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# Preliminary Performance Criteria for Stone Treatments for the United States Capitol

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U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Gaithersburg, MD 20899

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**NAL BUREAU OF STANDARDS**

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# PRELIMINARY PERFORMANCE CRITERIA FOR STONE TREATMENTS FOR THE UNITED STATES CAPITOL

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**NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director***

# EXECUTIVE SUMMARY

Restoration of the West Central Front of the United States Capitol began in 1983 after Congress voted money to strengthen, restore, and preserve that national monument. Restoration includes cleaning and repainting the stone masonry. Sandstone from Aquia Creek, Virginia, was used in the original construction. The sandstone demonstrated rapid decay soon after being put in place in the late 1700's. (A large amount of the deteriorated sandstone damaged in the 1814 fire was replaced between 1816 and 1819.) Paint, which was first applied to the stone surface in 1818, apparently has protected most of the stone from rapid decay.

Badly deteriorated sandstone is being replaced with Indiana limestone. During the current restoration work, stone treatments applied to the cleaned sandstone to extend the lives of both the stone and subsequently-applied paint has been considered. Stone treatments, however, have been known to increase or hasten the damage to stone caused by weathering processes. In view of the many factors which may affect the performance of treated stone, the Architect of the Capitol requested the assistance of the National Bureau of Standards in developing a technical basis to aid in deciding whether or not treatments should be used on the stone in the West Central Front. To meet this need the approach was to develop performance criteria for stone treatment and paint systems. The pertinent performance criteria could then be used for screening treatment and paint systems to minimize the problems caused by their use. It is understood that, for aesthetic reasons, the Capitol will continue to be painted. Therefore, any treatment used must become part of a superior treatment and paint system.

The selection of appropriate performance criteria for a stone-treatment and paint system was based on an analysis of the likely processes responsible for the deterioration of the sandstone in the West Central Front. Sandstone deterioration which has taken place since the first painting does not appear to have been caused by salt damage or from moisture migrating outward from the interior of the Capitol. Based on these considerations, it was concluded that the major deterioration of the stone involved processes that were associated with water which penetrated into the stone from its exposed outer surface.

Based on the analysis of the sandstone deterioration, it was concluded that if a treatment is to be used on the West Central Front of the Capitol, the treatment or stone-treatment and paint system should protect the outer surface of the sandstone from penetration by rain. Since painting the sandstone has effectively protected it, any stone-treatment and paint system should protect the stone against water entry for at least as long as the paint

alone would, and preferably much longer. Further, in case all the deteriorated sandstone has not been removed, a consolidation treatment must be able to penetrate the deteriorated layers. Based on these requirements, four performance criteria for treatments were identified that considered the:

1. effect of treatment on initial paint adhesion,
2. effect of treatment on paint adhesion following prolonged exposure to moisture,
3. ability of treatment to penetrate deteriorated sandstone, and
4. ability of treatment to consolidate deteriorated sandstone.

Because they have not been verified by long-term studies, the performance criteria proposed in this report are preliminary criteria.

Five stone-treatments and paint systems were selected for investigation by a consultant to the Office of the Architect of the Capitol. The stone treatments consisted of one acrylic material and four silanes. Their compliance with the critical performance criteria developed in this study were determined. Only one of the five treatments appeared to meet all the criteria.

Recommendations are presented regarding the use of stone treatments on the West Central Front. It is recommended that a treatment be used on areas of deteriorated sandstone needing consolidation and on stone located in areas where weathering is most severe.

ABSTRACT

The West Central Front of the United States Capitol is being restored, including cleaning and repainting the sandstone, and replacing badly deteriorated sandstone with Indiana limestone. Application of stone treatments was proposed as a way of extending the lives of the sandstone and paint. However, without adequate selection criteria there was no certainty of effective performance by stone treatments. The purpose of this study was to develop performance criteria to assist in the selection of stone treatments.

Based on an analysis of expected deterioration processes, it is concluded that deterioration of the sandstone was most likely associated with water penetration into the exposed outer surface. Therefore, if a treatment is to be used, it should protect the outer surface of the sandstone from rain penetration. In addition, the treatment should penetrate and consolidate any deteriorated stone. Also, treatment should not cause premature failure of the paint. Based on these requirements, four preliminary performance criteria were developed, but have not been verified by long-term studies. Of five stone treatments evaluated, one met the four criteria.

It is recommended that treatment be used on areas of deteriorated sandstone needing consolidation and on stone in areas subjected to the most severe weathering locations.



Keywords: Consolidation, limestone, paint, performance criteria, restoration, sandstone, United States Capitol, weathering.

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# 1. INTRODUCTION

## 1.1 Background

Restoration of the West Central Front of the United States Capitol began in 1983, the stone has been cleaned, and some stone has been replaced. The walls are to be repainted. This section of the Capitol was constructed in the late 18th and early 19th centuries using a sandstone from Aquia Creek, Virginia, which rapidly deteriorated. B.H. Latrobe observed (1) that it "..cracks and falls to pieces on exposure to air and sun. Sometimes, contrary to all expectations and appearance, the frost tears it to pieces. All of it expands when wet, and contracts when dry." Fire damaged stone was replaced between 1815 and 1819. In 1818 the stone walls were first painted to protect them from the weather. During the present restoration, an inspection of the stone revealed that deterioration was confined largely to the surface of the stone. Because of cracking combined with surface deterioration, approximately 30 percent of the sandstone had to be replaced with Indiana limestone. Over the years about 35 layers of paint had been applied to the sandstone; the paint never was thoroughly removed until the present restoration. The color of the repainted West Central Front will closely match those of the other natural marble walls of the Capitol.

Application of stone treatments to extend the lives of both the sandstone and paint had been suggested at the start of the current restoration. Materials most often used to treat stone are intended to serve as preservatives and consolidants. The materials, however, have been known to accelerate the deterioration of stone (2). In recent reviews of stone preservatives and consolidants (3,4), it was noted that some preservatives and consolidants increased the harm to stone caused by weathering processes. Because the Capitol will continue to be painted, any treatment used must become a part of a superior treatment and paint system.

## 1.2 Purpose of Project

The Architect of the Capitol asked National Bureau of Standards (NBS) to develop technical criteria to help decide whether or not treatments should be used on the stone on the West Central Front of the Capitol. The approach taken was to develop performance criteria for stone treatments. The benefits are that performance criteria describe what the stone treatments should accomplish and define how their performance might be measured. Treatments meeting the pertinent performance criteria could then be considered for use.

During the project, the Architect of the Capitol requested that the project's scope be enlarged to include evaluation of the performance of stone-treatment and paint systems. Thus, performance criteria were also developed for these systems.

The performance criteria proposed in this report are considered to be preliminary since they have not been verified by long-term field studies.

