MCCM

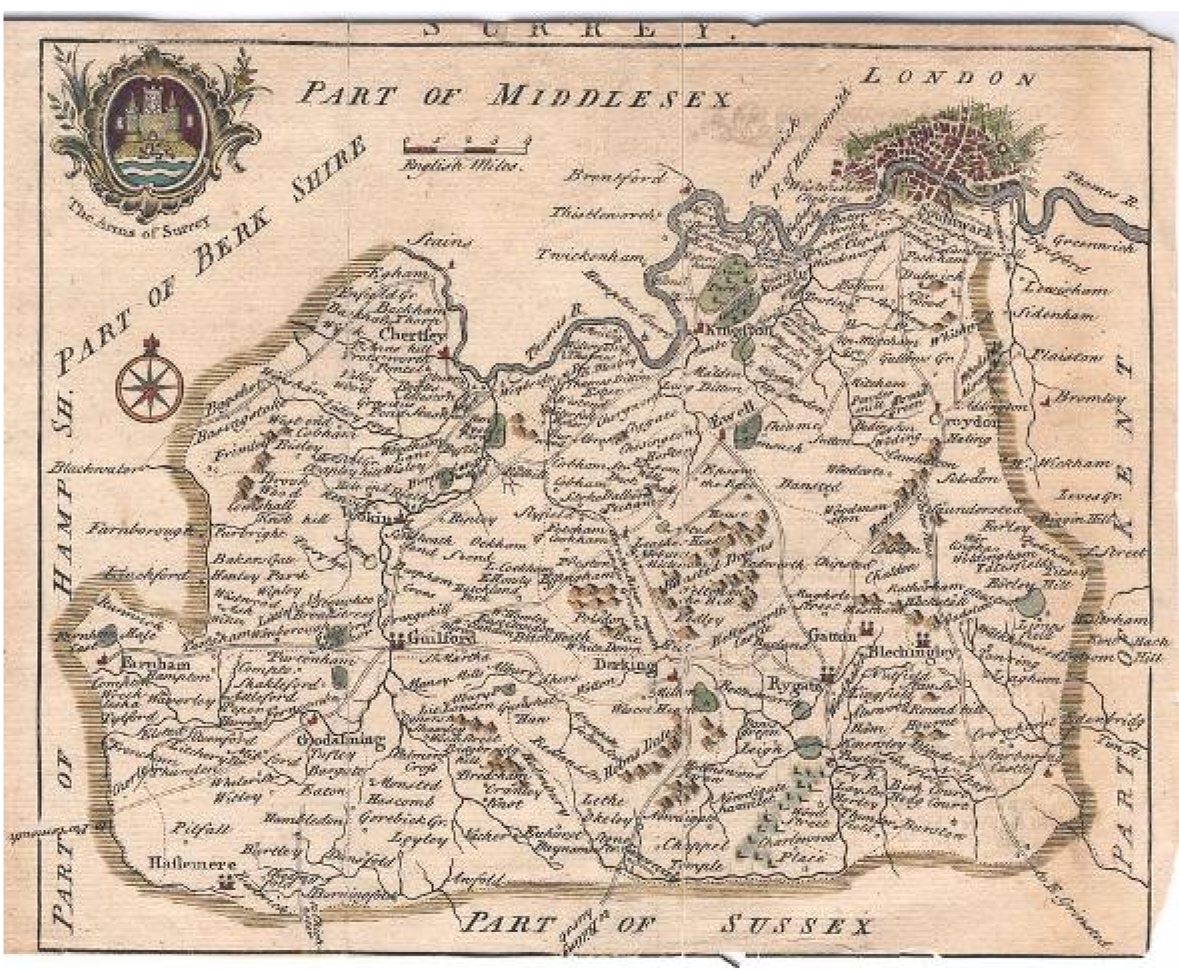
MCC MATERIALS, INC.

IRVING SLAVID
PROF. NORMAN R. WEISS

REIGATE STONE TRIALS

SEPTEMBER 2002

WINCHESTER PALACE
HM TOWER OF LONDON
WESTMINSTER ABBEY
HAMPTON COURT PALACE



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1. Introduction

This report summarizes a multi-year project on the conservation treatment of Reigate stone, undertaken by MCC Materials, Inc. (Colebrook, Connecticut, USA) for a consortium consisting of Historic Royal Palaces, Westminster Abbey and English Heritage. Our focus was on assessment, both in the laboratory and in the field, of the utility of a formulation recently invented by us for the stabilization of limestone and marble. The developmental research for this product, called HCT, began in 1996. Details of the chemistry of the product are provided in section 3., "HCT fundamentals".

In November 1998, brief discussions concerning the status of HCT research were held with Susan Bradley (British Museum), John Stewart (National Trust), John A. Fidler and Jeanne Marie Teutonico (English Heritage), and Dr. Clifford A. Price (Institute for Archaeology, University College, London). With specific reference to the problems of architecture and outdoor sculpture in the UK, both Price and Teutonico suggested that we consider some testing on Reigate stone. Teutonico--now with the Getty Conservation Institute--informed us of a study on Reigate stone for HRP that was underway.

The first public presentation on HCT, which is now sold worldwide by ProSoCo, Inc. (Lawrence, Kansas, USA), was made at the 5th International Symposium on the Conservation of Monuments in the Mediterranean Basin, held in Seville, Spain, on 5-8 April 2000. Immediately thereafter, we met again with Dr. Price, and with Bill Martin (EH), who was able to provide us with a large research sample of Reigate stone removed by HRP from the White Tower (HM Tower of London) earlier in the year (see Table 1 for all stone identification).

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MCC Materials began laboratory testing on this sample in May 2000, in our facilities in Connecticut. On 1 August 2000, Martin confirmed that HRP would include HCT in a set of field trials. At this point, EH was working to establish similar trials with us at Eltham Palace, but later in that year shifted their focus to Winchester Palace, in Southwark.

On 11 December 2000, we examined the south wall of the Great Hall (prob. ca. 1220) at Winchester Palace with Eleni Loizides (EH), and viewed the proposed trial areas. We then met at HM Tower of London with Richard Roberts (HRP), and the consultants on the Reigate Stone Recording and Research Project, Keith Garner, Robin Sanderson and Paul Sowan. (Garner is HRP's consulting architect and Reigate Project Co-ordinator. Sanderson and Sowan are London area geologists; Sanderson is the project's Consultant Geologist, and Sowan is Special Advisor on the archaeology of the Reigate stone mines.) We were shown the proposed trial area on the lower part of the south face of the Bell Tower (ca. 1190), and were also given an opportunity to examine several areas of previous stone treatment, including the interior of the Wakefield Tower, where some stones were consolidated experimentally with Brethane in the 1970's.

Also attending that meeting at the Tower, by invitation of Roberts, was George Burroughs (Westminster Abbey). On the following day, December 12, we visited the Abbey with Burroughs and Vanessa Simeoni (Westminster Abbey), and examined their proposed trial area, a small doorway on the upper wall (an apparent survival from the time of Edward the Confessor, ca. 1065) along the south side of the Cloister. They provided us with some additional Reigate samples and with two testing reports prepared by Sandberg (London, England).

On 13 December, we met with Roberts, Jo Thwaites (HRP), Garner and Sanderson at Hampton Court Palace. This group examined numerous features of Reigate stone in the Wolsey/Henry VIII portions of the Palace, and the blocks reused by Wren in the 1690's. We viewed the proposed trial area, the Reigate stone mullion heads of a window bay at the east end of Tennis Court Lane (dating to 1532). Roberts provided us with small samples of Reigate from Whitgift Almshouses and Chipstead Church. At this time, we also discussed the construction of two mock-up walls incorporating Reigate stone, one at Hampton Court and one at the Tower.

