

NBS TECHNICAL NOTE 941

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Stone Preservatives: Methods of Laboratory Testing and Preliminary Performance Criteria



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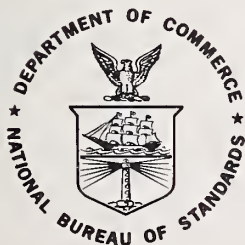
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Cover Photo: Pueblo Bonito, Chaco Canyon National Monument, New Mexico.

Stone Preservatives: Methods of Laboratory Testing and
Preliminary Performance Criteria

Gerald A. Sleater

Although numerous materials have been proposed as preservatives for stone in historic buildings and monuments, their efficacy is difficult to establish. In the work described here, a laboratory research program of accelerated simulated stone decay was used to obtain data on stone preservatives and to suggest criteria for their selection. Over 50 materials usable as stone preservatives were tested.

Tests to simulate stone decay were of two types:

1. exposure to combined weathering factors using a special test chamber for accelerated decay (CAD), in which chemical attack, salt and water action, and thermal effects were simulated in one operation;
2. exposure to single causes of stone decay using sulfurous acid fog, sodium chloride fog, water condensation/evaporation cycling, sodium sulfate penetration and crystallization, and ultraviolet radiation.

Methods for measuring the effects of the exposures are given together with the test data; these have been used to set limits of acceptable performance in preliminary performance criteria for the selection of stone preservatives. The behavior of each stone preservative tested in meeting these criteria is given. No one stone preservative met all criteria.

Key words: Accelerated laboratory testing; performance criteria; stone decay; stone preservatives.

1. INTRODUCTION

1.1 Stone Decay and Stone Preservation

Stone decay, the deterioration of stone from natural weathering or man-made environments is of increasing concern in the preservation of historic buildings and monuments throughout the world [1]. Although natural weathering processes will lead to the mineralogical re-adjustment of quarried stone to an atmospheric environment, the change is normally a slow process [2]. Whereas the increasing incidence of stone decay is often blamed on the industrial pollution of the 20th century [3], stone decay may occur where there is no pollution, and not occur where there is serious pollution [4]. One aspect of the problem of stone decay is, therefore, to determine the causes relevant to each specific case of decay and to measure and define the effects of this cause. International committees have been organized to carry out this latter task [5].

International committees are also active in a second aspect of the problem of stone decay - how to preserve stone [6, 7, 8]. One widely reported means of preserving stone has been through the use of stone preservatives [9]. Generally, stone preservatives are liquids, which when applied to the surface of the stone impart to the stone properties that will prevent or retard the decay. Stone preservatives have a long history of use [10], but information concerning the long-term effectiveness of these reported applications is lacking. Also, materials that have only recently been used as stone preservatives have not had time to prove their worth through long-term usage. Because of this lack of long-term performance data for stone preservatives, there should be discretion in their use.

The largest custodian of historic sites in the United States is the National Park Service, with over 300 national parks and monuments to administer. The use of stone preservatives in dealing with stone decay problems at historic sites in these parks and monuments is complicated by factors such as the different types of stone involved and their usage and condition, the diversity of climates and environments throughout the national park system, and the need to retain cultural or historic character of the site. It cannot be assumed that a stone preservative effective at one site will prove equally effective at another site where there is a different environment, type of stone, or degree of stone decay. For these reasons, the National Park Service has been reluctant to use stone preservatives without a solid base from which to judge their effectiveness.

To obtain such a base of knowledge, the National Park Service requested the National Bureau of Standards to investigate stone preservatives and develop methods that could be used to select stone preservatives for field use.

1.2 Program Objective

One way of meeting the needs of the National Park Service for stone preservatives would be through the development of performance criteria for their selection. Performance criteria have been applied in many areas of the building industry [11]; however, their application to stone preservatives would be new. The advantages of applying performance criteria to stone preservatives are that they will both describe what the stone preservative is to accomplish and will define how that accomplishment may be measured. Any material that meets the appropriate performance criteria can then be tried in the field to find out if it fulfills the need for a stone preservative at the local site.

For these reasons, the development of preliminary performance criteria for the selection of stone preservatives became the objective of the research program.

The program was carried out in three phases:

Phase 1. A literature survey of stone decay, stone testing, stone preservation, and the use of stone preservatives was done. Information from the survey was used in developing the testing phase of the program and in writing a state of the art report on stone preservation [12].

Phase 2. Practical knowledge of stone decay problems and stone preservation efforts was gained through on-site inspection of national monuments. The sites visited included the Lincoln and Jefferson Memorials, and Washington Monument in Washington, D.C., Federal Hall and Castle Clinton in New York, Chaco Canyon National Monument in New Mexico, Castillo de San Marcos in Florida, and the El Moro National Monument in Puerto Rico.

Phase 3. The major effort in the program was in the development and the application of methods for testing stone preservatives. Information gained through the first two phases gave a basis for selecting performance requirements for stone preservatives, and helped identify the tests needed for laboratory evaluation of preservation performance. The data obtained using the tests became the basis for the preliminary performance criteria for preservative selection.

2. DEVELOPMENT OF THE TESTING PROGRAM FOR STONE PRESERVATIVES

The laboratory testing program for stone preservatives was developed in five stages:

- (1) The identification of the causes of stone decay to be simulated in testing (Section 2.1).
- (2) The selection of laboratory procedures to simulate the relevant causes of stone decay and test methods for evaluating the effects of testing on preservative-treated stone (Section 2.2).
- (3) Selection of stone to be used as the substrate material in the testing of stone preservatives (Section 2.3).
- (4) Selection of representative stone preservatives for testing (Section 2.4).
- (5) Testing of treated stone (Sections 3 and 4).

2.1 Identification of Causes of Stone Decay

Stone decay in a structure can be classified as being either intrinsic or extrinsic in origin [13]. The intrinsic causes come from the nature and the use of the structures. They include causes, such as structural failures and improper usage of materials, the effects of which stone preservatives cannot prevent or protect against. Extrinsic causes include the climatic and environmental factors of natural weathering against which stone preservatives are intended to provide protection.

The following four extrinsic causes of stone decay were chosen for simulation in the testing program, as they are the most important causes of stone decay [14, 15].

1. Water Action. Water is necessary both for chemical degradation and the salt action degradation of stone. Water alone can cause stone decay through its expansion in the pores of the stone upon freezing, or through its dissolution or hydrolysis of mineral constituents of stone [16].

2. Chemical Attack. Chemical attack includes attack by any agent that reacts chemically with stone. The most important agents are the normal constituents of the atmosphere and air pollutants [17]. The normal constituents usually react slowly, as carbon dioxide does with carbonate-containing stone. On the other hand, air pollutants, such as sulfur dioxide and the oxides of nitrogen - both forming strong acids with water, are more destructive to stone. These acids dissolve the stone and form salt deposits that cause further stone decay through salt action.

3. Salt Action. The destructive action of salts upon building stone occurs in two ways. The first way is from insoluble salt deposits forming hard crusts on the surface of stone. If these deposits spall, they remove the surface of the stone to which they adhere and expose a fresh stone surface to weathering [18]. The second way for destructive salt action is through crystallization of salts in the pores beneath the surface of the stone. As these salt deposits build up, or as the salts crystallize in different forms, forces may develop within the stone which may eventually cause spalling to occur [19]. Salts may be the residue of chemical attack on the stone or on other building materials. Salts may also be transported into the structure by groundwater, or they may be carried by rain or wind.

4. Thermal Effects. Diurnal temperature variations cause expansion and contraction of stone, and can lead to differential expansion of mineral components or salts in the stone [20]. Below-freezing temperatures are necessary for freeze/thaw damage to water-saturated stone.

2.2 Laboratory Test Procedures: Simulation of Stone Decay and Evaluation of Test Results

Two approaches were used to simulate these causes of stone decay in laboratory testing:

(1) Combined Testing: Simulating several causes of stone decay in one test operation. This approach is described in Section 3.

(2) Single Tests: Simulating a single cause of stone or stone preservative decay in one testing procedure. This approach is described in Section 4.

2.3 Stone Substrate for Test Specimens

Indiana limestone (Standard Gray) was chosen as the substrate for the testing of stone preservatives. It is widely used as a building stone throughout the United States [21]. Its properties fitted the needs of the test program: it was susceptible to, but not rapidly destroyed by acid attack in the CAD (described in Section 3.1); the effects of CAD testing upon its properties, such as water absorption and hardness, were measurable; stone preservatives could penetrate into the stone.

Test specimens were cut at NBS from unweathered limestone purchased from a commercial quarry. After cutting, the specimens were washed several days with running tap water to remove stone dust, then dried at room temperature (22-25°C) under a vacuum of 5-10 mm of mercury for 8-16 hours. The dry specimens were then used for testing or stone preservative treatment.

2.4 Stone Preservative Materials

The materials tested for stone preservation treatments were obtained from commercial suppliers. Although most were primarily intended for treatment of stone, concrete, or masonry to produce water repellency, "waterproofing," or surface hardening, such materials may also be useful as stone preservatives and they are all described as stone preservatives in this report. Each stone preservative was given a two digit code number to identify it. Uncoated limestone specimens were coded 00.

Several materials, such as saturated calcium hydroxide solution (limewater) and linseed oil, that have long been used as stone preservatives [22] were also included in the study. They were bought from chemical supply houses.

Table 1 lists some important properties of these materials. The qualitative composition of a stone preservative was supplied by the manufacturer or determined, when possible, from its infrared spectrum. Nonvolatile content was the residue obtained by evaporating to dryness a weighed amount of the material at 105°C. The color of a preservative refers to its appearance in liquid form. "Waterwhite" describes a liquid which has the colorless, transparent appearance of water. Miscibility and reaction with water were determined by mixing equal proportions of stone preservative and water; after mixing, the pH of the water phase was determined with pH-indicating paper (only pH values above pH 7 or below pH 5 are recorded in table 1). The color and general physical characteristics of the dried film are those of the film or the deposit obtained by evaporation of the liquid preservative at room temperature.

The appearance and the surface texture of the Indiana Gray limestone, as determined by visual observation, and the percent weight increase in the specimens after application of the preservative treatment (Section 2.4.1) are given in table 2. Figure 1 illustrates the differences in appearance among preservative-treated specimens of Indiana gray limestone.

The depth of penetration of the stone preservatives into unweathered limestone (table 2) was determined by sawing the treated specimens in half. How deep the penetration of a preservative into unweathered limestone could be is illustrated in figure 2.

2.4.1 Application of Stone Preservative Material

Most suppliers of stone preservative materials recommended their application by brush or spray. In this program, the dry limestone specimens (Section 2.3) were treated only once, the preservative solution being brushed on all sides of a specimen (in a laboratory exhaust hood) until it was no longer absorbed by the stone. After air-drying in the hood, specimens were further dried at room temperature (22-25°C) under a vacuum of 5-10 mm of mercury for 8-16 hours.

Vacuum drying was used after most testing procedures to avoid possible damage to the preservative treatments by drying at oven temperatures (100°C). An exception were the sodium sulfate tests (Sections 4.2.2 and 4.2.3) in which a temperature of 105°C was needed to obtain anhydrous sodium sulfate.

3. EXPOSURE TO COMBINED WEATHERING FACTORS

By combining in one testing operation the major causes of stone decay -- water and salt action, chemical attack, thermal effects -- an attempt was made to simulate the synergistic action of stone decay factors sometimes found in nature, and as in nature, without being able to assign to each cause its contribution to the decay process.

The combined exposure testing was done using a special test Chamber for Accelerated Decay (CAD) and test cycle developed by the Illinois Institute of Technology Research Institute (IITRI).

3.1 Chamber for Accelerated Decay (CAD)

The CAD, built of stainless steel by IITRI, is illustrated in figure 3. Its features include: A) three infrared and six ultraviolet lamps; B) a water spray; C) a revolving eight-sided specimen drum; D) each face of the drum can hold up to 15 test specimens (each 10 cm x 10 cm x 2.5 cm) on removable plates; E) a motor and reduction gearbox for rotation of the specimen drum; F) an inlet for corrosive test solutions (60 liters of solution will fill the chamber to the bottom face of the specimen drum); G) an outlet through which the corrosive test solution can be pumped back to storage for reuse; H) a drain that can be completely closed by a valve when the corrosive test solution is being used, or only partially closed by a stand pipe during water-soaking of the specimens.

To simplify testing, only one size limestone specimen (10 cm x 10 cm x 2.5 cm) was used in CAD testing. Test specimens of other shapes and sizes (round sections cut from cores, for instance) could be used in the CAD by changing the mounting fixtures.

The CAD test cycle (table 3) combines chemical attack, salt and water action, and thermal effects in one operation. Freeze/thaw action was obtained by removing the plates holding the specimens from the CAD after they had been soaked in the sodium chloride/sulfurous acid solution and storing them overnight in a deep freezer at -40°C. As many of the stone preservatives contained organic resins, which might be degraded by solar ultraviolet radiation [23], ultraviolet lamps were used to simulate this radiation in the CAD.

Table 4 is a matrix showing the contribution of each feature of the CAD to the different causes of stone decay being simulated.

3.2 Evaluation of Specimens after Exposure in the CAD

Both preservative treated and untreated stone specimens were exposed in the CAD. Stone specimens were evaluated after periods of 10 and 20 CAD test cycles. Twenty test cycles were usually sufficient to prove lack of durability of the stone preservatives; evaluation of specimens after 10 cycles helped to detect the onset of degradation and trends in specimen behavior. Separate test specimens were used for each CAD test cycle of a stone preservative. (In some test runs duplicate specimens treated with the same preservative were used to test reproducibility of the evaluation measurements. This reproducibility was good.) After completion of the chosen number of test cycles, specimens were removed from the plates on which they had been mounted for CAD testing and were washed for 8-16 hours in running tap water. The specimens were then dried as described in Section 2.3.

The effects of stone decay are shown through surface degradation, internal alterations (visible, invisible), freezing breakage, and stains [24]. The effectiveness of a stone preservative is defined by how well it prevents or modifies these conditions. Measurement of certain surface or bulk properties of stone can be used to determine this effectiveness.

The effectiveness of stone preservatives in modifying the effects of CAD exposures on the limestone substrate was evaluated by measuring changes in six properties of the test specimens:

(1) Weight. The most serious effect of stone decay is the material breakdown of the stone. The breakdown of the stone and the consequent loss of stone particles can be measured by the weight changes caused by CAD testing. These changes are discussed in Section 3.2.1.

(2) Appearance. In natural weathering small changes in the appearance of stone are accepted, even wanted. Grosser changes due to surface grime or salt deposits can degrade the stone as well as be unsightly [25]. In CAD testing, changes in appearance were determined both by visual examination and by instrumentally measuring the changes in color and gloss. The results of these measurements are given in Section 3.2.2.

(3) Water Absorption. The passage of liquid water through stone can be an important factor in stone decay (Section 2.1). How well stone preservatives prevent the passage of liquid water through stone can be determined by measuring the water absorption of the treated stone. The water absorption values and their changes resulting from CAD testing are described in Section 3.2.3.

(4) Water Vapor Permeability. The passage of water vapor through stone and its evaporation from the surface is one way in which liquid water escapes from within a stone. If this "breathing" is prevented by stone preservatives or deposits that seal the surface, liquid water trapped within the stone can lead to enhancement of the problems from water action (Section 2.2). Water vapor permeability measurements are discussed in Section 3.2.4.

(5) Abrasion Resistance. Resistance to mechanical wear is one indication of the cohesion of the surface particles of stone; loss of surface particles ("sugaring") is frequently observed in stone degradation. How well a stone preservative hardens or toughens the surface of the stone to prevent "sugaring" can be measured by the abrasion resistance of the treated stone. Abrasion resistance measurements are described in Section 3.2.5.

(6) Surface Hardness. Measurement of the surface hardness (resistance to deformation) is a relative indication of the strengthening of stone by a preservative treatment. Surface hardness is covered in Section 3.2.6.

3.2.1 Weight Changes

After drying (Section 3.2) specimens were weighed to 0.1 milligram. Any change in specimen weight is expressed as weight percent. Weight changes from CAD testing are listed in table 2.

Loss of weight indicates how much of a test specimen (stone and/or stone preservative) has been lost through CAD testing. All test specimens lost some weight during CAD testing except those treated with preservative 51 -- these gained weight. Untreated limestone specimens lost approximately 1% of their initial weight after 10 cycles of CAD testing, and 2% after 20 cycles. Most treated specimens had less weight loss than the unprotected limestone specimens, although specimens treated with preservatives 4, 6, 10, 13, 49, and 50 lost slightly more weight.

3.2.2 Change in Appearance*

The effect of CAD testing upon the appearance of the test specimens was measured using ASTM D 2244-68 (27)**, Test for Instrumental Evaluation of Color Differences of Opaque Materials; and ASTM D 523-67 (27), Test for Specular Gloss.

The initial effects of the different stone preservatives treatments on Indiana limestone are shown in figure 1. CAD testing can lead to further changes in specimen appearance, varying from slight changes (figure 4) to extreme differences in color (figure 5). Figure 6 shows changes that came about as a preservative treatment was lost -- the residue adhering to the stone being one color, the body of the stone another color. Figure 7 is a close-up of the specimen in figure 6 after exposure to 20 CAD cycles.

Color values for the tested specimens were calculated from readings made with a three filter colorimeter (ASTM D 2244-68, Section 3.8.1.2). The color difference (E) between the tested specimens and untested specimens is given in figure 8 in bar graph form. Gloss measurements were made for stone preservatives that imparted a gloss to the specimen. However, all specimen gloss was destroyed during CAD testing, and no measurement of gloss could be made after CAD testing began.