### 1.3 Outline of Report

In Section 2, possible deterioration processes of the stone in the West Central Front are discussed. Analysis of possible deterioration processes is important in developing performance criteria for stone treatments. Section 3 reviews performances of stone treatments. Section 4 discusses performance requirements for stone treatments. Section 5 identifies materials used in the study. Section 6 describes test methods and test results. Section 7 describes preliminary performance criteria for treatments. Information on the compliance of candidate treatments with the performance criteria is also presented. Recommendations on the use of treatments on the Capitol are given in Section 8. Tables of the test data are in Appendix A.

## 2. STONE DETERIORATION

### 2.1 Condition of the Sandstone

According to Latrobe's report (1), the sandstone in the West Central Front of the US Capitol demonstrated rapid decay soon after being put into place in the late 1700's and early 1800's. Paint first applied to the stone surface in 1818, has apparently protected most of the stone from further rapid deterioration. Some 35 layers of previously applied paint were only thoroughly removed in the present restoration program. Undoubtedly the previous paints, mostly oil-based, formed a barrier of low permeability to the passage of moisture into or out of the stone. The depth of stone deterioration observed after paint removal was found in most areas to range up to 0.5 in (13 mm). In a few areas, where the exposure conditions were the most severe and carved stone was used (e.g., cornices) it was as deep as 1 in (25 mm). In most cases, the severity of the deterioration appears to be random over the surface of the walls and not associated with specific locations except where exposure conditions were the most extreme. During a scanning electron microscopy study no soluble salts were detected in the stone pores. However, had salts been present, they might have been removed during the paint removal. Veins of feldspar are present in the sandstone. During tests on the effect of treatments on moisture transport, surface staining of test specimens occurred.

Regarding the cause of stone deterioration, the sandstone was found by x-ray powder diffraction to be siliceous and thus should not be readily attacked by acid precipitation. The rate of deterioration since the stone was painted has been low, with a maximum loss of about 1 in (25 mm) of stone over some 170 years. Some of the observed deterioration may have taken place before the stone was painted. Records do not reveal how much stone was replaced between 1815 and 1819.

## 2.2 Causes of Sandstone Deterioration

Based on the present condition of the sandstone, the low rate of deterioration since the first painting, and the written record before 1818, it is concluded that the major deterioration of the stone in the West Central Front of the Capitol involved processes associated with water penetration into the stone's exposed outer surface. The justification for this conclusion is as follows: If salt damage occurred, it would not likely be random but would usually be concentrated near the source of the salt; for example, at the base of the wall if the source was ground water (fig. 1). Also, oil-based paint could aggravate the problem by trapping salts in the stone. Similarly, if moisture was migrating to the stone surface from the interior of the Capitol, freezing and thawing damage would be aggravated by the paint because it would reduce the rate of water evaporation; also, the resulting deterioration would not likely be random but would be concentrated near interior sources of moisture or at locations where thermal gradients would drive the moisture to cooler regions of the stone (fig. 1). However, the paint has effectively protected the sandstone from the rapid deterioration evidenced at the time when the stone was unpainted.

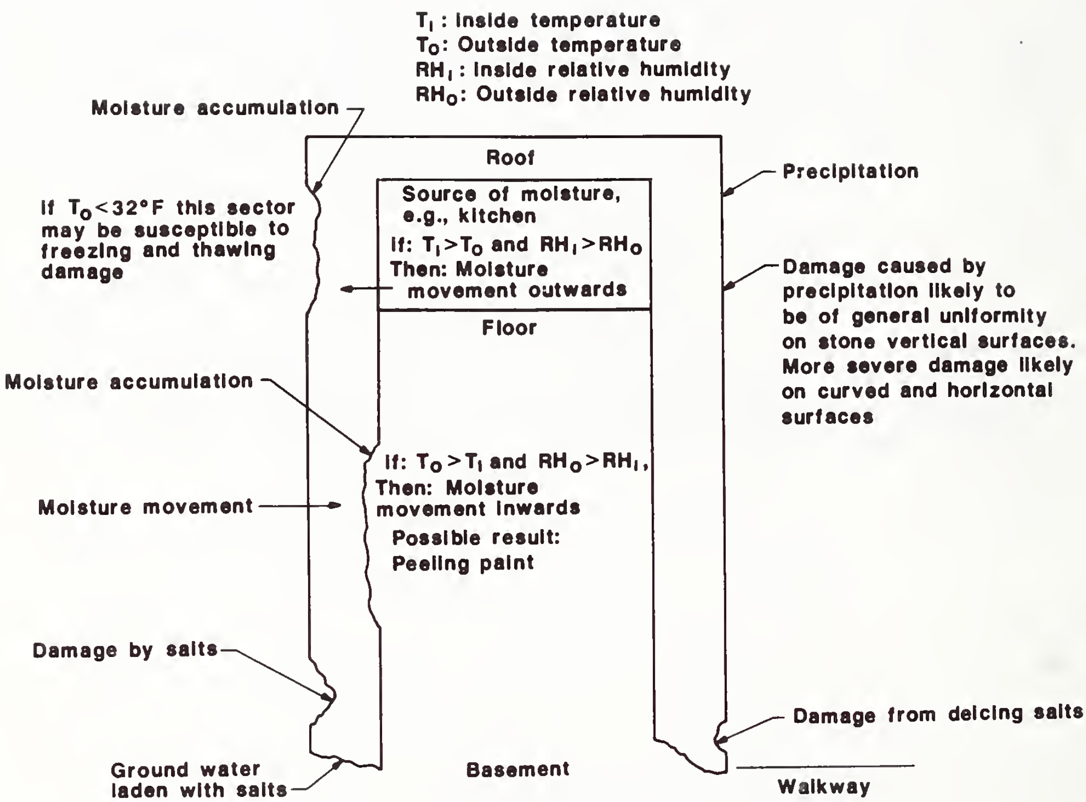


Figure 1. Schematic Showing Factors affecting Stone Deterioration Processes

## 3. PERFORMANCE OF STONE TREATMENT

Because the presence of moisture is associated with many of the common stone deterioration processes, "waterproofing" or "water repellent" materials have been applied to stone masonry for many years in an effort to reduce the rates of deterioration (3). In many instances, the use of these materials has been found to result in more stone damage than would normally occur by natural weathering processes. In some cases, their poor performance often has been attributed (4) to the formation of a thin layer of the material that is relatively impervious to liquid water but not to water vapor. This layer permitted the evaporation of aqueous salt solutions, rising from the ground by capillary action, to leave deposits of salts behind the surface of treated stone. Some waterproofing materials have been applied in such amounts that the pores of stone, at least those close to the surface, are filled. This impedes the passage of both liquid water and water vapor, thereby increasing the probability of freezing and thawing damage occurring under conditions in which moisture condenses behind the surface treatment. Moisture also could accumulate at the interface of a treated stone surface and a paint film thus, possibly reducing the adhesion of paint.

Paints themselves can perform as waterproofers. Since paint is unlikely to penetrate deeply into the stone, any resulting damage associated with the accumulation of moisture probably would be near the surface of the stone.