On 23 January 2001, MCC Materials sent reports to HRP, EH and Westminster Abbey, summarizing our laboratory research on Reigate stone, and defining the most appropriate treatment as HCT followed by an ethanol-based version of ProSoCo's Conservare OH. Our testing methods, performed on several different Reigate stone samples, included cyclical freeze/thaw, quantitative microabrasion, and pH-monitored acid rain simulation; additional data were submitted by Weiss in mid-March, during a brief visit to London (see section 4., "Laboratory evaluation of treatment").

The two mock-up walls, recently completed (and noticeably damp), were inspected at that time. The rubble stone wall at the Tower was built in the south moat. The Reigate stone included in it is from an interior wall of the New Armouries; additional material from this source was inspected in a storeroom, and some samples selected for further research. The brick and stone wall at Hampton Court was built in a service area to the north of the Pass Office. The Reigate stone is from Chipstead Church, Gatton Church, and Royal Earlswood Hospital.

Table 1: Reigate sample identification

	SOURCE
T.1	Tower of London, White Tower, S elev., nr. SW corner large sample, slight deterioration
T.2	Tower of London, New Armouries, interior wall
A.1	Westminster Abbey, interior of NW Turret of North transept fragile, scaling on rear face
A.2	Westminster Abbey, inside cloister (core), sound
A.3	Westminster Abbey, Cloister roof (core), sound
A.4	Westminster Abbey New, saw cut surface w/ traces of adhered mortar
X.1	Whitgift (from HRP), sound
X.2	Chipstead Church (from HRP) ornamental carved surface, incipient scaling in cross section

Following the granting of scheduled monument consent (required for the EH and HRP sites), field application of HCT was carried out on 26 April 2001 (Winchester Palace), 27 April (HM Tower of London and Westminster Abbey), and 1 May (Hampton Court). Details of the design and execution of the treatments are presented in section 5., "Field application/ Evaluation".

Participating in the work were David Boyer (ProSoCo) and the staff of Fisher Research (Enfield, England), ProSoCo's UK manufacturer, along with Simeoni and Thwaites.

On 23 June 2001, Slavid visited the Bell Tower with Jeremy Ashbee (HRP). The control (untreated) stones were considerably more "sugary" to the touch than those treated with HCT two months earlier. (At this time, Conservare OH treatment had not yet been done.)

We returned to London on 30 October 2001 for follow-up examination and some further treatment application. Completed trial areas at Winchester Palace and Westminster Abbey were examined on 30 October and 1 November, respectively. At Hampton Court, we examined the trial area on Tennis Court Lane and a set of hand samples on 2 November, and completed treatment of the mock-up wall. We examined the Bell Tower trial (now after OH treatment) on 3 November, and carried out treatments on the mock-up wall in the south moat.

2. Reigate stone

Reigate stone was mined in a band that runs east to west along the southern edge of the North Downs, in northeastern Surrey. It is a fine-grained rock of the Cretaceous Upper Greensand formation. Also known as Gatton stone, firestone and hearthstone, Reigate was used extensively in London and the Southeast from the 11th century onward, but largely fell out of favor by the time of Wren, who noted its poor durability.

A recent article by our colleagues Sanderson and Garner discusses the mineralogy of Reigate stone in the context of the patterns of weathering observed in the HRP study. It is attached to this report as Appendix 1 (Sanderson, Robin and Keith Garner, "Conservation of Reigate stone at Hampton Court Palace and HM Tower of London", J. Arch. Conservation, 7 (3), 7-23 (2001)).

Using optical microscopy and x-ray diffraction (XRD) analysis, they studied numerous fragments from historic buildings, along with core samples taken from the mines in 1999-2000. The predominant mineral phase is cristobalite (39 to 63% by volume), with some opal and quartz. (Both cristobalite and opal are relatively unstable forms of silica.) Micritic calcite ranges from 9 to 40% (by volume), unevenly distributed, along with the distinctive, dark green mineral glauconite, and (in some samples) up to 17% of smectite, a swelling clay.

Sanderson and Garner also report porosity ranging from 27.1 to 41.9%, saturation coefficients between 0.80 and 0.90, and moisture-induced expansion that can be an order of magnitude greater than for most English building stones (op. cit., pp. 17-18). In a private communication (undated), Sanderson shared with us some water absorption data, including a value of 14.9% for a sample that he took from the White Tower.

Our basic analytical data on three Reigate samples (designated T.1, A.4 and X.2) are presented in Table 2. We initially measured % acid solubles and % water absorption. The former involves digestion (after crushing) in 3M hydrochloric acid. The latter was done as a modification of ASTM C 97, in which the specimens are submerged in water for 48 hours.

Our values re: acid solubility range from 19.5 to 28.8%. Although we express our results as weight (rather than volume) percentages, these values seem to be in agreement with the Sanderson and Garner data for calcite content. Their volume % calcite for the White Tower, for example, is 29; our weight % for Reigate T.1 (White Tower, nr. SW corner) is 28.8.

We have reported three sets of water absorption data for Reigate T.1. The first two experiments--4 and 8 specimens, yielding values of 13.0 and 13.6%, respectively--were performed by air drying for several weeks, then soaking. The third experiment was performed on 5 specimens that had been stored in a dessicator at less than 10% RH.

Table 2: Basic data

SAMPLE	# specimens	water absorption	acid solubles	hygroscopic moisture content (7 days, 95% RH)
T.1	4	13.0%		
T.1	8	13.6%		
T.1	5	16.7%		
T.1	1		28.8%	
A.4	8	20.2%		
A.4	1		19.5%	
A.4	3			9.1%
X.2	4	21.8%		
X.2	1		21.0%	
X.2	3			10.0%

The higher value (16.7%) seemed to be indirect evidence of the hygroscopicity of Reigate stone, a characteristic that is easily anticipated from its unusual mineralogy. To confirm this, specimens of Reigate A.4 and X.2 were oven dried, cooled, and placed in a chamber at 95% RH. Hygroscopic moisture contents after 7 days were 9.1 and 10.0%, respectively, nearly half of their 48 hour absorption.

A larger study of hygroscopicity was done with triplicate specimens of five Reigate samples, T.1 and T.2, A.4, and X.1. and X.2. The data are graphically summarized in Figure 1. At seven days, the moisture content of Reigate T.1 was more than 50% higher than the mean value for all other samples. At 61 days, when the test was halted, the Reigate T.1 specimens were saturated (23.0%), at nearly twice the moisture content of T.2 (New Armouries).

A portion of Reigate T.1 was crushed, and stirred in de-ionized water. Solid material was collected by evaporation of the aqueous filtrate to dryness, and identified by XRD analysis as halite (sodium chloride; JCPDS # 5-0628). (This work was done for us by Dr. George Wheeler of the Metropolitan Museum of Art, New York, NY, USA.)

