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EXPERIMENTS ON EXTERIOR WATERPROOFING MATERIALS FOR MASONRY

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ABSTRACT

This investigation was concerned mainly with the effectiveness and durability of various types of waterproofings. Thirty-two samples of the more widely used commercial products and ten nonproprietary materials were studied. It is believed that practically all types were represented. Waterproofing effectiveness was rated by the ability of the treatments to reduce the absorption rate of masonry materials. Durability values were obtained by exposing treated specimens to the weather for various periods up to thirteen years. The preservative value of the treatments was studied incidentally.

Wax types were found to be the most durable, but showed the undesirable property of producing discolorations on most masonry materials. The insoluble soaps produced no appreciable discolorations but were not very durable. Fairly satisfactory durability was indicated for the thinned fatty oils and better lasting qualities were obtained for thinned fatty oils with a high melting point paraffin in solution. However, with such types it seems necessary to adapt the consistency of the treatments to the pore structure of the masonry. Treatments which produced a film on the surface, such as normal varnishes of thin consistency, lacquers, and wax emulsions, were not found to be very satisfactory. Treatments consisting of two reacting solutions which produce insoluble precipitates and also treatments which are intended to react with the masonry, were not found to be very effective.

Although the results of studies on preservative value were not sufficient in scope to be conclusive, some evidence was obtained that effective waterproofing materials retard the deterioration of masonry due to the more common weathering actions.

CONTENTS

I. INTRODUCTION

A series of tests to determine the effectiveness and durability of waterproofing treatments on limestone and sandstone was started in 1921. A report [1] was published in 1924 giving the results obtained during a two-year exposure period. The study has since been extended to a larger variety of treatments and a more representative range of masonry materials. Since there is a decided demand for information on the preservative value of masonry treatments, some further experiments were made to determine whether the treatments have merit in protecting masonry from common weathering agencies and from decay due to crystallization of water-soluble salts in the pores.

In addition to twenty-nine proprietary treatments, various nonproprietary treatments and a few purely experimental processes were included in the studies.

The assistance of H. H. Dutton, H. E. Newcomer, and W. H. Sligh in making several thousand determinations of absorption rates is gratefully acknowledged.

II. MATERIALS AND PROCESSES

1. WATERPROOFING MATERIALS

(a) PROPRIETARY TREATMENTS

Samples of the proprietary treatments were supplied by the producers at the Bureau's request. In some cases two or more samples under the same trade name were obtained at different times, which afforded a means of determining variations in composition of the products. The trade names of the proprietary treatments were as follows:

Aquabar
Aquabar no. 2
Aquapel
Anhydrosol
Aridol
Cresolac
Dehydratine no. 2
Dehydratine no. 22
Dehydratine no. 222
Gar-Kem
G. F. no. 100
G. F. no. 145
Glidden's Colorless Waterproofing
Hydrolox
Kemisol
Lastaseal
Lithol
Minwax Clear
N. W. Colorless Waterproofing
Pecora Colorless Waterproofing
Porseal
Protone
Pyramid
Reduced 1017
Seal-A-Pore
Toxloxpore
Transparent Driwal
Tremco
Whigheldt's Xterior Waterproofing

The approximate compositions of these treatments are given in table 1. Where two samples are designated by the same numeral followed by letters, as 1a and 1b, they are two samples of the same trade designation. In some cases the compositions of two samples

[1] Kessler, Tech. Pap. BS **18**, 1 (1924–1925) T248.

of the same trade designation were such as to necessitate their being put under different classifications. Examples of this are as follows: Sample 2 was received under the same trade name as sample 8, sample 11 the same as sample 27, and sample 4 the same as sample 12.

TABLE 1.—*Proprietary preparations*

[Classification and composition]

THINNED FATTY OILS

Designation	Waterproofing ingredients		Volatile thinner, solvent or suspension medium
	Percent	Nature	
1a	18	China wood oil	Mineral spirits.
1b	27	do	Do.
2	10	do	Do.
3	16	Fatty oil	Petroleum distillate.
4	29	Fatty oil and aluminum soap	Coal tar distillate.
5	10	do	Turpentine and petroleum distillate.

THINNED FATTY OILS AND PARAFFIN

6a	33	Fatty oil and paraffin (ratio 52:48)	Mineral spirits.
6b	33	China wood oil and paraffin (ratio 43:57)	Petroleum distillate.
7a	38	Fatty oil and paraffin	Do.
7b	26	Fatty oil and paraffin (ratio 83:16)	Do.
8	24	China wood oil and paraffin (ratio 64:35)	Mineral spirits.

THINNED VARNISHES

9	32	Oils and resins	Mineral spirits.
10	38	do	Do.
11	39	do	Do.
12	38	Coal tar resin	Coal tar naphtha.

ALUMINUM SOAP SOLUTIONS

13a	4	Aluminum soap	Mineral spirits.
13b	4	do	Mineral spirits and turpentine.
14	3.4	Aluminum soap and wax (ratio 8:1)	Mineral spirits.
15a	7	Fatty oil, aluminum soap, and resin	Petroleum distillate.
15b	12	Heavy mineral oil and aluminum soap	Do.
16	2	Aluminum soap	Do.
17	4.5	do	Petroleum distillate and ethyl acetate.
18	5	do	Coal tar distillate.
19a	4.7	Aluminum soap, fatty oil, and resin	Turpentine and petroleum distillate.
20	?	Aluminum soap and paraffin	Petroleum distillate.

AQUEOUS EMULSIONS

21a	5	Casein glue and aluminum soap	Water.
21b	3.2	Glue, aluminum soap, and wax	Do.
22	4	Glue and aluminum soap	Sodium soap solution.
23	12	Linseed oil	Sodium carbonate solution.
24	33	Paraffin	Sodium soap solution.

WAX SOLUTIONS

25a	14	Paraffin	Petroleum distillate.
25b	12	Paraffin and other waxy material	Do.
26a	7	Paraffin	Mineral spirits.
26b	15	do	Do.
27	7	Paraffin and other waxy material	Coal tar distillate.

TABLE 1.—*Proprietary preparations*—Continued

MISCELLANEOUS

Designation	Waterproofing ingredients		Volatile thinner, solvent or suspension medium
	Percent	Nature	
28a	7.5	Cellulose nitrate	Ethyl acetate and acetone.
28b	10	do	Amyl acetate and fusel oil.
29	8.5	Fatty matter and ammonium soap	Diethylene glocol.
30		Fatty oil (about 4 percent) and a high boiling petroleum distillate.	Some lower boiling petroleum distillate.
31	1	Petroleum grease	Petroleum distillate.
32	15	Magnesium fluosilicate	Water.

(b) NONPROPRIETARY TREATMENTS

Ten nonproprietary treatments were tried, but several of these did not give sufficient indications of merit in the preliminary trials to warrant further study. These processes are described in table 2. Process 33 is known as Sylvester's, 34 as Ransome's, and 40 as Caffall's. The others were experimental treatments modeled after those which have been used or suggested as being of possible value.

TABLE 2.—*Nonproprietary preparations*

Designation	Applications	Composition
33	First	2 oz. of alum in 1 gal. of water.
	Second	12 oz. of potassium soap in 1 gal. of water.
34	First	Sodium silicate solution (soda-silica ratio approximately 1:2), density 1.20.
	Second	Calcium chloride solution, density 1.15.
35	Two	Sodium silicate solution (soda-silica ratio approximately 1:2), density 1.20.
36	One	Sodium silicate solution (soda-silica ratio approximately 1:2), density 1.20.
	Followed by several	Limewater.
37	First	10% solution of barium chloride.
	Second	10% solution of aluminum sulphate.
38	One	10% solution of paraffin in gasoline.
39	One	10% solution of beeswax in benzol.
40	One	Paraffin applied with specially designed heating equipment.
41	First	4% solution of ammonium oxalate.
	Second and third	4% solution of calcium chloride.
42	First	10% solution of barium chloride.
	Second and third	10% solution of aluminum sulphate.

2. MASONRY MATERIALS

The greater part of the tests were made on sandstone and limestone of various textures with the expectation that such materials would be representative of the entire range of masonry. During the progress of the studies it was found that masonry materials with different pore structures were not susceptible to waterproofing to the same degree with a given type of treatment; hence a wider range of materials was added, including marble, brick, cast stone, and mortar, with the hope of finding what types of waterproofing were best adapted to various masonry materials. In table 3 the masonry materials are listed with notes on their texture and pore characteristics.

The specimens of natural stone, cast stone, and brick were all cored into cylinders approximately 2⅛ inches in diameter and 2½ inches high. The mortar specimens were cast in the form of cylinders of about the same size as the cored specimens.

TABLE 3.—*Description of masonry materials used in waterproofing tests*

Serial number	Grades and characteristics	Absorption of untreated material 30 minutes
	LIMESTONE	
		Percent
1	Oolitic, grade A	4.49
2	do	3.16
3	do	4.24
4	Oolitic, grade B	4.91
5	do	4.00
6	do	4.12
7	Oolitic, grade C	5.24
8	do	4.48
9	do	5.94
10	Fine grained sandy dolomite	4.50
11	Very fine grained chalky limestone	5.16
12	do	18.35
	SANDSTONE	
13	Medium grained, open pore structure	5.22
14	Fine grained, close pore structure	3.58
15	Coarse grained, open pore structure	6.34
16	Medium grained, open pore structure	4.13
17	Medium grained, close pore structure	3.14
18	Fine grained, open pore structure	6.00
19	Coarse grained, open pore structure	6.50
	MARBLE	
20	Medium grained, saccharoidal texture	0.10
21	Fossiliferous, fine grained matrix	.33
22	Large crystal, calcite	.03
23	Fine grained, calcite	.12
	BRICK	
24	Stiff mud, side cut shale, dense	4.27
25	Dry press, clay, very porous	15.40
26	do	19.09
	CAST STONE	
27	Marble aggregate, pressed, dense	5.04
	MORTAR	
28	1:3 portland cement and building sand	

3. APPLICATION OF WATERPROOFINGS

The proprietary treatments were applied to the specimens with a brush in accordance with the directions supplied by the producers. In most cases this consisted of two applications with an interval of at least 24 hours between coats. The specimens were thoroughly dried before being treated and the waterproofings were applied copiously, that is, until absorption appeared to cease. Since the specimens were treated under ideal conditions the results may be assumed to represent the maximum waterproofing values that can be attained with such applications.