Consolidants are treatments intended to re-establish cohesion of the particles of deteriorated stone (4). Experience with consolidants has shown that their ability to penetrate weathered stone is one of the main factors controlling their performance (5,6). Superficially-penetrating consolidants tend to fill the pores of stone surface layers, thereby reducing their permeability and causing problems similar to those described for waterproofing materials (4).

Because the performance of stone treatments often has not been satisfactory, stone treatments and application methods should be evaluated by laboratory or field tests which can simulate the conditions encountered in service, before they are used on actual structures. Both standard test methods and performance criteria need to be developed to form a basis for evaluating and selecting stone treatments.

## 4. PERFORMANCE REQUIREMENTS

Performance requirements for a material are general qualitative statements describing the performance expected of that material for fulfilling an intended purpose. Based on the requirements, tests may be selected to measure desired performance characteristics. Then, performance criteria may be developed from the test results, past experience, and from other relevant information. In this section, performance requirements for stone treatments are proposed as a basis for the selection of test methods. They were developed in collaboration with the Office of the Architect of the Capitol. The applicability of a particular requirement may depend on specific exposure conditions. Therefore some of the following requirements may not be critical for the West Central Front based on the analysis of the apparent major deterioration processes (Section 2.2). They are presented as requirements always to be considered in selecting stone treatments or paints. The requirements below are not necessarily all-inclusive and others may need to be specified depending on the exposure conditions.

The following two performance requirements should be met when stone treatments are used as preservatives: a) the treatment should increase the service life of the stone; and b) where paint is applied, the treatment should not reduce the performance of the paint compared to paint applied to untreated stone. These essential broad considerations were subdivided into more specific catagories for consideration in the study. The necessity of fulfilling specific requirements depends upon the likely causes of the deterioration of the specific stone in question.

### 4.1 Performance Requirements for Effect of Treatments on Stone.

Treatments should increase the service life of the stone. Test methods which are recommended to determine compliance are mentioned.

4.1.1 Moisture Accumulation. A stone treatment should not permit accumulation of moisture in the stone sufficient to cause moisture-related deterioration (e.g., from freezing and thawing). The effect of treatments on the accumulation of moisture in stone was investigated by measuring water absorption (Section 6.1) and water vapor transmission (Section 6.2) of treated and untreated stone specimens. Also measured was the water vapor transmission of stone-treatment and paint systems.

4.1.2 Resistance to Internal Expansive Forces. A stone treatment should increase the resistance to damage by internal expansive forces resulting from such processes as salt crystallization, freezing and thawing. However, some treatments have decreased the resistance of stone to such processes, thereby substantially increasing the severity of the processes (4). The resistance of treated stone to salt crystallization damage was investigated by the sodium sulfate crystallization test (Section 6.3). The freezing and thawing resistance was investigated in an accelerated test which combines several deterioration processes (Section 6.6).

4.1.3 Consolidating Ability. A stone treatment used as a consolidant should strengthen deteriorated surface layers of stone. The consolidating abilities of treatments were determined by measuring the compressive strengths of treated and untreated stone specimens (Section 6.4).

4.1.4 Penetration of Weathered Stone. A stone consolidant treatment should be able to penetrate, to a reasonable depth, weathered layers of stone. If the stone is deeply weathered then it would probably be replaced. The depth of penetration of treatments was measured in both laboratory and field studies (Section 6.5).

4.1.5 Durability. A stone treatment should withstand the service environment to which it is exposed for a sufficient time to be useful. The resistance to weathering of stone treatments was investigated by an accelerated test (Section 6.6).

## 4.2 Performance Requirements for Effect of Treatments on Paint.

In general, stone treatments should not accelerate failure of paint by any process. In the present study, the main concern was the effect of the treatment on the adhesion of the paint and on the effect of sustained exposure to moisture on paint adhesion.

4.2.1 Bond Development. Few if any studies have been carried out to establish acceptable levels of bond development between paints and stone. Because of the lack of bond criteria, it was decided to require that the strength of the bond developed between the stone treatment and stone should not be substantially less than the bond developed between the stone and paint. Bond was measured by using a pull-off adhesion tester (Section 6.7).

4.2.2 Bond Degradation. Bond between treatment and paint should not be degraded by moisture at a rate faster than the paint/stone bond. Effect of moisture on bond strength was determined by water condensation testing (6.8).

## 5. TEST MATERIALS

### 5.1 Stone

Sandstone specimens were taken from stone blocks removed from the Capitol and cut to size by NBS. The mineralogy of the sandstone was determined by x-ray powder diffraction and was found to consist predominantly of quartz with veins of feldspar.

Specimens of Indiana limestone were taken from stone blocks procured for replacement of deteriorated sandstone.

### 5.2 Stone Treatments and Paints

Stone treatments were selected for investigation by the Architect of the Capitol and his consultants. Altogether, five materials were selected for evaluation (Table A1); one acrylic (A) and four silanes (S1, S2, S3 and S4). Treatments S2, S3, and S4 were all from the same supplier. Generally, treatments were applied to stone specimens by the suppliers, since any large scale application on the Capitol would be in accordance with a supplier's instructions. Suppliers were requested to use application methods that would give specimens representative of field treated stones. In the case of the silanes, NBS also prepared some treated stone specimens, following the application instructions of the suppliers.

Paints (Table A2) were those recommended by the suppliers of the stone treatments for their compatibility with the treatments. Each paint is identified by the same code as the treatment with which it is to be used. NBS was provided with samples of paints and performed tests to identify possible application problems. All paints were easily applied with a brush and no unacceptable flow or curing characteristics were observed. Paint from the acrylic treatment supplier was acrylic-based, and paints from the silane suppliers were latex.

## 6. TEST METHODS AND RESULTS

Tests used to investigate the performances and characteristics of stone treatments and paints are described in this section. If an ASTM (American Society for Testing and Materials) standard test method was available, it was selected over other tests to facilitate the testing process. Also, the use of a standard test helps to indicate testing conditions and procedures. Whenever possible, data were obtained using specimens prepared by suppliers of the stone-treatment and paint systems.

## 6.1 Water Absorption

6.1.1 Test Method. The water absorptions of treated and untreated stone specimens were measured using 2 in (51 mm) cubes following the methods of ASTM C 97 (7). Water absorption values were calculated as mass gained during water immersion relative to the dry mass of the specimens. Then ratios of water absorptions of treated specimens to untreated specimens were calculated and expressed as percentages.

6.1.2 Results and Discussion. Water absorption values for treated stone specimens and untreated stone specimens are given in Table A3. All treatments were effective in reducing water absorption of both sandstone and limestone specimens. In comparing treatments, the silanes (treatments S1, S2, S3, and S4) were more effective in reducing water absorption of the sandstone (reduction to 4.3 to 8.6 percent) than the acrylic (treatment A, reduction to 53 percent). With limestone, results with the acrylic (reduction to 11.9 percent) were in the same range as with the silanes (reduction to 18.4 percent). Low water absorption could be important if the absorption of water (e.g., from rainwater) results in the manifestation of a deterioration process, e.g., freezing and thawing damage.