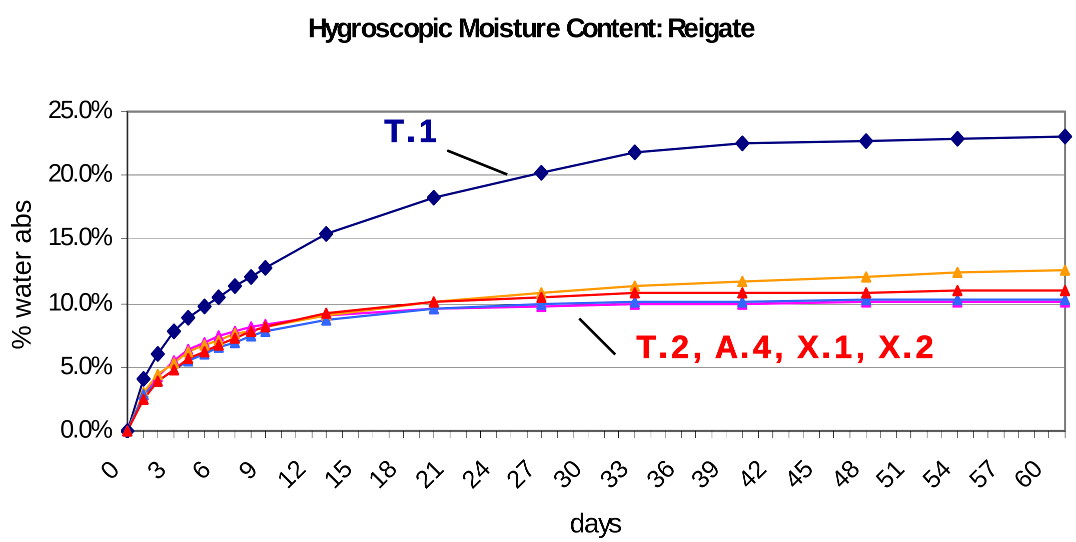


Figure 1

3. HCT fundamentals

HCT is a water-borne conservation formulation. Its development was in response to an observation made in the 1990's by many stone conservators--that treatment of carbonate rocks with ethyl silicate (despite its general acceptance since the 1970's for the consolidation of silicate building materials) gave widely varying results. This seems to be largely attributable to the absence of hydroxyl groups on the surface of calcite and dolomite, the two minerals of which limestones and marbles are primarily composed.

Our research thus began with a relatively simple concept: the formation of a stable hydroxylated conversion layer on carbonate minerals. HCT (which stands for "hydroxylating conversion treatment") works by chemical reaction with calcium ion in the surface of individual grains within the stone, forming a thin, well-adhered crystalline deposit. This material is formed at some depth from the stone surface, as HCT is a low viscosity liquid, incorporating a surfactant. The HCT reaction--resulting in formation of calcium tartrate tetrahydrate (CTT)--is surprisingly rapid, and can be carried out at ordinary ambient conditions.

Several experiments, especially those done on single crystals of calcite (Iceland spar), have established the validity of our fundamental concept that HCT would enhance the adhesion of silicate films to carbonate surfaces. We have further observed that HCT treatment alone can result in measurable improvements in cohesive strength in many limestones and marbles, presumably by intergrowth of CTT at points of carbonate grain contact. A particularly exciting aspect of HCT treatment is the ability of the conversion layer to impart passivation to acid rain exposure, considered to be a critical issue for many historic masonry materials, including mortars and renders.

Treatment consists of three or more saturating applications, followed by a Finishing Rinse that is designed to consume any residual unreacted HCT. A drying time of at least 30 minutes is required between applications. This permits a re-opening of the pore structure by evaporation, giving deep access for the "fresh" HCT of each application. The Finishing Rinse is utilized at least 30 minutes after the last HCT application.

HCT is an aqueous product (pH 4.0 ±0.2) and is odorless. There are no special requirements for handling or clean-up. It contains no carcinogens as listed by OSHA, IARC and NTT, nor are there any hazardous ingredients at concentrations greater than 1%. Product data and material safety data sheets for HCT are included as Appendix 2.

Details of the development of the HCT formulation and of some of our earlier testing are summarized in two documents that we prepared two years ago, attached as Appendices 3 and 4. Appendix 3 is an article published for a conference in Venice, in June, 2000 (Weiss, Norman R., Irving Slavid and George Wheeler, "Development and assessment of a conversion treatment for calcareous stone", in Vol. 2, Proceedings of the 9th International Congress on Deterioration and Conservation of Stone (V. Fassina, ed.). Elsevier Science: Amsterdam, 2000, pp. 533-540.). Appendix 4 is a draft version of a lengthier text which will appear in print at a later date. It was prepared in conjunction with our earlier presentation of HCT in Seville.

HCT was granted United States patent no. 6,296,905 (Slavid, Irving O. and Norman R. Weiss, "Method for protecting and consolidating calcareous materials") on 2 October 2001. Patent applications are in progress in thirty other countries, including the United Kingdom.

4. Laboratory evaluation of treatment

For several years, we have worked to quantify the cohesion effect that we have observed with HCT. In general, it has seemed odd to us that strength testing has played so small a role in the evaluation of stone consolidants since the 1970's. We have therefore made a considerable effort to evaluate a number of test methods--including indirect tension, three-point bending and cross-axial chisel splitting--and currently believe that our most reliable laboratory results are with quantitative microabrasion.

Figure 2: Microabrasion, 5 craters each specimen

This technique was first reported for the evaluation of conservation treatments by Phillips in 1982 (Phillips, Morgan W., "Acrylic precipitation consolidants", in <u>Science and Technology in the Service of Conservation</u>. London: IIC, 1982, pp. 56-60.). Our apparatus is the S.S. White Airbrasive Unit (Model-K). Fine alumina powder is carried in a narrow stream of air, resulting in measurable loss of stone by abrasion. The blasted area appears as a small crater (see Figure 2). To make the process reproducible, the orifice is positioned 20 mm from the surface of the specimen; a steel plate is used as a shield to control the precise time of the blast.

Data for five building stones (including Reigate T.1) currently being tested in our laboratory are presented in Table 3, which includes some other details of the experimental design. The mean weight loss (grams/ crater) for the untreated (control) specimens is divided by the same value as measured for the HCT-treated specimens. This number, which we have called the "comparative soundness", is a measure of the extent to which HCT enhances cohesion. The computed value for Reigate is 2.10, that is, the treated specimens are, on average, more than twice as resistant to abrasion than the controls.

We have also carried out microabrasion testing on specimens of Reigate T.1 treated with the ethanol-based version of Conservare OH and with HCT followed by OH. Treatment with OH only gave a comparative soundness of 1.69, less than that observed for HCT. Sequential treatment with HCT and OH gave 2.16, a bit more than for HCT alone (see Table 3).

Table 3: Microabrasion (Phillips test) on cut surfaces

SS White Airbrasive Unit (Model-K); 40 psi, 5x10 second exposures
AccuBRADE-50, powder flow 7
HCT-treated 3x4 minutes; all specimens at rm temp/ RH for 2 days

Stone	**Mean wt. loss, g/crater**		**Comparative soundness**		
	Untreated	**HCT treated**	**HCT**	**OH**	**HCT + OH**
Monks Park	0.0654 (40 craters)	0.0406 (20 craters)	1.61		
Indiana	0.0308 (40 craters)	0.0235 (20 craters)	1.31		
Reigate	**0.0342 (80 craters)**	**0.0163 (40 craters)**	**2.10**	**1.69**	**2.16**
Unknown marble	0.0194 (50 craters)	0.0146 (25 craters)	1.33	3.28	2.67
Howden Minster	0.0264 (30 craters)	0.0159 (15 craters)	1.66	1.32	2.12

Figure 3 is a graphic representation of more than two months of data on Reigate T.1 in a laboratory freeze/thaw test program that we have developed. Specimens are four sets of 2.5 cm (1 inch) cubes: untreated controls, treated with HCT only, with OH only, and with HCT followed by OH. Those treated with OH are given 28 days for room temperature curing. After weighing, the dried specimens are submerged in water for 48 hours, and re-weighed. Surplus water is drained, and the wet specimens are placed in a freezer at –15^{o}C for 16 hours; thawing is for 8 hours in water at room temperature. Specimens are then re-weighed, and returned to the freezer for another cycle.