The nonproprietary treatments were applied as indicated in table 2, except the wax solutions, which were applied like the proprietary treatments. In those treatments consisting of two reacting solutions, as in 33, 34, 37, 41, and 42, the applications were repeated in the order given so the specimens received two or more applications of each solution. Treatment 40 was made by heating the surface of the specimens with a gas flame several degrees above the melting point of the paraffin, after which the molten wax was applied copiously with a brush. The surface of the specimens was then reheated as before.

For each test on a given sample of waterproofing, three specimens of one masonry material were treated.

III. METHODS OF TESTING

The most important characteristics of a waterproofing are the initial effectiveness, E_{w1}, and the rate of deterioration, R_d. The waterproofing effectiveness was measured by the reduction in absorption of the treated material compared to the absorption of the untreated material. For convenience a comparatively short immersion period (30 minutes) was selected. If A_n=the 30-minute absorption before and A_t the 30-minute absorption after treatment, the waterproofing effectiveness, E_w, may be expressed (in percent) as follows:

$$E_w=\frac{(A_n-A_t)100}{A_n}$$

The treated specimens were stored in the laboratory for a few days to dry before the original absorption test was made. Those which showed fairly satisfactory E_w values were exposed to the weather on a roof for durability studies. They were brought into the laboratory at intervals for absorption determinations. A continuous drop in the E_w values during the exposure period indicated deterioration of the waterproofing. The effective period was considered to be the time during which the E_w values remained above 50 percent. The deterioration rate, R_d, may be expressed as the decrease in effectiveness between two determinations divided by the time interval, thus:

$$R_d=\frac{E_{w1}-E_{w2}}{T}$$

where T is the time in years between the determinations E_{w1} and E_{w2}. In determining the R_d values from the effectiveness curves given in figures 1 to 8 where a number of E_w values are available, a straight line, approximating the slope of the E_w curve, was drawn and the intercepts on any two convenient ordinates taken as E_{w1} and E_{w2}. In cases where the E_w curve crossed the abscissa $E_w = 50$ the slope of the line joining this intersection with the initial effectiveness value was used in computing R_d.

IV. RESULTS OF TESTS

1. EXPOSURE TESTS

In figures 1 to 8 the results of exposure tests on 67 series of tests are plotted for exposure periods ranging from one to twelve years. In some cases the tests were continued a few years after the treatments failed to show satisfactory waterproofing values, and in other cases the tests were not continued far enough to show the actual life of the treatments, several being in good condition at the end of twelve years.

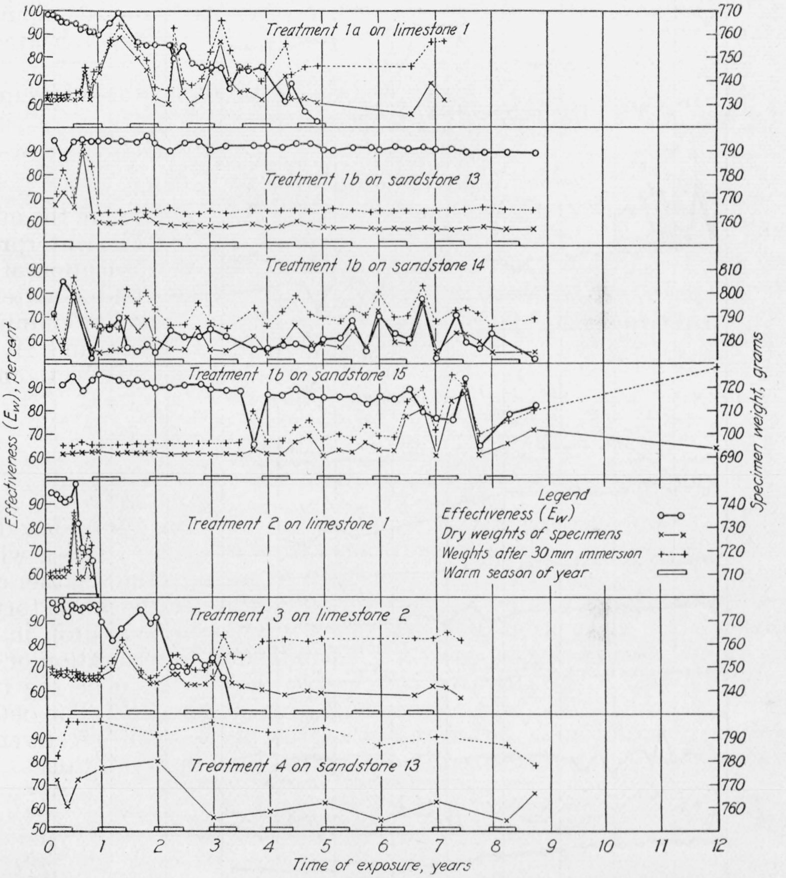

FIGURE 1.—*Results of exposure tests on thinned fatty-oil waterproofings.*

A considerable number of tests started from time to time during the progress of the original series were subjected to only occasional absorption tests. These results are not shown graphically but are described briefly. Since in some cases the seasons seem to influence the waterproofing effectiveness, the warm season of the year is indicated by black bars below the curves.

(a) THINNED FATTY OILS

In figure 1 are shown the results of seven series of tests on four proprietary materials of this class. Samples 4 and 5 differ from the others of this group in that they contain appreciable amounts of aluminum soap. Only one series of tests is shown for preparation 4, but two other series of tests were made with the same on sandstones 14 and 15, which gave results similar to those on sandstone 13. All three were very unsatisfactory. A series of tests with treatment 5 on lime-

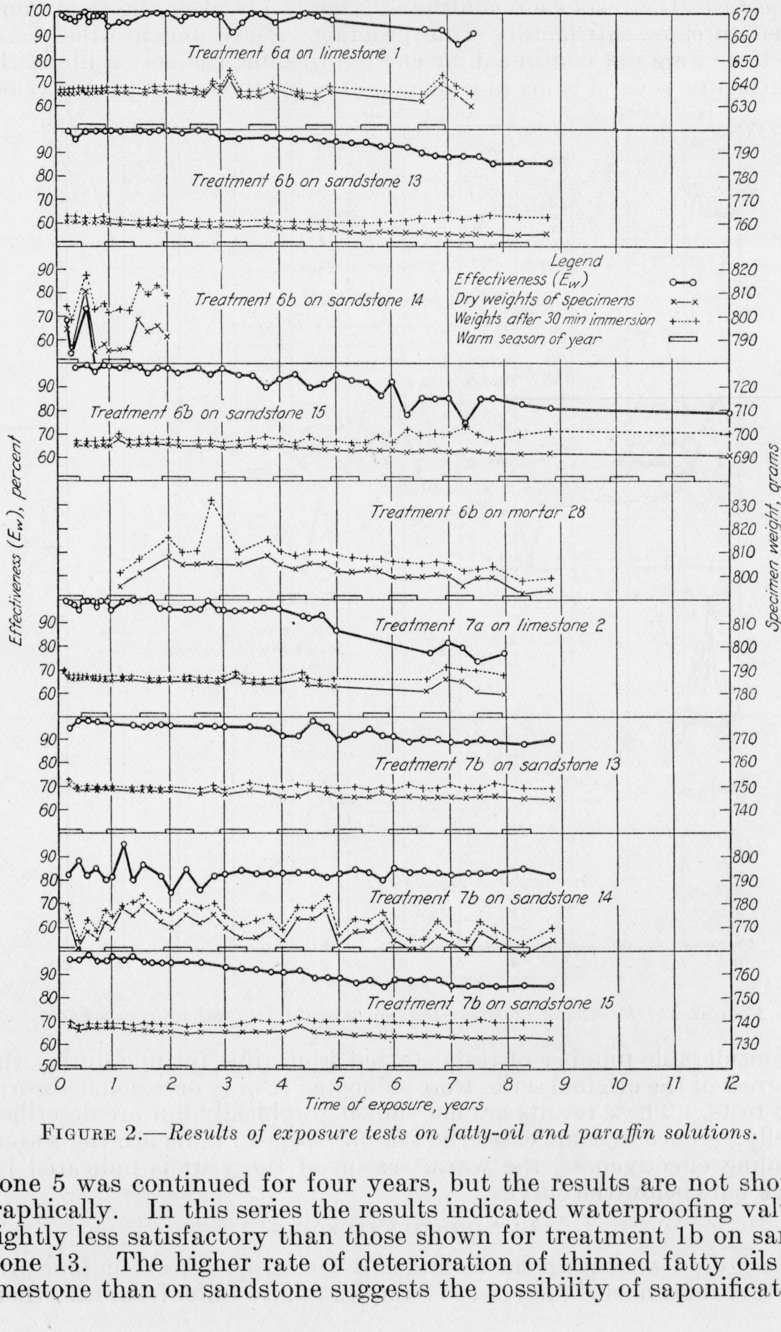

FIGURE 2.—*Results of exposure tests on fatty-oil and paraffin solutions.*

stone 5 was continued for four years, but the results are not shown graphically. In this series the results indicated waterproofing values slightly less satisfactory than those shown for treatment 1b on sandstone 13. The higher rate of deterioration of thinned fatty oils on limestone than on sandstone suggests the possibility of saponification of the oil in contact with limestone. Treatment 1b on sandstone 13 shows high waterproofing values throughout the duration of the tests and indicates that it is well adapted to such pore structures, although not satisfactory on close-pore structures like that of sandstone 14.