## 6.2 Water Vapor Transmission

6.2.1 Test Method. Water vapor transmissions (WVT) of treated stone, with and without a paint film, and of untreated stone were measured following the methods of ASTM E 96 (8). Test specimens with a diameter of 3 in (76 mm) and thickness of 0.25 in (6.4 mm) were used. The tests were carried out in a closed cabinet at a relative humidity of 88 percent and temperature of 90°F (32°C) for 8 days. Values were calculated as the percentage of mass of water vapor passing through treated stone specimens into the desiccant with respect to the water vapor moving through untreated specimens.

6.2.2 Results and Discussion. Results of the WVT studies are presented in Table A4 for specimens with treatments and paint films. Some trends are apparent in the data: treatments reduced the WVT's of limestone more than the WVT's of sandstone; painted and treated sandstone specimens had higher WVT's than corresponding limestone specimens. In a few cases WVT's higher than 1.0 were obtained with treated sandstone which are considered to be statistically insignificant. Under certain conditions, a low WVT can be harmful, because it can result in the accumulation of water behind the surface of the treated stone. If significant amounts of moisture in the form of vapor is entering untreated stone through its outer surface, a low WVT could be desirable.

## 6.3 Sodium Sulfate Crystallization Test

Resistance of stone to internal stresses, such as those produced by salt crystallization and freezing and thawing of water, can be tested by following procedures of ASTM Method C 88 (9). The test consists of immersing stone in saturated solutions of sodium sulfate, followed by oven drying, and repeating the process until the stone fails. Oven drying results in precipitation and dehydration of the salt in pores. Internal expansive stresses are produced by rehydration of sodium sulfate during repeated immersions and precipitation during drying. If tensile strength of stone is exceeded by expansive stresses produced by precipitation and rehydration of sodium sulfate, stone begins to crack. The failure mechanism intrinsic to this test may not be the same as that actually occurring in the field, however a more appropriate ASTM standard test method has not been developed.

6.3.1 Test Method. The methods given in ASTM C 88 (9) were followed. Failure was defined as spalling occurring on all sides. Often spalling was accompanied by a sudden loss of mass. In some tests, however, gain in mass by precipitated sodium sulfate exceeded the mass loss by spalling. Therefore, a specimen could be badly spalled but have only a modest mass change; thus a visual failure criterion was used.

6.3.2 Results and Discussion. Results of the sodium sulfate test (Table A5) indicate that treated stone specimens had higher resistances to the effects of the precipitation and rehydration of the salt than untreated stone. Treatments S3 and S4 were the most effective in improving the resistance of stone to sodium sulfate attack. For example, resistance of sandstone was increased from 7 to 13 cycles when treated with S3 and S4, and the resistance of limestone was increased from 4 to 10 cycles.

## 6.4. Consolidation Ability

The consolidation abilities of treatments were evaluated by measuring the compressive strengths of treated sandstone and limestone specimens.

6.4.1 Test Method. The compressive strength of 2 in (51 mm) cubes of treated and untreated sandstone and limestone were measured in accordance with ASTM C 170 (10).

6.4.2 Results and Discussion. Results of testing specimens prepared by suppliers of the treatments (Table A6) indicate that the treatments had varying effects on the compressive strength of sandstone. Apparent decreases occurred with treatments S1, S2, and S4. There was essentially no change with treatment A, and an increase of 16 percent with treatment S3. All the treatments increased the compressive strength of limestone, ranging from 9 percent with treatment S1 to 27 percent with treatment S2.

Only one sandstone specimen with treatment S1 was furnished by the supplier for compressive strength determination. Because of its low compressive strength (4400 psi (30.3 MPa)), further tests were performed. Three additional sandstone specimens using treatment S1 were prepared by NBS investigators. Treatment was applied using a paint brush until the surfaces were saturated. Average compressive strength of these specimens was 5,660 psi (39.0 MPa). The reason for low compressive strength of the specimen prepared by the supplier is not known, but possibly could be caused by a defect in the stone itself or by improper curing of the treatment. In a further investigation of the effect of application method on strength development, three sandstone specimens were immersed in treatment S1 for 10 minutes, allowed to cure for 4 days at 22·C, and tested in compression. The average compressive strength for these specimens was 8050 psi (55.5 MPa). These results indicate that strengths of treated stones are dependent on the depths of penetration of the treatments.

## 6.5 Depth of Penetration

### 6.5.1 Laboratory Test

Depths of penetration of treatments in 2 in (51 mm) sandstone cubes, fractured in compressive strength determinations, were measured. These specimens had been prepared by the treatment suppliers. Depth of penetration was estimated by treating surfaces with water and visually observing where water ceased to form droplets and started to penetrate fractured surfaces. Also, concentrated sulfuric acid was applied and the darkening (usually slight) of surfaces, denoting the presence of organic materials, was observed. Both methods gave similar values. The depths of penetration of treatments in sandstone specimens prepared by suppliers are given in Table A7. Depths ranged from 0.3 in (8 mm) to 1 in (25 mm). Penetration depended upon the method of application. For example, when applied in thin films with a brush by the supplier, treatment S1 penetrated to a depth of around 0.3 in (8 mm). However, when sandstone specimens were immersed by NBS investigators, in the silane for approximately 10 minutes, it penetrated to the center of the cube. When the silanes S2, S3, and S4 were applied by dipping sandstone specimens in them for 5 minutes, they penetrated nearly to the center of the cube (0.8 in (20 mm)). These treatments also penetrated to the center of 2 in (51 mm) cubes when sandstone specimens were immersed for approximately 10 minutes. Treatment A, which was injected under pressure by the supplier using proprietary equipment, was found to be present in the center of 2 in (51 mm) cubes.

6.5.2 Field Test. Treatments A, S1, and S4 were applied to both sandstone and limestone blocks located in the West Central Front Wall of the US Capitol. Treatments A and S4 were applied by their suppliers. The supplier of treatments S2, S3 and S4 regarded the penetration ability of S4 to be representative of the abilities of treatments S2 and S3. This assumption appeared to be reasonable because the three treatments are based on the same silicic ethyl ester with only small amounts of different additives to give desired performances. Further, they all gave essentially the same depth of penetration in the laboratory study. Recent field studies by the Architect of the Capitol's staff confirmed that S3 has the same penetration ability as S4. Treatment S1 was applied by NBS at the request of the supplier, closely adhering to the supplier's application guidelines. After the treatments had cured for the time specified by the suppliers, cores of 2 in (51 mm) diameter and 4 in (100 mm) length were removed from treated sections by a contractor. Depths of penetrations were measured by visually observing the location where water droplets ceased to form on the specimen's surfaces.

Test results are given in Table A8. Depths of penetration measured within 48 hours of application ranged from 0.7 in (18 mm) to 3.5 in (89 mm) in sandstone, and from 0.6 in (15 mm) to 3.1 in (79 mm) in limestone. Another set of cores were taken 30 days after application. In the case of sandstone, the penetration depths after 30 days of curing were all 1.0 in (25 mm) or greater, and had increased between 20 to 80 percent compared to initial measurements. Similar increases in depths of penetration of limestone after 30 days of curing were less than 11 percent. Depth of penetration depended on the application method in the order: pressure injection > spraying > brush application.