* Presently of great interest to the appearance of stone is the problem of graffiti. Two recent NBS publications that cover parts of the graffiti problem are listed in reference 26.

** The numbers in parenthesis following the ASTM Test Methods indicate what volume of ASTM Test Methods (1975 edition) the test method can be found in. ASTM Standards may be obtained by applying to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

Although the color difference E is useful for determining whether or not a specimen's appearance is within specified limits, a visual estimation of color difference is also recommended. For example, the specimens shown in figure 6 had color changes less than untreated limestone (7 units) after CAD testing, yet these specimens appear degraded. Changes in appearance must, therefore, be judged visually, and if possible, determined instrumentally.

3.2.3 Water Absorption*

The absorption of liquid water by stone specimens was measured by following the procedure of ASTM C 97-47 (19), Standard Methods of Test for Absorption and Bulk Specific Gravity of Natural Building Stone.

Water absorption was measured by immersing the test specimens completely in a stirred distilled water bath for 48 hours at 22-24°C as called for in the ASTM Test Method. The specimens were then removed from the water and quickly weighed, after wiping off the surface water on the specimens with a damp cloth. The results of the water absorption measurements are given in bar graph form in figure 9.

The effects of CAD testing on the water absorption of preservative-treated specimens can be classified in three categories: 1) little change, for those treated specimens which had a water absorption before testing approximately that of untreated limestone; 2) a great increase in water absorption after testing - the expected effect as the stone preservative treatment is lost; 3) fluctuations -- the water absorption values increasing then decreasing, or vice versa -- which may be due to blockage of the pores of the stone by salts or other materials.

The water absorption of untreated Indiana limestone was 6% before and after CAD testing. Water absorption remained below 6% for 20 CAD cycles for specimens treated with 24 of the 53 stone preservatives.

3.2.4 Water Vapor Permeability

The permeability of the test specimens to water vapor was measured using ASTM Test Method C 355-64 (18), Standard Methods of Test for Water Vapor Transmission of Thick Materials.

* Performance tests specifically for waterproofing materials are given in reference 27.

Cups (figure 10) to hold the square test specimens were fabricated from 1 millimeter thick aluminum sheet. The insides of the cups were protected against corrosion by the desiccant and made airtight by a coating of paraffin wax. To measure water vapor permeability calcium chloride desiccant was added to the cups and the test specimens taped to the cups with adhesive aluminum tape. The cups and attached specimens were kept in a constant humidity room ($50\% \pm 4\%$ RH and $23 \pm 1^\circ\text{C}$), and periodically weighed until the rate of water pickup by the desiccant was constant. The rate of weight increase measured the rate at which water vapor passed through the test specimen.

The Water Vapor Transmission (WVT), Permeance, and Average Water Vapor Permeability (WVP) of the specimens were calculated from the test data.

WVT = weight change in grams/test area (square meters)

Permeance = $\text{WVT}/S(R_1 - R_2)$

S = saturation vapor pressure at test temperature

R_1 = relative humidity in constant temperature room

R_2 = relative humidity inside cup

Average Permeability* = permeance x specimen thickness (cm).

Because specimen thickness varied between 2.30 cm and 2.50 cm, the test results were expressed as water vapor permeability (WVP). The WVP values are given in bar graph form in figure 11.

Water vapor permeability measurements show that both tested and untested specimens are permeable to water vapor. WVP changes caused by testing are similar to the changes observed in liquid water absorption (Section 3.3.3), the same preservative treatments often exhibiting the same trends. The type of variation in WVP during CAD testing were similar to the variations in water absorption: 1) little change between the tested and untested specimen WVP values; 2) an increase of WVP with testing; 3) fluctuations. The water vapor permeability of the untreated limestone decreased 25% during 20 CAD cycles -- from 3.3 metric perm-cm to 2.5 metric perm-cm. Specimens for stone preservative treatments 05, 09, 38, 55 remained at or decreased from this level during CAD testing.

* To convert from permeability in metric perms units to permeability in SI units (International System of Units), the following factors are used:

metric perm-cm (multiplied by 0.598) = perm-in (23°C)

perm-in @ 23°C (multiplied by $1.46 \text{ E-}12$) = kilogram/pascal-second-metre.

3.2.5 Abrasion Resistance

The resistance of test specimens to surface abrasion was measured using the equipment and test procedure described in ASTM D 1044-56 (35), Test for Resistance of Transparent Plastics to Surface Abrasion.

The abrasion testing machine (figure 12) was modified to hold the square test specimens by attaching four adjustable sides to the rotating stage of the machine. In testing for abrasion resistance the specimen (C) was firmly clamped to the rotating stage by the adjustable sides (D); two separate grinding wheels (B), each with a test load of 1 kilogram (A), were placed on the surface of the specimen and the stage rotated. Each rotation cycle was recorded by a counter. As the specimen rotated beneath them, the weighted grinding wheels abraded its surface. The weight loss (in milligrams, mg) of the specimen per rotation was used to measure the resistance of the specimen -- and preservative treatment -- to abrasion. The results of the abrasion resistance measurements are given in figure 13.

As with other properties of test specimens measured after CAD exposure, abrasion resistance increased, decreased, or fluctuated with CAD testing. Untreated limestone specimens increased in abrasion resistance (i.e. a decrease in surface matter abraded) with CAD testing; this increase may be due to the loss of loose surface granules during testing, or to the cementing action of residues or chemical reaction on the surface.

Many specimens treated with stone preservatives that did not penetrate deeply into the stone (see table 2) had an initial high abrasion resistance, but as the surface coating was lost with testing there was a sudden decrease in protection -- this behavior was shown by specimens treated with preservatives 24, 30, 34, 49. Some stone preservatives that penetrated deeply into the stone (09, 12, 18, 25) retained a relatively high abrasion resistance throughout CAD testing.

(In this project unweathered stone was used as test substrate. In testing where weathered stone or stone with a low abrasion resistance is used, the strengthening effect of stone preservatives on the stone should be much greater and more easily determinable by abrasion resistance measurements.)

3.2.6 Surface Hardness

Surface hardness was measured using the method and equipment described in ASTM E 18-67 (10), test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials.

Measurements were made with ROCKWELL-WILSON Hardness Tester*, Model 3-JR. Rockwell hardness values were read on the C-scale of the Tester dial gauge, using a steel ball surface penetrator (1.2 cm diameter) and a major test load of 60 kgf. The accuracy of the dial gauge was checked by measuring the surface hardness of a standard steel block.

In measuring surface hardness, one square face of the specimen was placed on the anvil of the hardness tester, and the penetrator pressed into the opposite face by a minor test load of 10 kgf. The pointer of the dial gauge was then set at zero, and the major load applied. The Rockwell hardness value is the depth of penetration of the penetrator caused by the major load. The measured Rockwell hardness values are given in bar graph form in figure 14.

The surfaces of all untreated and some treated limestone specimens did not correspond to the smooth, polished metal surfaces for which the hardness test is intended. This surface roughness was roughly compensated for by using a large diameter penetrator. The surface hardness of specimens for preservative 51 could not be read, because the penetrator continued to sink into the specimen and did not come to rest under the minor load.

During CAD testing, specimens treated with stone preservatives 09, 21, 35, 45 decreased in surface hardness 20% or more from the surface hardness of the untested limestone. The surface hardness of the other preservative treatments showed only slight variation consequent to testing.

3.3 Summary of CAD Testing

To summarize the usefulness of CAD testing in regard to studying stone preservatives:

- (1) CAD testing caused measurable deterioration of stone untreated with stone preservatives within the reasonable time of 20 CAD test cycles. This deterioration could be measured through changes in certain properties of the test specimens -- such as weight, appearance, water absorption, water vapor permeability, abrasion resistance, and surface hardness.
- (2) CAD testing caused, to varying degrees, changes in the same properties for preservative-treated stone specimens. The effectiveness of the preservative treatment on a specimen could be judged by how well it prevented changes in these properties equivalent to those for untreated stone.

* Description of a specific test instrument is to assist duplication of the testing procedure; it does not imply endorsement by the National Bureau of Standards.

(3) From evaluation of the effects of CAD testing of over 50 stone preservatives, appropriate upper limits of the property changes expected from the more effective preservatives can be identified. These limits are used in Section 5 to establish preliminary performance criteria for selection of stone preservatives for field testing. (How well the stone preservatives tested in Section 3 meet these criteria is indicated in table 12.)

4. INDIVIDUAL TESTS

In circumstances where it is determined that one cause is the major influence in stone decay, selection of a stone preservative can be based upon simulated testing involving that cause alone. In this project the causes of stone decay and stone preservative deterioration that were simulated individually were:

Chemical Attack: Sulfurous Acid Fog Test (4.1)

Salt Action: Sodium Sulfate Crystallization Test (4.2.1)

Sodium Sulfate Penetration Test (4.2.2)

Sodium Chloride Fog Test (4.2.3)

Water Action: Water Condensation/Evaporation Test (4.3)

Ultraviolet Radiation Degradation of Preservatives: Ultraviolet Radiation Test (4.4).

4.1 Sulfurous Acid Fog

The effect of a sulfurous acid environment on stone preservative treatments was evaluated by exposing test specimens to a sulfurous acid fog.

This test used the corrosion test chamber (Kesternich Cabinet) and the test procedure described in the German Industry Standard DIN 50 018, Testing of Materials, Structural Components and Equipment; Method of Test in Damp Heat Alternating Atmosphere Containing Sulfur Dioxide (Cabinet according to Kesternich) [28].

The test cabinet and its accessories are illustrated in figure 15. For each test cycle two liters of distilled water were poured into the test cabinet (A) to cover the lead-lined bottom. Two liters of sulfur dioxide gas from the gas cylinder (B), measured by the container (C), were passed into the closed cabinet through the gas inlet valve (D). Only then were the electrical heating elements in the bottom of the cabinet turned on. Within 90 minutes the temperature in the lower part of the cabinet reached 35°C, resulting in a fog of sulfurous acid that filled the cabinet and covered the test specimens. After 8 hours the heating was stopped and the cabinet cooled overnight (16 hours) with the door ajar. The water remaining in the cabinet was drained and replaced with fresh distilled water before starting a new test cycle.

Single test specimens (2 cm x 2 cm x 1 cm) were treated on five sides with each stone preservative; one large face of each specimen was left untreated in order to observe the effect of the test on untreated as well as treated stone. The specimens were placed on neoprene mesh midway between the top and bottom of the cabinet; they were not washed or otherwise cleaned between test cycles.

After 49 test cycles (8 hours heating and 16 hours cooling) the specimens were removed from the cabinet, washed three times with distilled water, and dried under vacuum (Section 2.3). The changes observed in the tested specimens are given in table 5. Figure 16 illustrates the changes in appearance consequent to this test. Figure 17 is a close-up of three specimens. Most specimens were affected to some degree by the sulfurous acid atmosphere. Although the surfaces of some specimens had blistered or changed color, the most common change was a visible white surface deposit of small (less than 0.1 mm) acicular crystals, presumably calcium sulfite or sulfate, too small for analysis. There was no great difference between the treated and untreated surfaces of specimens affected by testing.

The concentrations of sulfurous acid used in the sulfurous acid fog test or in the CAD weathering cycle did not physically destroy the limestone substrate material. The effect of concentrated sulfurous acid flowing over Indiana gray limestone is shown in figure 18; no similar destruction of the stone was observed in either the CAD or Kesternich tests.

The 60 liters of 0.01 molar sulfurous acid solution used in the CAD test had a concentration of approximately 800 parts per million (ppm) sulfur dioxide. Two liters of sulfur dioxide gas added to the Kesternich test cabinet (volume 300 liters) gave a fog with a sulfurous acid concentration of 15,000 ppm sulfur dioxide. Both concentrations are higher than the pollution levels of sulfur dioxide normally found in urban environments. For example, between 1954-64 the annual mean concentration of sulfur dioxide in London, Great Britain, was 0.12 ppm [29]. In the fog test the level of sulfur dioxide was over 100,000 times greater, and in the CAD test over 6,000 times greater.

4.2 Salt Action

Three types of salt action (see Section 2.1) on stone or stone preservatives were simulated: 1) crystallization of salt within stone (Sodium Sulfate Crystallization, 4.2.1); 2) penetration of salt solution through a preservative treatment (Sodium Sulfate Penetration, 4.2.2); 3) penetration through, or degradation of a preservative treatment by air-borne salt solution (Salt Spray Test, 4.2.3).

4.2.1 Sodium Sulfate Crystallization

The resistance of preservative treated specimens to the effects of salt crystallization was evaluated by cyclic sodium sulfate crystallization tests.

Testing followed ASTM C 88-69 (15), Standard Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate. Test specimens were limestone prisms (10 cm x 2.5 cm x 2.5 cm); single prisms were treated once on all sides with each stone preservative, which was brushed on until no more was absorbed by the stone.

Specimens were tested up to 10 cycles. Each test cycle consisted of immersing the test specimen for 16 to 18 hours in a stirred, saturated sodium sulfate solution kept at 22-24°C, removing the specimens from the solution and drying them at 105°C for four hours. The dried specimens were weighed, then re-immersed in the sodium sulfate solution.

During each test cycle, the sodium sulfate solution absorbed by the specimen is dehydrated by heating to 105°C. Upon re-immersion hydrated sodium sulfate is formed within the specimen, and the greater crystal volume of the hydrated salt exerts pressure on the confining stone. Untreated limestone prisms began to spall after two test cycles.

The cumulative specimen weight changes from cyclic sodium sulfate crystallization are given in table 6. Figure 19 illustrates how specimens increase in weight as sodium sulfate is absorbed, then begin to deteriorate and lose weight. The degradation of the test specimens by sodium sulfate crystallization is illustrated in figure 20.

Only specimens treated with 10 stone preservatives showed little or no weight loss after 10 test cycles; each of these preservatives penetrated 5 millimeters or more into Indiana limestone (see table 2).

4.2.2 Sodium Sulfate Penetration

The resistance of stone preservative treatments to the penetration of salt solutions was evaluated through modification of the Federal Specification for Water-Repellent, Colorless, Silicone Resin Base, SS-W-110C [30]. Section 4.3.5 of this Specification tests the resistance of specimens to efflorescence by immersing an unprotected surface of the test specimen in 10% sodium sulfate solution for 7 days. The specimen is then removed from the solution and dried, and any efflorescence on the treated surface opposite to the immersed, untreated surface noted.

To test for penetration of a salt through a stone preservative treatment, and to discover the effect drying or absorbed salts have on stone preservative treatments, the above procedure was reversed. Single test specimens (5 cm x 2.5 cm x 2.5 cm), treated on five sides for each stone preservative tested, were placed in a 10% sodium sulfate solution at 22-24°C with the one untreated surface uppermost; the bottom treated surface was just wetted by the solution. After seven days the specimens were removed from the solution, dried overnight (16 hours) at 105°C. After the specimens were dried, the untreated surfaces were inspected, and the specimen weighed. Specimens were tested for three cycles of seven days each.

The condition of the dry test specimens after three test cycles is given in table 7. The top, uncoated surface was degraded either through swelling and loosening of the stone granules, or by the appearance of dried white salt deposit (efflorescence). Degradation of the specimen's sides, which were treated with stone preservative, was through spalling of the treated zone from the body of the specimen. No change in appearance or physical state was found for test specimens of 20 of the 51 stone preservatives tested. There was no correlation between the weight of sodium sulfate absorbed by a test specimen (usually no more than a 1% increase in specimen weight) and degradation of the specimen. The destructive action of salt penetration on the stone is shown in figure 21.

4.2.3 Salt Spray Test

Stone preservative treated specimens were exposed to a salt (sodium chloride) spray environment by following the procedure of ASTM B-117-64 (9), Standard Method of Salt Spray (Fog) Testing.

Treated specimens were exposed to a sodium chloride atmosphere in order to determine what effect sodium chloride would have on the preservative treatments, and whether sodium chloride would be absorbed by the stone.

The test apparatus was a clear plastic chamber (1 m x 1 m x 1.1 m) maintained at 35°C. Compressed oil-free air, saturated with water vapor, was blown through a 5% sodium chloride solution in the chamber, resulting in a fine mist, or fog, of sodium chloride solution filling the chamber and covering the test specimens which were on a neoprene mesh in the top of the chamber. The test ran continuously for 6 months (180 days).

Single limestone test specimens (2 cm x 2 cm x 1 cm) were treated on all sides with each stone preservative. The test specimens were periodically inspected, and the presence of any surface deposits, blistering, or significant changes in the specimens were noted. Only two specimens (31, 38) showed visible changes - after 100 hours both had crystalline substances which gave a basic reaction (pH greater than 11). These deposits disappeared with further testing. After 180 days all specimens were removed from the chamber. Weight changes from salt pick up were insignificant. However, changes in appearance after washing and drying of the specimens were noticeable (figure 21).

4.3 Water Condensation/Evaporation

The effects of cyclic water condensation/evaporation on stone preservative-treated specimens were evaluated by using the test chamber and procedure described in ASTM D 2247-68 (27), Standard Method of Testing Coated Metal Specimens at 100 Percent Relative Humidity.

The test chamber is shown in figure 23. The test specimens were mounted over a water bath (B) kept at a temperature of 38°C. After 90 minutes of water condensation on the specimens, the water was evaporated from the face of the specimens by warm air blown through (C). Following 30 minutes of air-drying, water condensation was repeated. The 90 minutes of water condensation and 30 minutes of warm air-drying constituted one testing cycle.