(b) THINNED FATTY OILS WITH PARAFFIN

In figures 2 and 3 are shown the results of twelve series of tests with limestone, sandstone, and mortar. This type showed satisfactory

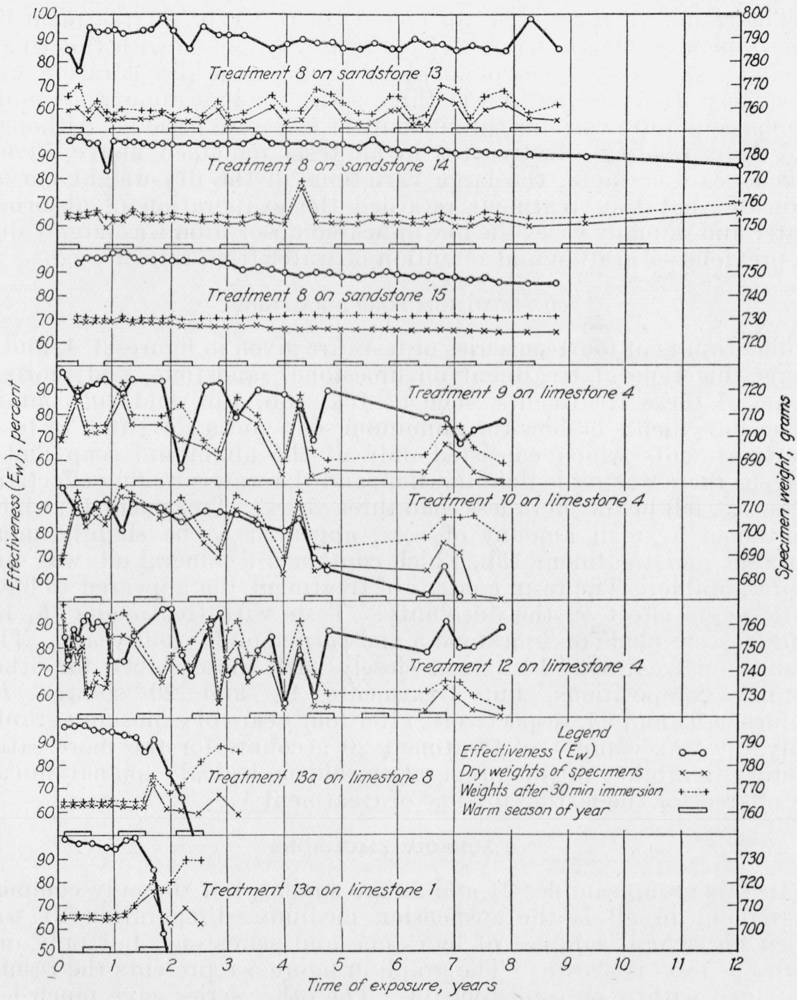

FIGURE 3.—*Results of exposure tests on fatty-oil and paraffin solutions, thin varnishes and aluminum-soap solutions.*

results in all the tests except treatment 6b on sandstone 14. Sandstone 14 in nearly all tests seemed to be particularly difficult to waterproof, but treatment 8 was evidently well adapted to such close-pore structures. Where this type of treatment showed high waterproofing values in the early stages of the exposure tests, the durability proved to be satisfactory. In the two series carried over a twelve-year period the E_w values were 80 and 85 at the end of the period. The effectiveness curve for the test on mortar is not shown because the original absorption of the mortar was not determined. However, the absorption curves indicate that the treatment was less effective on mortar than on sandstone and limestone.

(c) THINNED VARNISHES

The results of three series of tests with this type of treatment on limestone are given in figure 3. No tests were made with this treatment on the other types of masonry because the discoloration was excessive. The results of the three series on limestone were quite similar and quite variable throughout the exposure period. Although the waterproofing effectiveness in all tests remained above 50 for seven years or more, the large variations in the dry-weight curves indicate that the treatment retarded the evaporation of absorbed water and in many cases the low indicated absorption was largely due to previous saturation and retention of water from rains.

(d) ALUMINUM SOAP SOLUTIONS

The results of fourteen series of tests are given in figures 3, 4, and 5 using this type of treatment on limestone, sandstone, and mortar. Some of these treatments, such as 15a, 15b, 19b, and 20, contain other ingredients besides the aluminum soap and a solvent. In tests on treatments which consisted only of the aluminum soap and a solvent there was rapid deterioration, and the waterproofing effectiveness, E_w, fell below 50 in less than three years. Treatment 14, which contained a small amount of wax, appeared to be slightly more durable, and treatment 15b, which contained a mineral oil, was still more durable. The resin content of treatment 19a appeared to have little or no effect on the durability. Tests with treatments 16, 17, and 20 were made on limestone 5 and carried on for four years. The results on treatment 16 agreed closely with those shown for other similar compositions, but treatments 17 and 20 showed E_w values of 93 and 95, respectively, after four years of exposure. Probably the wax content of treatment 20 accounts for the more satisfactory durability of this preparation, but no logical explanation can be offered for the lasting quality of treatment 17.

(e) AQUEOUS EMULSIONS

In this group samples 21 and 22 are similar, but the only common ingredient in all is the suspension medium. Preparation 21 was tried on several samples of limestone and sandstone, but only one series of tests is shown. The graph in figure 5 represents the results obtained with it on sandstone 13. The other series gave much less satisfactory results. The results obtained on preparation 22 were like those for preparation 21 and are not given graphically. Preparation 23 was tried on limestones 5, 6, and 11, sandstone 17, and marble 22. These tests were carried for four years, but the only satisfactory result in the series was on limestone 6. Treatment 24 was tested on the same materials as treatment 23 and over the four-year period it gave good waterproofing results on all except limestone

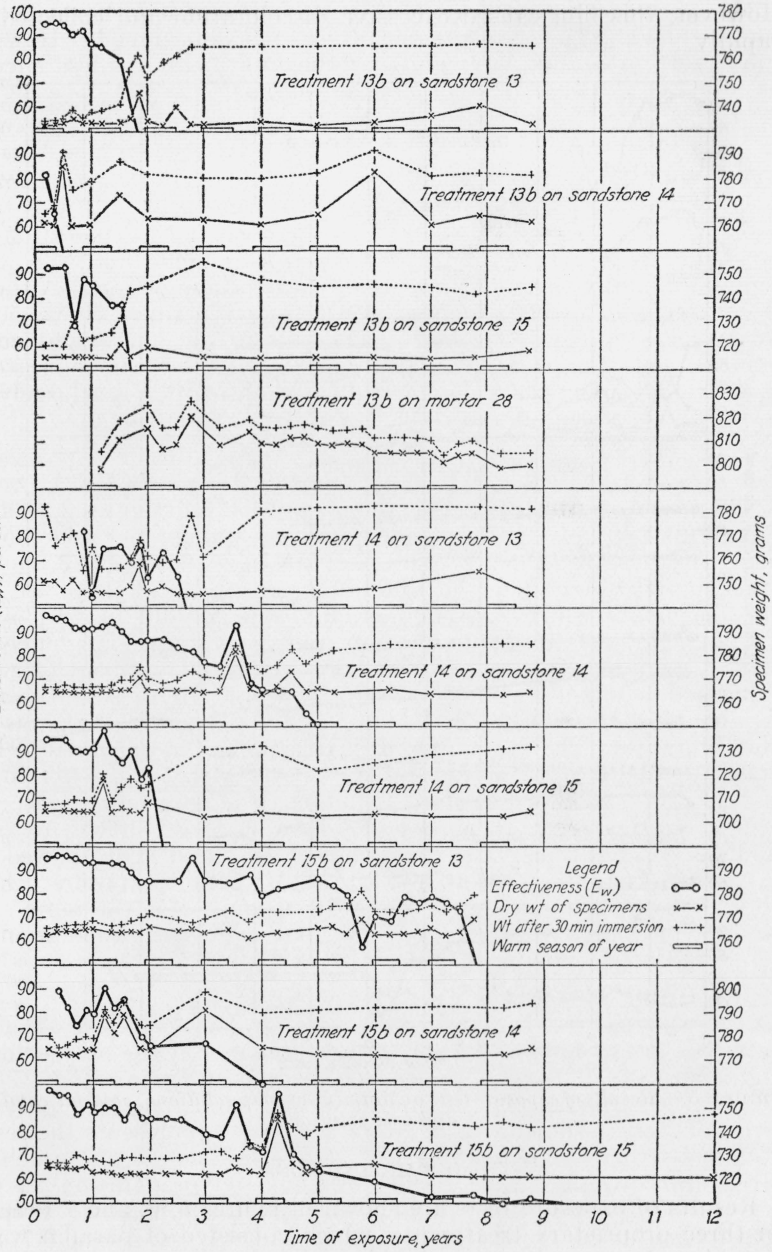

FIGURE 4.—*Results of exposure tests on aluminum soap solutions.*

10 and marble 22. This appeared to be due to a film of wax deposited on the surface of the specimens, as the penetration was very slight. However, this film caused excessive discoloration and collected dirt rapidly.

