

NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM  
REPORT **16**

# **PROTECTIVE COATINGS TO PREVENT DETERIORATION OF CONCRETE BY DEICING CHEMICALS**

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RESEARCH SPONSORED BY THE AMERICAN ASSOCIATION  
OF STATE HIGHWAY OFFICIALS IN COOPERATION  
WITH THE BUREAU OF PUBLIC ROADS

SUBJECT CLASSIFICATIONS:  
GENERAL MATERIALS  
MAINTENANCE, GENERAL

HIGHWAY RESEARCH BOARD OF THE DIVISION OF ENGINEERING AND INDUSTRIAL RESEARCH  
NATIONAL ACADEMY OF SCIENCES - NATIONAL RESEARCH COUNCIL 1965

## NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM

Systematic, well-designed research provides the most effective approach to the solution of many problems facing highway administrators and engineers. Often, highway problems are of local interest and can best be studied by highway departments individually or in cooperation with their state universities and others. However, the accelerating growth of highway transportation develops increasingly complex problems of wide interest to highway authorities. These problems are best studied through a coordinated program of cooperative research.

In recognition of these needs, the highway administrators of the American Association of State Highway Officials initiated in 1962 an objective national highway research program employing modern scientific techniques. This program is supported on a continuing basis by funds from participating member states of the Association and it receives the full cooperation and support of the Bureau of Public Roads, United States Department of Commerce.

The Highway Research Board of the National Academy of Sciences-National Research Council was requested by the Association to administer the research program because of the Board's recognized objectivity and understanding of modern research practices. The Board is uniquely suited for this purpose as: it maintains an extensive committee structure from which authorities on any highway transportation subject may be drawn; it possesses avenues of communications and cooperation with federal, state, and local governmental agencies, universities, and industry; its relationship to its parent organization, the National Academy of Sciences, a private, nonprofit institution, is an insurance of objectivity; it maintains a full-time research correlation staff of specialists in highway transportation matters to bring the findings of research directly to those who are in a position to use them.

The program is developed on the basis of research needs identified by chief administrators of the highway departments and by committees of AASHO. Each year, specific areas of research needs to be included in the program are proposed to the Academy and the Board by the American Association of State Highway Officials. Research projects to fulfill these needs are defined by the Board, and qualified research agencies are selected from those that have submitted proposals. Administration and surveillance of research contracts are responsibilities of the Academy and its Highway Research Board.

The needs for highway research are many, and the National Cooperative Highway Research Program can make significant contributions to the solution of highway transportation problems of mutual concern to many responsible groups. The program, however, is intended to complement rather than to substitute for or duplicate other highway research programs.

This report is one of a series of reports issued from a continuing research program conducted under a three-way agreement entered into in June 1962 by and among the National Academy of Sciences-National Research Council, the American Association of State Highway Officials, and the U. S. Bureau of Public Roads. Individual fiscal agreements are executed annually by the Academy-Research Council, the Bureau of Public Roads, and participating state highway departments, members of the American Association of State Highway

This report was prepared by the contracting research agency. It has been reviewed by the appropriate Advisory Panel for clarity, documentation, and fulfillment of the research plan. It has been accepted by the Highway Research Board and published in the interest of an effectual dissemination of findings and their application in the formulation of policies, procedures, and practices in the subject problem area.

The opinions and conclusions expressed or implied in these reports are those of the research agencies that performed the research. They are not necessarily those of the Highway Research Board, the National Academy of Sciences, the Bureau of Public Roads, the American Association of State Highway Officials, nor of the individual states participating in the Program.

NCHRP Project 6-3 FY '63

NAS-NRC Publication 1215

Library of Congress Catalog Card Number: 65-61650

## FOREWORD

*By Staff*

*Highway Research Board*

This report will be of interest to highway materials and maintenance engineers because of research results concerning the evaluation and development of protective coatings for concrete bridge decks, as well as other portions of structures, receiving frequent applications of deicing agents. Deteriorating effects are known to result when the deicing agents commonly in current use are applied to concrete. Because of diverse factors, all of which result in an ever-increasing use of deicing agents, impetus has been given to the requirement for protective coating materials of known capability in inhibiting deteriorating forces. On the basis of current knowledge concerning the mechanism of deterioration, this research has involved a critical evaluation of existing coating materials with a view toward developing new materials where potential has been indicated. A better knowledge has been gained in respect to the capabilities of existing materials, and the most promising have been delineated on the basis of protection and economy. Areas have been indicated where more detailed research on existing materials might be fruitful in developing improved and more durable coatings.

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It is generally accepted that concrete deterioration can occur in varying degrees due to attack by the deicing agents commonly used in snow and ice removal, and research continues to seek better agents as a solution to that particular problem. In the meantime, however, other research seeks to develop improved protective coatings, because the need for these is urgent and immediate in light of the many factors contributing to increased use of deicing agents in the northern tier of states. Foremost among these factors is the institution by many of the states of the so-called "bare pavement" policy, the net result being an environment in which deterioration can be accelerated. Design, construction practices, and construction control offer means whereby some compensation may be realized; however, until the research on deicing agents has been successfully consummated, the most positive assurance of protection lies within the province of coating materials of known protective capability.

The Battelle Memorial Institute conducted research to determine which of the potential protective coatings are most effective and economical and to develop new coatings meeting the same criteria. Laboratory freeze-thaw experiments were performed to evaluate the degree of resistance of the coatings to the attacking forces resulting from continued exposure to a 3 percent solution of sodium chloride brine. The degree of scaling was determined by visually comparing coated and uncoated specimens, both of which had air contents ranging from 1½ to 6 percent, with a standard set of specimens which the agency had prepared for this purpose and which exhibited varying degrees of scaling. Additional experimental studies were also conducted to determine whether physical or chemical processes, or any combination thereof, are responsible for scaling. The understanding of this phenomenon was held by the agency to be requisite to a rational approach in developing new protective coatings.

This report contains the details of evaluations which were made of 110 coatings representing a diverse assortment of organic and inorganic materials; however, only a few proved of value in protecting non-air-entrained concrete. Of these, linseed oil-based coatings performed best, with linseed oil solutions being superior to emulsions. Because of the degree to which other materials were surpassed in the experiments, research effort concentrated on the determination of optimum composition, application conditions, and application rate for linseed oil solutions. Investigations of different oils indicated that boiled linseed oils are more effective than raw and that variations may be encountered in boiled oils from different sources. Along with the linseed oil solutions, a sand-filled coal tar epoxy was found to perform in an excellent manner, the major difference being the increased cost over that of the solutions. Specimens coated with both of these materials sustained only moderate scaling in more than 200 freeze-thaw cycles.

Also contained in the report are the details of the experiments leading to the speculation that physical effects, rather than chemical, are primarily responsible for salt scaling, due to the existence of a gradient of salt concentration in the concrete. It is hypothesized that such a gradient results in excessive thermal and hydraulic pressures due to its effect on the freezing point of the liquid phase within the concrete.

This document constitutes a final report on the general subject matter of protective coatings for concrete structures. Although new, more effective coating materials have not evolved, the research is considered significant in other respects, one of these being the confirmation of the validity of current policies of many of the states in the use of linseed oil solutions. Conclusions have been reached concerning the relative merits of the various coating materials as compared with the performance to be expected from concrete which has been properly air-entrained and cured. Recommendations have been made for areas of further research in the development of coatings to protect structural concrete from attack by deicing agents. It is particularly felt that greater knowledge should be obtained of the specific mechanism by which linseed oil solutions work. Knowledge in this aspect could possibly provide guides for the development of new materials.

## **CONTENTS**

1	SUMMARY
2	CHAPTER ONE Introduction and Experimental Procedures Experimental Procedures
6	CHAPTER TWO Results of Coating Evaluations Linseed Oil Coatings Other Vegetable Oil Coatings Epoxy and Modified Epoxy Coatings Other Synthetic Resin Coatings Rubber-Base Coatings Inorganic Coatings and Treatments Miscellaneous Coatings
15	CHAPTER THREE Mechanism of Salt Scaling Experiments on Scaling Mechanism Conclusions from the Study of the Scaling Process
17	CHAPTER FOUR Conclusions and Recommendations General Use of Linseed Oil Coatings Further Research
19	REFERENCES
20	APPENDIX A Development of Salt-Scaling Evaluation Procedures
21	APPENDIX B Moisture Measurements

## FIGURES

- 3 Figure 1. Standard comparison slab No. 1, no scaling.
- 3 Figure 2. Standard comparison slab No. 2, slight scaling.
- 4 Figure 3. Standard comparison slab No. 3, moderate scaling.
- 4 Figure 4. Standard comparison slab No. 4, severe scaling.
- 5 Figure 5. Standard comparison slab No. 5, very severe scaling.
- 5 Figure 6. Partially-scaled coated slab.
- 16 Figure 7. Effect of presoaking concrete in brine on amount of scaling.
- 17 Figure 8. Thermally-scaled slab.

## TABLES

- 7 Table 1. Coatings Evaluated.
- 8 Table 2. Performance of Linseed Oil Solution Coatings.
- 10 Table 3. Effect of Pretreatment of Concrete on Effectiveness of Linseed Oil Solution.
- 11 Table 4. Absorption of Coated Concrete.
- 11 Table 5. Performance of Linseed Oil Emulsion Coatings.
- 12 Table 6. Performance of Vegetable Oil Coatings.
- 13 Table 7. Performance of Coatings Based on Synthetic Polymers or Elastomers.
- 14 Table 8. Performance of Inorganic Coatings.
- 14 Table 9. Performance of Coatings Based on Tars, Waxes, Greases, or Miscellaneous Organic Materials.
- 18 Table 10. Comparison of the Most Effective Coatings.
- 20 Table A-1. Effect of Air Content of Concrete on Scaling Rate.

## ACKNOWLEDGMENTS

The assistance and cooperation of many persons and agencies are acknowledged. The necessary concrete components and the various coating materials tested were cheerfully furnished to the project by the producers or manufacturers. Inquiries to various highway departments, universities, and private and public research groups yielded much helpful information.

M. Jack Snyder, Assistant Chief, Ceramics Division, Department of Materials Engineering, Battelle Memorial Institute, was the project director for the research reported herein. The responsible administrative officer for the project was W. H. Duckworth, Chief of the Ceramics Division. Other Institute staff members contributing to the work were C. L. Blankenship, B. G. Brand, G. E. Bundy, F. F. Fondriest, C. P. Johnson, and M. V. Weaver.

The Institute provided facilities and support conducive to the effective prosecution of the work.

# PROTECTIVE COATINGS TO PREVENT DETERIORATION OF CONCRETE BY DEICING CHEMICALS

## SUMMARY

The deterioration of concrete bridge decks and structures resulting from scaling caused by the increasing use of deicing chemicals has become an increasingly serious problem in the northern states. Although the entrainment of air in the concrete and adequate curing before exposure to severe deteriorating conditions provide a reasonable degree of resistance to deicing chemicals, for various reasons these desirable conditions are not always achieved in practice. Thus, a more positive protection is desirable. Previous studies and field trials have indicated that a number of protective coatings afford some degree of protection against deicing salt attack on concrete structures that have not been adequately protected by air entrainment. This report presents the results of a laboratory investigation to determine which of the potential protective coatings are most effective and economical and to develop new coatings that would provide maximum protection and economy.

Observations of the rate of scaling of a standard non-air-entrained concrete specimen subjected to repeated freezing and thawing while covered with a salt solution was the principal means used to evaluate the various coatings. The degree of scaling was visually rated on a 1 to 5 scale by comparison with a standard set of specimens that exhibited varying degrees of scaling.