Stone prisms (5 cm x 2.5 cm x 2.5 cm) were used as test specimens. Five sides of each prism were treated with stone preservative; the one large rectangular face that was not treated faced away from the water bath and was exposed to the room atmosphere.

Testing was continued for 5 months -- 2400 testing cycles. Water condensation/evaporation was used to simulate the cyclic water condensation/evaporation that is an important factor in natural weathering [31]. Evaluated in this test were: 1) absorption of water by the specimen; 2) breakdown of the preservative treatment. These effects were evaluated by periodically removing the specimens from the water bath, observing their appearance, testing the pH of the water condensate, and determining any weight increase.

Specimens that did increase in weight are listed in table 8. These specimens were treated with the same stone preservative treatments that had high water absorption in water absorption testing (Section 3.2.3).

Surface deposits formed on seven specimens during testing (table 9). These deposits appeared either on the untreated surface facing away from the bath, or just below the untreated surface on the side above a wetted zone extending up from the surface which faced the water bath. The deposit, illustrated in figure 24, probably comes from degradation of the preservative and transport of the degradation products by water through the specimen.

After 75 hours testing, the condensate on four specimens (10, 18, 31, 38) gave a basic pH reaction (pH greater than 10) when tested with pH-indicating test paper. The salt deposits that had appeared on the surfaces of specimens 31 and 38 disappeared with further testing.

4.4 The Effect of Ultraviolet Radiation Upon Stone Preservatives

The effect of ultraviolet radiation upon stone preservatives was determined by exposing preservative-treated specimens to ultraviolet radiation in a xenon-arc apparatus.

Method (A) of ASTM G 27-70 (41), Standard Recommended Practice for Operating Xenon-Arc Type Apparatus for Light Exposure of Nonmetallic Materials, was followed. A Model 60-WR Atlas Weather-Ometer with a 6000 watt water-cooled xenon-arc lamp and borosilicate inner and outer glass filters was used. Light exposure was continuous with no water spray on the specimens. Black panel temperature in the test chamber was kept at 62°C.

To better detect color changes in this test, stone preservatives were applied to a white substrate. White unglazed paper and white unglazed tile were both tried as substrates, but many preservatives were not absorbed by either material, either puddling or beading on their surfaces. A uniform absorption of preservatives was obtained with unpolished, sand-rubbed texture white Yule marble, and specimens of this stone (10 cm x 10 cm x 2.5 cm) were used as substrate.

The treated test specimens were hung with steel straps from the revolving specimen rack around the xenon-arc lamp. The positions of the specimens on the drum were changed periodically during testing to give them more uniform exposure to the xenon-arc.

Specimens were exposed to the xenon arc for up to 1100 hours. At periods of 300, 600, and 1100 hours, the specimens were removed and inspected for the appearance of yellowing. Only 13 of the 49 stone preservatives tested showed yellowing during the test (see table 10).

4.5 Suggested Performance Limits from Simulated Tests of Individual Causes of Stone Decay

In evaluating stone preservative performance, simulation of a single cause of stone decay can be useful when that cause is known or suspected to be involved in stone deterioration. As with CAD testing (Section 3.3), varying degrees of protection were given to the stone specimens by the preservatives.

By studying the effects of these tests on specimens treated with over 50 stone preservatives, a judgment can be made as what is acceptable stone preservative performance for each simulated cause of stone decay. These judgments are the basis for the preliminary performance criteria for stone preservatives given in Section 5. The behavior of the stone preservatives used in this program in meeting these limits is indicated in table 13.

5. PRELIMINARY PERFORMANCE CRITERIA FOR STONE PRESERVATIVES

Preliminary performance criteria for the selection of stone preservatives have been developed through knowledge gained in all three phases of the research program. The Phase I literature survey gave information about research that had been done in stone preservation, identified causes of stone decay, and indicated the needs in stone preservation work. The on-site inspections done in Phase II gave practical information about current problems in stone decay and stone preservation. The Phase III laboratory program showed that simulation of the environmental factors causing stone decay can be used (1) to distinguish differences in stone preservative behavior, and (2) to establish preliminary criteria to serve as guides to acceptable performance.

When choosing a stone preservative for a specific stone decay problem it is essential to identify the cause of decay. Only in this way is it certain that the work undertaken is appropriate to stopping, or at least modifying, the decay process. The performance criteria given in this Section require this step to be carried out. The user must therefore decide which performance requirements for stone preservatives fit his problem of stone decay, and which criteria should be applied in selecting preservatives.

Because these criteria will undoubtedly be revised as experience and testing give new information about stone preservative performance, they are designated as preliminary criteria. Until their relationship to field performance is more firmly established, it is recommended that their use be limited to the preliminary screening of materials for field testing.

The preliminary performance criteria are divided into three general categories:

- 5.1 -- General Requirements of Stone Preservative Treatments
- 5.2 -- Resistance of Preservative-Treated Stone to Specific Degradation Factors
- 5.3 -- Resistance of Preservative-Treated Stone to Combinations of Degradation Factors.

Important aspects of stone preservatives in each category are described by a performance statement. Each performance statement consists of four parts [29].

- (1) A Requirement, which states qualitatively what the stone preservative is to accomplish.
- (2) A Criterion, or Criteria, which expresses quantitatively the levels of performance needed to meet the Requirement.
- (3) A Test, which describes how compliance with the Criterion, or Criteria, can be established.
- (4) A Commentary, which may be added to explain or to clarify the Criterion.

The tests given in the following performance statements refer to the earlier sections of the report where the tests are described in detail. The matrix given in table 11 can be used as a guide in selecting tests and methods appropriate for evaluating a preservative's performance once the cause of stone decay is known and properties needed from a preservative treatment are identified. For example, if stone must be strengthened by a preservative, the effectiveness of the preservative in doing this can be evaluated by measuring weight changes, abrasion resistance, and surface hardness during testing. Tests and methods for evaluating resistance to specific environmental causes of stone decay -- the action of chemicals, salt, water -- can be selected in the same manner.

5.1 General Requirements for Stone Preservative Treatments

5.1.1 Chemical Compatibility of the Stone Preservative with the Stone

Requirement. The preservative should not harm the stone or any building materials contiguous to, or embedded in the stone.

Criterion. The stone preservative should not contain any substances that will harm the stone under the expected conditions of use, either upon application or with time.

Test 1. The chemical nature of the preservative should be determined, either by laboratory tests or from information provided by the supplier. This information on the composition should be reviewed by a consultant knowledgeable in stone decay, for its possible effect on the stone. A supplier who cannot divulge this information for a proprietary treatment should guarantee compliance with the criterion.

Test 2. The acidity or basicity of the water phase obtained when the preservative is mixed with an equal volume of distilled water should be within the pH range of 5 to 8.

Commentary. It is recommended that a list of the materials contiguous to or embedded in the stone, or otherwise likely to be contacted by the preservative treatment, should be given the supplier. He should describe the anticipated effects of the preservative treatments on these materials and recommend appropriate safeguards for protecting those materials that would be degraded.

The literature survey and field inspections demonstrated the importance of knowing the chemical nature of the stone preservative. The destructive chemical action of the preservative on the stone or the adjoining material is a serious problem. The deposition of soluble salts or their formation in the stone by the preservative treatment is also to be avoided.

The chemical nature of a preservative should also be considered as to its compliance with regulations covering worker safety and environmental protection.

5.1.2 Application of Stone Preservatives

Requirement. The stone preservative should be capable of being applied by the chosen method(s), so as to give the expected results from the preservation treatment.

Criterion. The method(s) to be used in applying the stone preservative should give a uniform treatment of the stone when applied with the expected level of workmanship, under the anticipated work conditions.

Test 1. The preservative supplier should specify practical methods of application; these should be followed throughout the treatment. These methods should be (1) evaluated by an independent expert in stone preservation, or (2) examined by laboratory and/or field tests by an agent of the user.

Commentary. The need for this Requirement and Criterion was apparent from literature surveys and field inspections. The results obtained from a preservative treatment may be dependent on how the treatment is carried out. This requirement is intended to minimize the likelihood of improper application. It should be noted that any special storage conditions (including shelf life), handling techniques, mixing directions, surface preparation, or safety precautions incident to the use of the preservative should be obtained from the supplier and considered as part of the Test. It is strongly recommended that the preservative be applied by workers experienced in applying it to stone. Further, before application of the preservative, the stone should be cleaned by a cleaning procedure in accord with the supplier's recommendations and all necessary repairs to the stone be completed.

5.1.3 Absorption of the Stone Preservative by Stone

Requirement. The stone preservative should be adequately absorbed by the stone upon application and should penetrate through any weathered surface layer of the stone. The absorbed preservative should not detract from the appearance of the stone, either initially or after a minimum period of on-site weathering for one year.

Criterion. Adequate absorption of the preservative by the stone is obtained if, when applied by the intended method at the intended spreading rate, the appearance of the stone after treatment is not less uniform than before treatment, and there are no signs of unabsorbed preservative on the surface.

Test 1. Changes in appearance are assessed by either the subjective or instrumental methods listed in Section 3.2.2.

Commentary. Once the preservative treatment is completed, objectionable differences in the appearance of the treated stone may occur unless the stone preservative was uniformly applied to and absorbed by the stone. Weathering can accentuate these differences. This weathering should be for at least one year - longer, if possible. To help detect changes in appearance, it is recommended that color photographs be taken of the stone test area before and after treatment, and again, after a minimum of one year's weathering under the expected service conditions. Both supplier and user should evaluate all photographs and agree on the acceptability of any changes in appearance. (A standard color chart included in the test areas before photographing them will help detect color changes due to the color film.)

5.1.4 Environmental Durability of Stone Preservative

Requirement. The stone preservative should withstand the environment to which it is to be exposed for a sufficiently long time to be useful as a preservative.

Criterion. The preservative should not change in appearance or evidence degradation when exposed, for the required test periods, to simulated or actual environments similar to those to which it will be exposed in use.

Test 1. Representative stone specimens should be treated with the preservative and exposed to laboratory testing that simulates the predominant environmental factors encountered at the site where the preservative will be used (see Section 4). When the predominant factors are unknown, a general test program will be used (see Section 3). The duration of testing and methods of evaluation will be defined by the test method being used.

Test 2. For on-site evaluation, an inconspicuous but representative area should be selected for application of the preservative. This area should be exposed to the full effects of the environment. Application of the preservative should follow the procedure recommended by the supplier for large scale application. The area should be inspected and photographed before and after application of the preservative, and periodically thereafter. After a minimum of one year, the test area should be evaluated for changes in appearance and evidence of deterioration of either the preservative or the protected stone (see 5.1.3, Commentary).

Commentary. Stone preservatives that have met laboratory criteria should be exposed also to on-site weathering. Although the laboratory tests will involve conditions more stringent than those found in natural weathering, only natural weathering will ensure that the preservatives are exposed to all environmental factors, in their normal intensity and combination. Whereas laboratory tests are done in relatively short times, defined by the test procedure, it is recommended that on-site weathering tests be carried out for at least one year to allow exposure to all seasonal variations of the climate.

5.2 The Stability of Stone Preservatives Exposed to Specific Degradation Factors

5.2.1 Stability of Stone Preservative Treatments to Air-Borne Chemicals

Requirement. The preservative treatment should protect the stone from attack by air-borne chemicals and remain unaffected by them for sufficiently long to be useful.

Criterion. Air-borne chemicals should not cause rapid physical changes nor changes in appearance of the preservative treated stone or of the preservative treatment when tested as described below.

Tests. As described in Section 4.1.

5.2.2 Stability of Stone Preservative Treatments to Salt Action

Requirement. Preservative treatments should protect the stone from salt action and be unaffected by the salts for sufficiently long to be useful.

Criterion 1. The stone preservative should protect the stone from deterioration due to salt crystallization within the stone for 10 test cycles when tested as described below.

Test. As described in Section 4.2.1.

Criterion 2. The stone preservative should prevent the penetration of the salt solution into the stone for at least 3 test cycles when tested as described below.

Test. As described in Section 4.2.2.

Criterion 3. The preservative treated stone should not change in appearance or the preservative treatment be degraded when exposed for 6 months to a salt water atmosphere in the test described below.

Test. As described in Section 4.2.3.

5.2.3 Stability of Stone Preservative Treatment to Water Condensation/Evaporation

Requirement. The stone preservative should resist deterioration when exposed to cyclic water condensation/evaporation.

Criterion. The surface of the preserved stone should not spall from the stone, decompose, change in appearance, or otherwise deteriorate when exposed to 1200 test cycles of water condensation/evaporation as described in the test below.

Test. As described in Section 4.3.

5.2.4 Stability of Stone Preservative Treatments to Solar Radiation

Requirement. The stone preservative treatment should not deteriorate rapidly during exposure to solar radiation.

Criterion. The stone preservative treatment should not discolor or lose its effectiveness when exposed to 500 test cycles of simulated ultraviolet radiation in the test described below.

Test. As described in Section 4.4.

Commentary. Although the effect of ultraviolet radiation upon a preservative has been considered only as to its effect upon the appearance of the preservative, visual changes indicating changes in other properties should also be considered and evaluated when necessary - the guide matrix (table 4) can be used in selecting appropriate procedures. Other test machines giving ultraviolet radiation of the intensity and wavelength of the machine described in Section 4.4 may also be used.

5.3 The Stability of Preservative Treated Stone to Combinations of Degradative Factors

Requirement. Preservative treated stone should withstand the expected degradative factors significantly better than untreated stone.

Criterion 1. Weight Loss - Preservative treated specimens of stone should have a weight loss no greater than 50% of the weight loss of untreated stone specimens when tested as described below.

Test. As described in Section 3.2.1.

Criterion 2. Appearance - Changes in appearance of the preservative treated stone specimens should be less than that of untreated specimens when tested as described below.

Test. As described in Section 3.2.2.

Criterion 3. Water Absorption - The absorption of liquid water by treated stone specimens should be no more than 50% of the water absorption of untreated specimens when tested as described below.

Test. As described in Section 3.2.3.

Criterion 4. Water Vapor Permeability - The water vapor permeability of preservative treated stone specimens should not decrease more than 25% from the water vapor permeability of untreated specimens when tested as described below.

Test. As described in Section 3.2.4.

Criterion 5. Abrasion Resistance - The abrasion resistance of preservative treated stone specimens should be greater than that of untreated stone specimens when tested as described below.

Test. As described in Section 3.2.5.

Criterion 6. Surface Hardness - The surface hardness of preservative treated stone specimens should not decrease by more than 10% from the surface hardness of untreated specimens when tested as described below.

Test. As described in Section 3.2.6.

Commentary. As discussed in Section 3.3, accelerated test procedures that combine several causes of stone decay attempt to simulate their synergistic action, as in nature, but with the drawback that the contribution of each degradation factor to the decay process cannot be determined. The properties and test methods referred to in the above criteria and the tests used in their evaluation can be applied to other methods simulating one or more causes of stone decay, providing that the test specimen is of a size and shape that can be used with the test instruments. Reference should be made to the matrix guide in table 13, to select those methods/tests that are appropriate to the needed requirement.

6. SUMMARY AND CONCLUSIONS

The development of preliminary performance criteria for selecting stone preservatives was the major goal of this program. This goal has been met through the recommendation of nine preliminary performance criteria in three general categories and the test procedures for establishing compliance with the criteria. Stone preservatives that meet the criteria called for by the stone decay problem will be those most likely to give adequate performance.

The test methods which are given are intended to simulate, both singly and in combination, the major causes of stone decay. A test which combines the most common causes of decay can be carried out with a special test chamber and selected evaluation procedures. Known test procedures can be used for test methods involving single causes of decay, and for the evaluation of both methods of testing.

It must be stressed that more remains to be done in stone preservative testing and evaluation. Much remains to be learned about the mechanisms of stone decay and the effects of stone preservatives on weathered stone. This knowledge may lead to the need of specific methods for specific stones and for specific exposure conditions. Test methods based on a knowledge of the microstructure of the stone to be preserved might be of great assistance in defining both the decay mechanisms and the most effective protective action. As new information about stone preservatives and their effectiveness is obtained and new test methods are developed, modifications may have to be made to the preliminary performance criteria.

The present preliminary criteria offer three benefits to those who need to use stone preservatives. First, they provide an objective basis for selecting stone preservatives, both those now available and those to be developed in the future. Second, they direct attention to properties that stone preservatives should have to be effective. Third, they provide a standard approach to the testing - and selection - of stone preservatives which field personnel can apply to their problems.

This research program leads to three conclusions for stone preservatives in general:

1. Test methods can be used to evaluate tentatively the performance of stone preservatives.
2. None of the stone preservative materials evaluated fulfilled all the proposed performance criteria.
3. A period of field testing must precede any large scale application of a stone preservative.

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APPENDIX A - CONFORMANCE OF STONE PRESERVATIVES TESTED TO CRITERIA

The purpose of the testing phase of the program was to use the test data and experimental procedures to develop preliminary performance criteria that could be used to select stone preservatives. The materials tested as preservatives were evaluated as to their performance. Two tables have been drawn up to summarize the performance of each material tested in the program, both in combined testing and in individual tests. They are:

Table 12 -- Results for General Testing (section 3).

Table 13 -- Results for Individual Testing (Section 4).

In each table, the numbers in the left hand column refer to the code numbers given each preservative. The column headings refer either to the change in property evaluated (Part 3, general testing), or to the specific test carried out (Part 4, individual testing).

Performance is expressed in one of three ways:

X -- the preservative met the criterion given in Part 5.

blank space -- the preservative did not meet the criterion.

N.T. -- the preservative was not tested.