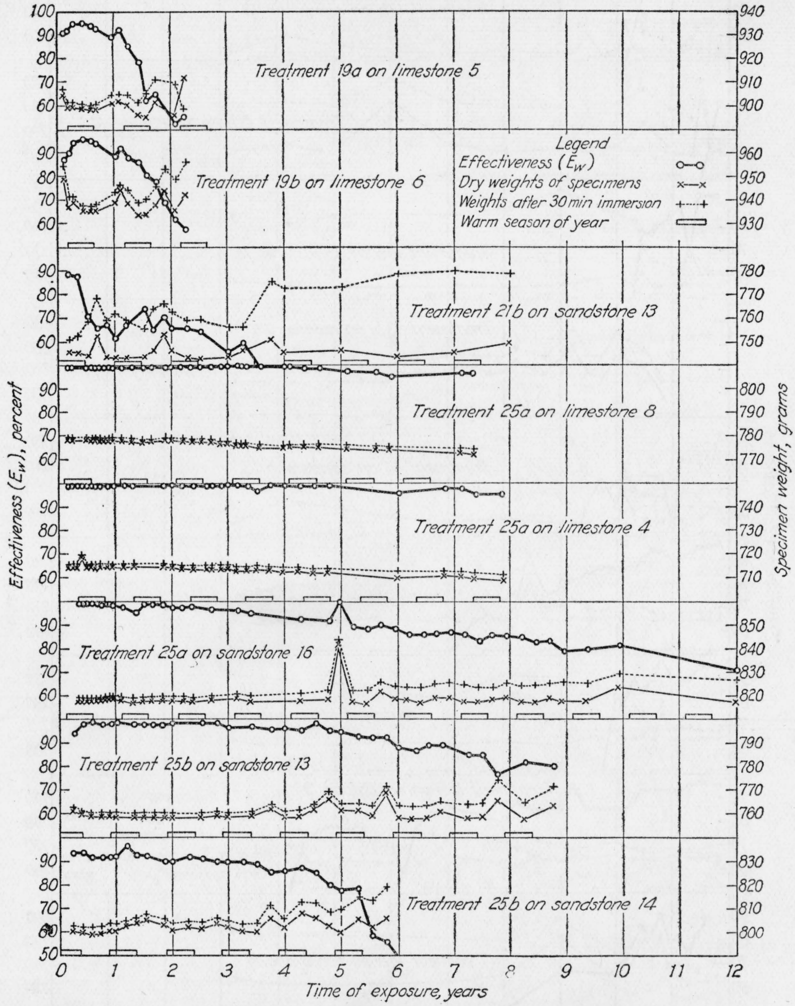

FIGURE 5.—*Results of exposure tests on aluminum soap solutions, aqueous emulsions, and wax solutions.*

(f) WAX SOLUTIONS

Results of exposure tests are shown in figures 5, 6, and 7 obtained on three proprietary treatments, which consisted of paraffin wax in suitable solvents. In general, this type of treatment showed quite satisfactory durability and high waterproofing values on materials with medium-sized pores. It does not seem to be well adapted to the very fine pore structures, and in some series of tests the results were not very good on the materials with large pores. Possibly a larger amount of wax in solution would prove more successful on the latter types of masonry. Some of the treatments were still showing good waterproofing values after twelve years of exposure. Occasionally it was found during the progress of the exposure tests that the speci-

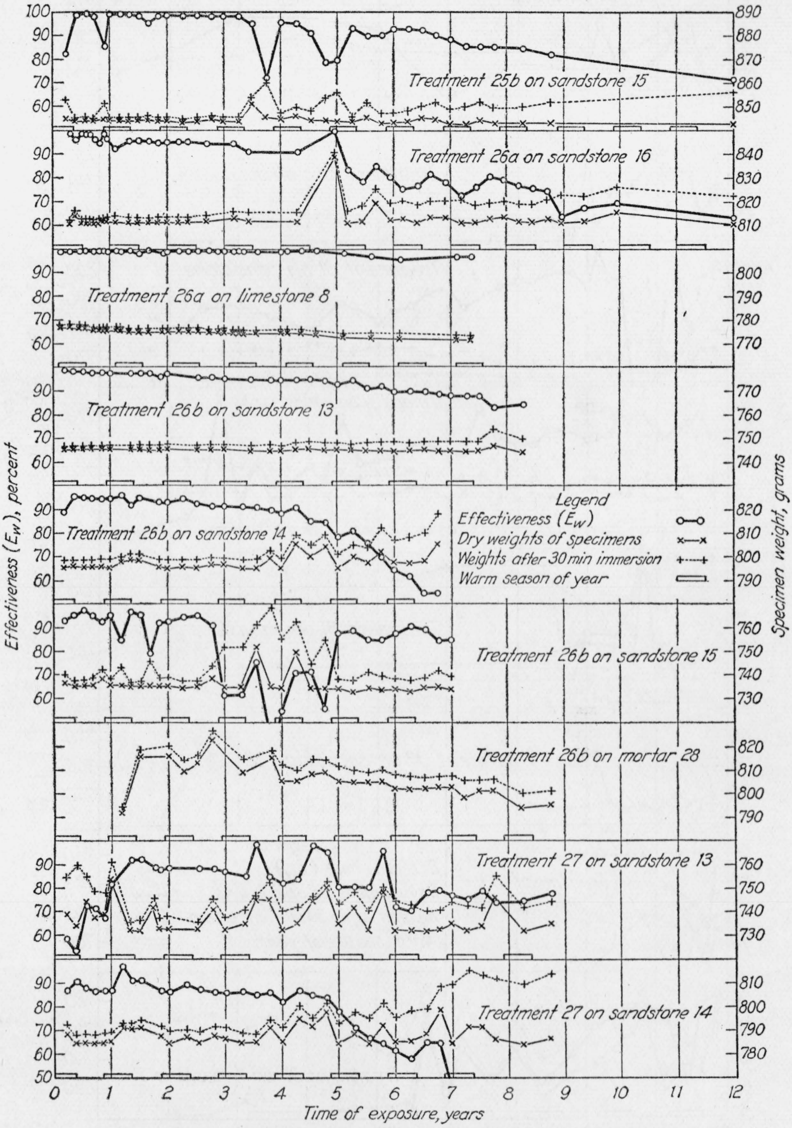

FIGURE 6.—*Results of exposure tests on wax solutions.*

mens had absorbed considerable moisture from rains. This is indicated in the graphs by a sharp rise in the weight curves. Usually these temporary breaks in the waterproofing effectiveness occurred in cold weather. It has been suggested that the wax may crystallize

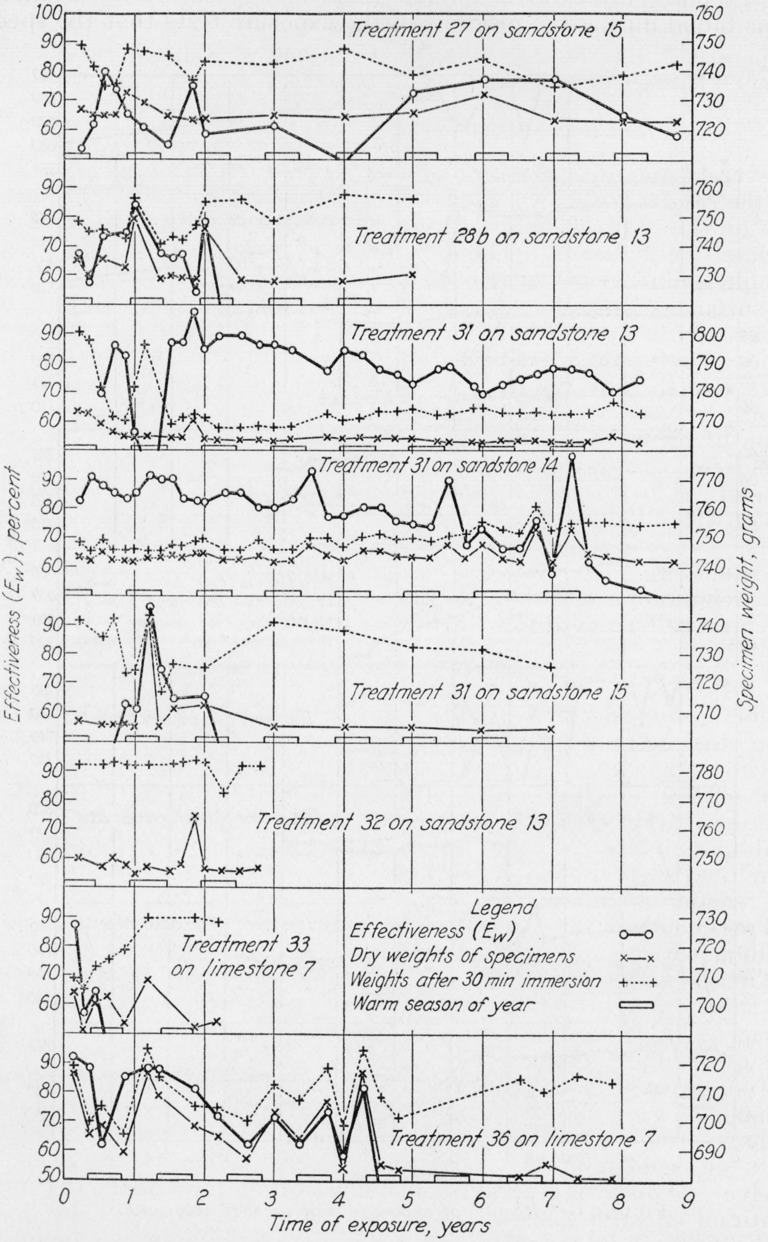

Figure 7.—*Results of exposure tests on wax solutions and miscellaneous compositions.*

at low temperatures and in the crystalline form it is not impervious. However, no prominent breaks in effectiveness are shown for the tests on specimens treated by process 40, and some experiments were made on specimens treated by this process by exposing them to low temperatures for several days, then making absorption tests in ice water. There was no increase in absorption due to this procedure.

