EVALUATION OF SEALERS FOR CONCRETE BRIDGES

DHT-21

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16. Abstract

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The following classes of sealers were evaluated: silanes and siloxanes, water-based epoxy, polyester, aluminum stearate, silicate, and linseed oil. The silane and siloxane group performed best in all phases of testing. Linseed oil performed nearly as well; however, some questions were raised concerning its long term durability due to its limited depth of penetration and to the reactivity of the oil in the alkaline environment of fresh concrete. Based on these results, it is recommended that *TEXAS STANDARD SPECIFICATION* Item 428 be supplemented to permit the use of silane and siloxane-based sealers on new construction.

The most effective screening procedure was shown to be a combination of accelerated weathering and indirect test methods including Weather-Ometer weathering coupled with 28 days of saline immersion, infrared spectroscopy, percent residue determination and depth of penetration testing. A detailed procedure is included in Appendix C.

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FOREWORD

The contents of this report reflect the views of the author who is responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views of policies of the State Department of Highways and Public Transportation. This report does not constitute a standard, specification, or regulation.

The identification of sealers by their trade or manufacturer's name will be supplied under separate cover to Transportation Authorities upon request. Producers participating in this evaluation will be given the identification of their material only.

The author wishes to express appreciation to Richard Hamilton for designing the batch mix used in preparing test specimens and for his assistance in testing the physical properties of concrete and to Ray B. Merrill for his assistance in the wet chemical procedures.

ABSTRACT

An evaluation of representatives of the most popular types of concrete bridge deck sealers was conducted using a two-pronged approach: performance testing via accelerated weathering coupled with water immersion, and indirect testing using instrumental and analytical techniques. The goals of the project were to assess the ability of the sealers to protect embedded reinforcing steel from corrosion and to develop an effective test procedure for screening commercial products for use by the Department on new construction.

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INTRODUCTION

Cracking and spalling of concrete caused by the expansion of corroding reinforcing steel are the most common causes of deterioration of bridge decks. The problems are most pronounced in regions where deicing salts are used and along coastlines where airborne chloride ions from saltwater contaminate the concrete. Migration of chlorides to the rebar can occur via two different mechanisms: by entering as a dissolved component of penetrating moisture, or by diffusing from the outside through stationary pore water. Once the chloride concentration at the depth of the rebar reaches a threshold level of approximately 1.5 pounds per cubic yard (1), corrosion of embedded steel is inevitable. The alkalinity of fresh concrete offers some protection from rusting; however, moisture in the capillary pores soon leaches free calcium hydroxide (laitance) to the surface and reduces the level of alkalinity. In a neutral pH environment, the chlorides can attack the steel causing it to corrode. The resulting expansion gives rise to localized tensile forces which in turn cause pop outs and spalling of concrete in that area. This it can be seen that for two reasons at least (i.e., transport of Cl and CaOH), the unabated permeation of moisture into concrete can have deleterious consequences.

Currently, *TEXAS STANDARD SPECIFICATION* Item 428.3 calls for the sealing of all concrete bridge decks with boiled linseed oil. Subsequent to the writing of this specification, alternative materials for sealing concrete have been developed which purport to be as effective as linseed oil treatment and should therefore be considered for use. There are hundreds of products representing some fifteen or so generic classes of penetrating sealers on the market for concrete (2). The goal of this project is to determine which of these provides equal or better protection of steel-reinforced concrete than linseed oil by evaluating representative brands of the most promising classes.

The quality of a concrete sealer is a measure of its ability to withstand the variety of persistent undermining forces encountered in the field. Among the most significant are the abrasive wearing of bridge decks by traffic, ultraviolet radiation and wet/dry and freeze/thaw cycling. The alkalinity of the concrete itself can cause saponification and deterioration of oil-based sealers (notably linseed oil). Clearly, the most reliable method to determine the effectiveness of a sealer would be to treat a bridge deck and then wait 10 years or so to check chloride profiles of cored samples and visually inspect the deck surface. The next best approach, the one used in this study, is to simulate the field environment under controlled conditions in a testing facility.

Part I of this project utilized accelerated weathering procedures to evaluate 15 candidate sealers. The procedure involved subjecting treated specimens to various forms of weathering and then evaluating the resultant durability of the sealer systems by immersing the specimens in saline solution for 28 days and measuring the weight gain. The following weathering programs were employed: WEATHER-OMETER exposure, freeze/thaw cycling, abrasion resistance testing and outdoor test rack weathering

Part II investigated "indirect" methods that could be used to supplement and/or improve on some of the performance-based procedures described in Part I. The most promising sealers from Part I were subjected to testing in Part II. Included in this program were the Rapid Chloride Permeability Test, depth of penetration testing and water vapor permeability testing. In addition, a procedure for the determination of weight percent of silicone residue in silane-based sealers was evaluated. Part II procedures were evaluated along with those used in Part I in order to come up with an effective combination of material and performance tests that can be used for routine product evaluations.

In summary, Part I of this project evaluates several of the most common generic classes of concrete sealers for use on bridge decks with an eye to supplementing the current *STANDARD SPECIFICATION* for linseed oil. Part II focuses on the test methods themselves. The object of Part II is to develop a concise, standardized procedure for the screening of new products from those generic classes demonstrating acceptable performances in Part I.

Specimen Casting & Preparation

Two types of specimens were used during the evaluation: 6" × 6" × 1" blocks made from a batch design similar to standard bridge deck concrete; and 6"× 3 × 3/8" mortar coupons cast from Type I cement and sand. Batch formulas can be found in Appendix A. All blocks and coupons were cast in forms, without the use of release agents. The bottom sides of the specimens were textured during casting by placing the forms on the bottom side of a section of indoor/outdoor carpet. The top surfaces were troweled smooth and textured in a similar manner by pressing a small section of damp carpet, grid side down, onto the top face of the specimens. The troweling procedure was standardized such that each specimen received the same number of passes with a magnesium trowel in an effort to standardize surface mortar content and hence surface permeabilities. All specimens were cured in a fog chamber for 24 hours, removed from molds, and then returned to the fog chamber for a minimum of 45 days.

Prior to the application of sealer, specimens were removed from the fog room, wire brushed and then rinsed to remove accumulated laitance and any loosely adhered particles that could subsequently rub off and affect weight measurements. The edges of the blocks and coupons were coated with a 100 percent solids epoxy to insure that moisture penetration could occur only through the sealed surfaces. The epoxy was allowed to cure for 24 hours before placing the specimens in a 140F oven for 48 hours and then storing at room temperature for 7 days. This preapplication step provided blocks containing approximately one percent free moisture and coupons with around 0.5 percent free moisture.

Sealer Application

With the exception of two late entries to the evaluation, all sealers were subjected in triplicate to each phase of testing. Sealers were applied to both sides of the specimens using a reciprocating spray machine to obtain exact wet film thicknesses thereby ensuring that redundant specimens were identical. Coverage rates were as prescribed by the producers. The spray device consisted of a conveyor which carried specimens under a fanned spray of sealer material at a fixed rate. Several passes were required to achieve the desired wet film thickness. When a range of rates were specified by the producer, the most conservative (i.e., thickest) coating was applied, except that materials from the same class of sealers and having the same solids content were applied at the same rate so that relevant comparison could be made. As an example: all of the 40 percent silanes were applied at 125 sq ft/gallon.

In order to evaluate the effect of reducing the application rate by 25 percent during selected phases of testing, the number of passes on the conveyor was commensurately reduced.

All sealed specimens were cured for 7 days at room temperature before being exposed to the various tests.

PROCEDURE

Part I

As previously indicated, the procedure for Part I involved treating concrete specimens with the various sealers and then subjecting them to a variety of simulated environmental conditions. Upon completion of each stress regimen, the blocks were weighed and then immersed in a 5 percent saline solution for 28 days to assess the sealers' ability to protect against the penetration of moisture.

Test Rack Aging

Three intervals of aging were observed: 0, 3, and 12 months. The unaged blocks were immersed in saline solution immediately upon completion of the 7-day sealer curing period. In addition, the sensitivity of sealers to variations in application rates was assessed by including specimens coated at 75 percent of the manufacturers' recommended coverage rate. The 3- and 12-month outdoor aging regimens involved mounting treated blocks on a test rack angled at 45 degrees with a south exposure. All blocks were flipped halfway through the aging process so that sealed surfaces received equal exposure to the environment. Upon completion of 3 and 12 months of aging, specimens were evaluated using the procedure described in the "Performance Testing" section.

WEATHER-OMETER Conditioning

Sealed mortar coupons were exposed to accelerated weathering in an Atlas XWR carbon-arc WEATHER-OMETER on a cycle of 102 minutes of sunshine followed by 18 minutes of rain and sunshine. Two durations of exposure were utilized: 500 hours and 1000 hours. The 1000-hour phase included the specimens coated at the 75 percent coverage rate as well as specimens coated at the manufacturer's recommended application rate. All coupons were rotated in their holders halfway through the conditioning so that each sealed surface received equal amounts of direct weathering. Upon completion of this phase of stressing, all specimens were performance tested via 28 days of saline immersion as described below.

Performance Testing

Upon completion of a given stress program, all coupons and blocks were stored at room temperature until their weights became constant before subjecting them to the 5 percent saline baths. In practice, constant weight implied that the weights of the blocks and mortar coupons changed less than 0.1 percent in a 24-hour period when stored in a climate-controlled room with relative humidity held at 55 ± 5 percent. An average of one week was required for specimens to equilibrate with the lab environment.

Saline solution was used in the immersion baths instead of pure water in an attempt to accentuate the weight gains of specimens. To increase saline uptake, the baths were fitted with heat lamps and timers so that the bulk temperature of the solution was cycled between 77 F (approximately room temperature) and 140 F every 24 hours. The baths were partitioned according to sealer type so that residue from a given group of specimens could not contaminate any of the other specimens.

After 28 days, the specimens were removed from the baths and placed in room temperature 5 percent saline solution for 4 hours to stabilize. Specimens were then lightly rinsed with tap water, towel dried to a saturated surface dry condition (3) and weighed to within ± 0.1 gram.

Freeze/Thaw Cycling

After obtaining weight gain data from 28 days of saline immersion, blocks from the 3-month aging regimen were exposed to freeze/thaw cycling in a Scientemp Environmental Chamber. The effect of 6 cycles/day for a total of 300 cycles were monitored by visual examination of the sealed surfaces.

Abrasion

The effect of aging and abrasion was tested using blocks that had been aged 3 months on the outdoor test rack. Each specimen was sandblasted using a blast cabinet which delivered 1200 grams of #2 sand at 45 psi to both front and back surfaces. The specimens were stored until their weights equilibrated with the lab environment and then were evaluated by immersion as described in the "Performance Evaluation" section above. The combined effects of abrasion and application rate variation were tested using a set of companion blocks coated at a rate of 25 percent less than the manufacturers/ recommended rate.

Part II

Rapid Chloride Permeation Testing

The Rapid Chloride Permeation Test was a modification of AASHTO Test Procedure T-277-83. The method involved impressing a voltage across a treated cylindrical specimen. The sealed face was ponded in 3 percent NaCl solution and the other, unsealed face was ponded in .3 N NaOH solution. The rate of diffusion of negatively charged chloride ions (equivalently the chloride-screening performance of the sealer) was tracked by the drop in resistance of the cylinder over a 6-hour period.

The test specimens were cast in a 2-inch-thick section of 4-inch-diameter P.V.C. pipe. One surface was trowelled and textured as described in the "Specimen Casting & Preparation" section above, the other surface was smooth cast against the bottom of the mold. No release agent was used. Cylinder curing and sealer application was accomplished in the same manner as previously described for the block specimens, including coating the sides with 100 percent solids epoxy.

Each sealed cylinder was desiccated to remove all air from the pore structures by placing it in a 1000 ml beaker and then applying vacuum at a pressure of 1 mm Hg for 3 hours. With the vacuum pump running, deaerated water was then introduced into the beaker via a separatory funnel attached to an orifice in the desiccator lid. After sufficient water was introduced to cover the top of the in other words, the indicated silanes and siloxanes all provided around 73 percent better water proofing than untreated concrete. Linseed oil and the silicates performed worst in this regimen.

3- And 12-Month Aging

Figures G2 and G3 show the performances of sealed blocks after 3 and 12 months of outdoor exposure respectively.

Discussion: The silane/siloxane group performed best after both 3 and 12 months of test rack aging. The exception to the rule, SM5, a 20 percent silane mixture, may have been too dilute to adequately seal the capillary pore system. No explanation is offered for the erratic trend of behavior of the water-based sealer, SW7.

The silicate group, represented by sealers S12 and S13, provided little or no protection beyond that of untreated concrete in this phase of testing.

The epoxy coating, E11, provided a consistent protection level from moisture penetration, absorbing aproximately 60 percent of that of untreated concrete.

The polyester sealer, P10, deteriorated over time such that at 12 months no protection beyond that of untreated concrete could be discerned.

The aluminum stearate sealer, A14, deteriorated over time.

The trend of linseed oil to perform better with time reflected the fact that linseed oil required ultraviolet light in order to cure. Evidently, the unaged specimens that were tested before being exposed to sunlight had not cured adequately. After 3 months on the outdoor test rack, the linseed oil specimens performed as well as the silanes and after 12 months, linseed oil surpassed the silane/siloxane group. The poor performance of linseed oil in the absence of ultraviolet light suggests that its use in parking structures and in certain light obstructing bridges may require special consideration.

An indicator of the reliability of the data generated in this section is provided by the moisture uptake of the control (unsealed) blocks in the unaged regimen. The average moisture uptake for five blocks was 2.34 percent with a standard deviation of only 0.08 percent. It can generally be assumed, then, that differences in performance between sealers on the order of a half of a percent or more (of absolute moisture uptake) can be attributed to properties of the sealers themselves rather than to experimental error.

Freeze/Thaw

After 300 cycles no evidence of delamination was observed in any of the specimens tested.

Discussion: The freeze/thaw test was included in this evaluation to address concerns that the silanes, in particular, tended to weaken the surface mortar of air-entrained concrete under the influence of freeze/thaw cycling (5). No differences were noted between the appearances of silane-treated specimens and any of the other specimens.

Abrasion Testing

Figure G4 shows the results of performance testing of blocks that had been test rack aged for 3 months and then sand blasted to simulate tire wear.

The epoxy semipenetrant, E11, showed little protection beyond that of the control group. The reduced effectiveness of this sealer due to abrasion of the treated surface reflects its negligible depth of penetration. The exposed concrete in the shape of the sand blast pattern evidently offered no resistance to moisture intrusion.

The same blast pattern shape could be seen in the polyester-coated specimens; like the epoxy sealer, and for the same reason, no protection was imparted.

