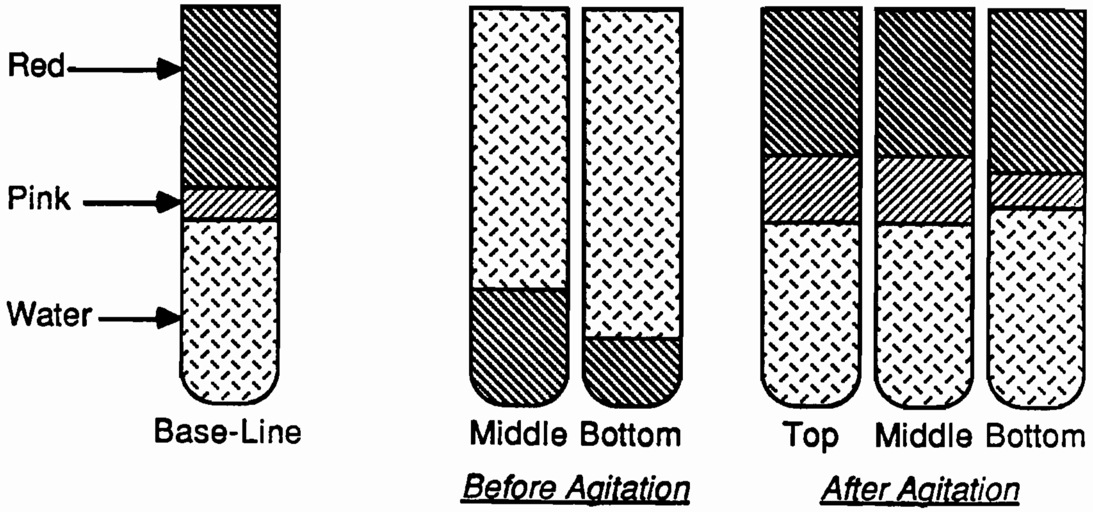
After six months of storage, the available data were somewhat more informative. With respect to solids content, both the previously mixed drum and the drum that had been undisturbed for the full six months exhibited remarkably similar behavior. In both cases, corresponding layers show very similar solids content values prior to agitation. After the agitation procedure solids content values at all layers of both drums are remarkably similar, as well as very close to the known baseline value. This, of course, is the state that would be expected in a properly agitated uniform mixture.

The six month visual test results were not quite as ideal. Drum number one produced visual test specimens that indicated a high degree of uniformity, and a favorable visual comparison after agitation with the base line material. Drum two, however, shows an irregularity in the visual samples taken after agitation. All three post-agitation samples contain an excess of the very viscous light pink compound component, and a deficit of both the fluid red upper layer and the lower layer of water. The problem occurred in the preparation of the specimens. This specific compound had a great tendency to lose the ability to re-emulsify in the 8-ounce nalgene sample bottles that were used. This problem did not occur in the drums themselves during the duration of the study, nor did it occur in all of the

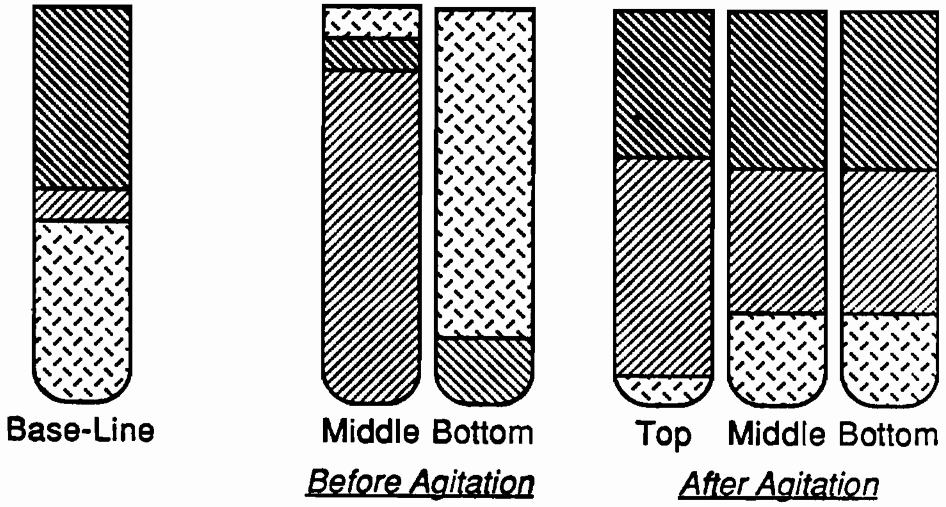
TABLE 9.36. SOLIDS CONTENT (PERCENT) OF S-1D CURING COMPOUND SAMPLES

	Drum No.	Before Agitation Top	Before Agitation Middle	Before Agitation Bottom	After Agitation Top	After Agitation Middle	After Agitation Bottom	Agitation Time (Min.)
Three Months	1	--	18.27	--	--	26.42	--	30
Six Months	1	--	16.09	10.59	26.16	25.90	25.52	30
	2	--	14.93	9.57	25.53	25.50	25.79	30
Nine Months	1	58.45	21.35	12.55	--	26.46	--	5
	2	56.29	23.60	14.38	--	26.30	--	5
	3	57.73	14.76	7.80	--	26.01	--	5
Twelve Months	1	57.00	11.89	0.84	--	25.82	--	10
	2	57.47	15.95	6.86	--	25.85	--	5
	3	55.58	14.16	10.15	--	25.79	--	5
	4	35.37	16.71	6.16	--	26.41	--	5

Base Line Solids = 25.88%



Drum One



Drum Two

Fig 9.22. 6-month S-1D shelf-life visual tests.

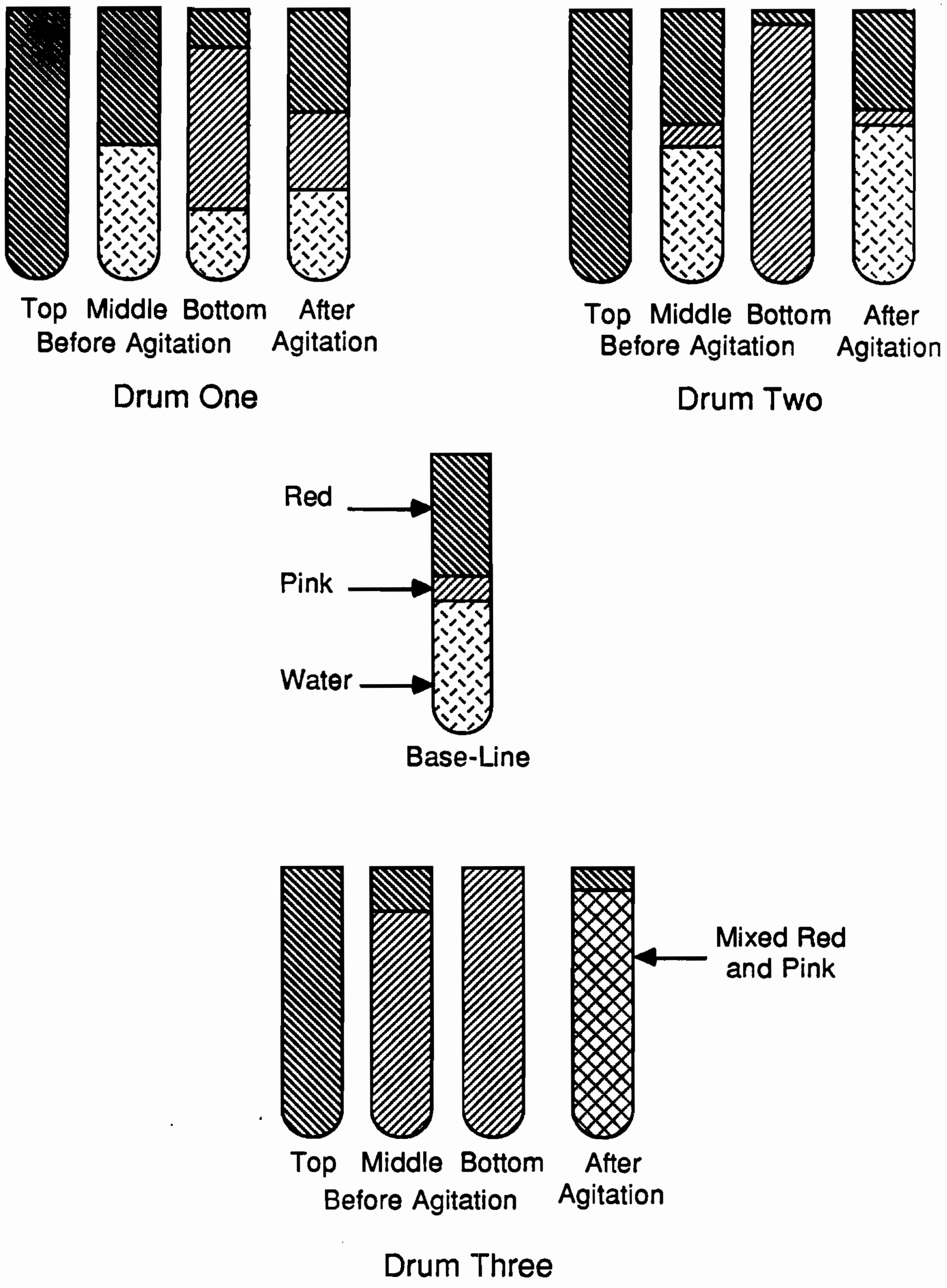


Fig 9.23. 9-month S-1D shelf-life visual tests.

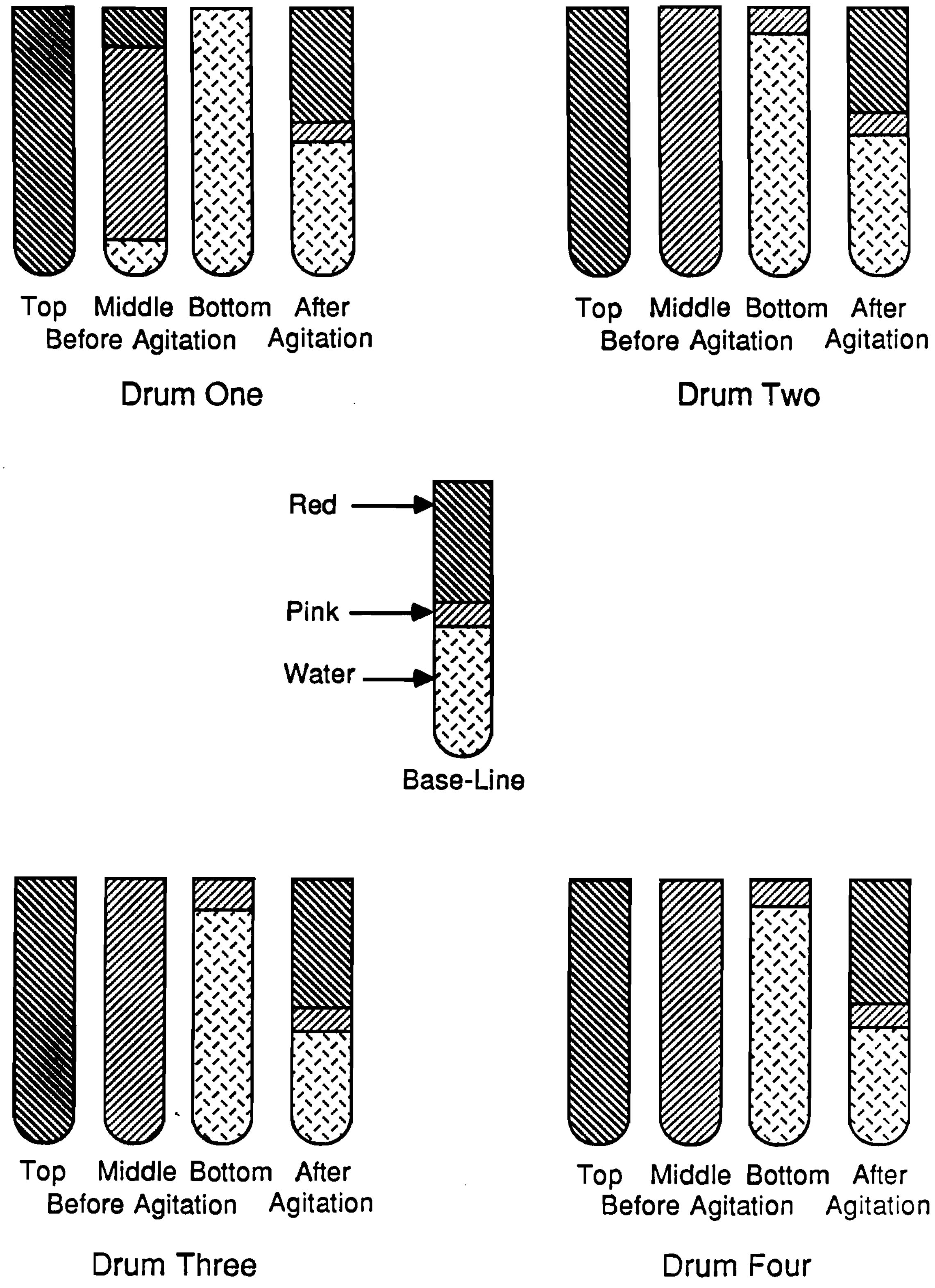


Fig 9.24. 12-month S-1D shelf-life visual tests.

8-ounce samples or in a predictable pattern. When the problem did occur, as in the samples from the sixth month agitation of drum 2, preparation of the test tube visual test sample became a very unreliable operation. Since the three different components were not mixed together, pouring them in proper proportions to their volumes was virtually impossible. As a result, the value of the visual test in such instances was severely reduced if not eliminated.

After nine months, the visual samples suffered the non-reemulsification problem, and so were of little value. The solids content data showed a very interesting pattern. Although agitation resulted in apparently properly mixed compound in all three drums, the two drums that had been agitated at earlier dates produced middle and top layer pre-agitation samples with similar solids content percentages. Interestingly, these percentages were significantly higher than the corresponding values from the previously undisturbed third drum. The most obvious interpretation of this information is that some of the effects of the earlier agitations remained in drums one and two, raising their upper and middle layer solid content rates. The most important observation to be made, however, is that all three drums of the compound were agitated to uniformity in 5 minutes.

After a full year of storage, the solids content test results did not support the apparent residual effect of earlier agitation efforts in the same manner as before. While there was no apparent pattern to the middle and bottom sample solids content test values, the three previously agitated drums all showed significantly higher top layer solids content values than drum four, previously unagitated. This may also have been the manifestation of some lingering effects of the earlier agitations. If this was the case, however, those effects do not appear to follow any consistent pattern. Again, however, all four drums were agitated to apparent uniformity and proper solids content percentage in a reasonably brief period of time.

It would then appear that fifty-five gallon drums of S-1D may be stored for at least one year after manufacture and returned to a usable state through reasonable effort at agitation. As a final note of interest, preparation for disposal of the research material revealed that the S-1D curing compound could not be reemulsified through simple agitation. These preparations took place seven months after completion of the experimental procedure involving the S-1D compound. Thus, the material became unusable at sometime between twelve and nineteen months of age.

The results of SDHPT standard curing compound tests are summarized in Table 9.37. At nine months, the type S-1D compound was shown to be unsatisfactory based on SDHPT requirements for holding a vertical surface and with regard to drying time, which was shown to be in excess of the required maximum of four hours.

With respect to moisture retention capabilities, both previously agitated drums (number 1 and 2) exhibited satisfactory properties, though these results were somewhat different from the original capabilities of the material. Though also failing the vertical spray and drying time requirements, the previously un-agitated drum number three exhibited moisture retention properties much more in line with those of the original samples.

After twelve months of storage, the S-1 material seemed more satisfactory. In this case, the samples from drums three (agitated once previously) and four (previously un-agitated) passed all SDHPT curing compound tests. Again, however, drum one and drum two samples both exhibited unsatisfactory vertical spray behavior, although drying time requirements were met in these cases.

At twelve months, the moisture retention tests showed a significant improvement over the base line values. The suspected cause of this was a sizeable loss of volatiles. This suspicion is supported by a very noticeable increase in solids content, flash temperature, and to a lesser degree, specific gravity. There was some indication of this three months earlier, but the specific values were less conclusive.

The reason for this loss of volatiles was not entirely clear. The actual tests were performed four months after the samples were taken. The samples were of sufficient size to fill their containers, however, and tight sealing of those containers had been made a point at the time of sampling. Furthermore, the loss was not likely caused by the repeated opening of the drums, since the loss of volatiles was noted as well in the sample from drum twelve, which had not been opened except for the taking of the sample tested

TABLE 9.37. RESULTS OF SDHPT TESTS ON TYPE S-1D CURING COMPOUND

Compound	Age at Test (mo.)	Vertical* Spray	Drying Time (min.)	Flash Temperature (°F)	Solids Content (percent)	Specific Gravity	24 Hour Moisture Loss (percent)	72 Hour Moisture Loss (percent)
Base Line Tests								
No. 1	--	S	60	98	25.88	0.949	0.75	1.53
No. 2	--	--	--	--	--	--	0.55	1.16
No. 3	--	--	--	--	--	--	0.68	1.09
No. 4	--	--	--	--	--	--	0.62	0.88
No. 5	--	--	--	--	--	--	0.44	0.89
9 Month Tests								
Drum 1	17	U	240+	114	27.70	0.939	1.09	1.66
Drum 2	17	U	240+	117	30.03	0.910	0.22	0.77
Drum 3	17	U	240+	121	32.18	0.926	0.61	1.15
12 Month Tests								
Drum 1	16	U	100	118	36.17	0.967	0.46	0.80
Drum 2	16	U	120	116	30.62	0.950	0.49	0.62
Drum 3	16	S	75	118	35.70	0.956	0.32	0.45
Drum 4	16	S	60	115	30.37	0.959	0.41	0.68

* Results are reported as "S" (satisfactory) or "U" (unsatisfactory)

by SDHPT. It seems likely, therefore, that the loss of volatiles was a symptom of age, occurring to the greatest degree between nine months of storage and an age of twelve months.

S-2 Curing Compound

Table 9.38 presents the tabulated solids content test results for type S-2 curing compound. Figures 9.25, 9.26, and 9.27 display the results of the visual tests on type S-2 compound after six, nine, and twelve months.

The three-month solids content data reveals the typical problems that accompanied reliance only on mid-drum solids content for verification of mixing effectiveness. Such a practice would label the drum as properly mixed even before agitation, when separation was clearly visible both through the appearance of a 7-inch deep transparent layer of yellow liquid at the top of the drum and through the layer of white solid pigment occupying the remainder of the drum, as detected through the stick probe.

As with the S-1D compound, the visual test was not employed at this stage of the study, and so could be of assistance in the analysis.

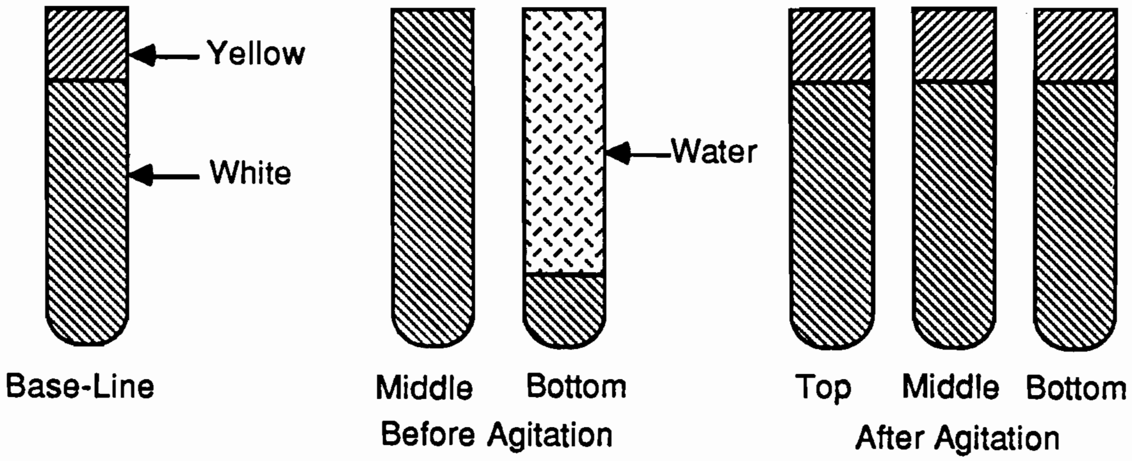
After six months of storage, both drums behaved in virtually the same manner. Interestingly, the pre-agitation bottom solids content was lower than that of the material in the middle of the drum. The implication was that the yellow resin at the top of an unmixed drum had a higher solids content than material containing the pigment material in the lower portions of the drum. Both drums were mixed to consistent solids content approximating the known base line value. Drum one was agitated for fifty minutes not out of necessity, but rather to leave no doubt of complete agitation. This was done to test the experimenters ability to judge thoroughness of mixing based on visual examination of the drum contents and material adhering the probe stick. This ability was confirmed and used during subsequent testing of all materials.