(g) MISCELLANEOUS TREATMENTS

Several series of tests were made with the cellulose nitrate solutions, but the results are shown in detail only on treatment 28b. The other tests on this type of material were less satisfactory than those shown in detail in figure 7. There is little penetration and the waterproofing value seems to depend mainly on the film which is left on the surface. No results are shown graphically for treatment 29, but it was tried on marble 20, limestones 5, 6, 9, 11, and 12, and sandstones 17, 18, and 19. These tests were continued for nearly three years, and three of the tests on limestone showed good waterproofing values, but the others were poor. A series of tests with treatment 30 on limestone 9 was continued for nearly four years. There was considerable deterioration during this period, with only fair values at the end. Three series of tests are shown in figure 7 on treatment 31, two of which indicate fair waterproofing values over a period of eight years. Several series of tests with treatment 32 on limestone and sandstone were started, but none indicated any particular value. The weight determinations for one series are shown in figure 7, which is typical of all the tests with this preparation.

(h) NONPROPRIETARY TREATMENTS

This group embraces ten diverse treatments. Treatment 33 has been used to some extent and is commonly known as Sylvester's process. Several preliminary experiments were made with it, but none showed much promise. A graph in figure 7 gives the results of a series of tests with this process on limestone. Since the precipitate is aluminum soap, it could not be expected to be more durable than aluminum soap applied in solution. The potassium sulfate formed in the reaction is undesirable.

Treatments 34, 35, 37, 41, and 42 were tried on limestone in some preliminary experiments, but they did not give promising results.

Treatment 36 was tried on limestone 7 and the detailed results are shown in figure 7. It was by far the most effective of any of the processes involving two reacting applications, but the discoloration was quite objectionable.

Five series of tests with process 38 on two grades of limestone and a sandstone are shown in figure 8. The tests on limestone showed rather variable results especially on the grade A stones. The two tests on sandstone 16 gave good waterproofing values over the twelve-year exposure period and indicate that the treatments might continue effective for several more years. Tests were also made with 7, 10, and 14 percent solutions of paraffin (melting point 57° C; 135° F) on bricks 24, 25, and 26 and cast stone 27, which were carried over a five-year exposure period.

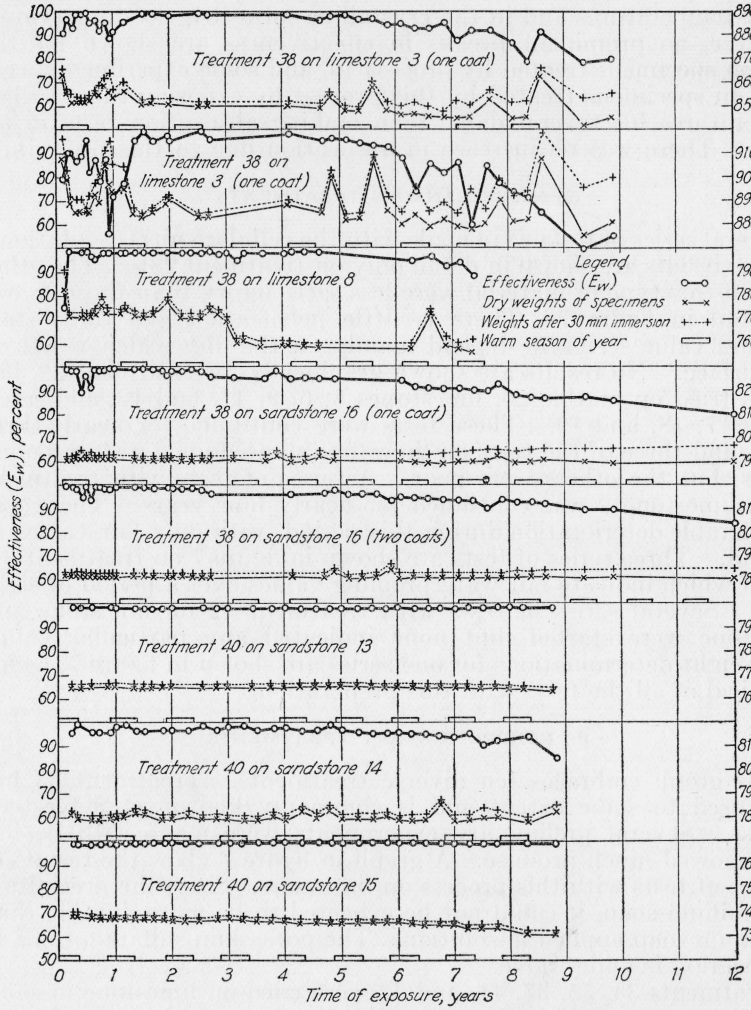

FIGURE 8.—*Results of exposure tests on nonproprietary treatments.*

The E_w values at the end of the period were as follows:

Material treated	Paraffin solution		
	7%	10%	14%
	E_w	E_w	E_w
Brick 24	96	79	72
Brick 25	96	98	98
Brick 26	35	29	15
Cast stone 27	79	82	84

All four of these masonry materials were somewhat like sandstone 14 in having very small pores. The results were quite satisfactory with the 7 percent solution on bricks 24 and 25, with all concentrations very poor on brick 26 and fair on the cast stone. The results were inconclusive as to the effect of solution strengths.

Tests on treatment 39 were carried no further than the preliminary stages because the discoloration was excessive.

Treatment 40 (Caffall process) was tried on three sandstones and carried over an exposure period of nearly nine years. The detailed results in figure 8 show practically no deterioration during this period for the medium and coarse-grained stones. The results were somewhat less satisfactory on the fine-grained stone. The gradual drop in the weight curve for sandstone 15 is believed to be due to the wax flowing out to some extent in hot weather, which indicates the necessity of using a higher-melting-point wax for materials with coarse-pore textures. In all of the tests on this material there was considerable discoloration by dirt collected on the surface. This indicates that it is necessary to remove the excess wax from the surface after treatment.

(i) SUMMARY OF RESULTS

In table 4 the waterproofing samples submitted to exposure tests are listed under the designations given in tables 1 and 2, with the observed initial effectiveness and computed deterioration rates for tests on the types of stone shown in column 2 of table 4.

TABLE 4.—*Initial effectiveness values and deterioration rates*

Waterproofing sample designation	Tested on—		Initial effectiveness (E_{wi})	Deterioration rate (R_d)	Remarks
	Material	Serial number in table 3			
		THINNED FATTY OIL COMPOSITIONS			
1a	Limestone	1	99	10	Produced oily discoloration.
1b	Sandstone	13, 14, 15	70 to 95	1 to 4	Do.
2	Limestone	1	97	48	Produced slight discoloration.
3	do	2	98	15	Produced oily discoloration.
4	Sandstone	13, 14, 15	54 to 78	15 to 36	Do.
5	Limestone	6	96	2	Do.
		THINNED FATTY OIL AND PARAFFIN COMPOSITIONS			
6a	Limestone	1	99	1	Produced oily discoloration.
6b	Sandstone	13, 14, 15	68 to 99	1 to 2	Do.
7a	Limestone	2	99	3	Do.
7b	Sandstone	13, 14, 15	83 to 97	1 to 2	Do.
8	do	13, 14, 15	86 to 97	1 to 2	Do.
		THIN VARNISHES			
9	Limestone	4	98	3	Produced oily discoloration.
10	do	4	99	7	Do.
12	do	4	84	3	Do.
		ALUMINUM SOAP SOLUTIONS			
13a	Limestone	1, 8	97 to 99	20 to 26	Very slight discoloration.
13b	Sandstone	13, 14, 15	81 to 95	25 to 60	Do.
14	do	13, 14, 15	20 to 96	9 to 20	Do.
15b	do	13, 14, 15	89 to 97	5 to 10	Do.
16	Limestone	5	80	16	Do.
17	do	5	98	1	Do.
18	Sandstone	19	99	7	Do.
19a	Limestone	5	91	17	Do.
19b	do	6	87	16	Do.
20	do	5	99	1	Slight discoloration.

TABLE 4.—*Initial effectiveness values and deterioration rates*—Continued

Waterproofing sample designation	Tested on— Material	Tested on— Serial number in table 3	Initial effectiveness (E_{w1})	Deterioration rate (R_d)	Remarks
		AQUEOUS EMULSIONS			
21a	Limestone	3	67	41	Slight discoloration.
21b	Sandstone	13, 14, 15	44 to 91	11 to 18	Do.
23	do	18	86	18	Produced oily discoloration.
24	Limestone	9	99	5	Produced greasy film.
		WAX SOLUTIONS			
25a	Limestone	4, 8	99	0.4 to 0.5	Produced oily discoloration.
25b	Sandstone	13, 14, 15	82 to 94	2 to 7	Do.
26a	do	16	99	3	Do.
26b	do	13, 14, 15	88 to 98	1 to 6	Do.
27	do	13, 14, 15	52 to 86	2 to 6	Do.
		MISCELLANEOUS COMPOSITIONS			
28a	Limestone	8	79	80	Produced glossy film.
28b	Sandstone	13, 14, 15	69 to 88	14 to 17	Do.
29	do	18	74	8	Produced oily discoloration.
30	Limestone	9	95	8	Do.
31	Sandstone	13, 14, 15	25 to 82	2 to 20	Very slight discoloration.
32	do	13, 14, 15	5 to 43		Do.
		NONPROPRIETARY PROCESSES			
33	Limestone	7	87	62	Very slight discoloration.
36	do	7	92	9	Produced white discoloration.
38	Limestone	3, 8	64 to 91	2 to 5	Produced oily discoloration.
38	Sandstone	16	99	1	Do.
40	do	13, 14, 15	95 to 99	0 to 2	Do.