A total of 110 coatings was evaluated in the program, including linseed oil solutions and emulsions, solutions of other vegetable oils, epoxy and modified epoxy coatings, a number of other synthetic resin coatings, rubber coatings, tars, asphalt, waxes, oils, and various inorganic coatings and treatments. Although the coatings represented an extremely diverse assortment of materials, good protection of non-air-entrained concrete was obtained only with a few. These few included coatings based on linseed oil or other vegetable oils and a sand-filled coal tar epoxy coating. The majority of the organic coatings failed rapidly by flaking or peeling from the surface of the concrete. Most of the inorganic coatings and some of the organic coatings were appreciably permeable to the salt solution and afforded protection only for a short time. Considering both economy and performance, the best results by far were obtained with vegetable oils and, particularly, linseed oil solutions.

In view of the excellent results with linseed oil solutions, attention was given to determining the optimum composition, application conditions, and application rates for linseed oil solutions. The results showed that the 50:50 mixture of boiled linseed oil and mineral spirits now used by a number of state highway departments was about the optimum composition and that the normally used application rates were satisfactory. Kerosene, which has a higher flash point than mineral spirits, could be substituted for the mineral spirits without affecting the

performance. In trials on partially-scaled concrete it was found that removal of loose scale and washing to remove absorbed salt solution was advisable.

Experimental studies of the salt-scaling process showed it to be intimately associated with the lowering of the freezing point of water by deicing chemicals. The effect is physical rather than chemical and arises from the development in the concrete of a depthwise gradient in the concentration of the deicing compound. The stresses that cause the concrete surface to scale probably are a combination of thermal and hydraulic stresses.

## CHAPTER ONE

# INTRODUCTION AND EXPERIMENTAL PROCEDURES

The deterioration of concrete bridge decks and structures resulting from scaling has become an increasingly serious problem in the northern states. Although other causes play a part in the deterioration process, it is clear that the major cause is the use of deicing salts to remove ice and snow (1, 2, 3, 4). Convincing evidence has been presented that salt scaling does not occur in concrete decks and structures that contain adequate amounts of entrained air and that have been properly cured (3, 5). Equally convincing evidence is provided by scaled bridge decks and structures that this knowledge of how to avoid salt scaling has not always been translated into practice (6, 7, 8, 9, 10). In view of the practical difficulty of insuring that a concrete bridge will indeed be immune to attack by deicing salts, a more positive protection is desirable. One approach is to apply a protective coating to the concrete. This approach has been followed in a number of previous investigations with varying degrees of success and with various findings concerning the merits of a large variety of coating materials (1, 9, 10, 11, 12, 13, 14, 15).

This report describes a laboratory investigation to determine which of the numerous coatings that have been proposed for protecting concrete against deicing salts are most effective and economical, and to develop new coatings that would provide maximum protection and economy. The research plan included (1) development of a suitable method for evaluating the protection afforded by coatings under controlled conditions, (2) the selection and evaluation of candidate coating materials, (3) attempts to develop superior coatings, and (4) field trials of any coatings indicated to be superior. To guide the evaluation and development studies, experiments were conducted in an attempt to define the mechanism by which salt solutions cause scaling of concrete.

## EXPERIMENTAL PROCEDURES

Observation of the resistance of a coated concrete slab to salt scaling is the principal means of evaluating the efficacy

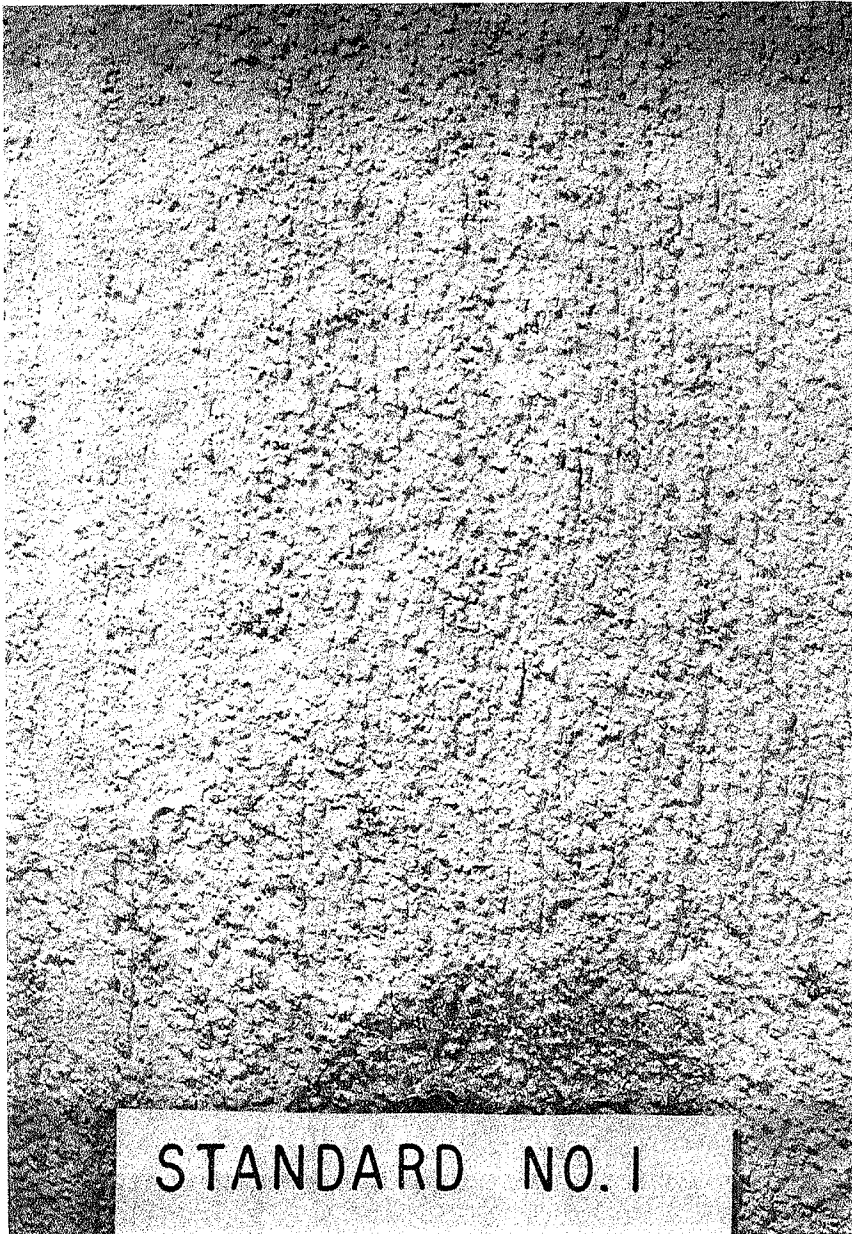
of a coating in protecting concrete structures from deicing salts. To permit evaluation of coatings in a reasonable period of time it is desirable that the exposure conditions be selected so as to promote rapid scaling of uncoated specimens without departing drastically from the conditions to which a concrete structure would be exposed in service. Likewise, the composition of the concrete and the curing conditions should be such as to give a reasonably rapid scaling rate without departing too far from normal structural concrete practice.

On the basis of information in the literature and discussions with other investigators who have conducted research on salt scaling, a set of conditions for specimen preparation and salt-scaling exposure was selected. After a period of experimentation in which the initially selected conditions were modified to provide more rapid evaluation and improved reproducibility, a standard procedure was established. Essentially the procedure involved subjecting non-air-entrained concrete slabs to repeated freezing and thawing while covered with a 3 percent sodium chloride brine. The degree of scaling was visually rated by comparison with a standard set of slabs exhibiting varying degrees of scaling. The effectiveness of a protective coating was evaluated in comparison with the scaling rate for an uncoated concrete slab and with the scaling rate for a slab coated with a 50-50 mixture of linseed oil and kerosene.

### Concrete Composition

The concrete used in preparing the specimens for evaluation corresponded to Ohio State Highway Department designation Class C, and was a 6.5-sack, 0.5-w/c mix. The coarse aggregate was a  $\frac{3}{4}$ -in. maximum size glacial gravel and the fine aggregate was a local concrete sand. Both were from sources consistently producing aggregates meeting specifications of the Ohio State Highway Department. The standard batch used contained 22.9 lb Type I portland cement, 42.4 lb fine aggregate, 75 lb coarse