### 6.6 Combination of Deterioration Processes

A test was developed to attempt to obtain evidence of detrimental effects of a combination of the following weathering processes: rapid temperature changes, wetting and drying, freezing and thawing, and aqueous dissolution. Independently, these processes may only cause slow rates of deterioration. When combined, the processes could have synergistic effects, thereby significantly increasing the rate of deterioration. Such an accelerated test with a single fixed cycle, however, would not permit isolation of the effects of individual processes.

6.6.1 Test Method. By using the apparatus illustrated in fig. 2, treated and untreated stone were exposed to accelerated weathering conditions and the combined effects of temperature cycles, wetting and drying, and freezing and thawing, on the performances of the treatments were investigated. Stone specimen blocks, with dimensions of 1.0 x 1.0 x 0.38 in (25 x 25 x 10mm) were subjected to cycles consisting of the following, in sequence: (1) heating at 104·F (40·C) for 15 minutes by a 1200 watt electric heater with a fan providing an air flow rating of 75 cu ft/min; (2) 3 minutes immersion in water at 72·F (22·C); (3) cooling at 4·F (-15·C) for 30 minutes; (4) drying by warm air as specimens leave the freezing chamber and enter the warm air chamber. A cycle required 70 minutes to complete, thus specimens were subjected to approximately 21 cycles in a 24-hour period. After testing, surfaces and interiors of specimens were examined visually and with an optical microscope at a magnification of 10 to determine if cracking had taken place or if there were any other sign of deterioration.

Conditions used in the accelerated test were selected based on analysis of weather conditions to which stone treatments would be subjected if applied to stone in the West Central Front. For example, the maximum temperature of 104·F (40·C) and minimum temperature of 4·F (-15·C) bracket the average monthly temperature in the Washington D.C. area, which ranges from around 30·F (-1·C) in January to around 80·F (27·C) in July (11).

Specimens were subjected to a total of 1660 test cycles which consisted of several runs of variable test cycles because specimens were added to the set of test specimens as they were received from their suppliers. The total number of cycles was established based on the need to complete the project within a specified time.

6.6.2 Results and Discussion. Treated and untreated sandstone and limestone specimens were subjected to 1660 cycles. No evidence of cracking deterioration was observed (Table A8). Some brownish surface staining on the surfaces of the specimens was observed.

Simulation of actual weather conditions by an accelerated test is difficult because short term fluctuations are often hidden in weather data. Also, the rate that conditions change is difficult to model. Correlation of 1660 cycles to actual weathering, therefore, can only be qualitative as follows. In an average winter in the Washington D.C. area, some 45 freeze-thaw cycles occur (11), thus 1660 cycles roughly corresponds to 37 "average years" relative to the number of freeze-thaw cycles. Maximum temperature exceeds 90·F (32·C) for an average of 30 days per year in the Washington D.C. area (11). Therefore, 1660 test cycles corresponds to 55 "average years" relative to the number of days per year exceeding 90·F (32·C). Such correlations, however, should not be taken to indicate an actual correspondence between test cycles and weathering conditions. Nevertheless, the test results do not suggest that the treated stone would be less durable than the untreated stone under temperature cycling or wetting and drying.

6.7 Adhesion between Paint and Stone

6.7.1 Test Method. The adhesion between paints and specimens of treated and untreated stone were measured following the method given in ASTM D 4541 (12). This method uses a pull-off adhesion tester. Briefly, a metal disk is secured to the coating surface with an epoxy. A testing apparatus is attached to the button and force applied perpendicular to the surface of the coating. The force is gradually increased until the coating material is detached or some other form of failure occurs. The test apparatus and method are described in more detail in Reference 13.

6.7.2 Results and Discussion. Test results are presented in Table A10. Data indicate that treatments can have a significant effect on the paint bond. For example, bond strength of paint applied over treatment S1 is substantially less than that applied to untreated stone. Also, the mode of failure is changed to an adhesive failure between the paint and treatment on treated stone from a cohesive failure of paint on the untreated stone. In the case of the paint applied over treatments S2, S3, and S4, the bond strengths were highest for the treated stones. Paint applied over treatment A exhibited failure in the paint film for both stone types.

6.8 Resistance of Paint to Moisture Condensation. Several accelerated test methods are available to study the effect of moisture on painted specimens. A major concern relative to the use of treatments on the Capitol is that moisture will accumulate at the interface between treated stone and paint resulting in loss of paint adhesion. The water vapor condensation test is a severe test (14) of the effect of moisture on the bond between paint and substrate and the tendency to blister.

6.8.1 Test Method. The apparatus and procedures described in ASTM Method D 2247 (15) were used. The apparatus provides water at a constant temperature as a source of vapor. Test panels are placed over the water vapor. The panels are at a lower temperature than the water resulting in condensation on the underside of the panels. Water temperature was set at 110·F (43·C). The panels were subjected to condensing moisture for 18 hours followed by drying for 6 hours to promote the formation of blisters.

Specimens were panels of nominal face size 6.75 in (171 mm) by 2.75 in (70 mm), and were 0.25 in (0.64 mm) thick. Paint films were applied to one face of each panel. Both painted and unpainted faces were exposed to water vapor. Triplicate specimens of each treatment-paint combination were tested for 28 days.