These conclusions, based solely on solids content tests, were confirmed by visual test results. The sample from both drums showed the same pattern. Before agitation, the middle samples show heavy concentrations of white pigment and no indication of either the yellow resin or the water that

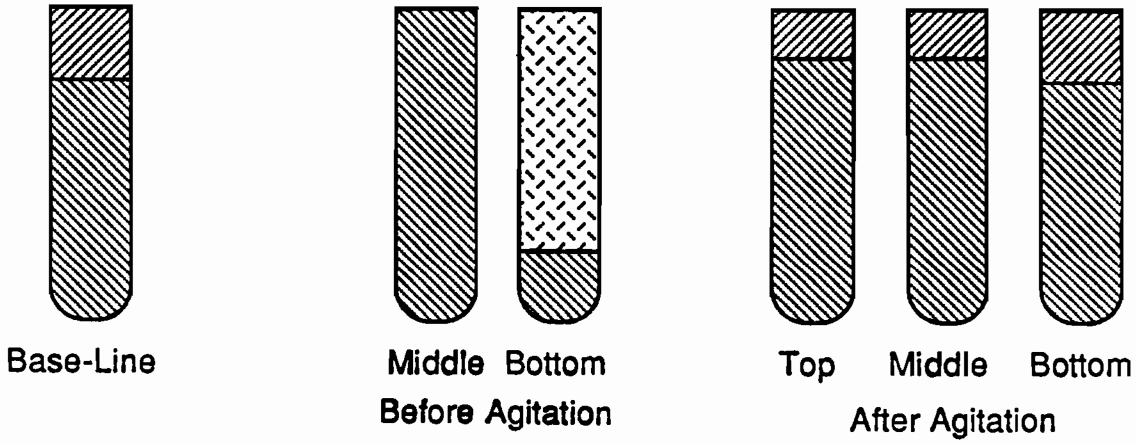
TABLE 9.38. SOLIDS CONTENT (PERCENT) OF S-2 CURING COMPOUND SAMPLES

	Drum No.	Before Agitation Top	Before Agitation Middle	Before Agitation Bottom	After Agitation Top	After Agitation Middle	After Agitation Bottom	Agitation Time (Min.)
Three Months	1	--	37.89	--	--	37.86	--	30
Six Months	1	--	39.18	30.72	40.10	40.08	40.60	50
	2	--	38.07	30.72	39.75	40.54	39.69	20
Nine Months	1	57.89	38.37	37.82	--	39.65	--	5
	2	59.12	39.02	36.09	--	39.42	--	5
	3	58.91	37.30	27.78	--	39.90	--	10
Twelve Months	1	56.34	38.45	35.16	--	39.26	--	5
	2	56.19	38.39	35.45	--	39.37	--	5
	3	57.45	37.75	35.31	--	39.11	--	5
	4	56.47	35.59	31.07	--	39.32	--	10

Base Line Solids = 39.27%



Drum One



Drum Two

Fig 9.25. 6-month S-2 shelf-life visual tests.

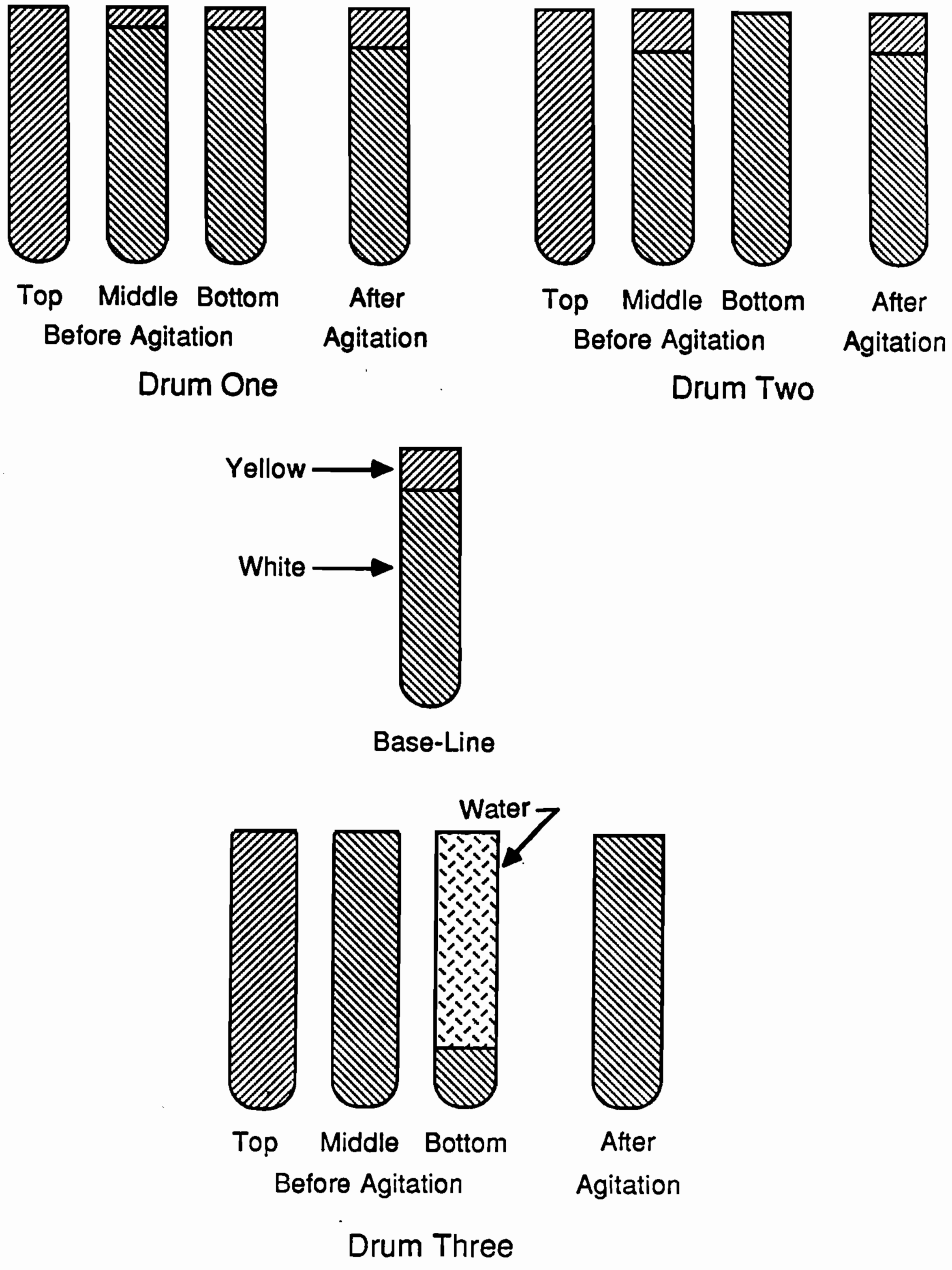


Fig 9.26. 9-month S-2 shelf-life visual tests.

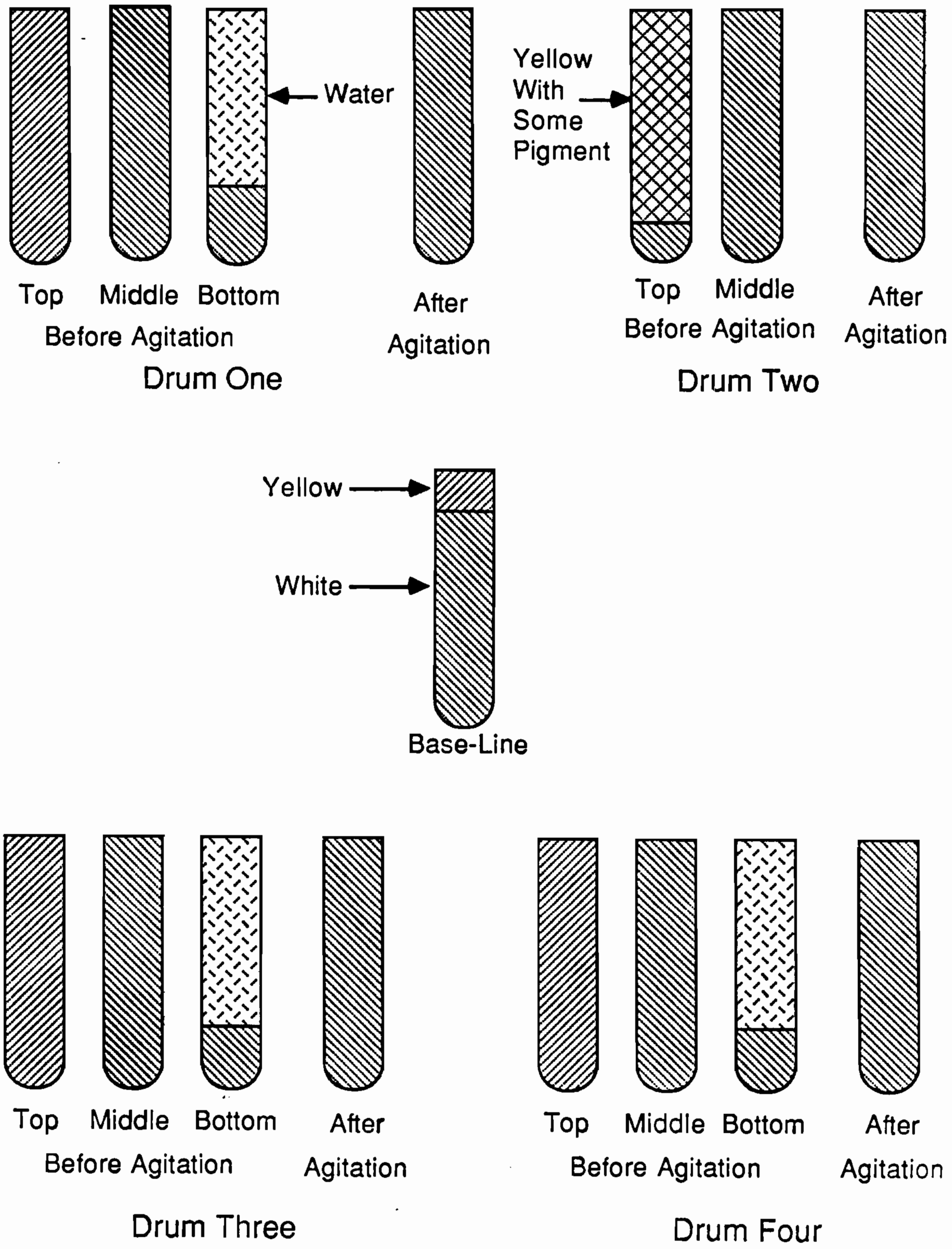


Fig 9.27. 12-month S-2 shelf-life visual tests.

constitute the rest of the compound. The bottom samples taken before agitation indicated the lack of any residual effect from the previous agitation, three months earlier, of drum two. Both bottom samples consisted largely of water, which explains the lower solids content value of the bottom samples.

After agitation, visual test results reinforced the confirmation of adequate agitation provided by solids content test results. Visual samples from both drums after agitation revealed a high degree of uniformity in the agitated compound, as well as good conformity to the separation pattern of the base line sample.

The nine-month battery of tests revealed some apparent residual effects of previous agitation. Drums one and two, which had been previously mixed, showed markedly higher solids content values in the lower third of the drum than did drum three, which had not been mixed since its manufacture. In the visual test, earlier agitation efforts appeared to have caused pigmentation material to remain in the lower third of the drum. This white pigment was evident in the bottom samples from drums one and two, which had been agitated before, to a great degree. Drum three, previously undisturbed, produced a sample from the bottom third, which, after separation, proved to consist primarily of water. This, of course, was reflected in the lower solids content of the third drum bottom sample, relative to the solids content values of the lower third samples from the two previously mixed drums.

Despite the apparently greater degree of separation in drum three, all three drums were agitated to uniformity with relative ease. During agitation, visual inspection of the three drums gave the impression of thorough mixing of all three drums. Solids content results also suggest that this was the case. The solids content test was not conclusive for this material, since mid-drum solids content rates were approximately of base line magnitude even before agitation. Visual separation patterns after agitation suggested uniformity in the two previously agitated drums, but not drum three.

It was necessary to agitate the third drum for ten minutes, as opposed to the five minutes necessary for drums one and two. This is not, however, a serious concern.

to the five minutes necessary for drums one and two. This is not, however, a serious concern.

The twelve month solids content tests were quite similar to the nine month results. Once again, drums which had been mixed previously had higher lower-third solids content values than the drum that had been left undisturbed for a year. The distinction between the drums that had been agitated before and the one that had not was not as great as it had been at nine months. Additionally, the supposed residual effect of earlier agitation was not demonstrated in the twelve month visual test specimen. At twelve months, the bottom samples from the previously agitated drums were virtually identical to tests from the drum not previously agitated. Specifically, all three pre-agitation bottom samples separated into two layers, a small layer of white pigment below a comparatively large volume of water. The pre-agitation bottom sample from drum two could not be prepared because of excessive viscosity. The reason for this was not clear.

As in the nine month tests, all drums were returned to a uniform state conforming to base line solids content values. Again, the drum that was not agitated at earlier dates required 10 minutes of agitation as opposed to the 5 minutes required for the other three drums.

For some reason, post-agitation visual separation patterns did not conform to the base line pattern. This might have indicated some change in the material, or sufficient time for settling might not have been allowed. On the basis of the tests performed, there was reason to limit shelf life of Type S-2 curing compound to less than one year. Agitation during the storage period did appear to result in some advantage in the nine month samples. The time saved in agitation just before use was only five minutes, however, and this saving was more than overcome by the time used for equipment preparation and actual agitation in performing the intermediate agitation during the storage period.

SDHPT performed the full range of curing compound qualifying tests on type S-2 compound as well. The results of these tests are summarized in Table 9.39. The findings of these tests were very confusing. At nine months, samples from all three drums were very thick. In fact, SDHPT found it impossible to conduct tests on the sample from drum number two because the

TABLE 9.39. RESULTS OF SDHPT TESTS ON TYPE S-2 CURING COMPOUND

Compound	Age at Test (mo.)	Vertical* Spray	Drying Time (min.)	Reflectivity	Flash Temperature (°F)	Solids Content (percent)	Specific Gravity	24 Hour Moisture Loss (percent)	72 Hour Moisture Loss (percent)
Base Line Tests									
No. 1	--	S	50	62.3	90	39.27	1.087	1.39	2.06
No. 2	--	--	--	--	--	--	--	1.36	2.32
No. 3	--	--	--	--	--	--	--	1.48	2.29
No. 4	--	--	--	--	--	--	--	1.34	2.54
No. 5	--	--	--	--	--	--	--	1.41	2.24
9 Month Tests									
Drum 1	17	S	150	77.6	115	49.65	1.125	5.12	6.06
Drum 2	17	Material Would Not Resuspend							
Drum 3	17	S	165	78.2	107	49.92	1.117	2.24	3.40
12 Month Tests									
Drum 1	14-16	S	105	64.2	113	40.39	1.119	0.46	0.73
Drum 2	14-16	Could Not Spray			116	41.06	1.121	Could Not Spray	Could Not Spray
Drum 3	14-16	Could Not Spray			113	45.00	1.124	Could Not Spray	Could Not Spray
Drum 4	14.16	Could Not Spray			112	42.29	1.018	Could Not Spray	Could Not Spray

* Results are reported as "S" (satisfactory) or "U" (unsatisfactory)

compound separated to such a degree that the viscous "solids" would not resuspend. The other two drums produced samples that had inadequate moisture retention properties, severely increased solids content rates, raised reflectivities, implying higher content of the reflective solids, and elevated flash temperatures. These symptoms all suggest high loss of volatiles, yet the added solids content would normally be expected to increase moisture retention, which was not the exhibited trend. It is considered likely that the properties displayed by the nine month S-2 SDHPT test samples is anomalous. The basis for this statement was the findings of the tests on the twelve month samples.

As can be seen in Table 9.39, the typical solids content and specific gravity values of the twelve month samples had returned from the high nine month values to almost the same values displayed by the original samples. Despite this, three of the four samples taken at twelve months were still not fluid enough to apply in the vertical spray and moisture retention tests. Obviously, this constituted a serious alteration in material properties.

It should be noticed that both the nine and twelve month tests were actually performed several months after the sampling date. The effects of long-term storage of such small volumes (8 ounces) of curing compound are not know, though one gallon samples used in the other parts of the study did not suffer similar thickening problems.

P-1D Curing Compound

In many ways, this was the most unusual of the six compounds tested. All the Type 2 compounds displayed base line solids content values typically on the order of 40 percent and also separated into easily distinguishable layers during storage. S-1D and M-1D compounds also separated very clearly, but had base line solids contents around 25 percent. The P-1D compound, however, virtually never displayed visible layer separation and also was typified by solids content values more to be expected from a Type 2 white-pigmented compound. Moreover, these solids content values were usually fairly uniform throughout a drum even before it was agitated. All of these factors, as well as stick probe observations and other, more qualitative, laboratory observations indicated that type P-1D compound undergoes only the slightest amount of separation during storage. Additionally, the agitation necessary for mixing to uniformity was very minimal relative to other compounds, and included no use of the agitator blades to scrape the interior surfaces of the drum.

After 3 months of undisturbed storage, both the solids content tests and the visual tests led to the same conclusion: the compound was essentially ready for use before agitation.

As can be seen in Table 9.40, the solids content of all samples, both before and after agitation, were approximately equivalent, and approximately the same as the known baseline value of 43.67 percent. Additionally, as can be seen in Fig 9.28, the three month visual test showed no visually distinguishable changes caused by agitation. The material looked, in all layers, like the baseline sample both prior to, and after, agitation. The material can best be described as having a consistency, viscosity, and texture very similar to glycerin, but with the deep red color of the fugitive dye. The only indication of separation before agitation was a layer of a much less viscous fluid at the top of the drum of compound. This layer was only a few millimeters thick, and mixed into the rest of the compound in the first few seconds of agitation.

After an additional three months of storage, the solids content and visual tests of both drums at that time led to essentially the same conclusions as were arrived at during the three month tests. Although the actual values are somewhat higher than those of the three month test battery, the six month solids content tests showed equivalent solids content throughout both drums one and two even prior to agitation. Again, these values closely reflected the base line value. The visual tests at six months were essentially the same as the three month tests. All samples from both drums appeared almost identical, as can be seen in Fig 9.29. In fact, the only indication that agitation was necessary was a thin layer of watery fluid similar to that at the top of the three month drum, present in both drums tested at six months of age.