The test data used in developing these tables should be accepted as valid only within the conditions of the test program.

These tables are intended only as a guide to selecting preservative materials for field application. It should be stressed that no one preservative met all performance criteria. Where a preservative has met the criterion or criteria relevant to a need in stone preservation, its use can be tentatively recommended. However, in all applications, preliminary field testing in situ will be necessary to adequately judge the material's performance.

TABLE 1. Stone Preservative Materials (2.4)

Stone Preservative Code #	Compositional Type	Non- Volatile Content	Liquid Preservative	Dry Preservative
			color; miscibility with water	color; physical state
01	Silicone	6.6%	water-white; immiscible	colorless; adherent, flexible film
02	Silicone	16.7%	water-white; immiscible	colorless; hard, brittle film
03	Methyl Methacrylate	20.7%	white, opaque; miscible	pale-white; flexible film
04	Methyl Methacrylate	18.5%	white; miscible	orange; brittle film
05	Silicone	10.0%	water-white; immiscible	white; crumbly film
06	Silicone	12.5%	water-white, miscible	did not dry
08	Methyl Silicone	6.7%	water white; miscible	pale yellow; brittle film
09	Methyl Silicone	80.0%	water-white; immiscible	colorless; brittle film
10	Methyl Silicone	33.0%	water-white; miscible (pH > 11)	white; brittle film
11	Methyl Silicone	53.0%	water-white; viscous, immiscible	--
12	Methyl Silicone	53.0%	white; miscible	white; waxy solid
13	Methyl Silicone	37.0%	water-white; immiscible	colorless; sticky film
14	Methyl Silicone	6.6%	water-white; immiscible	brown; crumbly, granular solid
15	Methacrylate Polymer	27.0%	water-white; immiscible	flexible, sticky mass
16	Methacrylate Polymer	30.0%	water-white; immiscible	colorless; flexible film
17	Methacrylate Polymer	39.0%	water-white; immiscible	pale white; tough, adherent, brittle film
18	Inorganic	26.0%	water-white; miscible, pH > 11	--
19	Inorganic	47.0%	water-white; miscible, pH 1	white; crystalline solid
20	Inorganic	45.0%	water-white; miscible, pH 3	dark brown; powder
21	Methyl Silicone	55.0%	water-white; immiscible	colorless; pliable film
22	Methyl Silicone	23.0%	water-white; immiscible	colorless; brittle film
23	Polyisobutyl Methacrylate	33.0%	colorless; immiscible	--
24	Acrylic Polymer	23.0%	water-white; immiscible	colorless; brittle film
25	Acrylic Polymer	10.0%	water-white; immiscible	white; crumbly film

TABLE 1. Stone Preservative Materials (continued)

Stone Preservative Code #	Compositional Type	Non- Volatile Content	Liquid Preservative	Dry Preservative
			color; miscibility with water	color; physical state
26	Acrylic Polymer	10.0%	water-white; immiscible	cream colored; powder
27	Methyl Silicone	23.0%	water-white; immiscible	colorless; brittle film
28	Methyl Silicone	55.0%	water-white; immiscible	colorless; pliable film
29	Polyisobutyl Methacrylate	31.0%	water-white; immiscible	--
30	Acrylic- Epoxy	39.0%	water white; miscible	pale yellow; flexible film
31	Sodium Methyl Siliconate	33.0%	water-white; miscible, pH > 11	white; powder pH > 11
32	Acrylic Polymer	10.0%	water-white; immiscible	water-white; flexible film
33	Acrylic Polymer	27.0%	water-white; solidified with H ₂ O	colorless; adherent flexible, tough film
34	Acrylic Polymer	21.0%	water-white; immiscible	colorless; flexible film
35	--	10.0%	water-white; immiscible	clear, dry, adherent, flexible film
36	Plastic Emulsion	3.0%	green-white; miscible	bluish; flexible film
37	Plastic Emulsion	28.0%	white; immiscible	faint yellow; tough, flexible film
38	Sodium Methyl Siliconate	31.0%	water-white; miscible, pH > 11	does not dry
39	Inorganic	--	water-white; miscible, pH > 10	--
40	Acrylic Copolymer	20.0%	water-white; immiscible	colorless; flexible, tough film
41	Silicone	4.9%	water-white; immiscible	--
42	Acrylic Resin	30.0%	water-white; immiscible	--
43	Fluorocarbon	2.0%	water-white; immiscible	colorless; clear, flexible film
44	Fluorocarbon	11.0%	water-white; ppt with water	colorless; clear, brittle film
45	Fluorocarbon	18.0%	white; miscible	yellow; brittle film
46	Fluorocarbon	2.0%	water-white; miscible	colorless; brittle film
47	Linseed Oil	103.0%	amber; immiscible	does not dry

TABLE 1. Stone Preservative Materials (continued)

Stone Preservative Code #	Compositional Type	Non- Volatile Content	Liquid Preservative	Dry Preservative
			color; miscibility with water	color; physical state
49	Sodium Silicate, 40° Baume	56.0%	water-white; miscible H ₂ O pH > 11	colorless; brittle film
50	Tetraethyl Ortho-Silicate	0.0%	water-white; immiscible	--
51	Acrylic Resin	47.0%	white; miscible	white; flexible, tough film
52	Methyl Silicone	7.0%	pale red-yellow; miscible	faint yellow; brittle film
53	Calcium Hydroxide	--	water-white; pH > 14	--
54	Ba (OH) ₂ + additives	30.0%	water-white; pH > 14	--
55	Fluorocarbon	--	--	--
56	Fluorocarbon	--	--	--

TABLE 2. Stone Preservative Treatments of Indiana Gray Limestone.
 Untested Stone: color; feel; weight changes; depth penetration
 of preservative. Stone Tested in CAD: color; feel; weight change.
 (2.4)

Stone Preservative	Untested Stone	Stone Tested in CAD	
	(color; feel; wgt. change; penetration)	(color; feel; wgt. change) 10 cycles	20 cycles
00	light gray; smooth	orange-brown; rough; -1.16%	dark brown; rough -2.06%
01	dark gray; smooth; +0.13%; 4 mm	light orange; rough; -0.38%	light brown; rough; -1.43%
02	medium gray; smooth; +0.72%; --	light gray-brown; rough; --	light orange- brown; rough; -1.93%
03	light gray-brown, glossy; smooth; +0.27%; <1 mm	light gray-brown; smooth; -0.09%	light orange- brown, no gloss; smooth; -0.29%
04	mottled orange, slight gloss; smooth; +0.24%; 1 mm	orange; --; -0.78%	dark brown; rough; -2.16%
05	light gray-brown; +0.22%; 2 mm	light orange; smooth; -0.45%	dark orange; smooth; -0.98%
06	dark brown; smooth; +0.50%; --	light brown; smooth; -1%	orange-brown; rough; -2.10%
08	light gray-brown, slight gloss; +0.15%; <1 mm	dark brown, no gloss; -0.56%	dark orange; rough; -1.27%
09	dark brown; +1.98%; 8 mm	light brown; rough; -0.77%	light brown; rough; -1.73%
10	dark gray; smooth +0.77%; 8 mm	light brown; rough; --	light brown; rough; -2.13%
11	light gray; +0.05%; <1 mm	light orange; rough; -1.02%	--
12	dark gray; smooth; +0.49%; 9 mm	orange coating; smooth; -0.68%	orange color; rough; -0.97%
13	dark brown; smooth; +0.69%; 3 mm	orange; smooth; -0.53%	orange; rough; -2.14%
14	dark gray; smooth; +0.06%; 3 mm	light orange; smooth; -0.61%	dark orange; rough; -1.44%
15	dark brown; smooth; +0.23%; 1 mm	light brown coating; smooth; -0.24%	dark orange; smooth; -1.10%
16	dark brown; smooth +0.32%; <1 mm	light brown; smooth -0.19%	light brown; smooth; -0.63%
17	dark brown-gray, gloss; smooth; +0.35%; 2 mm	medium brown, no gloss; smooth; -0.59%	light brown; rough; -0.62%
18	light brown; smooth; +0.63%; 5 mm	light brown; rough; -0.40%	light brown; smooth; -1.68%
19	light brown; smooth; +0.46%; <1 mm	light brown; smooth; --	light brown; rough; -2.1%

TABLE 2. Stone Preservative Treatments of Indiana Gray Limestone.
Untested Stone: color; feel; weight changes; depth penetration
of preservative. Stone Tested in CAD: color; feel; weight change.
(2.4) (continued)

Stone Preservative	Untested Stone	Stone Tested in CAD	
	(color; feel; wgt. change; penetration)	(color; feel; wgt. change) 10 cycles	(color; feel; wgt. change) 20 cycles
20	light gray; smooth; -- <1 mm	light gray; smooth; --	orange; smooth; -1.70%
21	dark brown, gloss; smooth; +0.75%; 1 mm	light brown; rough; -0.22%	light orange surface; -0.48%
22	medium brown; smooth; +0.53%; --	light brown; rough; -0.98%	light brown; rough; -1.95%
23	light brown, gloss; +0.36%; --	light brown, no gloss; smooth; -0.43%	light brown, no gloss; smooth; -0.25%
24	light gray-brown; smooth; +0.37%; <1 mm	light gray-brown; smooth; -0.08%	light gray-brown; smooth; -0.12%
25	light brown; smooth; +0.04%; 4 mm	light brown; smooth; -1.54%	light brown; smooth; -0.45%
26	light gray; smooth; +0.15%; 5 mm	light orange; rough; -0.88%	dark orange; rough; -2.04%
29	dark brown, gloss; smooth; +0.30%; --	--	medium brown, no gloss; rough; -0.23%
30	dark gray, gloss; smooth; +0.23%; <1 mm	dark gray, gloss; smooth; -0.49%	dark gray, gloss; smooth; -1.90%
31	light brown; smooth; +0.96%; 6 mm	light brown; rough; -0.97%	dark brown; rough; -1.22%
32	medium brown; smooth; +0.10%; 6 mm	--	light brown; smooth; -0.83%
33	medium brown, gloss; smooth; +0.44%; 3 mm	dark brown, some gloss; smooth; -0.40%	dark brown, no gloss; smooth; -0.22%
34	medium brown, gloss; smooth; +0.32%; <1 mm	orange-brown; rough; -1.09%	dark brown; rough; -1.88%
35	medium brown, gloss; smooth; +0.11%; <1 mm	medium brown, no gloss; smooth; -0.20%	medium brown; smooth; -0.29%
36	dark gray; smooth; +0.20%; <1 mm	dark orange; mottled; rough; -0.49%	medium orange; rough; -0.95%
37	light brown, gloss; smooth; +0.44%; <1 mm	light gray; rough; -0.23%	light gray; rough; -0.34%
38	medium gray; smooth; +0.34%; 8 mm	light gray; smooth; --	gray-brown; rough; -1.81%
40	medium brown, gloss; smooth; +0.34%; <1 mm	light brown, gloss; rough; -0.15%	light brown, no gloss; rough; -0.43%

TABLE 2. Stone Preservative Treatments of Indiana Gray Limestone.
 Untested Stone: color; feel; weight change; depth penetration
 of preservative. Stone Tested in CAD; color; feel; weight change.
 (2.4) (continued)

Stone Preservative	Untested Stone	Stone Tested in CAD	
	(color; feel; wgt. change; penetration)	(color; feel; wgt. change) 10 cycles	20 cycles
41	light gray; smooth; +0.11%; <1 mm	light brown; rough; -0.56%	dark orange; rough; -2.17%
42	light brown; smooth; +0.18%; 3 mm	orange; rough; -1.33%	orange; rough; -1.82%
43	light gray; smooth; +0.03%; 1 mm	light brown; rough; -0.93%	orange; rough; -2.05%
44	light gray; smooth; +0.15%; 1 mm	light gray; rough; -0.09%	orange; rough; -0.62%
45	light gray; smooth; +0.17%; <1 mm	light gray; smooth; -0.19%	orange-gray; smooth; -0.57%
46	light gray; smooth; +0.01%; 1 mm	orange-gray; rough; -0.24%	light brown; rough; -1.93%
47	dark brown; smooth; +1.41%; 6 mm	light orange; smooth; -1.37%	dark orange; rough; -1.59%
49	dark gray; smooth; +0.94%; <1 mm	dark gray; rough; -2.1%	orange-gray; rough; -3.90%
50	light gray; smooth; +0.05%; <1 mm	light brown; rough; -1.63%	medium brown; rough; -2.8%
51	medium gray, gloss; smooth; +0.75%; <1 mm	medium gray, some gloss; smooth; +0.28%	medium gray, some gloss; smooth; +0.06%
52	dark gray; smooth; +0.22%; <1 mm	light orange; rough; -0.96%	dark orange; rough; -1.42%
53	light gray; smooth +0.01%; --	light brown-gray; smooth; -0.54%	medium brown; rough; -1.54%
54	light brown; smooth; +1.25%	light brown; smooth; -0.88%	dark brown; smooth; -2.01%
55	light brown; smooth; --; 3 mm	light orange-brown; smooth; -0.35%	orange; smooth; -0.76%
57	dark gray; smooth; --; 5 mm	--	orange; rough; -0.92%
58	dark brown; smooth; --; 10 mm	orange; smooth; -0.04%	orange; smooth; -0.09%

TABLE 3. CAD Test Cycle (3.1)

Overnight	-- Specimens stored in deep-freeze at -40°C. <u>(16 hours)</u>
8:30	-- Specimens removed from deep-freeze, mounted on drum of CAD.
8:30 - 10:00	-- Specimens rotated with lamps (6-UV, 3-IR) on -- specimens' surface temperature reaching 50°C. <u>(1 1/2 hours)</u>
10:00 - 10:30	-- Water spray on, continued rotation of specimens with lamps on. <u>(1/2 hour)</u>
10:30 - 11:00	-- Water spray off; continued rotation of specimens with lamps on. <u>(1/2 hour)</u>
11:00 - 16:00	-- Lamps on, CAD drain closed; 3% sodium chloride/0.01 molar sulfurous acid solution pumped into CAD, specimens rotated, passing through solution for 1/8 of rotation. <u>(5 hours)</u>
16:00 - 16:30	-- Lamps off, continued rotation of specimens. <u>(1/2 hour)</u>
16:30	-- CAD drained, specimens removed and returned to deep-freeze for overnight storage.

TABLE 4. Contribution of Each Feature of the CAD to
Simulated Stone Decay

Simulated Cause of Stone Decay \ Feature of CAD Test	UV Lamps	IR Lamps	Water Spray	Water Soak	Sulfurous Acid Soak	Salt Soak	Deep Freeze
Water Action			X	X	X	X	X
Chemical Attack					X	X	
Salt Action		X		X		X	
Thermal Effects		X	X				X
Solar Radiation	X	X					

TABLE 5. Result of Specimens Exposed to 49 Cycles Sulfurous
Acid Testing (4.1)

00	*	28	white surface deposit
01	*	29	surface deposit
02	*	30	blistering of surface
03	* - white surface	31	white surface
04	original color lost	32	white surface
05	*	33	discoloration
06	blistering of surface	34	discoloration, flaking of preservative
08	* - white surface	35	white surface
09	*	36	no change
10	*	37	no change
11	*	38	*
12	flaking of preservative	39	blistering of surface
15	white surface	40	*
16	white surface	41	*
17	* - surface residue	42	surface deposit
18	discoloration	43	*
19	*	44	no change
20	*	45	*
21	white surface	46	white surface deposit
22	*	47	fading of color
23	*	49	brittle, cracked surface
24	no change	51	discolored
25	*	52	surface deposit
26	*	53	mottled appearance

*small acicular crystals on specimen surface

TABLE 6. Sodium Sulfate Crystallization Test (4.2.1)
(Cumulative percent weight loss of specimens)

Stone Preservative	Test Cycles									
	1	2	3	4	5	6	7	8	9	10
00	+2	0	-1	-8	-16	-21	-25	-29		
01	+1	+1	+1	+1	+1	-1	-1	-8	-17	-27
02	0	0	0	0	0	0	-1	-1	-1	-1
03	+2	+2	+3	-2	-12	-20	-26			
04	+3	+3	+2	-2	-5	-7	-10	-12	-23	-28
05	0	0	0	0	0	0	0	+1	0	+1
06	+2	+3	+3	+2	+3	+2	+1	+4	-11	-20
08	+1	+3	+3	+12	-20	-25	-29			
09	0	0	0	0	0	0	0	0	0	0
10	0	0	0	0	0	0	0	0	0	0
11	+1	-1	-8	-11	-14	-17	-21	-25		
12	+2	+1	-2	-8	-15	-9	-10	-15	-17	-17
13	+2	+1	+1	0	-4	-10	-15	-23	-26	
14	+2	+1	-3	-4	-5	-5	-6	-13	-15	-15
15	0	0	+1	-17	-24	-25				
16	+1	+1	-14	-15	-17	-23	-34			
17	0	+1	-9	-24	-30					
18	0	0	0	0	-1	-4	-6	-12		
19	+1	+1	+2	+1	-4	-10	-15	-17	-23	-26
20	+1	+1	+1	-1	-5	-9	-11	-13	-14	-15
21	0	0	0	0	0	+1	+1	+1	+1	-11
22	+1	+1	+1	0	0	0	0	0	-1	-6
23	+4	+4	+5	+3	+1	+1	-1	-6	-10	-11
24	0	0	+1	0	-1	-6	-22			
25	0	0	0	0	0	+1	+1	+1	+1	-8
26	0	0	0	0	0	0	0	0	0	0
29	+1	+3	+4	+2	-5	-13	-19	-24	-29	
30	+3	+3	+4	+4	+4	+5	-5	-13	-19	-21
31	0	0	0	0	0	0	0	0	0	0
32	+3	+2	+1	-1	-8	-23	-30			
33	0	0	0	0	+2	+2	-8	-20	-25	
34	+2	-2	-3	-15	-24	-29				
35	+3	-3	+3	-2	-5	-8	-13	-16	-20	-24
36	+2	+2	-3	-4	-34					
37	+1	+2	+2	-5	-9	-24	-32			
38	0	+1	0	0	0	0	0	0	0	0
39	+1	+1	+2	+2	+2	+2	+2	+2	+2	+2
40	+7	+3	0	-5	-8	-10	-12	-14	-19	-25

