EVALUATION OF CEMENTITIOUS INJECTION GROUTS FOR THE STABILIZATION OF

HOLLY TOWER SUPPORT ROCK, HOVENWEEP NATIONAL MONUMENT

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Table of Contents

| Chapter 1 | Introduction 1 |
|-----------|---|
| 1.1 | Background Summary 1 |
| 1.2 | Archaeological / Historical Context |
| 1.3 | Geological Context |
| 1.3.1 | Regional Geology |
| 1.4 | Climate |
| Chapter 2 | Site Conditions10 |
| 2.1 | Keeley Canyon |
| 2.2 | Condition of the Support Rock11 |
| Chapter 3 | Petrographic Analysis of the Support Rock15 |
| 3.1 | Sampling and Methodology15 |
| 3.2 | Petrographic Results15 |
| 3.3 | Conclusions / Discussion |
| 3.4 | Petrographic Description of Fabric Group17 |
| Chapter 4 | Literature Review |
| 4.1 | The Use of Mortar and Injection Grouts for Conservation |
| 4.2 | The Stabilization of Rock —Hewn Heritage Sites23 |
| 4.2.1 | Living Stone23 |
| 4.2.2 | Rock-Hewn Sites – Case Studies24 |
| 4.3 | The Shift toward Compatible Conservation Materials27 |

| 4.4 | The Use of Injection Grouts in a Structural Capacity | 28 |
|-----------|--|----|
| Chapter 5 | Injection Grouting | 30 |
| 5.1 | Grout Properties | 30 |
| 5.2 | Grout Components | 31 |
| 5.2.2 | 1 Binders | 31 |
| 5.2.2 | 2 Fillers – Inert and Reactive | 32 |
| 5.2.3 | 3 Additives | 33 |
| 5.2.4 | 4 Water Content | 33 |
| 5.3 | Grouting Techniques | 34 |
| 5.4 | Jahn M40 Crack Injection Grout | 34 |
| Chapter 6 | Summary of Previous Stabilization and Recent Research | 36 |
| 6.1 | Previous Stabilization Campaigns for Holly Tower | 36 |
| 6.2 | Recent Conservation Research | 36 |
| 6.2.7 | 1 Condition Survey | 36 |
| 6.2.2 | 2 Laboratory Testing of Stone Consolidant and Anti-Swelling Agent | 37 |
| 6.2.3 | 3 Preliminary On-Site Testing of Stone Consolidant and Anti-Swelling Agent | 38 |
| Chapter 7 | Methodology | 42 |
| 7.1 | Optimal Properties | 42 |
| 7.2 | Testing Matrix | 44 |
| 7.3 | Analysis of Jahn M40 Grout | 45 |
| 7.3.7 | 1 Gravimetric Analysis | 45 |
| 7.3.2 | 2 Particle Size Distribution | 49 |

| 7.3.3 | Results | 50 |
|------------|--|----|
| Chapter 8 | Stone Sample Preparation | 53 |
| 8.1 | Sample Collection | 53 |
| 8.2 | Sample Cutting, Labeling and Documentation | 53 |
| 8.3 | Sample Treatment | 54 |
| 8.3.1 | Pre-treatment with Funcosil Antihygro | 54 |
| 8.3.2 | Insertion of Gauge Studs | 58 |
| 8.3.3 | Treatment with Remmers KSE 300 E | 61 |
| Chapter 9 | Mold Preparation | 64 |
| 9.1 | Grout Mold Preparation | 64 |
| 9.1.1 | Splitting Tensile Strength Molds | 64 |
| 9.1.2 | Thermal Expansion / Drying Shrinkage / Water Immersion Molds | 65 |
| 9.2 | Stone / Grout Composite Mold Preparation | 66 |
| 9.2.1 | Shear Bond Strength and Frost Resistance Molds | 66 |
| Chapter 10 | Chamber Preparation | 69 |
| Chapter 11 | Grout Samples | 71 |
| 11.1 | Preliminary Preparations | 71 |
| 11.2 | Mixing | 72 |
| 11.3 | Pouring | 73 |
| 11.4 | Methodology for Numbering the Samples | 74 |
| Chapter 12 | Tests for Working Properties | 75 |
| 12.1 | Expansion and Bleeding | 75 |

| 12.1.1 | Purpose | 75 |
|--|--|----|
| 12.1.2 | Adaptations | 75 |
| 12.1.3 | Equipment and Materials | 76 |
| 12.1.4 | Preparation | 76 |
| 12.1.5 | Procedure | 76 |
| 12.2 F | low | 78 |
| 12.2.1 | Purpose | 78 |
| 12.2.2 | Adaptations | 78 |
| 12.2.3 | Equipment and Materials | 78 |
| 12.2.4 | Preparation | 79 |
| 12.2.5 | Procedure | 79 |
| Chapter 13 | Tests for Curing and Setting Properties | 81 |
| 13.1 V | isual Shrinkage | 81 |
| 13.1.1 | Purpose | 81 |
| 13.1.2 | Equipment and Materials | 81 |
| | | |
| 13.1.3 | Preparation | 82 |
| 13.1.3 13.1.4 | Preparation | |
| 13.1.3 13.1.4 13.2 D | Preparation Procedure rying shrinkage | |
| 13.1.3 13.1.4 13.2 D 13.2.1 | Preparation Procedure rying shrinkage Purpose | |
| 13.1.3 13.1.4 13.2 D 13.2.1 13.2.2 | Preparation Procedure rying shrinkage Purpose Adaptations | |
| 13.1.3 13.1.4 13.2 D 13.2.1 13.2.2 13.2.3 | Preparation Procedure rying shrinkage Purpose Adaptations Equipment and Materials | |

| 13.2 | 2.5 | Procedure | 84 |
|------------|-------|---|----|
| 13.3 | Time | e of Setting by Vicat Needle | 85 |
| 13.3 | 3.1 | Purpose | 85 |
| 13.3 | 3.2 | Adaptations | 85 |
| 13.3 | 3.3 | Equipment and Materials | 85 |
| 13.3 | 3.4 | Preparation | 86 |
| 13.3 | 3.5 | Procedure | 86 |
| Chapter 14 | 4 Te | ests for Hardened / Mechanical Properties | 88 |
| 14.1 | Shea | ır Bond Strength | 88 |
| 14.1 | 1.1 | Purpose | 88 |
| 14.1 | 1.2 | Adaptations | 88 |
| 14.1 | 1.3 | Equipment and Materials | 89 |
| 14.1 | 1.4 | Preparation | 89 |
| 14.1 | 1.5 | Procedure | 90 |
| 14.2 | Split | ting Tensile Strength | 91 |
| 14.2 | 2.1 | Purpose | 91 |
| 14.2 | 2.2 | Adaptations | 92 |
| 14.2 | 2.3 | Equipment and Materials | 92 |
| 14.2 | 2.4 | Preparation | 93 |
| 14.2 | 2.5 | Procedure | 94 |
| Chapter 15 | 5 Te | ests to Determine Stone/Grout Compatibility | 96 |
| 15.1 | Ther | mal Expansion | 96 |
| | | | |

| | 15.1 | .1 | Purpose | 96 |
|----|--|--|---|---|
| | 15.1 | .2 | Adaptations | 96 |
| | 15.1 | .3 | Equipment and Materials | 96 |
| | 15.1 | .4 | Preparation | 97 |
| | 15.1 | .5 | Procedure | 97 |
| Ch | apter 16 | Re | esults and Discussion | 99 |
| | 16.1 | Curin | g Conditions | 99 |
| | 16.2 | Q-tes | t | 99 |
| | 16.2 | .1 | Q-table | 100 |
| | 16.3 | Stan | lard Error of the Mean | 100 |
| | 16.4 | T-Tes | t | 101 |
| | 16.4 | .1 | Paired (dependent) T-Test: Type 1 Test in Excel | 101 |
| | 16.4 | .) | Unpaired (independent) T-Test: Type 3 Test in Excel | 102 |
| | | •2 | | |
| | 16.5 | F-Tes | t | 103 |
| | 16.5 16.6 | F-Tes Appe | .t | 103 103 |
| | 16.5 16.6 16.7 | F-Tes Appe Expa | rdices ndices nsion and Bleeding - ASTM C 940-10a | 103 103 104 |
| | 16.5 16.6 16.7 16.7 | F-Tes Appe Expa | ndices nsion and Bleeding - ASTM C 940-10a Observations and Results | 103 103 104 104 |
| | 16.5 16.6 16.7 16.7 16.7 | F-Tes Appe Expa 7.1 | t ndices nsion and Bleeding - ASTM C 940-10a Observations and Results Discussion | 103 103 104 104 105 |
| | 16.5 16.6 16.7 16.7 16.7 16.8 | F-Tes Appe Expa 7.1 7.2 Flow | rt ndices nsion and Bleeding - ASTM C 940-10a Observations and Results Discussion - ASTM C 939-10 | 103 103 104 104 105 105 |
| | 16.5 16.6 16.7 16.7 16.7 16.8 | F-Tes Appe Expa 7.1 7.2 Flow 8.1 | ndices nsion and Bleeding - ASTM C 940-10a Observations and Results Discussion - ASTM C 939-10 Observations and Results | 103 103 104 104 105 105 105 |
| | 16.5 16.6 16.7 16.7 16.8 16.8 16.8 | F-Tes Appe Expa 7.1 7.2 Flow 8.1 8.2 | tndices nsion and Bleeding - ASTM C 940-10a Observations and Results Discussion - ASTM C 939-10 Observations and Results Discussion | 103 103 104 104 105 105 105 106 |
| | 16.5 16.6 16.7 16.7 16.8 16.8 16.8 16.8 | F-Tes Appe Expa 7.1 7.2 Flow 8.1 8.2 Visua | tndices nsion and Bleeding - ASTM C 940-10a Observations and Results Discussion - ASTM C 939-10 Observations and Results Discussion I Shrinkage - ASTM C 1148-92a (2008) / ASTM C 474-13 | 103 103 104 104 105 105 105 106 108 |

| 16.9.1 | Observations and Results | 108 |
|--------------|--|-----|
| 16.9.2 | Discussion | 110 |
| 16.10 Dryi | ng Shrinkage - ASTM C 1148-92a | 111 |
| 16.10.1 | Observations and Results | 111 |
| 16.10.2 | Discussion | 114 |
| 16.10.3 | On Site Implications | 116 |
| 16.11 Time | e of Setting by Vicat Needle - ASTM C 953-10 / ASTM C 191-08 | 116 |
| 16.11.1 | Observations and Results | 116 |
| 16.11.2 | Discussion | 117 |
| 16.12 Shea | ar Bond Strength - ASTM D 905 / EN 196-1 | 118 |
| 16.12.1 | Test Modification | 118 |
| 16.12.2 | Observations and Results | 119 |
| 16.12.3 | Sample Inspection after Shear Bond Strength Test | 122 |
| 16.12.4 | Discussion | 125 |
| 16.12.5 | On-site Implications | 125 |
| 16.13 Split | tting Tensile Strength - ASTM C 496/496M-11 | 126 |
| 16.13.1 | Observations and Results | 126 |
| 16.13.2 | Sample Inspection after Splitting Tensile Strength Test | 129 |
| 16.13.3 | Discussion | 136 |
| 16.13.4 | On Site Implications | 137 |
| Thermal Expa | ansion - ASTM C 531-00 (2012) / ASTM D 4535-08 | 138 |
| 16.13.5 | Observations and Results | 138 |

| 16.1 | 3.6 | Discussion | 142 |
|------------|-------|--|--------------|
| 16.14 | Addi | itional Tests to be Completed | 143 |
| 16.1 | 4.1 | Hydric Expansion — RILEM 11.7 | 143 |
| 16.1 | 4.2 | Frost Resistance – RILEM V.3 | 144 |
| 16.1 | 4.3 | Capillary Absorption / Drying Index – NORMAL 11/85, 7/81and 29/88; ASTM C67-97 a | ind C948-94; |
| ARC | Laboi | ratory Handbook; ICCROM 1999 | 144 |
| 16.1 | 4.4 | Water Vapor Transmission – ASTM E96/E96M-12 and NORMAL 21/85 | 145 |
| 16.1 | 4.5 | Wet Density – ASTM C185-08 | 145 |
| Chapter 17 | C | onclusions | 146 |
| 17.1 | Gene | eral Summary | 146 |
| 17.2 | Reco | ommendations | 148 |
| Chapter 18 | R | ecommendations for Implementation | 150 |
| 18.1 | On-s | ite Preparations for Grouting | 150 |
| 18.1 | .1 | Construction of a Shading Device | 150 |
| 18.2 | Prep | arations of the Support Rock Prior to Grouting | 150 |
| 18.2 | .1 | Flushing of the Cavity | 150 |
| 18.2 | .2 | Temporary Facing at injection Holes and the Application of Clay Washes | 151 |
| 18.2 | .3 | Spacing and Configuration of Injection Holes | 152 |
| 18.2 | .4 | Temporary Stabilization of Slab | 154 |
| 18.3 | Addi | itional Measures to Maintain the Monolithic Integrity of the Support Rock | 156 |
| 18.3 | .1 | Sealing the Top of the Support Rock | 156 |
| 18.3 | .2 | Non-invasive Techniques for Void Detection | 156 |

| 18.3.3 Surficial and Sub-Surface Water Mitigation | 159 |
|--|-----|
| Chapter 19 Future Recommendations | 160 |
| 19.1 Cintec Harke Anchor System | 160 |
| 19.2 Grouting with Compacted Sand and Pre-stressed Bolts | 161 |
| Bibliography | 163 |
| Appendix A — Terminology | 169 |
| Appendix B- Testing Matrix | 173 |
| Appendix C- List of Samples | 174 |
| Appendix D — Gravimetric Analysis Data | 175 |
| Appendix E — Particle Size Distribution | 176 |
| Appendix F — Bleeding and Expansion | 177 |
| Appendix G – Flow | 178 |
| Appendix H – Drying Shrinkage | 179 |
| Appendix I — Time of Setting by Vicat Needle | 181 |
| Appendix J — Shear Bond Strength | 182 |
| Appendix K — Splitting Tensile Strength | 186 |
| Appendix L — Thermal Expansion | 190 |
| Appendix M — Product Information | 192 |
| Index | 205 |

List of Figures

| Figure 1. Context map for Hovenweep National Monument. Source: Hovenweep National Monument. General |
|--|
| Management Plan: Environmental Assessment. National Park Service, U.S. Department of the Interior, 2011 |
| Figure 2. View of Sleeping Ute Mountain from Hovenweep National Monument. Source: Department of Interpretive |
| Planning, Harpers Ferry Center and Hovenweep National Monument. Hovenweep National Monument: Long Range |
| Interpretive Plan. National Park Service, Department of the Interior, 2010. |
| Figure 3. Photo-geologic map of the Aneth-1 Quadrangle, San Juan County, Utah, Courtesy of the United States |
| Geological Survey, http://ngmdb.usgs.gov/Prodesc/proddesc_1409.htm |
| Figure 4. General context photographs of Keeley Canyon and Holly Tower. Source: Milot Berisha, 201311 |
| Figure 5. Vertical bedding orientation of the Holly Tower support rock. Source: Architectural Conservation Laboratory, |
| University of Pennsylvania, 201213 |
| Figure 6. Partially detached slab on the west face of the support rock. Source: overlay by author, photograph by the |
| Architectural Conservation Laboratory, University of Pennsylvania, 2012. |
| Figure 7. Views of partially detached slab. Source for images on left and top right: Lacombe Thesis, University of |
| Pennsylvania, 2013. Source for image on the bottom left: Architectural Conservation Laboratory, University of |
| Pennsylvania, 2012 |
| Figure 8. Comparison of porosity between sample HT.N1 (A) and sample HT.E1 (B) in PPL, magnification 50x; |
| migration of cement to surface of stone in sample HT.E1 as shown in PPL (C) and XPL (D), magnification 50x; images |
| by author20 |

| Figure 9. Alteration of grains in sample HT.N1 as shown in PPL (A), magnification 100x; overgrowth of quartz grains |
|---|
| and heterogeneous cement in sample HT.E1 as shown in XPL (B), magnification 100x; graphic intergrowth of quartz |
| and K-feldspar surrounded by hematite in sample HT.E1 as shown in PPL (C), magnification 200x; images by author. |
| |
| Figure 10. Demarcation of friable and non-friable areas and application of consolidant. Photographs by author, 2013. |
| |
| Figure 11. Preparations for RILEM water absorption test. Sources: Laura Lacombe and Milot Berisha, 201340 |
| Figure 12. Installation of stainless steel pins to monitor surface erosion. Sources: Laura Lacombe, Milot Berisha and |
| author, 201341 |
| Figure 13. Jahn M40 sand and microspheres clearly visible after water was added to the weighing boats46 |
| Figure 14. Sample preparation and levigation of microspheres and fines48 |
| Figure 15. Formation of white residue after pre-treatment with Funcosil Antihygro |
| Figure 16. Samples prepared for thermal expansion and water immersion tests prior to treatment and after applying |
| Funcosil Antihygro |
| Figure 17. Samples prepared for shear bonding strength test prior to treatment and after applying Funcosil Antihygro. |
| |
| Figure 18. Samples prepared for frost resistance test prior to treatment and after Funcosil Antihygro58 |
| Figure 19. Peck drilling the stone on the vertical mill |
| Figure 20. Drawing epoxy into a syringe60 |

| Figure 21. Aspirating holes in the stone; injecting epoxy into holes; inserting and rotating the gauge stud | 61 |
|---|-----|
| Figure 22. Applying Remmers KSE 300 E and blotting samples between cycles | 63 |
| Figure 23. Cutting PVC pipe for splitting tensile strength molds with chop saw | 65 |
| Figure 24. Preparation of molds for thermal expansion test | 66 |
| Figure 25. Construction of the shear bond strength molds | 67 |
| Figure 26. Fabrication of the aluminum plates for the shear bond strength test | 68 |
| Figure 27. Test chamber with potassium carbonate | 69 |
| Figure 28. Acrylic shelves for curing chambers | 70 |
| Figure 29. Wrapping and soaking stone prior to pouring | 72 |
| Figure 30. Pouring the grout | 74 |
| Figure 31. Expansion and bleeding test in progress | 77 |
| Figure 32. Leveling flow cone and performing flow test. | 80 |
| Figure 33. Aluminum plates used to distribute the load during shear bond strength test | 91 |
| Figure 34. Measuring the diameter of the grout cylinders with digital calipers. | 94 |
| Figure 35. Reference bar and grout sample inserted into length comparator. | 98 |
| Figure 36. Mold for thermal expansion and drying shrinkage tests. | 107 |
| Figure 37. Photograph of visual shrinkage test samples after 28 days of curing. | 109 |
| Figure 38. Detail photograph of cracking along the perimeter of the sample. | 110 |

| Figure 39. Plane of breakage in shear bond strength samples during curing process | 119 |
|---|-----|
| Figure 40. Sample A2.2 – Original grouted area: 2.850 sq. in. / Bond line area: 1.156 sq. in | 122 |
| Figure 41. Sample A3.1 – Original grouted area: 2.935 sq. in. / Bond line area: 1.022 sq. in | 123 |
| Figure 42. Sample B2.3 – Original grouted area: 3.039 sq. in. / Bond line area: 1.623 sq. in | 123 |
| Figure 43. Sample B3.1 – Original grouted area: 2.859 sq. in. / Bond line area: 1.534 sq. in | 124 |
| Figure 44. Sample B4.2 – Original grouted area: 3.021 sq. in. / Bond line area: 1.021 sq. in | 124 |
| Figure 45. Splitting tensile strength test in progress. | 129 |
| Figure 46. Splitting tensile strength procedure and fracture pattern for sample C2.2. | 131 |
| Figure 47. Splitting tensile strength procedure and fracture pattern for sample C2.3. | 132 |
| Figure 48. Splitting tensile strength procedure and fracture pattern for sample C2.4. | 133 |
| Figure 49. Splitting tensile strength procedure and fracture pattern for sample C2.5. | 134 |
| Figure 50. Splitting tensile strength procedure and fracture pattern for sample C2.6. | 135 |
| Figure 51. Grout and stone samples in desiccator. | 138 |
| Figure 52. Mural painting in cave 85 of the Mogao grottoes stabilized with temporary facing prior to grouting. | |
| Source: Stephen Rickerby et al. Los Angeles: The Getty Conservation Institute, 2010. | 152 |
| Figure 53. Custom press in cave 85 of the Mogao grottoes. Source: Stephen Rickerby et. Al. Los Angeles: The Get | ty |
| Conservation Institute, 2010. | 155 |

List of Tables

| Table 1. Abbreviated testing matrix; complete matrix is located in the appendix | 44 |
|---|-----|
| Table 2. Q-test for 95% confidence level | 100 |
| Table 3. Flow test results | 106 |
| Table 4. Calculations for drying shrinkage test. | 112 |
| Table 5. Results for paired t-test | 113 |
| Table 6. Shear bond strength test results | 121 |
| Table 7. Splitting tensile strength test results | 127 |
| Table 8. Comparison of splitting tensile strength values | 136 |
| Table 9. Test results for thermal expansion test | 140 |
| Table 10. Independent t-test for thermal expansion test results | 141 |
| Table 11. F-test results for thermal expansion test results. | 142 |

List of Graphs

| Graph 1. Weight percentage of the components of Jahn M40 | 51 |
|---|-----|
| Graph 2. Particle size distribution of microspheres in Jahn M40 | 52 |
| Graph 3. Particle size distribution of sand in Jahn M40 | 52 |
| Graph 4. Results for drying shrinkage test | 113 |
| Graph 5. Shrinkage curve for grout samples recorded over 28 days | 115 |
| Graph 6. Results of vicat test (initial set indicated with black dots) | 117 |
| Graph 7. Shear bond strength test results | 121 |
| Graph 8. Comparison of test results | 128 |
| Graph 9. Comparison of thermal expansion in the grout and stone samples | 141 |

Chapter 1 Introduction

1.1 Background Summary

This thesis addresses the testing and evaluation of the use of a cementitious injection grout for the reattachment of detached slabbing on the sandstone support rock of Holly Tower at Hovenweep National Monument. This research follows on a previous condition assessment and diagnosis of the deterioration of the sandstone support rock and the effects of consolidation on the rock as the first step in a multi-phase conservation program.¹

The case hardening and subsequent slabbing on the west side of the support rock has created a large inverted cavity behind a thick, brittle, partially detached slab. This slab has begun to behave independently of the larger rock mass, experiencing elongation and warping due to thermal deformation and creep.² While similar conditions exist on the east side of the rock as well, the testing described here has been designed for the inverted cavity, which is the area of greatest concern.