In addition, a solid crust of a light orange color and waxy texture had developed over the top of drum one, the drum that had been agitated three

TABLE 9.40. SOLIDS CONTENT (PERCENT) OF P-1D CURING COMPOUND SAMPLES

	Drum No.	Before Agitation			After Agitation			Agitation Time (Min.)
		Top	Middle	Bottom	Top	Middle	Bottom	
Three Months	1	40.80	40.30	41.26	41.99	41.08	40.27	40
Six Months	1	44.97	43.07	42.98	--	44.52	--	5
	2	44.36	44.74	42.64	--	43.05	--	5
Nine Months	1	--	41.17	38.94	--	39.89	--	5
	2	--	42.32	40.94	--	40.29	--	5
	3	--	42.14	41.27	--	39.22	--	5
Twelve Months	1	--	55.43	49.59	--	47.52	--	5
	2	--	51.26	51.42	--	52.62	--	5
	3	--	48.73	51.28	--	56.01	--	5
	4	41.49	48.28	45.13	--	49.80	--	5

Base Line Solids = 43.67%

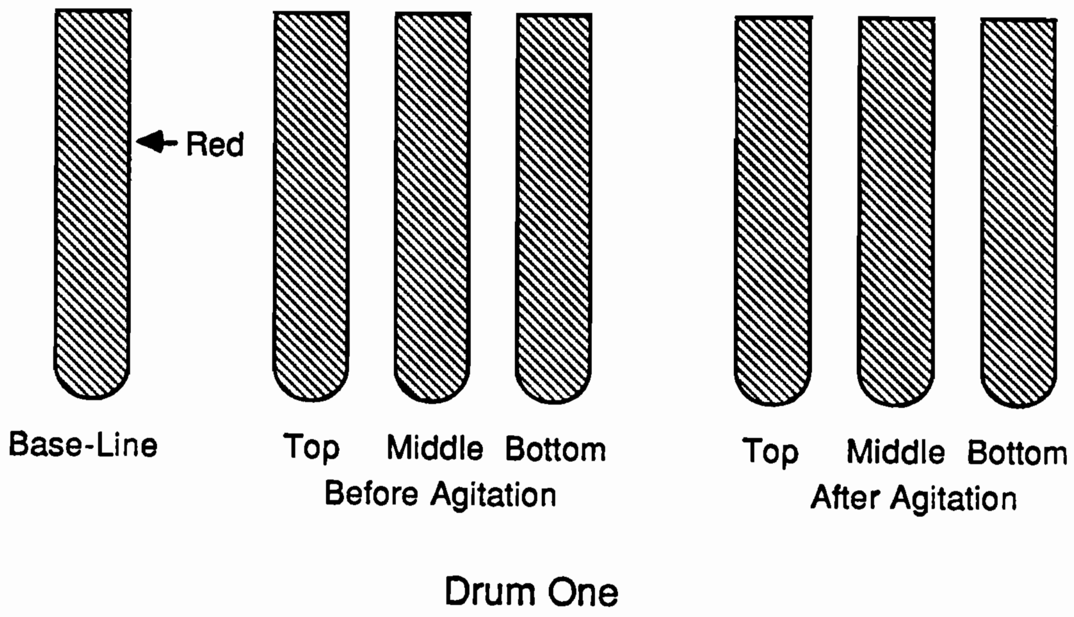
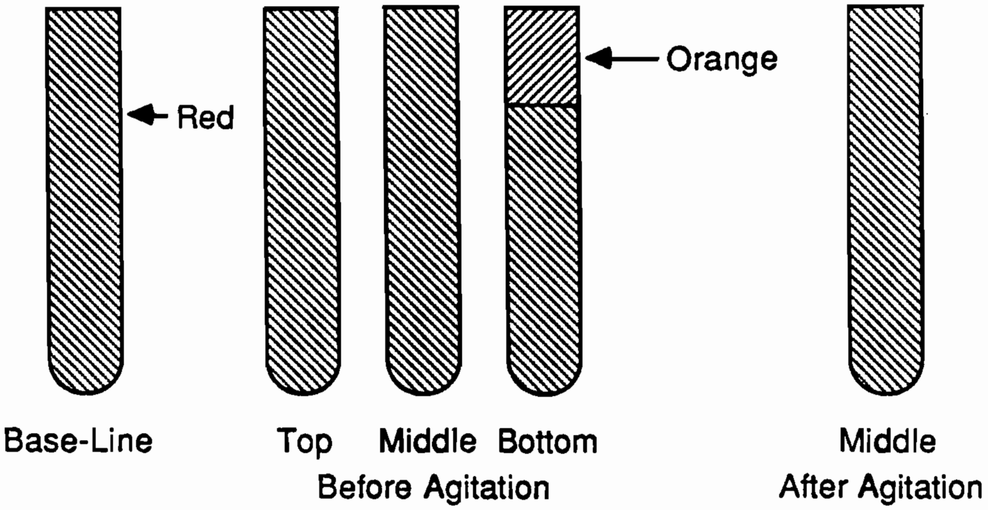
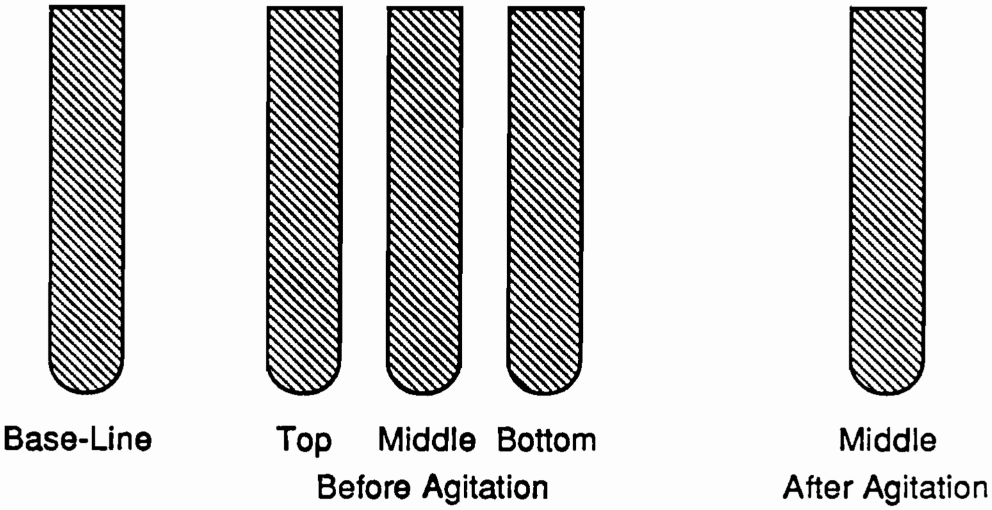


Fig 9.28. 3-month P-1D shelf-life visual tests.



Drum One



Drum Two

Fig 9.29. 6-month P-1D shelf-life visual tests.

months previously. This crust did not appear on the top of the compound in drum number two. The crust is shown in Fig 9.30.

The tests at nine months, yet again, produced little evidence of settlement during storage. Once again, solids content value for all samples from all three drums approximated the base line value. Visually, as seen in Fig 9.31, all three drums appeared uniform throughout, and similar to the base line sample. At the age of nine months, all three drums examined showed the typical watery fluid layer at the top of the drum. Again, however, this was quickly blended into the body of the compound. In addition, the two drums that had been agitated previously had the wax crust at the top of the compound. The third drum, which had not been previously agitated, did not have this crust. It is important to note that the crust in drum number one reformed since the six months agitation, since the crust noted at that time was broken up and dispersed throughout the drum during the six month agitation of the drum.

The twelve month test results included the first indications of an age effect. In terms of the solids content test, the experimental values again indicated compound uniformity prior to agitation. However, the actual value of solids content was 6 to 8 percent higher than the known value. This might have been caused by volatile evaporation during agitation in the first three drums, but the fourth drum had been sealed for the full year, yet still contained compound with the same unexpectedly high solids content rates. Additionally, the visual test, finally showed some variations with depth prior to agitation. In all four drums, the pre-agitation middle sample appeared the same as the base line sample, while the bottom sample contained a more opaque, lighter orange-colored material. See Fig 9.32 for a representation of the twelve months visual test results. The bottom samples, however, showed no evidence of separation into different components.

In all four drums, agitation resulted in a middle sample of a similar visual appearance to that of the base line value, but with a solids content above the base line value. This, as before, implies some loss of volatiles, but since it occurred even in the drum that had remained sealed for the full year, this loss could not be explained.

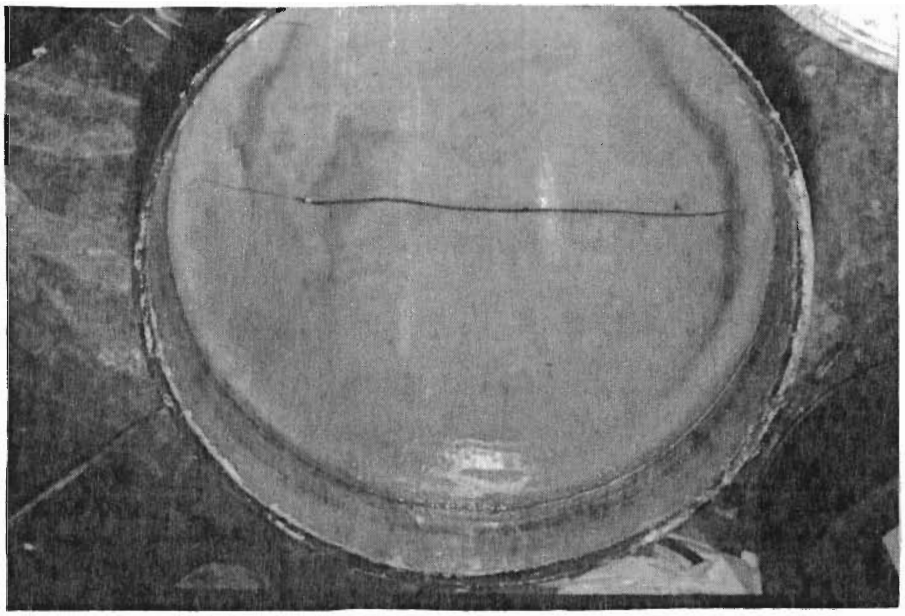


Fig 9.30. Crust that developed at the top of drums of type P-1D compound between agitations.

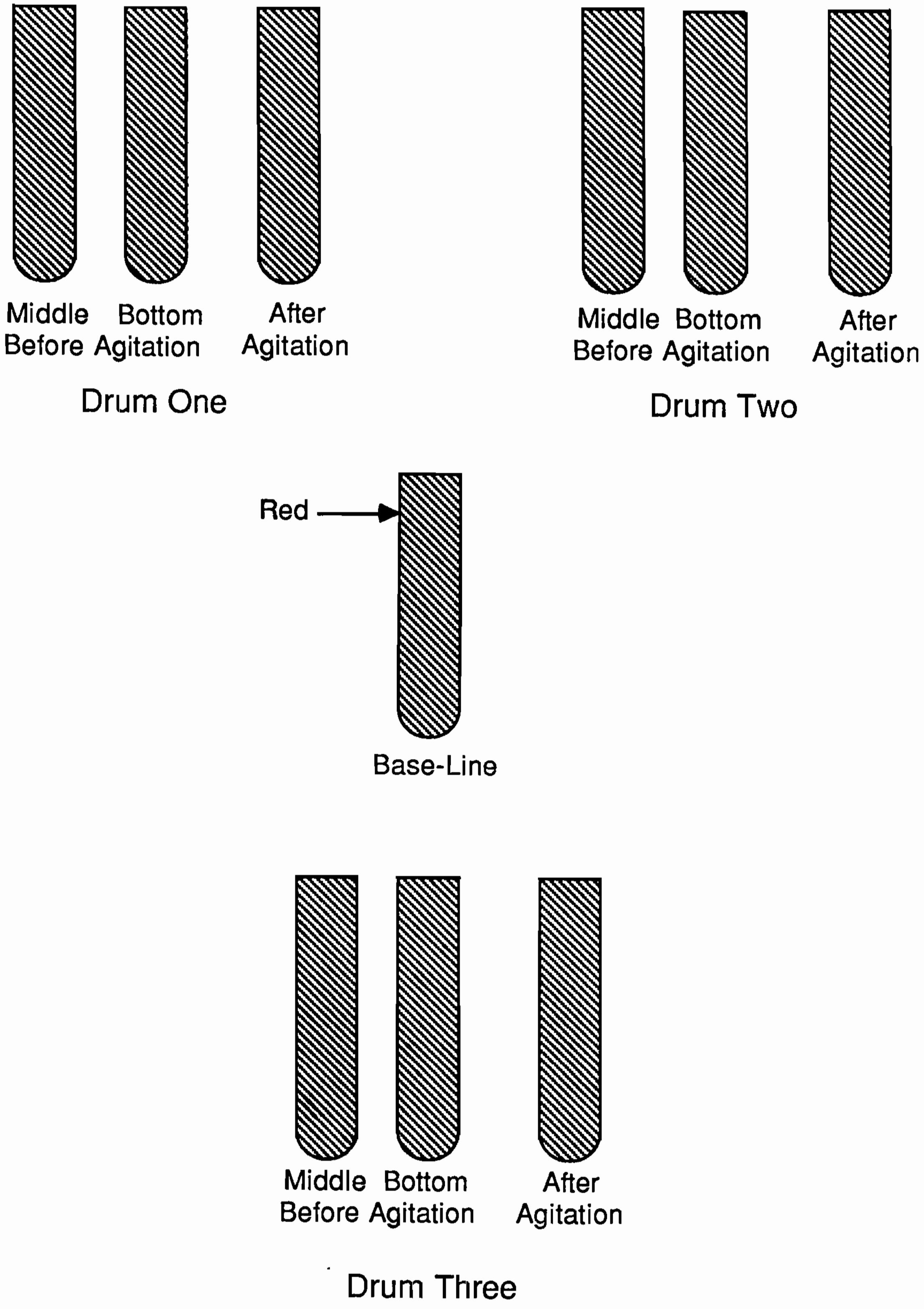


Fig 9.31. 9-month P-1D shelf-life visual tests.

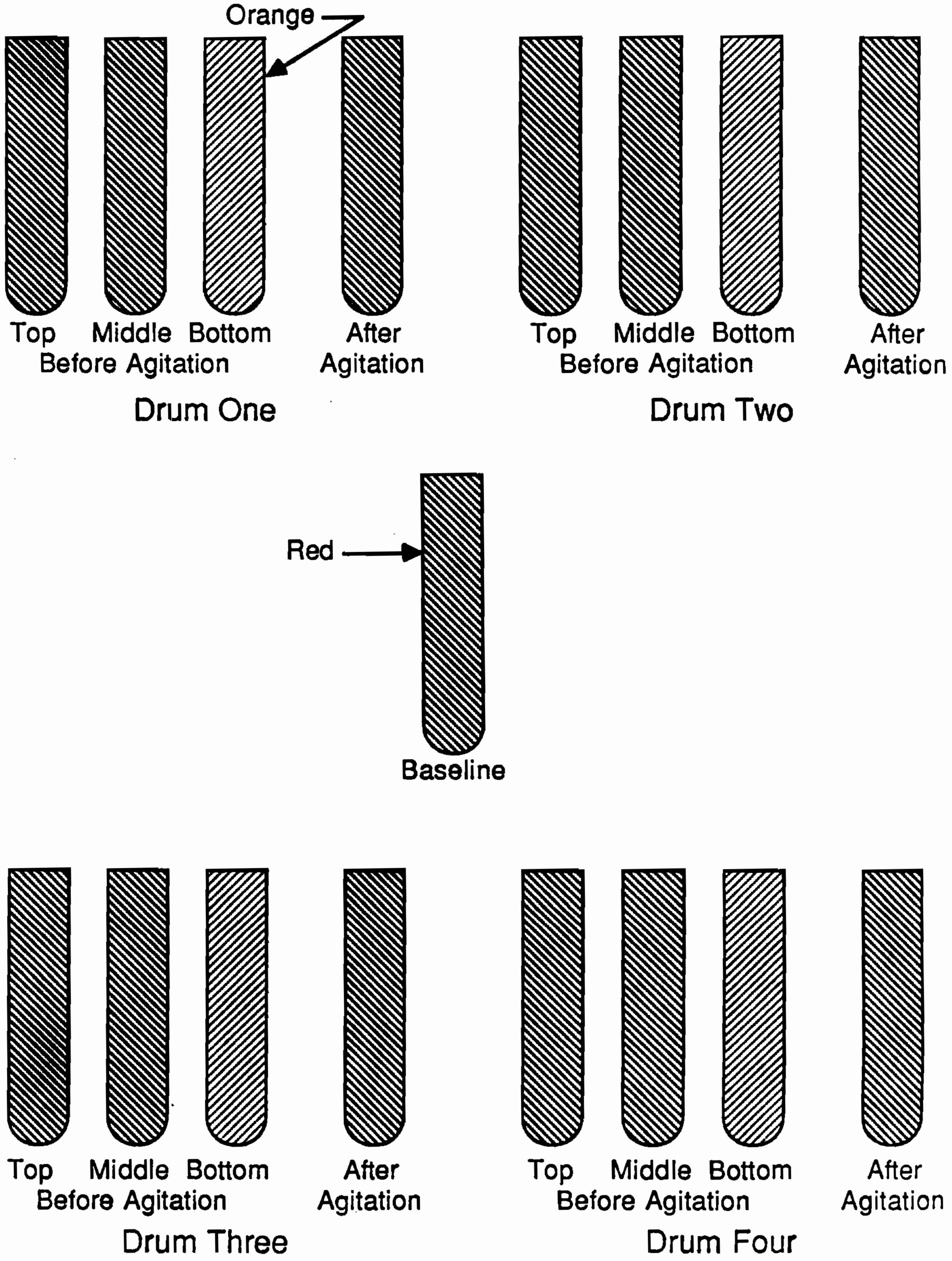


Fig 9.32. 12-month P-1D shelf-life visual tests.

The results of SDHPT curing compound tests on type P-1D are tabulated in Table 9.41. These results proved quite unusual.

At nine months of age all three drums tested exhibited properties quite comparable to those of the original material. The exception was the moisture retention test results. The drum that had not been previously opened displayed moisture retention properties quite comparable to those of the original material. Drums one and two, both of which had been opened and agitated previously, showed vastly superior moisture retention properties. The reason for this was unclear. All other properties were completely normal, giving no indication of an increased solids content or loss of volatiles.

The twelve month test results were quite similar to nine month results, except that all four drums showed superior moisture retention properties. The previously unopened drum also showed the superior moisture retention properties, which had not been the case three months previously. Again, these improved capabilities could not be explained. All other properties, including spraying ease, were not altered. Although these findings did constitute a change in material properties, this change was beneficial and the material was still acceptable by all SDHPT test criteria.

P-2 Curing Compound

In contrast to the type P-1D curing compound, the P-2 compound was the most difficult to agitate. This was primarily because the layer of settled white pigment at the bottom of the drum was more cohesive than that in the other Type 2 compounds and offered greater resistance to the agitator blades. This increased difficulty in agitation also makes sense in light of the fact that the P-2 compound had the highest solids content of the six compounds tested, and these solids were largely in the form of separating pigmentation material.

The solids content test results for type P-2 curing compound are presented in Table 9.42. The P-2 curing compound visual test results are presented in Figs 9.33, 9.34, 9.35, and 9.36.

TABLE 9.41. RESULTS OF SDHPT TESTS ON TYPE P-1D CURING COMPOUND

Compound	Age at Test (mo.)	Vertical* Spray	Drying Time (min.)	Flash Temperature (°F)	Solids Content (percent)	Specific Gravity	24 Hour Moisture Loss (percent)	72 Hour Moisture Loss (percent)
Base Line Tests								
No. 1	--	S	50	98	43.67	0.885	1.44	2.66
No. 2	--	S	--	--	--	--	1.29	2.36
No. 3	--	S	--	--	--	--	1.28	2.41
No. 4	--	S	--	--	--	--	1.48	2.81
No. 5	--	S	--	--	--	--	1.30	2.78
9 Month Tests								
Drum 1	12	S	45	101	43.09	0.889	0.52	0.78
Drum 2	12	S	40	103	43.76	0.889	0.54	0.80
Drum 3	12	S	65	111	48.26	0.901	1.85	2.32
12 Month Tests								
Drum 1	14	S	50	105	43.02	0.884	0.66	0.83
Drum 2	14	S	50	110	44.90	0.883	0.63	1.09
Drum 3	14	S	45	100	41.35	0.885	0.38	0.88
Drum 4	14	S	45	96	42.67	0.884	0.60	0.77

* Results are reported as "S" (satisfactory) or "U" (unsatisfactory)

TABLE 9.42. SOLIDS CONTENT (PERCENT) OF P-2 CURING COMPOUND SAMPLES

		Before Agitation			After Agitation			
	Drum No.	Top	Middle	Bottom	Top	Middle	Bottom	Agitation Time (Min.)
Three Months	1	22.79	35.26	60.14	35.58	32.59	46.48	30
Six Months	1	--	42.60	51.05	--	47.36	--	10
	2	--	49.27	59.13	--	47.02	--	10
Nine Months	1	20.30	51.12	59.63	--	47.55	--	15
	2	22.44	52.34	58.66	--	46.91	--	15
	3	21.99	43.19	60.32	--	45.67	--	15
Twelve Months	1	20.88	42.36	56.89	--	48.39	--	15
	2	23.35	57.92	57.46	--	47.95	--	15
	3	22.68	34.53	59.01	--	48.19	--	15
	4	22.69	36.55	61.46	--	38.62	--	15

Base Line Solids = 46.48%

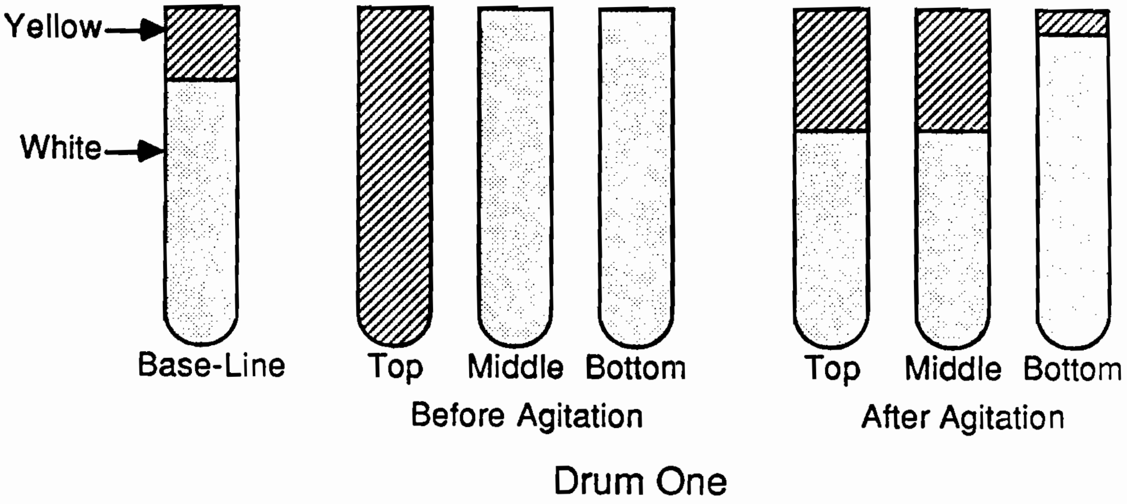
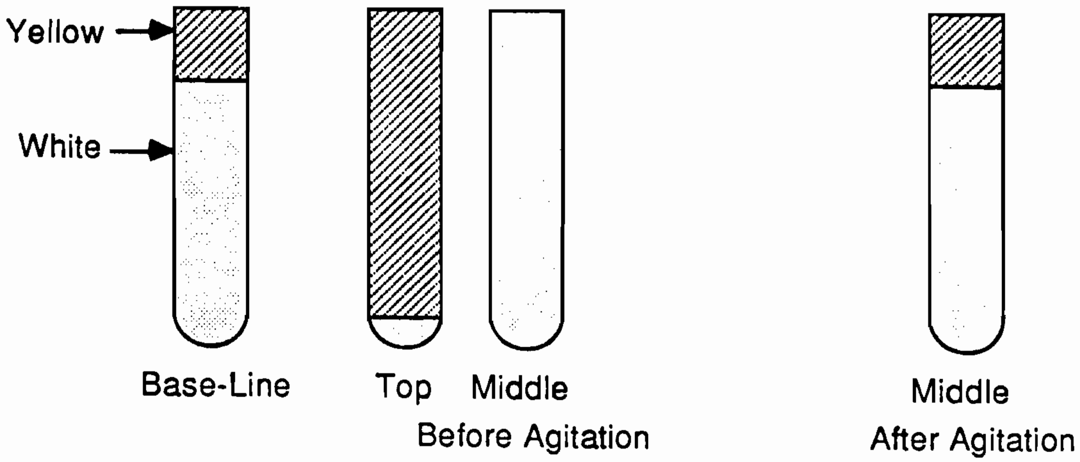
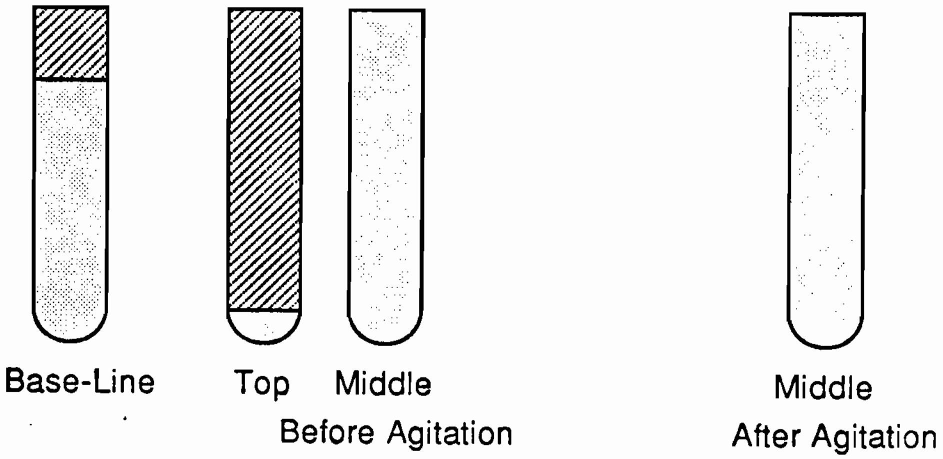


Fig 9.33. 3-month P-2 shelf-life visual tests.