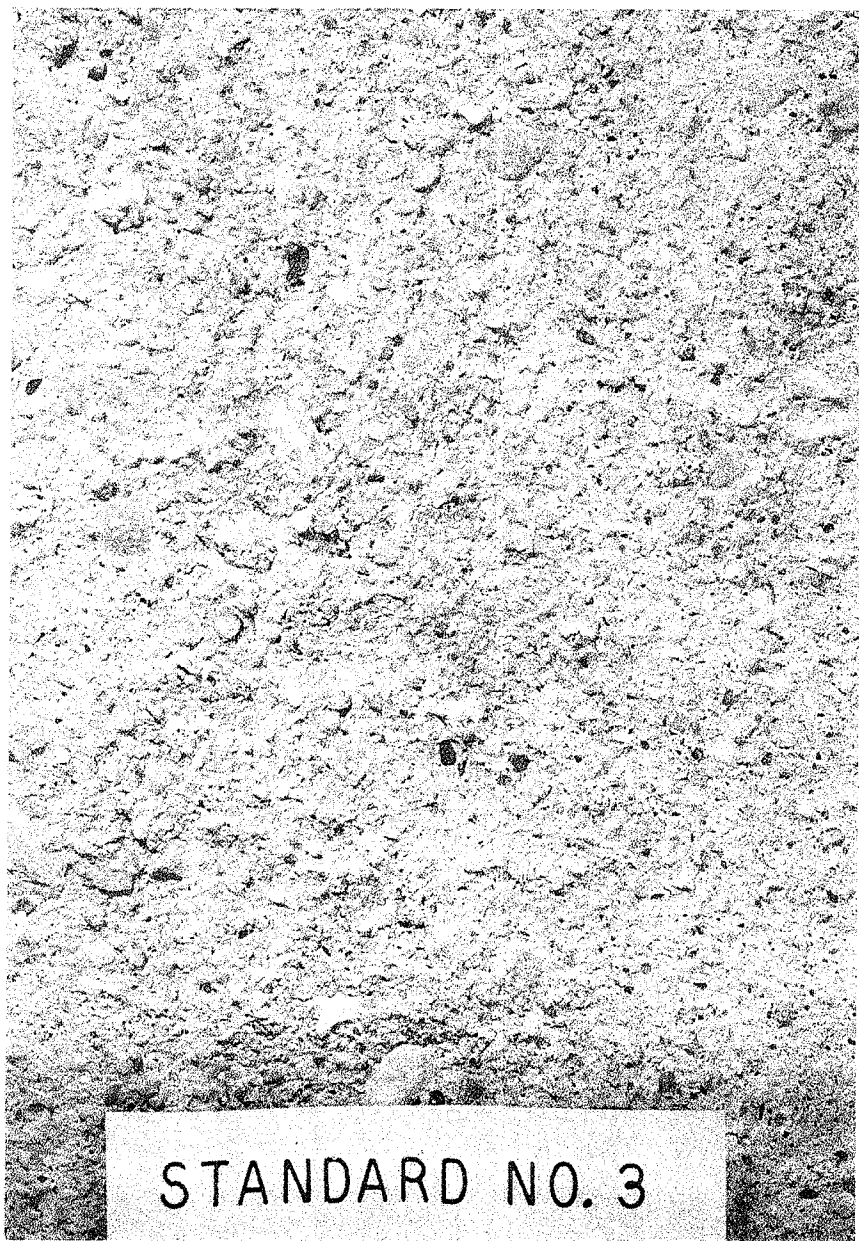




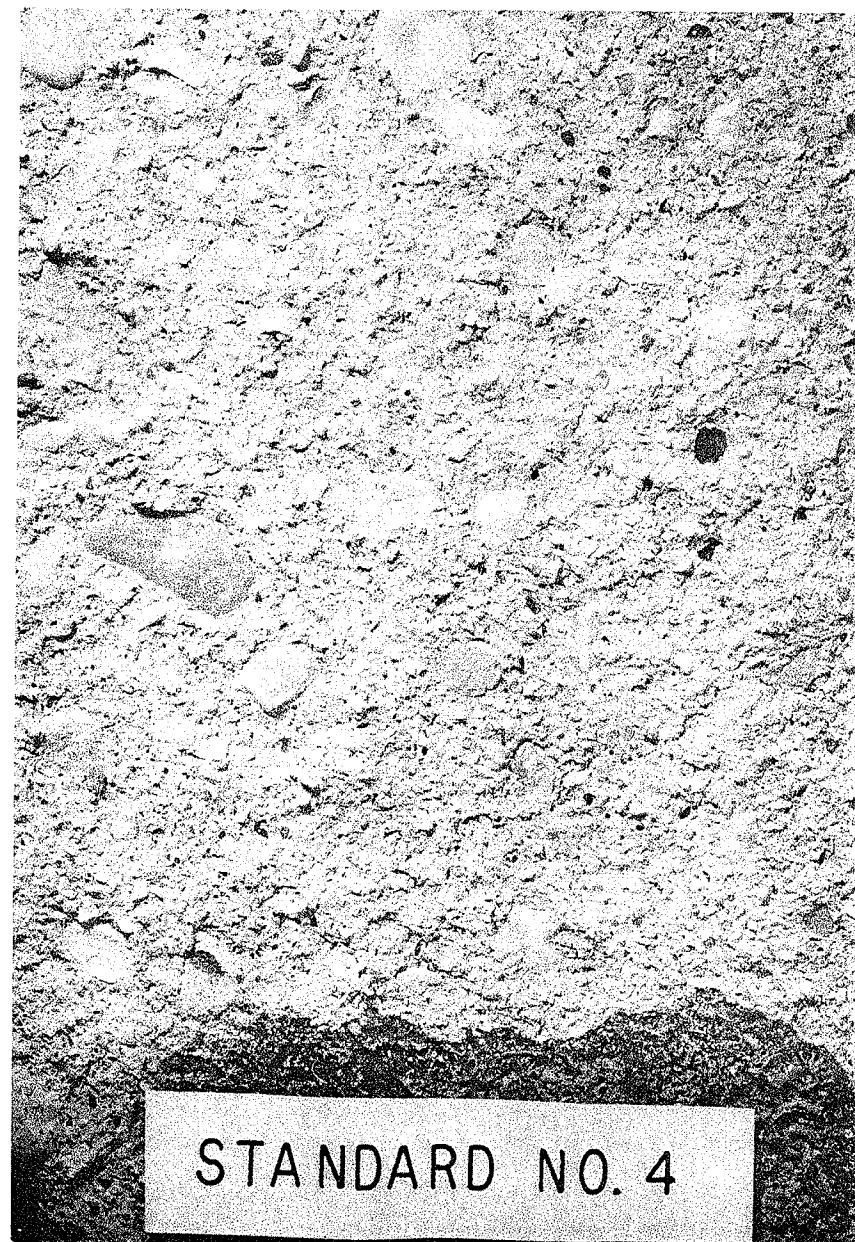
*Figure 1. Standard comparison slab No. 1, no scaling.*



*Figure 2. Standard comparison slab No. 2, slight scaling.*



*Figure 3. Standard comparison slab No. 3, moderate scaling.*



*Figure 4. Standard comparison slab No. 4, severe scaling.*



Figure 5. Standard comparison slab No. 5, very severe scaling.

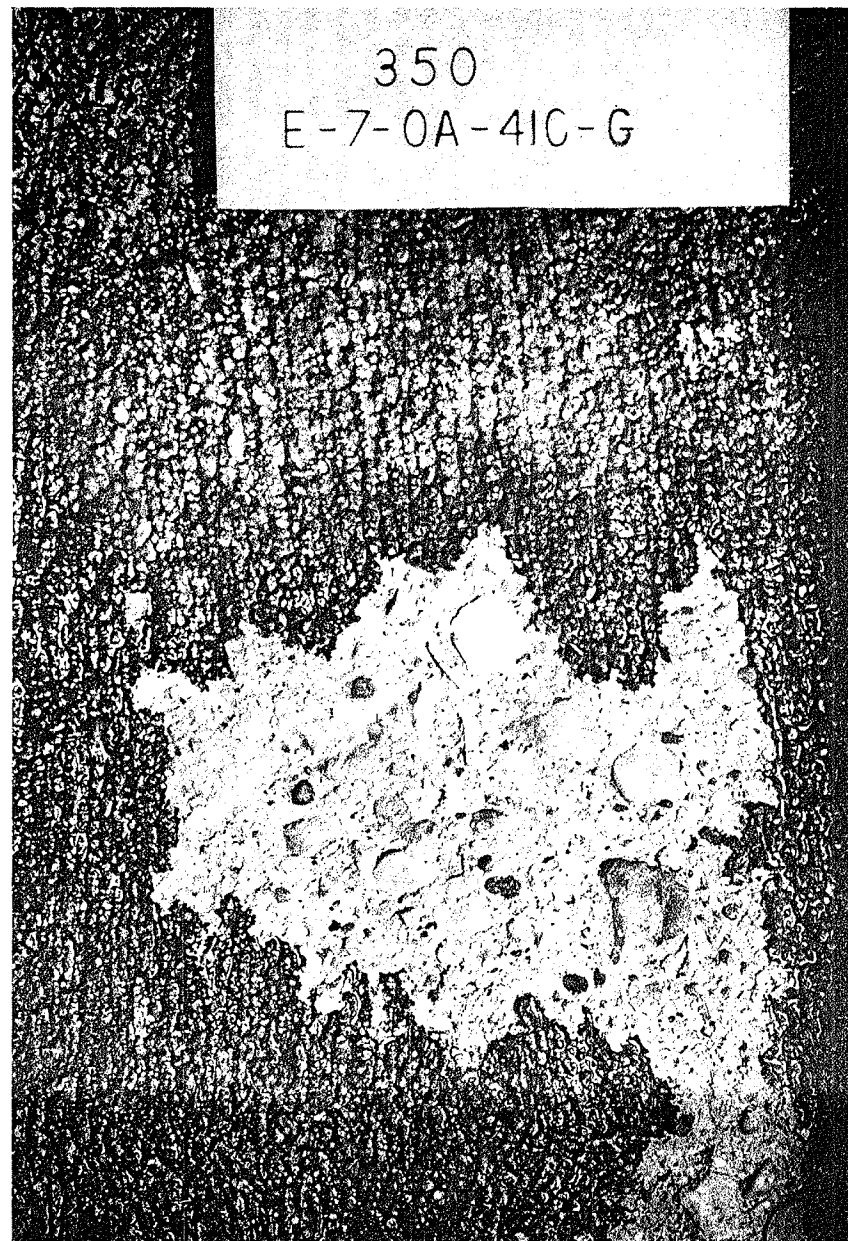


Figure 6. Partially-scaled coated slab.

aggregate, 11.45 lb water, and 3 oz retarder.\* The batch was mixed in a 2-cu ft capacity, rotating drum, concrete mixer.

#### *Specimen Preparation and Curing*

Slab specimens, approximately  $7\frac{1}{2} \times 11\frac{1}{2} \times 1\frac{1}{2}$  in., were cast in nominally 8 x 12 x 4-in. polyethylene trays on a vibrating table. The top was given a broomed finish after the initial set. The slabs were cured under damp cloths in the polyethylene trays overnight, then were stripped from the trays and placed in a fog room at 70-75 F and 100 percent humidity. Fourteen days after casting the slabs were removed from the fog room and allowed to dry in the laboratory air at about 80 F and 40 percent humidity for about 18 hr. Weight loss measurements indicated that under these drying conditions the moisture loss was about 1 percent, leaving a residual free moisture content of about 5 percent. Coatings were then applied, following suppliers' instructions where applicable, and normally were allowed to cure for about 60 hr at room temperature before exposing the slabs to salt scaling. Uncoated control slabs were allowed to stand in the laboratory for the same period before starting the salt-scaling evaluation.

The edges, sides, and bottom surfaces of the slabs were routinely protected from the salt solution by two coatings of linseed oil solution (50-50 boiled linseed oil and kerosene) applied during the last 6 to 8 hr of drying prior to applying the test coatings to the top of the slab. For some of the long-lasting coatings (notably linseed oil solutions or a sand-filled coal tar epoxy coating) attack at the sides and edges of the slab after about 150 cycles obscured the performance of the coating. To obtain a more positive evaluation in these cases, the experiments

\* Plastiment, supplied by Sika Chemical Co.

were repeated using slabs in which the sides and bottom surfaces were protected by an impermeable polyethylene film cemented with an asphaltic cement.

#### *Salt-Scaling Procedure*

The coated slabs and three uncoated control slabs were replaced in the polyethylene trays and covered with a 3 percent sodium chloride solution to a depth of about  $\frac{1}{2}$  in. above the top of the slab. The brine-covered slabs, supported on steel racks, were placed in an automatic freeze-thaw chamber and alternately frozen and thawed in air. The temperature was controlled between 0 and 40 F by means of a copper-constantan thermocouple in a glass protection tube immersed in the brine solution over one of the slabs near the middle of the freezing chamber. The total time for one cycle was 66 hr, including 3 hr of freezing and 3 hr of thawing. Once a week the racks were hoisted from the chamber, the brine solutions were poured off the samples, the samples were washed in tap water, and then examined for extent of scaling. Fresh brine solution was placed over the slabs and they were returned to the freeze-thaw chamber for further cycling.

#### *Evaluation*

The degree of salt scaling was visually rated on a scale from 1 to 5 in comparison with a set of standard slabs. The appearance of the standard scaled slabs is shown in Figures 1 through 5. In cases where the scaling was spotty, as was often the case with coated slabs, the rating of scaling was made in terms of both severity and percentage of area scaled. For example, the unevenly-scaled coated slab shown in Figure 6 would be given a rating of 75 percent No. 1, 25 percent No. 4.

## CHAPTER TWO

# RESULTS OF COATING EVALUATIONS

A total of 110 coatings was applied to non-air-entrained concrete slabs and the resistance of the coated slabs to the standard salt-scaling exposure was determined. The coatings and a general appraisal of their performance are given in Table 1.

Although the coatings represented an extremely diverse assortment of materials, good protection of non-air-entrained concrete was obtained only with a few. These few included coatings based on linseed oil or other vegetable oils and a sand-filled epoxy coating. The majority of the other organic coatings failed rapidly by flaking or peeling from the concrete surface, allowing the salt solution to penetrate into and subsequently cause scaling of the concrete. The majority of the inorganic coatings and some of the organic coatings were sufficiently permeable to the

salt solution that they afforded protection only for a short time.

The performance of the various coatings is discussed in greater detail in the following sections.