TABLE 6. Sodium Sulfate Crystallization (continued)

Stone Preservative	Test Cycles									
	1	2	3	4	5	6	7	8	9	10
41	+1	+2	0	-2	-5	-16	-25			
42	+2	+3	+1	-1	-4	-7	-9	-12	-18	-37
43	0	+1	+1	-5	-15	-21	-27			
44	0	0	-4	-6	-8	-12	-16	-24	-31	
45	+1	+2	-3	-11	-20	-29				
46	0	+1	+1	-1	-12	-23	-25			
47	+1	+1	+2	+2	+2	+1	+1	+1	+1	+1
49	0	+2	+2	+1	0	-2	-5	-8	-11	-18
50	+1	+2	0	-2	-4	-5	-8	-10	-11	-13
51	+1	+2	+3	+3	+2	+3	+4	+2	-26	
52	0	+1	+2	+2	-4	-13	-20	-28		
53	+2	+2	0	-4	-8	-14	-19	-23	-29	
54	+2	+2	+3	+3	+3	+3	+3	+2	+2	+1

TABLE 7. Sodium Sulfate Penetration Test (4.2.2)
Condition of dry test specimens after three test cycles.
Top of specimen untreated, other five sides treated with
stone preservative

Stone Preservative	Condition of Specimen	Stone Preservative	Condition of Specimen
00	top degraded	27	NC*
01	NC	28	efflorescence on top
02	NC	29	sides spalling
03	top degraded; sides spalling	30	top degraded
04	top degraded	31	NC
05	NC	32	top degraded
06	NC	33	sides spalling
08	top degraded; sides spalling	34	sides spalling
09	NC	35	sides spalling
10	NC	36	top degraded
11	top degraded	37	top degraded; sides spalling
12	NC	38	NC
13	top degraded	39	NC
15	NC	40	top degraded; sides spalling
16	NC	41	efflorescence on top
17	sides spalling	42	sides spalling
18	NC	43	top degraded; sides spalling
19	efflorescence on top	44	sides spalling
20	efflorescence on top	46	sides spalling
21	top degraded	47	NC
22	NC	49	NC
23	top degraded	50	top degraded
24	top degraded; sides spalling	51	top degraded
25	NC	52	efflorescence on top
26	NC	53	top degraded; sides spalling
		54	NC

* NC = no change in specimen.

TABLE 8. Water Absorbed by Specimens During
Condensation/Evaporation Testing (4.3)

<u>Stone Preservative</u>	<u>Percent Weight Gain</u>
00	6.3
03	6.7
04	5.2
06	4.8
08	5.1
11	5.4
12	1.4
13	4.7
14	4.0
19	5.2
20	4.8
30	3.6
32	3.7
34	5.6
35	3.5
36	6.3
37	6.6
42	4.3
45	5.4
47	4.5
49	5.1
50	5.9
51	5.0
52	5.9
53	5.7
54	4.3

TABLE 9. Surface Deposits on Specimens After Water
Evaporation/Condensation Testing (4.2.1)

<u>Stone Preservative</u>	<u>Description of Surface Deposit</u>
04	White deposit on sides of specimen above wet zone.
20	Dark deposits on sides of specimen above wet zone.
34	Very large white surface deposit on untreated surface of specimen.
36	Small white deposits on sides of specimen above wet zone.
37	White crust covering untreated surface.
47	Dark orange discoloration on sides of specimen above wetted zone.
50	White deposits on ends of specimen.
51	Spalling of preservative coating along untreated surface.
54	White deposits on untreated surface.

TABLE 10. Stone Preservatives Which "Yellowed" After Exposure to 1100 Hours Radiation in a Xenon-Arc Weathering Apparatus (4.4)

<u>Stone Preservative</u>	<u>Generic Composition</u>
04	Methyl Methacrylate
05	Silicone
08	Methyl Silicone
15	Methacrylate Polymer
16	Methacrylate Polymer
19	Inorganic Composition
20	Inorganic Composition
33	Acrylic Polymer
34	Acrylic Polymer
35	Organic Resin
40	Acrylic Copolymer
42	Unknown
47	Linseed Oil

TABLE 11. Guide to the Selection of Tests and Methods for Evaluating Stone Preservative Performance (5)

Method/Test to Determine Perf.		3.2.1	3.2.2	3.2.3	3.2.4	3.2.5	3.2.6	4.1	4.2.1	4.2.2	4.2.3	4.3	4.4
Required Results of Stone Preservative Treatment		Wgt. Change	Appear. Change	Water Absorp.	Water Vapor Permeability	Abrasion Resist.	Surface Hardness	Air-Borne Chemical	Salt Cryst.	Salt Penet.	Salt Atmosph.	Water Evap./Cond.	Solar Radiation
1.	Surface Strengthening	X*				X	X			?			
2.	Resistance to Chemical Action	X	X	X	X			X	X	X	X		
3.	Resistance to Salt Action	X	X						X	X	X		
4.	Resistance to Water Action		X	X	X								
5.	Resistance to Solar Radiation		X										X
6.	Retention of Appearance		X					X			X	X	X

* X = may be useful

TABLE 12. Extent to Which Stone Preservatives Met, After 20 Cycles of CAD Testing (Section 3), the Preliminary Performance Criteria of Section 5 (see Appendix A)

Stone Preservative	Appearance	Weight Loss	Water Absorption	Water Vapor Permeability	Abrasion Resistance	Surface Hardness
1				X*	X	X
2				X		X
3	X	X		X		X
4				X		X
5		X		X	X	X
6	X			X		X
8				X		X
9	X			X		
10	X			X		X
11				X	X	X
12		X		N.T.**	X	X
13				X		X
14				X	X	X
15				X		X
16		X		X		X
17		X		X		X
18	X			X		X
19		N.T.		X	X	X
20	X			X		X
21		X		X		
22	X			X		X
23		X		X		X
24		X				
25						X
26	X			X	N.T.	X
27	N.T.	N.T.		N.T.	N.T.	N.T.
28	N.T.	N.T.		N.T.	N.T.	N.T.
29		X		X	X	X
30						X
31				X		X
32		X		X	N.T.	X
33		X		X		X
34						X
35		X		X		
36		X		X		X

* X = met criteria.

** N.T. = not tested.

TABLE 12. CAD Testing (20 cycles) (continued)

Stone Preservative	Appearance	Weight Loss	Water Absorption	Water Vapor Permeability	Abrasion Resistance	Surface Hardness
37		X				
38				X		X
39		N.T.		N.T.	N.T.	N.T.
40		X				X
41	X			X		X
42				X	X	X
43				N.T.	N.T.	X
44		X				X
45		X		X		
46				X	N.T.	X
47				X		X
49				X		X
50				X		X
51				X	N.T.	N.T.
52				X		X
53	X			X		X
54				X		X
55		X	X	X		X
57		X		X		X
58		N.T.				X

TABLE 13. Extent to Which Stone Preservatives Met, for Individual Test Methods (Part 4), the Preliminary Performance Criteria of Section 5. (see Appendix A)

Stone Preservation	Water Condensation/ Evaporation	Sodium Sulfate Crystallization	Sodium Sulfate Penetration	Ultraviolet Radiation	Sulfurous Acid Fog	Sodium Chloride Fog
1	X*		X	X		X
2	X	X	X	X		X
3	X			X		X
4						X
5	X	X	X			X
6	X		X	X		X
8	X					X
9	X	X	X	X		X
10	X	X	X	X		X
11	X			X		X
12	X		X	X		X
13	X			N.T.**	N.T.	
14	X		N.T.	X	N.T.	N.T.
15	X		X			X
16	X		X			X
17	X			X		X
18	X		X	X		X
19	X					X
20						X
21	X			X		X
22	X		X	X		X
23	X			X		X
24	X			X	X	X
25	X		X	X		X
26	X	X	X	X		X
27	X	N.T.	X	X		X
28	X	N.T.		X		X
29	X			X		X
30	X			X		X
31	X	X	X	X		X
32	X			X		X
33	X					X
34						X
35	X					X
36				X	X	X

* X = met criteria.

** N.T. = not tested.

TABLE 12. Results for Individual Testing (see Appendix A) (continued)

Stone Preservation	Water Condensation/ Evaporation	Sodium Sulfate Crystallization	Sodium Sulfate Penetration	Ultraviolet Radiation	Sulfurous Acid Fog	Sodium Chloride Fog
37				X	X	X
38	X	X	X	X		X
39	N.T.	X	X	N.T.		X
40	X					X
41	X			X		X
42	X					X
43	X			X		X
44	X			X	X	X
45	X		N.T.	X		X
46	X			X		X
47		X	N.T.			X
49	X		X	X		
50				X	N.T.	X
51				X		X
52	X			X		N.T.
53	X			X		N.T.
54		X	X	X	N.T.	X
55	N.T.		N.T.	N.T.	N.T.	X
57	N.T.	N.T.	N.T.	N.T.	N.T.	N.T.
58	N.T.	N.T.	N.T.	N.T.	N.T.	N.T.

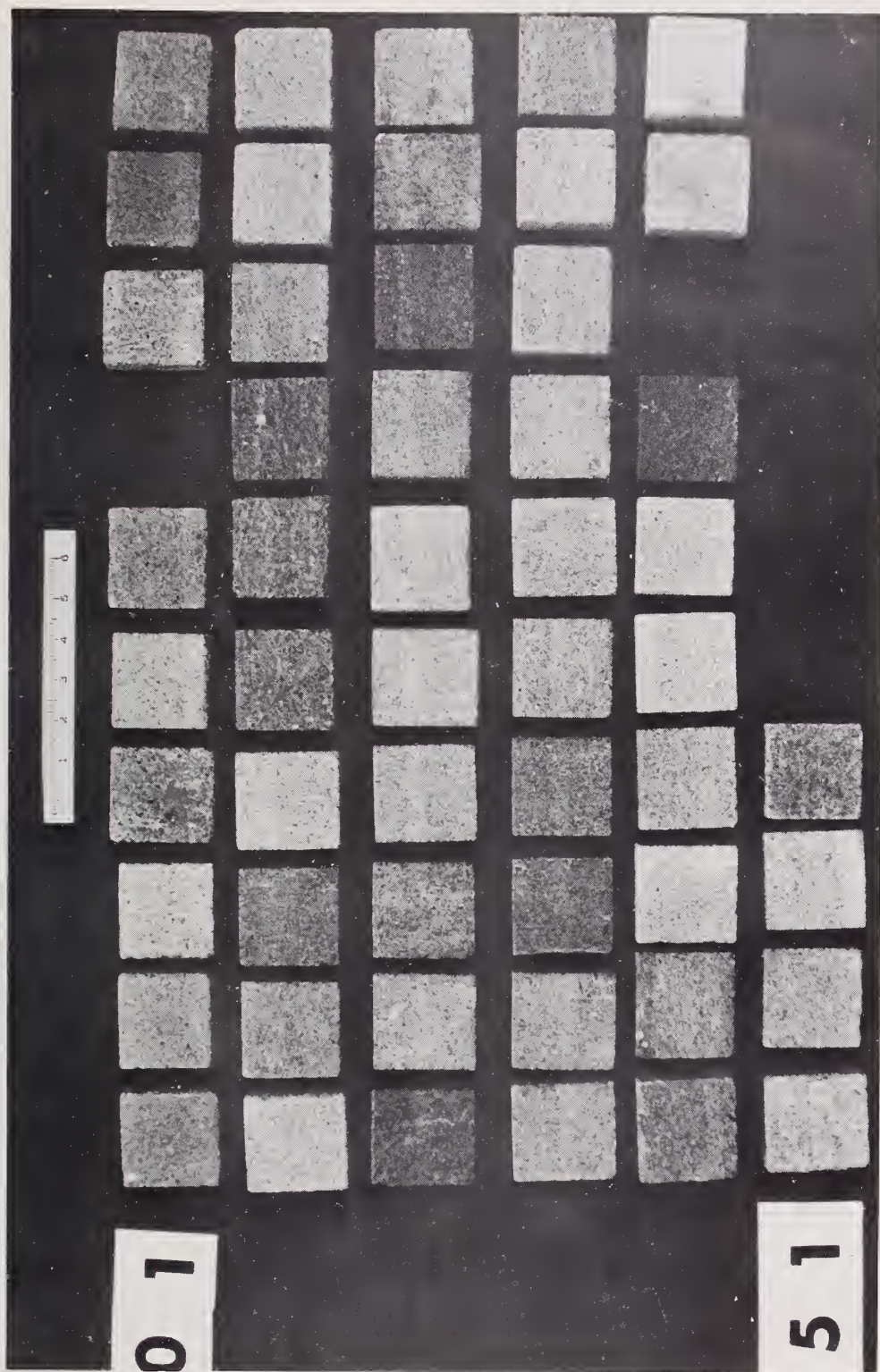


Figure 1. Change in Appearance (2.4): Limestone specimens treated with stone preservatives; code numbers for stone preservatives begin at upper left square (01) and end at bottom row with (54); first specimens of each row are 01, 11, 21, 31, 41, 51 respectively.

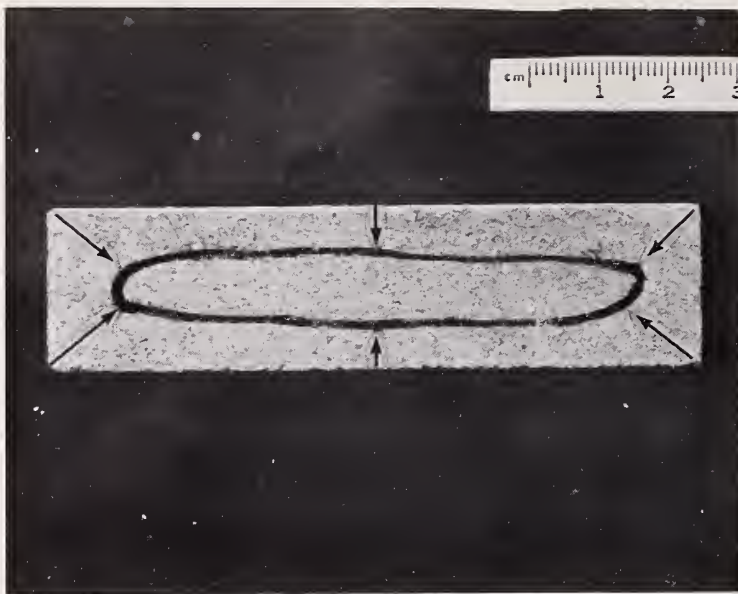


Figure 2. Penetration of Stone Preservative into Indiana Limestone (2.4): Section of stone specimen treated with stone preservative; black line indicates depth of penetration of preservative.

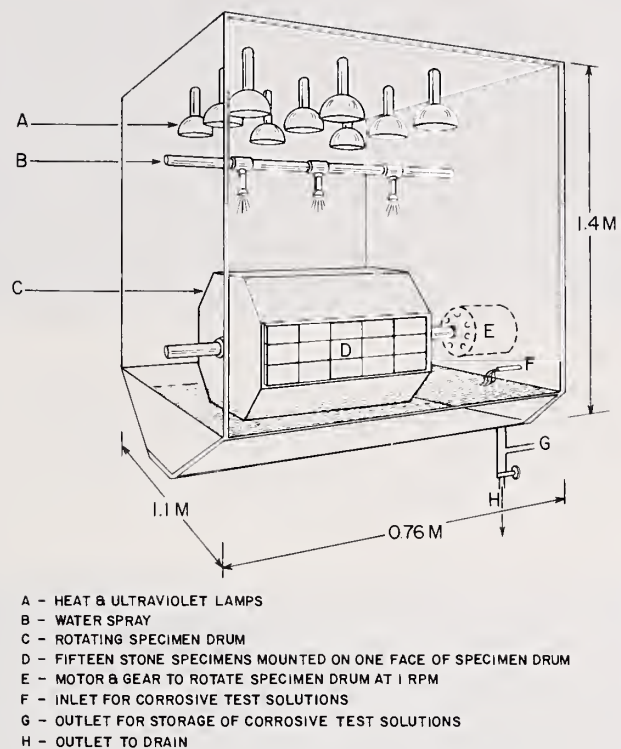


Figure 3. Chamber for Accelerated Decay (CAD) Section 3.1.