Drum One



Drum Two

Fig 9.34. 6-month P-2 shelf-life visual tests.

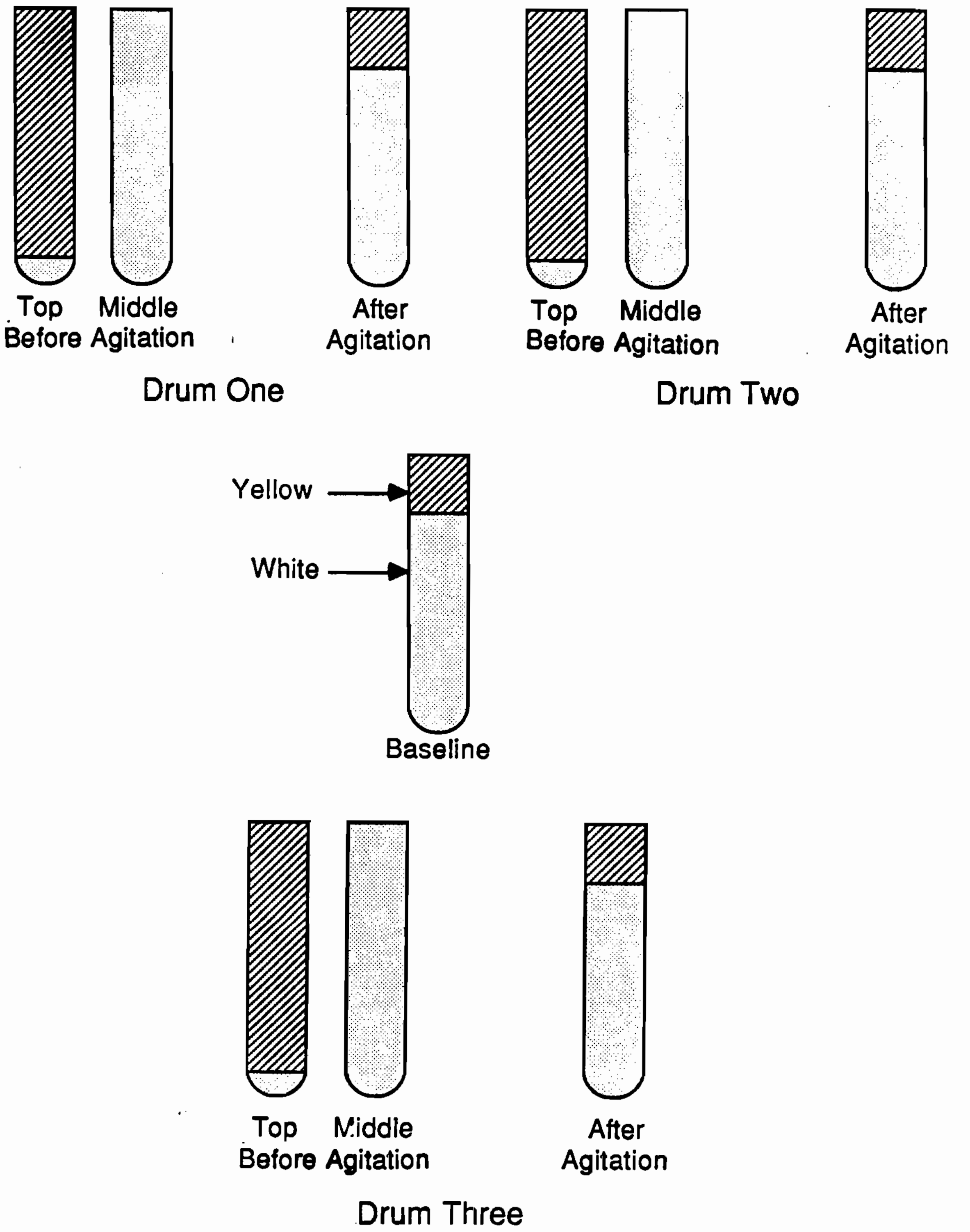


Fig 9.35. 9-month P-2 shelf-life visual tests.

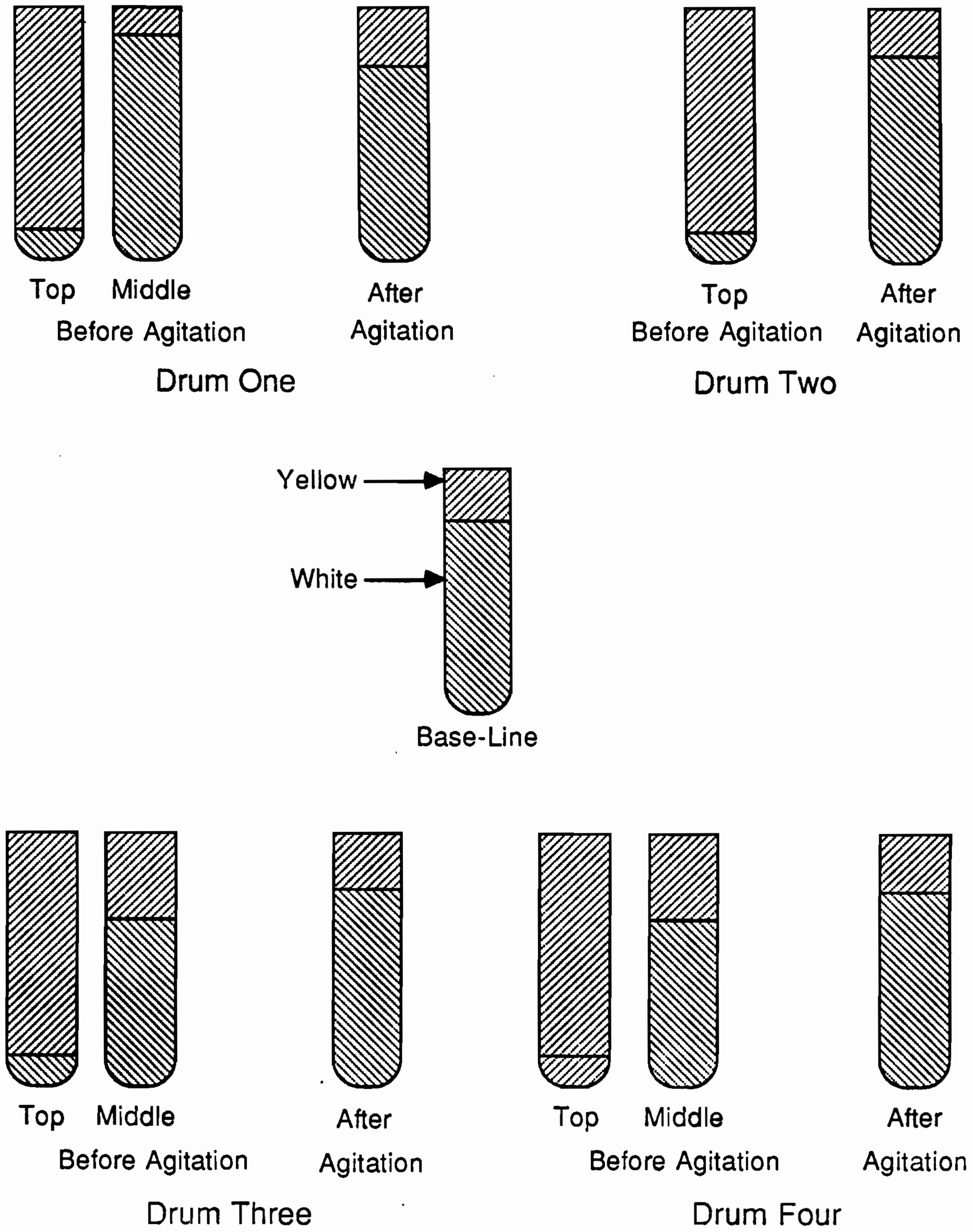


Fig 9.36. 12-month P-2 shelf-life visual tests.

The three month tests indicated that the agitation procedure was not complete. The three month P-2 samples were among the earliest taken, and the most effective agitation procedure had not yet been completely developed, or adequately practiced, when these samples were taken. The inadequate agitation was evident through three observations. First, post-agitation solids content values for the three locations in the drum were not equivalent. Second, the post-agitation middle sample solids content did not match the base line samples content values. Finally, the visual test indicated that the bottom third of the drum contained a significantly higher proportion of settled white pigmentation solids than did the top or middle samples.

By the time the six month samplings were performed, the agitation procedure had been perfected, as was evident in the results of the six month solids content and visual tests. The solids content tests indicated a higher solids content in the middle and bottom layers of drum number two than in drum number one. This was a possible indication of a greater degree of settling in the previously undisturbed drum than in the drum that had been agitated at both three as well as at six months. Despite this, both drums were agitated to virtually identical mid-drum solids content values. These values, in turn, were approximately equivalent to the base-line value.

At six months, the visual tests was somewhat less helpful. Pre-agitation bottom third visual samples could not be prepared because the material in this region was far too viscous to be poured into the glass test tube. The samples that were prepared, however, were virtually identical for the two drums. Before agitation, the top layers both were almost entirely made up of the yellow vehicle material. The pre-agitation middle layer samples, on the other hand, contained a large amount of white pigment which separated out, leaving an extremely shallow layer of the yellow vehicle at the top of the sample. After agitation, the middle sample from drum one separated into a visual twin of the base line sample, while the drum two middle sample consisted of white pigment almost exclusively.

The nine month solids content tests contradicted some of the observations made three months earlier. Drums one and two, both agitated previously, behaved similarly, but their middle sample had a higher solids content than the middle sample from the previously unagitated third drum. Since all three bottom samples had virtually the same solids content, it appeared that solids moved to the top third of drum one and two by previous agitation had settled into the middle layer, but not yet to the bottom. In any case, agitation thoroughly mixed all three drums in fifteen minutes, as can be seen in the after agitation middle layers solids contents, all three of which approximated the base-line value.

In terms of the visual test, all three drums behaved similarly to each other and to the six month tests described above. Again, bottom samples before agitation could not be prepared, middle samples were largely white pigment, and top samples were almost exclusively the yellow vehicle material. Also duplicating six month results, the middle samples taken after agitation all showed separation patterns duplicating that of the base line sample.

After the final three months of the year-long project term had elapsed, some interesting observations were made. In the earlier solids content tests, some apparent residual effects of earlier agitation had been observed. At twelve months, however, the three drums that had been agitated three months previously showed no real pattern of solids content. While top and middle samples from three drums were fairly consistent, the middle samples produced completely different solids content values. These three drums were, however, agitated to a "correct" mid-drum solids content value in 15 minutes. The fourth drum exhibited a more typical pattern of pre-agitation solids content values, with these values increasing with depth. However, after agitation, the middle sample solids content was significantly below the expected base line value of 46.48 percent. There was no explanation for this occurrence, since the drum appeared completely agitated, based on other observations, including the results of the visual tests.

These visual tests, like the solids content tests, revealed a distinct lack of any patterned behavior. Before agitation, drum one behaved as the earlier P-2 visual tests had, showing a top layer of primarily yellow vehicle and a middle layer primarily of white pigment material. In the second drum, the top layer was typical, but both the middle and bottom layers were too thick to be poured into the test tubes. The third and fourth drum test specimens behaved in virtually identical fashion before agitation. Top samples were, again, almost entirely yellow vehicle. Middle samples, while still mostly pigment material, contained a significant proportion of yellow vehicle.

After agitation, mid-drum samples from all four drums appeared virtually identical to each other and to the base line samples. Adequate agitation is thus indicated in all four drums, and no explanation for the solids content deficit in drum four can be provided.

The results of SDHPT tests on P-2 type curing compound are summarized in Table 9.43. This material behaved in a fashion very similar to the type P-2 compound. In this case, however, the results were somewhat more regular. After nine months of storage, all properties of the sample specimens, except flash temperature, were well within the values associated with their original material. The flash temperature changes were all increases, however, which is actually a desirable change. The nine month tests, then, do not suggest any damage to the type P-2 compound caused by storage of this duration.

The findings of the twelve month P-2 shelf life samples were virtual duplicates of the nine month results. All SDHPT acceptability criteria were satisfied for all four drums. Again, moisture retention rates were similar to original sample results. In fact, drums one, two, and four showed slightly better moisture retention properties than the original sample. Drum three, previously agitated once, was markedly superior in its moisture retention performance. Again, this behavior is not explainable based on other test results, but the changes are beneficial, so they do not affect the acceptability of the material under SDHPT criteria.

M-1D Curing Compound

The M-1D curing compound, like the P-1D, proved very easy to agitate. The M-1D compound did, however, separate into distinct layers, making confirmation of adequate agitation using the employed test procedures somewhat more conclusive. M-1D solids content test results are presented in Table 9.44.

TABLE 9.43. RESULTS OF SDHPT TESTS ON TYPE P-2 CURING COMPOUND

Compound	Age at Tast (mo.)	Vertical* Spray	Drying Time (min.)	Reflectivity	Flash Temperature (°F)	Solids Content (percent)	Specific Gravity	24 Hour Moisture Loss (percent)	72 Hour Moisture Loss (percent)
Base Line Tests									
No. 1	--	S	40	65.2	90	46.48	1.013	1.21	2.23
No. 2	--	S	--	--	--	--	--	0.55	1.08
No. 3	--	S	--	--	--	--	--	1.02	1.92
No. 4	--	S	--	--	--	--	--	0.82	1.56
No. 5	--	S	--	--	--	--	--	0.75	1.59
9 Month Tests									
Drum 1	12	S	90	66.4	118	53.72	1.037	1.18	1.38
Drum 2	12	S	40	65.0	114	48.97	1.015	0.94	1.34
Drum 3	12	S	60	64.0	113	48.03	1.014	0.81	1.27
12 Month Tests									
Drum 1	14	S	55	65.0	94	48.18	1.015	0.68	1.16
Drum 2	14	S	55	67.3	93	48.10	1.017	0.75	1.29
Drum 3	14	S	60	66.3	110	47.14	1.009	0.33	0.60
Drum 4	14	S	75	56.2	100	39.35	0.921	0.84	1.39

* Results are reported as "S" (satisfactory) or "U" (unsatisfactory)

TABLE 9.44. SOLIDS CONTENT (PERCENT) OF M-1D CURING COMPOUND SAMPLES

		Before Agitation			After Agitation			
	Drum No.	Top	Middle	Bottom	Top	Middle	Bottom	Agitation Time (Min.)
Three Months	1	30.77	30.36	0.97	22.57	22.75	22.43	30
Six Months	1	33.64	31.54	0.62	--	22.72	--	5
	2	39.60	31.05	0.60	--	22.81	--	5
Nine Months	1	34.92	29.06	0.59	--	21.81	--	5
	2	34.93	30.87	0.55	--	21.99	--	5
	3	38.10	32.20	0.31	--	22.07	--	5
Twelve Months	1	37.42	35.07	0.46	--	23.24	--	5
	2	37.90	34.70	0.69	--	23.19	--	5
	3	38.89	33.12	0.64	--	23.38	--	5
	4	40.77	27.93	0.58	--	23.12	--	5

Base Line Solids = 22.39%

At three months of age, drum number one showed solids content values in the top and middle layers that were approximately equal, but somewhat higher than the known baseline value of 22.30 percent. These were offset, however, by the solids content of the lower layer, which had a tested solids content of less than 1 percent. Visual testing as presented in Fig 9.37 both confirmed and explained these results. Prior to agitation, the top and middle samples appeared very similar, though the top sample contained a slightly higher proportion of the red watery fluid, while the middle sample was more heavily composed of more viscous light pink compound component. The bottom sample, however, showed almost no indication of solids, consisting almost entirely of water.

After agitation, solids content values and visual separation patterns from all three locations in the drum approximated the base line solids content value and the separation pattern of the base line sample. This separation pattern consisted of a thin layer of the red fluid over a large volume of the viscous lighter pink material. The absence of a water layer could not be explained except for the possibility that the slenderness of the glass tube aided the viscous compound component in trapping water within its mass. However, there was no detectable lightening of color to confirm this suspicion.

Finally, it should be noted that the 30 minute agitation time was employed only due to uncertainty at this time in the project, and proved unnecessarily long.

At six months, both solids content and visual testing indicated some residual effects of previous agitation. Top, middle, and bottom samples from drum one appeared virtually identical to those taken three months earlier. As represented in Fig 9.38, however, drum two, which had been undisturbed for six months, provided distinctly different samples. The bottom layer was still almost water, and the middle layer was still a combination of the two pink components, heavily weighted in favor of the light pink viscous material. The top layer, however, consisted almost entirely of the red, watery material. This material seemed to have a relatively high solids content, as seen in Table 9.44.