2. CONTROL TESTS

Two series of tests on untreated specimens were made to determine if the absorption of the stones varied appreciably during exposure. The results are shown in detail in figure 9. Rather wide variations in

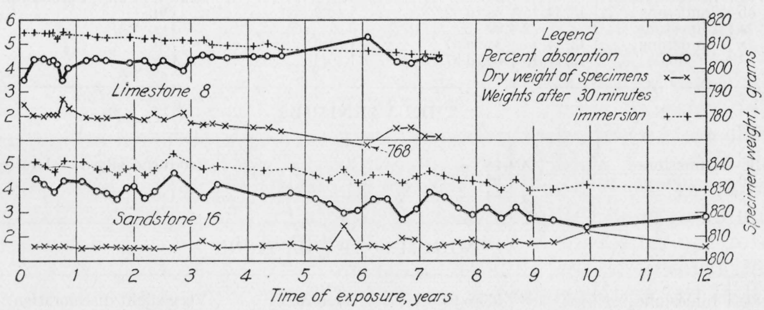

FIGURE 9.—*Exposure tests on untreated specimens.*

the absorption ratio are indicated, but as shown by the weight curves these are, in most cases, caused by the specimens being partly saturated at the first weighing. Another cause of variation was differences in temperature of the specimens and the immersion bath. In winter, when the specimens were brought in and tested before warming to room temperatures, the absorption values were too low. This was very likely due to expansion of the air in the pores of the specimens when placed in the water bath. This effect was shown very forcefully in a few cases when the treated specimens were immersed cold and partly saturated. In some such cases a negative absorption was indicated which showed that water had been forced out of the pores during immersion. It is also quite possible that the opposite effect came into play to some extent during summer, causing absorption values slightly higher than they normally should be. With a considerable number of determinations it is believed that the average is not far from correct. For the limestone the mean absorption ratio is shown to increase, while for the sandstone it decreased, but in both cases the changes are slight compared with the total absorption. The increase for the limestone is believed to be caused by the enlargement of the pores by the solvent action of rainwater. The decrease for sandstone is probably due to the pores near the surface becoming partly filled with dust.

3. TESTS OF PRESERVATIVE VALUE

Since waterproofing treatments are used occasionally for the purpose of preserving masonry, some studies were also made to determine their effect in reducing the deterioration from weathering. Water plays an important part in most weathering processes and if its penetration into the masonry can be prevented, masonry decay can presumably be reduced. The effects of such treatments in reducing weathering action were studied along the following lines: (1) Solvent action of rain water on calcareous masonry materials, (2) frost action, and (3) decay from crystallization of water-soluble salts.

(a) SOLVENT ACTION OF RAIN WATER

Rain water, being slightly acid, causes by its solvent action a gradual surface roughening of limestone and marbles. However, this surface effect is not of as much concern as the intrapore solution. This action increases the porosity and gradually weakens the bond between the component parts. Calcareous sandstones and other masonry materials consisting largely of inert ingredients cemented together with a matrix that is susceptible to acid action may be more seriously affected by this type of weathering than those that are entirely calcareous.

The rates of solution of a fairly porous limestone and a typical marble for an exposure period of twelve years are shown in figure 10. Both materials were freely exposed to the weather on the roof of a building at the National Bureau of Standards. The weights were determined at each test after thorough drying. The greater rate of solution of the limestone is believed to be due to deeper and more ready penetration of water, thus exposing more surface to the solvent.

All of the waterproofed specimens of limestone and marble showed weight losses during the exposure periods, indicating that even treatments which waterproofed effectively do not prevent surface solution of a calcareous material. Some information on the ability of the waterproofing to prevent intrapore solution may be gained by comparing the weight-loss rate of treated and untreated materials. Such a comparison can be made between limestone 8 in the original condition as shown in figure 10, and the same material treated with effective waterproofing materials such as treatments 25a and 26a, shown in figures 5 and 6. The weight loss of the untreated stone during seven and one-half years was 1.92 percent. The weight losses for the treated specimens for the same exposure were 0.65 percent in each case.

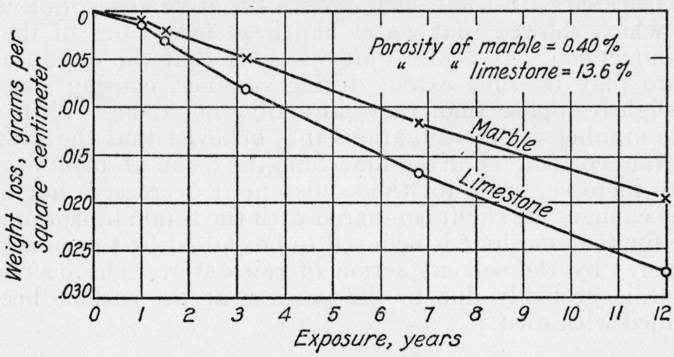

FIGURE 10.—*Solvent action of rain water on marble and limestone.*

Marble 20 (initial absorption 0.10 percent) was treated with seven different waterproofings and exposed to the weather for seven and one-half years for determinations of weathering effect. The percentage weight losses and final absorption of the specimens (each value is the average of three tests) were as follows:

	Waterproofing treatments—						
	1a	6a	13a	m	n	o	p
7½-year weight loss (%)[1]	0.80	0.70	0.92	0.62	0.97	0.72	0.68
Final absorption (%)	.17	.06	.24	.05	.24	.04	.05

[1] Weight loss for a surface area of 129 cm².

Treatments 1a, 6a, and 13a are described in table 1.

The other four were 10-percent solutions of paraffin waxes as follows: m, paraffin (melting point 50° C; 122° F) in gasoline; n, a low-melting-point paraffin in gasoline; o, paraffin (melting point 50° C; 122° F) in solvent naphtha, and p, paraffin (melting point 55° C; 131° F) in gasoline. None of the treatments on this marble gave very satisfactory waterproofing values at the beginning, and it will be noted in three cases the specimens were absorbing more after seven and one-half years than the original marble without treatment. The treatments which lowered the absorption rate appreciably were effective in reducing the solvent action. The loss on the untreated marble was 0.0016 g/cm² per year and for the marble treated with 6a, m, o, and p the average weight loss was 0.007. There were also appreciable differences in the appearance of the specimens, those with more effective treatments being smoother and usually cleaner.

FIGURE 11.—*Leaching test on limestone panel 1.*

Right-hand half treated with a 10-percent paraffin solution.

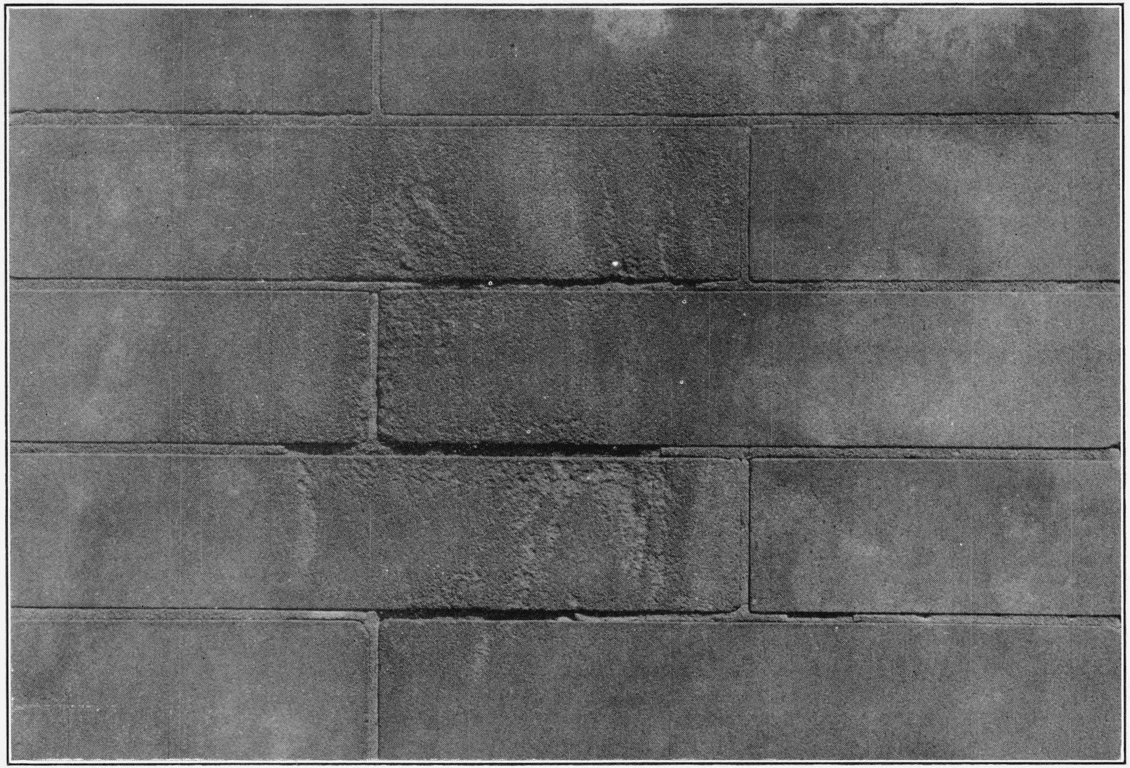

Figure 12.—*Leaching test on limestone panel 3.*

Right-hand half treated with a 4-percent solution of aluminum soap.