The extreme instability of the slabbing on the west side of the support rock requires remedial intervention that will retain and reattach the slab to the parent rock. Injection grouting was identified as a viable option for reattachment, requiring the development of an intensive testing regimen to determine the physical and mechanical properties of a commercial cementitious grout. Due to time constraints, hydraulic lime grouts were not included in this study, and only Jahn M40 crack injection grout was tested.

¹ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013).

² Ibid, 121-122.

From a cultural perspective, any treatment consideration must acknowledge the beliefs and values of the site's Native American tribal affiliates who have a primary role in the decision making process. In past and on-going projects in the region, specifically at Mesa Verde National Park to the east of Hovenweep National Monument, Native American tribes have expressed concerns about the use of synthetic materials on the ancestral sites they lay claim to. Many tribal communities believe that sites inhabited by their ancestors should be left undisturbed and reject the use of artificial or synthetic materials in the preservation process.

From a visual perspective, any proposed structural interventions should have as little impact as possible. Unlike masonry structures which have joints and an assembly of materials within which structural repairs can often be integrated, it is much more difficult to mask an intervention on a natural feature. The "untouched" appearance of the site which is highly valued by both the National Park Service and visitors should be maintained to the greatest extent possible.³

1.2 Archaeological / Historical Context

Located on the border of Colorado and Utah, Hovenweep National Monument was among the first group of archaeological sites to be protected under the Antiquities Act in 1923 by President Warren G. Harding. The Monument is comprised of the prehistoric remains of villages and their collective landscape built mainly during the Pueblo period (A.D. 750 to1300). Spread over 785 acres along the border of Colorado and Utah, it includes a range of structures built on both mesa tops and within the canyons.⁴ While no large settlements were present in the Hovenweep area during the Pueblo I period (A.D. 750-900), the population was thought to have greatly increased

 ³ Department of Interpretive Planning, Harpers Ferry Center and Hovenweep National Monument, "Hovenweep National Monument: Long Range Interpretive Plan," National Park Service, U.S. Department of the Interior (2010): 5-11.
 ⁴ Ibid, 5-11.

during the Pueblo II period (A.D. 900-1150) as expanding communities were forced to inhabit less desirable places with harsher climates and limited water access. During the Pueblo III period, many communities began to cluster, forming larger complexes which required increased agricultural production. By A.D. 1225, seven canyon head complexes became thriving communities which are now the most well-known archeological sites at Hovenweep. One of these complexes known as the Holly Unit was strategically located near the drainage from the surrounding mesa tops and a naturally occurring spring at the head of Keeley Canyon.⁵ The Hovenweep sites and many other villages in the Four Corners region were depopulated during the Pueblo IV period (after A.D. 1300).

Holly Tower, which was built with the local Dakota sandstone, was constructed between A.D. 1200 and 1300, and may have been used as a granary, a ceremonial structure, or a watchtower. ⁶ The tower was built on a detached sandstone "boulder", referred to the as the Holly Tower support rock, which is the focus of this report.⁷

⁵ Hovenweep National Monument, "General Management Plan: Environmental Assessment," National Park Service, U.S. Department of the Interior (2011): 92–99.

⁶ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013) 121-122.

⁷ The term boulder has been avoided in order to stress that the support rock is actually an adjacent canyon ledge rock that has detached, fallen and rotated 90° to its present position upon which the Ancestral Puebloans built their tower.



Figure 1. Context map for Hovenweep National Monument. Source: Hovenweep National Monument. General Management Plan: Environmental Assessment. National Park Service, U.S. Department of the Interior, 2011.



Figure 2. View of Sleeping Ute Mountain from Hovenweep National Monument. Source: Department of Interpretive Planning, Harpers Ferry Center and Hovenweep National Monument. Hovenweep National Monument: Long Range Interpretive Plan. National Park Service, Department of the Interior, 2010.

1.3 Geological Context

1.3.1 Regional Geology

Hovenweep National Monument is located in the Colorado Plateau Province, which is characterized by rounded

uplands separated by vast rangelands, and high plateaus. Beneath the rangelands lie large elliptical structural basins

with gently warped rock strata which display little deformation. Lateral monoclines, step-like folds which have formed the mesa tops and canyons, are another important geological feature on the Colorado Plateau. The mountain ranges surrounding the Hovenweep area to the north, south, and southeast are laccoliths. Laccoliths are formed by the sheet intrusion of magma between layers of sedimentary rock, which exerts upward pressure on the strata above and forces them upward. During the late Tertiary, these laccoliths were heavily eroded by wind, rain, and frost to form the mountain ranges which define this area.⁸

Based on the results of a geologic survey by the U.S. Department of the Interior, Keeley Canyon is composed of undifferentiated Burro Canyon Formation beneath Dakota sandstone, deposited during the Early and Late Cretaceous ages respectively. The Burro Canyon Formation chiefly consists of cross-bedded, stream-lain pebble conglomerate and coarse-grained conglomeratic sandstone. Shale and finer-grained sandstone layers are also present. The conglomerate pebbles mainly consist of light-gray to cream colored quartzite and light to dark grey chert. This formation is approximately 110 feet thick and was likely deposited across an alluvial plain by meandering streams which altered course over time.⁹

Dakota sandstone chiefly consists of cross-bedded sandstone in beds which range from several inches to several feet thick. Pebbly conglomerate, shale, mudstone and coal are also present in smaller quantities. The beds of Dakota sandstone are generally more uniform and thinner than the Burro Canyon Formation below. The fact that this formation was deposited in shallow water is evidenced by the presence of petrified wood, carbonized plant matter,

⁸ Hovenweep National Monument, "General Management Plan: Environmental Assessment," National Park Service, U.S. Department of the Interior (2011): 92-99.

⁹ United States Geological Survey, "Rock Formations: Their Attributes and Geologic Settings," Geological Survey Bulletin 1191, last modified March 28, 2006. http://www.cr.nps.gov/history/online_books/geology/publications/bul/1191/sec4e.htm.

impressions of twigs, worm burrows, and ripple marks.¹⁰ Dakota sandstone can therefore be characterized as fluvial sandstone.¹¹

¹⁰ United States Geological Survey, "Rock Formations: Their Attributes and Geologic Settings," Geological Survey Bulletin 1191, last modified March 28, 2006. http://www.cr.nps.gov/history/online_books/geology/publications/bul/1191/sec4e.htm.

¹¹ Mark A. Kirschbaum, "Geologic Assessment of Undiscovered Oil and Gas Resources of the Mancos/Mowry Total Petroleum System, Uinta-Piceance Province, Utah and Colorado, " in *Petroleum Systems and Geologic Assessment of Oil and Gas in the Uinta-Piceance Province, Utah and Colorado*, by USGS Uinta-Piceance Assessment Team, 1-33, Denver: U.S. Geological Survey, 2003.



Figure 3. Photo-geologic map of the Aneth-1 Quadrangle, San Juan County, Utah, Courtesy of the United States Geological Survey, http://ngmdb.usgs.gov/Prodesc/proddesc_1409.htm.

1.4 Climate

Hovenweep National Monument is located in a high desert region which is subject to extremes of temperature, both seasonally and sometimes daily. Temperature fluctuations exceeding 40 degrees have been recorded in a single day. In the summer, temperatures may exceed 100°F and late summer is referred to as the monsoon season, during which there are heavy downpours and electrical storms which may cause flash floods. In the winter, the high temperature ranges from 30°F to 50°F and the low temperature ranges from 0°F to 20°F. With the exception of the adjacent mountain ranges, there are rarely heavy snowfalls. During the spring and fall, April through May and mid-September through October respectively, the high temperature ranges from 60°F to 80°F and the low temperature ranges from 30°F to 50°F.¹²

¹² Hovenweep National Monument, "General Management Plan: Environmental Assessment," National Park Service, U.S. Department of the Interior (2011): 93.

Chapter 2 Site Conditions

2.1 Keeley Canyon

The effect of the hydrological processes at work in this landscape cannot be understated. While surface water has helped to shape the major geological features, groundwater is simultaneously eroding the mineralic cement which binds the sandstone, weakening and undercutting cliff faces which continue to collapse as the water freezes and expands. The permeable Dakota sandstone over the much less permeable shale component of the Burro Canyon Formation causes the horizontal flow of water. The downward infiltration of groundwater through the sandstone is blocked by the shale and is forced to find an outlet in the form of a seep or spring.¹³ The presence of a spring indicates that the ground water is very close to the surface. This is further supported by the concentration of trees in the canyon, as opposed to the more drought resistant vegetation which dominates the mesa top. The support rock is located in or near the path of the spring and the drainage from the mesa top, which suggests that the base of the rock is often saturated.

¹³ U.S. Department of the Interior, "Hovenweep National Monument: Geologic Resource Evaluation Report," Geologic Resources Division (2004): 3-41.



Figure 4. General context photographs of Keeley Canyon and Holly Tower. Source: Milot Berisha, 2013.

2.2 Condition of the Support Rock

Prior to the construction of Holly Tower, the support rock detached from the canyon rim and rotated roughly ninety degrees from its original orientation. As a result, the bedding planes are oriented vertically, allowing rain and snow to infiltrate between the layers of stone and accelerate deterioration. Petrographic analysis has revealed that the fine-grained heterogeneous cement binding the coarser mineral grains together is highly weathered and porous. As water leaches through the stone, the cement migrates to the surface and is re-deposited, resulting in a densified, less porous or "case-hardened" surface coupled with a mineral-depleted core beneath. Micrite, or microcrystalline calcite, accounts for a large percentage of the cement. Calcite exhibits anisotropic behavior when exposed to heat. The crystals do not expand equally in all directions when heated, causing dislocations along the interface between the crystals. This dislocation prevents the crystals from returning to their original positions when they cool, and the result

is an increase in volume and porosity. This increase in volume results in hysteresis, irreversible "growth" leading to deformation. This condition is particularly evident in thin slabs of stone, which are more susceptible to deformation from thermal stress.¹⁴

This combination of orientation and material composition has resulted in the formation of a large suspended slab on the west side of the support rock which is up to four inches thick, with an inverted cavity from depleted core loss behind it which is approximately two to three inches deep. Creep and thermal stress have further caused the slab to act independently of the larger rock mass, resulting in curvature away from the stone. ¹⁵ The surface of the slab is case-hardened as well and the cavity walls have become depleted of minerals. Continued water infiltration and leaching, as well as thermal fatigue, will eventually lead to complete detachment of the slab. The continued loss of material will in time will cause a continued wasting or reduction of the support rock's mass and eventually undermine the stability of the tower above. The tower's corners were built to the edge of the support rock leaving no tolerance for any loss of rock.

¹⁴ Sandy L. Cross, "Behind the Curtain: Mechanical Treatments for Bowed Marble Panels," (M.S. Thesis, University of Pennsylvania, 2005) 12-13.

¹⁵ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013) 39.



Figure 5. Vertical bedding orientation of the Holly Tower support rock. Source: Architectural Conservation Laboratory, University of Pennsylvania, 2012.



Figure 6. Partially detached slab on the west face of the support rock. Source: overlay by author, photograph by the Architectural Conservation Laboratory, University of Pennsylvania, 2012.



Figure 7. Views of partially detached slab. Source for images on left and top right: Lacombe Thesis, University of Pennsylvania, 2013. Source for image on the bottom left: Architectural Conservation Laboratory, University of Pennsylvania, 2012.

Chapter 3 Petrographic Analysis of the Support Rock

3.1 Sampling and Methodology

Two thin sections embedded in blue epoxy were used for this report. HOVE.HT.N1 was prepared from a rock sample collected from rock fall on the north side of the support rock, and HOVE.HT.E1 was prepared from a rock sample collected from the east face of the support rock. Both thin sections were prepared for previous research in 2013 and were again analyzed with a Zeiss Axioscope A.1 transmitted light microscope in the Ceramics Laboratory at the University of Pennsylvania Museum of Archaeology and Anthropology with assistance from Dr. Marie-Claude Boileau.

3.2 Petrographic Results

Samples HOVE.HT.E1 and HOVE.HT.N1 have been identified as a coarse-grained quartz arenite sandstone. The coarse quartz grains are well sorted, and while some of the grain boundaries are straight, many of them may be characterized as metamorphosed, which may indicate pressure dissolution during burial. Most grains exhibit overgrowth, which indicates that the stone is in the early stages of metamorphism. Undulose extinction is common and many of the grains appear stressed or fractured. This may be attributed to the preparation of the thin sections. Orthoclase is present in much smaller quantities, often displaying incipient alteration along cleavage planes. Some of the feldspar grains are pseudomorphs, which have been completely replaced by micrite and sericite. Microcline, chert, and calcium carbonate were the next most abundant, all displaying alteration. Plagioclase, polycrystalline quartz, muscovite laths and zircon, along with several unknown felsic and mafic minerals, were the rarest components of the coarse fraction. The cement is patchy and heterogeneous. Drusy calcite mosaic (micrite) is a major component (Figure 9, image B), often precipitating in fractures and cleavages in the grains and causing them to split. Due to the high porosity of the cement, it may be assumed that the calcite precipitated into the stone after the quartz overgrowths formed.¹⁶ While present in both samples, sericite is more abundant and the crystals were larger in sample E1, which was collected from the east face of the support rock. In addition to being a major component of the cement, sericite is also present as inclusions in many of the coarse grains. Micron-size hematite was observed as both thin rims around many of the coarse grains, and as dark concentrated clusters in the cement. Altered chert was also very abundant in the cement. Viewed as a whole, the cement reflects a series of dynamic processes which have occurred over the life of the stone. Flow patterns were visible in many parts of the coment, which is consistent with the migration of the cement to the surface of stone and the mineral depletion of the core (Figure 8, images C and D).

3.3 Conclusions / Discussion

The complexity of the sandstone's cement as a result of both precipitated and altered minerals and the abundance of grains currently displaying alteration further explain the friable nature of the stone. Continued inspection and the application of consolidants will be critical to the preservation of the Holly Tower support rock. Injection grouting as a technique for restoring the monolithic quality of the stone and protecting the exposed cavity is also necessary. It is a more viable option than pinning alone on a large scale, which will most likely cause wide scale fracturing and loss of the slab, exposure of the cavity and further disaggregation of the rock surfaces.

¹⁶ Maurice E. Tucker, *Sedimentary Petrography, 3rd ed.* (Malden: Blackwell Publishing Company, 2001).

The discovery of the presence of chert in the form of mineral grains and cement indicates that the support rock, and cliff face from which it detached, consists of stone which is at the interface between the Dakota sandstone and the Burro Canyon Formation layers. Several attempts to cut the stone revealed that the hardness varied from very soft (samples could be crushed by hand) to very hard (difficult to cut with the stone saw). Dark grey concentrations visible in the harder stone may in fact be chert concentrations.

Another major issue is the presence of swelling clays in the stone, which may be accelerating the rate of deterioration. Previous analysis of the stone with XRD only detected the presence of kaolinite, which was later confirmed with SEM. However, a methylene blue absorption test conducted in 2013 detected the presence of swelling clays such as smectite, so it is possible that the percentage of swelling clay in the stone is below the detection level of XRD.¹⁷ The use of Fourier Transform Infrared Spectroscopy (FTIR), which is capable of identifying and quantifying all of the molecular compounds, is required to make a final determination.

3.4 Petrographic Description of Fabric Group

Quartz arenite sandstone; very-fine grained buff to dark brown cement with quartz / feldspar / chert inclusions

2 samples: HOVE.HT.N1 / HOVE.HT.E1

Microstructure

Highly porous cement; distribution of inclusions range from close-spaced to single-spaced.

¹⁷ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013) 98-101.

Cement

Cement can be characterized as very fine and heterogeneous; buff to dark brown in PPL; mottled grey, deep brown, and black in XPL(100x); cement is primarily composed of kaolinite, micrite, sericite, hematite, and chert.

Inclusions

Inclusions are well sorted, with a unimodal grain size distribution, <0.73mm, r-a, mainly sr-r.

| Predominant | Monocrystalline quartz : sa-sr, equant to elongated, <0.42mm, mode 0.29mm. Most |
|-------------|---|
| | grains display overgrowth, undulose extinction common. Many of the grains appear |
| | stressed and/or fractured. |
| Few | Orthoclase: sr, equant, <0.18mm, mode 0.16mm. The boundaries of many grains |
| | have altered; a few grains display incipient alteration along cleavage planes; clay-sized |
| | mineral and sericite inclusions common. |
| Rare | Chert: a-r, equant to elongated, <0.48mm, mode 0.26mm. Many grains display |
| | growth (precipitation) between grains which forms part of the cement. |
| | Calcium carbonate: sr, equant to elongated, <0.34mm, mode 0.27mm. The |
| | boundaries of several grains display alteration. |
| | Microcline: sr-sa, equant to elongated, <0.52mm, mode 0.27mm. Mineral inclusions |
| | common; the boundaries of many grains have altered; many grains display incipient |
| | alteration along cleavage planes. |

Very Rare **Plagioclase:** sr-sa, equant to elongated, <0.73mm, mode 0.28. Some grains have partially altered.

Polycrystalline quartz: sr, equant, <0.40mm, mode 0.30mm.

Muscovite laths: elongated, <0.36mm.

Zircon: r, equant, <0.15mm.

<0.33mm. HOVE.HT.E1. Colorless foreground in relief in the shape of triangles (quartz) with a mottled pale red background (K-feldspar) in PPL; colorless foreground in relief in the shape of triangles with dark grey background in XPL; no pleochroism; high birefringence; 1st order interference colors; medium relief along grain boundary; hematite has replaced some of the grain boundaries.

Graphic (granophyric) intergrowth of guartz and K-feldspar: sr, elongated,

Glauconite?: r, elongated, <0.25mm. Weak pleochroism; pale olive green to slightly darker green in PPL; yellow-green with mottled, granular texture in XPL; low birefringence; high relief.

Felsic mineral: sa, elongated, <0.47mm, mode 0.41mm.

Colorless in PPL; light to dark grey in XPL; fibrous texture; 1 cleavage plane; 1st order interference colors; no pleochroism; high birefringence; the boundaries of several grains have altered.
Mafic mineral: r; strong pleochroism; pale green to olive green in PPL; 2nd and 3rd order interference colors in XPL; high birefringence; grain displays overgrowth, cleavage plane could not be detected.