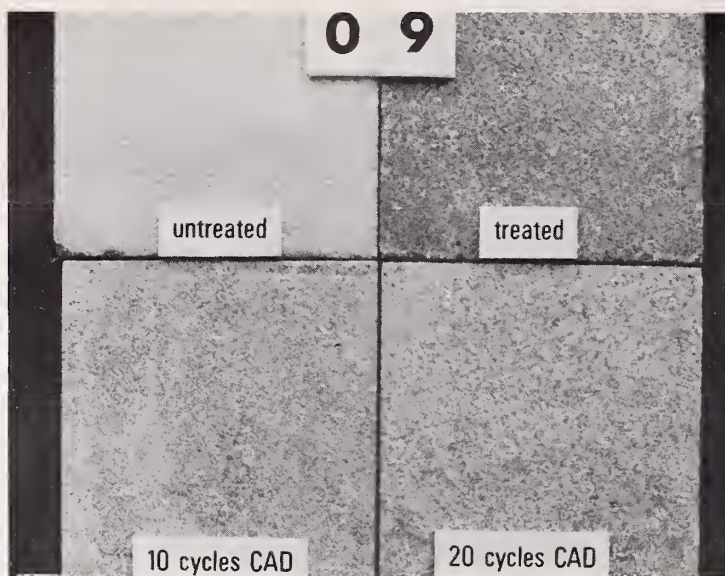


Figure 4. Change in Appearance (3.2.2): Slight change in appearance of treated stone. Top row -- untreated limestone specimen; limestone treated with preservative 09. Bottom row -- limestone specimens treated with preservative 09 after 10 and 20 CAD test cycles respectively.

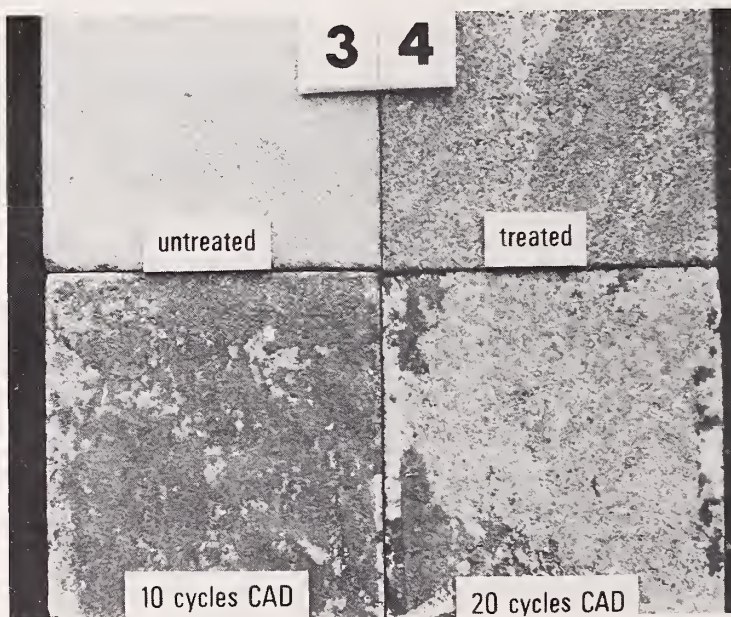


Figure 5. Change in Appearance (3.2.2): Extreme change in appearance of the treated stone. Top row -- untreated limestone specimen; limestone specimen treated with preservative 34. Bottom row -- limestone specimens treated with preservative 34 after 10 and 20 CAD cycles, respectively.

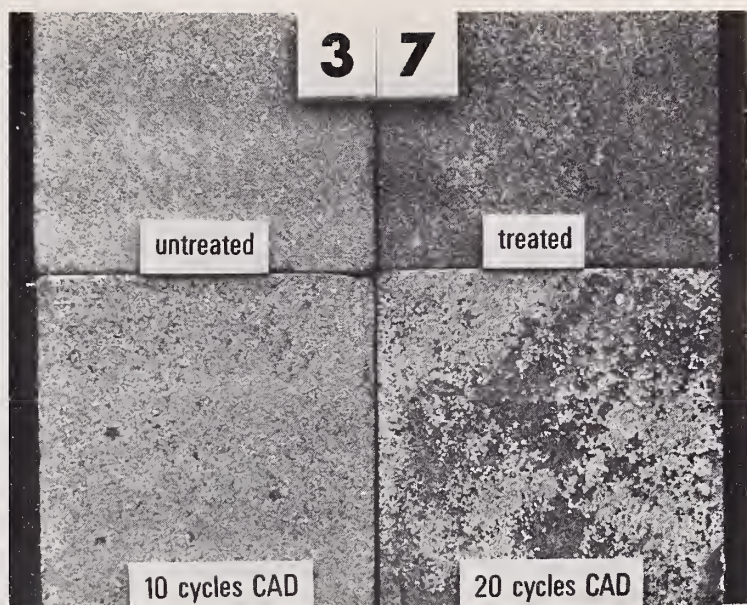


Figure 6. Change in Appearance (3.2.2): Change in appearance through loss of preservative coating. Top row -- untreated limestone specimen; limestone specimen treated with preservative 37. Bottom row -- limestone specimens treated with preservative 37, after 10 and 20 CAD cycles respectively.

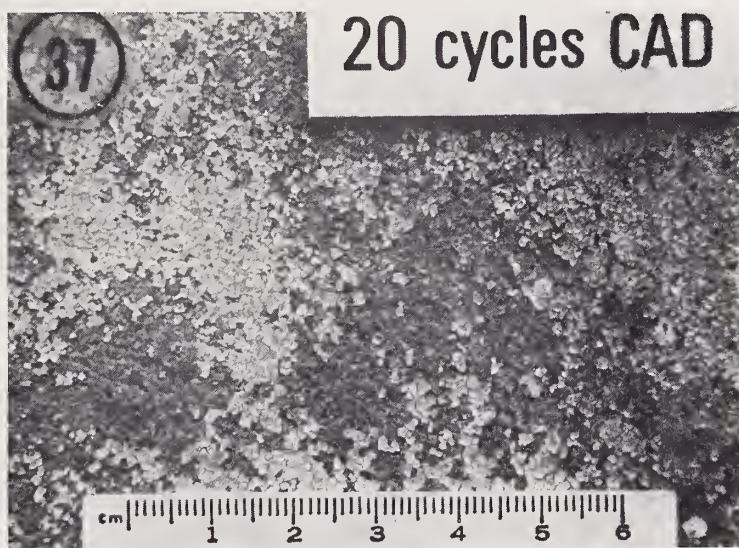


Figure 7. Change in Appearance (3.2.2): Close-up of surface of stone specimen treated with preservative 37, exposed 20 CAD cycles, from Figure 6.

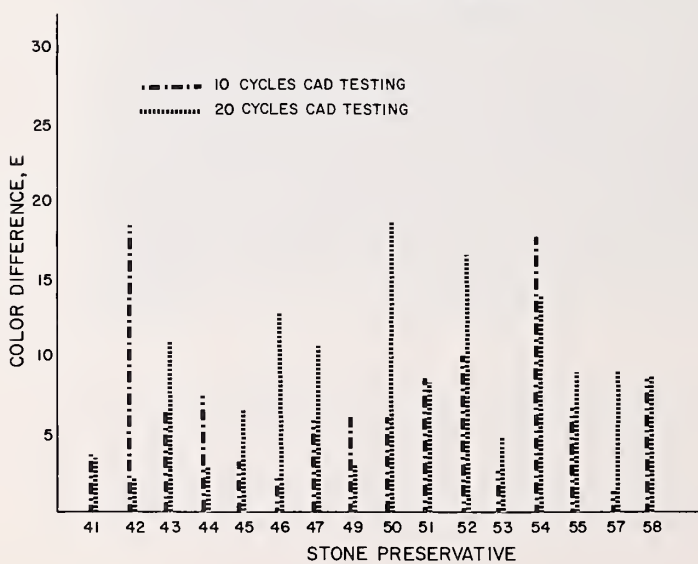
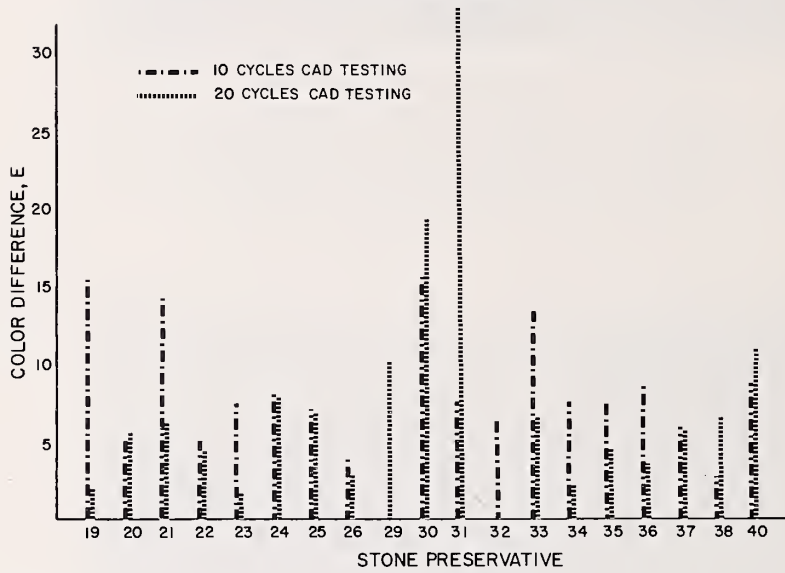
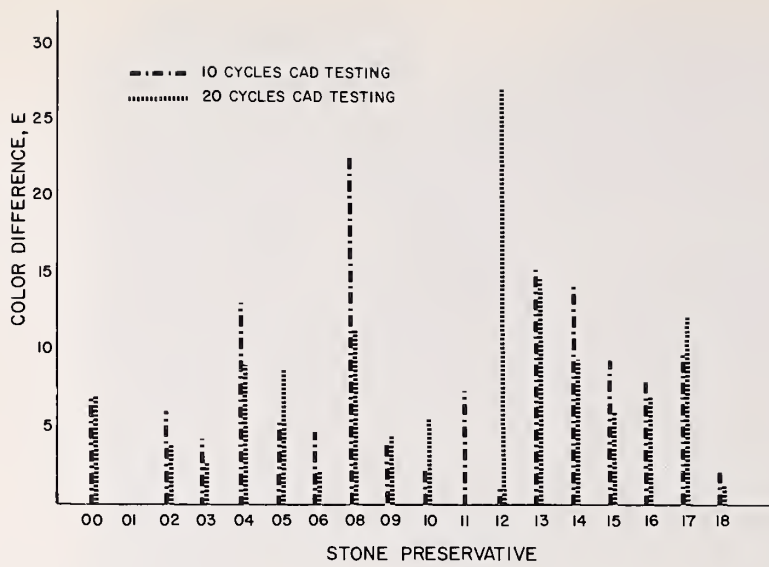


Figure 8. Change in Appearance (3.2.2): color differences (E) of stone preservative treated specimens after 10 and 20 CAD test cycles.

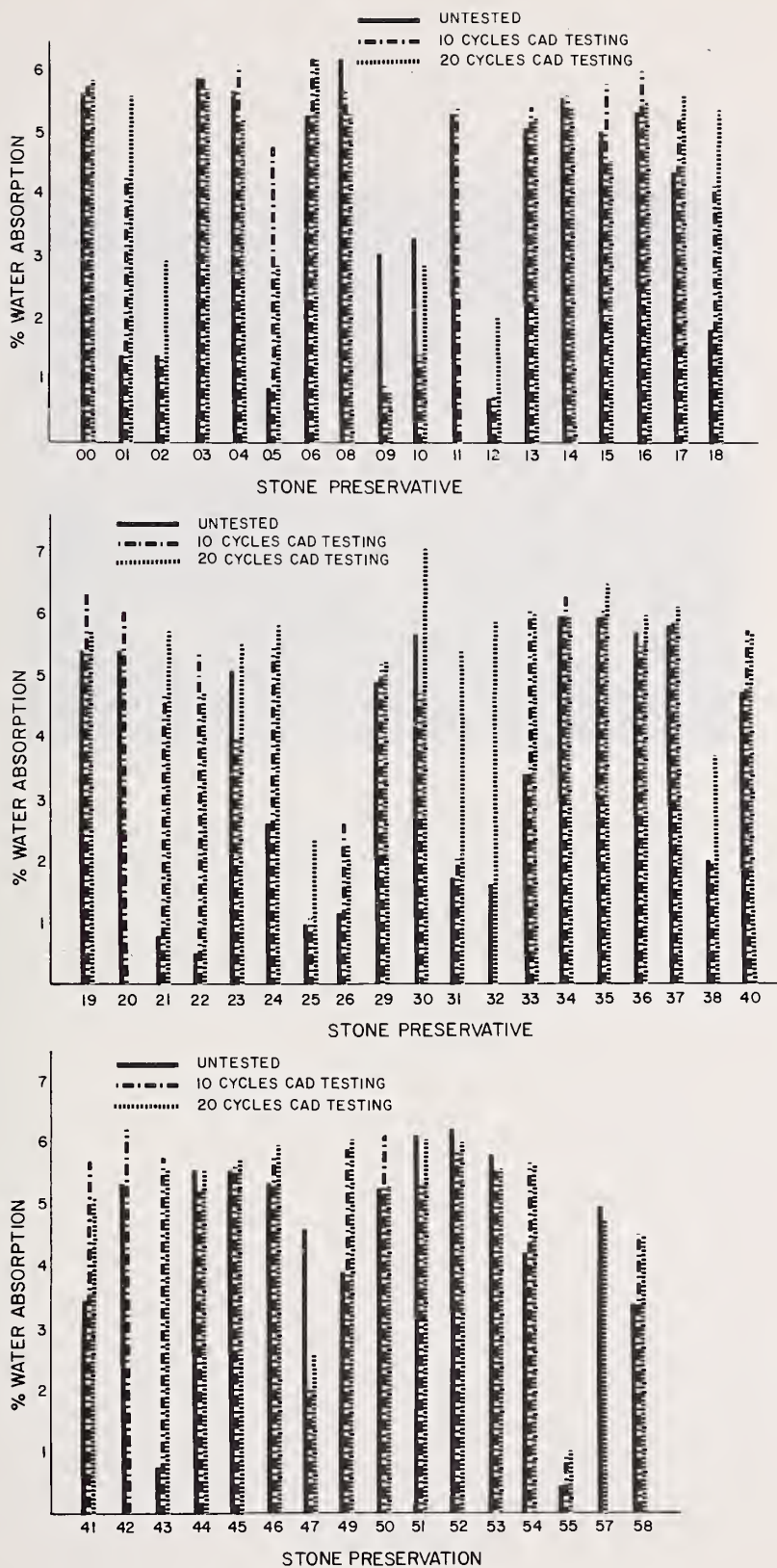


Figure 9. Water Absorption (3.2.3): Absorption of liquid water by stone preservative treated specimens before and after CAD testing.

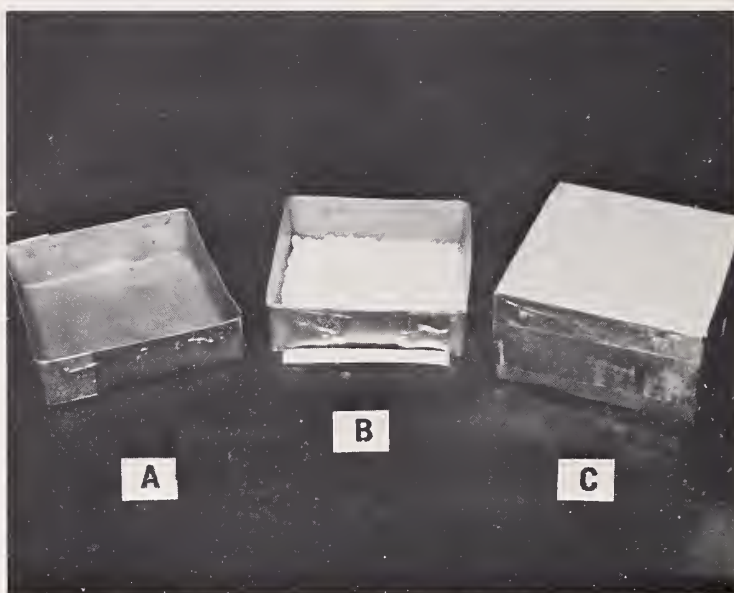


Figure 10. Water Vapor Permeability (3.2.4): Cups used for water vapor permeability measurement -- A) empty cup; B) cup with desiccant; C) cup with specimen attached with adhesive aluminum tape. (Cups approximately 10 cm on edge.)