(b) FROST ACTION

A few tests were made to determine if waterproofing treatments increase the resistance of limestone and sandstone to frost action. The treatment consisted of two coats of a 10-percent solution of high-melting-point paraffin dissolved in benzol. The tests were made under severe conditions, first soaking the treated specimens in water for 14 days and standing them in shallow pans of water while being frozen. They were thawed by immersion in water at about 20° C for 1 hour. Treated and untreated specimens were frozen under the same conditions and the results were as follows:

Material	Number of freezing and thawing cycles causing disintegration of treated and untreated stones			
	No. 15	No. 19	No. 10	No. 6
Treated stone	220	246	187	99
Untreated stone	57	144	95	34

Although these experiments are too meager in scope to warrant final conclusions, they may serve as evidence that an effective waterproofing treatment will increase the frost resistance of such materials. Since the moisture content of masonry walls above grade is usually much less than in the specimens during these experiments, it is safe to assume that the treatment would be more effective in increasing frost resistance under normal service conditions.

(c) DECAY FROM SALT CRYSTALLIZATION

To determine if surface waterproofings are of value in preventing decay from the effects of water-soluble salts crystallizing within the pores of masonry, a series of experiments was made on some panels of limestone-faced brick masonry. Each consisted of a four-inch facing of Indiana limestone backed by four inches of common clay brick, the latter all from a single source.

The entire back face of the brick was coated with a bituminous waterproofing. A coping of the same stone covered the entire top of each panel. Four typical panels are shown in figures 11 to 14. The stone facing is on the south side. The photographs show the present appearance of the facing. The right-hand half of each panel was treated with a waterproofing and the left half was untreated. The mortars used for setting the stone and brick are shown in table 5.

Two blocks of the same limestone as used in the panels were mounted on top of the coping block. Each of these was cored from the top face to give cavities for collecting rain water and causing an excessive amount of leaching through the stone and brick below. This leaching carried to the surface a considerable amount of water-soluble salts existing in the stone, brick, or mortar. After rains the salts were usually conspicuous on the surface of the panels as patches of efflorescence, and the crystallization of the salts within the pores caused more or less spalling or crumbling of the stone.

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TABLE 5.—*Construction of limestone panels* [1]

Panel	Setting mortar: Limestone	Setting mortar: Brickwork	Waterproofing
1	1 part normal portland cement; 2 parts white sand.	1 part normal portland cement, 2 parts white sand.	All faces of limestone except front and exposed ends painted with bituminous waterproofing material to ½ in. of front face.
2	do	do	None.
3	1 part cement, plus 10 percent lime; 2 parts white sand.	do	Do.
4	do	1 part slag cement, plus 10 percent lime; 2 parts white sand.	Do.
5	do	1 part normal portland cement, 2 parts white sand.	Back of limestone painted with bituminous waterproofing.
6	1 part natural cement; 2 parts white sand.	do	Do.
7	do	1 part natural cement; 2 parts white sand.	None.
8	1 part white portland cement; 2 parts white sand.	1 part normal portland cement; 2 parts white sand.	Do.
9	1 part white portland cement; 2 parts Potomac sand.	1 part white portland cement; 2 parts Potomac sand.	Do.
10	Lime mortar	Lime mortar	Do.
11	Duplicate of panel 1 with C grade limestone.		
12	Duplicate of panel 3 with C grade limestone.		

[1] Panels 1 to 10, inclusive, were faced with grade A limestone similar to limestone 3 in table 3; panels 11 and 12 were faced with grade C similar to limestone 8.

Eleven years have elapsed since the treatments were applied. The individual treatments and notes on the appearance of the stone and mortar are as follows.

Panel 1.—Treated with preparation 38, two coats.

Untreated part.—Noticeable scaling on three stones. The most prominent area of decay covers about four square inches and is one-sixteenth inch deep.

Treated part.—Badly discolored by treatment, but the stone is smooth and shows no decay.

Mortar shows shrinkage cracks along one horizontal joint and is scaling slightly. Treatment did not prevent decay of mortar.

Panel 2.—Treated with two coats of a 15-percent solution of paraffin in gasoline.

Untreated part.—Prominent scaling over one-fourth of surface, one-sixteenth inch deep in several places.

Treated part.—Badly discolored but smooth over most of the surface. About 3 square inches of one block had chipped off, but the appearance is not like that caused by salt crystallization.

Mortar was in good condition over entire surface.

Panel 3.—Treated with preparation 13b, two coats.

Untreated part.—Deep scaling on parts of five stones.

Treated part.—Deep scaling on parts of four stones. The decayed part was near the center of the panel, about two-thirds of the area being on the untreated part.

Mortar crumbled to depth of one-half inch at decayed area of stone and friable over entire panel.

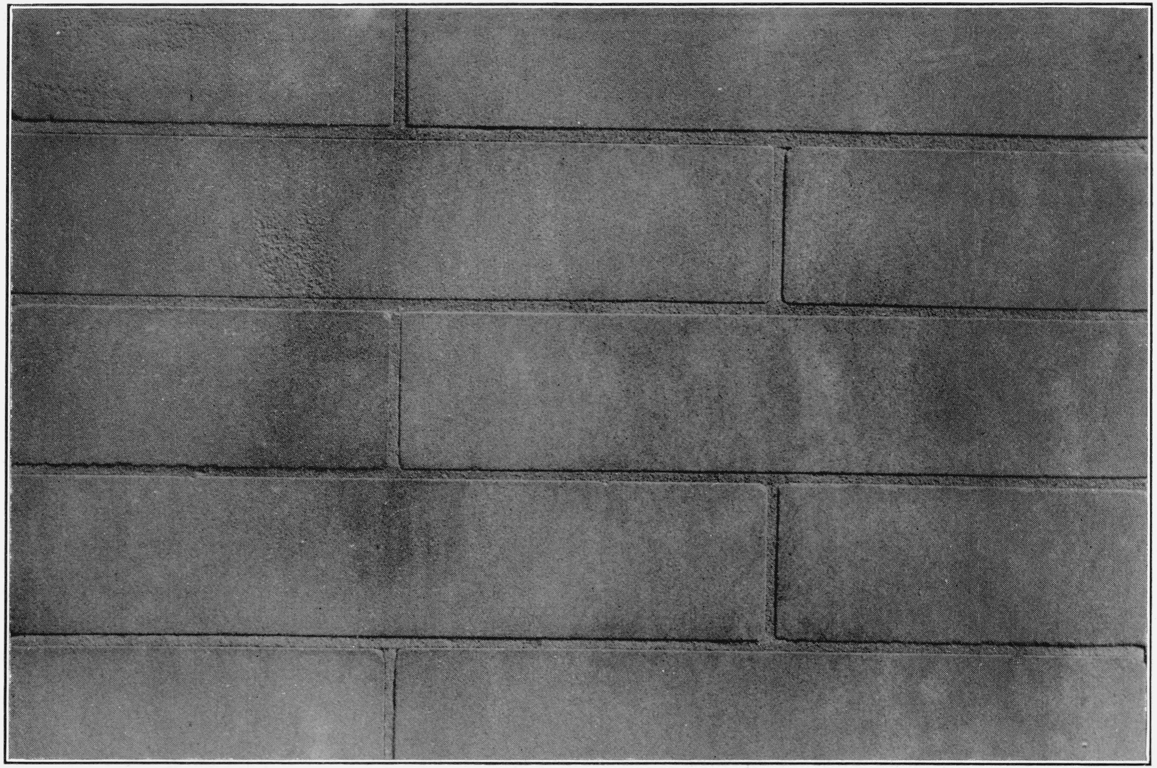

FIGURE 13.—*Leaching test on limestone panel 4.*

Right-hand half treated with a thinned china wood oil.

FIGURE 14.—*Leaching test on limestone panel 7.*

Right-hand half treated with a 15-percent paraffin solution.

Panel 4.—Treated with preparation 1b, two coats.

Untreated part.—Small areas of scaling on two blocks, rest in good condition.

Treated part.—Good condition.

Mortar is friable over entire surface of panel.

Panel 5.—Treated with preparation 13b, two coats.

Untreated part.—Prominent areas of scaling on three blocks, rest in good condition.

Treated part.—Similar to untreated part. Decayed area is near center of panel, about two-thirds being on untreated part.

Mortar is friable over entire panel and crumbled to depth of three-fourths inch in some places.

Panel 6.—Treated with preparation 27, two coats.

Untreated part.—Slight scaling on two blocks.

Treated Part.—Good condition and cleaner than untreated part.

Mortar is friable over entire panel but crumbled less on treated part.

Panel 7.—Treated with preparation 26b, two coats.

Untreated part.—Scaling slightly on three blocks.

Treated part.—Stone is discolored considerably, but smooth and in good condition.

Mortar is badly decayed over entire panel and crumbled to depth of one inch in places.

Panel 8.—Treated with preparation 31, two coats.

Untreated part.—Slight scaling on one block.

Treated part.—Good condition and somewhat cleaner than untreated part.

Mortar in good condition over entire panel.

Panel 9.—Treated with preparation 15b, two coats.

Untreated part.—Very slight scaling on one block.

Treated part.—Good condition, but soiled somewhat less than untreated part.

Mortar is sound, but shows a few shrinkage cracks.

Panel 10.—Treated with preparation 19b, two coats.

Untreated part.—Scaling on four blocks.

Treated part.—Slight scaling on one block.

Mortar is crumbling over entire panel.

Panel 11.—Treated with preparation 38, two coats.

Untreated part.—Good condition.

Treated part.—Discolored badly, but in good condition.