The linseed oil provided a slightly different scenario. As will be demonstrated later, the oil penetrated to a moderate extent; however, the UV from the sunlight, a requirement for proper crosslinking, was available only to the surface. The top layer cured adequately, as demonstrated by the 3- and 12-month-aged blocks (Figures G2 & G3), but was easily removed by the abrasion, thereby exposing uncured linseed oil and, thus, a permeable surface. The abraded linseed oil blocks absorbed 75 percent as much moisture as the untreated blocks. Fortunately, abrasive wear of bridge decks

UNAGED BLOCKS

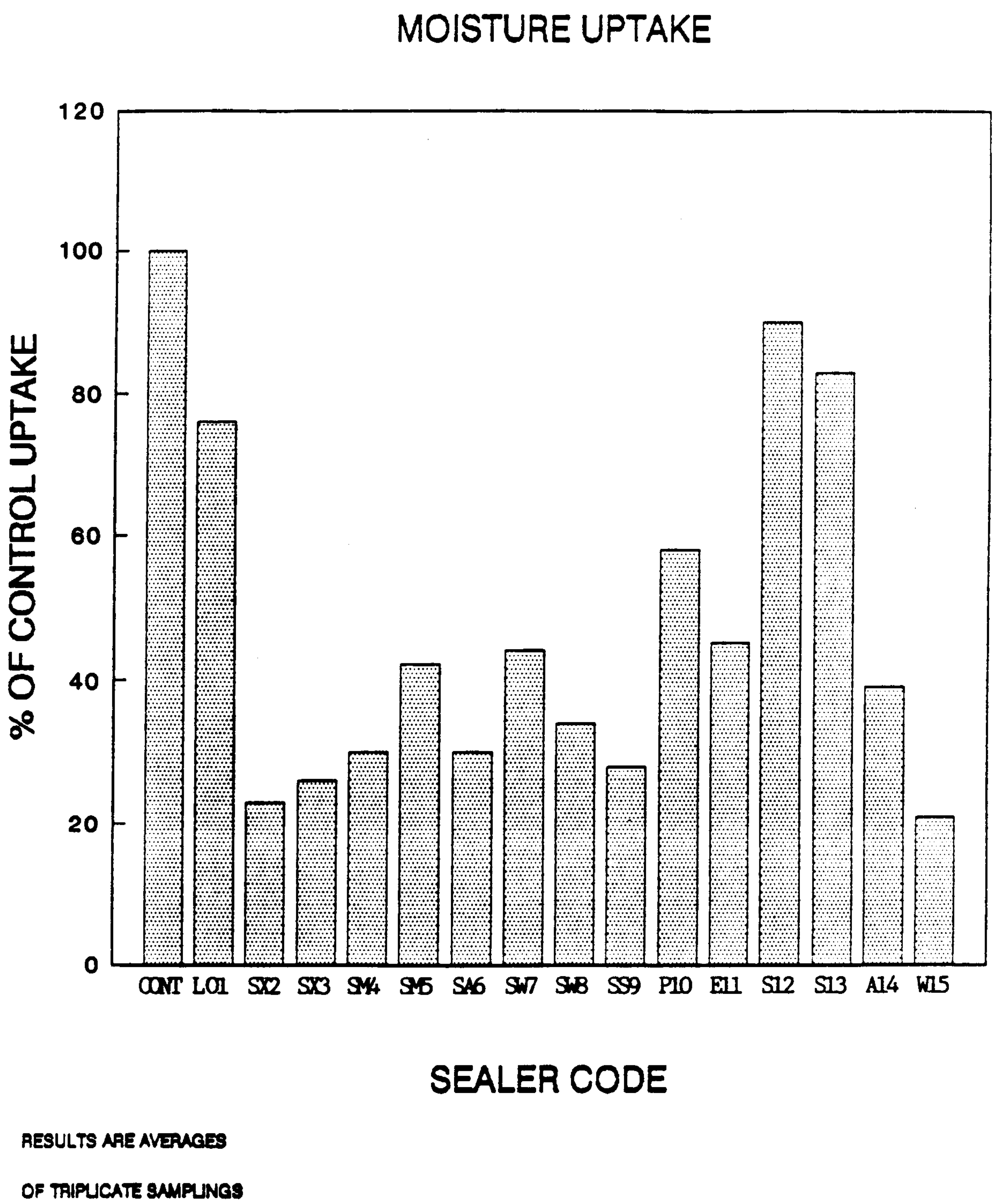
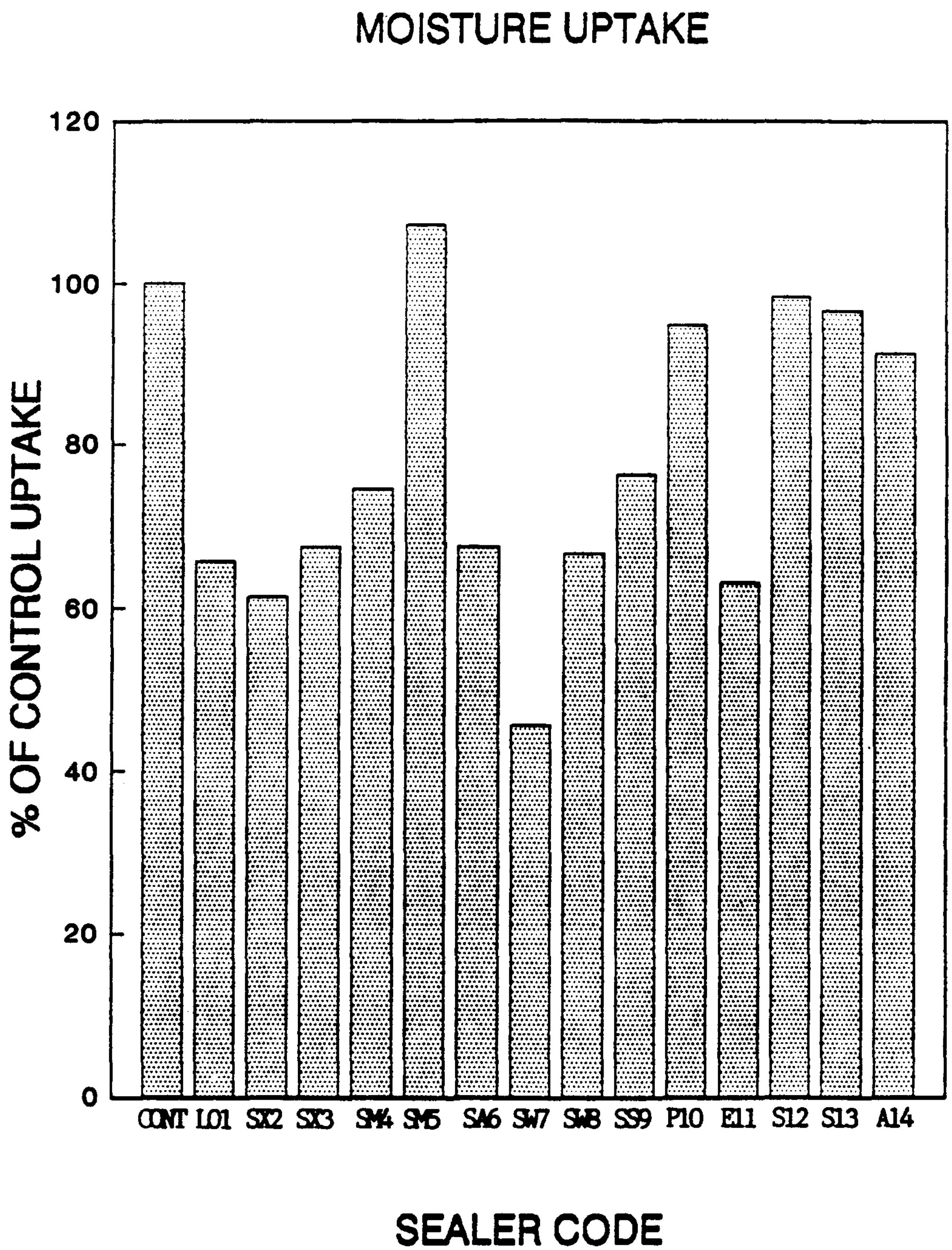


FIGURE G1

3 MONTH AGED BLOCKS

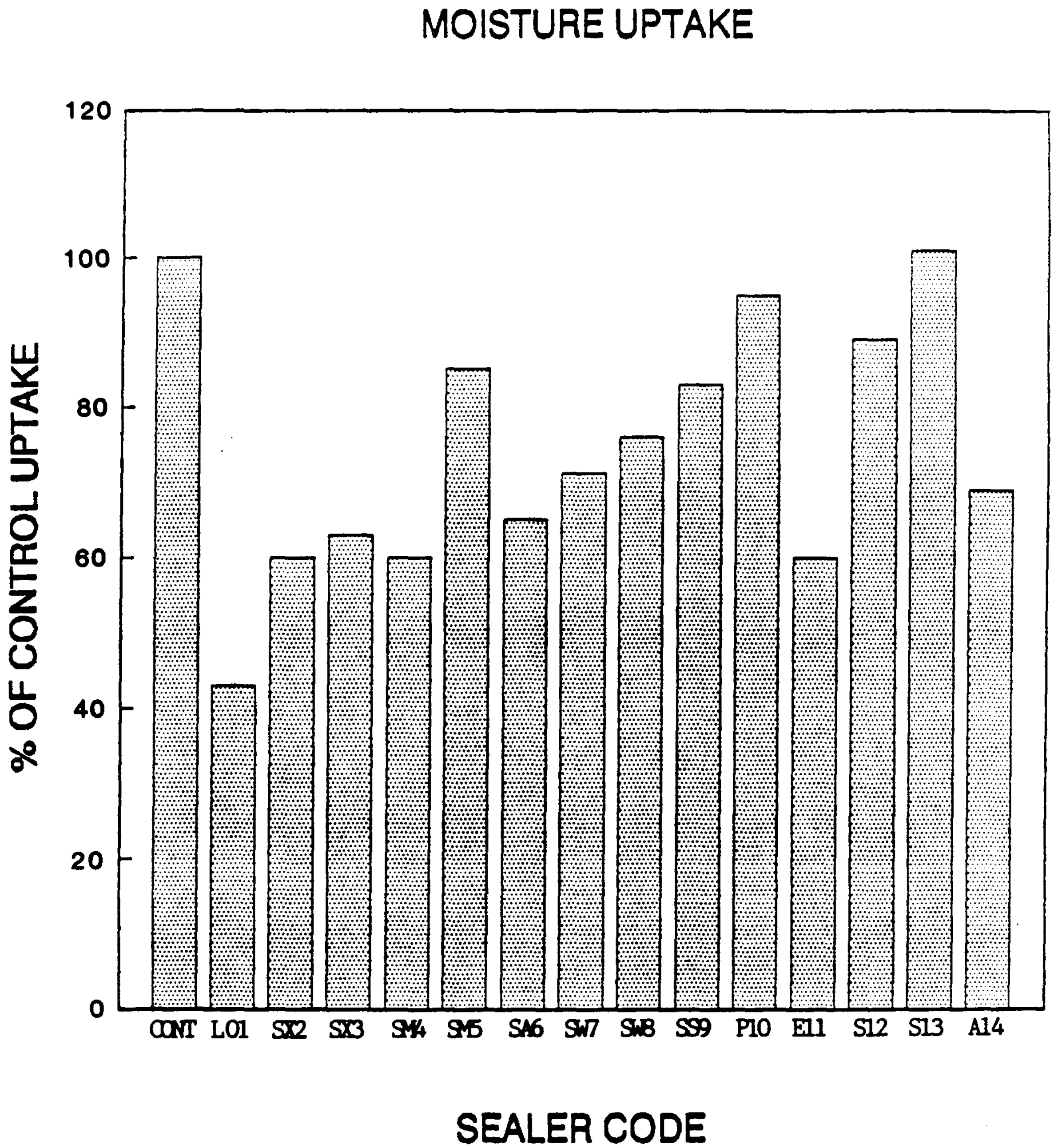


RESULTS ARE AVERAGES

OF TRIPLICATE SAMPLINGS

FIGURE G2

12 MONTH AGED BLOCKS



RESULTS ARE AVERAGES

OF DUPLICATE SAMPLINGS

FIGURE G3

3 MONTH AGED & ABRADED BLOCKS

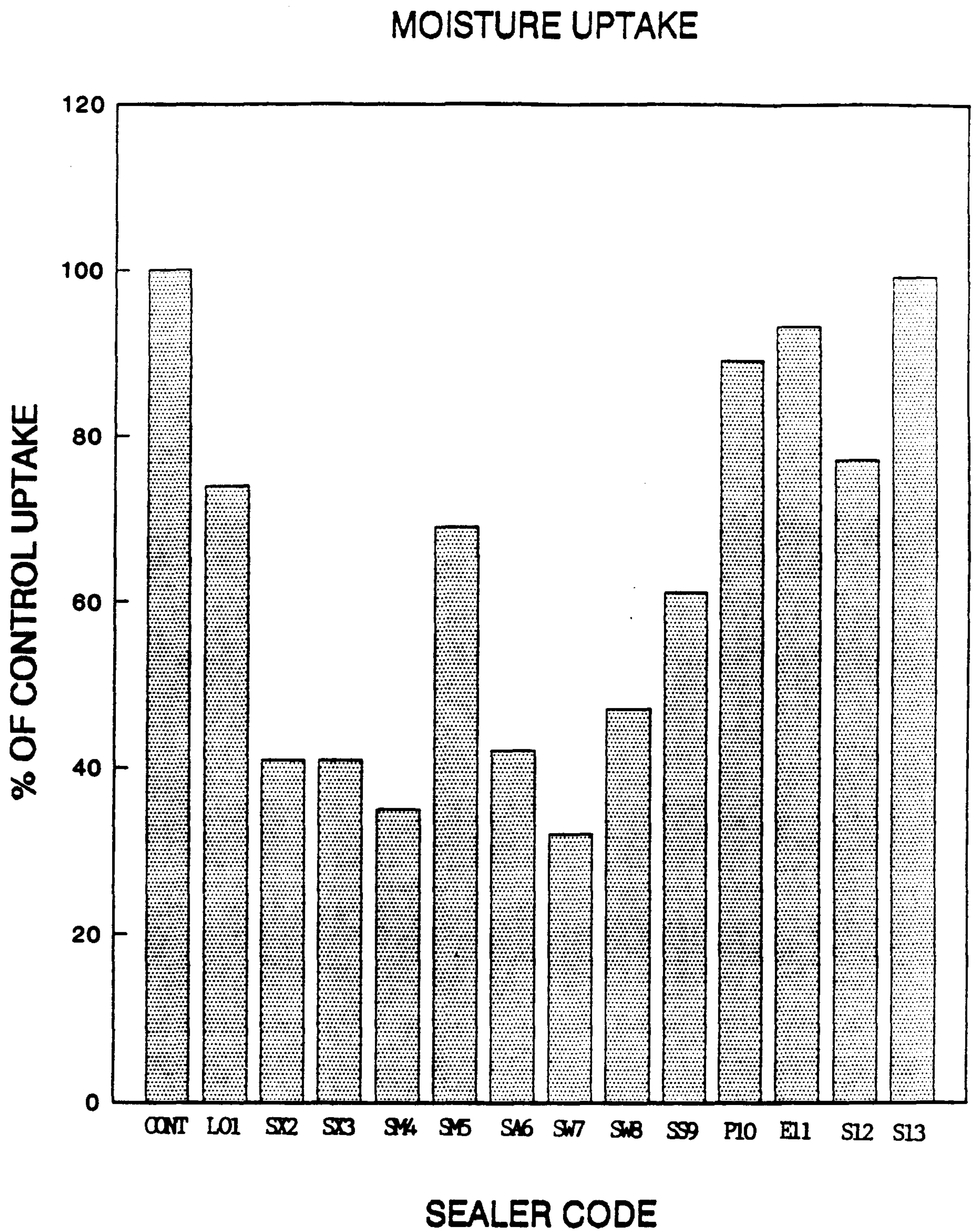


FIGURE G4

occurs more slowly than in this test so that as new surface is exposed, the linseed oil at that level presumably has an opportunity to cure.