Figure 8. Comparison of porosity between sample HT.N1 (A) and sample HT.E1 (B) in PPL, magnification 50x; migration of cement to surface of stone in sample HT.E1 as shown in PPL (C) and XPL (D), magnification 50x; images by author.



Figure 9. Alteration of grains in sample HT.N1 as shown in PPL (A), magnification 100x; overgrowth of quartz grains and heterogeneous cement in sample HT.E1 as shown in XPL (B), magnification 100x; graphic intergrowth of quartz and K-feldspar surrounded by hematite in sample HT.E1 as shown in PPL (C), magnification 200x; images by author.

Chapter 4 Literature Review

4.1 The Use of Mortar and Injection Grouts for Conservation

The widespread use of cement grout and mortar for historic and ancient structures in the 20th century has led to pervasive problems with compatibility, namely excessive strength, reduced porosity, and the formation of by-products which exacerbate and introduce new deterioration mechanisms such as soluble salts. ¹⁸ This trend also greatly impacted the conservation of ruins in the early 20th century. Portland cement mortar was used to cap walls to prevent further damage from water infiltration. It was also used as the binder for the grouts used to stabilize masonry walls by filling interior cavities. Lime-based products, though more compatible, required a longer curing time and increased construction costs, especially in wet, humid climates. The other key issue was the fact that lime-based mortars and grouts will not develop full strength and withstand weathering if the carbonation process is incomplete. Un-carbonated lime is water soluble, and therefore much more vulnerable to deterioration.¹⁹

An examination of past repairs at Holly Tower revealed that in 1948, cement was used to permanently stabilize the tower, while pigmented cement mortar was applied to areas with extensive mortar loss. Again in 1963, "brush-type" plastic cement was applied to the top of the tower to prevent further separation of the inner and outer courses due to moisture infiltration. It was not until 1986, when the lower courses were repointed with a stabilized earthen mortar that noticeable shift occurred as more compatible mortars were used. Following this, in 1998, twenty five percent of

 ¹⁸ Proceedings of the ICCROM Symposium Mortars, Cements and Grouts used in the Conservation of Historic Buildings, (Rome: ICCROM, 1982).
¹⁹ John Ashurst and Colin Burns, "Appendix 1: Materials and Techniques," in *The Conservation of Ruins*, ed. John Ashurst, (London; Burlington: Butterworth-Heinemann, 2007) 312.

the wall joints were repointed and chinking stones were inserted. In the same year, 95 percent of the areas repointed during earlier stabilization campaigns were replaced with soil mortar.²⁰

4.2 The Stabilization of Rock – Hewn Heritage Sites

4.2.1 Living Stone

Living stone differs from quarried stone in that it contains quarry sap (groundwater) and is prone to natural geomorphological processes. The quarry sap quickly evaporates after the stone is quarried and the surface of the stone becomes case hardened.²¹ The Holly Tower support rock, though detached from the canyon rim, has settled near the mouth of a spring at head of Keeley Canyon. While the stone no longer contains quarry sap, moisture is retained in the wind-blown soil and stone debris at the base of the tower. The lower portion of the support rock is saturated with ground water. The result is a rock monolith which is characterized by both rising and falling damp.

The Holly Tower support rock may be loosely classified along with other rock-hewn heritage sites, which are divided by typology. The first and most basic type includes caves, rocks and crevices which have been unaltered. Monolithic rock formations within this category include freestanding anthropomorphic features in the landscape which are often valued for ceremonial purposes.²² The relationship of the rock formation to the parent rock greatly affects structural stability and determines many of the prevalent deterioration mechanisms. Rock formations and structures which remain connected to the parent rock are much more susceptible to frequent dampness and are vulnerable to damage

²⁰ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (Master's thesis, University of Pennsylvania, 2013).

 ²¹ Christopher Gorse et al., *A Dictionary of Construction, Surveying and Civil Engineering*, (Oxford, Oxford University Press, 2013), accessed January 7, 2014, http://www.oxfordreference.com/view/10.1093/acref/9780199534463.001.0001/acref-9780199534463-e-5501.
²² Pietro Laureano, "Conservation Action Plan for the Rock Hewn Churches in Lalibela," World Monuments Fund, accessed January 9, 2014, http://issuu.com/zeb89/docs/wmf_final__report_eng_ott08.

due to the subsidence of the larger rock mass to which it remains connected. However, such formations are better able to withstand seismic shock and distribute loads.²³ Free-standing monolithic rock formations or structures in contrast which do not have the support of the parent rock as a buttressing mechanism are more vulnerable to seismic shock.²⁴ The Holly Tower support rock which has detached from the parent rock and settled on the canyon floor may continue to settle and shift over time until it reaches equilibrium in the environment.

4.2.2 Rock-Hewn Sites – Case Studies

Several international projects which involved the stabilization of rock-hewn structures were reviewed to assess conservation trends for this specialized typology within historic structures. The rock-hewn churches in Lalibela, Ethiopia were carved from a layer of tufa which rests on a layer of basalt. Cracking and interruptions in the geological strata were common and seismic activity presented a constant threat to the stability of the structures. Rainwater and groundwater were dissolving and weakening the stone, often resulting in deep cavities referred to as taffoni. Freestanding monoliths and walls were characterized with basal erosion due to saturation from groundwater.

Multiple restoration campaigns between the 1920's and the 1970's involved shifts between the use of compatible lime based mortars and grouts, and less compatible cement based products. Both natural and synthetic conservation materials were used to address water infiltration issues. Beginning in the 1990's, protective shelters were built over several structures to mitigate water damage. However, improper drainage resulted in further damage and created a sheltered environment for birds, which introduced bird guano as another mechanism of deterioration.

 ²³ Pietro Laureano, "Conservation Action Plan for the Rock Hewn Churches in Lalibela," World Monuments Fund, accessed January 9, 2014, http://issuu.com/zeb89/docs/wmf_final__report_eng_ott08.
²⁴ Ibid.

The rock hewn-churches at the Göreme circus in Turkey were carved out of andesitic tuff as early as 200 A.D. The highly porous stone has low compressive strength and the softer core is often topped with harder caps, which leads to differential erosion and collapse. While the vertical surfaces of the stone were often protected by moss and lichen, the horizontal surfaces were rapidly eroding. The migration of moisture through fractures in the rock and daily and seasonal variations in temperature resulted in a cycle of ice-jacking and the formation of hydraulic wedges which were formed as water filled the fractures. These processes were undermining the structural stability of the churches and jeopardizing the integrity of the murals on the interior.²⁵

To stabilize the surface, resins were tested, but the lack of adhesion and the nonporous quality of the resin led to the detachment of the consolidated surface from the weaker core material. Following this, mortar with a mixture of cement and lime was recommended with tuff as an aggregate.²⁶ To structurally stabilize the monolithic rock, it was suggested that cement grout be injected into cracks and voids with special attention to the amount of pressure used so as to prevent hydraulic jacking. In areas with overhangs or other configurations which required buttressing to avoid collapsing, the injection of cement or resin based grouts under pressure was recommended, as well as the insertion of stainless steel bars which would be pressure grouted as well.²⁷ Published in the 1990's this study clearly favored cement and hydraulic lime based grouts were not included for testing. Lime was viewed as a material which would not withstand erosion, but it was incorporated into the mortar formulation due to its lower cost and aesthetic appearance.

²⁵ Robert Bowen, "The Future of the Past at Göreme in Turkey," *Environmental Geology and Water Sciences*, Vol. 16, Issue 1 (July/August 1990):35-41.

²⁶ Ibid, 35-41.

²⁷ Ibid, 35-41.

In China, the conservation of the Mogao grottoes in the Dunhuang Province involved the use of many innovative techniques. The process of rock bolting, which usually involves drilling holes inclined upward, often presents problems in terms of completely filling the holes with grout. To address this problem, a technique referred to as cartridge-type grouting was developed. Highly absorbent paper cartridges, appropriately sized to fill the grout holes, were filled with a dry mix of fast-curing, non-corrosive thioaluminate cement and soaked in water immediately before insertion into the drilled holes. As the water mixes with the cement, the resulting grout became rigid within the cartridge within 1-3 minutes. The cartridges were then inserted into the holes, followed by the steel anchors, which were rotated into the holes to burst the cartridges and evenly distribute the grout within the hole.²⁸

At the Dafosi (Great Buddha Temple) Grotto in Xian, the capital of the Shaanxi Province, the temple and statues were carved from a sandstone cliff. Fractures and intrinsic weaknesses in the stone, exacerbated by carving the grotto into the cliff face, have resulted in stress concentrations and dynamic cracks which extended into the head of the eastern bodhisattva, threatening complete detachment. A rock anchor system developed by the Institute of Mining of the Russian Academy of Science in Novosibirsk was proposed to stabilize the head of the bodhisattva. Stainless steel or fiberglass bolts are pre-stressed with screw nuts and inserted into holes filled with compacted sand. This system may be used when the introduction of grout or mortar may adversely affect the stone. Furthermore, it is chemically neutral and the bolts may be removed with little or no damage to the stone.²⁹

²⁸ Zhong Shihang, "Application of Cartridge-Type Grouting in Grotto Conservation," in *Conservation of Ancient Sites on the Silk Road: International Conference on the Conservation of Grotto Sites, Mogao Grottoes at Dunhuang, October, 1993*, ed. Neville Agnew (Los Angeles: The Getty Conservation Institute, 1997) 181-182.

²⁹ Gerd Gudehus and Thomas Neidhart, "Geotechnical Stability Problems of the Dafosi Grotto," in Conservation of Ancient Sites on the Silk Road: International Conference on the Conservation of Grotto Sites, Mogao Grottoes at Dunhuang, October, 1993, ed. Neville Agnew (Los Angeles: The Getty Conservation Institute, 1997) 253-255.

4.3 The Shift toward Compatible Conservation Materials

In 1981, ICCROM sponsored a symposium specifically focused on the mortars, cements, and grouts used in historic buildings. A major concern expressed by many of the experts in the field was the lack of testing methods available to assess the mechanical and moisture related properties of the historic mortars, as well as the new mortars which were formulated based on ancient recipes. Most of the tests available at the time were designed for Portland cement grouts and mortars. The curing process and the materials used to create the molds did not simulate in-situ conditions, such as adjacent materials which might be in contact with the grout. The lack of appropriate tests prevented conservators from properly identifying the desirable properties of a historic mortar and then replicating those properties in new formulations.³⁰

The ICCROM Research Program (1979-1981) began an initiative to better define the desired properties for mortars specifically used in conservation. Partnering with the Istituto di Scienza delle Construzioni, the Centro di Studio cause di Deperimento e Metodi di Conservazione delle Opere d'Arte, and the Scuola di Specializzazione per le Studio ed il Restauro dei Monumenti, ICCROM outlined the potential problems with Portland cement, lime, lime-cement and hydraulic lime mortars within the context of conservation, identified the properties to be tested, and modified the tests designed for cement mortars.³¹

In addition to this research, ICCROM began investigating the use of injection grouting as a means of preserving architectural finishes architectural surfaces in-situ, and developed hydraulic lime grout formulations. Many variations of these formulations have since been developed, several of which are now commercially available. Beginning in

 ³⁰ Ingmar Holstrom, "Mortars, Cements and Grouts for Conservation and Repair: Some Urgent Needs for Research," Proceedings of the ICCROM Symposium Mortars, Cements and Grouts used in the Conservation of Historic Buildings, (Rome: ICCROM, 1982) 19-24.
³¹ S. Peroni, "Lime Based Mortars for the Repair of Ancient Masonry and Possible Substitutes," Proceedings of the ICCROM Symposium Mortars, Cements and Grouts used in the Conservation of Historic Buildings, (Rome: ICCROM, 1982) 19-24.

2004, the Getty Conservation Institute conducted a study to develop a testing regimen for the custom and commercial injection grouts that have been developed over the past twenty five years. By combining laboratory and field tests, which were modifications of standardized tests, a more thorough understanding of the properties of injection grouts may be gauged and compared. This study culminated in a GCI publication entitled *Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces*, published in 2013. It should be noted that there are currently no standardized tests for injection grouts used for conservation purposes.³²

4.4 The Use of Injection Grouts in a Structural Capacity

Grout injection restores the monolithic quality of masonry bearing walls, creating a more homogeneous system which reduces the variance of strength throughout the wall and therefore the risk of collapse. However, uneven filling of voids may result in a higher average strength but will ultimately result in a higher variance of strength, exacerbating existing structural problems. It is therefore critical that the voids are uniformly filled.³³

The International RILEM workshop on historic mortars in 1999 specifically addressed the formulation of injection grouts used in a structural capacity to stabilize ancient masonry. Injection grouts with lower quantities of Portland cement mixed with more traditional components such as lime and natural pozzolans were examined. It was concluded that hydraulic grouts composed of the mixtures described above had the potential to restore or improve the structural characteristics of ancient masonry, while possessing compatible qualities. ³⁴

³² "Injection Grouts for the Conservation of Architectural Surfaces: Research and Evaluation," Getty Conservation Institute, accessed December 25th, 2013, http://www.getty.edu/conservation/our_projects/field_projects/grouts/index.html.

 ³³ F. Van Rickstal, "Grout Injection of Masonry: Scientific Approach and Modeling," (PhD diss., Catholic University of Leuven, 2000): 22-23.
³⁴ E. Toumbakari et al., "Methodology for the Design of Injection Grouts for Consolidation of Ancient Masonry," Proceedings of the International RILEM Workshop oh Historic Mortars: Characteristics and Tests, (Paisley: RILEM, 1999) 395-405.

The most important characteristics of injection grouts were divided into two main categories, mechanical behavior and durability. Injection grouts with optimal mechanical behavior must bond well with the historic materials and must be easily injected. The grout must also develop final strength within an acceptable timeframe. Ninety days was considered a sufficient amount of time for both the modern and the more traditional materials to reach full strength.³⁵

To achieve durability, the grout must have a similar microstructure and similar hydration properties in relation to the historic material. They must also remain stable over time and have the ability to resist both natural and man-made environmental factors. Proper adhesion further combats environmental reactions and other mechanisms of decay.³⁶

 ³⁵ E. Toumbakari et al., "Methodology for the Design of Injection Grouts for Consolidation of Ancient Masonry," Proceedings of the International RILEM Workshop oh Historic Mortars: Characteristics and Tests, (Paisley: RILEM, 1999) 395-405.
³⁶ Ibid, 395-405.

Chapter 5 Injection Grouting

5.1 Grout Properties

Injection grouts are liquid mortars or other fluid materials introduced into cracks, voids or other discontinuities by gravity or pressure injection to reintegrate or reattach detached or voided material. The critical properties of any grout are low shrinkage, no segregation, continuous bond strength with the cavity or crack walls, adequate liquid and water vapor permeability and matched thermal properties with the surrounding material, no harmful degradation products, and good durability with resistance to moisture, salts, freeze-thaw and biological agents. Determining the actual performance data for these properties depends on the specifics of the material to be grouted, the environment and microclimate, and other related factors. The ability of the grout to address the remedial problem of detachment and its stabilization, as well as the compatibility of the grout and rock over the long term, are the critical factors to address in the selection of the method and materials.

Grouting assumes that the introduction of the grout will re-establish or at least improve the discontinuities in a monolithic mass, such as the support rock, or between intentional layers such as a multi-plastered wall. Grouting as a preferred technique to mediate the detachment of the formed slabbing on the Holly Tower boulder is remedial in that it addresses the damage existing in the form of large scale surface instability. Since any grout depends on good adhesion between the grout and its adherend, in this case the surfaces of the cavity and slab, the coterminous condition of friability, due to the mineral depletion of the rock in the area of the created cavity must be addressed first through consolidation. Previous testing in Phase I has shown this is possible with good results.

The current questions this thesis addresses are if a suitable grout can be found which will satisfy the criteria of reattachment of the slab and void filling of its associated cavity after consolidation. These in turn are determined by the composition of the grout and the technique of grouting, both of which are based on the alignment, scale and access to the cavities to be filled.

5.2 Grout Components

5.2.1 Binders

The binder is the active component in the grout and the unique properties of each binder must be considered before selecting a grout formulation in order to avoid incompatibilities, in this case, with the rock. Hydrated lime-based grouts require exposure to carbon dioxide (CO₂) in the air to allow for the conversion of calcium hydroxide to calcium carbonate, a process known as carbonation.³⁷ Hydraulic lime-based grout, in contrast, sets in the absence of air, is more durable, and develops strength more quickly. However, both hydrated and hydraulic lime-based grouts are prone to high shrinkage and poor injectability. It is for these reasons that quick-setting cement-based grouts are primarily used for structural purposes.³⁸

Portland Cement, the most widely used binder in the modern era, is often favored over lime-based grouts due to the fact that it develops early strength and sets quickly, allowing the construction process to proceed at a much faster rate. Due to the addition of gypsum in Portland cement manufacture, the introduction of soluble salts may sometimes be a problem. Stabilization agents are also often added to prevent bleeding and segregation which occurs

³⁷ John Ashurst and Colin Burns, "Appendix 1: Materials and Techniques," in *The Conservation of Ruins*, ed. John Ashurst, (London; Burlington: Butterworth-Heinemann, 2007) 312.

³⁸ Beril Bicer-Simsir et al, "Lime-based injection grouts for the conservation of Architectural Surfaces," Reviews in Conservation, Vol. 10 (2009): 4.

as the heavy cement particles have a tendency to sink in water, resulting in non-uniform strength. Super-plasticizers are often added as deflocculating agents that also reduce the amount of water needed without reducing the fluidity of the grout.³⁹ Natural cement, which is also hydraulic in nature, generally has a lower compressive strength, a significantly lower modulus of elasticity, and requires more time to develop final strength than Portland cement.⁴⁰

5.2.2 Fillers – Inert and Reactive

Fillers, which include aggregates, may be inert or reactive, and are added to grouts as bulking materials which reduce shrinkage and affect the mechanical strength of the grout. Fillers should be well graded as the smaller particles render the grout easily injectable, while the larger particles lend strength and stiffness to the grout. While sand is the most widely used filler, a range of inert fillers have been developed as alternatives for sand, which are lighter in weight and do not have the same tendency to segregate.⁴¹ Additional light-weight fillers, such as microspheres, which are present in Jahn M40 crack injection grout, may also be added to increase fluidity, but can adversely affect the mechanical properties of the grout.

Pozzolans are reactive fillers which also function as binders as they react with lime when water is introduced into the grout mix to form hydraulic stable insoluble compounds. Grouts containing pozzolans will set in water or wet environments and do not require carbon dioxide to cure. Despite these advantages, pozzolans affect both the

 ³⁹ F. Van Rickstal, "Grout Injection of Masonry: Scientific Approach and Modeling," (PhD diss., Catholic University of Leuven, 2000): 44.
⁴⁰ "The American Natural Cement Revival: Reintroducing a Historic Masonry Material and One of ASTM's Oldest Standards," ASTM International, accessed January 7, 2014, http://www.astm.org/SNEWS/JANUARY_2006/edison_jan06.html.
⁴¹ Beril Bicer-Simsir et al, "Lime-based injection grouts for the conservation of Architectural Surfaces," Reviews in Conservation, Vol. 10 (2009): 4.

working properties and performance characteristics of the grout and may render it highly viscous, less workable and too strong if the improper quantities are used, and may inadvertently introduce soluble salts. ⁴²

5.2.3 Additives

Additives are often used to modify specific properties of a particular grout formulation. Fluidizers (or plasticizers), which were mentioned earlier as additives in cement grout, are the most common additives, which improve the flow of grout, while reducing segregation as well as the required amount of water. Polymer emulsions were initially considered for this project as additives which would increase the tensile and adhesion strength of the grout. Polymer emulsions are pure liquids with a relatively low viscosity. The absence of solid particles prevents them from settling or thickening due to changes in water content or the injection technique used. Despite the suitability to injection and the increase in tensile and bond strength, polymers exhibit a very different stress-strain and thermal expansion behavior than many historic materials, and are impermeable to water. Furthermore, when polymers were used as binders for grouts they did not adhere well to wet surfaces.⁴³ These characteristics would have been unsuitable for the friable, highly porous sandstone of the Holly Tower support rock which must be moistened with water prior to injection to remove debris and to pre-wet the highly porous adherend surfaces. The other major concern with the use of polymers was the introduction of a synthetic material to a highly valued Ancestral Puebloan site.