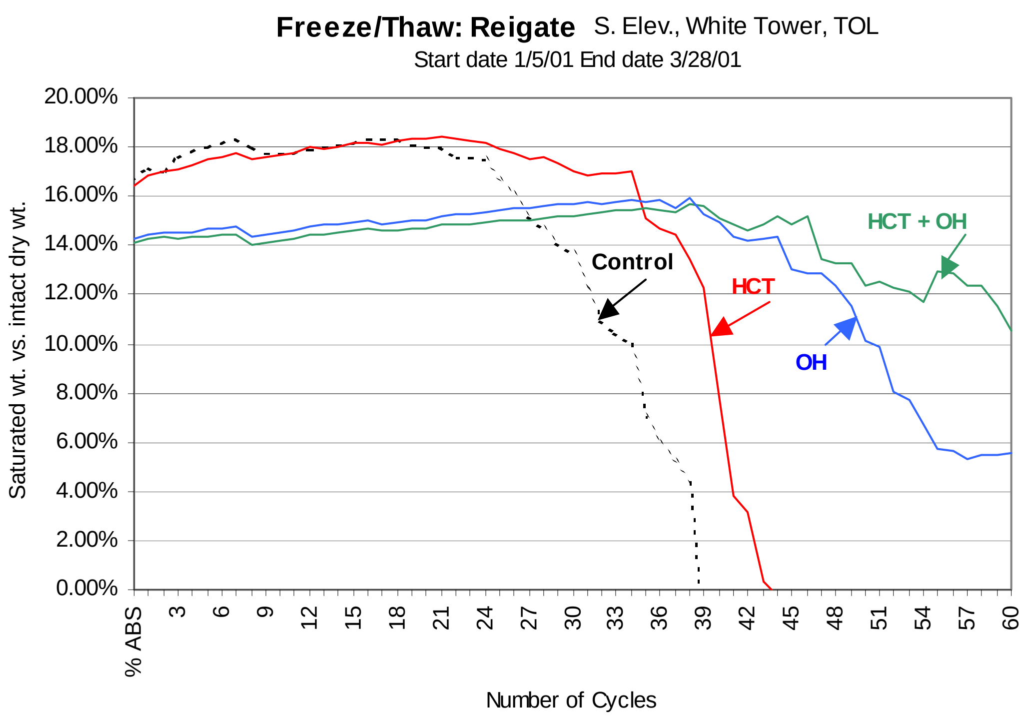


Figure 3

Y-axis values, plotted for each cycle, are computed as the mean wet weight (5 specimens) minus their original (i.e., intact) mean dry weight, divided by that dry weight, times 100. Thus, at the start of the test program, this value is simply the % water absorption (48 hours). As the test progresses, and specimens begin to exhibit some material losses from frost shattering, the diminishing specimen weights are seen as a descending curve.

What we observed initially for all sets is a relatively steady line. For the controls, the loss of material became quite dramatic by cycle 28. HCT-treated specimens did well for an additional 8 cycles, almost 30% longer. The best performance was exhibited by the set treated with HCT followed by OH, remaining relatively intact to beyond 50 cycles (see Figure 4), with the most gradual (i.e., the least dramatic) loss of material as the test was continued to 61 cycles.

Figure 4: Back to front:
HCT (dry) at cycle 34, controls (dry) at 26, HCT + OH (wet) at 50, OH (wet) at 50

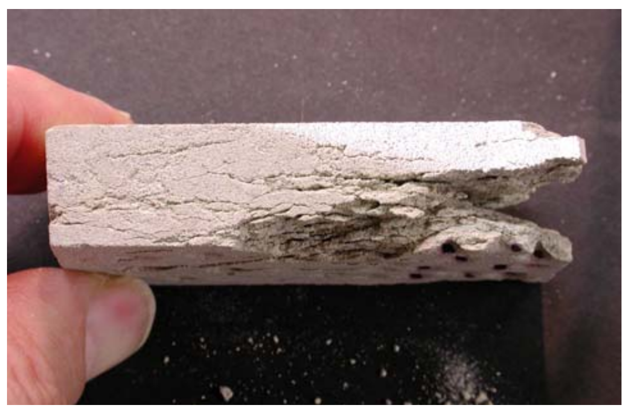
An important observation that we were able to make in our laboratory is that the appearance of specimens subjected to cyclical freeze/thaw is remarkably similar to that of deteriorated Reigate stone on buildings. This encourages us as to the value of our accelerated weathering testing.

A group of photographs (Figure 5) shows a number of aspects of the pathology of Reigate stone, comparing the decay of Reigate A.1 with the laboratory-induced behavior of Reigate T.1. Of particular note is the irregular scaling. (Viewed in cut cross-section, there is also a distinct zone of discoloration in Reigate A.1.)

Reigate A.1, scaling of rear face

Reigate T.1, laboratory induced scaling (freeze/thaw testing)

Reigate A.1, cross section showing incipient scaling



Reigate T.1, laboratory induced incipient scaling (freeze/thaw testing)

Figure 5

Using aggregates derived from Reigate T.2, we have been able to study the progress of the HCT treatment reaction, and to characterize the resistance of HCT-treated Reigate stone to environmental acidity. Crushed stone is sieved to pass a No. 50 screen (0.30 mm) and be retained on a No. 100 screen (0.15 mm), then washed and dried. 4 g of this material is treated with 20 ml of HCT, continuously stirred, for 8 minutes; pH is measured at 1 minute intervals. The solution is decanted, the aggregate quickly dried, and the process repeated twice, each time with fresh solution.

Increasing pH essentially represents neutralization of the HCT by reaction with the alkaline calcite. With repeated treatment on the same particles, the increase is progressively less, as there are fewer HCT-reactive sites left on the mineral surfaces, that is, the conversion treatment becomes more complete (see Figure 6).

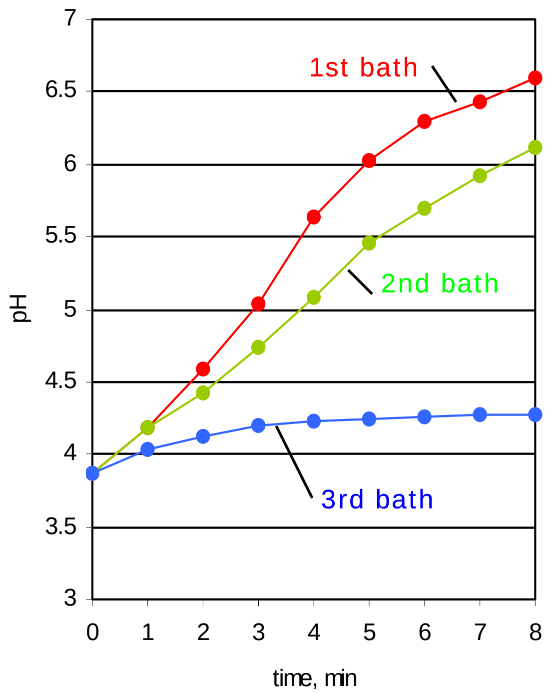


Figure 6: pH monitoring of treatment

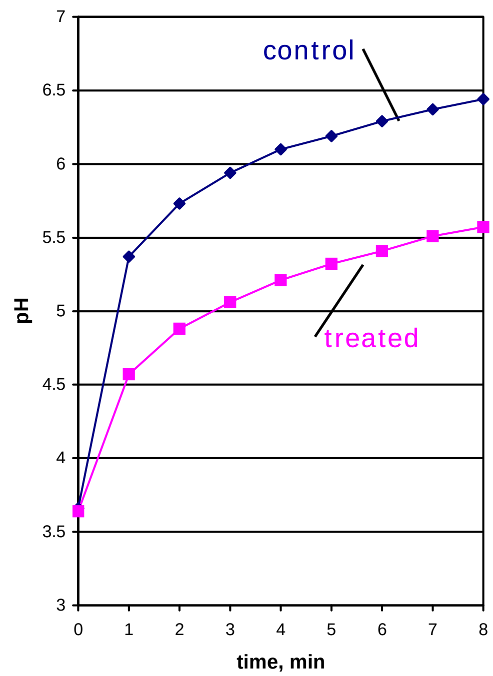


Figure 7: pH monitoring of acid rain simulant

Treated aggregate is then used to study the ability of the conversion layer (CTT) to impart some passivation to acid rain exposure. 4 g of treated and untreated (control) aggregates are separately stirred in 20 ml of an acid rain simulant (pH 3.6). Monitoring of pH (see Figure 7) now gives us a relative measure of the chemical resistance of treated aggregate to the simulant, which is dilute sulfuric acid saturated with respect to carbon dioxide. After 8 minutes, our data show a difference (treated vs. untreated) of 0.87 pH units. As pH is a measure of hydrogen ion activity on a base-10 log scale, this represents a reduction in sensitivity to acid exposure (via HCT treatment) by a factor of approximately 8.