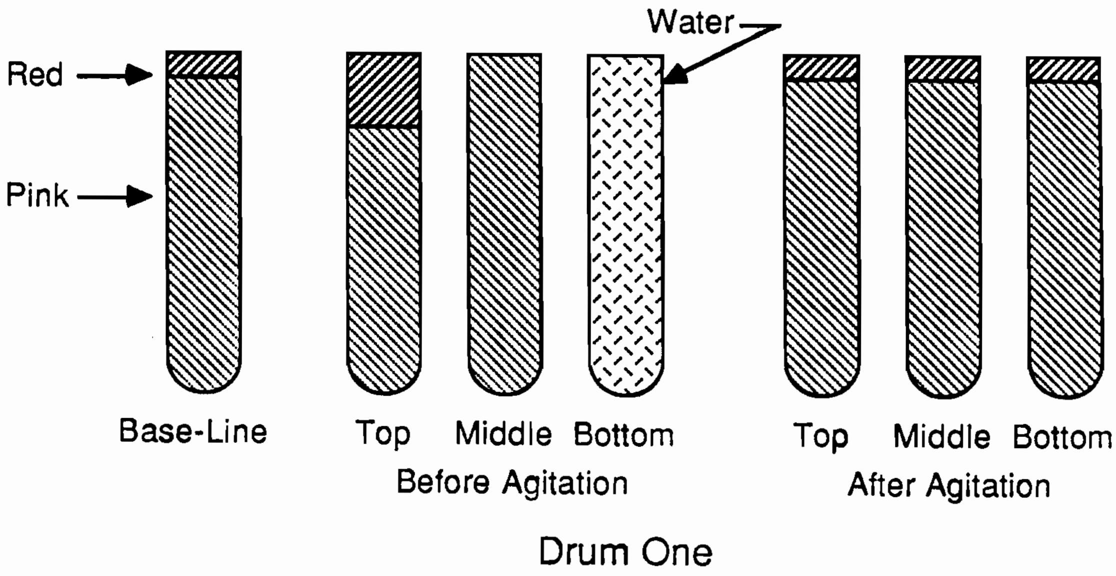
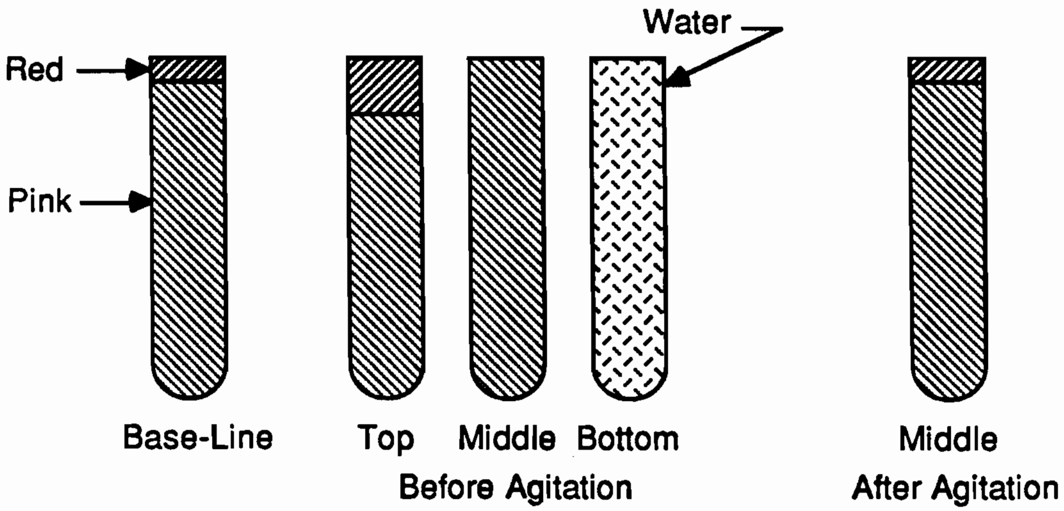
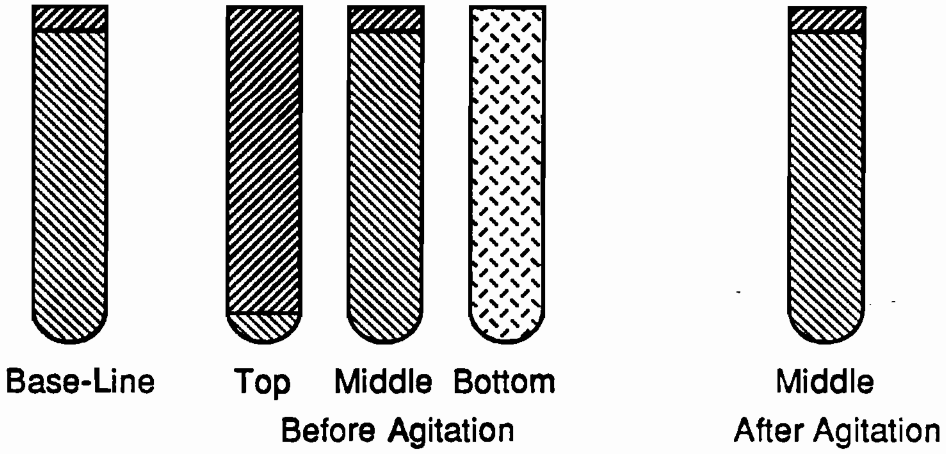


Fig 9.37. 3-month M-1D shelf-life visual tests.



Drum One



Drum Two

Fig 9.38. 6-month M-1D shelf-life visual tests.

While the bottom and middle samples from both drums agreed very well, the top layer of drum two had a solids content significantly higher than the top layer of drum one, which behaved as it had three months earlier, approximately duplicating the solids content value of the middle layer.

Both drums were agitated to solids content rates and visual separation patterns approximating those of the base line sample in 5 minutes. Thus, the apparent residual effect of the earlier agitation did not affect ease of agitation three months later.

The apparent residual effect of earlier agitation was confirmed by the nine month tests. In terms of solid content rates, drums one and two followed the pattern of previously agitated drums set by drum one, three months earlier. Drum three, not previously agitated, followed the pattern of high top layer solids content set by drum two at six months. As displayed in Fig 9.39, visual test results confirmed solids content observations. In the two previously agitated drums, top and middle samples were almost identical in terms of separation pattern. Drum three, however, supplied a top sample consisting almost exclusively of the red compound component. This was the same behavior exhibited by drum two three months earlier, when it had not been previously agitated.

As before, all three drums were agitated to approximate baseline solids content values and visual separation pattern in only 5 minutes. Again, effects of previous agitation, though evident, did not appear to shorten final agitation time.

After the full twelve month storage period had elapsed, some variation from the expected pre-agitation behavioral pattern was noted. In terms of solids content values, the top sample from the previously unagitated drum, number four, displayed the expected high solids content rate. However, this value was approximated by the top samples from the other three drums as well. Based on earlier data, these would have been expected to display solids contents 4 to 6 percent below that in drum four.

The analysis of twelve month middle samples resulted in something of a surprise. Earlier tests had indicated similar behavior in pre-agitation middle samples from drums that had been previously agitated and those that had not. At twelve months, however, the three previously agitated drums

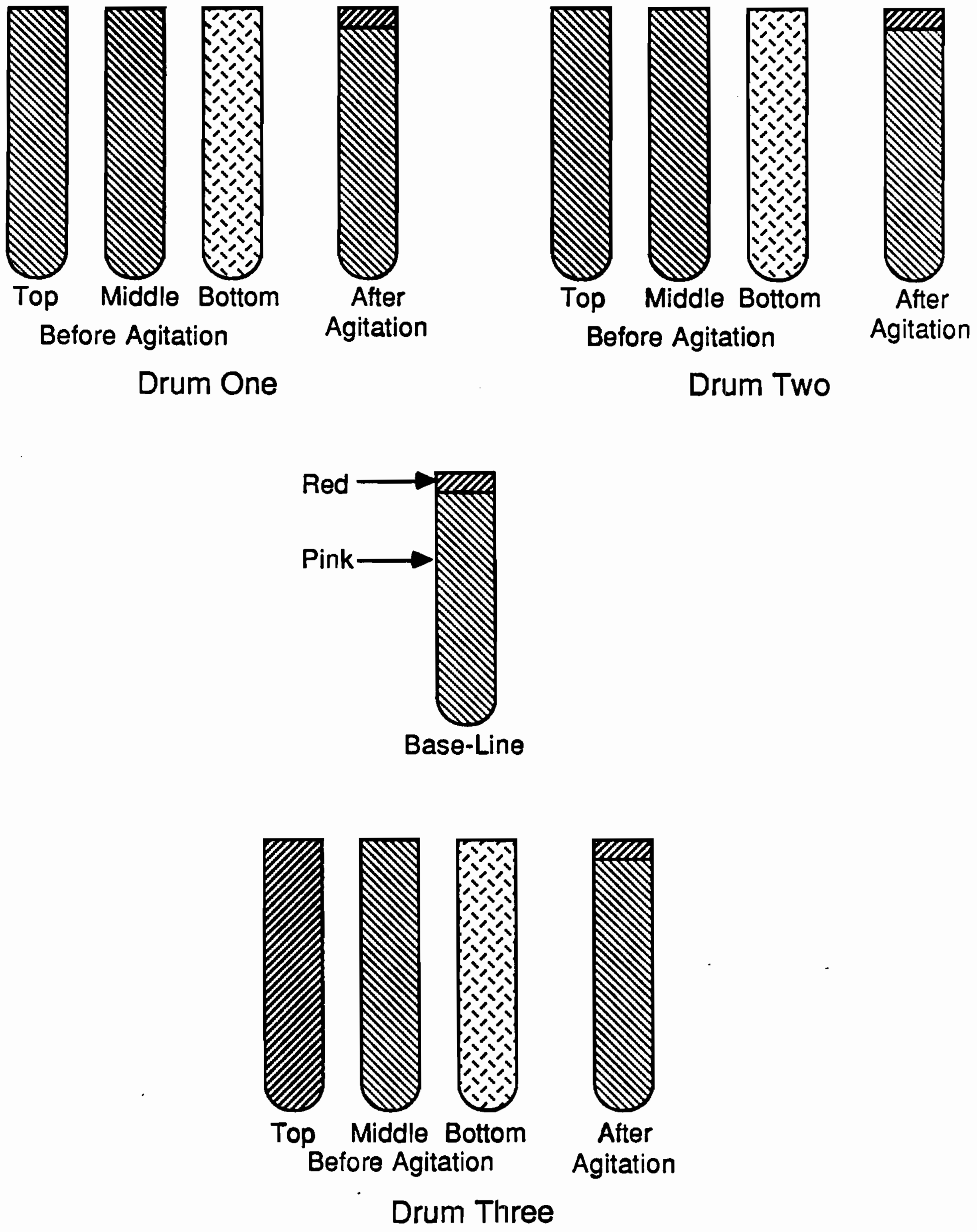


Fig 9.39. 9-month M-1D shelf-life visual tests.

produced middle samples with solids content rates slightly higher than expected. Drum four, however, produced a middle sample of unexpectedly low solids content. The only explanation provided for these irregularities is the possibility of some odd variation in compound separation pattern that was adequately described by the three specific locations sampled.

As presented in Fig 9.40, twelve month visual test results were exactly what would have been expected. Visual samples from drums one, two, and three followed the pattern of previously agitated drums with similar top and middle samples, the top samples containing a higher proportion of the red material. The previously unagitated fourth drum, however, supplied the expected top sampleconsisting almost entirely of the red compound component. All four drums produced pre-agitation bottom samples of almost pure water, and were agitated to uniformity in 5 minutes.

The patterned effect of previous agitation was thus confirmed. It should be noted that, at three months, drum one samples conform to this pattern, realizing that this compound was, in fact, "agitated" during manufacture at the plant (at zero months of age). Importantly, the effect of previous agitation was clear, but even after twelve months, material that had not been agitated at intermediate times was no more difficult to agitate than material that had been agitated three months earlier.

The SDHPT test procedures on the M-1 compound yielded the results presented in Table 9.45. With regard to the nine month shelf life tests, the only sample found unsatisfactory was that from drum number one (the drum that had been agitated for a total of four times during the year of storage). The problem was with the vertical spray test, which proved unsatisfactory. Most other test results were both satisfactory and closely in line with the original samples. The only exception to this was that all three drums showed 24 hour moisture losses significantly higher than those shown by the base line material. It should be recognized that the elevated values were still only twenty-five percent as great as the allowable maximum of two percent loss in 24 hours. Further, the 72 hour moisture loss rates were not noticeably different from the values found in the original material. Finally, all three nine-month samples showed longer drying times than the

TABLE 9.45. RESULTS OF SDHPT TESTS ON TYPE M-1D CURING COMPOUND

Compound	Age at Test (mo.)	Vertical* Spray	Drying Time (min.)	Flash Temperature (°F)	Solids Content (percent)	Specific Gravity	24 Hour Moisture Loss (percent)	72 Hour Moisture Loss (percent)
Base Line Tests								
No. 1	--	S	60	110	23.39	0.918	0.29	1.51
No. 2	--	--	--	--	--	--	0.26	0.73
No. 3	--	--	--	--	--	--	0.23	0.69
No. 4	--	--	--	--	--	--	0.30	0.97
No. 5	--	--	--	--	--	--	0.23	1.03
9 Month Tests								
Drum 1	12	U	110	112	22.98	0.927	0.56	0.90
Drum 2	12	S	95	118	23.70	0.935	0.44	0.70
Drum 3	12	S	75	115	23.29	0.936	0.50	0.87
12 Month Tests								
Drum 1	13-14	S	35	95	22.22	0.921	0.14	0.34
Drum 2	13-14	S	40	98	22.49	0.920	0.22	0.47
Drum 3	13-14	S	35	110	22.32	0.922	0.30	0.67
Drum 4	13-14	S	40	110	22.22	0.923	0.38	0.81

* Results are reported as "S" (satisfactory) or "U" (unsatisfactory)

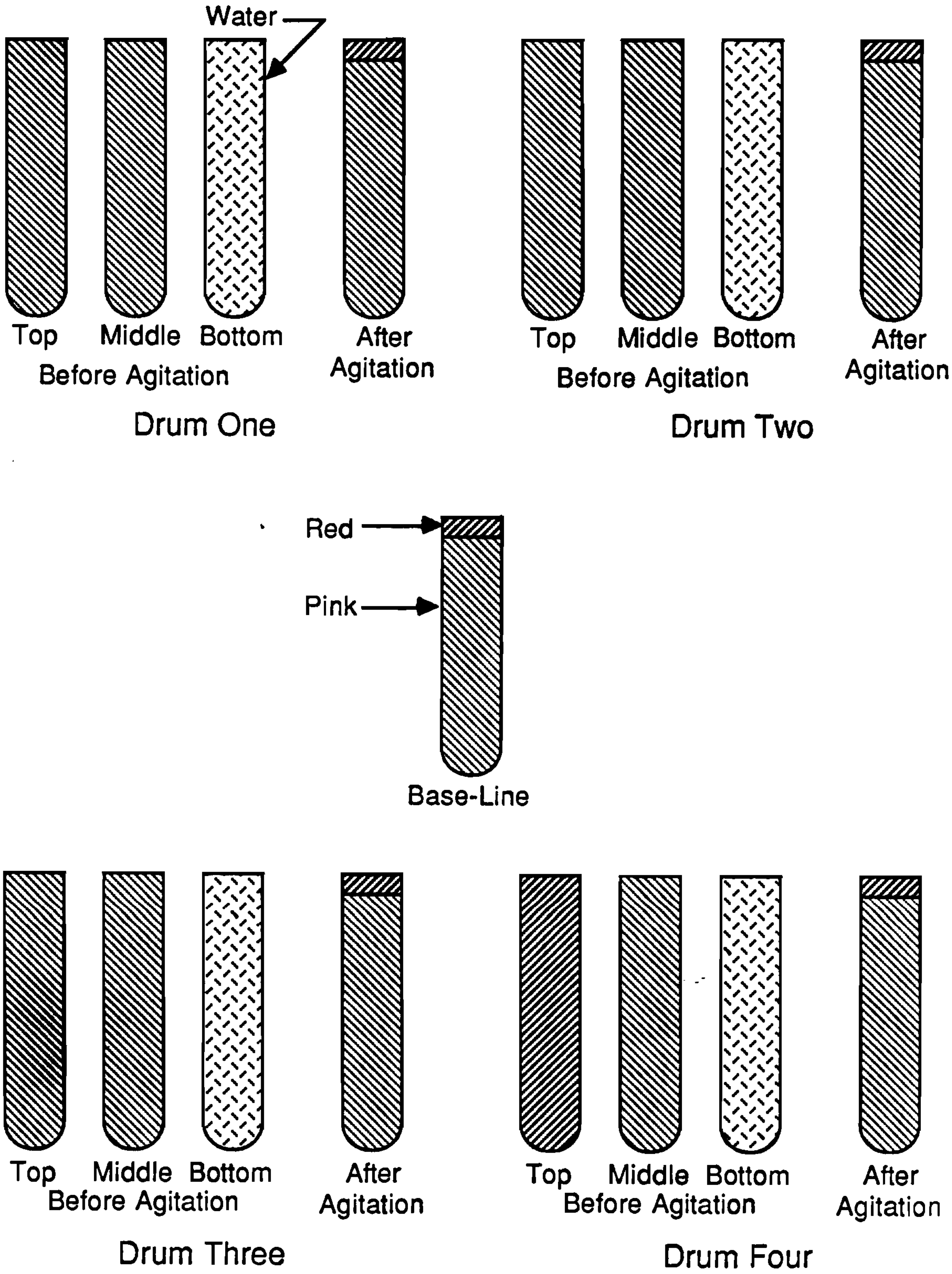


Fig 9.40. 12-month M-1D shelf-life visual tests.

original sample. Again, these drying times were still unsatisfactory, none being more than half of the four hour allowable. Furthermore, the SDHPT drying time test tends to result in highly variable results due to the nature of the test. Thus, apparently significant changes do not necessarily carry any importance.

At twelve months of age, the unsatisfactory vertical spray behavior of the material from drum number one that had appeared at nine months was no longer evident. In fact, the results of all tests indicated a material as good or better than the original product. Flash temperature, solids content and specific gravity all remained consistent with those of the original material. Drying time, however, was reduced (given the aforementioned qualifications to the significance of this statement, and 72 hour moisture loss rates also reduced slightly. The ranges of 72 hour twelve month moisture loss rates and those of the original material did overlap significantly, however, This, combined with the great degree of similarity between twelve month and original sample 24 hour moisture loss results indicated no reason to confidently conclude improved moisture retention capabilities from these tests.

M-2 Curing Compound

Results from M-2 solids content testing are presented in Table 9.46. M-2 compound was the only one of the six tested to exhibit deterioration prior to the first agitation. This deterioration took the form of translucent granules, about the size of table salt crystals, in the middle and bottom pre-agitation three month sample. These granules were not re-dissolved with agitation, and were of sufficient size to cause a blockage of typical spray mechanisms, rendering the compound unusable. Based purely on this observation, it appeared that the appropriate shelf life for this material was something less than three months. For the sake of rigor, however, solids content and visual test results were examined and analyzed.

The three month tests suffered from the inexperience of the experimenters. The agitation procedure had not yet been perfected, and so the drum was not completely agitated even after 30 minutes. The solids

TABLE 9.46. SOLIDS CONTENT (PERCENT) OF M-2 CURING COMPOUND SAMPLES

	Drum No.	Before Agitation			After Agitation			Agitation Time (Min.)
		Top	Middle	Bottom	Top	Middle	Bottom	
Three Months	1	--	48.03	22.58	44.22	47.83	41.18	30
Six Months	1	42.97	47.86	25.02	44.93	45.45	43.46	10
	2	42.55	45.24	39.64	43.10	43.51	43.66	10
Nine Months	1	39.87	44.21	26.50	41.64	41.14	41.60	5
	2	40.38	45.39	43.51	41.83	41.14	41.59	5
	3	40.36	42.16	38.81	42.94	42.06	43.37	20
Twelve Months	1	40.74	48.17	25.53	--	41.27	--	10
	2	39.90	50.21	42.87	--	39.51	--	10
	3	38.97	44.43	43.14	--	41.09	--	10
	4	39.95	43.74	40.76	--	41.47	--	20

Base Line Solids = 41.51%

content test results were somewhat confusing. Prior to agitation, the middle sample had a solids content somewhat above the base line value of 41.51 percent, while the bottom sample solids content was significantly below this value. After agitation, top and middle solids contents were about equal, while the bottom level value was somewhat lower than the other two. The visual test results are shown in Fig 9.41. Before agitation, high viscosity prevented preparation of middle or bottom layer visual test specimens. Even the top layer sample was mostly thick white pigment, topped by only a thin layer of yellow vehicle material.

After agitation, the solids content behavior was explained by visual test results. Top and middle samples were almost identical. The bottom sample contained a slightly larger proportion of pigmentation solids. The lower solids content of this sample was explained by the fact that the liquid in the bottom sample was water, contributing no solids, instead of the solid bearing yellow vehicle in the top and middle samples.

At six months, the proper agitation procedure had been developed and practiced. Test results reflected this fact. Before agitation, drum one behaved almost the same as it had three months earlier. Drum two, however was like drum one on top and in the middle, but had a much higher solids content in the bottom layer. This was reflected in the visual test (see Fig 9.42). Top and middle layers in the two drums showed virtually identical separation patterns, while the bottom sample from drum two contained significantly less water than did the bottom sample from drum one.

After agitation, all samples from both drums were virtually identical. In terms of solids content, both drums were roughly uniform and approximated the base line value. Visual tests confirmed these observations. Again, all samples from both drums appeared virtually identical, and were very similar to the baseline sample separation pattern.