5.2.4 Water Content

The water content in any grout must be carefully determined. Grouts with high water content are more easily injected, but may also cause the colloidal particles to segregate, ultimately resulting in bleeding and a loss of final

⁴² Beril Bicer-Simsir et al, "Lime-based injection grouts for the conservation of Architectural Surfaces," Reviews in Conservation, Vol. 10 (2009): 4-5.

⁴³ F. Van Rickstal, "Grout Injection of Masonry: Scientific Approach and Modeling," (PhD diss., Catholic University of Leuven, 2000): 42-43.

strength. Furthermore, an excess of water results in greater shrinkage and cracking as it evaporates during the curing process. The end result is a grout with higher porosity and lower mechanical strength.⁴⁴ The minimum amount of water recommended by the manufacturer was used when mixing the Jahn M40 crack injection grout used for testing.

5.3 Grouting Techniques

One of the primary injection techniques relevant to conservation projects is gravity grouting, in which grout with very low viscosity is introduced into a cavity through openings at the top, eventually filling the entire cavity.⁴⁵

The second technique involves injecting grout with the use of hand syringes, tubes and cannulae or by mechanical pumps. To fill large voids, the grout is injected in lifts, usually from bottom to top, displacing air with grout as the process proceeds. As each successive lift hardens and loses moisture, the process is repeated at a set distance above the previous one. ⁴⁶

5.4 Jahn M40 Crack Injection Grout

Jahn M40, a commercial grout manufactured by Cathedral Stone (USA) and designed specifically for the conservation of traditional masonry, was selected for testing as a possible candidate for use at Holly Tower. Jahn M40 crack injection grout is described as a mineral-based grout.⁴⁷ Gravimetric analysis revealed that it was primarily composed of well graded fine sand, well graded microspheres, and a binder which is presumed to be a mixture of natural and artificial cements (chapter 7.3). Its selection for consideration was based on previous testing and use on other

⁴⁴ Beril Bicer-Simsir et al, "Lime-based injection grouts for the conservation of Architectural Surfaces," Reviews in Conservation, Vol. 10 (2009): 6.

⁴⁵ Beril Bicer-Simsir and Leslie Rainer, *Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces*, (Los Angeles: Getty Conservation Institute, 2013) 2.

⁴⁶ Ibid, 2.

⁴⁷ "M40 Crack and Void Injection Grout," Cathedral Stone Products, accessed April 30, 2014, http://www.cathedralstone.com/products/jahngrouts/m40.

projects, including rock reattachment at El Morro National Monument.⁴⁸ Future testing of additional options will include other commercial and custom-grouts.

⁴⁸ Dawn M. Melbourne, "A Comparative Study of Epoxide Resin and Cementitious Grouts for the Delamination of Sandstone at El Morro National Monument, New Mexico." (Master's thesis, University of Pennsylvania, 1994).

Chapter 6 Summary of Previous Stabilization and Recent Research

6.1 Previous Stabilization Campaigns for Holly Tower

Holly Tower has undergone a series of stabilization campaigns over the last 70 years after park officials determined that the tower was in danger of collapsing. In 1941, temporary wood bracing was installed to stabilize both the walls and the footing of the tower. In 1948, cement, steel rods, and sandstone blocks were used to permanently stabilize the tower, while pigmented cement mortar was applied to areas with extensive mortar loss. In 1963, "brush-type plastic cement" was applied to the top of the tower to prevent further separation of the inner and outer courses due to moisture infiltration. In 1986, the lower courses were repointed with a stabilized earthen mortar. In 1998, twenty five percent of the wall joints were repointed and chinking stones were inserted. Ninety-five percent of the areas repointed during earlier stabilization campaigns were replaced with soil mortar.⁴⁹

6.2 Recent Conservation Research

6.2.1 *Condition Survey*

In contrast, very little has been done to address the rapid deterioration of the support rock. Although it was examined in the 1990's, a condition assessment and treatment plan was not developed until 2007. Despite this proposal, however, no treatments were implemented. During previous thesis research from 2012 to 2013, a condition survey was completed. A condition glossary was created which identified the conditions present on the surface of the support rock based on visual observation, dividing them into sub-categories which included the

⁴⁹ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013) 7-9.

methods of detachment or erosion, conditions produced as a result of material loss, and discoloration and surficial deposits. Particular importance was given to areas displaying detachment unrelated to the orientation of the bedding planes. These areas were often loose to the touch.⁵⁰ The most alarming method of detachment, however, was described as slabbing, which was detachment on a much larger scale and involved the formation of slabs several inches thick. These slabs were separating from the main body of the support rock and often occurred above areas displaying basal detachment.⁵¹ The most recent event of slabbing occurred on the northwest corner of the support rock and was attributed to freeze-thaw cycling and the erosion of a geological joint.⁵² Large slabs have also developed on the central area of the south face of the support rock, but it is the large partially suspended slab in front of an inverted cavity on the west side of the support rock which was identified as the area requiring immediate intervention.⁵³

When examined as a whole, the deterioration of the support rock was attributed to the varying mineralogical composition of the bedding strata, the presence of swelling clays and salts, the vertical orientation of the bedding strata, freeze-thaw cycling and the highly porous condition of the stone.⁵⁴

6.2.2 Laboratory Testing of Stone Consolidant and Anti-Swelling Agent

In the first stage of laboratory testing, a two-step treatment process was designed to simultaneously improve the cohesive strength of the friable surfaces of the stone and to reduce the swelling of the clay minerals intrinsic to the stone which are subject to hydric expansion, particularly during periods of heavy precipitation during the summer

⁵⁰ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013) 46.

⁵¹ Ibid, 52.

⁵² Ibid, 53.

⁵³ Ibid, 54.

⁵⁴ Ibid, 128.

monsoon season. Funcosil Antihygro, an anti-swelling agent, was applied first using the brush method and allowed to cure for 7 days. This product was chosen due to the fact that it would not adversely affect the penetration depth of the stone consolidant, and it had the potential to reduce hygroscopic expansion by 40-60%. Following this, Remmers KSE 300 E, an elastified ethyl silicate stone consolidant, was also brush applied and allowed to cure for 28 days. This product was selected to increase the modulus of elasticity of the stone by depositing silica within the pore and at the intergranular boundaries of the sandstone grains to increase cohesion. It was especially suited to medium and coarse-pored stone such as Dakota sandstone.⁵⁵

The results of the tests indicated that the treatments dramatically increased the cohesive strength of the stone when tested with the resistance drill by 400% when the stone was wet, and 300% when the stone was dry. The ability of the stone to withstand freeze-thaw cycling also increased by 400%. Furthermore, the porosity of the stone decreased by 48%, the water vapor transmission decreased by 49%, the capillary absorption decreased by 96% and the drying rate decreased by 7%. These results indicated that if the treatments were implemented on-site, the cohesion of the stone would improve, the amount of water which would be absorbed from precipitation and groundwater would dramatically decrease, and the rate of evaporation (drying) of the stone would be minimally affected and the stone would not retain moisture significantly longer than the untreated stone.⁵⁶

6.2.3 Preliminary On-Site Testing of Stone Consolidant and Anti-Swelling Agent

In the summer of 2013, small, friable areas on the east and west faces of the support rock were demarcated and treated with Funcosil Antihygro and Remmers KSE 300 E using the brush method. Due to time limitations, the

⁵⁵ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013) 79.

⁵⁶ Ibid, 129-130.

Remmers was applied 24 hours after the Funcosil instead of allowing for a seven day cure time between treatments as recommended by the manufacturer. Stable or non-friable areas adjacent to the treated areas were also demarcated, and RILEM water absorption tests which conformed to RILEM Test No. 11.6 were also performed in these areas. During these tests, the ambient temperature and relative humidity were recorded using a Fluke 971 temperature humidity meter. Following this, a cordless drill was used to drill 3 holes in both the treated and untreated demarcated areas. The holes were positioned in a triangular configuration which was measured so that each hole was the same distance from the other two holes. Each hole was drilled to the same depth and loose particulate matter was removed using a compressed air canister. Stainless steel pins (bone screws) were inserted into each hole to the same depth using an allen wrench and the depth of each pin was measured with digital calipers. The pins were left in place to monitor surface erosion.



Figure 10. Demarcation of friable and non-friable areas and application of consolidant. Photographs by author, 2013.



Figure 11. Preparations for RILEM water absorption test. Sources: Laura Lacombe and Milot Berisha, 2013.



Figure 12. Installation of stainless steel pins to monitor surface erosion. Sources: Laura Lacombe, Milot Berisha and author, 2013.

Chapter 7 Methodology

The goal of the testing regimen was to determine the suitability of Jahn M40 for the specific and unique site conditions at the Holly Tower support rock. Given the previous testing of Jahn M40 on similar projects such as Inscription Rock at El Morro National Monument in New Mexico⁵⁷, it was considered a suitable grout for this project.

7.1 Optimal Properties

The optimal properties of injection grouts were determined by the composition and condition of the sandstone support rock, the localized conditions of the inverted cavity to be filled, and the environmental conditions of the site. Optimal properties of the grout were identified and standardized tests were then selected to determine not only the performance of the grout, but the grout and stone as a composite system.

| Working Properties: | Tests for Working Properties: | | | |
|--|---|--|--|--|
| Non-segregating/non-expanding | Expansion and Bleeding - ASTM C 940-10a | | | |
| Good fluidity (complete filling of voids) | Flow - ASTM C 939-10 | | | |
| Curing/Setting: | Tests for Curing/Setting Properties: | | | |
| | | | | |
| Low shrinkage (as determined by the propagation of cracks) | Visual shrinkage - ASTM C 1148-92a | | | |

⁵⁷ Dawn Marie Melbourne, "A Comparative Study of Epoxide Resin and Cementitious Grouts for the Delamination of Sandstone at El Morro National Monument, New Mexico," (M.S. Thesis, University of Pennsylvania, 1994).

Low shrinkage (as determined by changes in length)

Early Set

Hardened/Mechanical Qualities:

Good bond strength relative to shear forces to restore monolithic quality to support rock

Good strength when tensile forces are applied

Stone/Grout Compatibility:

Thermal expansion

Drying shrinkage - ASTM C 1148-92a (2008)

Time of Setting by Vicat Needle (ASTM C 953-10 / ASTM C 191-08)

Tests for Hardened/Mechanical Properties:

Shear Bond Strength ASTM D 905-08 (2013) / EN 196-1 (2005)

Splitting Tensile Strength - EN 1771 (2004)

Tests for Compatibility:

Thermal Expansion - ASTM C 531-00 (2012)

7.2 Testing Matrix

| Test Matrix for Evaluation of Grouting of Dakota Sandstone at Holly Tower Support Rock | | | | | | | | | |
|--|------------------------------------|---|-------|-------|----------|---|---|--|--|
| Property Categories | Test | Standard | Grout | Stone | Assembly | Period | Samples Used | | |
| | Expansion and Bleeding | ASTM C 940-10a | Yes | N/A | N/A | 1 day | N/A | | |
| Working Properties | Flow | ASTM C 939-10 | Yes | N/A | N/A | 1 day | N/A | | |
| | Visual Shinkage | ASTM C 1148-92a (2008) / ASTM C 474-13 | Yes | N/A | N/A | 28 days total | (4.1, (4.2, (4.3, (4.4 (4.5 | | |
| Properties during Curing and Setting | Drying Shrinkage | ASTM C 1148-92a (2008) | Yes | N/A | N/A | 28 days total | C1.1, C1.2, C1.3, C1.4, C1.5, C1.7 | | |
| | Time of Setting by Vicat Needle | ASTM C 953-10 / ASTM C 191-08 | Yes | N/A | N/A | 1 day | N/A | | |
| Mechanical / Hardened Properties | Shear Bond Strength | ASTM D 905-08(2013) reapproved EN 196-1 (2005) | N/A | N/A | Yes | pre-soak stone for 24 hrs prior to pouring grout min 28 days to cure + 1 day testing | A2.2 A3.1 B2.3 B3.1 B4.2 | | |
| | Splitting Tensile Strength | ASTM C496/C496M-11 | Yes | N/A | N/A | min 28 days to cure + test on day 28 | (2.1, (2.2, (2.3) (2.4, (2.5, (2.6) (2.7, (2.8) | | |
| Compatibility | Thermal Expansion | For grout/stone: ASTM C 531-00 (2012) For stone: ASTM D4535-08 | Yes | Yes | N/A | 2-3 days testing | B7.1, B7.2, B7.3, B8.1, B8.2, B8.3, C1.1, C1.2, C1.3, C1.4, C1.5, C1.6 | | |

Table 1. Abbreviated testing matrix; complete matrix is located in Appendix B.

7.3 Analysis of Jahn M40 Grout

Prior to testing, the grain size distribution of the Jahn M40 grout was determined using a two-step process of gravimetric analysis and standard sieving methods based on ASTM standards C 136-06 and C 144-11. This information will be used for comparative testing of other grouts in the future which will be tested for the site. By creating other grout formulations with a similar particle size distribution and rate of flow, the performance of these formulations may be compared to Jahn M40.

7.3.1 Gravimetric Analysis

After an initial attempt to dry sieve the Jahn M40, excessive electrostatic attraction between the particles, specifically the clay-sized particles, caused them to cling to the sand and microspheres and thereby distorted the dry sieving results. After the contents of each sieve screen were placed in small weighing boats, several drops of water were added to each weighing boat to separate the sand and microspheres from the clinging particles of clay and the contents were observed under the Leica MZ16 stereo binocular microscope. The microspheres in each weighing boat floated to the surface and it was then clear that a range of microsphere sizes were used in the M40 formulation.



Figure 13. Jahn M40 sand and microspheres clearly visible after water was added to the weighing boats.

In order to obtain a more accurate grain size reading, an adaptation of the gravimetric analysis of mortars based on ASTM standards C 136-06 and C 144-11 was used to determine the proportion of the sand, microspheres and claysized binder by weight. 30.42 grams of Jahn M40 was left to dry in the oven at 60°C for 25 hours. It was then placed in the desiccator at a temperature of 20.7°C with a relative humidity of 21% to cool to room temperature. Following this, the sample was placed in a 600mL glass beaker to which 300mL of deionized water and a magnetic stirring bar was added. The beaker was placed on a stirring plate on a setting of 4 for 19 hours (Figure 14). The contents of the beaker were then left to settle for 2 hours until the water was relatively clear. Due to the hydraulic nature of the binder, the components of the grout had to be separated before they began to set. Glass funnels were set into a ring clamp attached to an instrument stand and one sheet of 24cm diameter #4 grade filter paper was pre-weighed, labeled and folded into quarters before being placed in the funnels over 500mL Erlenmeyer flasks. One sheet of filter paper was prepared for the clay/binder, microspheres and sand respectively, and each was pre-wet with deionized water.

Using a glass stirring rod to direct the flow of water, the microspheres, which floated to the top, were first poured off into the first funnel with filter paper (Figure 14). This step was repeated in 15 minute intervals to prevent the binder from mixing with the microspheres. When the microspheres were removed, the glass stirring rod was used to agitate the grout mixture to levigate the lighter clay-sized particles, separating them from the heavier sand particles. Immediately after agitation, the fine particles were poured off into the next filter paper which was pre-wet just before use. This process was repeated until only the sand remained at the bottom of the beaker. Finally, the beaker was held at an angle over the third filter paper and deionized water was squirted into the beaker until all of the sand was removed. The filter papers were then placed on watch glasses and placed in the oven at $60^{\circ}C \pm 5^{\circ}$ for 24 hours to dry.



Figure 14. Sample preparation and levigation of microspheres and fines.

After removing the samples from the oven, they were placed in the desiccator at a temperature of 19.1°C with a relative humidity of 25% until they cooled to room temperature. Using a Sartorius digital scale, the weight of the filter paper and the contents were taken to obtain the dry weight. The weight of the fines, sand and microspheres could then be obtained by subtracting the weight of the filter paper and the total weight percentage of each component could then be determined.

7.3.2 Particle Size Distribution

Legend:

 $M_{CT} = Mass of container used for the total sample$ $<math>M_T = Mass of original total sample + container$ $<math>M_{ST} = Mass of original total sample$ $<math>M_{CX} = Mass of container X$ $M_X = Mass of sample X plus container X$ $M_{SX} = Mass of sample X$ $\% M_{SX} = Sample X percent of total sample$ $\% M_{rt} = Percent of total mass retained$ $\% M_{pt} = Percent of total mass passed$

After obtaining the weight percentage, the microspheres and sand were placed in the oven for 24 hours at 60°C $(\pm 5^\circ)$, transferred to pre-weighed weighing boats and the net weight of the sand and microspheres was determined using the following formula:

$$M_{ST} = M_T - M_{CT}$$

Following standard sieving methods based on ASTM C 136-06, additional pre-weighed weighing boats were prepared and labeled for each sieve size. Each sample was then placed in the top of the sieve stack, which was covered and agitated at a 20° angle from horizontal for 15 minutes. The stack was rotated approximately every 25 agitations and lightly tapped to separate the particles. The contents of each sieve were weighed and the net weight of the contents of each sieve followed by the percentage of the total amount was calculated using the following two formulas respectively:

$$M_{SX} = M_X - M_{CX}$$

$$\% M_{SX} = \frac{M_{SX}}{M_{ST}} \times 100$$

The percentage of material lost during the sieving process was determined to ensure that not more than 0.1% was lost, and then the total percentage of material which was retained on each sieve, as well as the total percentage of material which passed through each sieve was calculated using the following formulas:

$$\% M_L = \frac{M_{ST} - \Sigma M_{SX}}{M_{ST}} \times 10$$

$$\% M_{rt} = \frac{M_{coarser}}{M_{ST}} \times 100 = \Sigma \% M_{SX(on \, or \, above)}$$

$$M_{pt} = 100 - M_{rt}$$

Using this information, the graph below was generated which illustrated the particle size distribution of the sand and microspheres.

7.3.3 Results

Based on the results of the gravimetric analysis, it was determined that Jahn M40 is composed of approximately 72% fines, 27% sand and 1% microspheres by weight (Graph 1).



Graph 1. Weight percentage of the components of Jahn M40.

Based on the results of the dry sieving procedure, the sand and the microspheres were retained in sieve #50,100, 200 and pan suggesting a particle size range of $<75\mu$ m to 300 μ m and the shape of the sand can be characterized as ranging from sub-rounded to sub-angular.



Graph 2. Particle size distribution of microspheres in Jahn M40.



Graph 3. Particle size distribution of sand in Jahn M40.

Chapter 8 Stone Sample Preparation

8.1 Sample Collection

Rock fragments were collected from the base and slope of the tower in October of 2012 by Professor Frank G. Matero and John Hinchman and labeled according to where they were collected. The HOVE.HT.B series refers to fragments taken from the rock fall of the support rock, while the HOVE.HT.A series refers to those collected in the immediate vicinity of the support rock.

8.2 Sample Cutting, Labeling and Documentation

After their arrival at the University of Pennsylvania, the fragments were first squared off in the fabrication lab by Dennis Pierattini using a diamond bladed wet stone saw. This preliminary step revealed fractures which helped determine which portions of the stone were unsuitable for testing. The texture of the stone varied widely from finegrained, dense stone which was difficult to cut, to coarser, friable stone. To the extent possible, the samples used for testing were selected to reflect this range in properties. Following this, most of the stone was cut into smaller samples sizes using the same saw, lightly brushed with a natural bristle brush to remove excess particulate matter, rinsed with de-ionized water, and allowed to air dry. It should be noted that all of the samples were cut to allow testing in the same bedding orientation.

In addition to dividing the samples into two series based on sample location as noted above, the faces of the sides were numbered to ensure that the treatments were properly applied. This was done with a fine-tipped, permanent,

non-bleeding marker. A labeled backdrop was created in AutoCAD and placed beneath the samples, which were then photographed in both jpeg and RAW format before and throughout the treatment process.

8.3 Sample Treatment

Since the inverted cavity on the west face of the support rock would first be flushed and treated with consolidant prior to grouting, it was necessary to treat the samples in a similar fashion. Following on previous research⁵⁸, the treatment was a two-step process which addressed both the friability of the mineral-depleted surface, and the tendency of the rock's intrinsic clay minerals to expand with moisture. Funcosil Antihygro, an anti-swelling agent, was first applied as a pre-treatment, and allowed to cure for seven days. Following this, Remmers KSE 300 E, an elastified stone consolidant was applied and allowed to cure for a minimum of 28 days due to the fact that the consolidant temporarily imparts hydrophobic properties to the stone.