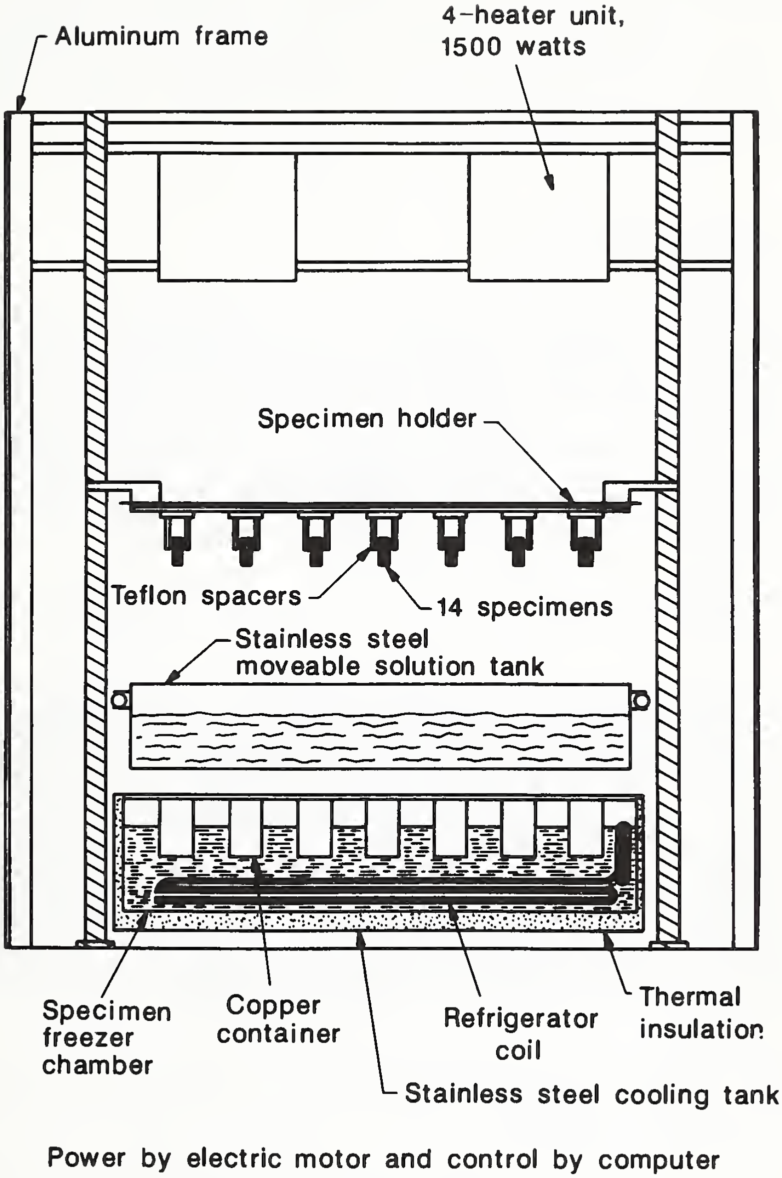


Figure 2. Accelerated Weathering Apparatus.

6.8.2 Test Results. Test results are given in Table A11. After 40 days of testing, no evidence of paint blistering was observed. Brown stains appeared on the outer sides of stone panels within the first week of testing. Similar stains were not observed with limestone specimens. The paint submitted by the supplier of stone treatment S1 became soft and could easily be removed with a spatula. There were no obvious changes in the hardness of the other paints.

# 7. PERFORMANCE CRITERIA

The selection of appropriate performance criteria for a stone-treatment and paint system should be based on an analysis of likely exposure and service conditions. Then these performance criteria will form a set of criteria which a treatment should be required to meet. The performance criteria proposed in this report are considered to be preliminary criteria as they have not been verified by long-term field studies.

## 7.1 Performance Criteria

Based on the analysis of sandstone deterioration given in Section 2, it is concluded that if a treatment is to be used on the West Central Front of the United States Capitol, the treatment or stone-treatment-and-paint system should protect the outer surface of the sandstone from penetration by rainwater. Since painting the sandstone has effectively protected it, a treatment should not cause premature paint failure (i.e., failure earlier than that occurring when paint is protecting untreated stone). Since all deteriorated stone may not been removed, a treatment should consolidate the sandstone by penetrating "to the depth of deterioration."

Performance requirements and criteria for stone treatment and paint systems considered for use on the West Central Front of the Capitol are given below.

7.1.1 Paint Adhesion. The adhesion between the treated stone and the paint should be adequate for sustained acceptable performance. In the absence of other information, initial adhesion should not be less than the adhesion between the paint and untreated stone. Otherwise premature failure of the paint may occur. If a cohesive failure occurs in the stone's surface, then strength of the treated stone is the limiting factor, not the strength of the paint-stone bond. Therefore, a cohesive failure is considered to indicate that the paint adhesion to treated stone is acceptable. Adhesion was measured as described in Section 6.5 and test results are given in Table A10.

7.1.2 Resistance of Paint to Moisture. Adhesion between the paint and the treated stone should not be degraded by moisture at a rate faster than the adhesion between paint and untreated stone. Effects of treatments on paint adhesion were evaluated by exposing for 40 days test specimens of painted stone to condensing moisture. To meet the requirement, the amount of visible loss of adhesion, such as blistering, occurring between paint and treated stone should not be more than that occurring between paint and untreated stone when the testing is carried out as described in Section 6.8. The test results are given in Table A11.

7.1.3 Consolidation of Weathered Stone. Stone treatment should consolidate surface deteriorated stone. Gauri recommended (17) that the compressive strength of treated weathered stone should be at least 10 percent above that of untreated and unweathered stone. Because of the subjectivity in deciding what constitutes weathered stone, a 10 percent increase in compressive strength of treated stone above that of untreated and unweathered stone was selected as the criterion. The results with sandstone are used in determining compliance with the criterion. The purpose of using a consolidant would be to treat weathered stone, and the nonweathered limestone was only recently put in place. The compressive strength of stone was measured as described in Section 6.5 and test results are given in Table A6.

7.1.4 Depth of Penetration. Stone treatment should be able to penetrate beyond the depth of deteriorated stone. Based on a visually observable maximum depth of deterioration, except in a few cases where exposure conditions were most severe, of 0.5 in (13 mm), a required depth of 1 in (25 mm) after 32 days was selected. This required depth of penetration should help to ensure that deteriorated stone is bonded to sound (unweathered) stone. Sandstone that had deteriorated to a depth greater than 1 in (25 mm) was replaced with Indiana limestone. The results of the field tests on sandstone (Section 6.5.2) were used in determining if treatments complied with the criterion. Test results are given in Table A8.

## 7.2 Compliance with the Preliminary Critical Performance Criteria

The extent of compliances of the treatments, applied to the sandstone, with preliminary critical performance criteria are given in Table 1. Treatments S2 and S3 were not included in the field test as their supplier elected to apply only S4. Results of the laboratory study on depth of penetration (see Table A7) indicate that the penetrating abilities of S2 and S3 are similar to that of S4. Therefore, S3 appears to meet the criterion for depth of penetration, in addition to the other criteria.

Table 1. Compliance of Treatments, When Applied to Sandstone, with Proposed Performance Criteria

| Proposed Performance Criteria | Treatment Designation[a] | | | | |
|---|---|---|---|---|---|
| | A | S1 | S2 | S3 | S4 |
| Paint Adhesion | F[b] | F | C[c] | C | C |
| Resistance of Paint to Moisture Exposure | C | C | C | C | C |
| Consolidation | F | F | F | C | F |
| Depth of Penetration (based on field testing | C | F | C[d] | C | C |
| after 32 days) | C | C | C[d] | C | C |

[a]Where appropriate, includes paint-treatment system.

[b]F indicates failure to comply with the criterion.

[c]C indicates compliance with the criterion.

[d]Indirect evidence of compliance as treatment not included in field test. However, laboratory results indicate that the depth of penetration of S2 is similar to S3 and S4.

# 8. SUMMARY AND RECOMMENDATIONS

## 8.1 Summary

The main purpose of this project was to develop a technical basis to aid in deciding if treatments should be used on the stone in the West Central Front of the U.S. Capitol. The approach taken to meet this need was to develop performance criteria for stone treatments based on analysis of the most probable processes responsible for the deterioration of sandstone in the West Central Front of the Capitol. The main processes responsible for the deterioration of the sandstone appear to be associated with exposure of the outer surface to precipitation. There is no evidence that moisture migration from the interior of the Capitol to the outer surfaces of the sandstone has caused water-related deterioration. Also, rising ground water leading to the deposition of soluble salts does not appear to have been a problem.