#### LINSEED OIL COATINGS

Solutions or emulsions of linseed oil have been reported in many of the previous studies to provide good protection against salt scaling. A number of state highway departments specify the application of linseed oil solutions or emulsions on bridge decks and structures. The excellent performance of linseed-oil-based coatings was confirmed by the results of the present study. Solutions of linseed oil were found to be superior to water-base emulsions; it is



TABLE 1

## COATINGS EVALUATED

COATING NUMBER	GENERAL DESCRIPTION	PERFORMANCE	CYCLES TO PRODUCE MODERATE SCALING	COATING NUMBER	GENERAL DESCRIPTION	PERFORMANCE	CYCLES TO PRODUCE MODERATE SCALING
1	Linseed oil solution	Excellent	>200	57	Acrylic emulsion	Poor	<27
3	Linseed oil emulsion	Poor	27-54	58	Hydrocarbon	Poor	27
4	Coal tar product	Poor	27-54	59	Linseed oil solution	Good	108
5	Epoxy	Poor	54-81	60	Linseed oil solution	Good	81
6	Coal tar epoxy	Fair	~81	61	Coating No. 1 +5% petroleum resin	Good	Edge erosion
6A	Sand-filled coal tar epoxy	Excellent	>200	62	Modified linseed oil emulsion	Poor	54
7	Linseed oil emulsion	Poor	54	63	Modified linseed oil emulsion	Poor	54
8	Polyvinyl butyral	Poor	27-54	64	Modified linseed oil emulsion	Poor	54
9	Linseed oil emulsion	Poor	27-54	65	Modified linseed oil emulsion	Poor	54
10	Linseed oil solution	Very good	~200	66	Modified linseed oil emulsion	Fair	54-81
11	"Water soluble" linseed oil	Good	108-135	67	Coating No. 1 +10% acid	Poor	54
12	Emulsifiable linseed oil	Good	81	68	Coating No. 1 +10% wetting agent	Fair	54-81
13	Coal tar epoxy	Poor	<27	69	Portland cement paint	Poor	<27
14	Hydrocarbon resin	Poor	<27	70	No. 69 +5% bentonite	Poor	<27
15	Modified castor oil	Excellent	>200	71	Sodium silicate	Poor	54
16	Rubber solution	Poor	27	72	Low-alkali silicate	Poor	<27
17	Hydrocarbon resin emulsion	Poor	<27	73	Coating No. 1 +10% petroleum resin	Fair	54-81
19	Polyvinylidene chloride dispersion	Poor	<27	74	Flame-sprayed glass	Poor	<27
20	Polyvinylidene chloride dispersion	Poor	27	75	Low-alkali silicate	Poor	<27
21	Polyvinylidene chloride dispersion	Poor	54	76	Sodium silicofluoride solution	Poor	27
22	Acrylic resin emulsion	Poor	54	77	Autoclaved slab, no coating	Poor	<27
23	Acrylic resin emulsion	Poor	54	78	No. 20 motor oil	Poor	54
24	Coating No. 10 diluted	Good	81	79	Lead phosphate	Poor	<27
25	Coating No. 15 diluted	Poor	54	80	Lead acetate	Poor	<27
26	Oleic acid solution	Poor	27	81	Lead nitrate	Poor	<27
27	Silicone resin solution	Poor	<27	84	Wax	Poor	<27
28	Rubber solution	Poor	<27	86	Fluorinated hydrocarbon spray	Poor	<27
29	Polyurethane resin	Poor	<27	90	Silicone grease	Poor	27
30	Kerosene	Poor	<27	91	Creosote	Poor	27-54
31	Linseed oil	Good	81	92	Mixture of rubber latex and sodium silicate	Poor	27
32	Polyurethane resin	Poor	54	93	Coating No. 92 +chromium oxide	Poor	27
34	Polyethylene-polyurethane copolymer	Poor	54	95	Mixture of No. 1 and creosote	Good	135
35	Modified epoxy	Fair	54-81	96	Mixture of No. 1 and silicone resin	Poor	54
36	Solution of No. 35 in toluene	Fair	54-81	97	Cottonseed oil	Excellent	189
37	Modified epoxy	Poor	<27	98	Soybean oil	Excellent	189
38	Solution of No. 37 in toluene	Poor	<27	99	SS-1 asphalt	Poor	27
39	Polysulfide-modified epoxy	Fair	54-81	100	RC-1 asphalt	Poor	27
40	Polysulfide-modified epoxy	Fair	81	101	Hydrocarbon resin	Poor	27
41	Air-entrained cement mortar	Poor	<27	102	Hydrocarbon resin	Poor	27
42	Acrylic emulsion	Poor	27	103	Hydrocarbon resin	Poor	<27
43	Polymeric resin	Poor	27	105	Rubber solution	Poor	54
44	Acrylic emulsion	Poor	27	106	Epoxy enamel	Poor	54
45	Epoxy	Poor	<27	107	Rubber latex	Poor	54
46	Epoxy	Poor	<27	109	Linseed oil solution	Excellent	189
47	Epoxy	Poor	<27	111	Linseed oil solution	Excellent	189
48	Polyethylene-modified linseed oil	Poor	<27	112	Linseed oil solution	Very good	162
49	Linseed oil solution	Good	81	113	Linseed oil solution	Very good	162
50	Linseed oil solution	Good	Edge erosion	114	Linseed oil solution	Good	81
51	Linseed oil solution	Good	Edge erosion	117	Polysulfide-modified epoxy penetrating sealer	Good	108
52	Linseed oil solution	Good	81	118	Polysulfide-modified epoxy penetrating sealer	Good	81-108
53	Linseed oil solution	Good	81	119	Linseed oil solution	Very good	162
54	Penetrating epoxy coating	Poor	54	120	Coating No. 1 +25% petroleum resin	Very good	162
55	Penetrating epoxy coating	Poor	<27	121	Cottonseed oil solution	Good	81
56	Epoxy	Poor	<27	122	Soybean oil solution	Good	108

believed that the relatively poorer performance of emulsions resulted from re-dispersion of the linseed oil in the salt solution and consequent displacement of the linseed oil from the concrete.

### Solutions

Early experiments in this program showed that a 50-50 mixture of boiled linseed oil and kerosene resulted in excellent protection of non-air-entrained concrete. This coating was subsequently used as a standard for comparison. As the study progressed and it became apparent that few, if any, other coatings approached the performance of the linseed oil solution, effort was devoted to determining the optimum composition, application condi-

tions, and application rate for linseed oil solutions. Variables studied included the kind and source of the linseed oil, the kind of solvent, the concentration of the linseed oil, the coating weight, the addition of a third component to the coating solution, the moisture content of the concrete at the time of coating, the temperature of the concrete at the time of coating, and the exposure of the concrete to salt solution prior to applying the coating. For these relatively long-lasting coatings, in most cases it was difficult to reach a definite conclusion about the effects of minor compositional or treatment changes. The effects of such changes were obscured by erosion of the sides and bottoms of the slabs. The results of these investigations are summarized in Tables 2 and 3.

TABLE 2  
PERFORMANCE OF LINSEED OIL SOLUTION COATINGS

COATING NUMBER	COATING COMPOSITION							WEIGHT APPLIED (GM) <sup>a</sup>
	OIL			SOLVENT		ADDITIONS		
	KIND	SOURCE	PARTS (%)	KIND	PARTS (%)	KIND	AMOUNT (%)	
None	—	—	—	—	—	—	—	—
(a) KIND AND SOURCE OF OIL AND KIND OF SOLVENT VARIED								
1	Boiled linseed	A	50	Kerosene	50	—	—	8
49	Boiled linseed	A	50	Min. spirits	50	—	—	8
52	Boiled linseed	B	50	Kerosene	50	—	—	8
53	Boiled linseed	B	50	Min. spirits	50	—	—	8
59	Boiled linseed	C	50	Kerosene	50	—	—	8
60	Boiled linseed	C	50	Min. spirits	50	—	—	8
109	Raw linseed	A	50	Kerosene	50	—	—	8
111	Raw linseed	B	50	Kerosene	50	—	—	8
(b) CONCENTRATION OF OIL AND COATING WEIGHT VARIED								
31	Boiled linseed	A	100	—	—	—	—	5
114	Boiled linseed	A	67.5	Kerosene	32.5	—	—	6
119	Boiled linseed	A	67.5	Kerosene	32.5	—	—	8
50	Boiled linseed	A	60	Kerosene	40	—	—	4
								8
								10
51	Boiled linseed	A	40	Kerosene	60	—	—	4
								8
								10
113	Boiled linseed	A	37.5	Kerosene	62.5	—	—	12
112	Boiled linseed	A	25	Kerosene	75	—	—	16
24	Boiled linseed	A	25	Kerosene	75	—	—	5
30	—	—	—	Kerosene	100	—	—	5
(c) ADDITIONS TO LINSEED OIL SOLUTIONS								
61	Boiled linseed	A	47.5	Kerosene	47.5	Petrol. resin	5	8
73	Boiled linseed	A	45	Kerosene	45	Petrol. resin	10	8
120	Boiled linseed	A	25	Kerosene	50	Petrol. resin	25	8
67	Boiled linseed	A	45	Kerosene	45	Oleic acid	10	8
68	Boiled linseed	A	45	Kerosene	45	Wetting agent	10	8
95	Boiled linseed	A	25	Kerosene	25	Creosote	50	8
96	Boiled linseed	A	25	Kerosene	25	Silicone resin solution	50	8

<sup>a</sup> Grams per slab.

<sup>b</sup> Isolated spots.

<sup>c</sup> Severe erosion of edges.

<sup>d</sup> Severe erosion.

<sup>e</sup> Spots of 5.

<sup>f</sup> Spots of 4.

## KIND AND SOURCE OF OIL

The standard No. 1 coating, a mixture of 50 percent by volume of boiled linseed oil from Supplier A and 50 percent by volume of kerosene, when applied to the standard non-air-entrained concrete slab in two coats for a total coating weight of 8 gm per slab (corresponding to a coverage of 40 sq yd/gal for the first coat and about 67 sq yd/gal for the second coat), delayed the onset of scaling for between 54 and 81 cycles and retarded scaling to the extent that 216 cycles were required to reach the stage of severe scaling. Table 2 gives the results of experiments in which the source of the linseed oil and the kind of solvent was varied and in which raw linseed oil was compared with boiled linseed oil while the concentration

of the oil and the coating weight were held constant. Severe erosion of the sides of some of the slabs, because of inadequate coating of the sides, may have caused premature scaling of the surface in those slabs and so it is not possible to reach a firm conclusion about the relative effectiveness of linseed oils from different sources. The results suggest, however, that boiled linseed oil is more effective than raw linseed oil and that boiled linseed oils from different sources may vary. Minor differences in the volatility of the solvent seemed to have no significant effect on the performance of the coating.

## CONCENTRATION OF OIL AND COATING WEIGHT

Table 2 also gives the results of experiments in which the concentration of boiled linseed oil and the weight of coating applied were varied while all other factors were held constant. These results clearly indicate that the extreme compositions—all linseed oil and all kerosene—were definitely inferior to mixtures of linseed oil and kerosene. The results for mixtures close to 50 percent of linseed oil were obscured by edge erosion, but the data indicate that within the range of 37.5 to 67.5 percent of linseed oil the concentration is not critical. Likewise, within this concentration range, variations in coating weight were not particularly critical. Observations of the degree of coverage and of the penetration of the coating into the concrete as coating weight and concentration were varied indicated that as the concentration of oil was decreased below 50 percent it was necessary to apply a greater volume of solution to achieve adequate protection; as the concentration was increased beyond 50 percent, the rate of penetration decreased and it was more difficult to achieve uniform coating. The 50-50 mixture appeared to have about the optimum spreading and penetrating characteristics to achieve good coverage and the proper rate of penetration for good protection. These observations suggest that in field applications it might be desirable to adjust the concentration of the linseed oil solution to fit the absorption characteristics of the specific concrete being treated.