The stone samples that were prepared for the frost resistance, thermal expansion and water immersion tests were treated on all six sides to more fully understand the properties of the treated stone when exposed to extremes in weather and precipitation events. The samples tested for shear bond strength in contrast were only treated on the side which was in direct contact with the grout, thereby imitating the in-situ conditions of the cavity.

8.3.1 Pre-treatment with Funcosil Antihygro

To prepare the samples for treatment, they were placed in an oven at 43°C for 38 hours to remove excess moisture and then moved to a desiccator as they cooled to room temperature. Metal trays with absorptive pads were used as the working surface. Eight inch long glass rods were arranged at one inch intervals and taped down to prevent the

⁵⁸ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013).

samples from moving if the tray was shifted. The samples were then placed on the rods and spaced several inches apart to allow for ease in handling and rotating.

A small amount of Antihygro was poured into a plastic beaker and a natural bristle paintbrush was used to apply the treatment. Antihygro was applied to each face of the sample in a series of cycles which may be broken down as follows:

2 quick strokes = 1 application

5 applications = 1 cycle

The temperature in the lab was 23.1°C and the relative humidity was 27%. One cycle of treatment was applied to the same face of all of the samples at the same time. Following this, they were blotted and rotated after waiting at least two minutes to inspect the samples for pooling on the surface. The stone samples which were treated on all six sides required between 3 and 4 cycles before pooling was observed. The samples which were only treated on one side required 15 cycles. When the samples were inspected 7 days after pre-treatment, a thin white residue was observed on the majority of the stones, resulting in a slightly lighter color (Figure 15).


Figure 15. Formation of white residue after pre-treatment with Funcosil Antihygro.



Figure 16. Samples prepared for thermal expansion and water immersion tests prior to treatment and after applying Funcosil Antihygro.



Figure 17. Samples prepared for shear bonding strength test prior to treatment (top) and after applying Funcosil Antihygro (bottom).



Figure 18. Samples prepared for frost resistance test prior to treatment(top) and after Funcosil Antihygro (bottom).

8.3.2 Insertion of Gauge Studs

Before the consolidant could be applied to the prism-shaped samples prepared for the thermal expansion and water immersion tests, Humboldt gauge studs had to be inserted into each end of the prisms to allow them to be inserted into a length comparator. To achieve this, 5/8" deep holes had be drilled into each end which were slightly larger

than the gauge stud diameter to allow room to insert the epoxy around the entire stud. The center of each end of the prism was determined by drawing two diagonal lines at opposing corners. In order to protect the stone and minimize vibrations during the drilling process, a vertical jig designed by Dennis Pierattini in the fabrication laboratory was clamped to the bed of a Bridgeport Vertical Mill. A 9/32" diameter round shank masonry drill bit from McMaster Carr was inserted into the mill, which was run with a spindle speed of 660 RPM. The mill was set to drill to a depth of 5/8" plus the depth of the v-shaped tip of the drill bit to ensure that the gauge stud was inserted to the correct depth. A technique referred to as peck drilling was used during the drilling process in which the drill bit was repeatedly retracted while compressed air was directed at the tip to continuously remove loose material and prevent the drill bit from overheating.



Figure 19. Peck drilling the stone on the vertical mill.

Following this, it was necessary to determine the amount of epoxy which would be needed to fill the residual space between the gauge stud and the hole. To prevent excessive spreading, a small weighing boat was filled with equal parts of J-B Weld Original Cold Weld Steel Reinforced Epoxy and thoroughly mixed. A series of holes were drilled into a piece of scrap stone with the same diameter as those drilled into the prisms. Epoxy was drawn into a BD 1 mL TB syringe without a needle and various amount were inserted into the holes until it was determined that ± 0.35 mL was the optimal amount.



Figure 20. Drawing epoxy into a syringe

Following this, the holes in the prisms were aspirated with compressed air to remove loose particulate matter and the prisms were braced with a test tube rack to keep them level during as the epoxy set. Epoxy was inserted into each of the holes and the gauge studs were then placed in the hole with tweezers and rotated slightly to evenly distribute the grout. The epoxy was left to set for 23 hours before rotating the prisms to insert studs into the opposite side.

To enable the prisms to stand upright after they were rotated, a stand was constructed. A perforated desiccator tray was inverted and covered with coated paper. Several perforations were made in the paper corresponding with those in the tray with a diameter slightly larger than that of the gauge studs. Test tube racks were placed on the tray and

taped to prevent them from shifting or falling. The prisms were then placed over the perforations and the entire process of inserting the epoxy and gauge studs was repeated. After the epoxy set, the prisms were then ready to be treated with consolidant.



Figure 21. Aspirating holes in the stone; injecting epoxy into holes; inserting and rotating the gauge stud.

8.3.3 Treatment with Remmers KSE 300 E

Seven days after the samples were pre-treated with Funcosil Antihygro, Remmers KSE 300 E was applied in a similar fashion with some modifications. Due to the toxic nature of the consolidant, all work was done under the fume hood. Plastic trays were lined with absorptive pads, and eight inch glass rods were spaced and taped in the same manner as

those used with the Funcosil Antihygro. The Remmers KSE 300 E was first slightly agitated by inverting the sealed container 1-2 times. A small amount was poured into a plastic beaker and a natural bristle paintbrush was used to apply the treatment to the same sides previously treated with Antihygro. Remmers was applied to each face of the sample in a series of cycles which may be broken down as follows:

3 slow strokes = 1 cycle

The brush was slowly passed over the surface of each sample to maximize the amount of consolidant absorbed during each cycle. The temperature in the lab was 23.8°C and the relative humidity was 22%. Following the application of one cycle to the corresponding face on each sample, the stone was blotted and rotated after waiting at least fifteen minutes to inspect the surface of the samples for pooling. The stone samples which were treated on all six sides required 3 cycles before pooling was observed. The samples which were only treated on one side required between 7 and 10 cycles. The remaining samples which were treated on all six sides required 3 cycles.

Immediately after treatment, all of the samples were blotted and brushed with methyl ethyl ketone to remove excess consolidant from the surface. Due to the toxicity of the Remmers KSE 300 E and the methyl ethyl ketone, the samples were left under the fume hood for 24 hours.



Figure 22. Applying Remmers KSE 300 E and blotting samples between cycles.

Chapter 9 Mold Preparation

9.1 Grout Mold Preparation

9.1.1 Splitting Tensile Strength Molds

Molds for the splitting tensile strength test were based on sample dimensions specified in ASTM C 496/496M-11 and were created using PVC pipe with an inside diameter of 2 inches. The molds were cut to 4 inch lengths using a chop saw and cut once along the length of the pipe with the band saw to allow the molds to pried open slightly during the de-molding process. Additional ³/₄ inch lengths of PVC pipe with the same diameter with vertical slits were attached to the top of each mold with electrical tape to allow the molds to be over-poured and trimmed just before final set.



Figure 23. Cutting PVC pipe for splitting tensile strength molds with chop saw

9.1.2 Thermal Expansion / Drying Shrinkage / Water Immersion Molds

Prismatic molds for the drying shrinkage, and thermal expansion and water immersion tests were based on sample dimensions specified in ASTM...and created using a mold prototype designed by Scott Pons during his thesis work in 2005. Using ³/₄" thick Philippine mahogany for the body and ¹/₄" plywood with Philippine mahogany veneer for the

base, 316 stainless steel #6 flat head Phillips screws from McMaster Carr were used to assemble the molds. Predrilled holes with a slightly smaller diameter than the Humboldt gauge studs and the 316 stainless steel 1/4"-20 socket head screw caps were created. A ¼ - 20 tap was then twisted into the holes to cut spiral grooves into the holes to allow the gauge studs to be twisted into place.



Figure 24. Preparation of molds for thermal expansion test.

9.2 Stone / Grout Composite Mold Preparation

9.2.1 Shear Bond Strength and Frost Resistance Molds

Molds for the shear bond strength test were based on a recent publication by the Getty Conservation Institute and were adapted from the specifications in ASTM D 905. A $3'' \times 3''$ plywood base with a $\frac{1}{2}''$ offset was surrounded by acrylic sheets on all four sides, two of which were screwed on, and two of which were attached with duct tape to

allow the samples to be easily de-molded. The acrylic sides were held in place with electrical tape (Figure 25). The frost resistance molds were constructed in the same manner with exception that the plywood base was flat. The molds were designed and built by John Hinchman at the Architectural Conservation Laboratory (ACL).



Figure 25. Construction of the shear bond strength molds

To maintain the vertical orientation of the shear bond strength samples during the test, a 4" x 5 1/4" x 3/8" thick aluminum plate with a 1 1/32" x 3 1/32" central slot was designed by LRSM supervisor, Dr. Alex Radin. Using a Bridgeport steel end mill with a speed of 1750 RPM, a drill bit was lowered incrementally to remove material until a central slot was created. A second drill bit with a smaller diameter was used in a similar manner to remove additional material from the corners. Following this, a metal file and sandpaper were used to smooth the interior edges of the slot.



Figure 26. Fabrication of the aluminum plates for the shear bond strength test.

Chapter 10 Chamber Preparation

In an effort to replicate the testing methods of the manufacturer, a chamber was prepared to maintain a relative humidity between 30 and 40%. Potassium carbonate solution was filtered through 24 cm diameter #4 grade filter paper and evaporated on a hot plate. The dried salt was slightly moistened with deionized water and placed in a plastic bin where the relative humidity level was monitored (Figure 27).



Figure 27. Test chamber with potassium carbonate

After determining that the potassium carbonate was suitable for the curing chamber, ¼" thick acrylic shelves with ¼" diameter holes spaced 2 inches apart were designed with AutoCAD and cut with the laser cutter in the fabrication

laboratory (Figure 28). One version of the shelves was designed to fit into an existing stationary acrylic curing chamber. The second version was design to fit into Rubbermaid Clever Store Basic Latch 95 Quart plastic bins which would be used as portable curing chambers to store the molds just after they were filled with grout, and for various tests, such as the visual shrinkage and vicat tests.



Figure 28. Acrylic shelves for curing chambers

After constructing the shelves, the potassium carbonate in powder form was placed in both pyrex and petri dishes and moistened with deionized water to create a relative humidity between 30 and 40%.

Chapter 11 Grout Samples

11.1 Preliminary Preparations

Prior to mixing and pouring the grout, the molds for the thermal expansion and drying shrinkage tests were repeatedly coated with mineral oil on all sides several days before to prevent the grout from sticking to the molds. An acrylic sheet was cut to size to create a non-absorptive surface for the splitting tensile strength molds. Plumbers putty was used to attach the splitting tensile strength molds and the vicat molds to their non-absorptive bases. The interior surfaces of these two types of molds were also coated with petroleum jelly.

The plywood base on the shear bond strength molds was lightly coated with mineral oil twice before pouring and then blotted. During a previous iteration of the molds in which the wood was heavily saturated with mineral oil, the oil began to seep into the stone. This should be taken into account in the final results of the shear bond strength test. The stone samples that were used for this test were wrapped with electrical tape on the perimeter to prevent the grout from sticking to the stone after pouring. The stone was then soaked in deionized water for 27 hours prior to pouring. Additional preparations will be discussed later when the flow test procedure is described.



Figure 29. Wrapping and soaking stone prior to pouring

11.2 Mixing

According to the manufacturer's recommendations, the grout was initially mixed using 2.5 parts dry grout to 1 part water by volume. The minimum amount of water recommended was used to reduce the risk of shrinkage. A Milwaukee corded drill with an RPM range of 0-850 was maintained at a medium speed to comply with the manufacturer's specifications of 400-600 RPM. A "Jiffy" paddle mixer attachment was used to mix the grout in a Vollrath 12.5 quart stainless steel tapered dairy pail. The minimum mixing time of 3 continuous minutes was increased to 4 ½ minutes as there were lumps still visible after 3 minutes. Due to the number of samples required for testing, the grout had to be poured in multiple batches.

11.3 Pouring

Immediately after mixing, the grout was poured into plastic funnels to direct the flow more accurately. A stirring rod was used to continuously agitate the grout during the pouring process. The molds were poured to overflowing to account for shrinkage and settling. They were then rapped sharply on the tray to remove air bubbles. After placing the samples in the curing chamber, they were left undisturbed until just before final set, which was determined by the vicat test. Following this, they were removed from the chamber and were trimmed using a handheld hacksaw, putty trowels and painter's tools to scrape off excess grout and level the top surface. Additional information is provided in the individual test descriptions.



Figure 30. Pouring the grout.

11.4 Methodology for Numbering the Samples

Due to the number of molds, the several batches of grout had to be prepared. The grout samples were therefore labeled according to their batch number. The HOVE.HT.C1 series refers to samples produced in the first batch. As with the stone, the grout samples were labeled with a fine-tipped, permanent, non-bleeding marker.

Chapter 12 Tests for Working Properties

12.1 Expansion and Bleeding

12.1.1 Purpose

The purpose of this test, which is in compliance with ASTM C 940-10a, is to determine the amount of expansion and the accumulation of bleed water on the surface of grout just after mixing. Grouts which exhibit bleeding and segregation often become clogged during the injection process and the loss of homogeneity alters the behavior of the grout. By observing grout during the first few hours after mixing, the amount of bleed water which develops is indicative of the degree to which the water and grout remain thoroughly mixed during the initial stages of the curing process. ⁵⁹ Expansion of the grout, which will determined by measuring the total volume of the grout during the initial stages of curing, is also critical to determine prior to injecting grout into the inverted cavity at Holly Tower as any significant expansion will exert outward pressure on the suspended slab, possibly causing complete detachment and collapse.

12.1.2 Adaptations

The only adaptation to the ASTM standard was the reduction of volume of grout from 800 \pm 10 mL to 200 \pm 10 mL based on the available equipment.

⁵⁹ Beril Bicer-Simsir and Leslie Rainer, Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces, (Los Angeles: Getty Conservation Institute, 2013) 18.

12.1.3 Equipment and Materials

- 1. (2) 250 ml graduated glass cylinders accurate to 10 ml
- 2. (2) 50 ml graduated glass cylinders accurate to 1 ml
- 3. pipet or 10 ml syringe with cannula (#20 or smaller) to decant bleed water
- 4. thermometer accurate to 1°c
- 5. parafilm or plastic wrap

12.1.4 Preparation

Two grout samples were tested simultaneously for this test. Prior to mixing the grout, the temperature of the laboratory, the mixing water and all dry materials was maintained at $23.0\pm2^{\circ}$ C. The grout was then thoroughly mixed, as any lumps may have altered both the bleeding and expansive behavior of the grout. Preparations were also made to begin taking volume measurements within three minutes of mixing the grout.

12.1.5 Procedure

The temperature of the grout was recorded immediately after mixing, and the grout was then transferred to (2) 250mL graduated cylinders until the volume of each sample (Vo) was 200 ± 10 mL (or 200 cc). The graduated cylinders were placed on a flat surface which was not subject to vibration. The time and initial volume of the sample was recorded and parafilm was placed over the cylinders to prevent the bleed water from evaporating.

Readings were taken at 15 minute intervals and recorded to the nearest 1 mL of both the upper surface of the grout (Vg) and of the bleed water (Vt) until two successive readings showed no further change in the volume of grout, confirming that there was no bleeding or expansion. Upon completion of the test, the bleed water was decanted into

a 50 mL graduated cylinder by tilting the grout mixture and removing the water with a pipet or syringe without agitating the grout. The volume of the bleed water (Vw) was then recorded to the nearest 0.5 mL.



Figure 31. Expansion and bleeding test in progress

12.2 Flow

12.2.1 Purpose

This test, which conforms to ASTM C 939-10, measures the rate of flow of a specific volume of grout through a standard flow cone, thereby providing information about the fluidity of the grout formulation being tested. This is particularly important during the injection process to ensure that the grout is thoroughly mixed and does not contain lumps which would retard the rate of flow and cause the rate of flow to vary. This test is suitable for grouts which contain fine aggregate which completely passes through a number 8 sieve and has a total efflux time of a maximum of 35 seconds.

12.2.2 Adaptations

No adaptations have been made to the ASTM standard.

12.2.3 Equipment and Materials

- 1. 178mm (7.0 inch) diameter non-corroding metal flow cone with a stainless steel discharge tube which conforms to the dimensions in figure 1 of ASTM C 939-10.
- 2. container to receive grout with a minimum capacity of 2000 ml
- 3. instrument stand to support flow cone
- 4. ring clamp
- 5. carpenter's level
- 6. stop watch with a minimum time increment reading of 0.2 seconds or less.

- 7. Vollrath 12.5 quart stainless steel tapered dairy pail
- 8. Milwaukee corded drill with a range of 0-850 rpm
- 9. "jiffy" mixer attachment

12.2.4 Preparation

The flow cone was positioned in the instrument stand with the ring clamp and a carpenter's level was placed on the flow cone in three different directions to ensure that the top of the flow cone was level. The discharge tube was sealed with a thumb and 1725 mL of water was poured into the cone. The point gauge was calibrated to correlate with the surface of the water by placing it on several points around the circumference of the cone and adjusting the height as necessary. The stop watch was started at the exact moment that the finger was removed from the bottom of the flow cone and it was stopped at the first break in the continuous flow of water. The time recorded represented the efflux (or rate of flow) of the water. This preliminary test was completed twice before testing the flow of the grout.

12.2.5 Procedure

After mixing the grout, 1725 mL was poured into a bucket and used for the flow test. The temperature of the grout, the time and the ambient temperature were then recorded. While the grout was being mixed, the flow cone was filled with water and drained one minute prior to pouring the grout. A thumb was used to plug discharge tube, the grout was poured into the flow cone until the upper surface corresponded with the point gauge, and the flow test commenced within one minute of mixing the grout. The same process used for the water was repeated and the stop watch was stopped at the watch at the first break in the continuous flow of grout, at the moment light was visible

through the discharge tube. Two flow tests were conducted for each batch of grout mixed and the results were considered satisfactory if the time of efflux for each test did not differ by more than 2.49 seconds. The time of completion of mixing to the time of the test completion was also recorded.



Figure 32. Leveling flow cone and performing flow test.

Chapter 13 Tests for Curing and Setting Properties

13.1 Visual Shrinkage

13.1.1 Purpose

This test, which is loosely based on ASTM C 1148-92a (2008) and C 474-13, helps to determine the amount of shrinkage of grout which may be caused by an excessive amount of water or an inappropriately sized aggregate. This test relies on visual observation, specifically the development of cracks, during the 28 day curing period.

13.1.2 Equipment and Materials

- 1. 600 ml of dry grout
- 2. 240 ml of water
- 3. 600 ml glass beakers to measure out grout and water
- 4. Vollrath 12.5 quart stainless steel tapered dairy pail
- 5. stainless steel milkshake mixing cup
- 6. (5) 90 ml tapered unglazed terra cotta saucers ½" deep x 3 5/16" bottom diameter x 3 11/16" upper diameter
- 7. Milwaukee corded drill with a range of 0-850 rpm
- 8. "jiffy" mixer attachment
- 9. parafilm or plastic film which will not stick to the grout after it begins to dry
- 10. moist chamber

11. broad knife

13.1.3 Preparation

The saucers were immersed in deionized water for 72 hours prior to testing. Following this, they were removed from the water and blotted dry immediately before the grout was mixed and poured.

13.1.4 Procedure

After mixing the grout according to the manufacturer's recommendations, it was transferred into the stainless steel milkshake cup. Following this, the grout was slightly over-poured into the clay saucers. After air bubbles were removed by rapping the saucer on the countertop, the broad knife was used to strike the surface of the grout, making it flush with the top of the saucers. All of the samples were lightly covered with parafilm to prevent excessive evaporation and placed in the curing chamber with a relative humidity between 30 and 40% and left to cure. It was then visually examined to look for signs of shrinkage 24 hours, 48 hours and 28 days after pouring.

13.2 Drying shrinkage

13.2.1 Purpose

This test, which conforms with ASTM C 1148-92a, will help to determine the unrestrained drying shrinkage of the grout during the initial 28 day curing process. The amount of shrinkage was determined with the use of a length comparator as opposed to visual inspection which was used in the previous test. While the laboratory curing conditions differ from the field conditions, which are often more dynamic and fluctuating in nature, this test will help

determine whether the grout will shrink to an extent that will lead to differential movement between the stone and the grout during the curing process, exacerbating the existing condition.