With the exception of SM5, the silanes and siloxanes retained their effectiveness suggesting excellent depths of penetration. The continuing poor performance of SM5 poses the likelihood that a silane content of 20 percent is too dilute to adequately protect concrete from moisture intrusion.

The silicates demonstrated marginal protection relative to the untreated blocks.

Addendum: An abbreviated abrasion resistance test was performed on the aluminum stearate, A14, and the paraffin/alkyd, W15. Representative sealed blocks from the unaged regimen correspond to Figure G1. Immediately upon completion of processing in that test phase, these blocks were sandblasted, weighed and immersed in saline solution. Given the higher initial moisture content of these blocks compared with those subjected to the full fledged abrasion test, they should have had an advantage over the other sealed specimens. At the end of 7 days of immersion however, the aluminum stearate had already absorbed 50 percent of the moisture absorbed by the control blocks in the original test. The paraffin/alkyd, absorbing 45 percent, showed similar reduction of effectiveness. The findings verified that neither of the sealers penetrated to any great extent. (This conclusion is corroborated by Depth of Penetration Testing, discussed later.)

WEATHER-OMETER Weathering

500 Hours

Figure G5 shows the influence of 500 hours of accelerated weathering on sealer performance.

1000 Hours

Figure G6 shows the influence of 1000 hours of accelerated weathering on sealer performance.

Discussion: The effectiveness of the WEATHER-OMETER exposure in accelerating the rate of weathering is demonstrated by comparing the numbers generated in the test rack-aging phase, Figures G1, G2 and G3, with Figures G4 and G5. Whereas after a year of test rack aging, most of the sealers were still providing between 30 and 40 percent improvement of protection over unsealed specimens, most of the specimens exposed to the WEATHER-OMETER absorbed on the order of 90 percent of the amount taken up by control specimens.

As in the test rack-aging phase, the silane/siloxane group did the best job. The silanes retained their effectiveness over the full 1000 hours slightly better than the two siloxanes.

The linseed-oil-treated specimens performed on par with the silanes. Apparantly the ultraviolet light exposure in the WEATHER-OMETER assisted in the crosslinking of the linseed oil.

The deterioration in the performance of the polyester sealer, P10, with time reflected the vulnerability of unstabilized polyester resins to ultraviolet degradation.

The epoxy sealer performed poorly under the accelerated weathering conditions.

Application Rate Testing

Unaged Blocks

Figure G7 illustrates the influence of application rates on the performance of sealers evaluated during the unaged blocks regimen.

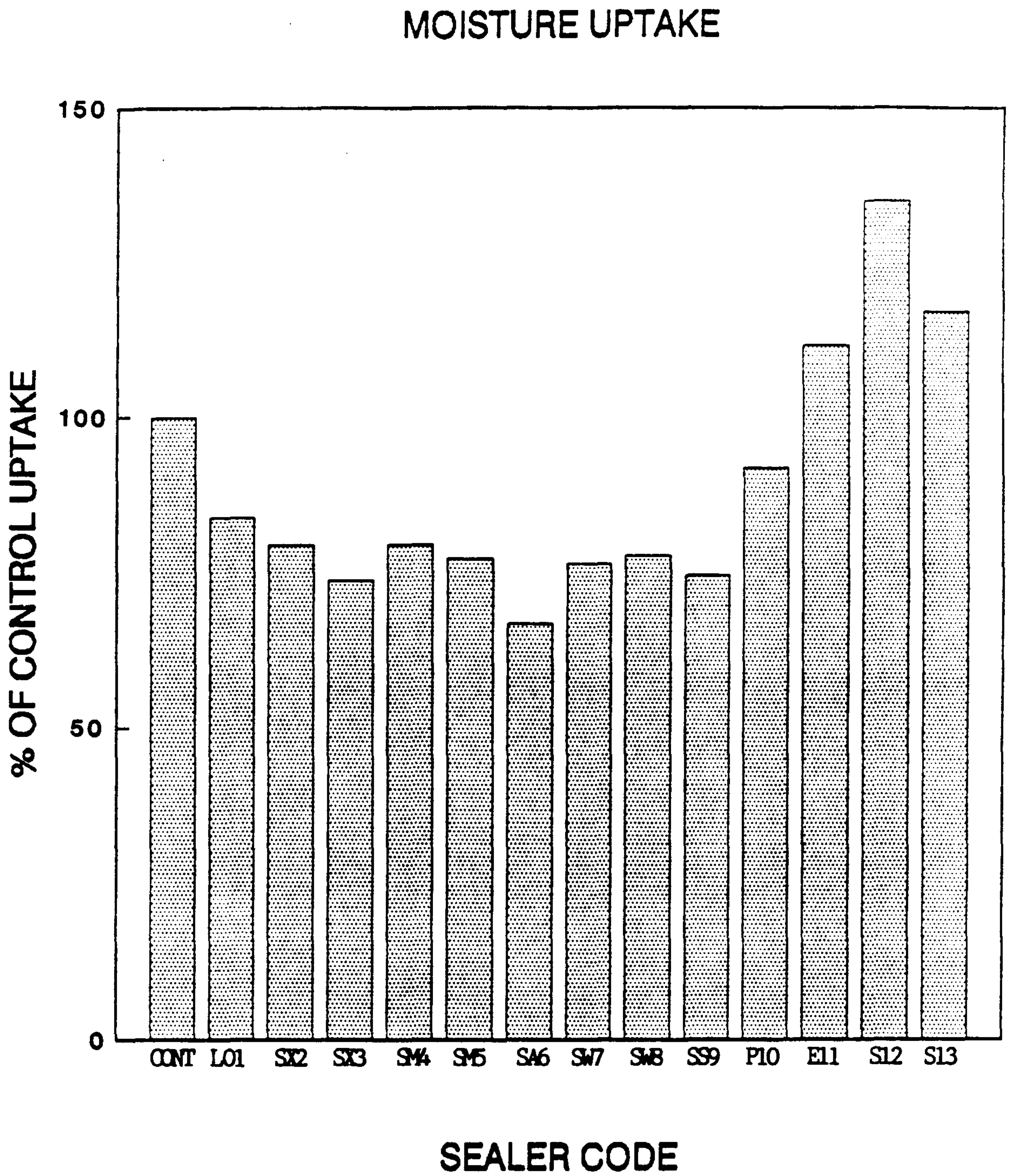
WEATHER-OMETER—1000 Hours

Figure G8 illustrates the influence of application rates in the moisture-sealing ability of sealers evaluated during the WEATHER-OMETER—1000-hours regimen.

Discussion: The silane/siloxane group showed a positive dependence on the rate of application. The most marked example was SM5, the 20 percent mineral-spirits-cut silane, which showed an appreciable reduction of performance in both of the application rate testing phases (i.e., unaged blocks and WEATHER-OMETER—1000 HOURS) when the application rate was cut by 25 percent..

The other group showing a marked sensitivity to application rate was the silicate group. Silicate sealers act by absorbing free pore water under alkaline conditions to form an "aero-silica gel." The nature of this pore-plugging gel is such that if it partially dehydrates, it can be reconstituted back to its original form by replacing the lost water. In effect, the gel acts more as a water reservoir than a barrier. Furthermore, due to the wetting ability of the silicate—sodium silicate is used as a wetting agent in the coating industry—the depth of penetration is limited only by the application rate: the