## ADDITIONS TO LINSEED OIL SOLUTIONS

A few experiments were conducted to determine whether the excellent performance of linseed oil solutions could be improved by the addition of resins that might produce a tougher film, or wetting agents that might promote better penetration into the concrete, or materials that might produce a more waterproof film. The results are given in Table 2.

Partial replacement of the linseed oil with a petroleum resin showed promise. Edge erosion obscured the effects of small replacements to some extent, but replacement of 25 percent of linseed oil by the petroleum resin resulted in nearly as good performance as the unmodified linseed oil solution. The lower cost of the petroleum resin compared to linseed oil makes this an attractive possibility for further investigation. Additions of oleic acid, of an oil-soluble wetting agent, or of a silicone resin solution were clearly detrimental. Addition of creosote to the linseed oil solution impaired the effectiveness of the linseed oil coating.

## DEGREE OF SCALING AFTER INDICATED NO. OF CYCLES

24	54	81	108	135	162	189	216	243
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4-5								
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1	1	1½	3 <sup>b</sup>	3 <sup>b</sup>	3 <sup>b</sup>	2	3	—
1	1½	1½	2	2	2-4	2-5	3	—
1	1	3-5 <sup>c</sup>						
1	2	3-5 <sup>c</sup>						
1	1½	2-5 <sup>c</sup>						
1	1	2-5 <sup>c</sup>						
1	1	1½	2	2	<sup>a</sup>	<sup>a</sup>	4-5	
1	1	1½	<sup>c</sup>	<sup>c</sup>	<sup>a</sup>	<sup>a</sup>	3-5	

1	2	2-5						
1	1½	2-4	4					
1	1½	1½	2	2	2-3			
1	1½	1½	<sup>c</sup>					
1	1	1½	<sup>c</sup>					
1	1½	1½	<sup>c</sup>					
1	1½	1½	<sup>c</sup>					
1	1½	1½	<sup>c</sup>					
1	1	1	1	2	3-4	4		
1	1	1½	1½	2-3	3-4	3-4	4	
1½	2 <sup>c</sup>							
4	5+							

1	1½	<sup>c</sup>				
1	2	5	<sup>c</sup>			
1	1½	1¾	2	2½	2-3	
1½	3	<sup>c</sup>				
1½	2	5	<sup>c</sup>			
1½	2	2½	2½	3-4		
1½	3					

TABLE 3  
EFFECT OF PRETREATMENT OF CONCRETE ON EFFECTIVENESS OF LINSEED OIL SOLUTION <sup>a</sup>

PRETREATMENT OR CONDITIONING INVESTIGATED	CONDITION, TIME OF COATING		DEGREE OF SCALING AFTER INDICATED NO. OF CYCLES						
	MOISTURE (%) <sup>b</sup>	TEMP. (°F)	27	54	81	108	135	162	189
None, standard conditions	80	75	1	1	1½	1½ <sup>c</sup>	1½ <sup>c</sup>	1½ <sup>c</sup>	2
Moisture content:									
No drying	100	75	1½	1½-3	4 <sup>d</sup>	4 <sup>e</sup>			
Short drying	88	75	1	1½	1½-2	1½-2	†		
Normal drying	80	75	1	1-1½	1-1½	1-1½	†		
Longer drying	72	75	1	2	2-2½	2½-3			
	66	75	1	—	2½-3	2½-3			
	58	75	1	1½	2½	2½-3			
Oven dried	0	75	1	1-1½	1-1½	1½	†		
Application temperature:									
Low temperature	80-90	40	1½	1½	1½-3	3-4	3-5		
High temperature	50-70	120	1½	1½-5	2-5	5			
Partial scale, no cleaning:									
Normal drying	80	75	1½-2	2½-3	2½-3½				
Oven dried	0	75	1½-2	2½	5				
Dried, sandblasted	<50	75	2-3	3-5	5				
Partial scale, washed:									
Normal drying	80	75	1+	1½-2	2				
Brine soaked, no cleaning:									
No drying	100	75	1	1½-3	4				
Dried	0	75	1½	1½-2	2½				
Brine soaked, washed:									
Normal drying	80	75	1	1	1½	†			

<sup>a</sup> Coating No. 1 used.

<sup>b</sup> Percent of total free moisture.

<sup>c</sup> Isolated spots of 3.

<sup>d</sup> 25 percent.

<sup>e</sup> 75 percent.

† Discontinued.

#### PRETREATMENT OF CONCRETE

Experiments to determine the effect of variations in the moisture content of the concrete and the temperature at the time of applying the standard linseed oil solution are summarized in Table 3. The protection against further scaling afforded by applying the linseed oil solution to a partially-scaled slab also is shown by data in Table 3.

The moisture content of the slab at the time the coating was applied had a significant effect on the scaling rate of the coated slab. The coating provided only moderate protection when applied to wet concrete. As the moisture content of the concrete was decreased, the protection improved, reaching a maximum value at a moisture content of about 80 percent of the saturated moisture content. Further drying of the concrete prior to application of the linseed oil resulted in more rapid scaling. It is believed that the linseed oil solution penetrated to a greater depth in the drier concrete, with the result that the top surface was inadequately sealed by the linseed oil film. Thus, the critical moisture content for scaling was reached in a relatively short time. When the free moisture content of the slab was reduced to essentially zero by oven-drying the slab prior to applying the linseed oil coating, the coated slab had excellent scaling resistance. In this case, it is believed that the very dry concrete did not approach the critical saturation value until after a large number of freeze-thaw cycles.

The limited study of the effect of the temperature at time of application showed that better protection was obtained by applying the coating at temperatures of about

75 F than at either low or high temperatures. Application at 120 F appeared to be more detrimental than application at 40 F. The apparent temperature effect may have been accentuated by differences in moisture content, as the slabs coated at 120 F may have dried below the optimum moisture content while being heated to the application temperature.

Experiments in which the linseed oil coating was applied to a partially-scaled slab showed that the coating retarded further scaling but that the subsequent scaling rate was much more rapid than for concrete which had been coated before exposure to salt solution. This result suggests that salt solution trapped within a concrete slab continues to cause scaling even when further ingress of salt solution is prevented.

To determine whether the situation might be remedied by cleaning the partially-scaled slab before applying the linseed oil solution, experiments were conducted in which partially-scaled slabs or brine-soaked but unscaled slabs were washed, or dried, or sandblasted to remove potentially damaged concrete at the surface, before applying the linseed oil solution. Salt-scaling exposures of the coated slabs showed that, of these procedures, only washing was effective. Drying caused cracking of the slabs and accentuated the scaling damage. Removal of the loose or weakened surface by sandblasting was not helpful.

Thus, these results show that if it is desired to protect concrete structures from deicing salt damage by application of linseed oil solutions, the solution should be applied before the concrete is exposed to deicing salts. If it is not



TABLE 4  
ABSORPTION OF COATED CONCRETE <sup>a</sup>

COATING NUMBER	TYPE OF COATING	LIQUID	AMOUNT OF LIQUID ABSORBED <sup>b</sup> (%) AFTER IMMERSION FOR INDICATED NO. OF DAYS				
			1	4	8	12	50
—	None	Water	9.5	10.3	10.3	10.5	10.8
		Salt sol. <sup>c</sup>	10.7	10.9	11.2	11.4	11.4
1	Linseed oil	Water	6.3	8.3	8.7	9.4	9.7
		Salt sol. <sup>c</sup>	3.0	4.4	5.8	6.5	9.2
27	Silicone resin	Water	0.5	1.8	2.1 <sup>d</sup>	10.3 <sup>d</sup>	10.7
		Salt sol. <sup>c</sup>	0.8	4.4	10.3	10.7	11.5

<sup>a</sup> High-porosity concrete used to facilitate absorption measurements.

<sup>b</sup> Specimens completely immersed in water or salt solution.

<sup>c</sup> Concentration 3 percent.

<sup>d</sup> A similar temporary delay in water absorption with silicones was observed by Klieger and Perenchio (13) for low-porosity concrete.

feasible to apply the coating until after the concrete has been exposed to deicing salts and is beginning to scale, the surface of the concrete should be scrubbed and flushed with clean water to remove as much of the absorbed salt as possible. The washed concrete then should be allowed to dry for about one day at temperatures of 60 to 90 F and relative humidities of 20 to 60 percent before the linseed oil solution is applied.

#### WATER PERMEABILITY OF LINSEED OIL COATINGS

In an attempt to determine why linseed oil is so effective in retarding salt scaling, the effectiveness of linseed oil in preventing absorption of water by concrete was investigated (Table 4). Surprisingly, it was found that the application of linseed oil solution (No. 1 coating) to concrete caused only a moderate reduction in the rate of water absorption when the coating was dried as usual for salt-scaling evaluations. On the other hand, a commercial silicone resin that was relatively ineffective in retarding

salt scaling substantially reduced the water absorption of concrete.

In view of the indication from other experiments (described later) that the critical factor in salt scaling is the development of a gradient in salt concentration, the effect of the linseed oil coating on the absorption of salt water was investigated. Measurements revealed that linseed oil (No. 1 coating) reduced the rate at which salt water is absorbed by concrete. Thus, although the linseed oil coating was relatively ineffective as a barrier against pure water, it was resistant to penetration by salt.

#### Emulsions

Application of linseed oil to concrete as an aqueous emulsion would be preferable to application from solution in volatile organic solvents because of the fire hazard associated with the low flash-point solvents used. A number of commercially available linseed oil emulsions or emulsions prepared from commercial emulsifiable linseed oils and

TABLE 5  
PERFORMANCE OF LINSEED OIL EMULSION COATINGS

COATING NUMBER	DESCRIPTION OF EMULSION	DEGREE OF SCALING AFTER INDICATED NUMBER OF CYCLES							
		27	54	81	108	135	162	189	216
None	None	4-5							
1	Linseed oil solution <sup>a</sup>	1	1	1½	1½ <sup>b</sup>	1½ <sup>b</sup>	1½ <sup>b</sup>	2	3
3	Commercial linseed oil emulsion	1	5						
7	Commercial linseed oil emulsion	1	4						
9	Commercial linseed oil emulsion	1-3	5						
11	"Water soluble" linseed oil <sup>c</sup>	1	1	1	1 <sup>a</sup>	1 <sup>a</sup>	1 <sup>a</sup>	4-5	
12	Emulsifiable linseed oil <sup>c</sup>	1	1½	2-3	5				
48	Polyethylene-mod. linseed oil emulsion	4							
62	Exper. linseed oil emulsion	1½	3						
63	Exper. linseed oil emulsion	1½	3						
64	Exper. linseed oil emulsion	1	3						
65	Exper. linseed oil emulsion	1	3						
66	Exper. linseed oil emulsion	1	1½	5					

<sup>a</sup> Standard for comparison.

<sup>b</sup> Isolated spots of 3.

<sup>c</sup> Diluted with equal weight of water.

<sup>d</sup> Isolated spots of 4.