13.2.2 Adaptations

The molds used in this test were constructed from Philippine mahogany (also known as meranti or luan) instead of the steel molds specified in ASTM C 490. The porous nature of the wood molds more closely resembled materials which would typically be adjacent to the grout in historic structures. Due to the fragile nature of the grout when cast in a thin prismatic shape, the molds were constructed per the adaptations specified in ASTM C 490 to create 1" x 1" x 6 ¼" grout prisms with a gauge length of 5inches (the distance between the innermost ends of the gauge studs). Due to the higher probability of the samples breaking during de-molding, additional samples were cast. Rather than de-molding the prisms 48 hours after pouring and taking the first measurements 72 hours after pouring, they were left to cure for seven days and were then de-molded and tested on the same day. Lastly, the relative humidity of the curing chambers was modified to replicate the grout manufacturer's laboratory testing conditions.

13.2.3 Equipment and Materials

- 1. prismatic molds to create 1" x 1" x 6 ¼" prisms
- 2. Humboldt gauge studs
- 3. straight-edged steel trowel
- 4. length comparator with a digital micrometer accurate to 0.0001in. (0.002mm)
- 5. reference bar to calibrate length comparator with an overall length of $65/8 \pm 1/8$ in.
- 6. curing chamber with a temperature of $23.0 \pm 2.0^{\circ}$ c and a relative humidity between 30 and 40%

13.2.4 Preparation

Prior to pouring, the gauge studs were inspected and screwed in to the appropriate length which would ensure that they were embedded in the grout prisms to a depth of 5/8 in. Immediately after pouring, the molds were overpoured, rapped on the counter sharply to remove air bubbles, and placed in the curing chamber. Just before final set, excess grout was removed and the top surface of the prisms was scraped flush with the top of the mold. After replacing the samples in the curing chamber, they were de-molded 7 days after pouring.

13.2.5 Procedure

Prior to measuring the samples in the length comparator, the reference bar was placed in the length comparator so that the digital micrometer read 0.0000 in. The grout prisms were marked on one end of each prism to ensure that they were placed in the length comparator with the same orientation throughout the testing process and to establish the initial position for taking readings throughout the test. The specimens were then immediately placed in the length comparator with the same end up each time and slowly rotated through one complete rotation while the gauge reading was taken. The smallest length, as well as the length at the initial position indicated by the mark, was recorded. The room temperature was recorded throughout the testing process and the reference bar was used to calibrate the length comparator whenever the ambient temperature varied by more than $\pm 1.0^{\circ}$ C from the previous use of the length comparator and just prior to measuring a set of prisms. The length of the samples was measured on day 7, 14, 21 and 28 after pouring.

13.3 Time of Setting by Vicat Needle

13.3.1 Purpose

This test, which is in compliance with ASTM C 953-10 and C 191-08, will determine the initial and final set time of the grout. The results will provide valuable information for on-site implementation, such as how long the grout may be injected before the rate of efflux decreases, and when the grout will begin to harden, allowing for the injection of the next lift of grout and the removal of the non-staining clay which may be used to seal the bottom of the cavity.⁶⁰

13.3.2 Adaptations

The first reading was taken 3 hours and 30 minutes after mixing. This time was determined by conducting a preliminary vicat test. The preliminary test further indicated that the time between initial and final set was just over one hour and therefore required that the intervals between measurements had to be decreased from 10 minutes until initial set was reached and 5 minutes until final set was reached. Plumber's putty rather than paraffin wax was used to seal the bottom of the mold prior to pouring the grout.

13.3.3 Equipment and Materials

- 1. vicat apparatus
- 2. 100mm square plane non-adsorptive plate
- 3. flat trowel with straight edged steel blade 100-150mm in length
- (2) 40mm high x 70mm (inside bottom diameter) x 60mm (inside top diameter) truncated conical ring molds per grout type

⁶⁰ Beril Bicer-Simsir and Leslie Rainer, Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces, (Los Angeles: Getty Conservation Institute, 2013) 58.

- 5. minimum of 350 ml of mixed grout (per grout type)
- 6. putty trowel
- 7. petroleum jelly
- 8. plumber's putty
- 9. curing chamber with a temperature of $23.0 \pm 2.0^{\circ}$ c and a relative humidity between 30 and 40%

13.3.4 Preparation

A curing chamber with a temperature of 23.0 \pm 2.0°C and a relative humidity between 30-40% was prepared prior to testing. Prior to pouring, the inside of the molds were coated with petroleum jelly, placed on non-adsorptive plates, and the bottom edge was sealed with plumber's putty. The temperature of the mixing water, grout, molds and base plates were maintained at 23.0 \pm 2.0°C. The ambient temperature and relative humidity recorded.

13.3.5 Procedure

The grout was slightly over-poured into the molds within two minutes after mixing, rapped sharply on the counter to remove air bubbles and placed in the curing chamber. A timer was started immediately after pouring to begin measuring the initial set time and the ambient temperature was recorded. The molds were then placed in a curing chamber for 3 hours and 30 minutes without being disturbed. Just prior to taking the first reading, the trowel was used to strike the top of the grout flush with the top of the mold by holding it an oblique angle as it was passed over the grout. The surface was then further smoothed out with the trowel as required, without compressing the grout. After 3 hours and 30 minutes elapsed, the molds were placed below the vicat apparatus and the penetration rod was positioned so that it just touched the surface of the grout. The rod was then released and allowed to settle for 30

seconds before the measurement was taken. Each measurement should be taken at least 5mm away from any previous penetration and at least 10mm away from the inner side of the mold. The needle was also wiped clean between every measurement.

Initial set was determined by taking measurements at 10 minute intervals until a penetration depth of 25mm was reached. Following this, measurements were taken at 5 minute intervals until final set was reached. Once the penetration rod reached a depth less than 0.5mm, it was determined that final set was reached. Final set was confirmed by taking two additional measurements in different parts of the sample which were obtained within 90 seconds of the first measurement.

Chapter 14 Tests for Hardened / Mechanical Properties

14.1 Shear Bond Strength

14.1.1 Purpose

The purpose of this test is to determine the bond strength between the grout and the stone when subjected to shear forces. The bond strength of an injection grout should not exceed that of the adjacent historic material, or in this case, the Dakota sandstone, due to the fact that the weaker material will fail and crack. It is therefore preferable that failure occurs either within the grout or at the grout/stone interface.⁶¹

14.1.2 Adaptations

This test is an adaptation of ASTM D 905 and complies with EN 196-1. The procedure and sample dimensions were based on the earlier El Morro grout testing program and a recent publication by the Getty Conservation Institute (GCI) which provided guidelines for testing injection grouts.⁶² The dimensions of the stone specified in the Getty publication were modified based on the stone saw that was used but the area (3" x 1 ½") at the grout/stone interface was maintained. Due to the fact that the stone was treated with consolidant, it was fully immersed in deionized water for a minimum of 24 hours to ensure that the stone was fairly saturated. After attempting to funnel grout into syringes prior to testing, air pockets in the syringe caused the grout to spatter and could not be evenly injected. It was then decided to funnel the grout directly into the molds. The curing time was reduced to 28 days due to the use of a cementitious rather than lime grout as tested by the GCI.

⁶¹ Beril Bicer-Simsir and Leslie Rainer, Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces, (Los Angeles: Getty Conservation Institute, 2013) 62. ⁶² Ibid, 62.

14.1.3 Equipment and Materials

- 1. stone assembly; (2) $3'' \times 1 \frac{1}{2}'' \times 1''$ thick pieces of treated stone per assembly
- 2. (1) plywood / acrylic composite mold with $\frac{1}{2}$ spacer per assembly
- 3. electrical tape
- 4. mineral oil
- 5. funnel
- 6. pyrex dishes filled with deionized water
- 7. Instron universal testing instrument
- 8. 5" x 5" x 3/8" thick aluminum plate with a 1 1/32" x 3 1/32" central slot
- 9. additional 3/8" thick aluminum plates to distribute load during test; sizes varied
- 10. oven
- 11. putty trowel
- 12. curing chamber with a temperature of $23.0 \pm 2.0^{\circ}$ c and a relative humidity between 30 and 40%

14.1.4 Preparation

Prior to assembly, the stone samples were oven dried for 48 hours at 60°C. Due to the fact that the stone was treated with Funcosil Antihygro, which is an organic compound, the suggested temperature of 105°C had to be modified. Following this, the stone was placed in the desiccator and allowed to cool to room temperature. Due to the stepped profile of the composite sample, the stone was wrapped with electrical tape to prevent the grout from adhering to the stone in the wrong areas. The stone was immersed in deionized water for 27 hours prior to assembly and pouring to prevent the stone from wicking moisture out of the grout during the initial curing process. It should be noted that the tape was wrapped around the stone prior to soaking the stone due to the fact that the tape would not adhere to the stone if it was wet.

14.1.5 Procedure

While the grout was being mixed, the stones were removed from the deionized water and placed into the molds. The grout was then funneled into the molds and over-poured. Each assembly was rapped on the countertop to remove air bubbles and more grout was added to ensure that the grout would not sink below the top of the stone. Following this, the molds were placed in the curing chamber. Just prior to final set, the top of the molds were scraped with a putty trowel and the electrical tape on the top of the stone was removed. After returning them to the chamber, they were de-molded 7 days after pouring and the remaining electrical tape was removed.

After 28 days of curing, the samples were inserted into the Instron universal testing instrument and secured using aluminum plates, which not only stabilized the samples, but distributed the load. After testing them under a displacement rate of 0.01 inches per minute, the point of failure was recorded and the samples were visually examined to determine whether the failure occurred in the stone, the grout, or the stone/grout interface. Lastly, the area of the grout/stone interface of each sample was measured.



Figure 33. Aluminum plates used to distribute the load during shear bond strength test.

14.2 Splitting Tensile Strength

14.2.1 Purpose

This test, which conforms to ASTM C 496/496M-11, is used to determine the tensile strength of cured grout through an indirect tension test. Tensile, rather than compressive stresses are a major concern given the conditions of the
inverted cavity at the Holly Tower support rock. The grout should have a similar (compatible) tensile strength which is slightly lower than that of the stone to prevent further damage and crack propagation.⁶³

14.2.2 Adaptations

The samples were cured in a grout chamber which complied with the manufacturer's recommendations. Balsa wood rather than plywood strips were used to distribute the weight of the Instron bearing plates during the test.

14.2.3 Equipment and Materials

- 4 inch lengths of PVC pipe with an inner diameter of 2 inches and a vertical incision along the full length of the pipe to allow the samples to be de-molded
- ³/₄" inch lengths of PVC pipe with an inner diameter of 2 inches and a vertical incision along the full length of the pipe to allow the samples to be de-molded
- 3. putty trowel
- 4. handheld hacksaw
- 5. electrical tape
- 6. plumber's putty
- 7. plastic funnel
- 8. ¹/₄" thick acrylic sheet
- 9. petroleum jelly
- 10. mineral oil

⁶³ Beril Bicer-Simsir and Leslie Rainer, Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces, (Los Angeles: Getty Conservation Institute, 2013) 28.

- 11. Instron universal testing machine
- 12. 1" wide x 1/8" thick balsa wood strips which extend slightly past the edge of either side of the PVC pipe
- 13. drafting tape
- 14. stirring rods
- 15. curing chamber with a temperature of $23.0 \pm 2.0^{\circ}$ c and a relative humidity between 30 and 40%

14.2.4 Preparation

The 4 inch and ¾ inch lengths of PVC were taped together with electrical tape to allow the molds to be over poured and prevent the grout from settling below the top of the 4 inch PVC pipe. The interior of molds were coated with petroleum jelly and attached to the acrylic sheet with plumber's putty. Mineral oil was also lightly brushed on the acrylic below each mold. The molds were initially placed on an absorbent drywall sheet, but due to the excessive amount of water that was drawn out of the grout, the samples settled and displayed excessive shrinkage. When the molds were prepared for the second time, it was decided to place the samples on a non-absorbent surface.

Immediately after mixing, the grout was funneled into the molds and allowed to overflow. The acrylic plate was rapped on the counter sharply to remove air bubbles. Following this, the molds were placed in the curing chamber until just before final set. The ³/₄" lengths of PVC pipe were removed, a handheld hacksaw was used to saw off most of the excess grout and a putty trowel was used to strike the surface of the grout, making it flush with the top of the molds. The samples were again placed in the curing chamber and de-molded 7 days later.

14.2.5 Procedure

After 28 days of curing, the samples were removed from the chamber and diametral lines were drawn on each end of the samples to ensure that the balsa wood strips were placed in the same axial plane. The diameter of each sample was determined by averaging three measurements taken with the digital calipers at the ends and middle of each sample to the nearest 0.01 inches. The length was then determined by averaging two measurements to the nearest 0.1 inches taken between the diametral lines on each side of the sample. Balsa wood strips were centered on the diametral lines and adhered with tape.



Figure 34. Measuring the diameter of the grout cylinders with digital calipers.

Each sample was placed in the Instron universal testing machine so that the wood strips were vertically aligned and in contact with the upper and lower bearing plates. Using a displacement rate of 0.1 inches per minute, the samples were subjected to indirect tensile stress until failure occurred. In addition to the data recorded by the machine, the appearance of the grout and the type of failure was documented.

Chapter 15 Tests to Determine Stone/Grout Compatibility

15.1 Thermal Expansion

15.1.1 Purpose

This test, which conforms to ASTM C 531-00 (2012) and ASTM D 4535-08, will determine the coefficient of thermal expansion for both the grout and the sandstone. This test is particularly important for the support rock at the Holly Tower support rock. The inverted cavity is west facing and therefore exposed to direct sunlight with temperatures exceeding 38°C (100°F) in the summer months. It is critical to determine whether the stone and the grout have a similar coefficient of expansion, thereby minimizing the risk of cracking and detachment.

15.1.2 Adaptations

Due to the anisotropic nature of the stone, ASTM D 4535-08 recommended using samples with different bedding orientations, however, due to the size and orientation of the bulk samples taken from the site, only thermal expansion parallel to the bedding planes could be tested.

15.1.3 Equipment and Materials

- 1. length comparator with a digital micrometer accurate to 0.0001in. (0.0025mm)
- 2. reference bar to calibrate length comparator with an overall length of $65/8 \pm 1/8$ in.
- 3. constant temperature oven capable of maintaining a temperature to \pm 3°F (\pm 1.5°C) and up to 210°F (99°C).
- 4. scale capable of weighing to $\pm 0.3\%$ accuracy

15.1.4 Preparation

The oven was calibrated to a constant temperature of 99°C and the reference bar was placed in the length comparator and the digital micrometer was adjusted until it read 0.0000 in.

15.1.5 Procedure

The samples were heated to a constant length in the oven at 99°C for 16 hours and then conditioned at 23°C for a minimum of 16 hours. After recording the length of each sample at 23°C using the length comparator, they were again placed in the oven and heated at 210°F for a minimum of 16 hours. Using a similar methodology for the drying shrinkage test, the samples were removed one at a time and measured in the length comparator, being careful not to allow the oven temperature to drop. After measuring each sample, they were placed in the desiccator to cool to room temperature.



Chapter 16 Results and Discussion

16.1 Curing Conditions

While the relative humidity in the curing chambers was generally maintained between 30-40%, there were several fluctuations in humidity both below and above this range. This was in part due to fluctuations in the ambient conditions and excessive amounts of moisture which were released from the samples during the initial stages of curing.

16.2 Q-test

This test was used to identify and discard outliers in the test results. It should be noted that this test can only be used once per set of test results and will only identify one outlier. The formula for the q-test is as follows:

$$Q = \frac{gap}{range}$$

gap = the difference between the suspected outlier and nearest value to the outlier range = the difference between the highest and lowest values

Using a 95% confidence level and the total number of samples, a q-table (Q_{tab}) was referenced. If $Q > Q_{tab}$, the suspected outlier was discarded. The q-test was not used for tests results involving composite grout/stone assemblies, as the variability of the stone resulted in a wider range of results.

16.2.1 *Q-table*

For the purposes of this report, only the critical Q values for a 95% confidence level were referenced as seen in the following table.⁶⁴

| Table of Critical Values of Q | | | | | | | | |
|-------------------------------|-------|-------|------|-------|-------|-------|-------|-------|
| Number of values (N) | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| Qcrit CL 95% | 0.970 | 0.829 | 0.71 | 0.625 | 0.568 | 0.562 | 0.493 | 0.466 |

Table 2. Q-test for 95% confidence level.

16.3 Standard Error of the Mean

To measure the variability of the sample mean, the standard error of the mean test results for each test were

calculated using the following formula:

$$\frac{S\bar{x}}{\sqrt{n}}$$

 $S\overline{x}$ = standard deviation of the mean

 $n = \text{sample size}^{65}$

⁶⁴ "Dixon's Q-test: Detection of a Single Outlier," University of Athens, Department of Chemistry, accessed, April 18, 2014, http://www.chem.uoa.gr/applets/AppletQtest/Appl_Qtest2.html.

⁶⁵ Deborah J. Rumsey, *Statistics for Dummies*, 2nd ed., (Hoboken: Wiley Publishing, Inc., 2011), 166.

16.4 T-Test

16.4.1 Paired (dependent) T-Test: Type 1 Test in Excel

The dependent or paired t-test was used to analyze the results of two sets of data taken within the same sample population under two different conditions. This only applied to the drying shrinkage test, in which the measurements taken with the length comparator on the same material at the beginning of the test were compared against those taken at the end of the test. ⁶⁶ The formula is as follows:

$$t = \frac{\bar{d}}{\sqrt{s^2/n}}$$

 \bar{d} = mean difference

 s^2 = sample variance (squared standard deviation of the mean difference) n = sample size⁶⁷

16.4.1.1 Methodology

After the test results were entered, the t statistic, degrees of freedom, and the critical value at a 95% confidence level were automatically generated in excel. If the value of the t-statistic exceeded the critical value, it was determined that the means of the two sets of results were statistically different, and the null hypothesis was rejected.

⁶⁶ "Paired t-Test," Statistical Help, accessed April 19, 2014,

http://www.statsdirect.com/help/default.htm#parametric_methods/paired_t.htm.

⁶⁷ "Paired Sample t Test," Real Statistics Using Excel, accessed April 20, 2014, http://www.real-statistics.com/students-t-distribution/paired-sample-t-test/.

16.4.2 Unpaired (independent) T-Test: Type 3 Test in Excel

The independent or unpaired t-test was used to analyze the results of data taken from two different materials when subjected to the same test. This only applied to the thermal expansion test, in which the linear coefficient of thermal expansion of the stone prisms was compared to that of the grout prisms.

$$t = \frac{\overline{x_1} - \overline{x_2}}{\sqrt{\frac{s_1^2 + s_2^2}{n}}}$$

 \bar{x}_1 = mean of first sample

 \bar{x}_2 = mean of second sample

 s_1^2 = variance (squared standard deviation) of data set 1

 s_2^2 = variance (squared standard deviation) of data set 2⁶⁸

16.4.2.1 Methodology

After the test results were entered, the t statistic, degrees of freedom, and the critical value at a 95% confidence level were automatically generated in excel. If the value of the t-statistic exceeded the critical value, it was determined that the means of the two sets of results were statistically different, and the null hypothesis was rejected.

⁶⁸ "Independent t-Test," Statistics Tutorial, University of Glasgow, accessed April 19, 2014, http://www.gla.ac.uk/sums/users/narjis/stroke/indept2.html.

16.5 F-Test

The f-test, which was also automatically generated in excel, was only used for the results of the thermal expansion test to determine whether there was more variation in the expansion of the stone samples relative to the grout samples. The formula for the f-test is as follows:

$$F = s_1^2 / s_2^2$$

 $s_1 =$ standard deviation of data set 1

 $s_2 =$ standard deviation of data set 2

 $s_1^2 =$ variance of data set 1

 $s_2^2 =$ variance of data set 2^{69}

If the value of F exceeded the critical value at the 95% confidence level, this indicated that the standard deviation of the two groups of samples tested displayed different amounts of variation.

16.6 Appendices

While calculations and charts are included in this section, the complete calculations and additional graphs are included in the appendices at the end of the report.

⁶⁹ "Comparison of Standard Deviations: The F-Test," Chemistry Department, University of Toronto, accessed April 19, 2014, http://www.chem.utoronto.ca/coursenotes/analsci/StatsTutorial/ftest.html.