Four critical performance criteria were identified: 1) the effect of treatments on the performance of paint, 2) the ability of treatments to consolidate deteriorated sandstone, 3) the ability of treatments to penetrate and 4) the consolidation of the layers of weathered sandstone. The performance criteria are considered preliminary because they have not been verified by long-term field exposure testing. Therefore, compliance with the criteria does not unequivocally ensure that treatments will perform as intended. However, on the basis of current knowledge, it is expected that treatments that meet the criteria will perform satisfactorily.

## 8.2 Recommendations

In developing recommendations regarding the use of stone treatments, specific needs for them were analyzed as follows: Painting the sandstone has been an effective approach to reducing the extent of deterioration and for aesthetic reasons both sandstone and replacement limestone will be painted. Also, most of the severely deteriorated sandstone has been replaced with Indiana limestone. Therefore, it does not appear to be necessary to treat all the sandstone in the West Central Front. However, the use of consolidants could be of significant benefit if deteriorated layers are still present on some sandstone. Another reason to use a treatment would be to protect areas which are most likely to be subjected to severe weathering, such as cornices and tops of parapet walls. <u>Therefore, it is recommended that treatment S3 be used on the West Central Front on areas of deteriorated sandstone needing consolidation and on stone located in areas subjected to the most severe weathering.</u> No evidence was found that the application of stone treatments to newly placed limestone would be detrimental. Therefore the treatment could be applied to selected regions of the West Central Front or to the complete stone masonry.

## 9. ACKNOWLEDGEMENTS

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## APPENDIX A. Tables of Experimental Data

Table A1. Description of Stone Treatments

| Treatment Designation | Generic Type | Comments |
|---|---|---|
| A | Acrylic | prepolymer, dissolved in solvent |
| S1 | Silane | ethyl silicate[a], with hardener |
| S2 | Silane[b] | ethyl silicate[a], with hardener and water repellent. |
| S3 | Silane[b] | ethyl silicate[a], with water repellent |
| S4 | Silane[b] | ethyl silicate[a], with hardener |

[a]Reacts with water, in the presence of a catalyst, to form hydrated silica.

[b]Treatments S2, S3, and S4 were obtained from the same supplier.

Table A2. Description of Paints

| Paint Designation[a] | Generic Type | Comments |
| --- | --- | --- |
| A | Acrylic based | Applied by supplier, coverage not known. |
| S1 | Latex | Two coats recommended. Coverage, 200-275 $ft^2$/gal., first coat and 150-200 $ft^2$/gal. second coat. |
| S2, S3, and S4[b] | Latex | Two coats recommended. Solid content is 45%. Coverage, 125-150 $ft^2$/gal., first coat and 150-175 $ft^2$/gal. second coat. |

[a]Paints were obtained from suppliers of stone treatments and are identified by the same designation code as the treatments with which they are used.

[b]Stone treatments S2, S3, and S4 were obtained from the same supplier. The same paint was applied over each of these three silanes.

Table A3. Water Absorption of Treated Stone

| Treatment Designation[b] | Ratio of Water Absorption of Treated to Untreated Stone[a] | |
|---|---|---|
| | Sandstone, percent | Limestone, percent |
| A | 53 | 11.9 |
| S1 | 4.3 | 4.1 |
| S2 | 5.3 | 3.4 |
| S3 | 5.3 | 18.4 |
| S4 | 8.4 | 10.6 |

[a]Measured using ASTM C 97 (7)

[b]Treatments were applied by their respective suppliers.

Table A4. Water Vapor Transmission (WVT).

| Treatment Designation[a] | Ratio of WVT of Treated and/or Painted Stone to WVT of Untreated and Unpainted Stone | | | | |
|---|---|---|---|---|---|
| | Treated Stone | | Treated Stone with Paint | | Untreated Stone with Paint |
| | Sandstone | Limestone | Sandstone | Limestone | Sandstone[b] |
| A | 1.01 (0.14)[c] | 0.26 (0.07) | N.T.[b] | 0.26 (0.04) | N.T. |
| S1 | 1.07[d] (0.04) | 0.39 (0.01) | 1.07 (0.05) | 0.34 (0.07) | N.T. |
| S2 | 1.03 (0.01) | 0.68 (0.01) | 0.63 (0.20) | 0.35 (0.01) | 0.95[e] (0.07) |
| S3 | 1.0 (0.01) | 0.73 (0.01) | 0.40 (0.14) | 0.31 (0.07) | 0.95e (0.07) |
| S4 | 0.73 (0.01) | 0.57 (0.01) | 0.48 (0.13) | 0.34 (0.07) | 0.95e (0.07) |

[a]Specimens prepared by treatment supplier, except where noted otherwise.

[b]N.T. denotes not tested because specimen or material not available.

[c]Values in parenthesis are standard deviations for triplicate specimens.

[d]Specimens prepared by NBS by applying treatment and paint with brush. Coverage approximately 200 $ft^2$/gal.

[e]Same paint used with treatments S2, S3, and S4.

Table A5. Resistance to Sodium Sulfate[a]

| Treatment Designation[a] | Number of Cycles to Failure[bc] | |
| --- | --- | --- |
| | Sandstone | Limestone |
| A | 8 | 7 |
| S1 | 9 | 6 |
| S2 | 11 | 8 |
| S3 | 13 | 10 |
| S4 | 13 | 10 |
| Untreated | 7 | 4 |

[a]Treatments applied to stone specimens by respective suppliers.

[b]Data obtained by using ASTM Designation C 88 (9)

[c]Results are average of two specimens.

Table A6. Compressive Strengths of Treated and Untreated Stones

| Material Designation | Compressive Strength[a] | | | | | |
|---|---|---|---|---|---|---|
| | Sandstone | | | Limestone | | |
| c.v.b. % | psi | MPa | c.v.[b] % | psi | MPa | |
| A | 5720 | 39.4 | 5.1 | 5737[c] | 39.6 | |
| S1 | 4400[c] | 30.3 | | 5640[c] | 38.9 | |
| | 5660[d] | 39.0 | | | | |
| | 8050[e] | 55.5 | 10.1 | | | |
| S2 | 5150 | 35.2 | 8.0 | 5875 | 40.5 | 8.2 |
| S3 | 6700 | 46.2 | 1.4 | 6320 | 43.6 | 1.3 |
| S4 | 5540 | 38.2 | 0.5 | 6580 | 45.4 | 3.3 |
| untreated | 5790 | 39.8 | 10.7 | 5170 | 35.6 | 3.3 |

[a]If not specified, triplicate specimens (2 in (51 mm)) prepared by supplier of treatments were tested according to ASTM Designation C 170 (10).

[b]Coefficient of variation, in percent, designated by c.v. obtained by dividing the standard deviation for a set of data by the average of the set.

[c]Single specimen provided by treatment supplier.