TABLE 6  
PERFORMANCE OF VEGETABLE OIL COATINGS

COATING NUMBER	COATING COMPOSITION				DEGREE OF SCALING AFTER INDICATED NUMBER OF CYCLES							
	OIL		SOLVENT									
	KIND	PARTS	KIND	PARTS	27	54	81	108	135	162	189	216
None	—	—	—	—	4-5							
1	Linseed	50	Kerosene	50	1	1	1½	1½ <sup>a</sup>	1½ <sup>a</sup>	1½ <sup>a</sup>	2	3
31	Linseed	100	None	—	1	2	2-5					
121	Cottonseed	50	Kerosene	50	2	2	2-3	3-5	3-5	5		
98	Cottonseed	100	None	—	1	1	1¾	1¾	1¾	2	2-4	2-5
122	Soybean	50	Kerosene	50	1½	2	2½	3-5	3-5	3-5		
97	Soybean	100	None	—	1	1	1	1½	1¾	1¾	2-4	2-5
15	Mod. castor	100	None	—	1	1	1	1½	1½	1½	2	2
25	Mod. castor	50	Kerosene	50	1	2-3	3					

<sup>a</sup> Isolated spots of 3.

several experimental linseed oil emulsions \* were applied to the standard concrete slabs at coating rates recommended by the suppliers of the materials. The salt-scaling resistance of the coated concretes is compared with that of concrete coated with a linseed oil solution in Table 5.

None of the emulsions was as effective as the linseed oil solution. One of the emulsions, prepared from a "water soluble" linseed oil, provided excellent protection for 81 cycles, but slabs coated with it began to scale deeply in isolated spots thereafter and the scaling had become widespread by 189 cycles; concrete coated with the linseed oil solution on the other hand showed only moderate scaling after 189 cycles.

The linseed oil coatings deposited from the emulsions in the pores and on the surface of the concrete apparently were easily reemulsified, as they were observed to be displaced and to float free on the salt solution. If allowed to dry for a longer period before being exposed to the salt solution, or if formulated so as to be less stable, the linseed oil emulsions might have exhibited better performance.

#### OTHER VEGETABLE OIL COATINGS

The excellent performance of linseed oil coatings suggested that other vegetable oils might be effective in protecting concrete from deicing salts. The performances of coatings based on cottonseed oil, soybean oil, and a modified castor oil, are given in Table 6. When applied undiluted, cottonseed oil, soybean oil, and the modified castor oil all provided excellent protection against salt scaling and compared favorably with the linseed oil solution. However, all were appreciably less effective when diluted with kerosene and thus did not provide a more economical protection than the linseed oil solution.

#### EPOXY AND MODIFIED EPOXY COATINGS

In view of the reported excellent performance of epoxy and modified epoxy resins as protective coatings on con-

crete in laboratory and field trials, several proprietary epoxy coatings were evaluated. The coatings included epoxy materials specifically formulated for highway and bridge deck protection. The modified epoxies included coal-tar-modified epoxies, polysulfide-modified epoxies, epoxy enamels, epoxy compositions formulated to penetrate into concrete, and others which were identified only as modified epoxies. In addition, a sand-filled epoxy coating was tried that was formed in place by first applying a layer of the epoxy and then broadcasting sand onto the tacky epoxy coating and after the epoxy had hardened, brushing off the excess sand. The results of salt-scaling evaluations of the various epoxy coatings are given in Table 7.

With the exception of the sand-filled epoxy coating, all of the epoxy and modified-epoxy coatings provided only relatively short-term protection. Invariably, the coatings failed by pinholing, flaking, or peeling so as to expose the concrete to the salt solution. Scaling started at these places and then proceeded underneath the intact coating, loosening it and progressively exposing more and more of the concrete. The typical spotty failure of the epoxy coatings is shown in Figure 6. It is believed that the primary cause of failure was the stresses set up by the difference in thermal expansion of the coating and the concrete coupled with the brittleness of the epoxy coatings at low temperature.

The excellent performance of the sand-filled epoxy coating lends weight to that hypothesis. In the first trials of the sand-filled epoxy coating no signs of scaling were observed in the central areas of the slab up to 135 cycles other than a moderate loss of sand. However, extensive scaling and undercutting had occurred at the edges of the slab. Observations of cut sections of the coated slab showed that the scaling had proceeded inward from the edges nearly to the center. In later trials where the edges of the slab were sealed by cementing a polyethylene film over the bottom and sides of the slab, no deterioration of the sand-filled epoxy coating was observed up to 189 cycles. However, observations of cut sections at 216 cycles showed that scaling was taking place underneath the visually intact sand-epoxy coating. Thus, unless the edges of

\* The experimental emulsions were supplied through the courtesy of the Northern Regional Research Laboratory, U. S. Department of Agriculture.

the concrete are sealed, the sand-filled epoxy coating will fail in a relatively short time by scaling that starts at the edges and moves underneath the coating. If the edges are adequately sealed, the attack is delayed further. The evidence from the laboratory tests, which do not take account of any acceleration in the deterioration that might result from traffic stresses, indicates that the sand-filled epoxy coating does not provide substantially longer protection than the much less costly linseed oil coating.

#### OTHER SYNTHETIC RESIN COATINGS

Many other varieties of synthetic resins have been suggested as protective coatings for concrete. The results of salt-scaling evaluations of coatings representative of several

types of resins, including polyurethanes, acrylics, polyvinyl butyral, polyvinylidene chloride, hydrocarbon resins, and a silicone resin, are given in Table 7.

None of the synthetic resin coatings protected the concrete for more than 81 cycles. Most of the coatings peeled or flaked badly within the first 27 to 54 cycles.

#### RUBBER-BASE COATINGS

Inasmuch as the synthetic resin coatings failed by flaking or peeling, presumably because of differential expansion stresses, a more extensible coating such as a rubber-base coating would appear to offer more promise. A few rubber-base coatings, three solutions and one latex, were tried (Table 7) but the results were uniformly discouraging.

TABLE 7  
PERFORMANCE OF COATINGS BASED ON SYNTHETIC POLYMERS OR ELASTOMERS

COATING NUMBER	GENERIC DESCRIPTION	DEGREE OF SCALING AFTER INDICATED NUMBER OF CYCLES							
		27	54	81	108	135	162	189	216
5	Epoxy	1	<sup>a</sup>	5					
6	Coal tar epoxy	1	1	<sup>b</sup>	4½				
6A	Sand-filled coal tar epoxy	1	1	1	1	1	1	1	<sup>c</sup>
13	Coal tar epoxy	<sup>d</sup>	5						
35	Modified epoxy	1	<sup>d</sup>	5					
36	Modified epoxy <sup>e</sup>	1	<sup>d</sup>	5					
37	Modified epoxy	<sup>d</sup>	5						
38	Modified epoxy <sup>f</sup>	1-3	5						
39	Polysulfide-modified epoxy	1	<sup>d</sup>	4-5					
40	Polysulfide-modified epoxy	1	1½	<sup>d</sup>	5				
45	Epoxy	<sup>d</sup>	5						
46	Epoxy	<sup>d</sup>	5						
47	Epoxy	<sup>d</sup>	5						
54	Penetrating epoxy coating	1	5 <sup>g</sup>	5					
55	Penetrating epoxy coating	<sup>d</sup>	5						
56	Epoxy	<sup>d</sup>	5						
106	Epoxy enamel	1	<sup>d</sup>	5					
117	Polysulfide-modified epoxy	1	1	<sup>h</sup>		5			
118	Polysulfide-modified epoxy	1	1	<sup>d</sup>	5				
29	Polyurethane resin	1	<sup>d</sup>	5					
32	Polyurethane resin	<sup>d</sup>	5						
34	Polyethylene-polyurethane copolymer	1	<sup>d</sup>	5					
22	Acrylic emulsion	2	<sup>d</sup>	5					
23	Acrylic emulsion	2	<sup>d</sup>	5					
42	Acrylic emulsion	3½	5						
44	Acrylic emulsion	3½	5						
57	Acrylic emulsion	4							
8	Polyvinyl butyral solution	2	<sup>d</sup>	5					
14	Hydrocarbon resin	<sup>i</sup>	5						
17	Hydrocarbon resin emulsion	<sup>i</sup>							
19	Polyvinylidene chloride dispersion	<sup>i</sup>							
20	Polyvinylidene chloride dispersion	<sup>i</sup>	5						
21	Polyvinylidene chloride dispersion	2	<sup>i</sup>	5					
27	Silicone resin solution	4							
43	Polymeric resin	3	5						
101	Hydrocarbon resin	2	4						
102	Hydrocarbon resin	2	4						
103	Hydrocarbon resin	4							
16	Rubber solution	1½-5	5						
28	Rubber solution	4							
105	Rubber solution	1	<sup>d</sup>	5					
107	Rubber latex	1	<sup>d</sup>	5					

<sup>a</sup> Flaking. <sup>b</sup> Extensive flaking. <sup>c</sup> Deteriorated under coating. <sup>d</sup> Peeling. <sup>e</sup> No. 35 diluted 50-50 with kerosene.  
<sup>f</sup> No. 37 diluted 50-50 with kerosene. <sup>g</sup> Spots. <sup>h</sup> Slight peeling. <sup>i</sup> Peeled. <sup>j</sup> Extensive peeling.

The rubber-base coatings that penetrated into the concrete resulted in a permeable coating that was quickly penetrated by the salt solution. The rubber-base coatings that formed an impermeable coating failed by peeling within the first 54 cycles.

#### INORGANIC COATINGS AND TREATMENTS

A number of inorganic coatings or treatments expected to be more compatible with the concrete than the organic

coatings were investigated. The results (Table 8) were not encouraging. The best protection was achieved with a low alkali content silicate solution, but it was decidedly inferior to the linseed oil coating.

#### MISCELLANEOUS COATINGS

Several coatings composed of tars, waxes, greases, and mineral oil were evaluated. None of these (Table 9) was particularly effective in protecting concrete from salt scaling.

TABLE 8  
PERFORMANCE OF INORGANIC COATINGS

COATING NUMBER	GENERIC DESCRIPTION	APPLICATION <sup>a</sup>	DEGREE OF SCALING AFTER INDICATED NO. OF CYCLES		
			27	54	81
41	Air-entrained cement mortar	1/16 to 1/8 in.	<sup>b</sup>		
69	Slurry of fine-ground portland cement	20 gm	4		
70	No. 69 + 5% Wyoming bentonite	15 gm	4		
71	Sodium silicate solution	10 gm	1	3	5
72	Low-alkali silicate solution	10 gm	4		
74	Flame-sprayed borosilicate glass	0.05 in.	4		
75	Low-alkali silicate	10 gm	4		
76	10% sol. sodium silico fluoride	8 gm	3	5	
77	Uncoated slab <sup>c</sup>	—	4		
79	10% sol. lead phosphate	8 gm	3 1/2-4		
80	10% sol. lead acetate	8 gm	3-4		
81	20% sol. lead nitrate	8 gm	3-3 1/2	5	
92	Sodium silicate sol. and rubber latex <sup>d</sup>	8 gm	2	4	
93	No. 92 + 10% chromic oxide	8 gm	2-3	5	

<sup>a</sup> Thickness, or weight per slab.

<sup>b</sup> Spalled off.

<sup>c</sup> Steam cured at 335 F for 8 hr.

<sup>d</sup> Equal volume mixture.

TABLE 9  
PERFORMANCE OF COATINGS BASED ON TARS, WAXES, GREASES, OR  
MISCELLANEOUS ORGANIC MATERIALS

COATING NUMBER	GENERIC DESCRIPTION	DEGREE OF SCALING AFTER INDICATED NO. OF CYCLES		
		27	54	81
4	Coal tar product	2-3	5	
58	A hydrocarbon	3	5	
84	Saturated hydrocarbon mixture	4		
86	Fluorinated hydrocarbon spray	5		
90	Silicone grease	2	5	
91	50% creosote sol. in naphtha	1 1/2-2	4	
99	SS-1 asphalt	<sup>a</sup>	4	
100	RC-1 asphalt	<sup>a</sup>	5	
26	50% oleic acid sol. in kerosene	3	4 1/2	
78	No. 20 motor oil	1 1/2	3	

<sup>a</sup> Peeling.