We have also had excellent (and dramatic) results re: acid resistance by another test method. Drops of 0.09 M sulfuric acid (pH 1.1) placed on HCT-treated Reigate aggregate show no reaction. By comparison, evolution of carbon dioxide gas--evidence of reaction of the acid with calcite--for untreated Reigate aggregate is vigorous (see Figure 8). More importantly, a difference between treated and untreated Reigate can be observed when drops of 0.09 M sulfuric acid are placed on larger specimens of stone.

Reigate aggregates, untreated on left, HCT-treated on right

Reaction with .09M sulfuric acid, pH 1.1

Figure 8

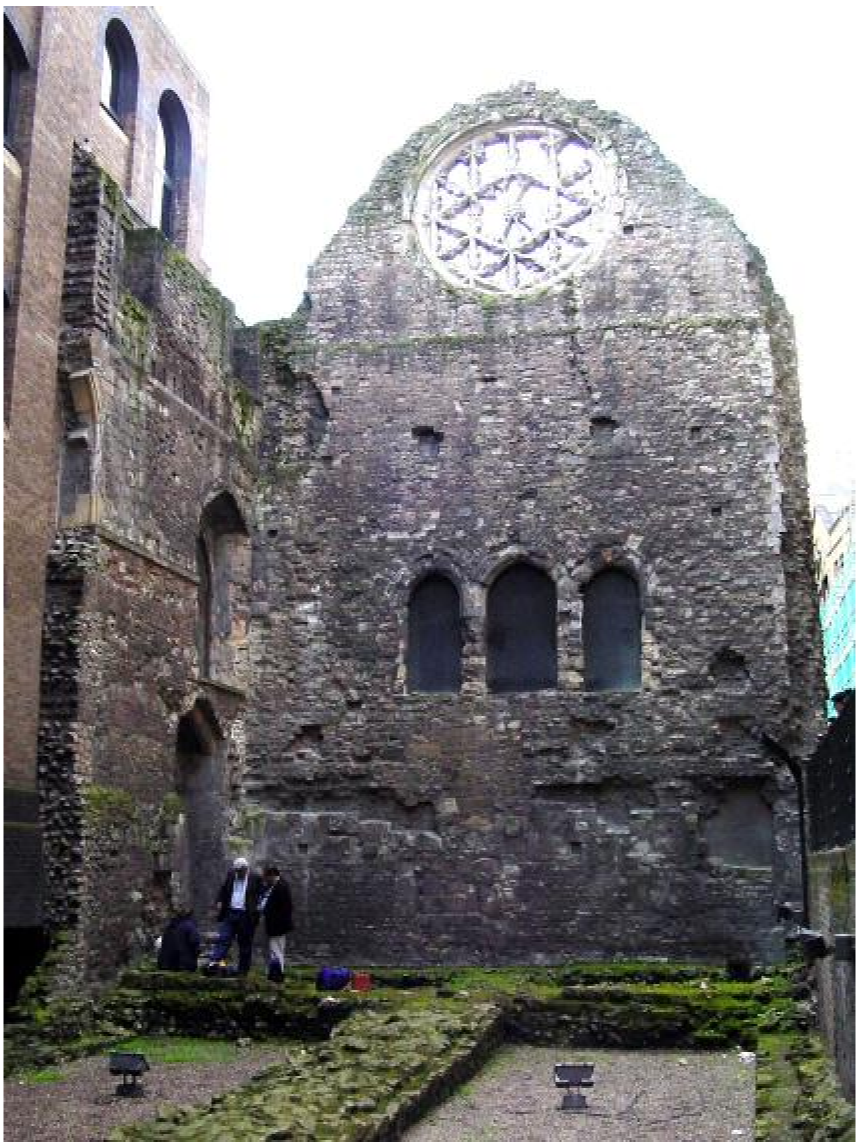
Supplemental technical evaluation of the treated-related improvement of Reigate stone was done with the use of pulse velocity ultrasound. As this testing was performed in the field, it is presented in 5.1 and 5.3 of section 5., "Field application/Evaluation". Our apparatus is the portable PUNDIT, by CNS Electronics (Borehamwood, England). The instrument records the transit time, in microseconds, of a pulse between hand-held transducers. The distance traveled, in mm, is divided by the time, to compute the velocity of sound in km/sec. In general, ultrasound velocity correlates with material characteristics such as compactness and cohesive strength. Comparison of pre– and post-treatment values at these sites shows substantial improvement in velocity as a result of treatment with HCT (36%, Winchester Palace) and HCT followed by OH (40%, Westminster Abbey).

5. Field application / Evaluation

5.1 Winchester Palace

26 April 2001

Trial area was near the center of the south wall of the Great Hall. Reigate stones designated for treatment constitute the lower portion of the east (left-hand) jamb of the lowest window opening. (Another approved area, at the north end of the west elevation, was determined to be unsuitable for trials, due to its proximity to the roadway along Clink Street.) HCT treatment was carried out by: Irving Slavid and Norman Weiss (MCC Materials) and David Boyer (ProSoCo). Also present as observers were: Eleni Loizides (English Heritage); Richard Roberts, Jo Thwaites and Jeremy Ashbee (Historic Royal Palaces); and Iwan Fisher and Jagoba Mariscal (Fisher Research).



Winchester Palace (cont.)

Description/Condition: Three (designated 5, 6 and 7) of the seven Reigate stones in the trial area to be treated with Conservare HCT. (This portion of the area is approx. 45 by 50 cm.) Stones 1 and 7 to be treated with Conservare OH; 2, 3 and 4 to serve as (untreated) controls. Color: all are pale grey-green, with considerable surface discoloration. Ancillary wall materials: Caen stone, brick/concrete repairs. Previous conservation treatment: there is no documentary record, but the hardness of the (flaking) surface crust suggests some prior chemical treatment. In some areas, the crust is mechanically unstable; flakes could be detached with the slightest manual pressure. Much of the lighter-colored stone beneath the crust (and where it has been lost) is friable and “sugary”.

Winchester Palace (cont.)

Surface preparation: Loose crusts and granular stone debris were removed from the surface with a long-fibered brush. Brushing was carried out slowly and with very light pressure, the process referred to by EH staff as “rationalizing” the surface. This work was done on all seven stones until a finger placed directly on the surface did not cause the loosening of any additional grains. Surface conditions made it very difficult to determine ultrasound pulse velocity. Only stone 5 permitted proper placement of the transducers, providing transit time data that could be compared with post-treatment values (see entry for 2 November, 2001, below).