WEATHER-OMETER – 500 HOURS

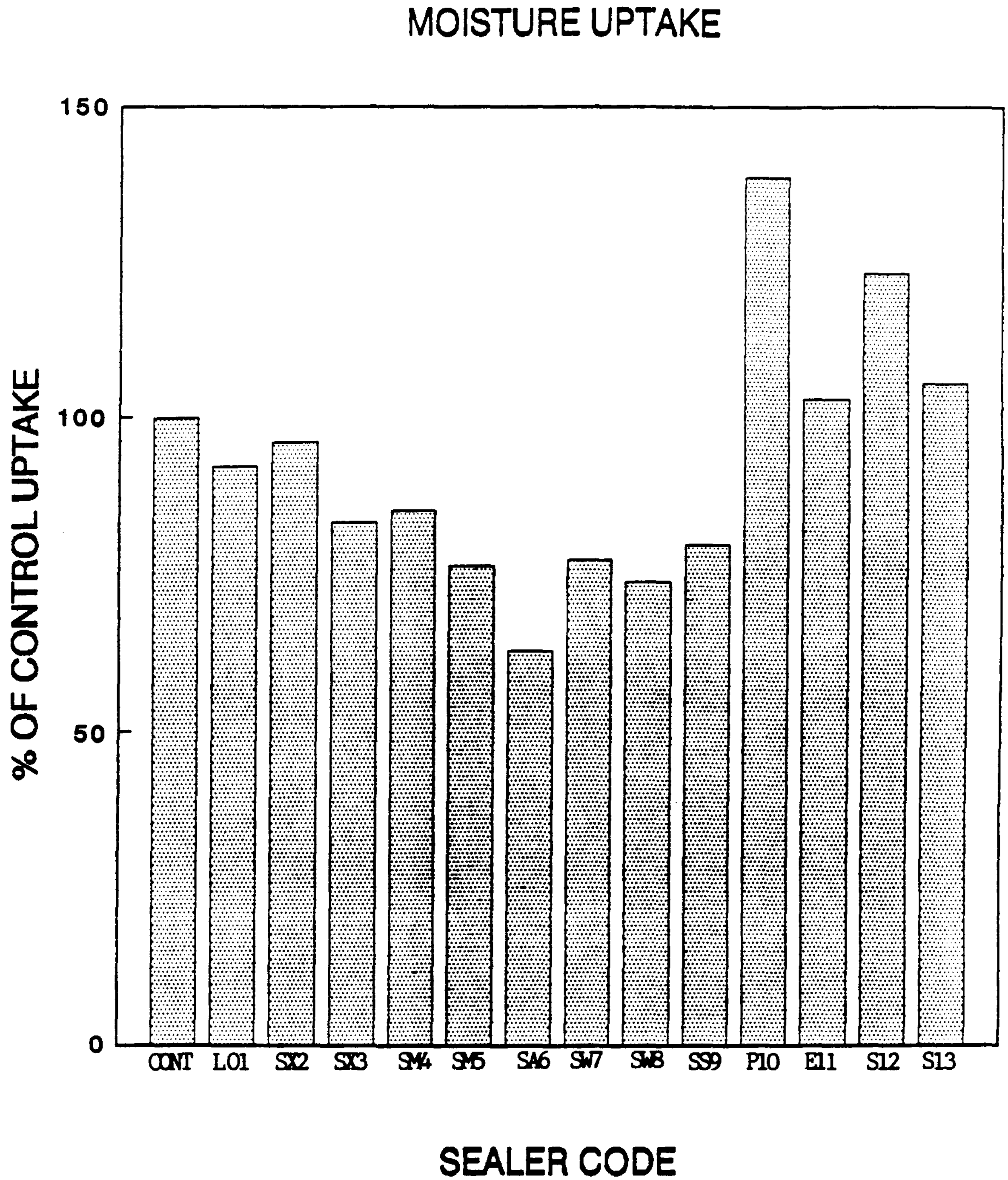


RESULTS ARE AVERAGES

OF TRIPLICATE SAMPLINGS

FIGURE G5

WEATHER-OMETER — 1000 HOURS

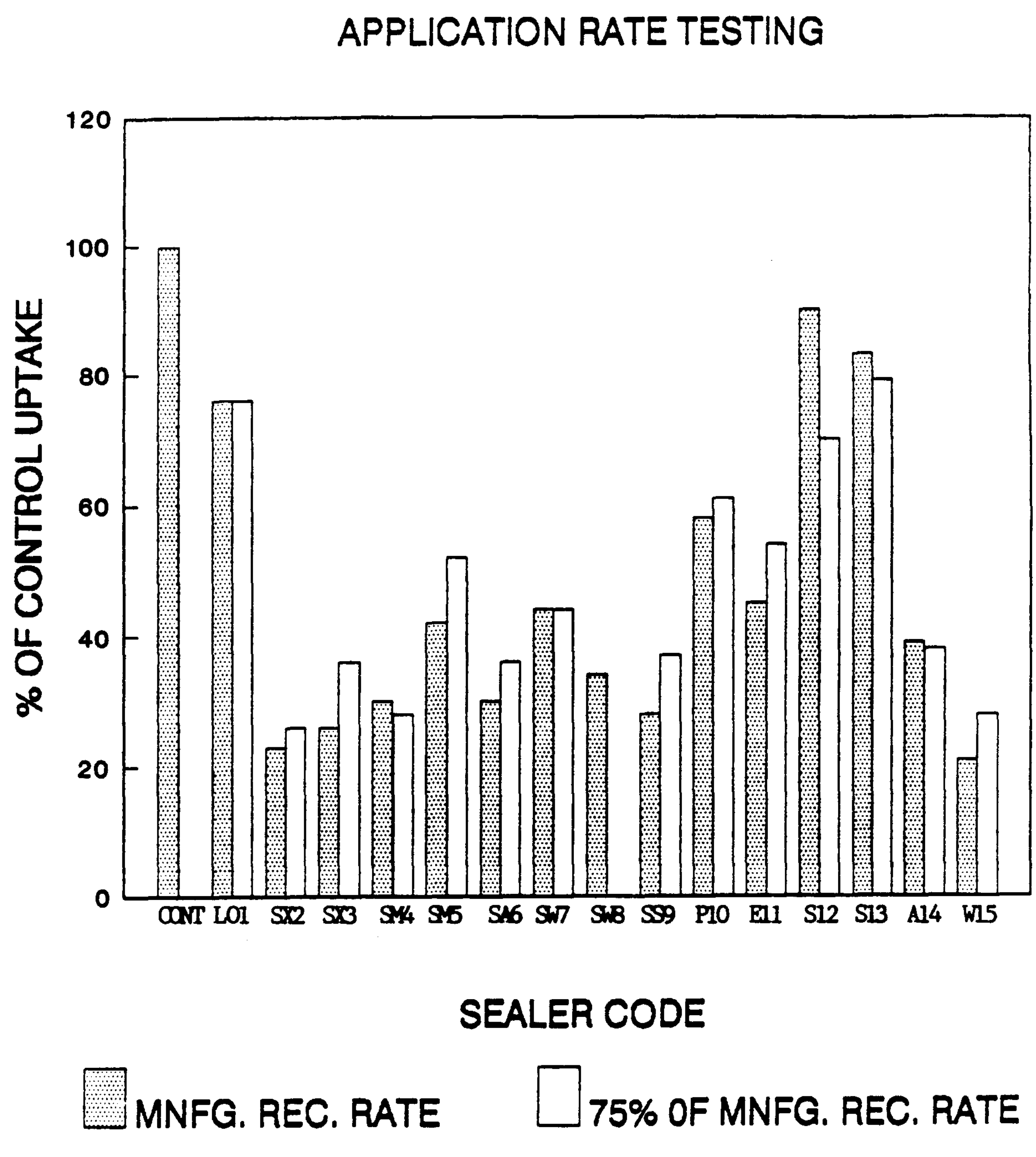


RESULTS ARE AVERAGES

OF DUPLICATE SAMPLINGS

FIGURE G6

UNAGED BLOCKS

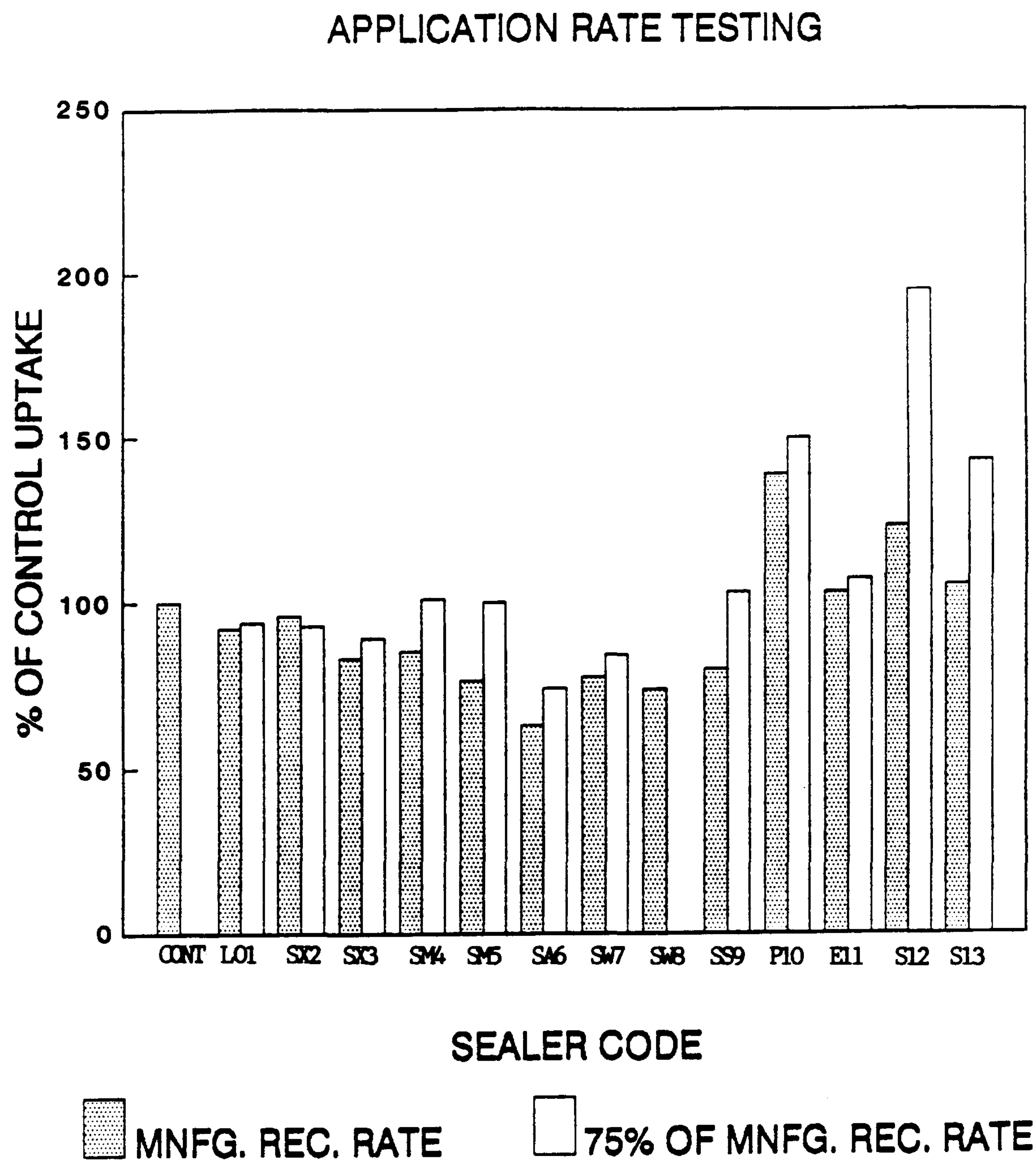


RESULTS ARE AVERAGES

OF TRIPLICATE SAMPLINGS

FIGURE G7

WEATHER-OMETER—1000 HOURS



RESULTS ARE AVERAGES

OF DUPLICATE SAMPLINGS

FIGURE G8

3 MONTH AGED & ABRADED BLOCKS

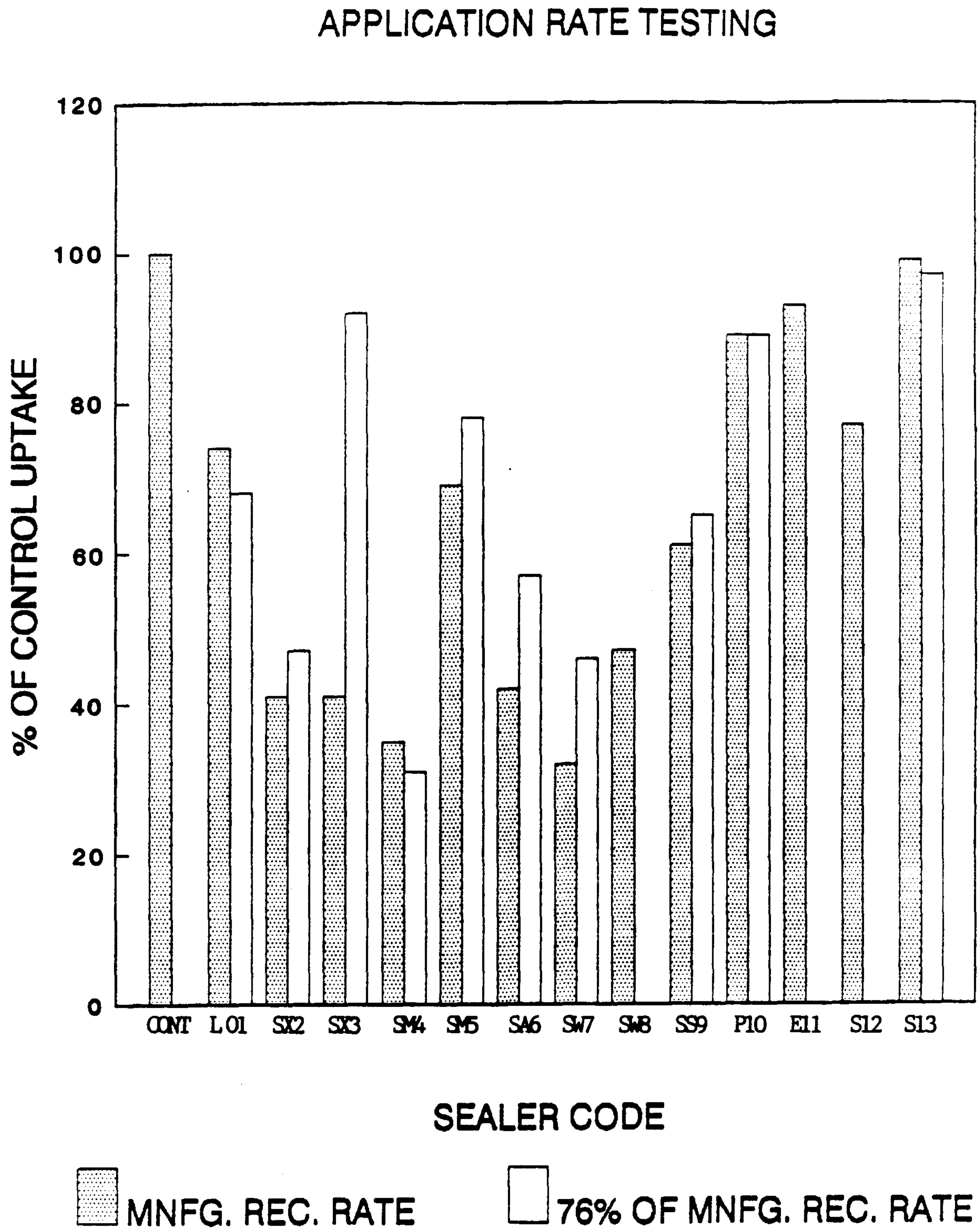


FIGURE G9

more you apply, the deeper it penetrates. Theoretically then, a departure from the behavior of the traditional moisture barrier-type sealer is anticipated; varying application rates can yield unexpected results. Figure G7 bears this out: the specimens coated at the manufacturers' recommended application rate absorbed substantially more moisture than those treated with 25 percent less.

Figure G8 demonstrates the fact that sealed blocks can actually absorb more moisture than unsealed, control blocks. The "reservoir" nature of the silicate group discussed previously explains their sponge-like absorption behavior. No explanation for similar behavior in the polyester, P10, has been determined.

Abrasion

Figure G9 shows the results of varying the application rate on waterproofing performance of blocks subjected to sandblast abrasion. No results are available for sealers SW8, E11 or S12.

The performance of the 10 percent siloxane sealer (SX3) dropped off dramatically with the 25 percent reduction in application rate. This behavior suggests that the 10 percent concentration of siloxanes may be too dilute for adequate, long term protection. All of the silane/siloxane group showed some dependence on the coverage rate during abrasion testing. The reduction of effectiveness of silanes and siloxanes when applied at light coverage rates and abraded to simulate tire wear (both phenomena occur in field applications), calls into question the assertion made by producers that these products never require reapplication.

The topic of application rates is considered further in connection with the Depth of Penetration Test.

Part II

Rapid Chloride Permeation Test

Sealers SM4 and SA6 were selected for testing using the Rapid Chloride Permeability Test (RCPT). Two untreated control cylinders were also run to assess the repeatability of the procedure and to provide a basis of comparison for the sealed specimens.

The data from the control runs are presented in Figures G10 and G11. The calculated values of total coulombs passed for the two runs differ by approximately 9 percent.

Figures G12 and G13 show the results of runs on SM4 and SA6.

In contrast to the 28-day immersion procedure which measures moisture protection, the RCPT provides a measure of the chloride-screening ability of a sealer. In either case, meaningful results can be obtained only in comparison with control specimens made from the same batch of concrete as the test specimens.

Taking an average for the RCPT control specimens gives 1738 coulombs. The ratios of results from sealers SM4 and SA6 to the control average are 41 percent and 37 percent respectively. The corresponding ratios obtained in the unaged blocks regimen, i.e., moisture uptake of sealed specimens ratioed with control uptake (See Figure G1) were 30 percent for both SM4 and SA6. These results indicate that neither of the two sealers tested effectively screen chlorides.

It is important to note that the RCPT measures electrophoretically induced chloride diffusion; an assumption has to be made that this transport mechanism is indicative of the potential for bulk convection and Fickian Diffusion of chlorides.

The RCPT requires careful sample preparation in order to obtain reproducible results. Such variables as sealer coverage rates, curing time of the concrete specimens, surface preparation and sealer curing time can exert disproportionate influence on the test results. It is unclear what effect metal-containing sealers (aluminum stearate, sodium silicate, etc.) would have on this test.

Water Vapor Permeability

Figures G14, G15 and G16 show the results of the water vapor permeability study. The data are presented for each specimen in terms of evaporation of moisture as a percentage of the 28-day moisture uptake. An arbitrary permissible threshold level of 50% of the permeability of the unsealed (control) specimen has been superimposed on each plot. None of the sealers exhibit vapor permeability problems using the 50% criterion.