## CHAPTER THREE

## MECHANISM OF SALT SCALING

Although the principal aim of this study was to evaluate existing protective coatings and develop improved coatings, incidental attention was given to the mechanism of salt scaling to provide guidance for selecting and developing improved coatings. A number of hypotheses have been proposed by various investigators to explain the destructive action of deicing salts on concrete. Mardulier (16) and Hartmann (17) reviewed previous hypotheses attributing salt scaling to (1) crystallization of deicing salts within pores in the concrete, (2) chemical reactions between the salt and cement compounds, (3) osmotic pressures superimposed on hydraulic freezing pressures, and (4) heat of solution effects that result in temperature differentials during thawing. Miss Hartmann eliminated these various effects on the basis of experimental evidence and calculations of osmotic pressures to be expected in practical situations and proposed a new hypothesis that scaling resulted from thermal stresses in the concrete surface caused by rapid cooling of the surface layer as heat was extracted from it by the rapid melting of ice when the deicing salt was applied. This hypothesis, of course, does not take account of the fact that the application of salt to an ice layer on concrete may be a sufficient condition to cause scaling, but it is not a necessary condition. Scaling can occur in the absence of the rapid thawing associated with applying salt: repeated freezing and thawing of a salt solution impounded on a concrete surface, by slowly changing the ambient temperature, will result in rapid scaling if the concrete is not adequately air-entrained. Experimental evidence by Verbeck and Klieger (2) indicated that salt scaling was principally a physical phenomenon rather than a chemical effect. The same type of scaling was produced by a number of chemically dissimilar materials and intermediate concentrations of the deicing chemicals were more destructive than highly concentrated or very dilute solutions.

The evidence, thus, seems clear that chemical effects are minor, that the major cause of scaling is physical, and that this effect is related to the lowering of the freezing point by the deicing chemical. Consideration of the probable course of events when concrete is contacted with a salt solution leads to the speculation that a concentration gradient would develop from the surface toward the interior of the concrete; the surface pores would be filled with a solution having a higher concentration of salt than pores toward the interior. The freezing point of the solution in the capillaries of the concrete would exhibit a corresponding gradient. The liquid in the interior capillaries would freeze at a higher temperature than the liquid on the surface and in the surface capillaries.

## EXPERIMENTS ON SCALING MECHANISM

Some experiments were conducted to determine if such a gradient in freezing temperature might exist during the

salt-scaling cycles, whether scaling might be influenced by the presence or absence of a concentration gradient, and whether scaling might be induced in the absence of deicing chemicals by establishing the proper temperature gradient.

*Temperature Measurements*

Two concrete slabs, 3 in. thick, were prepared containing three thermocouples imbedded at depths of approximately  $\frac{1}{4}$ , 1, and  $1\frac{1}{2}$  in. below the top surface. One slab was covered with water, the other with a 3 percent solution of sodium chloride. The temperatures at the top surface and within the slabs were recorded as the slabs were slowly frozen in a chamber maintained at about 0 F.

The temperature measurements revealed interesting differences in the freezing process between the two specimens. In the case of the slab covered with water, the interior temperature did not fall below the freezing point until the covering layer of water had frozen. Thus, freezing began at the surface and proceeded inward toward the center of the slab. In contrast, the temperature recorded by the uppermost thermocouple in the slab covered with brine showed that freezing occurred within the slab before the brine solution had cooled to its freezing point.

This difference in freezing behavior could account for the more severe deterioration associated with deicing salts. As freezing proceeds inward from the water-covered concrete surface, the hydraulic pressure associated with ice formation can be relieved by water movement toward the interior of the concrete. However, when the topmost, brine-saturated layer of concrete in the brine-covered slab begins to freeze, the hydraulic pressure is blocked by the layer of ice that formed earlier within the slab and by the overlying frozen brine. Thus, destructive pressures are set up that can result in scaling unless the topmost layer is adequately cushioned by air entrainment.

*Concentration Gradients*

Several experiments were conducted in which different gradients in the salt concentration from the top toward the center of the slab were established and the effect on the scaling rate was observed when the concrete was repeatedly frozen and thawed.

A uniform salt concentration in the surface layer of the concrete was established in two ways: (1) by using a 3 percent sodium chloride brine as the mix water, and (2) soaking the standard concrete slab for several weeks in a 3 percent sodium chloride solution. When covered with the normal 3 percent sodium chloride solution and repeatedly frozen and thawed as usual, the non-air-entrained slabs that were soaked in or made with salt solution scaled very slowly, reaching a scale rating of only  $1\frac{1}{2}$  after 54 cycles. When covered with distilled water rather than the salt solution, the uniformly salt-saturated slabs also

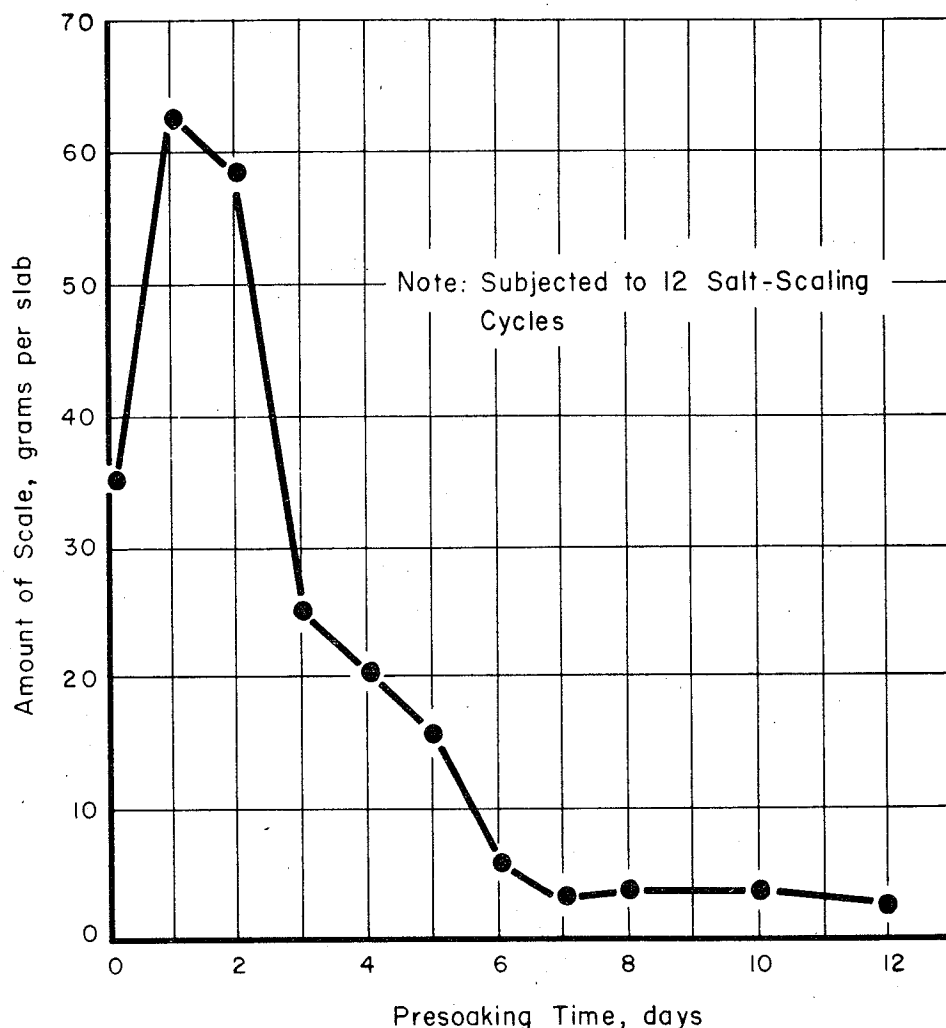


Figure 7. Effect of presoaking concrete in brine on amount of scaling.

scaled very slowly. One would expect scaling of the salt-saturated slabs to proceed rapidly (even more rapidly than the rate in the standard test procedure) if salt scaling were a chemical effect or caused by osmotic pressures.

The destructive effects of a concentration gradient were accidentally demonstrated in the first attempts to produce a uniformly-saturated slab by use of brine as the mix water. When the brine-saturated slabs were allowed to dry for the normal period before they were subjected to salt scaling, the drying resulted in migration of salt to the surface of the concrete and accidental development of a concentration gradient. When subjected to the salt-scaling exposure, these slabs scaled rapidly. In the later experiments, migration of the salt was avoided by eliminating the drying step.

The concentration gradient was varied also by soaking standard concrete slabs in a 3 percent sodium chloride solution for various periods of time, subjecting the slabs to a fixed number of salt-scaling cycles and accurately measuring the degree of scaling by filtering and weighing the scaled portion.

The results (Figure 7) showed that the amount of scaling during 12 cycles went through a maximum as the soaking time was increased. The effect can be explained by the following reasoning. When the concrete was first covered with salt solution, the solution slowly diffused into the concrete and a concentration gradient developed. As diffusion proceeded, eventually the concentration gradient decreased and ultimately the concentration of salt in the surface layer of the concrete was essentially uniform. Thus, the initial increase in scaling and the subsequent decrease that was observed is completely consistent with the hypothesis that salt scaling is caused by a concentration gradient.

#### Temperature Gradient

If the concentration gradient causes scaling to occur because of its effect on the freezing sequence as was postulated from the earlier temperature measurements, the crux of the sequence is the fact that the freezing of the topmost portion of the concrete is delayed until after the portion immediately below it has frozen. An experiment was

conducted to determine if scaling could be produced in the absence of any deicing chemicals by delaying the freezing of the surface layer. The delay was produced by the use of an electric immersion heater which heated the surface of the water-covered concrete until the interior had frozen. The temperature at about  $\frac{1}{4}$  in. below the surface of the concrete was monitored by a thermocouple as the slab was alternately frozen and thawed in the automatic freeze-thaw chamber. The thermocouple output was fed to a controller that turned the heater on and off. The controller was set so that heat was applied to the top of the slab until the interior froze; then the heat was turned off so that the water and the upper surface of the concrete froze.

The water-covered slab, which in the absence of the delayed-freezing mechanism would require more than 54 cycles to show appreciable scaling, scaled relatively rapidly, reaching a scale rating of about 3 in 54 cycles. The scaling proceeded relatively slowly thereafter, perhaps because the thermocouple was now too close to the surface. The appearance of the thermally-scaled slab after 210 scaling cycles is shown in Figure 8.

#### CONCLUSIONS FROM THE STUDY OF THE SCALING PROCESS

These experiments prove that "salt" scaling is principally a physical effect rather than a chemical effect. Osmotic pressure is probably not an important effect in view of the very slow scaling of concrete uniformly saturated with a 3 percent sodium chloride solution. The development of a concentration gradient through the depth of the concrete is the primary cause of scaling. The ultimate effect of the concentration gradient probably is to produce excessive thermal and hydraulic pressures through its effect on the freezing point of the liquid phase within the concrete.