At nine months, there arose some reason to believe that drum number one was atypical. Before agitation, drums two and three both behaved much as drum two had three months earlier, displaying very close solids content values even before agitation. Drum one, however, showed the same low bottom layer solids content it had on previous test dates. Visually, as seen in Fig 9.43, drum three displayed a bottom layer with a much greater proportion of

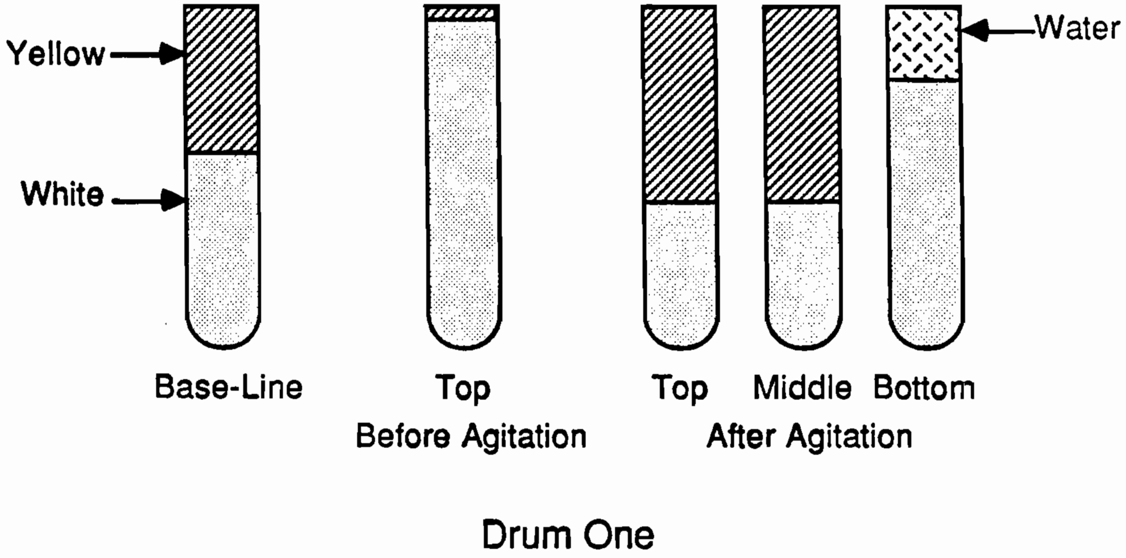
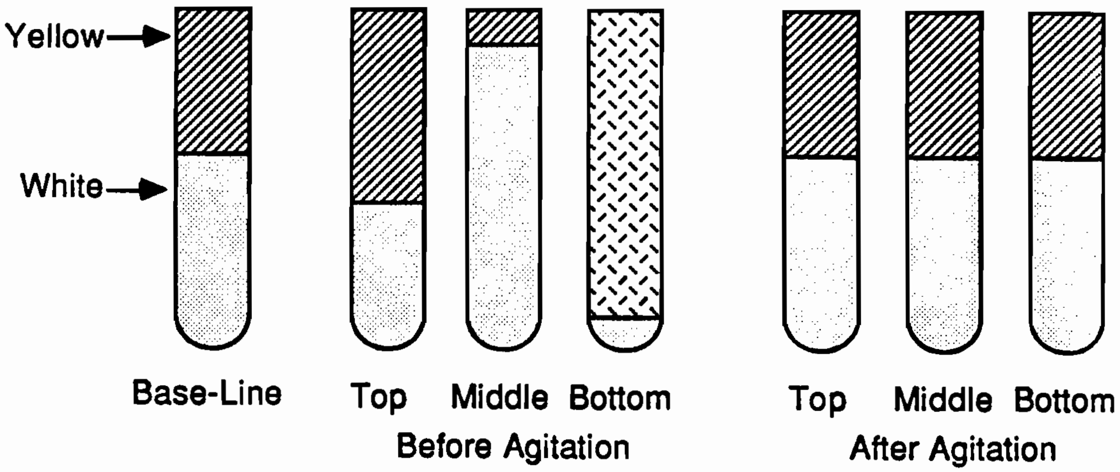
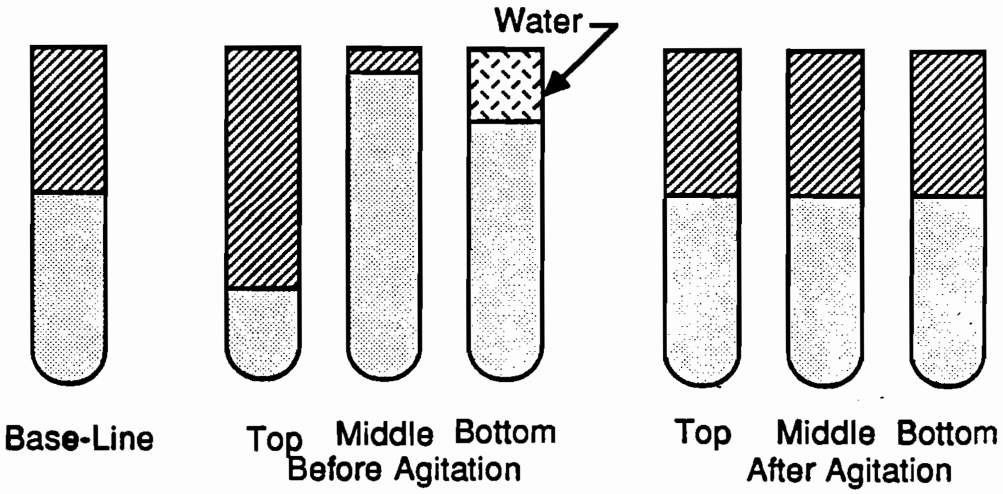


Fig 9.41. 3-month M-2 shelf-life visual tests.



Drum One



Drum Two

Fig 9.42. 6-month M-2 shelf-life visual tests.

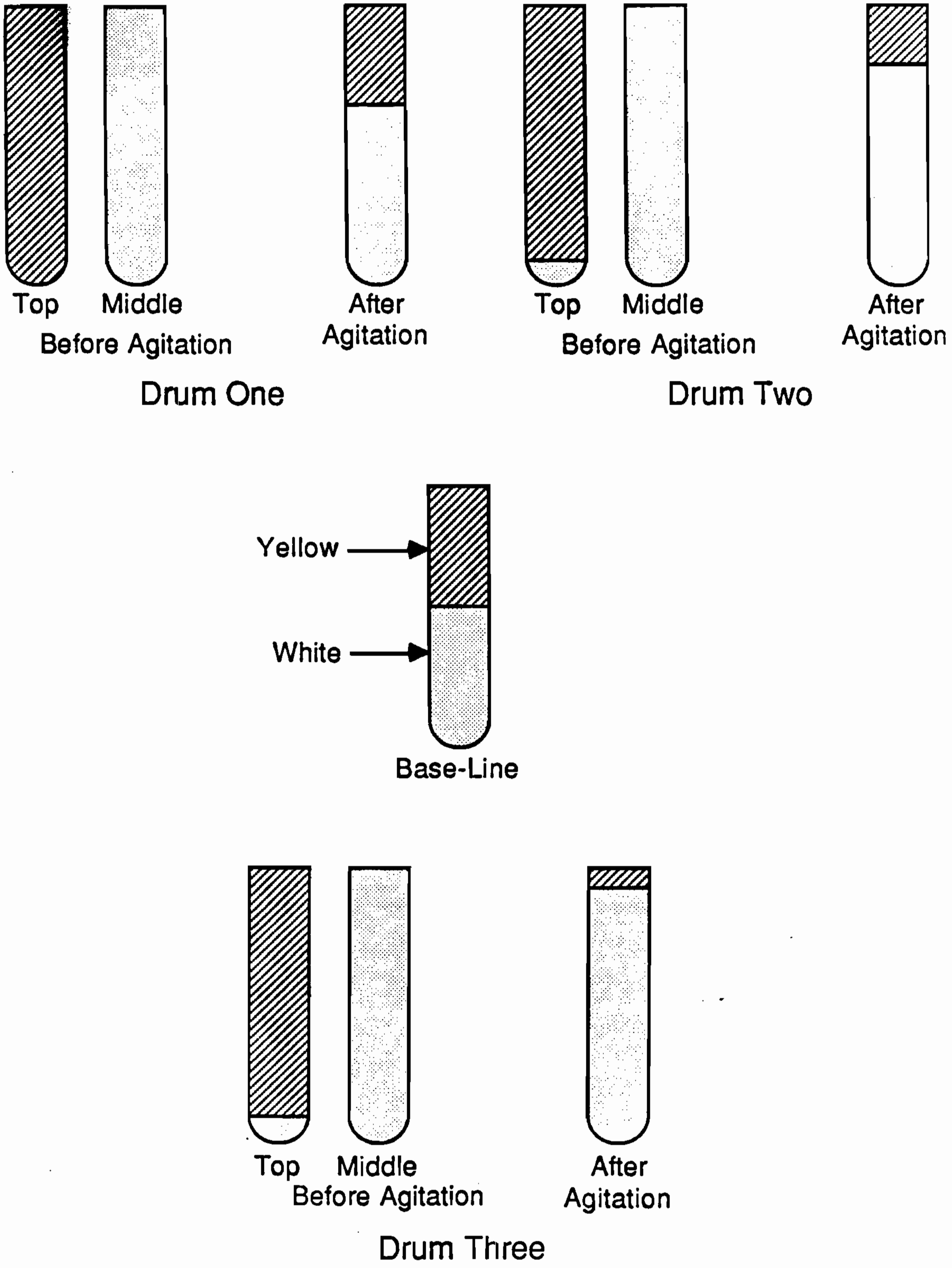


Fig 9.43. 9-month M-2 shelf-life visual tests.

pigments than the bottom layers of drums one and two. This, of course, contradicts solids content data, instead of explaining it.

Despite the apparent irregularity of drum one, it, and the other two drums, were all agitated to uniformity, and test results confirmed base line solids content and visual separation pattern except for a tendency for slightly higher proportions of pigmentation material. It should be noted, however, that the previously agitated drums received only 5 minutes of agitation, while drum three required 20 minutes.

The twelve month test battery confirmed all of the observations made at nine months. Before agitation, drums two, three, and four all exhibited the "normal" pattern of relatively constant solids throughout. Visual tests (see Fig 9.44) showed at twelve months that these approximately equivalent solids content values did not result from similar composition, however. Drum one, for the fourth time, exhibited low bottom layer solids content resulting from abnormally high water content in that layer. The visual sample could not be prepared due to the high viscosity of the material. Inspection of the material, however, did indicated the high water content mentioned.

Agitation produced results very similar to those at nine months. Again, all four drums displayed approximately base line solids content rates. Visually, the separation pattern was as it had been at nine months, showing a white pigment layer topped by a yellow vehicle layer from which not all the solids would separate. There was no clear explanation for this lack of separation, but it might have been an indication of the deterioration of the compound, as the granules described above had been found in all tests of material from all four drums. Finally, it should be noted that drum four required 20 minutes of agitation, while the three drums that had been agitated three months earlier required only 10 minutes.

Following these tests by CTR, SDHPT performed its standard battery of curing compound tests on type M-2 shelf life samples. The results of these tests are recorded in Table 9.47.

At nine months, all three drums sampled showed reflectivity and solids content increases of significant proportions. These finding would suggest the possibility of superior moisture retention capabilities. This suspicion

TABLE 9.47. RESULTS OF SDHPT TESTS ON TYPE M-2 CURING COMPOUND

Compound	Age at Test (mo.)	Vertical* Spray	Drying Time (min.)	Reflectivity	Flash Temperature (°F)	Solids Content (percent)	Specific Gravity	24 Hour Moisture Loss (percent)	72 Hour Moisture Loss (percent)
Base Line Tests									
No. 1	--	S	90	69.7	120	41.51	1.06	0.84	2.16
No. 2	--	--	--	--	--	--	--	0.91	1.81
No. 3	--	--	--	--	--	--	--	0.98	1.87
No. 4	--	--	--	--	--	--	--	1.14	2.03
No. 5	--	--	--	--	--	--	--	0.95	1.77
9 Month Tests									
Drum 1	15	S	90	74.9	132	51.87	1.102	0.27	0.81
Drum 2	15	S	60	76.1	112	49.93	1.095	0.30	0.73
Drum 3	15	S	40	78.5	118	54.71	1.116	1.45	2.04
12 Month Tests									
Drum 1	12	S	50	72.2	107	42.63	1.074	0.50	0.90
Drum 2	12	S	50	72.7	110	40.86	1.028	0.42	0.82
Drum 3	12	S	120	73.3	115	43.13	1.086	1.01	1.59
Drum 4	12	S	75	74.5	116	44.87	1.097	0.74	1.34

* Results are reported as "S" (satisfactory) or "U" (unsatisfactory)

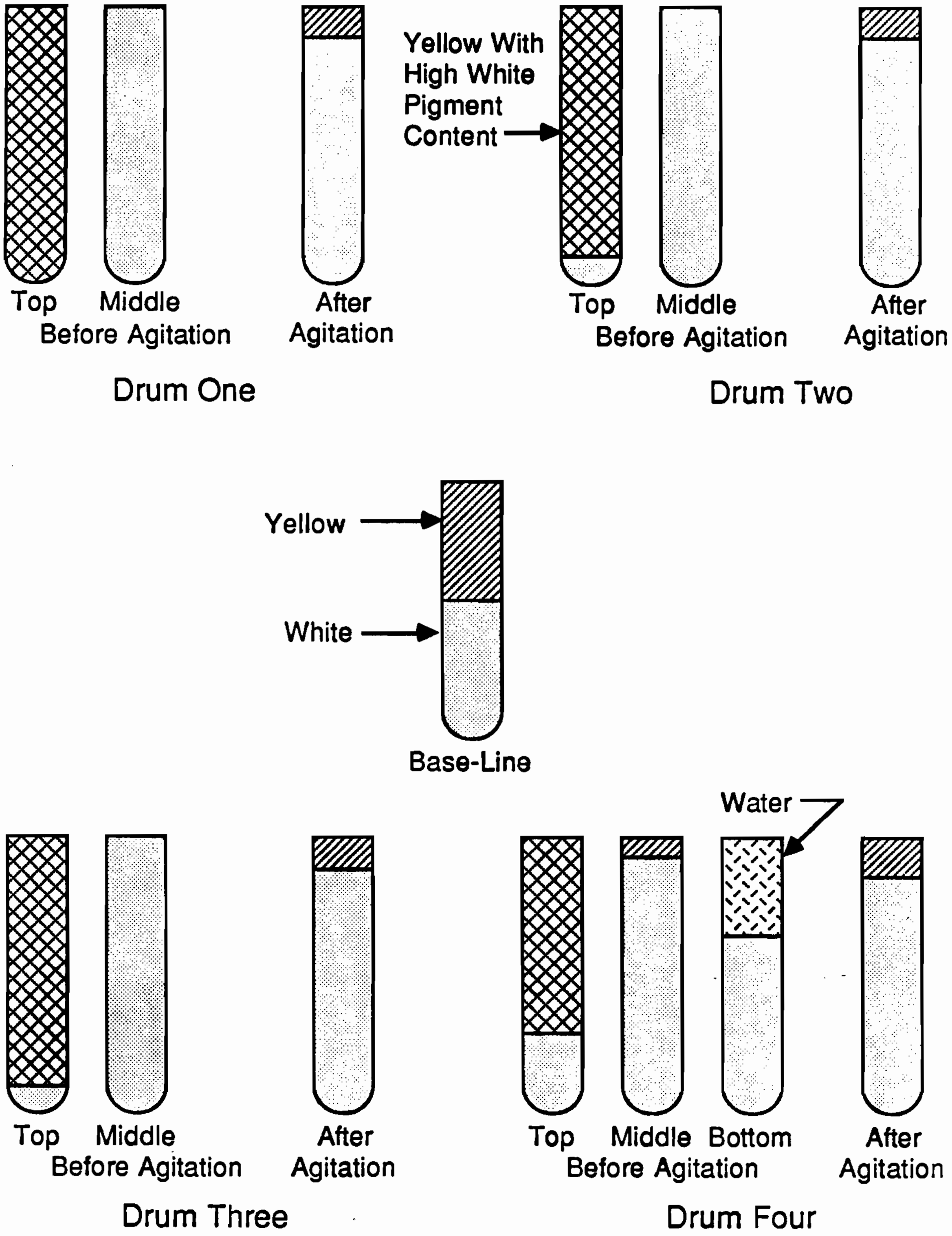


Fig 9.44. 12-month M-2 shelf-life visual tests.

was confirmed at both 24 and 72 hours by the samples from drums one and two, both previously agitated, but not by the tests on the material from drum number three, which had not been previously agitated. The results from this drum's sample were somewhat contradictory. The 24 hour loss rate was quite a bit higher than the original value for the M-2 compound, though still well within the range of acceptability. The 72 hour moisture loss value was fairly typical of base line values, however, suggesting little change in the material.

After twelve months of storage, both the reflectivity and specific gravity values reported for samples from all four drums were much closer to base line values than the nine month values had been. With regard to moisture retention test results, the 24 hour tests showed some return toward "normal" values in drums one and two. There was a similar tendency in the 72 hour tests on these two drums, but the trend was of smaller magnitude and so, less convincing.

Drum number three, which had exhibited slightly elevated 24 hour moisture loss when it was first agitated three months previously now produced a sample with both 24 and 72 hour moisture loss rates that were quite comparable to the base line values.

Drum number four, which had not been agitated previously, showed a sort of intermediate moisture loss behavior. At both 24 and 72 hours, the sample from drum four showed higher moisture losses than the abnormally low losses of the drum one and two samples. However, drum four's moisture loss values were not in the range fo the base line values, as drum number three's sample had been.

Both the nine and twelve month SDHPT tests on type M-2 compound suggest an anomaly in the material from drum number three. Despite this, none of the tests suggest any reason to reject this material for use even after twelve months of storage.

APPLICATION PATTERN

As described in Chapter 6, liquid membrane-forming curing compound is typically applied by a spray bar travelling longitudinally along the pavement. This study included a comparison of the effect on moisture retention of six alternative compound application patterns. These patterns were described in Chapter 6 as two through seven. The standard pattern of a single longitudinal pass was also described in Chapter 6, and was referred to as pattern number one.

In order to determine the most effective application pattern, statistical tests for significance were performed on the moisture loss test data for the seven different application patterns. These data are tabulated in Table 9.48.

The statistical testing employed involved both "F" tests and "t" tests for significance of differences of means for each of the six alternative application patterns (patterns two through seven) to the standard pattern (pattern one). The results of these statistical tests are as follows.

First, the "F" test showed no significant difference in means for any of the six alternative patterns, in comparison to the standard pattern, at any level of confidence greater than or equal to 90 percent.

The "t" test, on the other hand, showed differences in means for application patterns five and six, with a 95 percent level of confidence; pattern seven, with a confidence level of 97.5 percent, and pattern three, with a 99 percent confidence level. However, the mean moisture losses using these four alternative patterns were all higher than the moisture loss using the standard application pattern. This suggested, of course, that, though there were significant differences in those four patterns, the differences were deleterious, instead of producing the hoped-for improvement in moisture losses.

During the testing, it was noticed that the moisture loss recorded for the "standard" test (using application 1 in all cases) varied somewhat for each set of test specimens. It was decided that the test results would be normalized to the mean value of the moisture losses of the standard specimens found in tests of patterns two through six. The standard blocks from tests

TABLE 9.48. 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	1.044	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
C.V. (Percent)	15.0	17.4	14.8	42.0	8.8	13.8	19.5

of patterns one and seven were excluded from calculation of that mean. This was done because Dixon's test for exclusion of extreme values confirmed that those two values should be excluded with confidence levels of 98 and 99 percent, respectively. Because these two values were excluded, the test data accompanying them could not be normalized, and so those data were treated as if the standard specimen in the test had exhibited moisture loss equivalent to the mean of the other five standard specimens, this value being 1.254 kg/m^2. All other data were normalized to this mean by multiplying them by 1.254 kg/m^2, and then dividing them by the moisture loss found in the standard specimen that had been cured with them. In this manner, possible fluctuation in curing environment, spray bar behavior, or other factors could be corrected for.

Statistical testing of the normalized test data produced results almost identical to those found during examination of the non-normalized test data. The normalized data are presented in Table 9.49 and the statistical test results are summarized below.

The "F" test for differences of means again showed no significant difference between any of the alternate application patterns and the standard pattern.

The "t" test, once again, did not concur with the F test results. In the case of the normalized test data, pattern six produced different mean moisture loss with 90 percent confidence; pattern five was different from pattern one to a 95 percent confidence level; pattern seven was statistically different with a 97.5 percent level of confidence, and pattern three varied from pattern one with 99 percent confidence.

As with the analysis of the non-normalized data, however, all of these statistically different application patterns mean moisture losses greater than those produced when the standard pattern was employed. Thus, none of the statistically different means would be improvements on present procedure if they were employed in the field.

TABLE 9.49. NORMALIZED MOISTURE LOSS (KG/M^2) FOR DIFFERENT APPLICATION PATTERNS

	↑	→	↑↑	⇉	⇅	⇄	↑↘
Specimen	Pattern 1	Pattern 2	Pattern 3	Pattern 4	Pattern 5	Pattern 6	Pattern 7
1	1.044	0.816	1.372	1.602	1.270	1.028	1.753
2	0.775	1.145	1.797	0.696	1.078	1.357	1.399
3	0.979	0.926	1.446	0.995	1.275	1.182	1.194
Standard Deviation	0.140	0.167	0.227	0.462	0.112	0.165	0.283
Mean	0.933	0.962	1.538	1.098	1.208	1.189	1.449
C.V. (Percent)	15.0	17.4	14.8	42.1	9.3	13.9	19.5

REFLECTANCE STUDIES

As described in Chapter 7 of this report, the first step in the reflectance study was to develop a set of standard reflectance curves for the prototype reflectance meter. Four surfaces with known reflectances were employed. Those known reflectances were 2 percent, 18 percent, 82 percent, and 90 percent. Curves were developed for two conditions:

(1) reflectance meter standardized to read zero at 18 percent reflectance,

(2) reflectance meter standardized to read zero at 90 percent reflectance.

These two reflectance values were chosen because they are commercially available surfaces, available in any good photographic accessory store.

Reflectance readings were taken under different lighting conditions and these readings are presented in Table 9.50. The curves generated from these data are shown in Fig 9.45.

One should note the relatively large variation in readings that are associated with the same surface. For example, considering the values associated with the 18 percent standard curve, a reflectance reading of -150 mV could be associated with an actual reflectance of anywhere between 52 and 68 percent. See the dashed lines on Fig 9.45 for graphical explanation of this example. This uncertainty manifested itself very clearly during the experimental procedures.

The standard curve data collected for reflectance readings from different application rates of P-2 curing compound are presented in Table 9.51. Figure 9.46 shows two curves for readings standardized to 18 percent reflectance. The solid curve is merely a connection of the mean readings for the three application rates. The dashed curve was generated by linear regression of all data points. Figure 9.47 shows the same two curves generated for data standardized to 90 percent reflectance.