16.7 Expansion and Bleeding - ASTM C 940-10a

16.7.1 Observations and Results

The following equations were used to calculate expansion and bleeding:

Expansion,
$$\% = \frac{V_g - V_0}{V_0} \times 100$$

Bleeding, $\% = \frac{V_t - V_g}{V_0} x \ 100 \ at \ prescribed \ intervals$

Combined Expansion,
$$\% = \frac{V_t - V_0}{V_0} \times 100$$

Final Bleeding,
$$\% = \frac{V_w}{V_0} \times 100$$

 V_0 = volume of the sample at the beginning of the test, mL

 V_t = volume of the sample at set intervals, mL

 V_g = volume of the grout portion of the sample at set intervals, at the upper surface of the grout, mL

 V_w = volume of decanted bleed water, mL

The expansion and bleeding test was conducted using 2 grout samples simultaneously, both with a volume of 200 mL \pm 1 mL. The grout temperature was recorded at 22.8°C immediately after mixing and the ambient temperature of the room was recorded at 22.5°C. A small amount of bleed water developed on the surface of each sample by the time the first reading was taken 15 minutes after pouring. However, the total volume of the grout remained the same throughout the duration of the test. After this initial separation occurred, the volume of bleed water remained constant. In summary, all of the readings remained constant after the first 15 minutes and the test was concluded

after 45 minutes (3 readings) to ensure that no further bleeding or expansion would occur. 0.5 mL of bleed water from each sample was decanted into 50 mL graduated cylinders using a pipet. Based on these results, there was no expansion, and the final bleeding for both samples was 0.2%. A grout formulation is acceptable when the percentage of bleeding does not exceed 5%.⁷⁰

16.7.2 Discussion

The results indicate that Jahn M40 exhibits minimal bleeding within the first 15 minutes of pouring, but does not expand during the curing process. This implies that the consistency and mechanical properties of the grout will remain uniform throughout the inverted cavity and that there is minimal risk of further or complete detachment of the suspended slab due to the expansion of the grout.

16.8 Flow - ASTM C 939-10

16.8.1 Observations and Results

Using the minimum amount of water recommended by the manufacturer to minimize shrinkage and maximize mechanical strength, a ratio of 2 ½ parts of grout to 1 part water was used for this test.⁷¹ As was mentioned earlier, the mixing time was increased from the minimum duration of 3 minutes specified by the manufacturer to 4.5 minutes to remove all of the lumps and ensure that the flow was even. A total of 4 flow tests were conducted, which corresponded to the number of batches needed to fill all of the molds. Two readings were taken for each test and averaged. The average rate of efflux for the four tests ranged from 13.3 to 14.7 seconds. In compliance with the

⁷⁰ E. Toumbakari, "Lime-Pozzolan-Cement Grouts and their Structural Effects on Composite Masonry Walls," (PhD diss., Katholieke Universiteit Leuven, 2002).

⁷¹ Beril Bicer-Simsir et al, "Lime-based injection grouts for the conservation of Architectural Surfaces," Reviews in Conservation, Vol. 10 (2009): 7.

ASTM standard, none of the results differed by more than 2.49 seconds. All of these results passed the q-test, and the

standard deviation was 0.6 seconds with a standard error of 0.3.

| Flow Test Jahn M40 | | | | | | | | |
|--------------------|--|--------------------------------------|---------------------------|----------------|--|--|--|--|
| | | | | | | | | |
| | | | | | | | | |
| Volume of grou | t and water used for all tests 1725 mL ± ! | 5 mL | | | | | | |
| 2 Readings A/B | were taken for each test | | | | | | | |
| Time of mixing | to test completion 9 min ± 1 min. | | | | | | | |
| Solid to water ra | atio: 2.5 parts grout to 1 part water | | | | | | | |
| | | | | | | | | |
| Test Number | Rate of Efflux for Grout (seconds) | Avg. Rate of Efflux for Grout (sec.) | Standard Deviation (sec.) | Standard Error | | | | |
| 1A | 14.5 | 14.7 | | 0.3 | | | | |
| 1B | 14.9 | 14.7 | | | | | | |
| 2A | 13.0 | 12.2 | 0.6 | | | | | |
| 2B | 13.5 | | | | | | | |
| 3A | 13.9 | 14.0 | 0.0 | | | | | |
| 3B | 14.0 | 14.0 | | | | | | |
| 4A | 14.1 | 12.0 | | 1 | | | | |
| 4B | 13.4 | 13.0 | | | | | | |

Table 3. Flow test results.

16.8.2 Discussion

When compared with flow tests for hydraulic lime grout with a rate of efflux ranging from 5.47 to 10.43 seconds,⁷² Jahn M40 had a significantly slower rate of efflux, but it was still within the acceptable parameters of the ASTM standard, which specifies a maximum of 35 seconds. The grout had the consistency of a milkshake when the mixing was complete and flowed evenly after the mixing time was increased to 4.5 seconds. When the grout was poured into the molds after the flow test was complete, it was fluid enough to evenly fill both the 2 inch and 1 inch wide molds which are similar in width to the inverted cavity. After de-molding the 1" x 1" x 6 ¼" grout prisms for the

⁷² Victoria Pingarron, "Performance Analysis of Hydraulic Lime Grouts for Masonry Repair," (Master's thesis, University of Pennsylvania, 2006): 45, and Kelly Wong, "Assessment of the Grout used for the Structural Stabilization of the Early Phrygian Citadel Gate at Gordion, Turkey," (Master's thesis, University of Pennsylvania, 2006): 127.

drying shrinkage and thermal expansion tests, it was observed that the grout did not completely fill the immediate area beneath the gauge studs on all of the samples when the grout was injected from above (Figure 36).



Figure 36. Mold for thermal expansion and drying shrinkage tests.

The gauge studs may be likened to protrusions within the cavity, and based on these results, it is recommended that the cavity be filled from the bottom upwards in successive lifts as opposed to the gravity grouting technique which involves injecting grout from the top of the cavity. This will help to ensure that the areas below protrusions in the cavity will be completely filled during the injection process.

The other important observation was that the viscosity of the grout increased significantly several minutes after mixing unless the grout was continuously agitated. While this may expected with hydraulic materials, it is also possible that there are components in the grout formulation which were added to induce thixotropic behavior.

Thixotropy may be described as the propensity of a wet grout to become more viscous and gel-like when allowed to rest, with the ability to return to a less viscous, liquid-like state when agitated or pressurized.⁷³ While this property allows for greater control during the injection process, as the grout will remain in place after injection, it also has the potential to interfere with the injection process by reducing the rate of flow.⁷⁴ Materials such as bentonite clay, fly ash and fumed silica impart thixotropic properties to grout formulations.⁷⁵ Due to the fact that the grout did not exhibit expansion during the expansion and bleeding test, it may be assumed that bentonite, a swelling clay composed primarily of montmorillonite, is not a component of the grout formulation. The more extreme temperature and humidity conditions on the site may further accelerate this increase in viscosity due to the accelerated evaporation of water, and further flow tests which simulate these conditions may be necessary.

16.9 Visual Shrinkage - ASTM C 1148-92a (2008) / ASTM C 474-13

16.9.1 Observations and Results

The grout samples did not exhibit any significant cracking when observed 24 hours, 48 hours and 28 days after pouring. Minor cracking was observed at the rim, indicating that the grout had contracted toward the center of the saucer (Figure 38).

⁷³ A. C. Houlsby, *Construction and Design of Cement Grouting* (New York, Chichester, Brisbane, Toronto, Singapore: John Wiley and Sons, Inc., 1990), 79-81.

⁷⁴ Ibid, 81.

 ⁷⁵ F. Van Rickstal, "Grout Injection of Masonry: Scientific Approach and Modeling," (PhD diss., Catholic University of Leuven, 2000): 77;
S. Akbulut and A. Saglamer, "The Effects of Silica Fume in Cement Grouting," Proceedings of the ICE - Ground Improvement, No. 1, (2003): 37-44.



Figure 37. Photograph of visual shrinkage test samples after 28 days of curing.



Figure 38. Detail photograph of cracking along the perimeter of the sample.

16.9.2 Discussion

The absence of cracks in the center of the samples, the area with the greatest thickness, suggests that each lift of grout injected into the inverted cavity will contract slightly toward the center but will remain intact. The question that remains is how this type of shrinkage will affect the grout as it is poured in lifts which must be spaced several hours apart to allow the grout to lose moisture and harden to the extent that it will be able to support the load introduced by the next lift of grout.⁷⁶

⁷⁶ Beril Bicer-Simsir and Leslie Rainer, *Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces*, (Los Angeles: Getty Conservation Institute, 2013) 2.

16.10 Drying Shrinkage - ASTM C 1148-92a

16.10.1 Observations and Results

Due to concerns with the samples breaking, the first readings were taken on day 7 rather than on day 3, as specified in the ASTM standard. The formula used to calculate the percent shrinkage is as follows:

$$S = \left[\frac{L_1 - L}{L_0}\right] x 100$$

S = Percent shrinkage (%)

L₀ = Effective (nominal) gauge length (in.)

- $L_1 =$ initial measurement after removal from molds on day 7
- L = final measurement taken on day 28

After performing the q-test, no outliers were detected among the final results. The average percent shrinkage was 0.075%, with a standard deviation of 0.005 and a standard error of 0.002. The paired (or dependent) t-test was used to compare the mean percent shrinkage of the samples by comparing the initial measurements taken on day seven against the total shrinkage recorded on day seven when the test was completed. Due to the fact that the t Stat value (39.4613) exceeded the critical value at the 95% confidence interval (2.5706) at 5 degrees of freedom, the null hypothesis was rejected, indicating that there was a difference in the mean between the initial length measurements and the final measurements taken after curing.

| Drying Shrinkage Test | | | | | | | | |
|-----------------------|-------------------|---------------|---------------|----------|--|--|--|--|
| Calculations | | | | | | | | |
| Comple # | Percent Shrinkage | Avg. Percent | Standard | Standard | | | | |
| Sample # | (%) S | Shrinkage (%) | Deviation (%) | Error | | | | |
| (1.1 | 0.076 | | | | | | | |
| (1.2 | 0.082 | | 0.005 | 0.002 | | | | |
| (1.3 | 0.078 | 0.075 | | | | | | |
| C1.4 | 0.070 | 0.075 | | | | | | |
| (1.5 | 0.076 | | | | | | | |
| C1.7 | 0.070 | | | | | | | |

Table 4. Calculations for drying shrinkage test.



Graph 4. Results for drying shrinkage test.

| t-Test: Paired Two Sample for Means | | | | | | | |
|-------------------------------------|-----------------|----------------------|--|--|--|--|--|
| | Initial Reading | Final Reading | | | | | |
| Mean | 6.65E+00 | 6.65E+00 | | | | | |
| Variance | 2.80E-04 | 2.76E-04 | | | | | |
| Observations | 6 | 6 | | | | | |
| Pearson Correlation | 1.00E+00 | | | | | | |
| Hypothesized Mean Difference | 0 | | | | | | |
| df | 5 | | | | | | |
| t Stat | 39.46131171 | | | | | | |
| P(T<=t) one-tail | 9.84992E-08 | | | | | | |
| t Critical one-tail | 2.015048373 | | | | | | |
| P(T<=t) two-tail | 1.96998E-07 | | | | | | |
| t Critical two-tail | 2.570581836 | | | | | | |

Table 5. Results for paired t-test.

16.10.2 Discussion

The average percent shrinkage of 0.075% was minimal, and the majority of the shrinkage occurred between day 7 and day 14 (Graph 5). Due to the higher modulus of elasticity of cementitious grouts such as Jahn M40, shrinkage, however minimal, raises more concern about detachment at the grout/stone interface than traditional lime-based grouts.

Dakota sandstone, which ranges from coarse-grained and highly porous to fine-grained with low porosity, has the potential to draw a large amount of moisture out of the grout if the stone is not properly moistened prior to grouting. Based on the results of previous capillary water absorption tests, untreated stone absorbed the majority of the total water gained during the first three hours. In contrast, the stone treated with consolidant and an anti-swelling agent absorbed moisture very slowly during the first 4 days, after which the absorption rate increased. This was attributed to the fact that the water had exceeded the penetration depth of the consolidant on the fourth day and was then able to flow more freely into the untreated core of the stone samples.⁷⁷ These results were further confirmed when composite stone/grout samples for the shear bond strength test were prepared. Due to the fact that the stone samples dried too quickly, they had to be immersed in a deionized water bath for a minimum of 24 hours and were placed in the molds immediately before pouring the grout.

Based on these observations, several recommendations may be made. Firstly, further tests may be needed to gauge how much water the stone will draw from the grout during the initial stages of curing. ASTM standard C1506-09, the standard test for water retention of hydraulic cement-based mortars and plasters, was considered, but section 4.3

⁷⁷ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013) 104-105.

specifically states that the results obtained from the test would not reflect the water retention of the grout when used with masonry units. An adaption of this test may be necessary to properly gauge the amount of water retained by a specific grout formulation which is in direct contact with Dakota sandstone from two sides, simulating the conditions in the inverted cavity.



Graph 5. Shrinkage curve for grout samples recorded over 28 days.

16.10.3 On Site Implications

Regarding on-site implementation, if the inverted cavity is treated with consolidant, it may be necessary to devise a method to repeatedly moisten the stone for several days prior to grouting, possibly wrapping the stone with an impermeable textile between periods of wetting. To discourage biological growth and increase wetting, denatured alcohol may need to be added to the water.

16.11 Time of Setting by Vicat Needle - ASTM C 953-10 / ASTM C 191-08

16.11.1 Observations and Results

As mentioned in the methodology, the readings for 2 samples were taken simultaneously at 10 minute intervals until initial set was reached and at 5 minute intervals until final set was reached. Due to the rapid setting time, the graph was plotted in 5 minute intervals which began 3 hours and 30 minutes (210 min.) after the grout was poured into the molds. Sample A reached initial set 4 hours and 4 minutes (244 min.) after pouring, and sample B reached initial set 4 hours and 12 minutes (252 min.) after pouring. Sample A reached final set 5 hours and 5 minutes (305 min.) after pouring, and sample B reached final set 5 hours and 10 minutes (310 min.) after pouring. It should be noted that the penetration depth was not consistent throughout the samples and the test was concluded when a depth of less than 0.5mm was recorded in 3 different locations.



Graph 6. Results of vicat test (initial set indicated with black dots).

16.11.2 Discussion

The results of the test indicate that the grout will remain workable for approximately 4 hours and then rapidly reach final set 5 hours after injection. This implies that each lift of grout must be injected into the cavity at least 5 hours after the preceding lift. Due to the fact that lifts are injected from the bottom upwards, each lift must have time to cure to an extent that it retains its form (is no longer workable) and has developed sufficient strength to support the next lift without being damaged. Final set represents this threshold in the curing stage.

Cementitious grouts reach final set at a much faster rate than traditional lime-based grouts. While this will allow for each lift to be injected into the cavity more quickly, it also raises concerns. The outward-pushing hydrostatic pressure exerted by each lift of grout will fluctuate during the injection process as each new lift is injected and begins to cure. The rapid setting time means that the grout will reach final set well before the injection process is complete, therefore, any movement of the partially suspended slab due to hydrostatic pressure and changes in temperature may ultimately affect the bonding process between the grout and the stone. Further tests should be conducted at the site to determine the impact of the temperature and relative humidity on the setting time.

16.12 Shear Bond Strength - ASTM D 905 / EN 196-1

16.12.1 Test Modification

During the curing process, detachment between the one stone adherend with restricted lateral movement and the grout occurred in all but one of the samples (Figure 39). The results of the visual shrinkage test demonstrated that the grout contracted towards the center during the curing process, separating from the rim of the terracotta saucers. When the grout was injected into the molds for the shear bond strength test, the grout pulled away from the rigidly fixed adherend and pulled the unrestricted adherend inward as it shrank, allowing the stone/grout bond to be maintained. The test was modified to test the bond strength between the remaining intact portion of the samples.



Figure 39. Plane of breakage in shear bond strength samples during curing process.

16.12.2 Observations and Results

After several trial tests, the testing parameters for the Instron universal testing machine were programmed as follows:

Load:
$$1 \text{ volt} = 25 \text{ lbs}.$$

Displacement: 1 volt = 0.002 inches

The formula used to calculate shear bond strength is as follows:

$$f_{SB} = \frac{F}{A_B}$$

 $f_{SB} =$ shear bond strength (psi)

F = breaking load (lbs.)

AB = bond line area (in.²)

A total of five samples were tested and the all of the samples failed at the grout/stone interface. The relatively clean break left the stone completely intact, exhibiting ideal failure. The average shear bond strength was 34.25 psi, with a standard deviation of 31.50 and a standard error of 14.09. A dramatic difference was noticed between the coarser grained samples with a higher porosity (A) and the finer grained samples with a lower porosity (B) (Graph 7). The range in shear bond strength between the A samples was approximately 55-79 psi, while the range in the B samples was between 7-18 psi. Based on these results, it may be inferred that the coarser stone with higher porosity allowed for a much stronger bond due to the fact that the grout was able to penetrate further into the stone due to suction.

The results were compared with shear bond strength tests conducted during previous research on the use of Jahn M40 crack injection grout to reattach delaminated Zuni sandstone at El Morro National Monument in New Mexico. The average bulk density of the Zuni sandstone was 2.17cm³, and based on this result, it was determined whether the shear bond strength between the grout and the stone would be adequate to support the weight of the reattached stone. The mean shear bond strength was 72.2 psi, which was considered an acceptable strength.⁷⁸ This result roughly corresponded to the results of the coarse grained Dakota sandstone samples and the Jahn M40 injection grout for this project. Based on this comparison, the fine grained stone samples did not possess an acceptable level of shear bond strength to support the partially detached slab.

⁷⁸ Dawn M. Melbourne, "A Comparative Study of Epoxide Resin and Cementitious Grouts for the Delamination of Sandstone at El Morro National Monument, New Mexico." (Master's thesis, University of Pennsylvania, 1994): 45-57.

| ihear Bond Strength Calculations | | | | | | | | |
|----------------------------------|----------------|-----------------|-----------|----------|--|--|--|--|
| | Shear Bond | Avg. Shear Bond | Standard | Standard | Stone Characterization | | | |
| Sample | Strength (psi) | Strength (psi) | Deviation | Error | C = coarser grain size/higher porosity | | | |
| | fsb | Avg. fsb | (psi) | | F = finer grain size/lower porosity | | | |
| A2.2 | 55.23 | | | | C | | | |
| A3.1 | 79.22 | | | | C | | | |
| B2.3 | 7.90 | 34.25 | 31.50 | 14.09 | F | | | |
| B3.1 | 10.74 | | | | F | | | |
| B4.2 | 18.17 | | | | F | | | |

Table 6. Shear bond strength test results.



Graph 7. Comparison of shear bond strength test results.

16.12.3 Sample Inspection after Shear Bond Strength Test

Examination of the samples after the test revealed that only 34-54% of the grout was in full contact with the stone during the test. As a result, the load applied during the test was concentrated within and therefore limited to the area of full contact, or the bond line area. The calculations for shear bond strength were therefore modified to apply to the bond line area rather than the original grouted area. The shaded red areas in the images below represent the bond line area and the dashed red lines indicate the original grouted area. The samples were also examined to determine if there were any recurring patterns in the location of the bond line area in relation to the top or bottom of the sample to further understand the uneven bonding, possibly due to uneven rates of drying during the curing process. The bottom of each sample has been indicated in each of the images below, but no recurring patterns were detected.



Figure 40. Sample A2.2 – Original grouted area: 2.850 sq. in. / Bond line area: 1.156 sq. in.



Figure 41. Sample A3.1 – Original grouted area: 2.935 sq. in. / Bond line area: 1.022 sq. in.



Figure 42. Sample B2.3 – Original grouted area: 3.039 sq. in. / Bond line area: 1.623 sq. in.



Figure 43. Sample B3.1 – Original grouted area: 2.859 sq. in. / Bond line area: 1.534 sq. in.



Figure 44. Sample B4.2 – Original grouted area: 3.021 sq. in. / Bond line area: 1.021 sq. in.

16.12.4 Discussion

While the use of mineral oil on the plywood base of the molds may have affected the bonding process, the fact that all of the samples failed at the grout/stone interface during the test, and that the bond line area was limited to 34-54% of the original grouted area, warrants further investigation. Not only should the design and preparation of the molds be revisited, but the test should be repeated using both treated and untreated samples to determine whether the consolidant treatment affected the bonding process. Bulk density tests should be conducted to determine the minimum acceptable shear bond strength value. It should also be taken into account that the smooth profile of the stone blocks, which did not simulate the site conditions, may have also affected the test results due to the reduced surface area between the grout and the stone.