RAPID CHLORIDE PERMEABILITY TEST

SPECIMEN: CONTROL #1

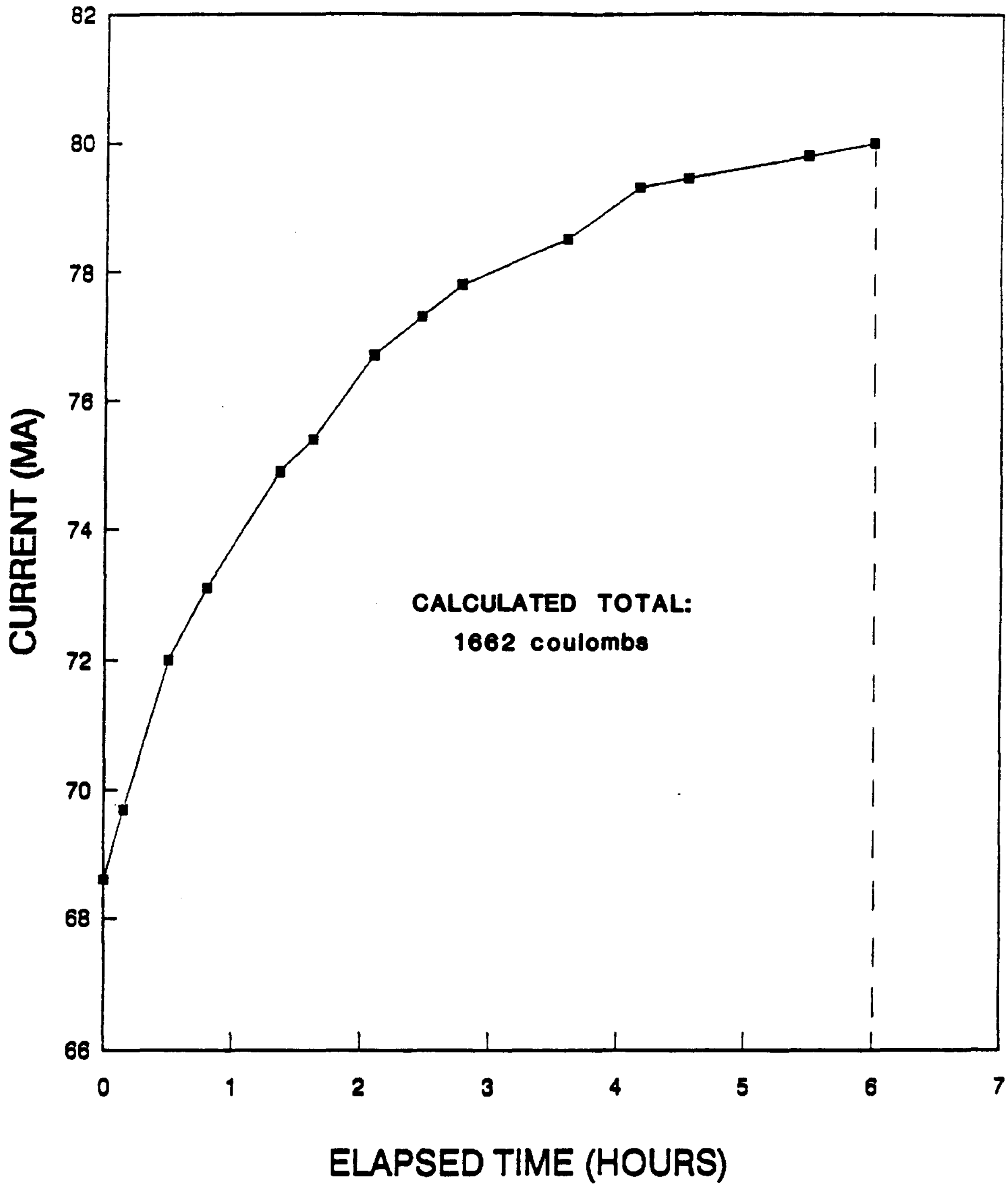


FIGURE G10

RAPID CHLORIDE PERMEABILITY TEST

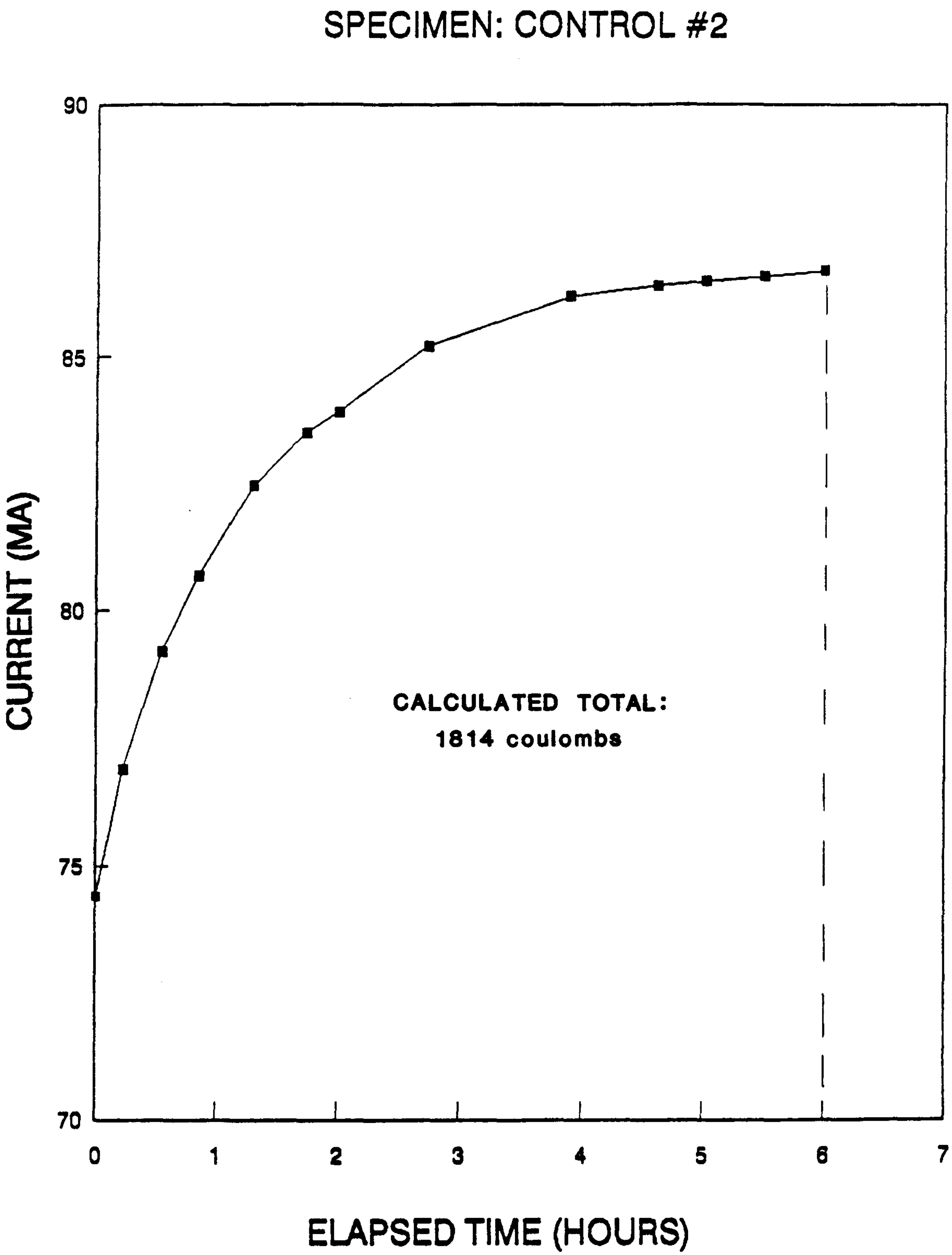


FIGURE G11

RAPID CHLORIDE PERMEABILITY TEST

SPECIMEN: SM4

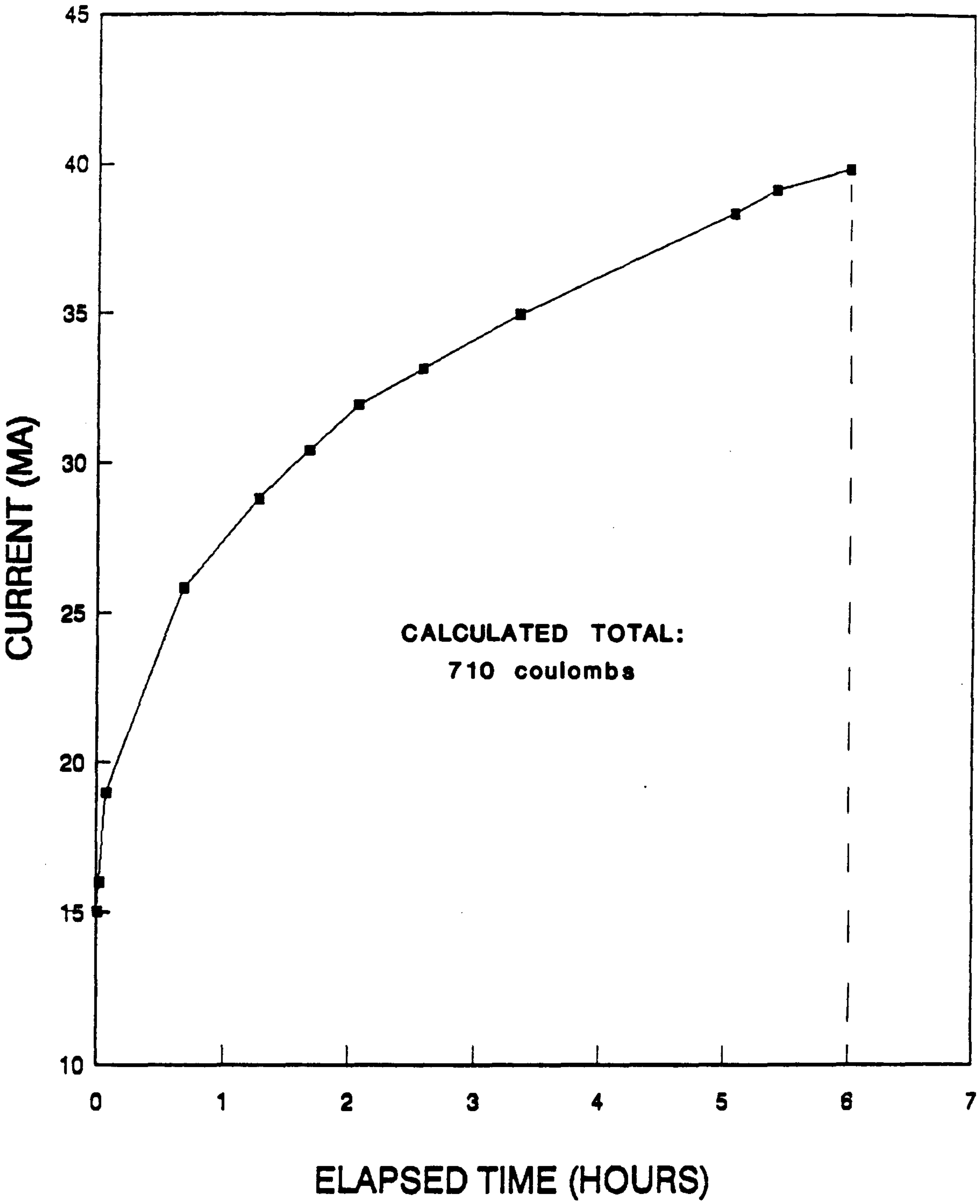


FIGURE G12

RAPID CHLORIDE PERMEABILITY TEST

SPECIMEN: SA6

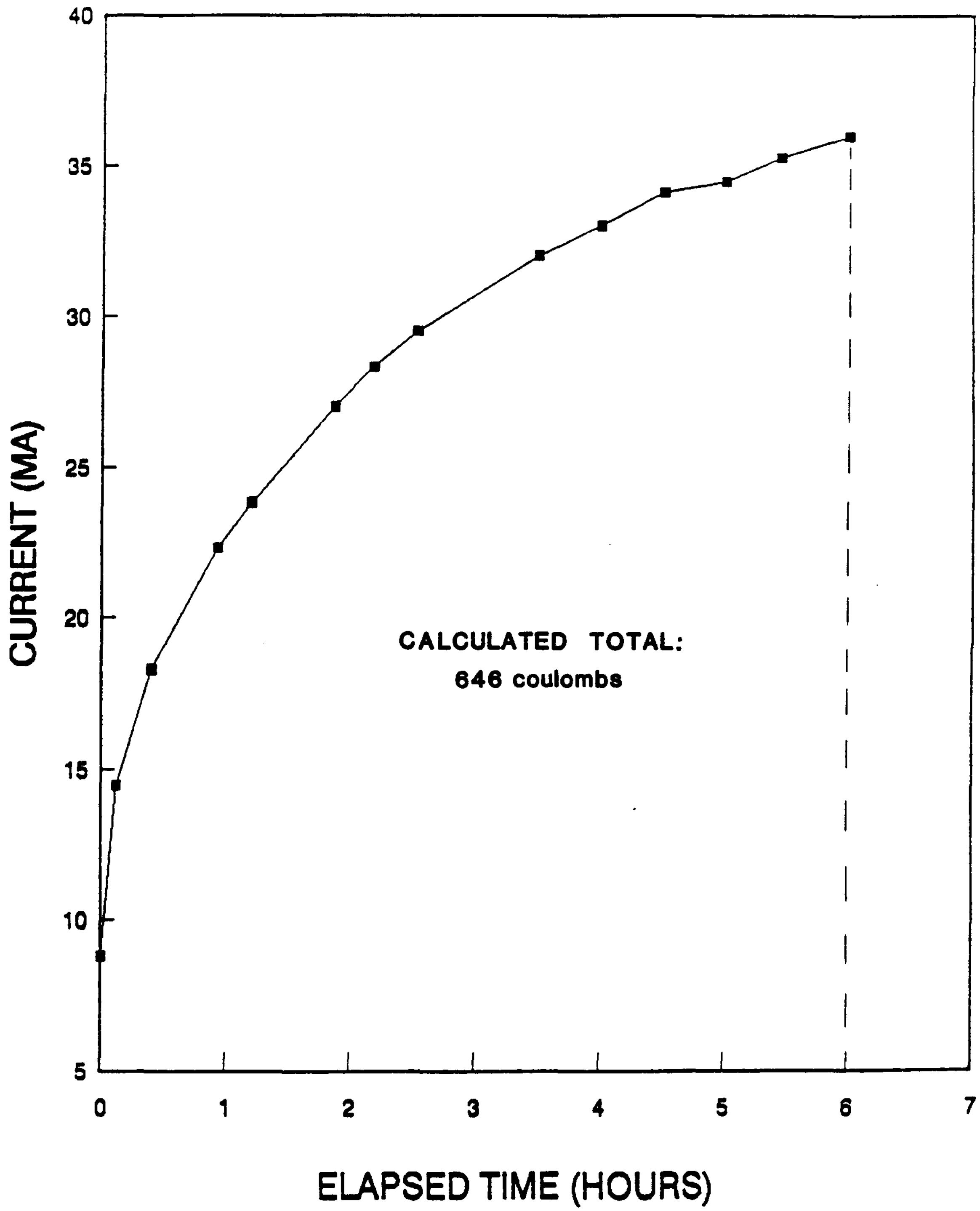


FIGURE G13

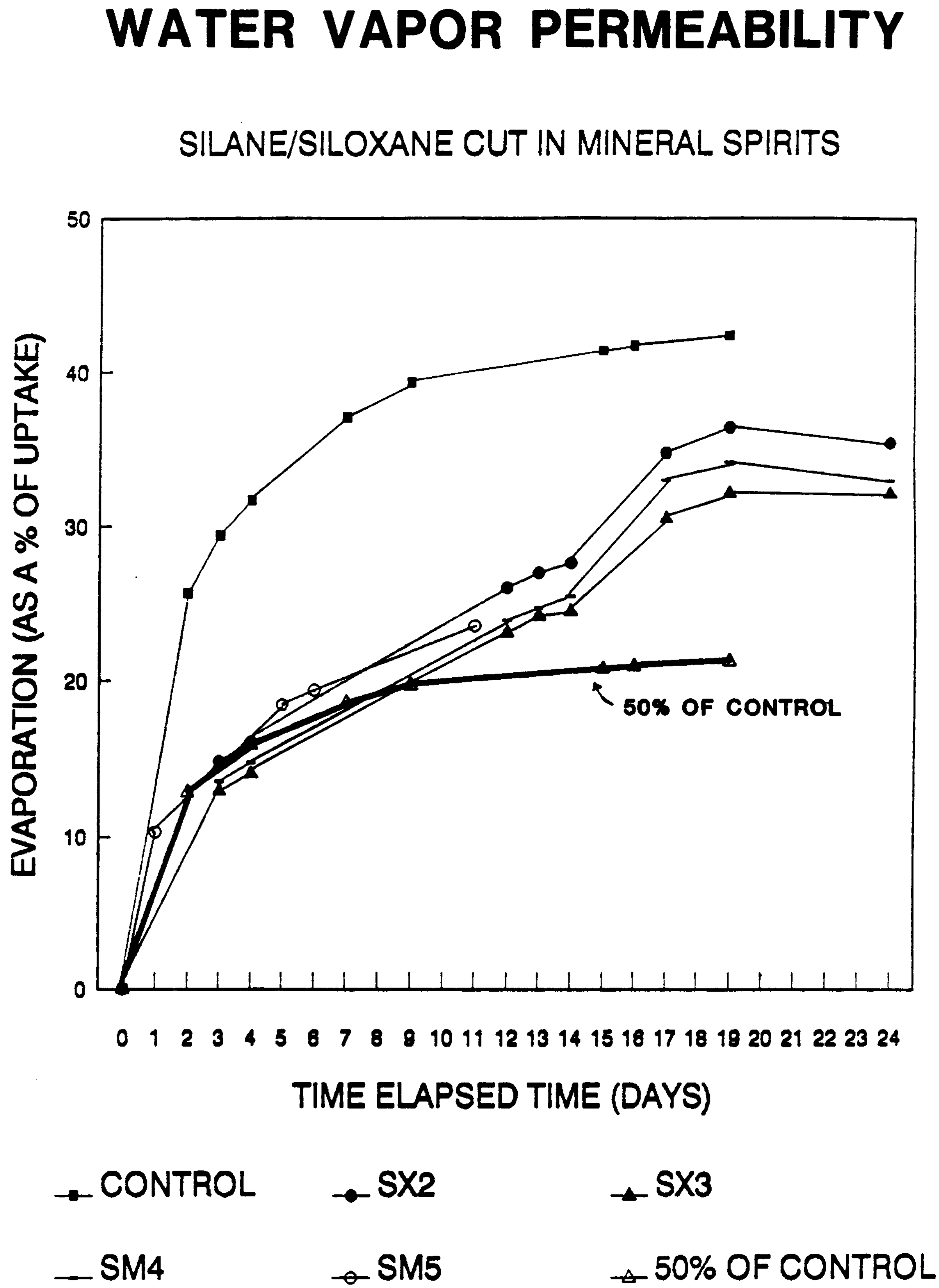

WATER VAPOR PERMEABILITY
SILANE/SILOXANE CUT IN MINERAL SPIRITS
EVAPORATION (AS A % OF UPTAKE)
0
10
20
30
40
50
50% OF CONTROL
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24
TIME ELAPSED TIME (DAYS)
CONTROL
SX2
SX3
SM4
SM5
50% OF CONTROL

FIGURE G14

WATER VAPOR PERMEABILITY

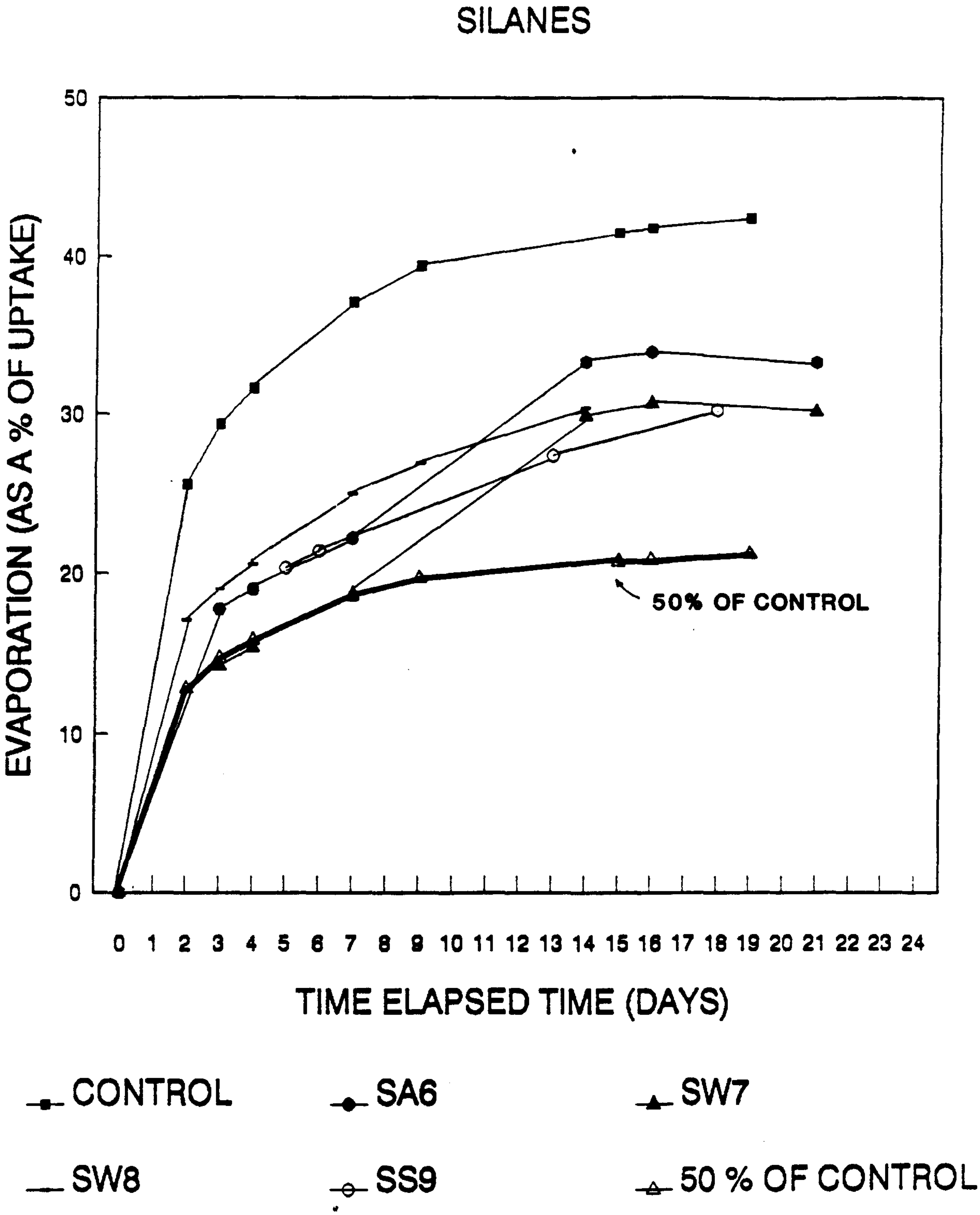


FIGURE G15

WATER VAPOR PERMEABILITY

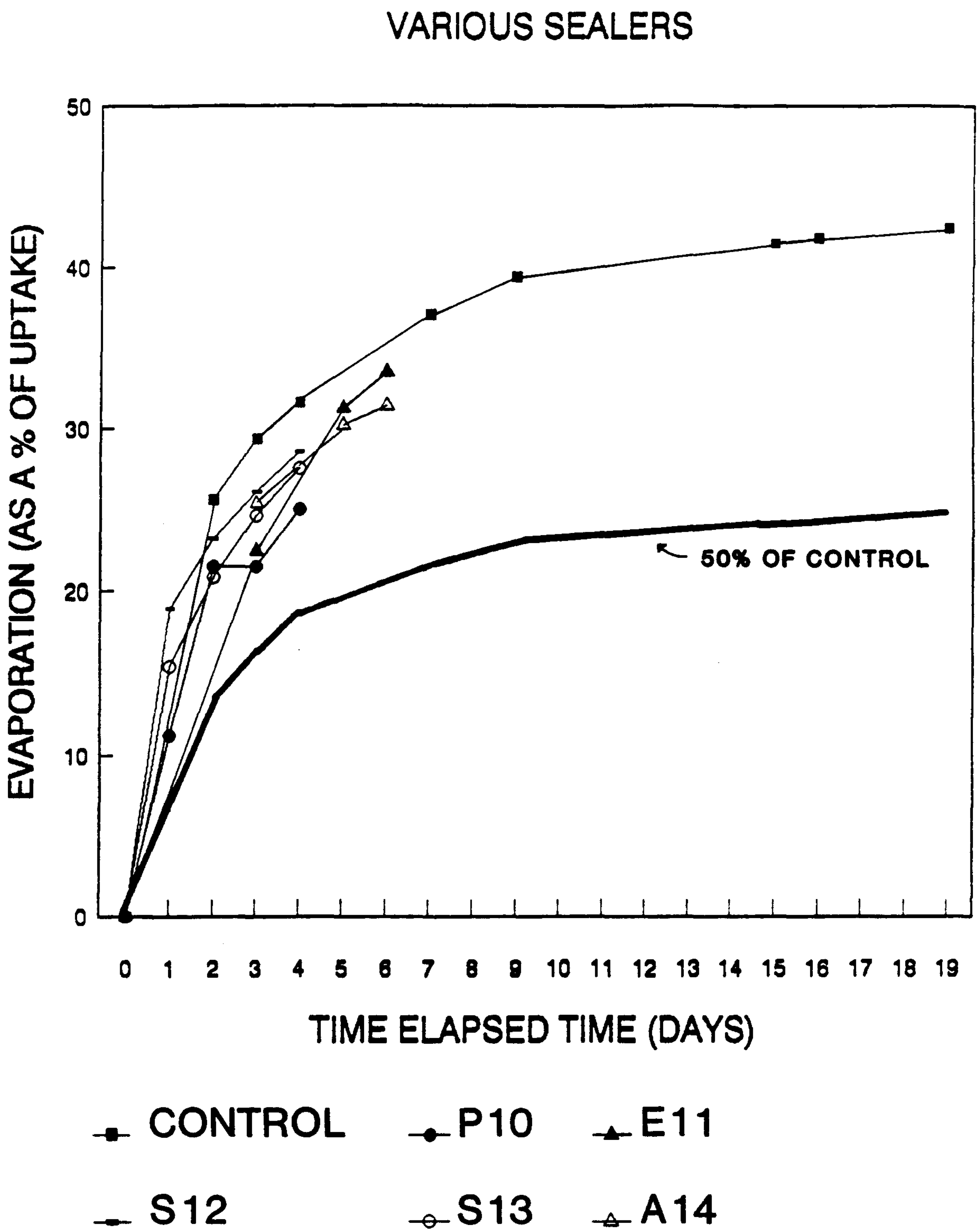


FIGURE G16

Instrumental Analysis of Silane-Sealers

Infrared Spectroscopy

Spectra of silane and siloxane sealers are included in Appendix D. Of particular interest is the characteristic Silicon-Oxygen-Silicon stretching vibration at wavenumber 1075 on the spectra of both siloxanes, SX2 and SX3. This peak can be used to indicate the presence of hydrolyzed material in a vendor sample.

Determination of Percent Residue in Silane/Siloane Sealers

Table 2 shows the results of residue determinations of the silanes and siloxanes tested in Part I. Also listed are the major solvent carriers, specific gravities and the percent nonvolatile material (NVM) contents. The NVM was obtained by placing a known weight of sealer in a 100 degree C oven for 3 hours and then weighing the remaining material. The value of the NVM was calculated by ratioing the remaining material with the initial weight.

TABLE 2 RESULTS OF RESIDUE TEST

MATERIAL	TYPE	SOLV	RESIDUE	GRAVITY	%NVM	PRODUCER CLAIM
SX2	Siloxane	MS	13.56	0.82	11.8	Proprietary
SX3	Siloxane	MS	13.74	0.97	12.5	10%
SM4	Silane	MS	16.45	0.9	16.1	Proprietary
SM5	Silane	MS	26.02	0.86	26.4	20%
SA6	Silane	IPA	21.57	0.82	2.33	40%
SW7	Silane	H_2O	28.3	0.95	8.1	40%
SW8	Silane	H_2O	26.76	0.95	*	40%
Isopropanol				0.78		
100% Silane				0.87		
MS				0.78		

* Not applicable: due to the kinetics of polymerization, the nonvolatile content of the water/silane mixture, SW8, depends upon the elapsed time subsequent to mixing.