TABLE 9.50. REFLECTANCE READINGS (mV) FOR DEVELOPMENT OF STANDARD CURVES

Environment	Known Reflectance (Percent)	Standardized to 18 Percent Reflectance	Standardized to 19 Percent Reflectance
	2	+241	+396
Flourescent	18	000	+234
Light	82	-234	+009
	90	-249	+001
	2	+190	+328
Sunlight	18	+002	+181
(12 noon)	82	-172	+016
	90	-189	000
	2	+195	+349
Sunlight	18	-003	+188
(2 p.m.)	82	-176	+022
	90	-195	000
	2	+205	+341
Sunlight	18	+004	+198
(4 p.m.)	82	-194	+025
	90	-216	+003
	2	+192	+329
Shade	18	-001	+183
(4 p.m.)	82	-181	+015
	90	-194	000

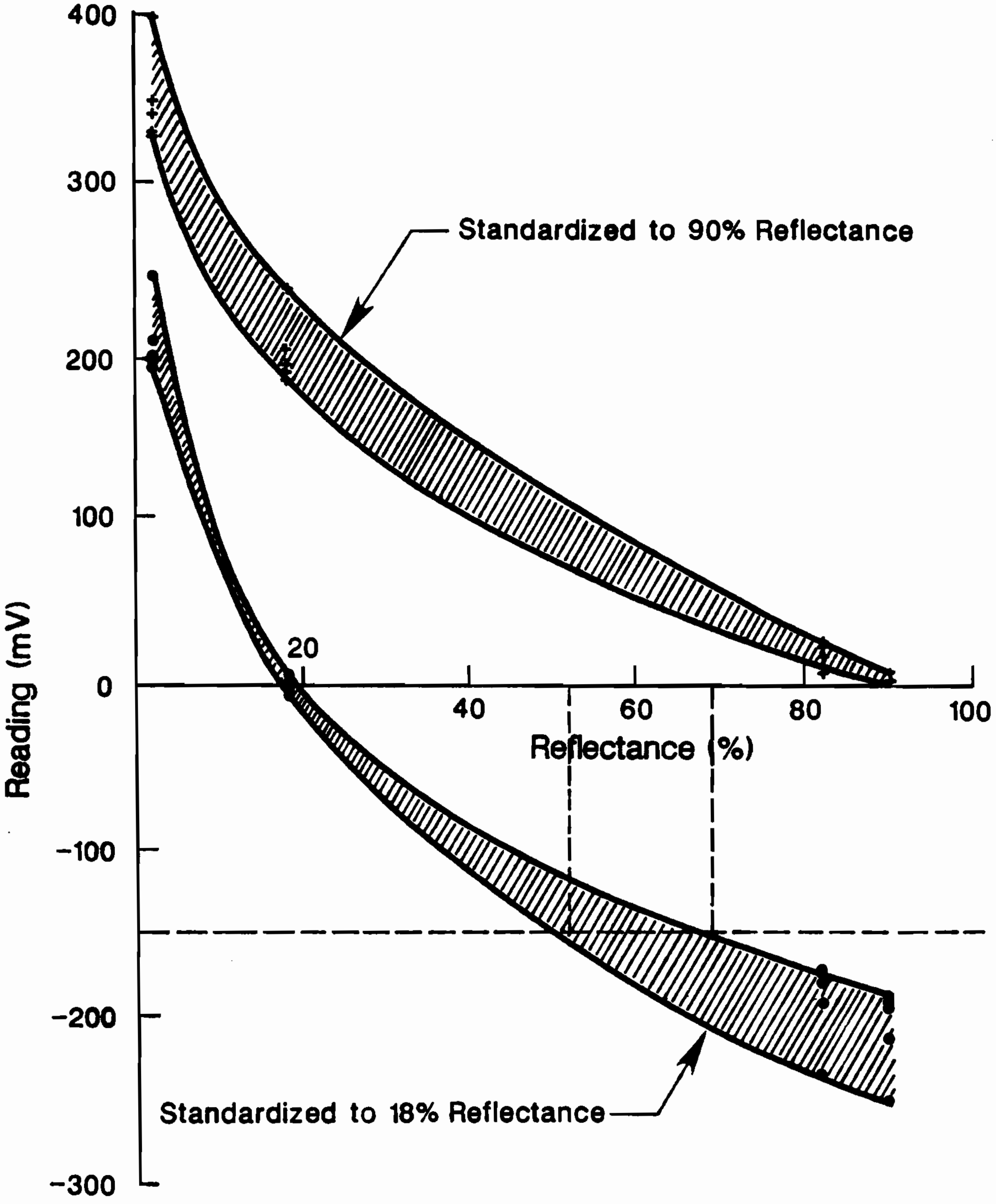


Fig 9.45. Standard reflectance curves for prototype reflectance meter.

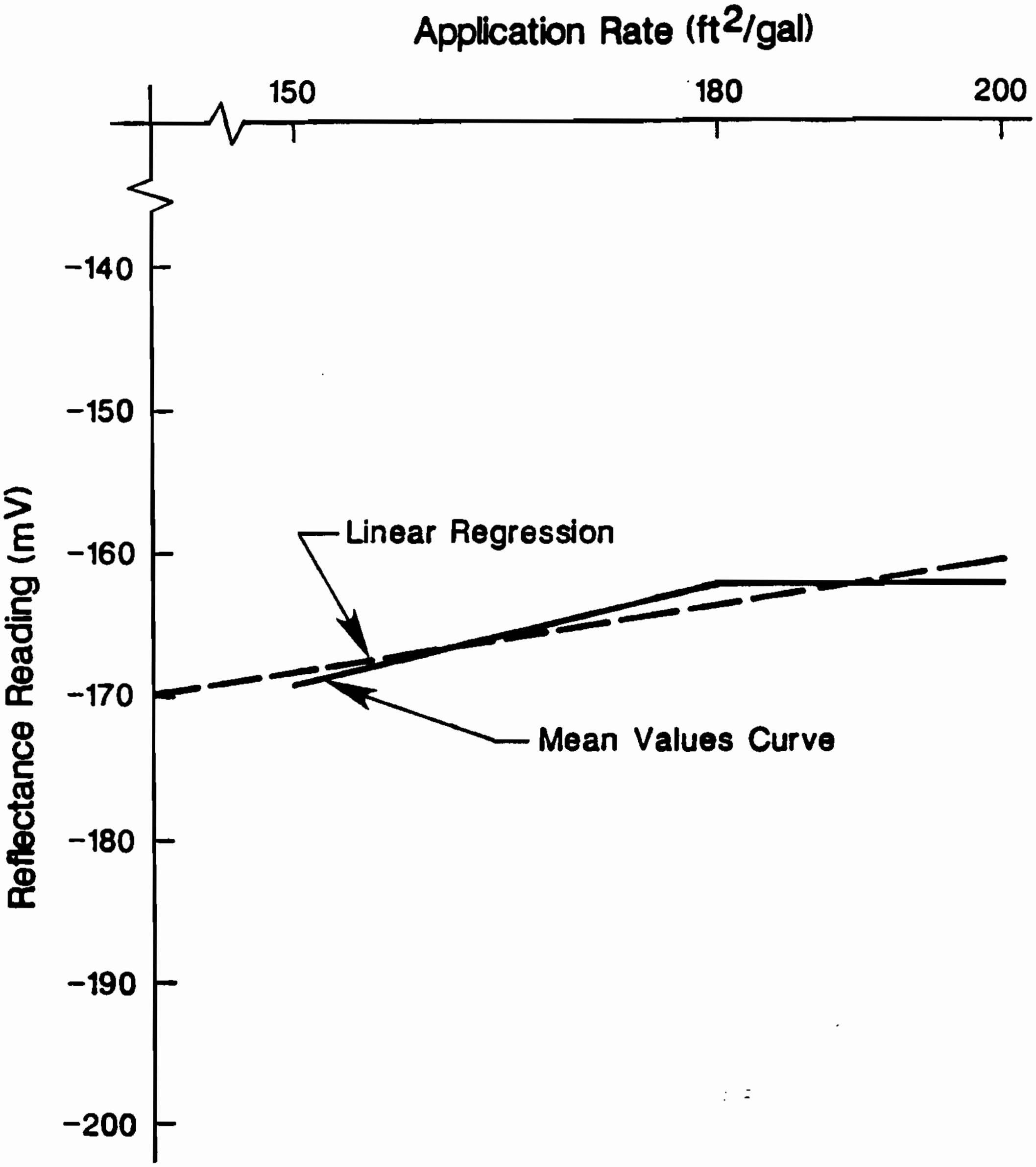


Fig 9.46. Correlation of reflectance readings and P-2 compound application rates with meter standardized to 18 percent reflectance.

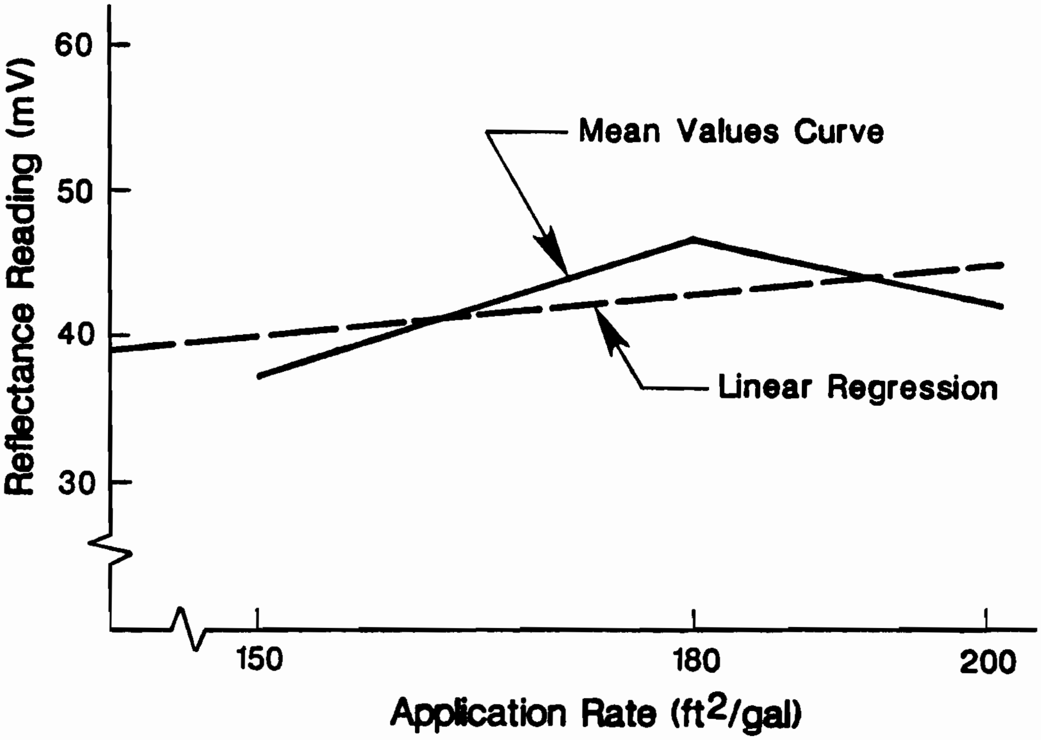


Fig 9.47. Correlation of reflectance readings and P-2 compound application rates with meter standardized to 90 percent reflectance.

The results from the 18 percent standardized data led to several observations:

(1) For any application rate, the typical spread of readings for a single surface grossly exceeded even the largest spread between mean readings for different application rates,

(2) Slope of the linear regression was only 0.15 $mV/ft^2/gal$,

(3) Linear regression correlation coefficient was only +0.199, indicating very low correlation at best.

These observations led to rejection of the use of reflectance to measure application rate.

This decision to reject the method was confirmed by the data produced by the reflectance study of P-2 compound with the reflectance meter standardized to a 90 percent reflectance reflectant surface. Examining Table 9.51, Fig 9.45 and Fig 9.47, four major observations are made:

(1) As with the 18 percent standardization tests, the range of readings for any one surface far exceeded even the largest difference between mean readings for different application rates,

(2) Mean values did not consistently increase or decrease,

(3) The slope of the linear regression curve was only 0.09 $mV/ft^2/gal$,

(4) The regression correlation coefficient was only +0.287, indicating, again, low correlation between reflectance and application rate.

With regard to an explanation of this lack of correlation, several possibilities were considered, and all may have contributed. First, the P-2 compound tested, as well as four of the five other compounds involved in this study were opaque. As a result, once the concrete was covered by the curing compound, any additional compound merely covered other compound, not altering the reflective nature of the surface.

Second, minor variations in light affected readings fairly significantly. For example, though a shadow was never cast over an area being tested, minor arm motions by the experimenter while recording data or

TABLE 9.51. STANDARDIZED REFLECTANCE READINGS (mV) FROM CONCRETE TREATED WITH P-2 CURING COMPOUND

	Standardized to 18 Percent Reflectance		
Reading No.	1 gal/150 ft^2	1 gal/180 ft^2	1 gal/200 ft^2
1	-164	-158	-171
2	-172	-155	-199
3	-169	-162	-151
4	-191	-152	-149
5	-160	-193	-143
6	-154	-155	-153

	Standardized to 90 Percent Reflectance		
Reading No.	1 gal/150 ft^2	1 gal/180 ft^2	1 gal/200 ft^2
1	+39	+55	+40
2	+43	+45	+51
3	+33	+44	+50
4	+39	+47	+29
5	+41	+38	+45

adjusting the reflectance meter could typically alter neighborhood reflection patterns enough to raise or lower reflectance readings by as much as 15 mV. Obviously, this would skew results to the point of complete unreliability on a busy jobsite.

Third, although all outdoor data employed here were taken on cloudless days, other investigation showed that variable cloud density also caused significant variations in readings from the same surface.

Finally, the specific photocells used had a specific spectral response curve (see Fig 9.48). It is possible that differential atmospheric wavelength absorption, depending on weather conditions, could have reduced photocell sensitivity, causing inconsistent reflectance readings.

Moisture Loss Study

Table 9.52 is a presentation of the data obtained during the moisture loss comparison described in Chapter 7. It should be noted that one of the three specimens treated at each rate was insufficiently treated due to a mechanical problem with the spray bar. Despite this, several interesting observations can be made about these data.

(1) The higher application rate actually showed a slightly higher average moisture loss. This observation is valid whether one includes the errant values or not.

(2) Both "F" and "t" tests confirm that the difference in means (excluding the errant values) is statistically significant. However, the validity of these tests themselves is highly questionable, given an effective sample size of only two.

(3) Reliance on the statistical testing is also questionable from an intuitive point of view. The exhibited trend in moisture retention does not conform logically to the expectation that a higher application rate will produce a greater membrane thickness, leading to greater moisture retention.

(4) It was also noted that, at the application rate of 1 gallon per 150 sq ft, curing compound began to pool in surface indentations. This

TABLE 9.52. MOISTURE LOSS RATES (KG/M^2) USING APPLICATION RATES OF 1 GALLON PER 180 FT^2 AND 1 GALLON PER 150 FT^2

Specimen No.	Application Rate 1 gal/180 ft^2	 1 gal/150 ft ^{2}
1*	1.6136	1.719
2	0.904	1.171
3	1.033	1.139
Average	1.183	1.343
C.V. (Percent)	31.95	24.27
Average neglecting Specimen No. 1	0.969	1.155
C. V. neglecting Specimen No. 1	9.41	1.96

* (Nozzle on spray bar passing over these specimens was operating improperly.)

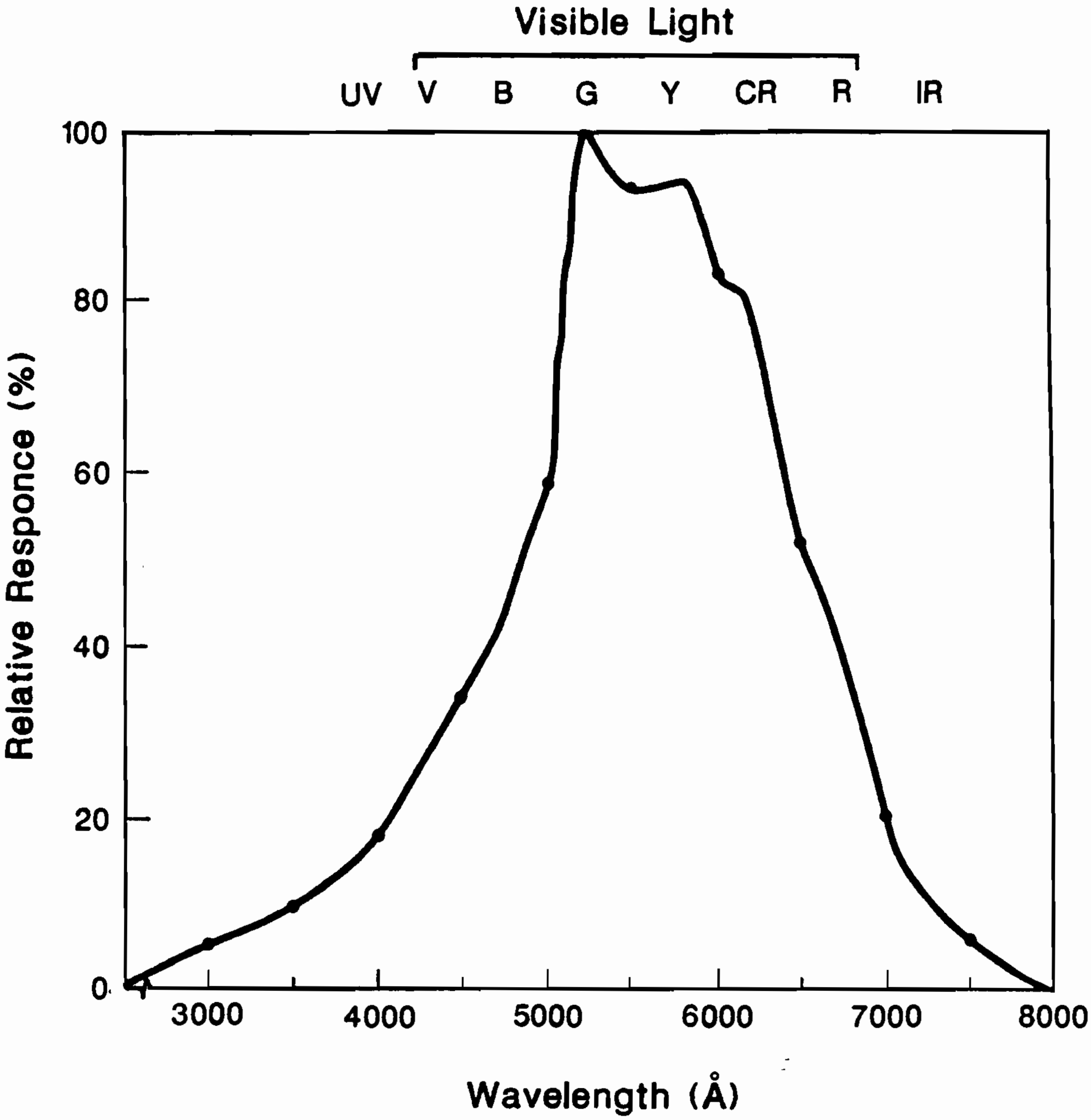


Fig 9.48. Spectral resonse pattern of Radio Shack #276-116A cadmium sulfide photocells used in prototype reflectance meter.

inspection also led to the observation that the groove surfaces seemed very sparsely covered at either application rate.

Based on these observations, it appears logical to conclude that application rates greater than 1 gallon per 180 sq ft do not seem to be helpful in reducing moisture loss. Further, it appears that the high moisture loss may be the result of inadequate membrane development within the surface grooves. Yet this problem is not solved by varying the pattern of application, as indicated earlier in this chapter.

IN-LINE VERSUS DRUM SAMPLE COMPARISON

S-1D Curing Compound

Table 9.53 presents the in-line versus drum sample test battery results. Results of vertical spray and drying time tests were satisfactory in all cases. In batch one, the drum sample showed a significantly higher flash temperature than the in-line sample. This difference was minimal in the other two batches, however, and the direction of the difference was not consistent in indicating a higher drum value than in-line value.

In all three batches, the in-line sample produced a sample with approximately 50 percent higher solids content value than the corresponding drum sample. This finding was virtually identical in all three batches and very probably indicates a meaningful difference between the two sample types.

With regard to the specific gravity tests, the drum sample showed a higher value than the in-line sample on a consistent basis, but this difference was only between 0.004 and 0.010, constituting only a 1 percent variability even in the worse case.

In the moisture retention tests, the trend of the data affirmed the findings of the solids content tests. The drum samples, having the lower solids content values, displayed much higher 24-hour moisture losses. These losses were between 3.9 and 5.0 times the corresponding in-line sample. Even the drum sample values were within the 2 percent acceptability criterion, but reliance on in-line sample data on a less retentive compound exhibiting a

TABLE 9.53. IN-LINE SAMPLE VERSUS DRUM SAMPLE SDHPT TEST RESULTS FOR TYPE S-1D CURING COMPOUND.