[d]Triplicate specimens prepared by NBS using a paint brush. Treatment applied until surface was saturated.

[e]Triplicate specimens prepared by NBS by immersing specimens in treatment.

Table A7. Depth of Penetration of Treatments in Sandstone Specimens[a], Laboratory Testing

| Treatment Designation | Depth of Penetration[b] inch | mm | Method of Application |
|---|---|---|---|
| A | 1 | 25 | pressure injection |
| S1 | 0.3 | 8 | brush |
| S2 | 0.8 | 20 | dipping |
| S3 | 0.8 | 20 | dipping |
| S4 | 0.8 | 20 | dipping |

[a]Specimens were 2 in (51 mm) cubes. Treatments were applied by suppliers.

[b]Estimated error in measurement is 0.13 in (3.3 mm).

Table A8. Depth of Penetration of Treatments Applied in Field

| Treatment Designation | Sandstone inch | Sandstone mm | Limestone inch | Limestone mm | Method of Application |
|---|---|---|---|---|---|
| A | <1 week after application[b] | | | | |
| | 3.5 | 89 | 2.9 | 74 | pressure injection, |
| | (0.20)[a] | (5.1) | (0.13) | (3.3) | by supplier |
| | 32 days after apploication | | | | |
| | 4.4 | 112 | 3.3 | 84 | |
| | (0.3) | (7.6) | (0.04) | (0.1) | |
| S1 | <1 week after application | | | | |
| | 0.65 | 17 | 0.60 | 15 | two coat application |
| | (0.03) | (0.76) | (0.03) | (0.76) | brush, performed by NBS |
| | 32 days after application | | | | |
| | 1.0 | 25 | 0.62 | 16 | |
| | (0.09) | (2.3) | (0.09) | (2.3) | |
| S4 | <1 week after application | | | | |
| | 1.5 | 38 | 1.3 | 33 | sprayed, by |
| | (0.08) | (2.0) | (0.07) | (1.8) | supplier |
| | 32 days after application | | | | |
| | 2.2 | 56 | 1.4 | 36 | |
| | (0.2) | (5.0) | (0.05) | (1.3) | |

[a]Values in parenthesis are standard deviations of a set of triplicate measurements.

[b]Within one week after application.

Table A9. Results of Accelerated Weathering Test[a]

| Treatment Designation[b] | Condition after 1660 Cycles |
| --- | --- |
| A | Sandstone: No cracking, brown stains on surfaces.<br>Limestone: No cracking, no change in appearance. |
| S1 | Sandstone: No cracking, brown stains on surfaces.<br>Limestone: No cracking, no change in appearance. |
| S2, S3, & S4 | Sandstone: No cracking, brown stains on surfaces.<br>Limestone: No cracking, no change in appearance. |
| untreated stone | Sandstone: No cracking, brown stains on surfaces.<br>Limestone: No cracking, no change in appearance. |

[a]Duplicate specimens tested using apparatus described in Section 6.6. No painted specimens were tested.

[b]Treated stone prepared by supplier of treatment.

Table A10. Results of Adhesion Testing of Paints[a]

| Test Specimen | A | S1 | S2 | S3 | S4 |
|---|---|---|---|---|---|
| | Treatment and/or Paint Designation | | | | |
| Sandstone | | painted, untreated | | | |
| PSI | N.A.[c] | 288 | 619 | 619[d] | 619[d] |
| MPa | | 2.0 | 4.3 | 4.3 | 4.3 |
| C.V.[b] | | 4.5 | 16 | 16 | 16 |
| Failure | | P[e] | S[e] | S | S |
| Sandstone | | painted, treated | | | |
| PSI | 482 | 227 | 604 | 567 | 619 |
| MPa | 3.3 | 1.5 | 4.2 | 3.9 | 4.3 |
| C.V. | 40 | 37 | 6.3 | 4.4 | 3.9 |
| Failure | P | P | S | S | S |
| Limestone | | painted, untreated | | | |
| PSI | N.A. | 307 | 526 | 526[d] | 526[d] |
| MPa | | 2.1 | 3.6 | 3.6 | 3.6 |
| C.V. | | 16 | 0.8 | 0.8 | 0.8 |
| Failure | | P | S | S | S |
| Limestone | | painted, treated | | | |
| PSI | 475 | 228 | 507 | 528 | 537 |
| MPa | 3.3 | 1.5 | 3.5 | 3.6 | 3.7 |
| C.V. | 47 | 25 | 2.6 | 4.5 | 6.9 |
| Failure | P | P | S | S | S |

[a]Tested according to ASTM Designation D 4541.

[b]Coefficient of variation in percent. Triplicate specimens.

[c]Painted specimen or paint not submitted by supplier.

[d]Same paint as with treatment S2.

[e]P indicates failure in paint film, S indicates failure in stone.

Table A11. Results of Water Vapor Condensation Testing of Painted and Treated Stone[a]

| Paint-Treatment Designation | Condition of Paint after 40 Days of Testing |
|---|---|
| A[b] | No blistering of paint on either treated or untreated sandstone and limestone test panels. Brown stains on top (unexposed) surface of sandstone test panels. |
| S1[c] | No blistering of paint on either treated or untreated sandstone or limestone test panels. Brown stains on top (unexposed) surface of sandstone test panels. Paint had softened and was easily removed with a spatula. |
| S2, S3, & S4[d] | Same paint for S2, S3 and S4. No blistering of paint on any test panel. Brown stains on top surface of sandstone test panels. No visually observable deterioration of paint. |

[a]Tested using apparatus and procedures described in ASTM Designation D 2247.

[b]Test stone panels prepared by supplier of paint-treatment system. Tested in triplicate.

[c]Test stone panels prepared by NBS. Tested in triplicate. Paint coverage was approximately 200 $ft^2$/gal.

[d]Test stone panels prepared by NBS. Tested in duplicate. Paint coverage was approximately 150 $ft^2$/gal.

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11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

The West Central Front of the United States Capitol is being restored, including cleaning and repainting the sandstone, and replacing badly deteriorated sandstone with Indiana limestone. Application of stone treatments was proposed as a way of extending the lives of the sandstone and paint. However, without adequate selection criteria there was no certainty of effective performance by stone treatments. The purpose of this study was to develop performance criteria to assist in the selection of stone treatments.

Based on an analysis of expected deterioration processes, it is concluded that deterioration of the sandstone was most likely associated with water penentration into the exposed outer surface. Therefore, if a treatment is to be used, it should protect the outer surface of the sandstone from rain penetration. In addition, the treatment should penetrate and consolidate any deteriorated stone. Also, treatment should not cause premature failure of the paint. Based on these requirements, four preliminary performance criteria were developed, but have not been verified by long-term studies. Of five stone treatments evaluated, one met the four criteria. It is recommended that treatment be used on areas of deteriorated sandstone needing consolidation and on stone in areas subjected to the most severe weathering locations.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

consolidation, limestone, paint, performance criteria, restoration, sandstone, United States Capitol, weathering

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