Removing loose surface material by lightly brushing

Applying treatment with low pressure spray

Treatment application: Conservare HCT Hydroxylating Conservation Treatment, 1:00 to 3:50 PM. 16 to 22^{o}C, 54% RH; unstable weather with brief periods of rain. Three saturating applications, initially by brush, but switching almost immediately to low pressure spray. Drying times of 1 to 1 and ½ hours between applications, with 25 minutes after the third application. Conservare HCT Finishing Rinse, one saturating application by low pressure spray, 4:15 to 4:16 PM. All seven stones were then washed down with water; runoff from the untreated portion of the trial area appeared to contain minute particles of stone.

Winchester Palace (cont.)

25 July 2001

Treatment application: Conservare OH (ethanol as solvent), 3 “cycles”, carried out by Richard Turk (Tensid). Application to stones 1 and 7. Total volume used approx. 2 liters.

2 November 2001

Inspection/Evaluation: Present: Irving Slavid and Norman Weiss. Inspection was by touch, with a 10X lens and a 30 X field microscope, and with ultrasound. Control stone 4 was somewhat fragile to the touch after 6 months of weathering.
Stone 1, treated with OH only, was only in slightly better condition. Stones 5, 6 and 7 were considerably sounder than 4, with 7 the best of the group. There was no discernable alteration of surface appearance (color, gloss) as a result of treatment. Summary of ultrasonic testing, stone 5: transducer distance is 126 mm. Apparatus is portable PUNDIT.

Pulse velocity (km/sec), before treatment		2 November data	
	1.27		1.73
	1.30		1.69
	1.28		1.80
mean =	**1.28**	**mean =**	**1.74**

Treated Reigate stone (5) shows a mean velocity increase of 36%.

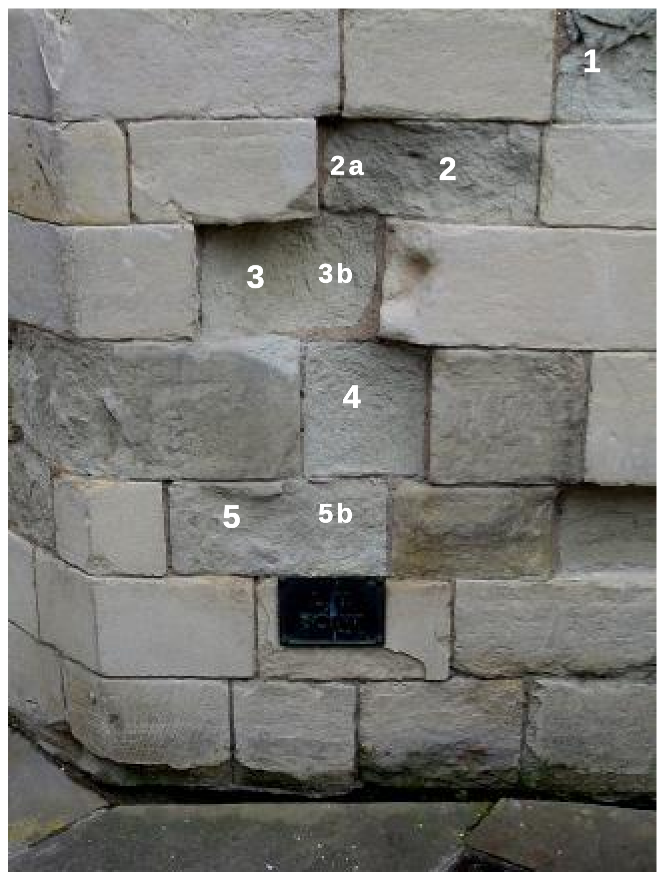
Measuring ultrasonic transit time with PUNDIT

5.2a Bell Tower, HM Tower of London

27 April 2001

Trial area was on the exterior of the Bell Tower, at ground level, facing southeast. Designated Reigate stones are 0.75 to 1.5 m above grade, in a heavily trafficked location, close to the main visitor entry point for the Tower. HCT treatment was carried out by: Irving Slavid and Norman Weiss, and David Boyer. Also present as observers were: Richard Roberts, Jo Thwaites and Jeremy Ashbee; Robin Sanderson and Keith Garner (consultants to HRP); and Anthony Fisher (Fisher Research) and Jagoba Mariscal.

Description/Condition: Three contiguous Reigate stones (designated 2, 3 and 4), arranged vertically, to be treated with Conservare HCT. Reigate stones above (1) and below (5) to serve as controls. A vertical band of treatment with Conservare OH to be done subsequently, so as to superimpose OH on the left-hand portion of 2 (2a), on 3b, on all of 4, and on 5b (5 not given HCT). Depth of stones unknown; thought to have rubble backing. Color: all are pale grey-green. Ancillary wall material: Caen stone. Previous conservation: there is no clear physical evidence or documentary record of prior chemical treatments. The three stones are in generally similar condition. Significant loss of material (15–50 mm) with concave weathering in each stone block. Current surface is friable, with only minor scaling and delamination. There are no gypsum crusts. The advanced state of decay may be in part because these stones are touched by visitors.

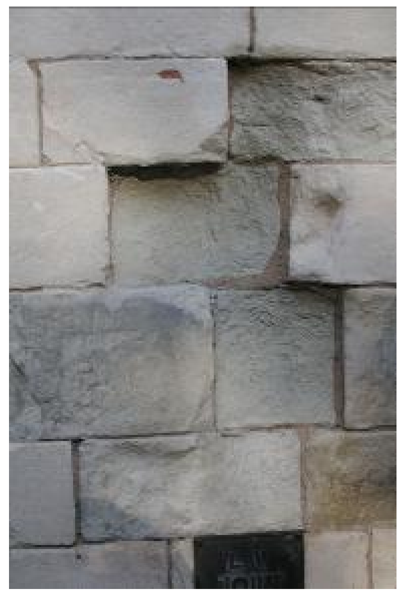


Bell Tower, HM Tower of London (cont.)

Surface preparation: Fine dust was removed from the surface of the test stones and adjacent control stones with a soft, long-fibered dusting brush, as described in section 5.1, above.

Treatment application: Conservare HCT Hydroxylating Conversion Treatment, 8:30 to 11:24 AM. 12^{o}C, 43% RH; partly cloudy to sunny. Three saturating applications done by low pressure spray, with approximately 1 hour drying time between applications. Stone 5 was "masked" with small volumes of fresh water to prevent absorption of run-down. Conservare HCT Finishing Rinse, one saturating application by low pressure spray, 12:00 noon to 12:02 PM. All stones (including 1 and 5) rinsed with fresh water, low pressure spray, 12:10 PM.

Application of treatment with low pressure spray



Condition of wall, 3 November 2001

23 June 2001

Inspection/Evaluation: Present: Irving Slavid and Jeremy Ashbee. Inspection of HCT-treated trial area was visually and by touch. Treated surfaces appeared to be significantly consolidated (vs. controls). No visual difference between treated and untreated areas could be discerned.

6 July 2001

Treatment application: Conservare OH (ethanol as solvent), 3 "cycles", carried out by Jo Thwaites.

3 November 2001

Inspection/Evaluation: Present: Irving Slavid and Norman Weiss; Jo Thwaites; Deborah Carthy (Carthy Conservation); Keith Garner and Robin Sanderson. Inspection was by touch and with 10X lens and 30 X field microscope. Consensus was that treated surfaces appeared to be significantly consolidated.

5.2b Mock-up wall, HM Tower of London

27 April 2001

A mock-up wall (incorporating Reigate stone from an interior wall of the New Armouries), constructed several weeks earlier, was inspected by the team assembled to do the HCT treatment on the Bell Tower. It displayed variable and excessive moisture. There was agreement that the mock-up wall be allowed to dry more fully before carrying out treatments.

Mock-up wall, HM Tower of London (cont.)