Batch Number	Sample*	Vertical Spray**	Drying Time**	Flash Temperature (Degrees F)	Solids Content (Percent)	Specific Gravity	24-Hour Moisture Loss (Percent)
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

* "I" implies in-line sample, "D" implies drum sample.

** Reported as "S" (satisfactory), or "U" (unsatisfactory).

similar trend in moisture retention values could lead to use of sub-standard materials in the field.

S-2 Curing Compound

As in the case of the S-1D compound, all vertical spray and drying time tests on all samples of S-2 compound proved satisfactory. As can be seen in Table 9.54, all three batches of this compound showed higher reflectivity values in drum samples than in corresponding in-line samples. These differences averaged only about 6 percent from the in-line value, however, and so no reliable conclusion can be made based on the data at hand.

In the flash temperature, solids content and specific gravity tests, there was no general trend of findings. In all three batches, the differences between in-line and corresponding drum sample test results were consistently less than 10 percent of the in-line value, and typically less than 5 percent of that value. In addition, the direction of the differences switched between favoring in-line data to favoring drum sample data. This was true of all three batches tested.

With regard to the moisture retention test data, the drum sample showed higher moisture loss in two of the three batches tested. In batch two, the drum sample lost over twice as much moisture as the corresponding in-line sample. In the first batch, however, the drum sample actually supplied superior moisture retention properties as compared to the corresponding in-line value. This, combined with the high degree of variability associated with the moisture retention test, suggests that no reliable preference for drum or in-line samples can be developed from this data.

P-1D Curing Compound

Only two batches of type P-1D curing compound were subjected to in-line and drum sample acceptability test result comparison. All samples from both batches passed both the vertical spray and drying time tests. In addition, the total range of specific gravity values was 0.005 out of an average value of 0.8995. As can be seen in Table 9.55, there was somewhat greater

TABLE 9.54. IN-LINE SAMPLE VERSUS DRUM SAMPLE SDHPT TEST RESULTS FOR TYPE S-2 CURING COMPOUND.

Batch Number	Sample*	Vertical Spray**	Drying Time**	Reflectivity (Percent)	Flash Temperature (Degrees F)	Solids Content (Percent)	Specific Gravity	24-Hour Moisture Loss (Percent)
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

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* "I" implies in-line sample, "D" implies drum sample.

** Reported as "S" (satisfactory), or "U" (unsatisfactory).

TABLE 9.55. IN-LINE SAMPLE VERSUS DRUM SAMPLE SDHPT TEST RESULTS FOR TYPE P-10 CURING COMPOUND.

Batch Number	Sample*	Vertical Spray**	Drying Time**	Flash Temperature (Degrees F)	Solids Content (Percent)	Specific Gravity	24-Hour Moisture Loss (Percent)
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

* "I" implies in-line sample, "D" implies drum sample.

** Reported as "S" (satisfactory), or "U" (unsatisfactory).

variability in the other test results. In both batches, the drum sample flash temperature was significantly below the corresponding in-line value. In the case of the second batch, this difference was a full 16° F. The solids content values of the drum samples were both somewhat above those of the corresponding in-line samples. These differences averaged only about 3 percent, however, and could not be used to justify a definite conclusion concerning relative sample validity.

Finally, with regard to moisture retention test results, the second batch results from in-line and drum samples were virtually identical. In the case of batch number one, the drum sample gave a superior performance to that of the in-line sample. This difference was not great enough to be conclusive, however, given the high degree of variability of this test.

P-2 Curing Compound

Four different batches of type P-2 curing compound were tested for in-line versus drum sample discrepancies. The results of these tests are presented in Table 9.56. Vertical spray and drying time tests proved satisfactory for all samples tested. In addition, reflectivity and specific gravity tests on in-line and drum samples were essentially the same for any one of the four batches tested.

For any given batch, there were reasonably large differences between in-line and drum sample solids content and 24 hour moisture retention. These differences, however, were not consistent in their direction among the four batches tested. In three of the four batches, the in-line moisture loss was greater than the drum sample loss. Also, in three of the four cases, drum sample solids content was higher than that of the corresponding in-line sample. These solids content differences were relatively minor, however, and did not correspond to the cases of higher in-line sample moisture loss.

M-1D Curing Compound

Table 9.57 constitutes the test data for in-line and drum sample SDHPT quality control tests performed on three batches of type M-1D curing compound. In all three batches, all in-line and drum samples were found to

TABLE 9.56. IN-LINE SAMPLE VERSUS DRUM SAMPLE SDHPT TEST RESULTS FOR TYPE P-2 CURING COMPOUND.

Batch Number	Sample*	Vertical Spray**	Drying Time**	Reflectivity (Percent)	Flash Temperature (Degrees F)	Solids Content (Percent)	Specific Gravity	24-Hour Moisture Loss (Percent)
1	I	S	S	61.4	100	47.61	1.015	1.71
1	D	S	S	61.7	113	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	0.998	1.54

* "I" implies in-line sample, "D" implies drum sample.

** Reported as "S" (satisfactory), or "U" (unsatisfactory).

TABLE 9.57. IN-LINE SAMPLE VERSUS DRUM SAMPLE SDHPT TEST RESULTS FOR TYPE M-1D CURING COMPOUND.

Batch Number	Sample*	Vertical Spray**	Drying Time**	Flash Temperature (Degrees F)	Solids Content (Percent)	Specific Gravity	24-Hour Moisture Loss (Percent)
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

* "I" implies in-line sample, "D" implies drum sample.

** Reported as "S" (satisfactory), or "U" (unsatisfactory).

be satisfactory with regard to both drying time and the requirements of the vertical spray test.

In all three batches, the flash temperature was found to be higher in the in-line sample than in the drum sample. This difference varied from 2°F to 12°F, averaging 6.67°F. The solids content tests revealed no general trend. In the case of two batches, the in-line solids content value was above the drum sample, but the differences were ooth much less than one-half of one percent. In batch two, the difference was almost four full percentage points. The direction of this difference, however, was opposite that found in the other two batches.

In two of the three batches tested, differences in drum and in-line specific gravity values were on the order of only three or four thousandths of one percent (out of values near 0.93 percent). The third batch tested displayed a more significant difference in specific gravity values. This difference was still only about 0.075 percent, out of values around 0.9 percent. Because of this relatively small difference, and the fact that there as no general trend toward such a difference in all three batches, there was no reason to suspect a true difference in the specific gravities of in-line and storage drum samples.

Finally, with regard to the moisture retention tests on in-line versus drum samples; only negligible differences were found in the samples from batches one and three. Batch two, however, displayed a drum sample moisture loss value more than double the in-line sample value. The true significance of this finding, however, is questionable due to two factors: first, the phenomenon was not observed in the other two batches of this material and, second, the variation, though larger, was not unusual given the high variability associated with the test (as discussed earlier in this report).

M-2 Curing Compound

As can be seen in Table 9.58, only two batches of type M-2 compound were tested for drum versus in-line sample differences. Both samples from each batch were found satisfactory with regard to the vertical spray test and the drying time test. In addition, both batches showed virtually identical in-

TABLE 9.58. IN-LINE SAMPLE VERSUS DRUM SAMPLE SDHPT TEST RESULTS FOR TYPE M-2 CURING COMPOUND.

Batch Number	Sample*	Vertical Spray**	Drying Time**	Reflectivity (Percent)	Flash Temperature (Degrees F)	Solids Content (Percent)	Specific Gravity	24-Hour Moisture Loss (Percent)
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

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* "I" implies in-line sample, "D" implies drum sample.

** Reported as "S" (satisfactory), or "U" (unsatisfactory).

line and drum sample results of the flash temperature, solids content, and specific gravity tests. The reflectivity and moisture retention tests, however, were quite a different story. Both batches displayed severely inferior drum sample reflectivity and moisture retention test results.

With respect to the reflectivity test, both batches were found to display at 10 percent lower reflectivity when drum samples were tested than when in-line samples were checked. Although the lower value was still acceptable, a similar trend in a batch that tested borderline in-line samples, could lead to use of substandard materials.

The same can be said of the moisture retention test results. In this case, however, even the drum sample losses were drastically below the allowable 24-hour loss limit of 2 percent. The implication is that a borderline material could be significantly different from the two batches tested,and so there is no reason to expect the same trends in the test results.

CHAPTER 10. CONCLUSIONS AND RECOMMENDATIONS

SUMMARY

This study presents the results of an investigation performed in order to determine the adequacy of the current Texas State Department of Highways and Public Transportation (SDHPT) specifications related to liquid membrane-forming curing compounds for portland cement concrete with regard to moisture retention, settlement rate of the solids, and quality of agitation. In addition, the study reports the results from the development of a new test for moisture retention test for curing compounds.

Manufacturers in recent years have questioned the adequacy of Test Method Tex-219-F, "Testing of Curing Materials," a procedure for evaluating the moisture retention of curing compounds, stating that it is not consistent and not as reliable as the ASTM method. To investigate this, a series of multiple moisture retention tests on six different curing compounds was performed by the Center for Transportation Research using Test Method ASTM C 156-80. A similar set of tests on the same compounds was performed by the Bituminous Section of D-9 of the SDHPT using Test Method Tex-219-F. Results from the two test procedures were compared and analyzed for statistical characteristics including variability and confidence.

Current specifications require curing compounds be storable for at least six months without deterioration. Fugitive dye or water in Type 1-D and white pigment in Type 2 compounds are prone to separation during storage. This could be a serious problem because it reduces the moisture retention capability of the compound and limits the ability to detect nonuniform application. In addition, if excessive cake or crust is developed, the material becomes unusable because spraying is impossible. CTR performed agitation tests on the same six curing compounds that were used for the moisture retention tests. Testing included 3- and 6-month mechanical agitation of compounds contained in fifty-five gallon drums, using four different types of mixing blades applicable through a 2-inch diameter bunghole. Quality of mixing of each agitator was determined by solids content and visual inspection tests of compound samples obtained before and after mixing.

The study included a search for experimental verification of the currently accepted six month recommended shelf life of liquid membrane forming curing compound. To this end, samples of all six curing compounds were drawn before and after agitation at ages of three, six, nine, and twelve months. These samples were tested by CTR for solids content and visual separation pattern. Similar samples were also sent to D-9 and subjected to the normal full battery of curing compounds tests.

Curing compound application patterns and rates were also examined. Seven different application patterns were tested by comparing the moisture lost from standard ASTM C-156 test specimens textures with the SDHPT standard pavement surface texture, and treated in the specific pattern being examined.

Application rate and uniformity were examined by direct measurement of moisture loss of ASTM C-156 specimens treated at application rates of 1 gallon per 180 sq ft and 1 gallon per 150 sq ft. Attempts were also made to develop an optical reflectance test of application rate and uniformity.

For the development of a new moisture retention test a simple apparatus was manufactured and several membranes were cured with compound in order to determine one that had similar permeability to the evaporation of cured concrete as determined from multiple moisture retention tests.

CONCLUSIONS

After reviewing the experimental results, the following conclusions can be made.

Moisture Retention Tests

(1) The ASTM C 156-80 and Tex-219-F moisture retention tests have slightly different procedures: (a) the Texas method expresses moisture loss in terms of water in the mortar at the time of

application, while ASTM expresses loss based on a constant surface area, and (b) the Texas method applies a faster and simpler test for the determination of loss in volatile matter from compounds during curing.

(2) These differences in procedure have been shown to cause no difference in the results from the two methods. Texas results and Texas values converted into ASTM units had the same coefficient of variation.

(3) The use by Texas of the water present at the time of application for expressing moisture loss does not significantly improve the results and does require recording of additional weights which adds to the complexity of the test.

(4) The use by Texas of aluminum pans in which compound is poured and not sprayed for the determination of loss in volatile matter is an accurate substitution of the ASTM procedure and made the test less complicated and easier to perform.

(5) The variability of the ten specimens tested for each compound was high; the average C. V. of the five compounds was 33 percent and 21 percent for the ASTM and Texas methods, respectively. Considering the tests performed for each compound (each ASTM test consisted of 3 specimens and Texas of 2), the average C.V. changes to 20 percent and 15 percent, respectively. Thus, on an average basis, ASTM exhibited a relatively higher variability. In addition, ASTM test had higher C. V. in four our of the five compounds tested.

(6) From the statistical "F" test for variances using specimen values, it was determined that one ASTM test exhibited higher variability than the Texas test at a significance level of 5 percent. The variability of ASTM and Texas in the other four tests was the same.

(7) Statistical tests for moisture loss differences of mean ASTM and Texas values converted into ASTM units (considering all specimens) determined that results did not follow a definite trend and showed that each test is equally likely to produce higher losses than the other. Tests on ASTM and Texas average test values indicated that

both methods gave equal values in three compounds, while Texas gave higher values in two compounds.

(8) Statistical tests for differences of mean ASTM and Texas moisture loss values expressed as a percentage of the maximum loss allowed by the two tests (considering all specimens and average test values) indicated that each method is equally likely to produce higher (stricter) values than the other.

(9) The variability of the moisture retention tests was reduced when moisture loss of cured specimens was expressed as a function of the moisture loss of a specimen without curing compound cast at the same time. Based on these results it appears that it would be beneficial to express the Texas test moisture loss in the same manner.

(10) One of the contributors to the variability of the ASTM moisture retention test is the variable moisture loss potential of mortar specimens prepared with different brands of portland cement.

(11) Routine moisture loss tests on nine compounds (blind samples) showed that the Texas procedure produced higher results in all the compounds despite the fact that Texas losses were reported in 24 hours and ASTM in 72 hours. Of the nine compounds tested, five failed the Texas test, but none of them failed the ASTM test. These results contradict the conclusions derived from the five multiple moisture retention tests and no viable explanation can be offered.

Agitation Tests

(1) The solids test was found to be an accurate method of determining the extent of mixing of curing compounds, even though no relationship was found between the moisture retention ability of curing compounds and their solids content. Results of solids content tests were misleading only when separated compound portions had the same level of solids contents. This was the case in M-2 curing compound.

(2) The proposed visual inspection test was also found to be an accurate method of determining mixing quality of curing compounds. Both solids content and visual tests gave the same results concerning the mixing quality, except in M-2, where only the visual test gave a correct indication of the quality of mixing.

(3) Separated compound portions at the top and bottom positions of the drums were the most difficult layers to be reincorporated into the mix in both types of curing compounds. Therefore, samples taken only from the middle of the drums gave false results concerning the quality of mixing.

(4) Samples should be obtained from top, middle, and bottom drum levels in both Type 1-D and Type 2 curing compounds, in order to examine quality of mixing. For perfect mixing the solids content or separated portions in the three levels should be the same. In order to check if the compound lost any volatiles, the three samples should be compared to the base line values.

(5) Agitator type D produced a "best" mixing in all Type 1-D curing compounds, except in P-1D which developed a crust after 3 months of storage. Mixing times were 30 minutes for S-1D, and 10 minutes for P-1D and M-1D. This agitator had 2-inch diameter non-folding blades which run at a very high speed (1400 rpm). The high speed seemed to be the most important factor in Type 1-D curing compounds.

(6) Agitators A, B, and C performed "best" mixing only in compounds P-1D and M-1D. Mixing times were the same as with agitator D, except in M-1D where agitator A mixed the compound in 20 minutes. In the case of compound S-1D, agitator A did not cause any redispersion of separated ingredients, while agitators B and C failed to redisperse the top layer into the mix.

(7) Type 1-D curing compounds stored up to six months can be easily remixed to a homogeneous consistency using the Type D agitator.

(8) In Type 2 curing compounds 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, either after 3 or 6 months of storage. In general, agitator B produced the best quality of mixing. This agitator had folding blades that opened to 4 3/4 inches in diameter and ran at a higher speed than C and lower than D. The high shearing action of this agitator was apparently the key factor of good mixing. Mixing times of all the agitators were 20 minutes for S-2 and M-2 and 30 minute for P-2 curing compounds.

(9) Agitators C and D had about the same performance in Type 2 curing compounds. Agitator C produced somewhat better mixing in M-2 curing compound, while agitator D performed better in S-2 and P-2 compounds. Agitator A did not produce any mixing in Type 2 curing compounds.

(10) Three-month mixing of Type 2 curing compounds did not prevent any further separation of compound ingredients. Quality of mixing at six months was lower than the quality at three months as evidence by both solids and visual inspection tests.

Shelf Life Tests

(1) CTR tests of visual separation patterns and solids content suggested that type S-1D compound could be used after one year of storage. SDHPT curing compound tests did suggest some alteration of compound properties. The compound consistently failed vertical spray and drying time tests at nine months, but not at twelve months (though two samples at twelve months did fail these tests). Because some samples passed all tests at twelve months, and because of the possible deleterious effects of the delay between sampling and testing, these results do not constitute sufficient cause to set S-1D shelf life at less than one year.

(2) CTR tests again indicated no unusual visual separation pattern on solids content changes in type S-2 compound after one year of storage. SDHPT tests, however, showed a severe thickening of the compound after nine months of storage. This was evident, to a more

severe degree, at twelve months. This problem was not believed to have been caused entirely be delay between sampling and testing. Thus, a shelf life of more than six months could not be recommended for type S-2 curing compound.

(3) CTR tests on type P-1D curing compound indicated no material degradation after one year of storage provided the material was not agitated until just before use. If the material is agitated, it should be entirely used at that time, and not allowed to sit and then be re-agitated at a later date. SDHPT tests confirmed these conclusions. If anything, these tests suggested an improved moisture capability in this material after one year of storage.

Recommended shelf life for type P-1D compound was thus determined to be not less than one year.

(4) Throughout the CTR tests on P-2 compound, both solids content and visual separation patterns were quite similar to those of base line samples even after one year of storage. The only exception to this was low solids content in one of the twelve month samples. SDHPT test confirmed both this general behavior and the specific anomaly. However, these tests showed the P-2 material to be completely satisfactory under all test criteria even after one year of storage. Thus, extension of allowable shelf life to twelve months did seem justified for the P-2 curing compound.

(5) All CTR tests on type M-1D curing compound showed that this material could be agitated to uniformity with relative ease after up to one year. There was some evidence that intermittent agitation during storage facilitated agitation just prior to use. However, this benefit would not be sufficient to justify the effort expended on that intermittent agitation.

(6) Type M-2 compound was the least satisfactory with regard to the findings of CTR tests. At both nine and twelve months, agitation did not regenerate separation patterns similar to the base line sample. More importantly, large, hard particles were discovered in the compound when it was first tested at three months of age.

These particles were of sufficient size to cause clogging of typically used field application equipment.

SDHPT was able to conduct its test despite these particles, and the material did pass all tests. This material, like the M-1D compound, showed some tendency to improve its moisture retention capacity with age. This does not eliminate the other problems cause by the suspended particles, however, and so the evidence supporting extension of shelf life to a period of more than six months does not seem adequate.

(7) Based on these observations, no shelf life extension is recommended for compounds S-2 and M-2. However, the shelf life of P-2 and all Type 1-D compounds was shown to be at least one year.

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) "t" tests suggested significant differences in results 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 7 (one pass parallel to grooves and one pass perpendicular to grooves). These differences were detrimental with regard to 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 1 gallon per 150 square feet to 1 gallon per 200 square feet varied by significantly less than the typical same-surface variation noted in (1) above.

(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 1 gallon per 180 square feet and 1 gallon per 150 square feet 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 (4) above, suggested that compound pooling in surface depressions had begun with the use of the 1 gallon per 150 square feet application rate.

New Moisture Retention Test

Celanese microporous film K-442 was found to exhibit potential in the development of a new moisture retention test for membrane-forming curing compounds. Type 2 curing compounds are likely to give better results than Type 1-D when tested with the new test.

Drum Versus In-Line Sample Comparison

(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 as compared to in-line samples. This suggests that drum samples are preferable.

(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 preferability of the use of drum samples.

(3) Flash temperature data from type M-1D compound tests also suggested lower drum sample values. This reaffirmed conclusion (2) above.

(4) Drum samples of type M-2 curing compound were found to be inferior to corresponding in-line samples with respect to reflectivity and moisture retention capability, again reaffirming (2) above.

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

RECOMMENDATIONS

Based on the results and conclusions 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 percent moisture retained should be equal to or greater than 80 percent.

(2) Recommended speed and blade diameter of agitators used for Type 1-D curing compounds are 1400 rpm and 2 inches, respectively. Mixing should be continued until a homogeneous consistency at the top, middle, and bottom of the drum is accomplished, as determined by a solids content or visual inspection test. For the curing compounds tested, the mixing range was 10 to 30 minutes.

(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 and the use of Celanese membrane K-442, a small testing program is required for each curing compound to determine the suitability of the new test, the applicability of the membrane, and the appropriate run time.

(4) Since 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 six months for Type 2 compounds, but extended to twelve months for Type 1-D compounds.

(5) The data collected during this study do not support specification of a standard application pattern, since no significant differences were noted among the various patterns tested.

(6) The human eye appeared to be more sensitive and able to survey larger areas than the reflectance meter. Therefore, it is recommended that visual inspection be continued as the standard procedure.

(7) No significant increase in moisture retention was noted with the increase in application rate to 150 sq ft per gallon. Therefore, it is recommended that the current standard rate of 180 sq ft per gallon 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 more similar history to field material than does an in-line sample suggested recommendation of drum samples for use in SDHPT curing compound acceptability tests.

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