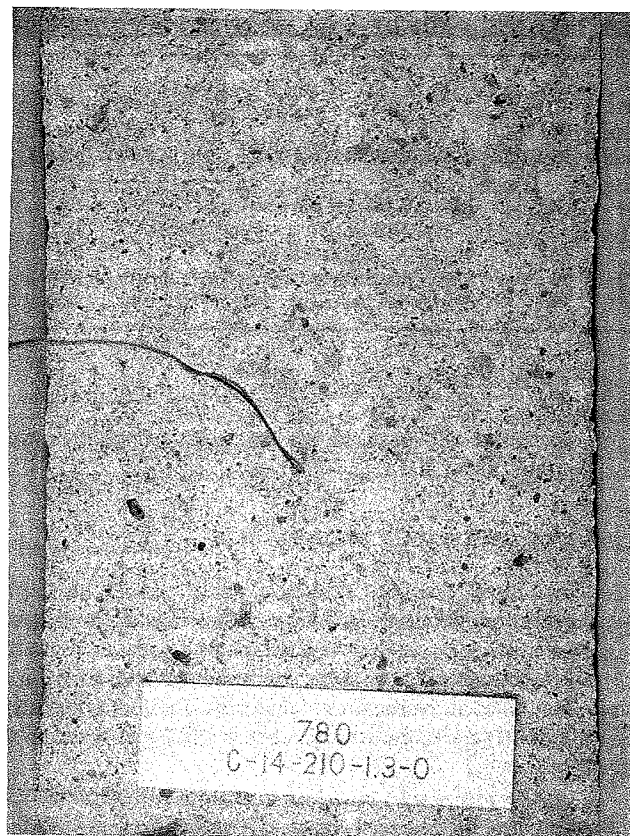


Figure 8. Thermally-scaled slab.

#### CHAPTER FOUR

## CONCLUSIONS AND RECOMMENDATIONS

The information gathered from the literature and produced in the experimental program lead to the following conclusions and recommendations.

#### GENERAL

Evidence from other studies and from practical experience was confirmed by experiments in this program that concrete which contains on the order of 6 percent of entrained air and which is properly cured is essentially immune to salt scaling.

Experimental studies of the salt-scaling process showed that salt scaling is intimately associated with the lowering of the freezing point of water by deicing chemicals. The effect is physical rather than chemical; it arises from the development of a depthwise gradient in the concentration of the deicing compound, and the stresses that result in scaling probably are a combination of thermal and hydraulic stresses.

Of the many protective coatings that were tried, best results, on the basis of protection and economy, were obtained with a solution of linseed oil. The relative per-

TABLE 10  
COMPARISON OF THE MOST EFFECTIVE COATINGS

COATING NUMBER	DESCRIPTION	CYCLES TO PRODUCE NO. 2 SCALE RATING	EST. COST OF COATING CONCRETE (\$/SQ YD)	
			MATERIALS	APPLIC.
1	Linseed oil solution	~189	0.03	0.04-0.09
6A	Sand-filled coal tar epoxy	200	1.50-2.00	2.50-4.00
15	Modified castor oil	189	0.08	0.10-0.16
97	Cottonseed oil	162	0.03	0.04-0.09
98	Soybean oil	189	0.04	0.05-0.12

formance and costs of the five most effective coatings are summarized in Table 10.

#### USE OF LINSEED OIL COATINGS

The linseed oil coating preferably should be applied to the cured concrete structure before it is exposed to deicing salts. In view of the present lack of assurance that adequate air entrainment will be achieved in the surface of the concrete, it is recommended that linseed oil coatings be applied to all new structures that will be exposed to deicing salts.

The preferred linseed oil coating is a 50-50 (by volume) mixture of boiled linseed oil and kerosene. Two coats, the first applied at a rate of about 40 sq yd per gallon, the second at a rate of about 67 sq yd per gallon, are recommended. The first coat should be dry (few hours at 70-80 F) before the second coat is applied.

The coating should not be applied to wet concrete. Wet concrete should be allowed to dry for a day or two at temperatures of 60 to 90 F and humidities of 20 to 60 percent before the coating is applied. If the concrete is extremely dry, either it should be wetted thoroughly and allowed to dry for a day or two, or a third coat of linseed oil should be applied to insure that the surface is adequately sealed.

If the coating is to be applied to concrete which has been exposed to deicing salts or is partially scaled, all loose scale should be brushed away and the surface of the concrete should be scrubbed and flushed with clean water to remove as much of the absorbed salt as possible. The washed concrete then should be allowed to dry for a day before the coating is applied.

#### FURTHER RESEARCH

In view of the excellent protection that is achieved with proper air entrainment, research on foolproof air-entraining agents or other methods of achieving the requisite pressure relief is recommended. If the latter approach is taken, it is suggested that attention be concentrated on developing the proper microstructure in the upper surface layers.

A more detailed and comprehensive investigation of the mechanism by which linseed oil coatings protect concrete should provide leads to guide the development of improved and more durable coatings. The possibility of preventing the ingress of the deicing salt into the capillary pores of the concrete through coatings that act as semi-permeable membranes has been suggested by the limited study made so far.

Another approach to protecting the concrete which is suggested by the excellent results with linseed oil coatings is the development of impregnating coatings that are permanently tough and ductile at both high and low temperatures.

Some specific areas worthy of investigation to facilitate the utilization of the present linseed oil solutions include:

1. Development of a method of measuring the moisture content of the concrete *in situ* to permit application of the coating at the optimum moisture content.
2. Development of more complete information about proper balance between penetration and surface coverage to guide the applicator of the coating.
3. Development of modified emulsions that are less susceptible to re-emulsification.
4. Further exploration of the possibilities of extending linseed oil with lower cost materials.



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## APPENDIX A

### DEVELOPMENT OF SALT-SCALING EVALUATION PROCEDURES

One of the steps in developing the procedure for evaluating the salt-scaling resistance of coated concrete specimens was to determine the scaling rate of uncoated concrete. The effect of varying the air content of the concrete on the scaling rate was determined to establish the air content that would result in a reasonably rapid scaling rate if the concrete were adequately cured but not protected otherwise. Data on the scaling rate of uncoated and linseed oil-coated slabs as air content was varied from 1.5 to 6 percent are summarized in Table A-1.

The data show that concrete containing 6 percent of entrained air remained essentially scale free for an extremely large number of cycles. Concrete containing 3 percent of entrained air scaled relatively rapidly and the first salt-scaling evaluations accordingly were conducted on concrete specimens containing 3 percent of air. However, as more experience was gained it became evident that the scaling rates of uncoated specimens containing 3 percent air varied erratically. Apparently an air content of about 3 percent was the critical value for the concrete composition used in this study. Subsequently, non-air-entrained concrete, containing 1 to 2 percent of accidental air, was selected as the standard. Uncoated non-air-entrained concrete scaled to a rating of 4-5 in one week (27 cycles) but was adequately protected by the linseed oil coating.

Concurrently with the variations in air content, variations in the concrete composition and the curing time were investigated in the attempt to develop a reproducible and rapid evaluation method.

The concrete mix originally selected was a 6.5-sack mix with a water/cement ratio of 0.5, a maximum aggregate size of  $\frac{3}{4}$  in., and a coarse-to-fine-aggregate ratio of 1.85:1. Slab specimens 6 x 12 x 1 $\frac{1}{2}$  in. were cast in oiled wooden molds from the concrete mix, using a vibrating table to consolidate the specimens. After the initial set, the top surface of the slabs was brushed lightly and the slabs were cured under damp cloths overnight in the molds. The stripped slabs were cured in a room at 75 F

and 100 percent relative humidity. In the initial trials the standard curing time was 28 days.

Plexiglas strips were attached to the specimens with modeling clay to form dikes to impound a layer of brine on the top surface. Repeated freezing and thawing of a 3 percent sodium chloride brine between 0 and 40 F was selected for the salt-scaling exposure. The temperature cycling was done in an automatic freeze-thaw chamber on a 3-hr cycle.

Considerable difficulty was encountered with leaking of the brine solution in these early experiments and a number of alternative dike materials were tried. Mortar dikes, used in past studies by most investigators, were less prone to initial leakage than other dike materials tried, but deteriorated in the salt-scaling exposure and ultimately leaked to about the same extent as the plastic dikes. The problem of leakage was finally eliminated by casting the slabs in polyethylene trays; the trays were sufficiently deep that their sides formed a natural dike around the slabs.

At this stage of the development of an evaluation procedure, the difficulty with erratic scaling rates for concrete containing 3 percent of air was recognized. The possibility that the erratic results were a result of variations in the freezing-and-thawing conditions was investigated.

Temperature measurements were made with thermocouples at various locations in the freeze-thaw chamber and at various spots on and within concrete slab specimens as the chamber was cycled through the standard 0-40 F freeze-thaw program. These measurements showed that with a full load of specimens in the chamber and the control thermocouple attached to the bottom of one of the slabs (the standard location) the temperature of the surface exposed to the brine solution only cycled between about 15 and 25 F. Increasing the cycle period from 3 hr to 6 hr increased the temperature range at the brine-concrete interface slightly. Immersing the control couple in the brine increased the range to almost the desired 0-40 F cycle, but the temperature indicated by the control

TABLE A-1  
EFFECT OF AIR CONTENT OF CONCRETE ON SCALING RATE

AIR CONTENT (%)	COATING APPLIED	DEGREE OF SCALING AFTER INDICATED NUMBER OF CYCLES								
		27	54	81	108	135	162	189	216	355
1.5	None	4-5								
3.0	None	3	4	4						
6.0	None	1	1	1	1	1+	1½	1½	1½	1½
1.5	No. 1	1	1	1½	1½ <sup>a</sup>	1½ <sup>a</sup>	1½ <sup>a</sup>	2 <sup>a</sup>		
3.0	No. 1	1	1	1½	2	2	2	2		
6.0	No. 1	1	1	1	1	1	1½	1½	2	2

<sup>a</sup> Isolated spots of 3.

couple lagged behind the temperature called for by the program controller and after a few cycles the minimum temperature reached stabilized at about 20 F. Investigation disclosed that the refrigeration system was low on refrigerant and had been leaking for an unknown time previously. Repair of the leak and recharging with refrigerant cleared up this problem. With the refrigeration system in proper working order and the control couple (in a glass protection tube) immersed in the brine solution on a slab placed approximately in the middle of the chamber, the temperature at the brine-concrete interface cycled between 0 and 40 F in phase with the program controller on a 6-hr cycle. Temperature measurements on slabs at various locations in the chamber showed a maximum deviation of 10 F from the control couple.

In an alternate approach to improving the reproducibility and accelerating the evaluation process, the effect of using a lower quality concrete composition was explored.

The original composition selected was a 6.5-sack, 0.5-w/c concrete. The lower quality concrete tried was a 5.5-sack, 0.6-w/c concrete.

Slabs made from the lower quality concrete with no air entrainment and cured for 5 to 7 days scaled very rapidly. After only 11 cycles the uncoated slabs had scaled completely down to the coarse aggregate. Specimens from the same lot coated with linseed oil had partially scaled after 11 cycles.

In view of the extremely rapid scaling with the Class E concrete, subsequent effort was concentrated on the higher quality Class C concrete originally selected as being representative of the concrete used in highway structures. The procedure described in the body of the report, which used Class C concrete containing no entrained air and cured for 14 days in a fog room, was subsequently adopted as the standard.

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## APPENDIX B

### MOISTURE MEASUREMENTS

Initial rough measurements indicated that it might be possible to monitor the moisture content of concrete slabs by dielectric measurements. An instrument was assembled to investigate further this means of monitoring moisture content. In the first trials with this apparatus, encouraging results were obtained; the difference in readings between a saturated slab and a dry slab was reasonably large. However, experiments with slabs having intermediate moisture contents indicated that the relationship between moisture content and capacitance is nonlinear, and that most of the capacitance change occurs below 1 percent of moisture. Thus, the measurement is relatively

inaccurate for moisture contents in the range of greatest interest, 3 to 6 percent. Also, there was considerable scatter in the data.

The method ultimately adopted for measuring the moisture content of the concrete specimens was the direct weighing method. The concrete slabs were weighed when removed from the curing room and weighed again just prior to applying the coating. The moisture content at the time of applying the coating was calculated as a percentage of the total free moisture in the saturated slab on the basis of the average total free moisture from weight measurements on oven-dried specimens.

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