Description: Eight Reigate stones (designated 1 through 8) were incorporated in the rubble stone wall (Kentish rag, with brick back-up). Prior to installation, each stone was cut vertically. The halves (designated a and b) were installed with a semi-rigid membrane in between, to permit application of different treatments to portions a and b of the same stone. Construction of the wall is described in a drawing ("Research Panel 2") prepared by HRP. Average surface dimensions of the Reigate stones are given therein as 6 by 23 cm. Stones 1a, 2a, 3a and 4a to serve as (untreated) controls.

Mock-up wall, HM Tower of London (cont.)

3 November 2001

Mock-up wall is in the south moat, facing south. HCT and OH treatment was carried out by: Irving Slavid and Norman Weiss. Also present as observers were: Jo Thwaites; Deborah Carthy; and Keith Garner and Robin Sanderson.

Treatment application: Conservare HCT Hydroxylating Conversion Treatment, 11:37 AM to 1:21 PM. 14°C, 66% RH. Three saturating applications done by brush, with at least 30 minutes drying time in between. Application was to the b (right-hand) portion of all Reigate stones. Conservare HCT Finishing Rinse, one saturating application by brush, 2:28 to 2:30 PM. Conservare OH (ethanol as solvent), 11:42 AM to 2:36 PM; 2 and 2/3 “cycles” (i.e., eight saturating applications) by brush, to stones 5a, 6a, 7a and 8a. (As designed, this field test was to include OH treatment of stones 5b, 6b, 7b and 8b by HRP; that work was never accomplished.)

After HCT treatment (b of all stones), 3 November 2001, still wet

After OH treatment (5a-8a), 3 November 2001, still wet

5.3 Westminster Abbey

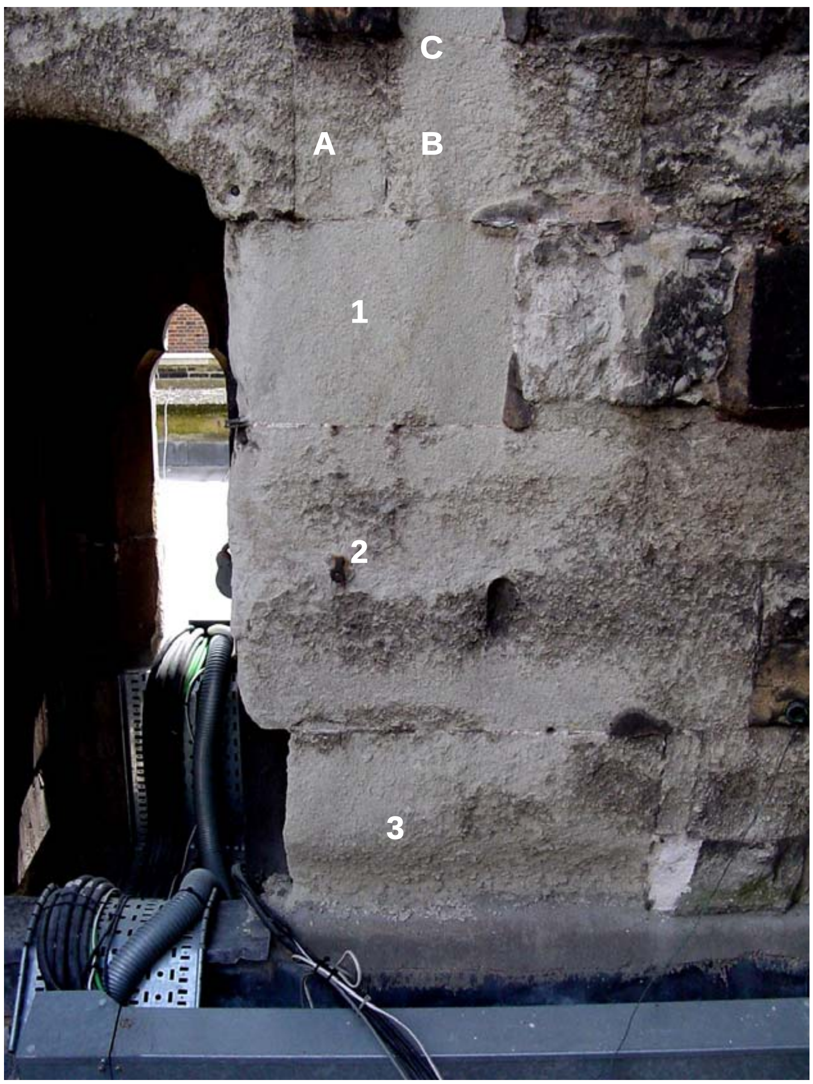
27 April 2001

Trial area was at the west end of the wall above the south Cloister roof, on the north face of the wall. This area has been protected from rain with corrugated plastic sheet since December 2000. Reigate stones designated for treatment are to the right of the door opening. HCT treatment was carried out by: Vanessa Simeoni (Westminster Abbey); Irving Slavid and Norman Weiss; and David Boyer. Also present as observers were: Richard Roberts, Jo Thwaites, Sarah Ferraby (Historic Royal Palaces) and Jeremy Ashbee; Robin Sanderson and Keith Garner; and Jagoba Mariscal.

Covered trial area at upper right

Westminster Abbey (cont.)

Description/Condition: Three contiguous Reigate stones (designated 1, 2 and 3), constituting the right-hand edge of the doorway. Smaller Reigate stone blocks immediately above (designated A, B and C) to serve as controls. Approx. depth of stones 13 cm. Color: all are pale grey-green, with some brown discoloration thought to be iron oxide. Ancillary wall materials: Caen stone, flints, brick. Previous conservation: there is no clear physical evidence or documentary record of prior chemical treatments**.** The three stones differ in condition. Stone 1 (top) is uniform in appearance, with a friable surface. Stone 2 (middle) also has a similar surface, with a thin weathering crust over approx. 40% of its area. Stone 3 (bottom) has the crust over approx. 25% of the surface and is somewhat concave, possibly due to backsplash from the surface of the roof immediately below.



Detail of trial area
A, B, & C are controls; 1, 2, 3 are to be treated

Westminster Abbey (cont.)

Surface preparation/Ultrasonic testing: Loose grains and other debris were removed from surface of the test stones and adjacent control stones with a soft, long-fibered dusting brush. The area of stone 1 was visually "divided" into four quadrants. Ultrasonic transducers placed in the center of each quadrant were used to determine pre-treatment pulse velocity in the two lateral and two diagonal directions. (These data are shown in the entry for 1 November 2001, below.)

Treatment application: Conservare HCT Hydroxylating Conversion Treatment, 2:40 to 5:00 PM. 12°C, 44% RH; clear day, becoming overcast. Three saturating applications done by low pressure spray, with approx. 30 minutes drying time between applications. Total volume used approx. 2.5 liters

Application of HCT

Treated stones (wet)

30 April 2001
Treatment application: Conservare HCT Finishing Rinse, one saturating application, carried out by Vanessa Simeoni.

4 July 2001
Treatment application: Conservare OH (ethanol as solvent), 3 "cycles", carried out by Vanessa Simeoni. Total volume used approx. 7.5 liters.

Westminster Abbey (cont.)

1 November 2001

<u>Inspection/Evaluation</u>: Present: Irving Slavid and Norman Weiss; Vanessa Simeoni. Inspection of treated area was by touch, with 10x lens and 30x field microscope, and with ultrasound. Considerable improvement in surface soundness (vs. control stones) was readily apparent. No visual difference (color, gloss) could be discerned as a result of treatment.

Summary of ultrasonic testing, stone 1: a-b , c-d transducer distances are 230 mm; a-d, b-c distances are 270 mm. Apparatus is portable PUNDIT.