Mortar shows shrinkage cracks along all joints and is slightly more friable on treated area.

Panel 12.—Treated with 15 percent paraffin in gasoline, two coats.

Untreated part.—One small area of scaling, but rest is in good condition.

Treated part.—Badly discolored; two small areas spalled, but appearance is not like that caused by salt crystallization.

As noted above, six panels indicated that the waterproofing treatments were effective in preventing decay of the limestone by salt crystallization. The three panels which showed decay from salt action on the treated stone had been treated with a type which has been shown to be effective for only about two years. Two panels treated with a 15 percent solution of paraffin each showed a small area of spalled stone about one inch inside the border between the treated and untreated parts. The appearance of these areas suggests the action of frost rather than salt crystallization. One panel faced with grade C limestone showed no decay on either part. This may possibly be due to the relative capillary properties of the stone and brick. The bituminous waterproofing originally placed on the entire back (exposed) face of the brick did not remain effective for long, but soon scaled off. Hence, it was possible for the moisture which entered the masonry to pass out either through the limestone face or through the brick backing. It seems probable that the moisture was drawn either to the limestone face or to the brick face depending on which material exerted the stronger capillary action.

The bituminous waterproofing applied to the unexposed faces of each limestone block before setting in four of the panels did not show any definite value in preventing decay of the limestone face. Three of these, viz, panels 1, 5, and 6, showed appreciable disintegration of the limestone, but panel 11 did not.

4. DISCOLORATION EFFECTS

The discoloration of masonry surfaces by exterior waterproofing applications was studied by treating half of the face of each slab and comparing it with the untreated portion. There are two considerations in this connection, the initial discoloration and the appearance after weathering. One series of tests made on limestone slabs treated with the various types showed that initially the aluminum soap solutions stained very little, and the thin varnishes most of all. Thinned fatty oils and wax solutions stained in proportion to the amount of wax or oil in the treatment. The molten paraffin process discolored more than the wax solutions, and the cellulose nitrate solution produced a shiny film and splotchy effect. After a few months of weathering most of the discolorations disappeared, and the treated parts of the slabs appeared lighter in color than the untreated parts. After longer exposure there was no appreciable difference between the untreated portions and those with the less durable treatments. However, those with durable treatments remained cleaner for several years.

A similar series of tests was made by applying the treatments on sandstone 13, and marbles 20 and 21. These materials gave a range in color from gray to white, and a range in porosity from 0.5 to 20 percent. The tests indicated that the discoloration by the oil or wax types is greater on the more porous materials. On the very dense and slow-absorbing materials the treatments usually leave a film on the surface. Oil films soon weather away, but a film of paraffin is apt to remain for several years and collect dust.

The limestone panels (described in sec. IV–3–c) which were treated with wax solutions soiled very badly after a few months. This was evidently caused by applying the treatment too profusely, resulting in a film of wax on the surface which held dust particles and caused a soiled appearance. After six or eight years the film started to scale, and caused a mottled appearance.

The lower part of panel 1 was cleaned with an abrasive grit and water. The appearance is shown in figure 11. A moderate amount of scouring served to remove the deposit of dust. An attempt made to clean panel 2 with a wax solvent left a film of about the same appearance as the newly applied treatment.

V. ADAPTABILITY AND SELECTION OF TREATMENTS

As already shown, certain treatments are not equally effective on all types of pore structure. Limestones 10, 11, and 12, sandstone 14, marbles 20, 21, 22, and 23, brick 26, and mortar 28 represent types with finer pore structure that were difficult to waterproof effectively. Oolitic limestones 1, 2, and 3, the finer textures, were not as effectively waterproofed in most of the tests as limestones 4 to 9 with coarser textures. Many tests were made on sandstone 14 with various treatments and in most cases the results were much less satisfactory than the same treatments on sandstone 13, which had a relatively open-pore structure. However, treatments 8 and 14 gave somewhat better results on sandstone 14. Some experiments with 10 percent paraffin and 5 percent China wood oil in high-flash naphtha, applied to various materials of fine-pore structure like sandstone 14, indicated that this composition was well adapted to the type of pore structure that is difficult to waterproof with most of the present proprietary treatments.

It may be expected that treatments having low amounts of nonvolatile matter would prove ineffective on very porous masonry because the residue would not fill the pores to a satisfactory depth. Some evidence in support of this is gained by comparing sandstones 14 and 15 treated with preparations 14, 27, and 31, in figures 4, 6, and 7.

Occasionally manufacturers recommend certain products for use on stone, others for use on brick, and still others for use on concrete. Such designations are not well enough defined since any one class of masonry may have a wide range of pore structure, and waterproofings should be adapted to pore structure rather than to types of masonry. It seems best to determine a suitable treatment for any particular masonry by preliminary experiments with samples of the masonry.

In such preliminary experiments the main points to be determined are waterproofing effectiveness, penetration, and discoloration effects. Rough, dry fragments of the masonry two or three inches in diameter may be treated with the trial composition, and after a drying period of two days subjected to absorption tests of thirty minutes to determine the waterproofing effectiveness. One of the treated fragments broken open and dipped in water will show the approximate penetration of the treatment. Discoloration can be judged by comparing the treated and untreated material. One coat of the waterproofing should reduce the thirty-minute absorption at least 90 percent. Depth of penetration will vary according to the pore structure. For common types of brick, limestone, and sandstone this should be from one-eighth to one-fourth inch, but for denser materials, one-sixteenth inch should prove satisfactory. In cases where no appreciable discoloration is permitted the choice is limited to the stearate type. Since this type is not very durable a compromise between discoloration and durability may be desirable. The addition of paraffin wax (55° C melting point) to the stearate solution increases both durability and discoloration in proportion to the amount of paraffin added. Where durability is the first consideration and cost secondary, the Caffall process may be chosen. However, there is probably a limit to the size of pores that will successfully retain paraffin at high temperatures even though a high-melting-point wax is used. In some of the tests the wax flowed out and the specimens soon turned black with dirt accumulations. An economical treatment that is very durable may be made by dissolving from six to twelve ounces of a high-melting-point paraffin to the gallon of solvent, such as mineral spirits, naphtha, gasoline, etc. This usually gives high waterproofing values on materials of medium to coarse textures. For fine-pore structures it will be desirable to add from three to six ounces of China wood oil to the gallon of gasoline.

All of the solutions should be applied only when the masonry is dry, and in warm weather. Exponents of the Caffall process claim satisfactory results can be obtained with it on damp walls because the preliminary heating drives the moisture back from the surface. No experiments were made to determine the truth of that claim. Weather temperature with this treatment is evidently not an important consideration.

VI. CONCLUSIONS

The exterior waterproofings of the compositions studied show quite varied effectiveness and durability values. By proper selection of the treatment for any particular masonry it seems possible to obtain good waterproofing results, durability, and some measure of preservative value.

The study has indicated that some compositions may give good waterproofing values when applied to masonry of certain pore structures but inferior results for other types of pore structure. The problem of adapting the waterproofing to the masonry seems to be one of securing adequate penetration. Usually the more viscous solutions and particularly the emulsions do not penetrate to a sufficient depth when applied to masonry having small pores. Specific conclusions relating to the merits of the various types are as follows:

1. The thinned fatty-oil type of treatment appears to be fairly satisfactory on materials of medium texture but not well adapted to the fine-grained or coarse types of masonry. There is probably a slow rate of saponification in contact with calcareous materials. On sandstones of the variety which are satisfactorily waterproofed by this type of treatment, the rate of deterioration is low. Discolorations of an oily appearance are produced by this type.

2. Treatments consisting of fatty oils and paraffin in volatile solvents gave high waterproofing values and satisfactory durability in most of the tests. Those treatments with higher amounts of oil gave less satisfactory waterproofing values on materials of fine pore structure. Discolorations were about the same as for the thinned fatty oils.

3. Thin varnishes did not prove to be very effective and showed a tendency to prevent the escape of absorbed water. The discolorations were more pronounced than for the thinned fatty oils.

4. Aluminum soap solutions usually gave satisfactory initial waterproofing but showed a high rate of deterioration. The discolorations produced by this type were very slight.

5. Aqueous emulsions of waxes and oils gave unsatisfactory results in most of the tests. There seems to be an unsatisfactory penetration of such treatments when applied to most types of masonry, and the film of wax and oils remaining on the surface not only discolors but collects dirt.

6. Paraffin wax dissolved in volatile solvents proved to be very effective and durable on the materials to which the treatment is adapted. For use on fine-pore structures the effectiveness may be improved by small additions of fatty oil. The melting point of the wax should be sufficiently above summer wall temperatures to prevent the flow of wax out of the masonry. For most localities a melting point of 57° C (135° F) is satisfactory. Discolorations of an oily appearance are produced by the treatment.

7. Molten paraffin applied to masonry materials which have been heated somewhat above the melting point of the wax gave very high waterproofing values and had excellent durability. The melting point of the wax should be 57° C (135° F) or higher. Usually a film of wax remains on the surface and should be removed to prevent excessive discoloration and accumulation of dirt.

8. Applications consisting of two separate aqueous solutions that react to produce insoluble precipitates in most cases gave poor waterproofing values and in some cases showed a tendency to cause disintegration.

9. Treatments of the pyroxylin type showed little penetration and the film produced on the surface soon weathered away. This type produces a somewhat glossy and splotchy appearance.

10. The magnesium fluosilicate treatment gave no indications of waterproofing value in any of the tests.

11. Effective waterproofing treatments were of value in increasing the resistance of limestone to frost action, in reducing the destructive effects accompanying efflorescence, and in reducing the solvent action of rain water on calcareous materials.

WASHINGTON, January 9, 1935.