A detailed discussion of the chemistry of this procedure is presented in Appendix C. The significance of this test can be understood by considering the data in Table 2. The standard procedure in the coatings industry for the determination of the percentage of "active" ingredient in a coating or sealer is the NVM procedure described above. This method is intractable for solutions where the "active" material is as volatile as its carrier. The data for SA6 and SW7 demonstrate the disparity between NVM and residue determinations. In polymerizing the monomeric silanes and the oligomeric siloxanes into a nonvolatile mass and accounting for the weight of the inert polymerizing catalyst, an accurate nonvolatile content may be calculated.

It is important to note that the residue content determined by this method can be markedly different from the percentage of active ingredient (silane, siloxane or a blend thereof) as formulated by the producer. The reason for this is that the hydrolysis and subsequent polymerization of two silane molecules result in the net loss of two volatile alcohol molecules. This hydrolysis and condensation polymerization is the same reaction that occurs when silanes and siloxanes are applied to concrete. For that reason, the residue content of a sealer is actually a more important parameter than the original silane or siloxane content because it indicates how much sealer will be left in the cured film.

The variety of inert or nonvolatile admixtures that are used in proprietary formulations preclude the use of this method to test selectively for silane content: the inert content shows up in the results with the silane. The test can be used effectively as a pass/fail for minimum silane or siloxane contents, however. It can also be used for quality control in that it can detect changes in proportions of formula components by the producer that would otherwise escape detection by qualitative IR spectroscopy.

RESULTS SUMMARY

Materials

The silicate group, represented by sealers S12 and S13, provided little discernable protection in any of the tests in which they were included. The assertion made by the silicate industry that silicates can effectively cure as well as seal was tested using Test Method Tex-219-F: "Testing of Concrete Curing Materials - Moisture Retention." The silicate tested, S13, lost 6 percent of the bleed water in 24 hours. *TEXAS STANDARD SPECIFICATION* Item 526 allows a maximum of 2 percent loss in 24 hours.