Pulse velocity (km/sec), before treatment			1 November data
a-b	.91		1.40
a-d	.90		1.33
c-d	.89		1.16
b-c	.95		1.23
mean =	**.91**	**mean =**	**1.28**

Treated Reigate stone (1) shows a mean velocity increase of 40%.

5.4a Tennis Court Lane, Hampton Court Palace

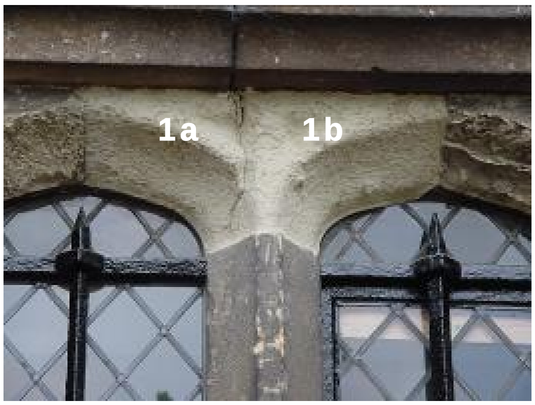
1 May 2001

Trial area was a window bay at the east end of Tennis Court Lane, at the ground floor level. Designated stones are about 2.5 m above grade. HCT treatment was carried out by: Irving Slavid and Norman Weiss, and David Boyer. Also present as observers were: Richard Roberts; Keith Garner and Robin Sanderson; and Brian Klelund (Tensid UK).

Description/Condition: Two Reigate stones, individual elements of two arched window heads. Stones designated 1b and 2a to be treated with Conservare HCT; adjacent stones 1a and 2b to serve as controls. HCT-treated stones to be treated subsequently with Conservare OH. Color: all are pale grey-green. Ancillary wall materials: Bath stone, brick. Previous conservation: considerable stone replacement. No documentary record of prior chemical treatments, but the initial difficulty in treating No. 1b may be indirect evidence of some residue. The two stones are in generally similar condition. Both exhibit considerable erosion; surfaces are friable and "sugary", with some curled crusts.

Tennis Court Lane, Hampton Court Palace (cont.)

Surface preparation: Stones 1a and 1b were "rationalized" with a soft, long-fibered dusting brush. 2a and 2b were prepared with the brush and a small plastic spatula.



Treatment application: Conservare HCT Hydroxylating Conversion Treatment, 11:10 AM to 1:15 PM. 14°C, 85% RH; overcast with showers. (Although applications were conducted during periods of rain, the trial area did not become heavily wetted.) Three saturating applications done by low pressure spray, with approximately 1 hour drying time between applications. (First application to stone 1b was absorbed slowly; the second was considerably better.) Conservare HCT Finishing Rinse, one saturating application by low pressure spray, 2:50 to 2:55 PM. All stones (including vertical elements below) rinsed with fresh water, low pressure spray, 3:00 PM.

22 June 2001

Treatment application: Conservare OH (ethanol as solvent), apparently 1 and 1/3 "cycles" (4 applications), carried out by Nimbus Conservation, and observed by Richard Roberts. Application was by brush. Volumes recorded by Roberts totaled approx. 0.4 liters for the two stones, almost certainly an insufficient quantity of consolidant. There is no Nimbus report.

2 November 2001

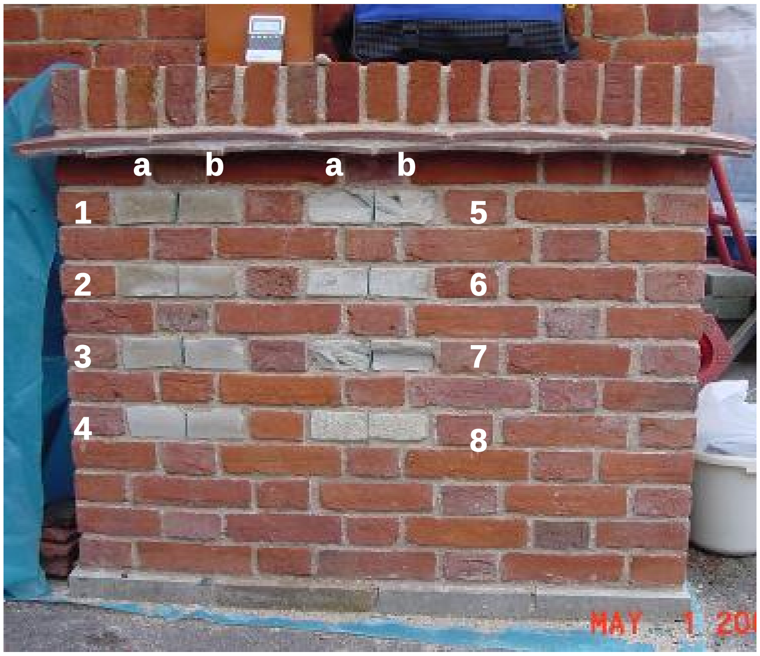
Inspection/Evaluation: Present: Irving Slavid and Norman Weiss; Richard Roberts: Keith Garner and Robin Sanderson. Inspection was by touch and with 10X lens and 30X field microscope. Consensus was that treated surfaces appeared to be significantly consolidated.

5.4b Mock-up wall, Hampton Court Palace

1 May 2001

Mock-up wall is in a service area to the north of the Pass Office, facing south. HCT treatment was carried out by: Irving Slavid and Norman Weiss, and David Boyer. Also present as observers were: Richard Roberts; Keith Garner and Robin Sanderson; and Brian Klelund (Tensid UK).

Description: Eight Reigate stones were incorporated in a brick wall. The stones were from Chipstead Church (1, 3, 5 and 7), Gatton Church (2 and 6), and Royal Earlswood Hospital (4 and 8). Prior to installation, each stone was cut vertically. The halves (designated a and b) were installed with a semi-rigid membrane in between, to permit application of different treatment to portions and a and b of the same stone. Construction of the wall is described in a drawing ("Research Panel 1") prepared by HRP. Average surface dimensions of the brick-shaped Reigate stones are given therein as 6 by 23 cm.



Treatment application: Conservare HCT Hydroxylating Conversion Treatment, 11AM to 1:05 PM. 14^{o}C, 85% RH; overcast with showers. Three saturating applications done by brush, with approximately 1 hour drying time in between. Application was only to the b (right-hand) portion of 5 though 8. Conservare HCT Finishing Rinse, one saturating application by brush, 3:00 PM, followed by a rinse with fresh water. (The wall was only recently constructed, and seven of the eight other individual pieces of Reigate stone were visibly damp, as seen in the photo above.) Future HCT treatment of the corresponding right-hand halves (b) of 1 through 4, and OH treatment of 5 through 8 (a and b) would then leave stones 1a, 2a, 3a and 4a as (untreated) controls.

Mock-up wall, Hampton Court Palace (cont.)

22 June 2001

Treatment application: Conservare OH (ethanol as solvent), 2 applications by brush, 11:00 AM to "mid pm", carried out by Richard Roberts. Mistakenly done to 1 through 4 (a and b), utilizing a total of 0.03 liters (30 ml).

2 November 2001

Treatment application: Conservare HCT Hydroxylating Conversion Treatment, 11:15 AM to 1:07 PM. Three saturating applications done by brush, with at least 30 minutes drying time between. Application was to stones 1b, 2b, 3b and 4b. (Absorption was satisfactory, despite the 22 June treatment.) Conservare HCT Finishing Rinse, one saturating application (to the same stones) by brush, 2:16-2:17 PM. Both sides (a and b) of 5 through 8 treated with Conservare OH (ethanol as solvent), 11:25 AM to 2:36 PM; three "cycles" (i.e., nine saturating applications) by brush.