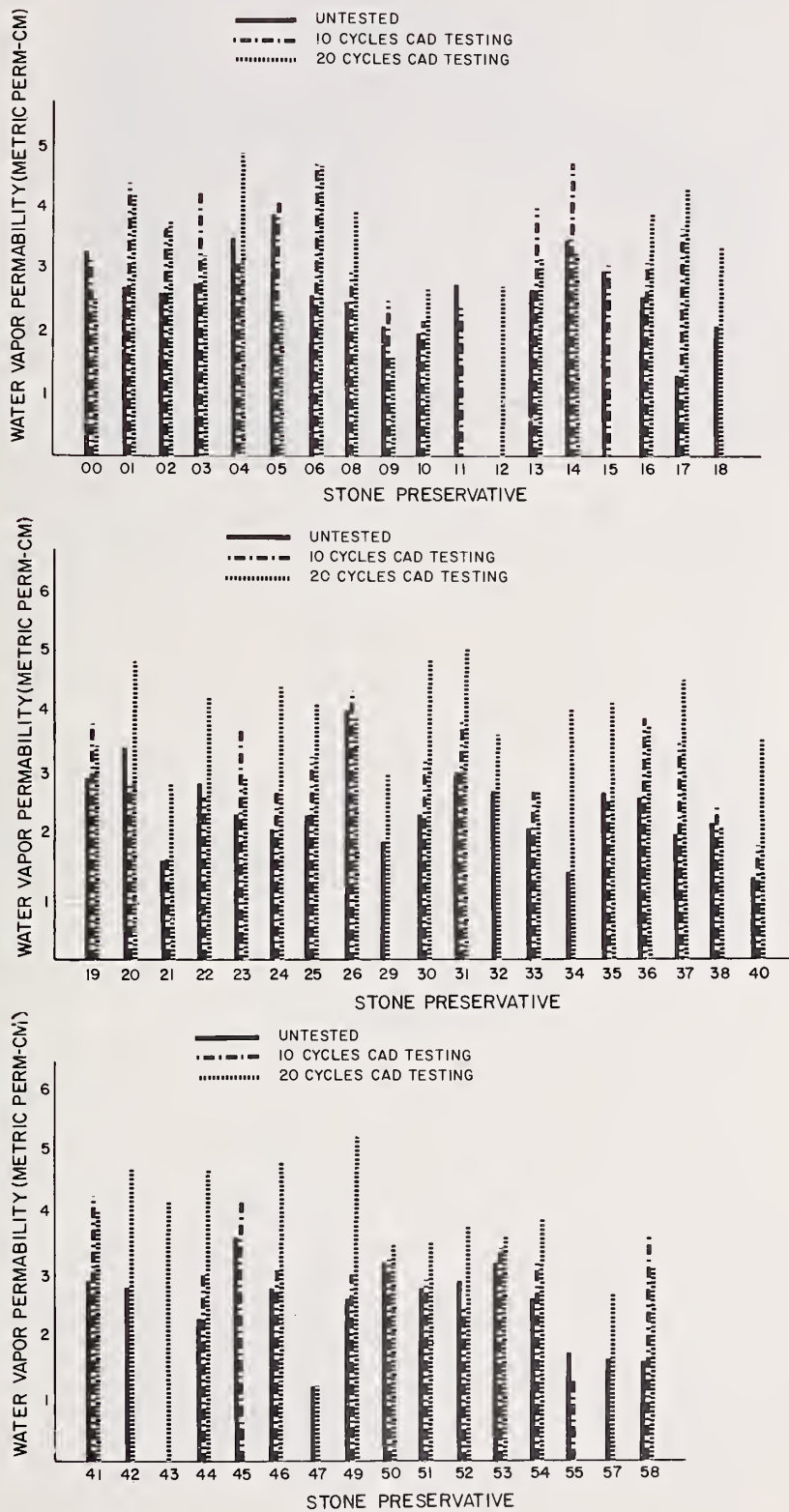


Figure 11. Water Vapor Permeability (3.2.4): Water vapor permeability of stone preservative treated specimens before and after CAD testing.

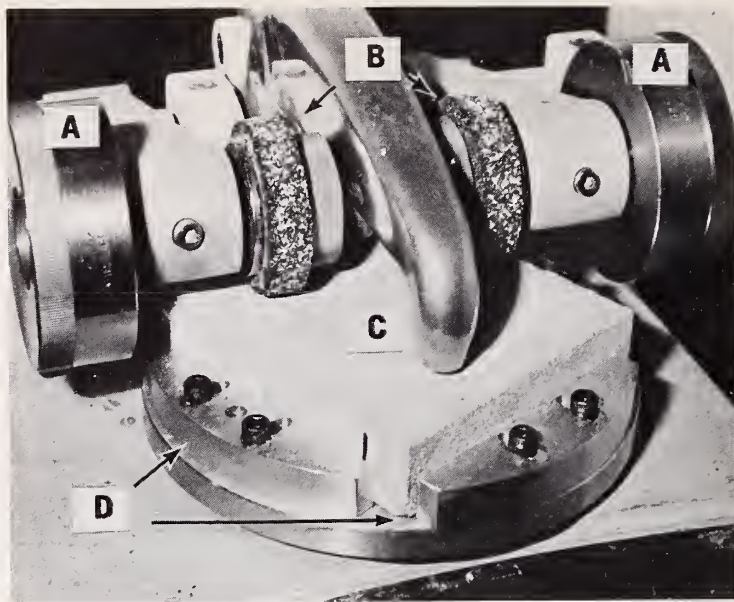


Figure 12. Abrasion Resistance (3.2.5): test machine --
A) 1 kg test loads; B) grinding wheels; C) stone test
specimen; D) adjustable sides to hold specimen on rotating
table.

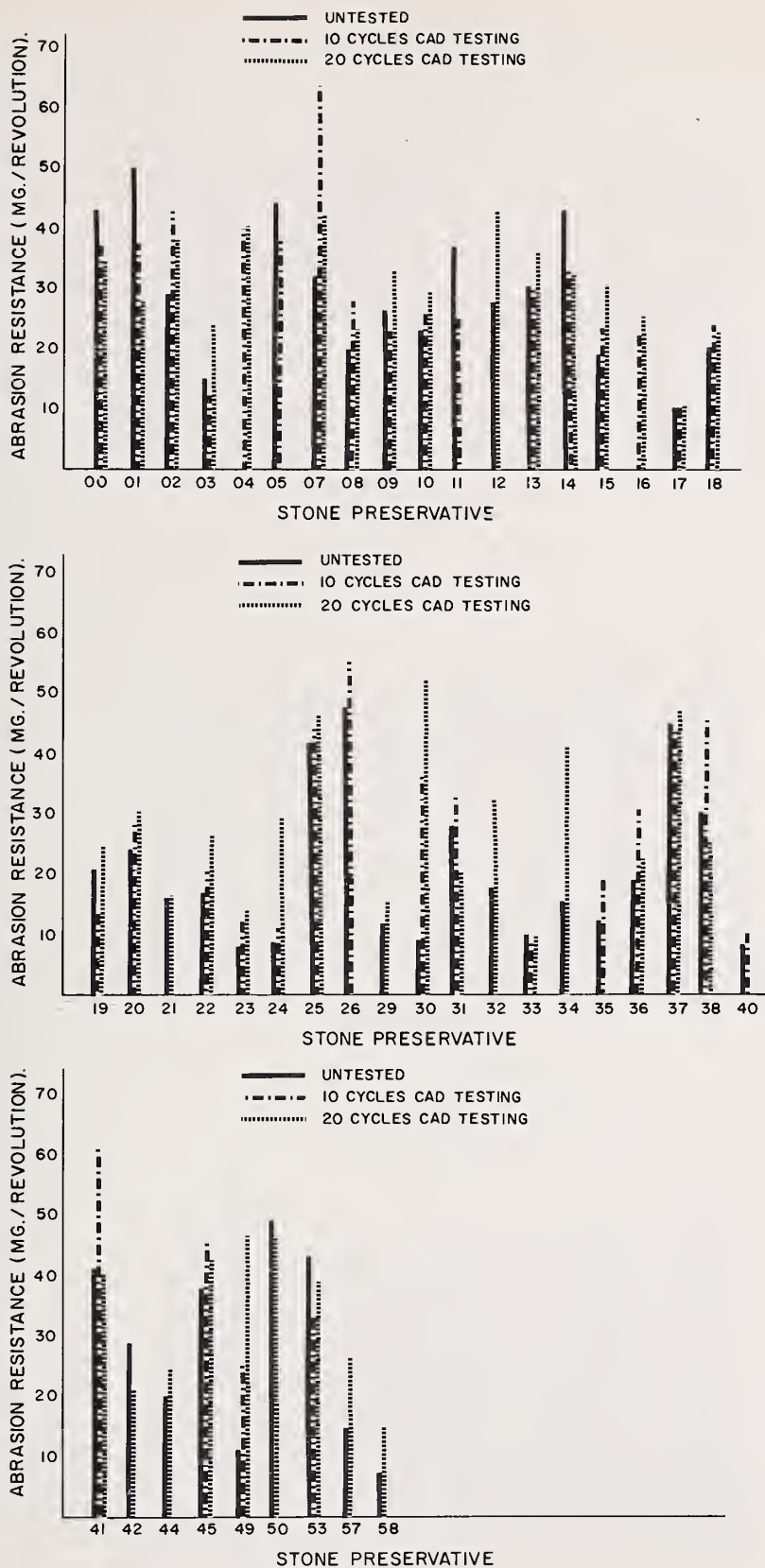


Figure 13. Abrasion Resistance (3.2.5): Abrasion resistance of stone preservative treated specimens before and after CAD testing.

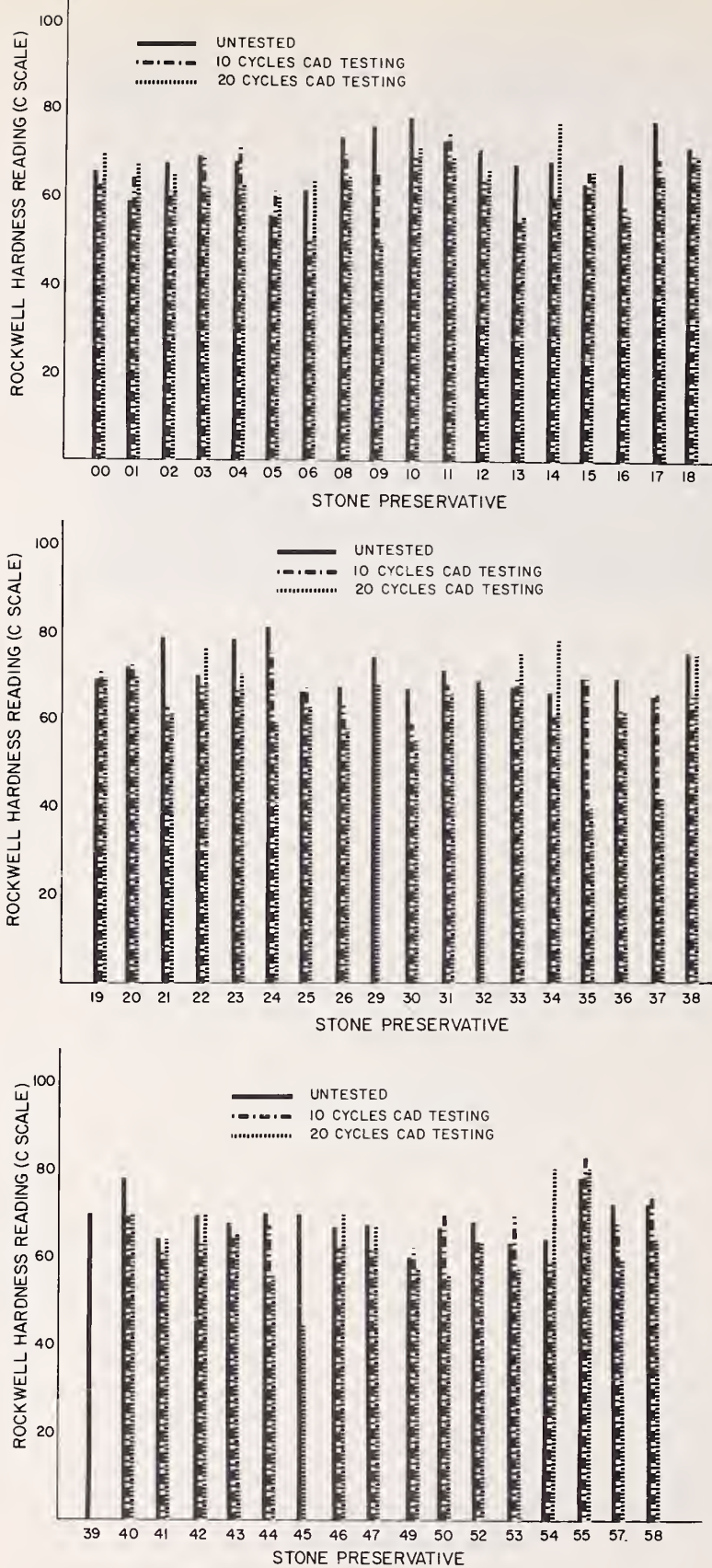
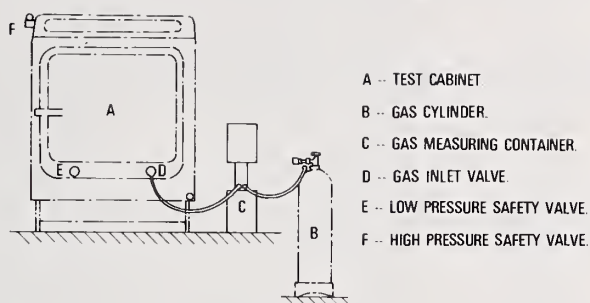
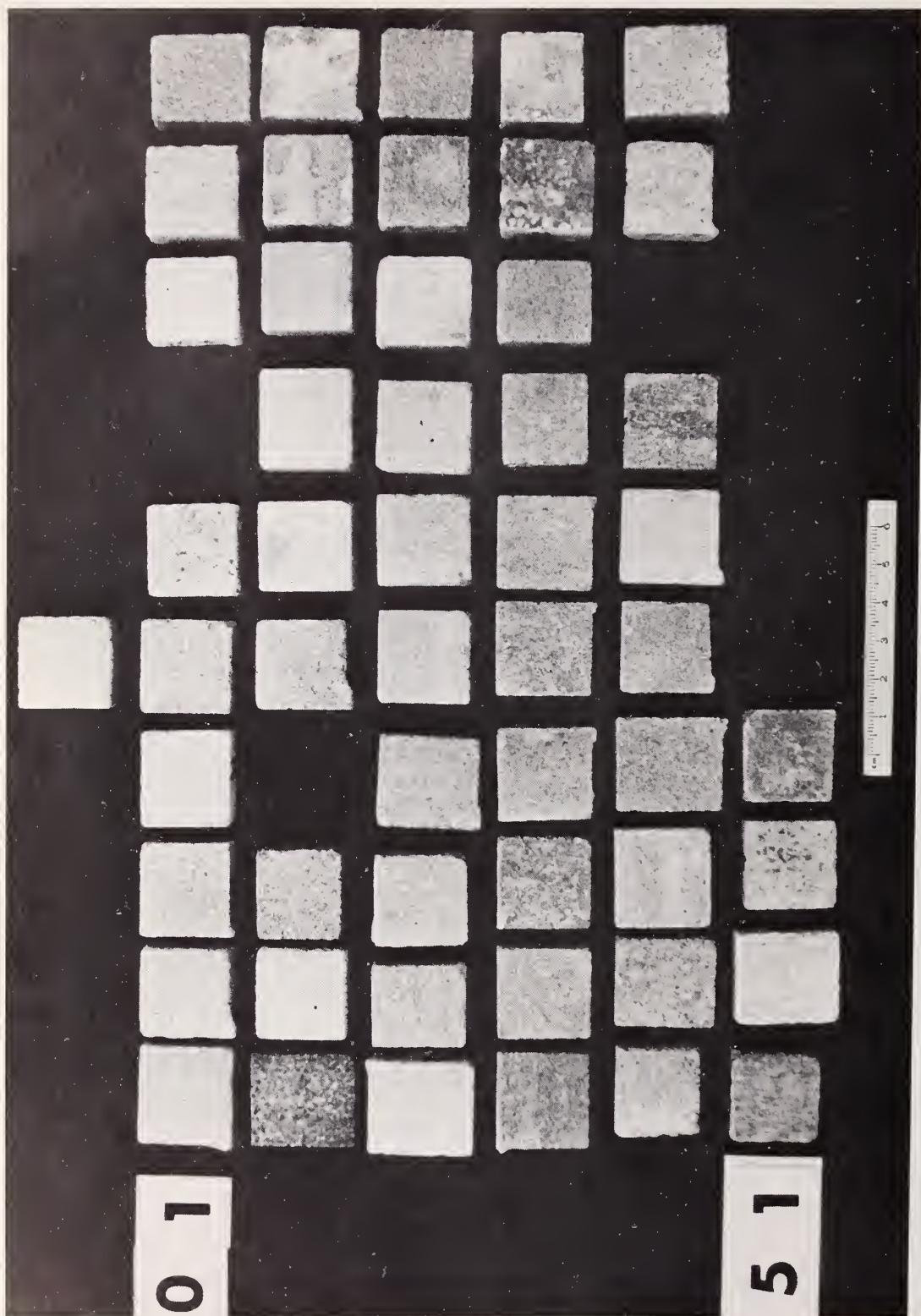


Figure 14. Surface Hardness (3.2.6): Surface hardness of stone preservative treated specimens before and after CAD testing.



SULFUROUS ACID FOG TEST

Figure 15. Sulfurous Acid Fog (4.1); Representation of test cabinet used in sulfurous acid fog test.



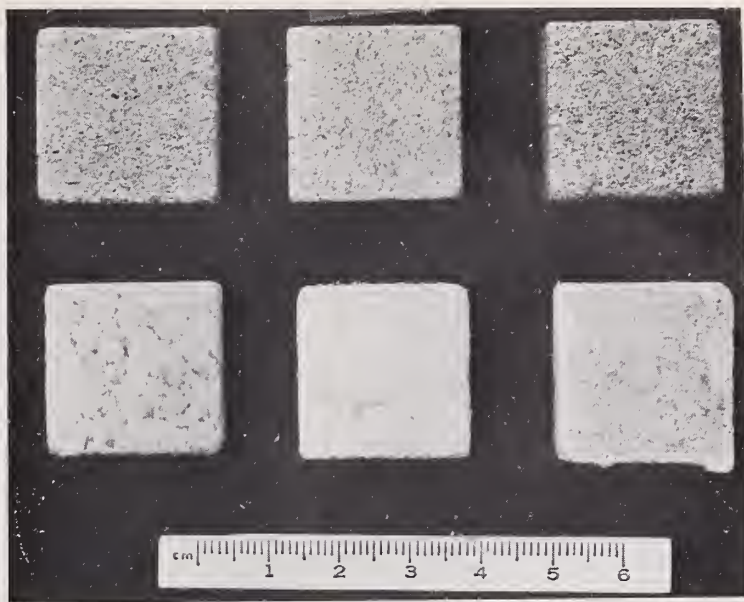


Figure 17. Sulfurous Acid Test (4.1): treated limestone specimens exposed to 49 cycles of sulfurous acid testing. Top row -- treated but unexposed test specimens. Bottom row -- exposed specimens with same preservative treatment as the unexposed specimens above them.