The epoxy and the polyester groups, represented by E11 and P10, are both unacceptable for use on riding surfaces because they tend to build on the surfaces rather than penetrate. Skid problems notwithstanding, the numbers collected during this evaluation show these sealers to be ineffective for long tern use as a protective sealant.

The paraffin/alkyd combination, W15, coats rather than penetrates also. The material didn't hold up well to an abbreviated abrasion resistance test. As with the epoxy and the polyester, skid problems are at issue.

The aluminum stearate, A14, appeared to penetrate slowly during application and the surfaces of coated specimens showed no signs of film build up; however, only minimal penetration (i.e., less than 1/32 inch) was found in any of the treated specimens. The abbreviated resistance test verified the penetration problems characteristic of this material.

SS9, a blend of silanes, siloxanes, aluminum stearate and oils showed problems with test rack aging and abrasion resistance. The material penetrated a fraction of the depth of the other silanes and siloxanes and is more accurately categorized as an aluminum stearate and oil spiked with silanes and siloxanes rather than as a silane-based material.

Silanes and Siloxanes

Sealer SW8, a mixture of 40 percent isobutyl-trimethoxy silane and water prepared at the time of application performed surprisingly well; however, its erratic penetration profile precludes it from serious consideration as a long term bridge deck protection strategy.

The water-based silane, SW7, is an environmentally attractive option. The material held up well in all of the courses except for depth of penetration where the application rate may not have been sufficient to allow comparable penetration with the other silanes. Some questions have been raised that need to be addressed concerning shelf life and the effect of freezing temperatures on the material while in storage .

The 20 percent silane, SM5, demonstrated problems in tests where the other silanes held up well. The implication is that 20 percent may be too low a concentration of silane to effect adequate protection.

SM4, the mineral-spirits-based silane, performed exceptionally well considering its relatively low residue content of 16 percent. The formula of this material is proprietary; however, its high specific gravity (see Table 2) suggests the possibility that it contains some siloxane or that the side group on the silane molecule is an octyl rather than the more commonly used isobutyl substitution. The IR spectrum of SM4 is inconclusive regarding either of these speculations.

Both of the siloxanes performed well in all phases of testing. The low concentration of siloxanes in the formulations compared with the silanes results in lower production costs. Speculations that the large molecule size of siloxanes restricts its penetration into concrete were not borne out in either the Abrasion Test or the Depth of Penetration Test.

EVALUATION OF TEST PROCEDURES

The application rate used when evaluating sealers should be based on residue content (either percent nonvolatile material or as determined using the silane residue test) rather than the manufacturer's recommendation. In order to standardize the evaluation and prequalification process, a coverage rate corresponding to 0.01191 lb. of residue per square foot should be used.

WEATHER-OMETER conditioning of sealed mortar coupons provided essentially the same test results as the test rack but in a fraction of the time. Since the mortar coupons are smaller and easier to handle, redundant sets of specimens can be run to increase the accuracy of statistical data.

The Depth of Penetration Test gives the same results as the Abrasion Resistance Test and incurs much less labor and testing time.

The Freeze/Thaw Test provides no information when used in conjunction with air-entrained concrete specimens regardless of the type of sealer applied.

The Rapid Chloride Permeability Test requires exacting preparation technique in order to obtain reproducible results even for untreated specimens.

Absolute values of coulombs passed are meaningless for the purpose of routine evaluations of sealers because the resistivity of concrete specimens vary from batch to batch. As a consequence, untreated companion specimens have to be run along with test specimens in order to obtain a relative indication of the performance of the sealer that could be rated against some performance standard.

The RCPT provides an indication of chloride permeability of concrete sealers but does not address the issue of waterproofing ability and therefore cannot be used effectively as a substitute for a water immersion test.

The Residue Content Test gives an indication of the silane and siloxane content of a given sealer; however, inert fillers, stabilizing agents and other admixtures can cause the result to be unrealisticly high. This test should therefore be used only as a minimum quality standard. In other word, if a sealer residue is found to be lower than a required standard, then that result can be used as a basis for rejection.

RECOMMENDATIONS

It is recommended that *TEXAS STANDARD SPECIFICATION* Item 428 be supplemented to permit the use of silane and siloxane-based penetrating sealers on new construction. The preparation of the deck surface should be the same as that required for linseed oil application as described in Item 428.3.

FURTHER WORK

Further research is needed in the area of long-term durability of both linseed oil and the silane/siloxane class of sealers. The substantial difference in material costs between the two classes of sealers (6) demands that, for a given level of protection, the silanes/siloxanes must last 10 times longer than linseed oil to justify their use.

The subtle differences between the silanes and the siloxanes warrant further investigation, particularly with regard to longevity. The evidence indicates that isooctyl siloxanes can be applied more sparingly (i.e., at lower concentrations) than silanes but, here again as with linseed oil, the cost benefit cannot be considered in a vacuum—it must be supported by longevity.

REFERENCES

1. BABAEI, K., and HAWKINS, N., "Evaluation of Bridge Deck Protective Strategies", NCHRP Report 297, 1987.

2. Speciality Products Evaluation List, AASHTO, 1985.

3. Test Method Tex-403-A, September 1984, *MANUAL OF TESTING PROCEDURES*, Texas State Department of Highways and Public Transportation.

4. HAGEN, Arnulf, "Silanes: Synthesis, Properties and Reactions", University of Oklahoma Press, No Date Cited.

5. PERENCHIO, William, "Durability of Concrete Treated with Silanes", *Concrete International* (November 1988): 34-40.

6. PFEIFER, D., and SCALI, M., "Concrete Sealers for Protection of Bridge Structures", *NCHRP Report 244*, 1981.

APPENDICES

APPENDIX A: Batch mix design for Concrete Block Specimens & Mortar Coupon Specimens

Concrete Block Batch Mix Design. Modified form Class S, Standard Specification Item 421.9, this design uses #4 aggregate exclusively in lieu of the specified four different grades.

Material	Quantity
#4 Aggregate	13.7 lbs
Type 1 cement	4.14 lbs
Fine Aggregate*	9.51 lbs
Deionized water	1.98 lbs
Vinsol Resin (A.E.A.)	1.2 cc
Con Ad N (plast.)	5.0 cc

* Fineness modulus range = 2.3 – 3.1
7 day strength = 4823 psi.
Air content = 4.5%

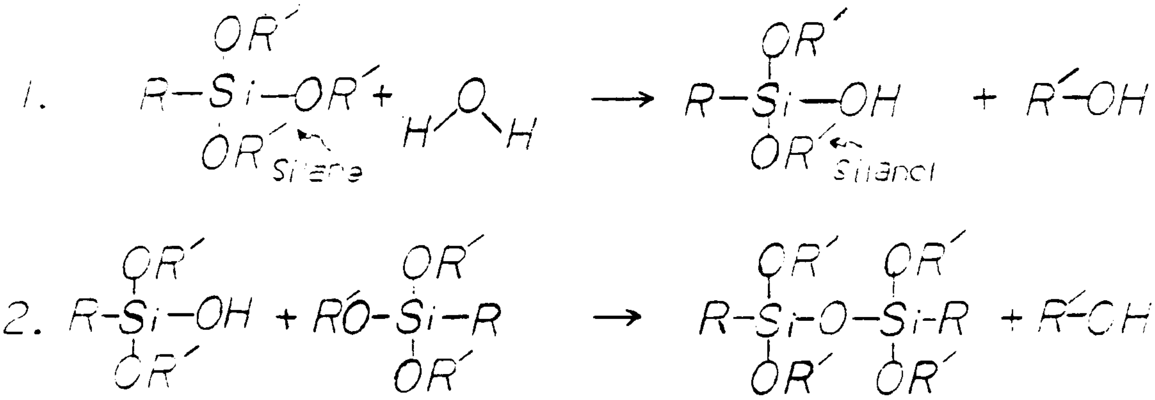
Mortar Coupon Batch Mix Design. Based on design used for Test Method Tex-219-F: Test for Vertical Sag of Concrete Curing Membranes.

Material	Quantity
Fine Aggregate*	408.6 grams
Type 1 cement	365.4 grams
Deionized water	129. grams

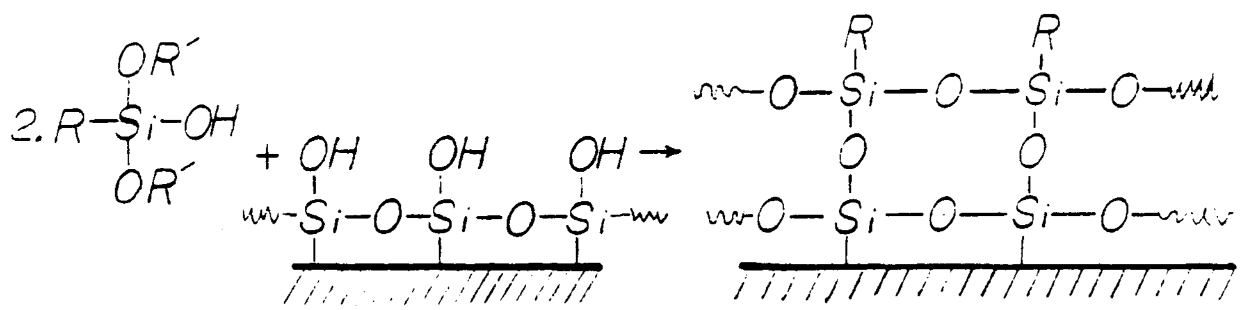
* Fineness modulus range = 2.3 - 3.1

APPENDIX B: Silane and Siloxane Chemistry

The alkylalkoxy silane molecule is a monomer which forms polysiloxanes in the presence of water as follows:



In treating concrete with silanes or siloxanes, the silanol or polysilanol, respectively, reacts with the silica in the concrete to form a rigid bond. When fully cured, the silane/siloxane sealer consists of a tightly crosslinked silicone resin with hydrophobic hydrocarbons—the R groups in the diagram—extending into the pore complex:



Most producers use either isobutyl or isooctyl R groups in their formulations. The isooctyl groups impart a higher level of hydrophobicity; the cost being that a 40 percent by weight isooctyl silane contains only 75 percent of the molar concentration of a 40 percent isobutyl silane. The lower molar concentration implies a lower level of silicon bonding with possible implications in terms of durability and longevity.

APPENDIX C: Test Method for Silane- and Siloxane-Type Sealers

Infrared spectroscopy is used to verify the silane or siloxane nature of the candidate sealer. The spectrum is retained for a standard to check for changes in formulation in subsequent submissions. Once a material is placed on an approved list, IR spectroscopy becomes the most important component of quality monitoring of routine samples.

Percent residue testing determines the weight percent of the active ingredient—silane or siloxane—plus any inert material. Test results are compared against a specified minimum residue content.

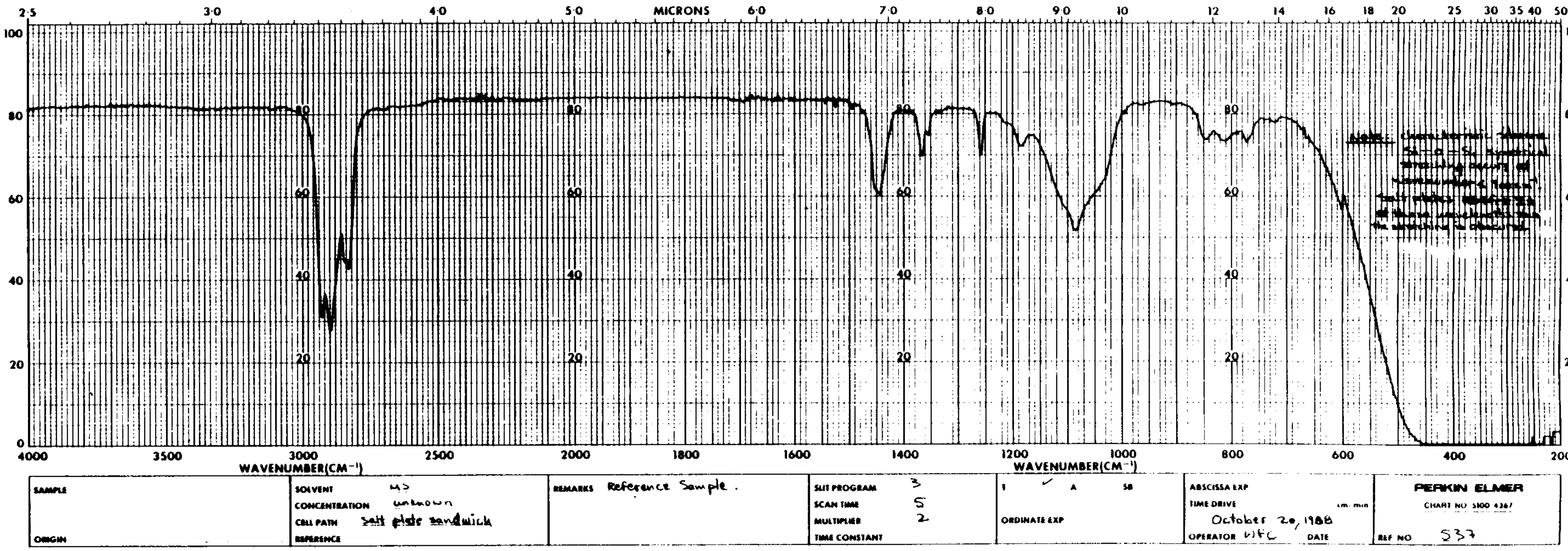
The results from the residue determination are used to calculate a normalized coverage rate for a Depth of Penetration Test. Sealers are applied to specimens sawed from a 6" × 6" concrete beam at a coverage rate corresponding to 0.01191 lb. of residue per square foot. Actual coverage rates to be used must be back-calculated using percent residue contents previously determined. Sealers are allowed to cure at room temperature for 7 days before the specimens are fractured and the exposed edges are dipped in water to reveal the depth of the nonwetted margin. Depths of penetration are measured at three locations and the average is compared to a minimum standard.

Finally, a WEATHER-OMETER Program tests for long term durability. 3" × 6" × 3/8" mortar coupons are dried to constant weight in a 140 F oven and then are allowed to equilibrate under conditions of controlled humidity so that specimen weights fluctuate less than 0.1 percent in a 24-hour period. Edges of specimens are waterproofed with epoxy or other suitable coating. Sealers are applied to front and back surfaces at the same application rates as used in the Depth of Penetration Test and allowed to cure in lab conditions for 7 days before weathering.