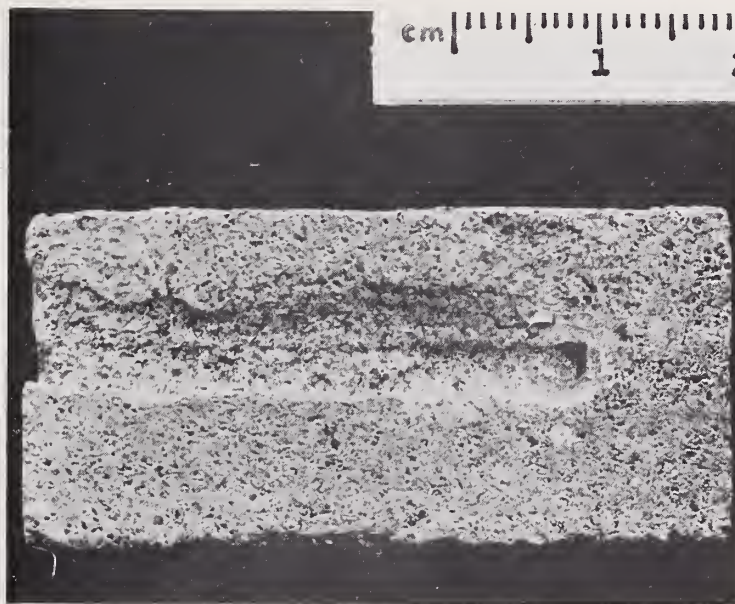


Figure 18. Concentrations of Sulfurous Acid Used in Testing (4.1): Indiana limestone (standard gray) exposed 4 hours to flow of sulfur dioxide saturated water solution over surface (flow rate, 5 ml/min). Weight loss of specimen, -2.50%. Depth of erosion, 2 millimeters. Experiment run at 22°C.

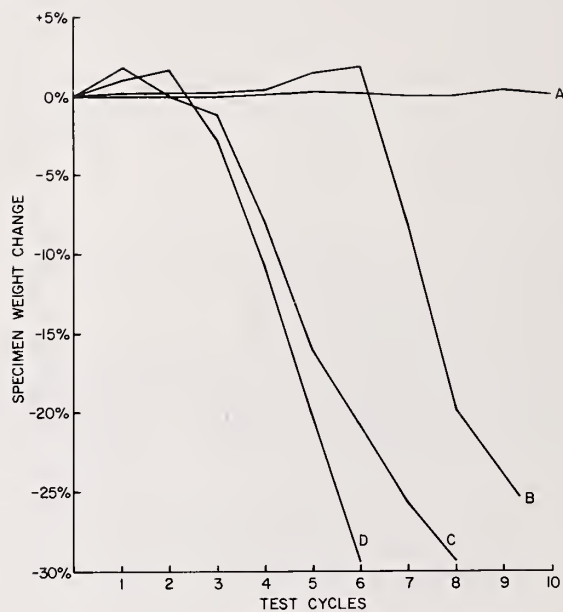


Figure 19. Sodium Sulfate Crystallization (4.2.1): Weight changes in stone preservative treated specimens exposed to sodium sulfate crystallization test. Specimen A -- unaffected after 10 test cycles; specimen B -- onset of degradation after 6 test cycles; specimen C -- onset, then gradual degradation after 1 test cycle; specimen D -- onset of degradation after 2 test cycles.

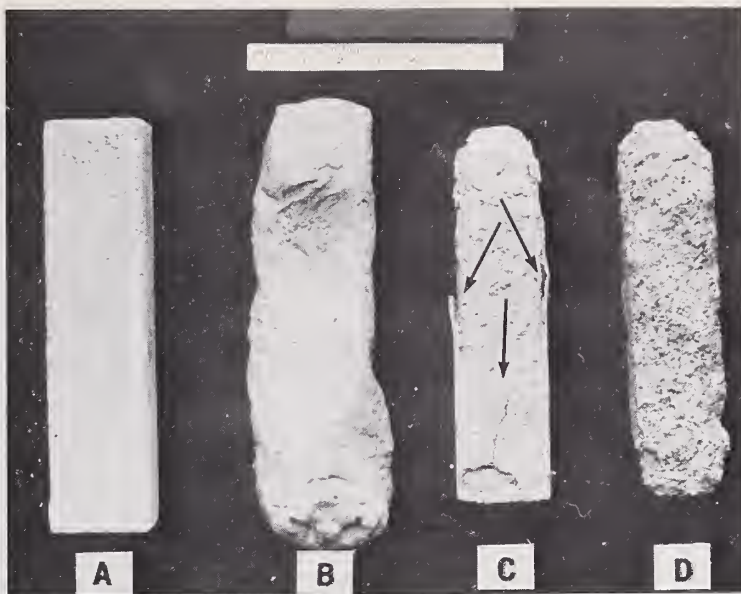


Figure 20. Sodium Sulfate Crystallization (4.2.1): Specimens exposed to varying lengths of cycles of sodium sulfate crystallization test. A) -- preservative treated specimen, intact after 10 test cycles; B) -- preservative treated specimen, preservative coating flexible when wet, stone degraded within preservative shell; C) preservative treated specimen, beginning to degrade after 10 test cycles (arrows indicate spalling and cracking preservative treated layer); D) untreated limestone after 3 test cycles.

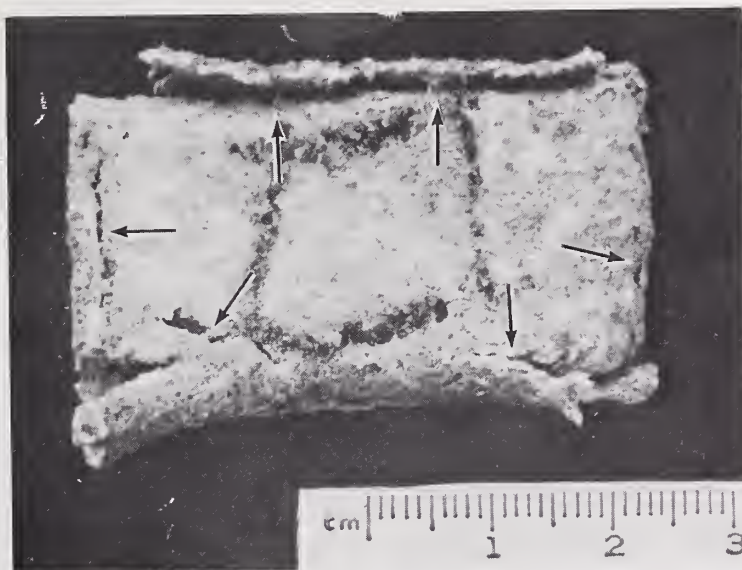


Figure 21. Sodium Sulfate Penetration (4.2.2): Top, untreated surface of preservative treated specimen after 3 test cycles of sodium sulfate penetration. (Arrows indicate spalling layers of preservative treated stone.)

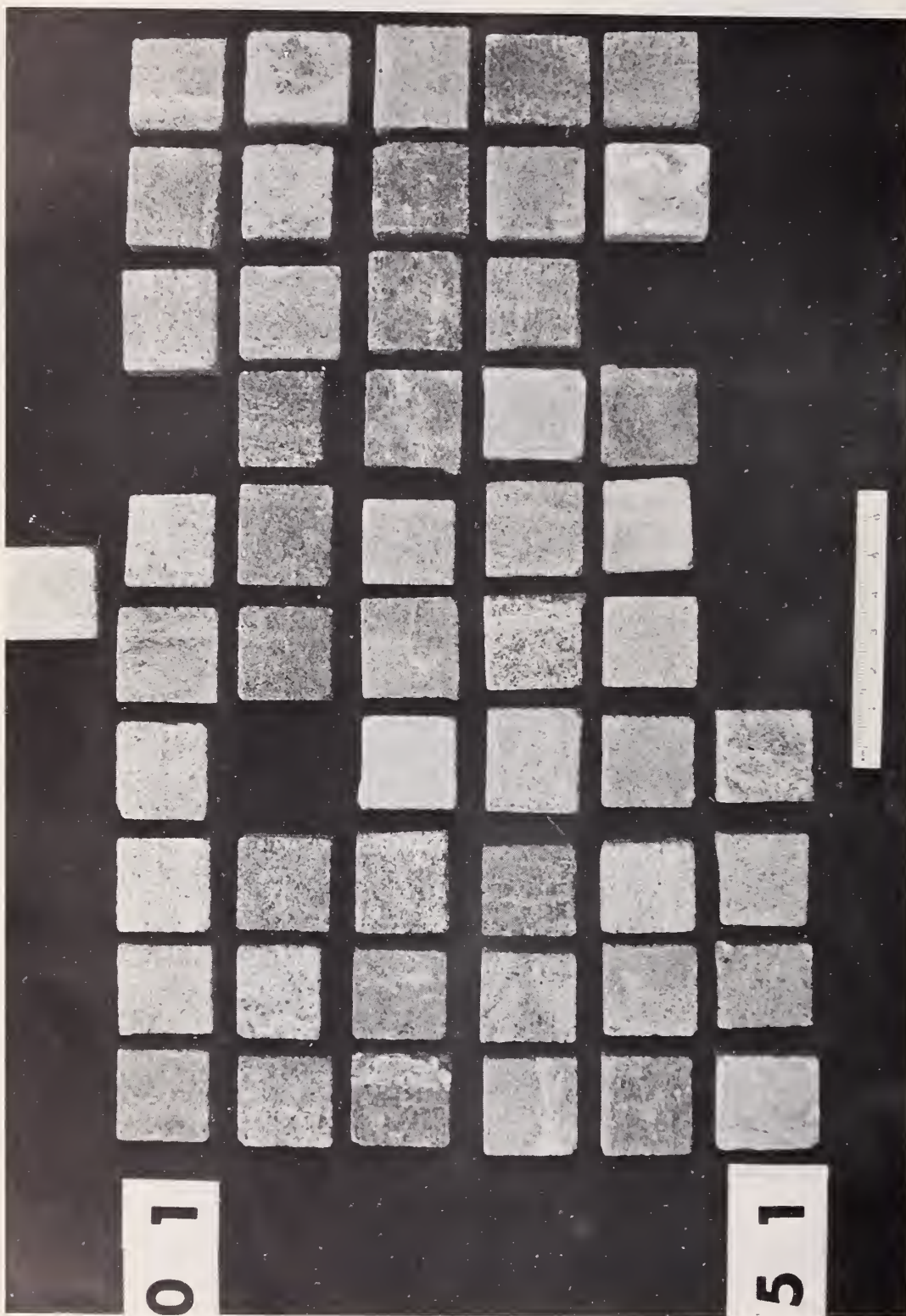


Figure 22. Salt Spray Test (4.2.3): limestone specimens treated with stone preservatives exposed for 6 months to salt spray test. Code numbers for stone specimens begin at upper left with 01 and end at bottom row with 54; first specimens of each row, starting from top, are 01, 11, 21, 31, 41, 51, respectively. Untreated, exposed limestone specimen at top of picture.

- A -- TEST SPECIMENS
B -- WATER BATH
C -- DUCT FOR FORCED-AIR DRYING

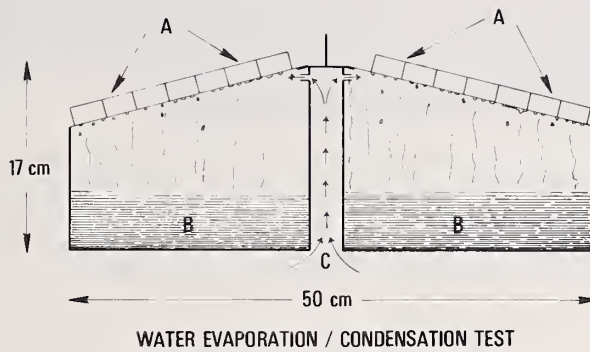
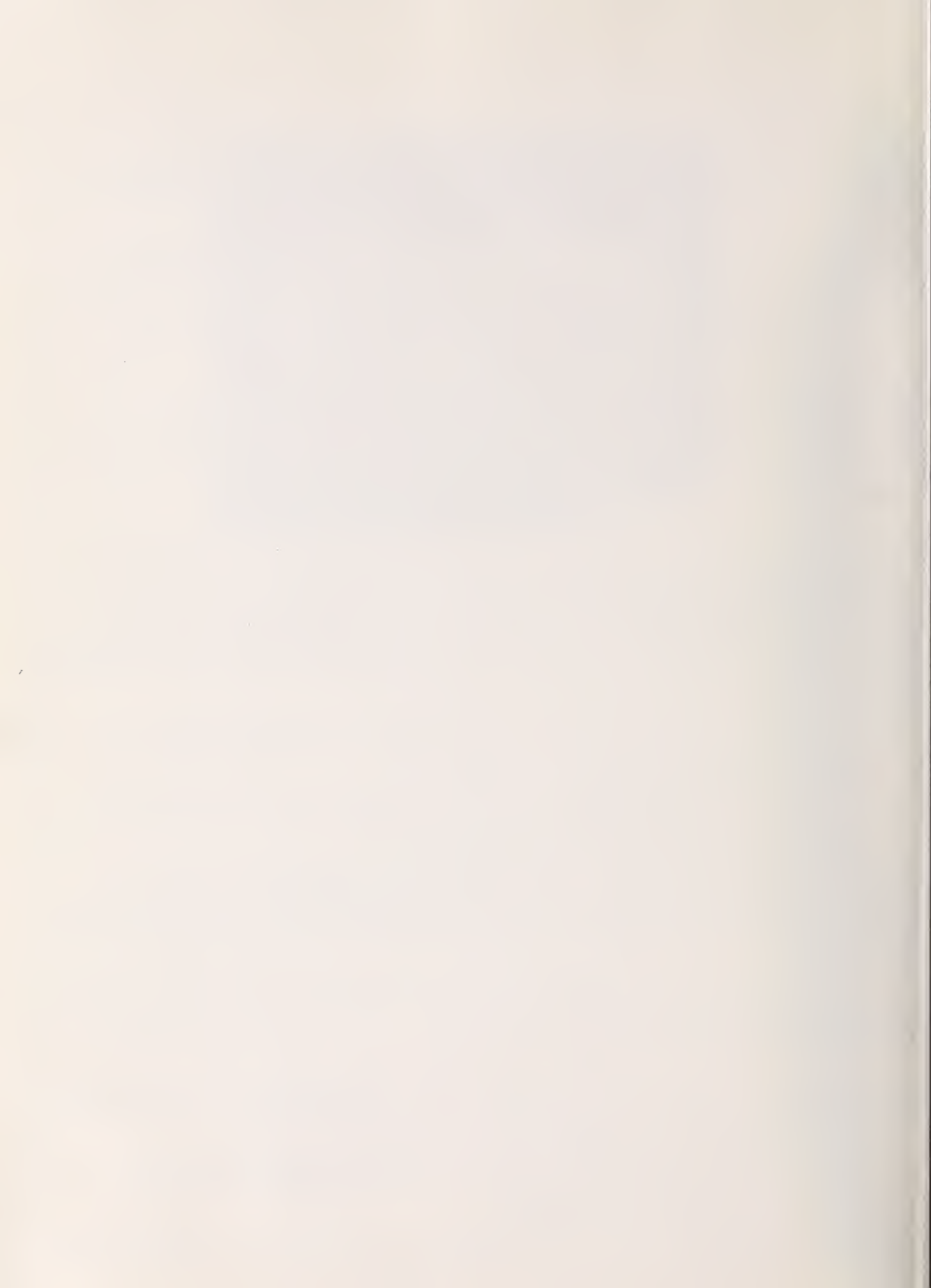


Figure 23. Water Condensation/Evaporation (4.3):
Schematic of water bath used in test.



Figure 24. Water Condensation/Evaporation (4.3): degradation and transport of stone preservative treatment by water condensation/evaporation testing (2400 cycles). Side view of specimen. Arrows indicate direction of water migration from bottom, coated surface of specimen to the top, uncoated surface.

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<p>16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)</p> <p>Although numerous materials have been proposed as preservatives for stone in historic buildings and monuments, their efficacy is difficult to establish. In the work described here, a laboratory research program of accelerated simulated stone decay was used to obtain data on stone preservatives and to suggest criteria for their selection. Over 50 materials usable as stone preservatives were tested.</p> <p>Tests to simulate stone decay were of two types:</p> <ol style="list-style-type: none"> 1. exposure to combined weathering factors using a special test chamber for accelerated decay (CAD), in which chemical attack, salt and water action, and thermal effects were simulated in one operation; 2. exposure to single causes of stone decay using sulfurous acid fog, sodium chloride fog, water condensation/evaporation cycling, sodium sulfate penetration and crystallization, and ultraviolet radiation. <p>Methods for measuring the effects of the exposures are given together with the test data; these have been used to set limits of acceptable performance in preliminary performance criteria for the selection of stone preservatives. The behavior of each stone preservative tested in meeting these criteria is given. No one stone preservative met all criteria.</p>			
<p>17. KEY WORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)</p> <p>Accelerated laboratory testing; performance criteria; stone decay; stone preservatives.</p>			
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