WEATHER-OMETER exposures using cycles of 18 minutes of sunshine and rain followed by 102 minutes of sunshine have proven effective. The 500 hours of exposure are followed by weight equilibration as described in the preceding paragraph. The specimens are then immersed in 5 percent saline solution for 28 days whereupon they are removed, towel-dried so that no surface moisture is visible, and weighed. The results are reported as percent moisture uptake.

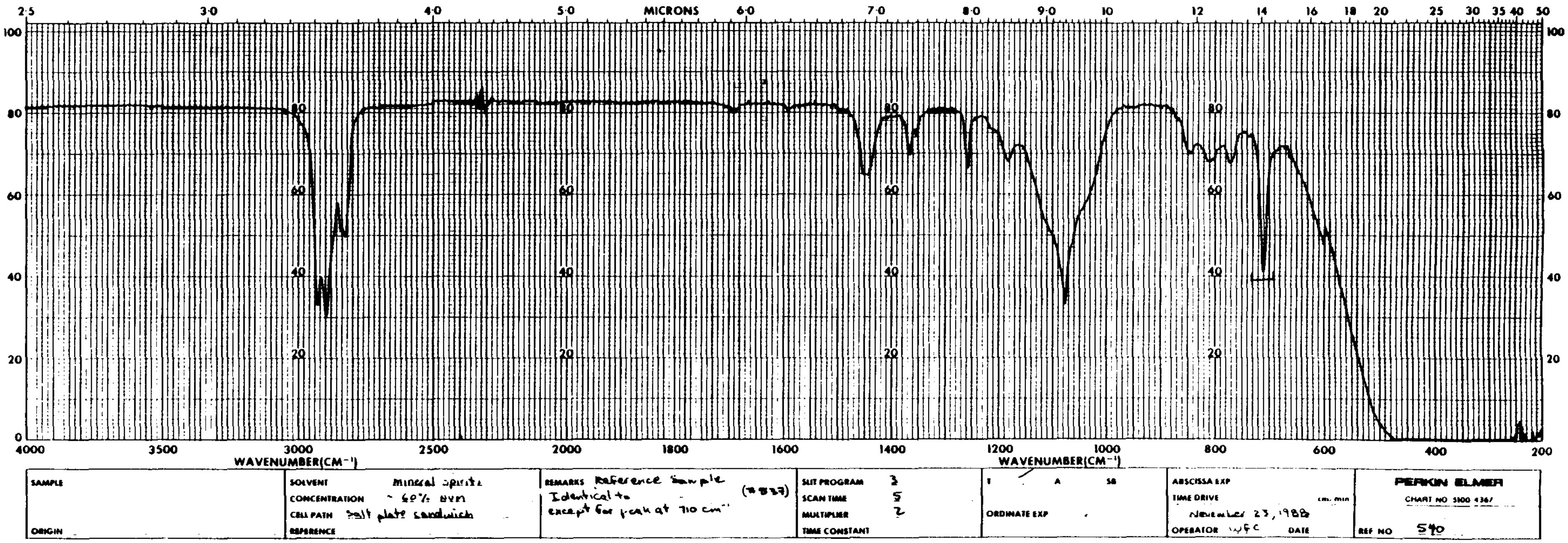
APPENDIX D: Residue Determination

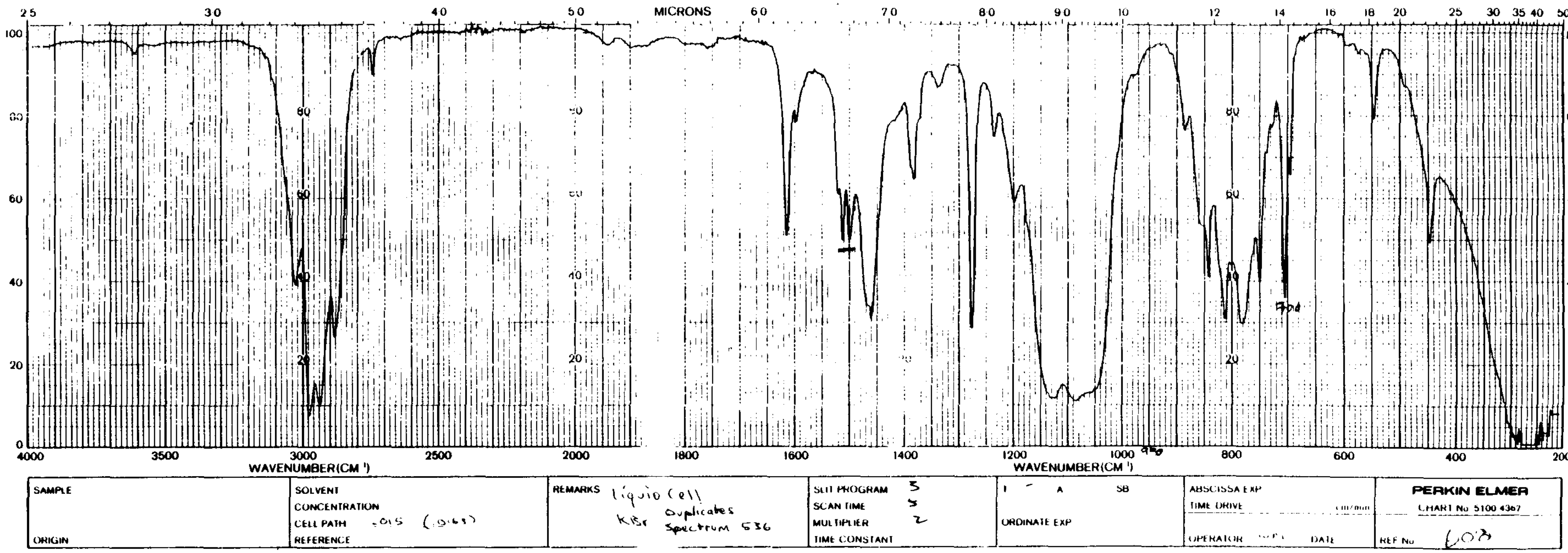
The residue determination is accomplished by hydrolysis and subsequent polymerization of silanes or siloxanes as described in steps 1 and 2 of Appendix B. The hydrolysis and polymerization reactions are driven to completion by using paratoluene sulfonic acid as a catalyst. With the active ingredient thus rendered nonvolatile, the two alcohol groups generated in steps 1 and 2 and the carrier solvent are removed via drying in an oven. If the identity of the alcohols is known, the original weight percent of the silane or siloxane material may be back-calculated using the percent residue data (providing the sealer contains no inert solids). In the more general case involving a proprietary formulation of silanes or siloxanes (or both), only the percent residue can be determined. It should be noted that the residue content is the more significant parameter since it relates how much material is available ultimately for the protection of the concrete. As an example, a 25 percent solution of isobutyl silane would yield more cured end product than a 30 percent solution of isooctyl silane.


MICRONS
WAVENUMBER(CM⁻¹)
SAMPLE
ORIGIN
SOLVENT MS
CONCENTRATION unknown
CELL PATH salt plate sandwich
REFERENCE
REMARKS Reference Sample.
SLIT PROGRAM 3
SCAN TIME 5
MULTIPLIER 2
TIME CONSTANT
ORDINATE EXP
ABSCISSA EXP
TIME DRIVE
October 20, 1988
OPERATOR
DATE
PERKIN ELMER
CHART NO 5100 4367
REF NO 537

SEALER SX2

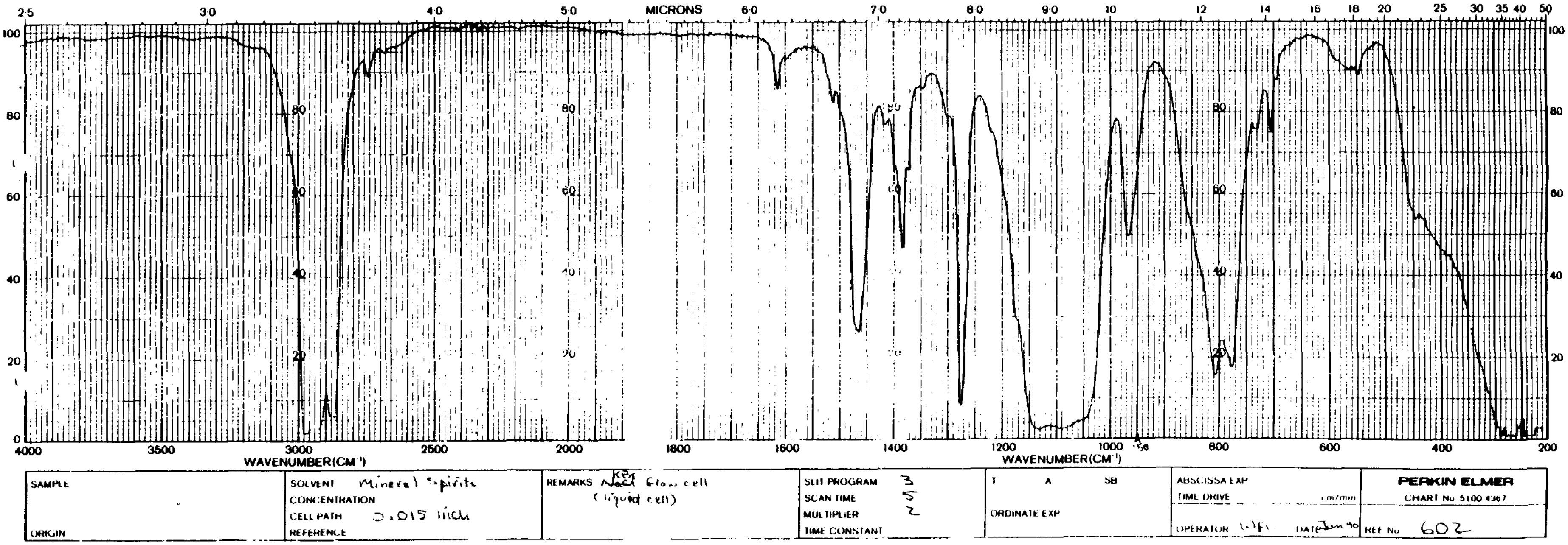
SEALER SX3


MICRONS
WAVENUMBER(CM⁻¹)
SAMPLE
ORIGIN
SOLVENT mineral spirits
CONCENTRATION ~ 60% w/v
CELL PATH salt plate sandwich
REFERENCE
REMARKS Reference Sample
Identical to (#537)
except for peak at 710 cm⁻¹
SLIT PROGRAM 3
SCAN TIME 5
MULTIPLIER 2
TIME CONSTANT
ORDINATE EXP
ABSCISSA EXP
TIME DRIVE
November 23, 1988
OPERATOR WFC DATE
PERKIN ELMER
CHART NO 5100 4367
REF NO 540


MICRONS
WAVENUMBER(CM⁻¹)
SAMPLE
ORIGIN
SOLVENT
CONCENTRATION
CELL PATH .015 (.0163)
REFERENCE
REMARKS liquid cell
Duplicates
KBr Spectrum 536
SLIT PROGRAM 5
SCAN TIME 5
MULTIPLIER 2
TIME CONSTANT
ORDINATE EXP
ABSCISSA EXP
TIME DRIVE
OPERATOR
DATE
PERKIN ELMER
CHART No 5100 4367
REF No 603

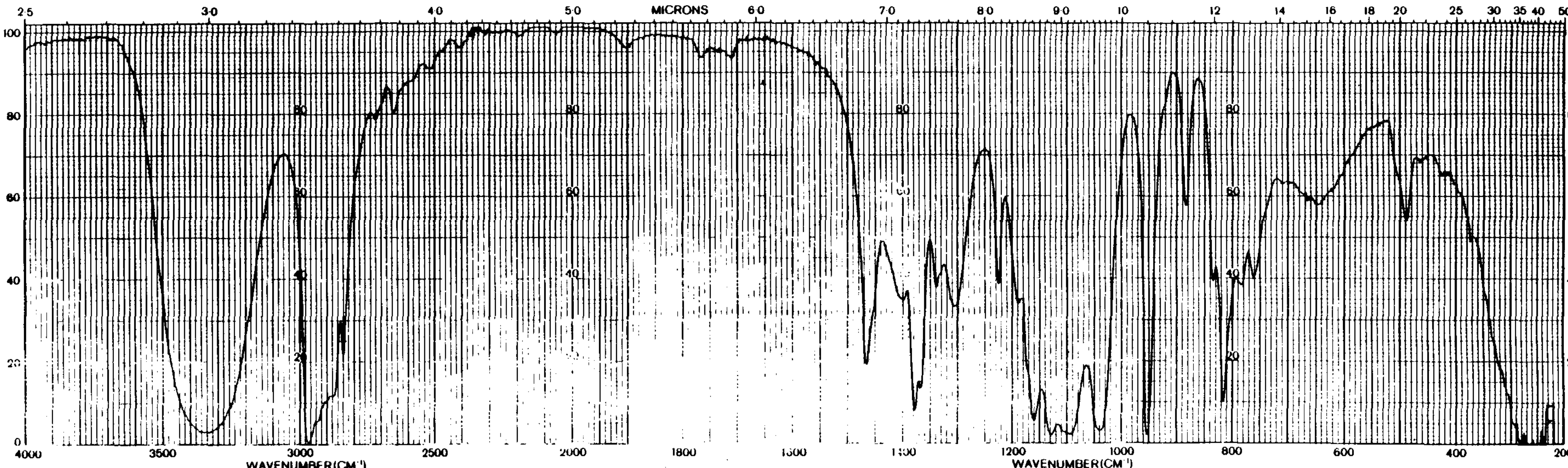
SEALER SM4

SEALER SM5



MICRONS
WAVENUMBER(CM⁻¹)
SAMPLE
ORIGIN
SOLVENT Mineral Spirits
CONCENTRATION
CELL PATH 0.015 inch
REFERENCE
REMARKS NaCl KBr Flow cell (liquid cell)
SLIT PROGRAM 3
SCAN TIME
MULTIPLIER 2
TIME CONSTANT
T A SB
ORDINATE EXP
ABSCISSA EXP
TIME DRIVE
OPERATOR
DATE
PERKIN ELMER
CHART No 5100 4367
REF No 602

41



SAMPLE ORIGIN	SOLVENT isopropanol CONCENTRATION ~40% NVM CELL PATH .0163 inch REFERENCE N/A	REMARKS liquid cell. Duplicates Spectrum S34	SLIT PROGRAM 3 SCAN TIME 5 MULTIPLIER 2 TIME CONSTANT	T A SB ORDINATE EXP	ABSCISSA EXP TIME DRIVE cm/min OPERATOR DATE 1/31/90

PERKIN ELMER
CHART No. 5100 4367
REF No. 609

SEALER SA6