16.12.5 On-site Implications

The repeated clean detachment of the grout from the stone samples which were held in a rigid position in the molds during the curing process may provide insight into how the grout would perform on site. The stone/grout/stone configuration of the samples (Figure 39) reflects the in-situ condition of the inverted cavity on the west face of the support rock. The larger intact mass of the support rock may be likened to the stone adherend which is restricted from moving laterally, and the suspended slab which has begun to behave and deflect independently may be likened to the unfixed piece of stone. Based on the shrinkage and detachment observed in the laboratory, it is possible that the grout injected into the inverted cavity will exhibit the same pattern of shrinkage and detach from the larger mass of the support rock which is unable to move laterally. Not only would this add substantial weight to the partially suspended slab, but it could result in total detachment. Further testing is required to make this determination.

16.13 Splitting Tensile Strength - ASTM C 496/496M-11

16.13.1 Observations and Results

After several trial tests, the testing parameters for the Instron universal testing machine were programmed as follows:

Load: 1 volt = 500 lbs.

Displacement: 1 volt = 0.05 inches

The formula used to calculate splitting tensile strength is as follows:

$$T = 2P/\pi LD$$

T = splitting tensile strength (psi)

P = maximum applied load (lbs.)

L = average length of sample (in.)

D = average diameter of sample (in.)

During the test, the first sign of failure was a vertical crack through the entire sample which was on or near the diametral lines. Samples C2.3 and C2.4 also displayed a large curved crack from the edge of the balsa wood strip under the sample towards the center. Following this, additional vertical cracks formed until complete failure occurred. The q-test was applied to the test results to detect outliers, but none of the results were discarded. The average splitting tensile strength was 199 psi., with a standard deviation of 24.9 and standard error of 10.2.

| Splitting Tensile Strength Test | | | | | | | | | |
|---------------------------------|------------------------------|--------------------------------|-------------------------------|------------------------------------|--|--|--------------------------------|-------------------|--|
| Sample # | Average Length (in.) L | Average Diameter (in.) D | Length x Diameter L x D | Maximum Applied Load (lbs) P | Splitting Tensile Strength (psi) T | Avg. Splitting Tensile Strength (psi) Avg. T | Standard Deviation (psi) | Standard Error | |
| (2.1 | 4.04 | 2.07 | 8.36 | 2416.70 | 184 | | | | |
| (2.2 | 4.04 | 2.06 | 8.32 | 2889.59 | 221 | | | | |
| (2.3 | 4.05 | 2.08 | 8.42 | 2161.57 | 163 | 100 | 24.0 | 10.2 | |
| Q.4 | 4.05 | 2.07 | 8.38 | 2839.48 | 216 | 199 | 24.9 | 10.2 | |
| (2.5 | 4.05 | 2.06 | 8.34 | 2435.22 | 186 | | | | |
| C2.6 | 4.02 | 2.04 | 8.20 | 2894.43 | 225 | | | | |

Table 7. Splitting tensile strength test results


Graph 8. Comparison of test results.



Figure 45. Splitting tensile strength test in progress.

16.13.2 Sample Inspection after Splitting Tensile Strength Test

The fragments of each sample were examined after the test and most of the breaks were clean, running through the entire length. Several air bubbles were observed in sample C2.3 (Figure 48). While this sample exhibited the lowest splitting tensile strength, the result was not low enough to be considered an outlier when the q-test was conducted.



Figure 46. Splitting tensile strength procedure and fracture pattern for sample C2.1.



Figure 47. Splitting tensile strength procedure and fracture pattern for sample C2.2.



Figure 48. Splitting tensile strength procedure and fracture pattern for sample C2.3.



Figure 49. Splitting tensile strength procedure and fracture pattern for sample C2.4.



Figure 50. Splitting tensile strength procedure and fracture pattern for sample C2.5.



Figure 51. Splitting tensile strength procedure and fracture pattern for sample C2.6.

16.13.3 Discussion

While splitting tensile strength tests have not yet been performed on sample stone cores from the site treated with consolidant, it is necessary to gain at least a preliminary understanding of splitting tensile strength of Jahn M40 relative to Dakota sandstone. An environmental impact report was prepared for the U.S. Army Corps of Engineers regarding the surface stabilization of Dakota sandstone at Kanopolis Lake in Kansas, the site of the Faris Cave petroglyphs. Conservare OH, another ethyl silicate stone consolidant, was applied to cored samples which were then tested for compressive strength in psi.⁷⁹ By converting these values into splitting tensile strength, it is possible to compare untreated, and treated stone samples against the Jahn M40 grout samples.⁸⁰

| Compressive Strength of Dakota Sandstone Relative to Jahn M40 Injection Grout | | | | | | |
|---|-------------------------------|-------------------------------------|---|--|--|--|
| | Compressive Strength (psi) | Splitting Tensile Strength (psi) | Percent Increase in Strength for Dakota Sandstone (%) | | | |
| Jahn M40 Grout samples | 1417 | 199 | N/A | | | |
| Untreated Stone | 1,080 | 164 | 0 | | | |
| 1 treatment cycle of OH | 2,550 | 302 | 184 | | | |
| 2 treatment cycles of OH | 3,050 | 343 | 209 | | | |
| 3 treatment cycles of OH | 4,750 | 470 | 287 | | | |

Table 8. Comparison of splitting tensile strength values⁸¹

⁷⁹ David Grisafe, "Stabilization of Dakota Sandstone Surface of the Faris Cave Petroglyphs, Kanopolis Lake Project, Kansas," Department of the Army, U.S. Army Corps of Engineers, (1992): 16.

⁸⁰ N.J. Carino and H.S. Lew, "Re-examination of the Relation between Splitting Tensile Strength and Compressive Strength of Normal Weight Concrete," *ACL Journal*, Vol. 79, No.3, (1982): 214-219.

⁸¹ "Strength Converter," ACPA, accessed April 19, 2014, http://apps.acpa.org/apps/StrengthConverter.aspx.

Previous testing was conducted on the Dakota sandstone at the Holly Tower support rock with DRMS (the drilling resistance measurement system), which determined the amount of force necessary to drill to a specific depth, while maintaining a constant rotation speed and penetration rate.⁸² While a direct relationship to between the splitting tensile strength and the drilling resistance of the stone cannot be made, the results of the drilling resistance test indicated that the strength of the stone treated with three cycles of Remmers KSE 300 E increased by 300% when the stone was dry, which roughly corresponded with the 287% percent increase of the splitting tensile strength of the stone of the stone of the stone of the splitting tensile strength of the stone treated with the 287% percent increase of the splitting tensile strength of the stone of the stone of the splitting tensile strength of the stone of the stone of the splitting tensile strength of the stone of the stone of the splitting tensile strength of the stone of the stone of the splitting tensile strength of the stone when treated with three cycles of Conservare OH.

16.13.4 On Site Implications

While no conclusions can be drawn at this point, it may be inferred that the grout samples have a higher splitting tensile strength than the untreated stone, but a lower splitting tensile strength than the treated stone.⁸³ This relationship of splitting tensile strength means that failure will occur in the grout before the treated stone, preserving the historic fabric.

⁸² Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013) 92.

⁸³ The compressive strength values shown for the untreated stone represent the maximum value of the cores tested.

Thermal Expansion - ASTM C 531-00 (2012) / ASTM D 4535-08



16.13.5 Observations and Results

Figure 52. Grout and stone samples in desiccator.

The formula used to calculate the linear coefficient of thermal expansion is as follows:

$$C = (Z - Y - W)/T(W - X)$$

- C = linear coefficient of thermal expansion (°F)
- Z = length of sample, including the studs, at elevated temperature (in.)

Y = length of stud expansion (in.) = $X \times T \times k$ (where k is the linear coefficient of thermal expansion per °F of the studs

- W = length of sample, including the studs, at lower temperature (in.)
- T = temperature change (°F)
- X = length of two studs at lower temperature (in.)

The linear coefficient of thermal expansion used for the 316 stainless steel gauge studs was 8.90E-06.⁸⁴ After the test was completed and the coefficient of expansion determined, the results of the q-test determined that none of the test results were outliers. The average linear coefficient of thermal expansion for the grout samples was 4.07E-06 in./in. °F, with a standard deviation of 2.46E-07 and a standard error of 1.00E-07. The average linear coefficient of thermal expansion for the stone samples was 5.60E-06 in./in. °F, with a standard deviation of 1.98E-07 and a standard error of 8.06E-08. The unpaired (or independent) t-test was used to compare the mean coefficient of expansion of the grout relative to the stone. Due to the fact that the t Stat value (11.8966) exceeded the critical value at the 95% confidence interval (2.2281) at 10 degrees of freedom, the null hypothesis was rejected, indicating that there was a difference in the mean between each material. Following this, the f-test was conducted to compare the degree of variation in the test results between the grout and stone samples. Due to the fact that the F value (0.6455)

⁸⁴ "316/316L Stainless Steel Data Sheet," AK Steel, accessed April 19th, 2014,

http://www.aksteel.com/pdf/markets_products/stainless/austenitic/316_316l_data_sheet.pdf.

exceeded the critical value at the 95% confidence interval (0.1980) at 5 degrees of freedom, the null hypothesis was rejected, indicating that there was a difference in the variation of the results. The general conclusion was that the stone samples exhibited a higher coefficient of thermal expansion than the grout, while the grout displayed greater variation in the degree of expansion of the samples tested.

| Thermal Expansion Test Calculations | | | | | |
|-------------------------------------|----------|-------------------------|-------------------|-----------|----------|
| | | Coefficient of thermal | Average | Standard | Standard |
| | Sample # | expansion of sample (C) | Coefficient of | Deviation | Error |
| | | (IN./IN.°F) | thermal expansion | | |
| | C1.1 | 3.67E-06 | 4.07E-06 | 2.46E-07 | 1.00E-07 |
| | C1.2 | 3.95E-06 | | | |
| out | C1.3 | 4.37E-06 | | | |
| Gr | C1.4 | 4.11E-06 | | | |
| | C1.5 | 4.26E-06 | | | |
| | C1.6 | 4.06E-06 | | | |
| | B7.1 | 5.63E-06 | 5.60E-06 | 1.98E-07 | 8.06E-08 |
| | B7.2 | 5.34E-06 | | | |
| one | B7.3 | 5.93E-06 | | | |
| Sto | B8.1 | 5.62E-06 | | | |
| | B8.2 | 5.47E-06 | | | |
| | B8.3 | 5.62E-06 | | | |

Table 9. Test results for thermal expansion test



Graph 9. Comparison of thermal expansion in the grout and stone samples.

| t-Test: Two-Sample Assuming Unequal Variances | | | | | | |
|---|-------------|-----------|--|--|--|--|
| | Stone | Grout | | | | |
| Mean | 5.60167E-06 | 4.07E-06 | | | | |
| Variance | 3.90167E-14 | 6.044E-14 | | | | |
| Observations | 6 | 6 | | | | |
| Hypothesized Mean Difference | 0 | | | | | |
| df | 10 | | | | | |
| t Stat | 11.89660211 | | | | | |
| P(T<=t) one-tail | 1.58449E-07 | | | | | |
| t Critical one-tail | 1.812461123 | | | | | |
| P(T<=t) two-tail | 3.16897E-07 | | | | | |
| t Critical two-tail | 2.228138852 | | | | | |

Table 10. Independent t-test for thermal expansion test results.

| F-Test Two-Sample for Variances | | | | | |
|---------------------------------|-------------|-----------|--|--|--|
| | Stone | Grout | | | |
| Mean | 5.60167E-06 | 4.07E-06 | | | |
| Variance | 3.90167E-14 | 6.044E-14 | | | |
| Observations | 6 | б | | | |
| df | 5 | 5 | | | |
| F | 0.64554379 | | | | |
| P(F<=f) one-tail | 0.321371761 | | | | |
| F Critical one-tail | 0.1980069 | | | | |

Table 11. F-test results for thermal expansion test results.

16.13.6 Discussion

The difference between the average linear coefficient of expansion between the grout and stone was 1.53E-06. This difference was considered minimal, indicating that Jahn M40 grout and Dakota sandstone are compatible. It was surprising that there was greater variation in the results of the grout samples, despite the fact that all of the samples were made with the same batch of grout from a proprietary material. It should be noted, however, that all of the stone samples were cut from the same piece of stone with a fine-grained texture. To better understand the thermal expansion properties of the stone, it may be necessary to repeat this test using stone cut from both coarse and fine-grained rock fragments. Furthermore, due to the anisotropic nature of the calcite present in Dakota sandstone, ASTM D 4535-08 recommends testing samples cut at different orientations to the bedding layers (x, y, and z directions) to assess the degree of anisotropy.

In a paper presented at the 9th International Congress on Deterioration and Conservation of Stone in 2000, a study was conducted on the weathering of marble due to thermal expansion. It was determined that stone texture (the lattice preferred orientation of calcite) determined the direction of thermal dilatation. Furthermore, grain size was identified as a major factor in the formation of thermally induced micro cracks, and marble samples with larger grain sizes exhibited thermal cracking at lower temperatures.⁸⁵ Due to the range of grain sizes present in the stone samples tested for this project and fact that calcite, a mineral which exhibits highly anisotropic thermal dilatation, is a major component of the cement in Dakota sandstone, a similar study may be required to fully understand the behavior of Dakota sandstone when subjected to thermal stress.

16.14 Additional Tests to be Completed

16.14.1 Hydric Expansion – RILEM 11.7

During the summer "monsoon" season in the Four Corners region of the American Southwest, brief but intense downpours are common. For this reason, it is critical to understand the hydric expansion properties of Dakota sandstone in relation to Jahn M40 grout and other formulations which will be tested in the future. Hydric expansion is essentially a volumetric increase, which may be exacerbated by the presence of clays or a large percentage of micropores.⁸⁶ Given the variability in the texture and mineral content of Dakota sandstone, and the fact that the methylene blue adsorption tests during previous research detected the presence of swelling clays, the water immersion test (RILEM 11.7) will provide critical information which will inform subsequent conservation efforts.⁸⁷ The 1"x1"x6 ¼" prismatic stone and grout samples used for the drying shrinkage and thermal expansion tests may also be used for the water immersion test.

⁸⁵ S. Siegesmund, "Control of Marble Weathering by Thermal Expansion and Rock Fabrics," in *Proceedings of the 9th International Congress on Deterioration and Conservation of Stone*, Venice 19-24 June, 2000, vol. 1, ed. Vasco Fassina, (Amsterdam: Elsevier Science B.V., 2000), 205-213.

⁸⁶ S. Siegesmund, and H. Dürrast, "Physical and Mechanical Properties of Rocks," in *Stone in Architecture: Properties, Durability*, 4th edition, ed. Siegfried Siegesmund and Rolf Snethlage, (Heidelberg, Dordrecht, London, New York: Springer, 2011), 165.

⁸⁷ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument," (M.S. Thesis, University of Pennsylvania, 2013) 98.

16.14.2 Frost Resistance – RILEM V.3

The seasonal and diurnal fluctuations in temperature on the site place additional strain on the rock due to freeze/thaw cycling. Previous frost resistance tests on the stone revealed that untreated samples had visibly deteriorated after 10 freeze/thaw cycles, while the treated samples exhibited no visible deterioration after 40 cycles.⁸⁸ Given this information, it is necessary to determine the effect of freeze/thaw cycling on grout samples, as well as grout/stone composite samples which simulate the conditions in the inverted cavity. 3″x3″x 1.5″ composite stone/grout samples were made for the frost resistance test and future tests will reveal the ability of the grout and stone to remain intact.

16.14.3 Capillary Absorption / Drying Index – NORMAL 11/85, 7/81and 29/88; ASTM C67-97 and C948-94; ARC Laboratory Handbook; ICCROM 1999

In addition to precipitation events, the Holly Tower support rock lies in the drainage path of the mesa top runoff and is adjacent to a spring, indicating that the water table is close to the surface. Additionally, windblown sediment which has accumulated around the base of the tower walls retains moisture at the top of the support rock, and the result of all of these conditions is both rising and falling damp. Given these factors which are accelerating the deterioration of the support rock, the rate of capillary absorption and drying of both the stone and grout must be understood to ensure compatibility. Previous capillary water absorption and drying index tests conducted on the stone revealed that the consolidation treatment reduced capillary absorption by 96% and drying rates by 7%.⁸⁹ The

 ⁸⁸ Laura Lacombe, "Condition Assessment and Treatment Recommendations at Holly Tower Support Rock, Hovenweep National Monument,"
 (M.S. Thesis, University of Pennsylvania, 2013) 108.
 ⁸⁹ Ibid, 129.

next step is to test Jahn M40 and additional grout formulations to determine which formulations exhibit capillary absorption and drying rates which are the closest to the treated stone.

16.14.4 Water Vapor Transmission – ASTM E96/E96M-12 and NORMAL 21/85

Previous tests were conducted to determine the effect of the consolidant on the water vapor permeability of the stone, which might have a negative impact on its drying behavior. Due to the fact that the support rock is subjected to seasonal and possibly continuous saturation as discussed in the previous section, any significant reduction in the water vapor permeability would inhibit the drying process, further accelerating the rate of deterioration. The results of the test indicated that the water vapor transmission rate of the treated stone decreased by 49%, which was consistent with the reduction in liquid water transport, and was therefore considered acceptable. The same test must be repeated for Jahn M40 and additional grout formulations to determine which formulations exhibit water vapor transmission rates which are the closest to the treated stone.

16.14.5 Wet Density – ASTM C185-08

The purpose of this test is to determine if the weight introduced by grouting the cavity will result in increased instability or failure of the partially detached slab. This test is particularly relevant for on-site conditions which involve filling large internal voids.⁹⁰

⁹⁰ Beril Bicer-Simsir and Leslie Rainer, Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces, (Los Angeles: Getty Conservation Institute, 2013) 21.

Chapter 17 Conclusions

17.1 General Summary

The test results have been summarized as follows:

1. Expansion and Bleeding

The average percentage of final bleeding was 0.2%, which was well below the maximum acceptable percentage of 5%, and no expansion was observed.

2. <u>Flow</u>

The average rate of efflux of the grout for the 4 tests conducted ranged from 13.3 to 14.7 seconds. While slower than several hydraulic lime grout formulations tested during previous research which ranged from 5.47 to 10.43 seconds, the results were well below the limit of 35 seconds specified in ASTM C939-10.

3. <u>Visual Shrinkage</u>

Aside from minor cracks at the rim due to the inward contraction of the grout, no cracks were observed within the main body of the samples.

4. Drying Shrinkage

The average percent shrinkage was minimal at 0.075% and the majority of the shrinkage occurred between day 7 and day 14. This result, while minimal, must be assessed along with the higher modulus of elasticity typical of cementitious grouts, which makes the material more brittle and prone to sudden failure.

5. <u>Time of Setting by Vicat Needle</u>

The average time of initial set was 4 hours and 8 minutes (248 mins.) and the average time of final set was 5 hours and 8 minutes (308 mins.) The grout will therefore remain workable for approximately 4 hours and will harden and retain its form approximately 5 hours after pouring.

6. <u>Shear Bond Strength</u>

The average shear bond strength between the grout and the stone was 34.25 psi. The range in shear bond strength between the coarse grained samples with a higher porosity was approximately 55-79 psi, while the range in shear bond strength between finer grained samples with a lower porosity was between 7-18 psi. The results for the coarse grained stone roughly corresponded to the mean shear bond strength of 72.2psi., which was recorded for Jahn M40 crack injection grout and Zuni sandstone at El Morro National Monument and was considered an acceptable strength. Based on this comparison, the fine grained stone samples did not possess an acceptable level of shear bond strength to support the partially detached slab.

7. Splitting Tensile Strength

The average splitting tensile strength of the grout was 199 psi. When the results were compared to tests conducted on Dakota sandstone samples taken from Kanopolis Lake in Kansas, which were treated with another ethyl silicate consolidant, it appeared that the Jahn M40 grout samples had a higher splitting tensile strength than untreated stone, but a lower splitting tensile strength than treated stone. This relationship of splitting tensile strength implies that failure will occur in the grout before the treated stone, preserving the historic fabric.

8. <u>Thermal Expansion</u>

The average linear coefficient of thermal expansion for the grout samples was 4.07E-06 in./in. °F, and the average linear coefficient of thermal expansion for the stone samples was 5.60E-06 in./in. °F. The difference of 1.53E-06 in./in. °F was minimal, indicating that the two materials are compatible.

17.2 Recommendations

1. It is currently inconclusive whether or not the consolidation treatment applied to the stone affected the ability of the stone to bond with the grout in the molds for the shear bond strength test. The shear bond strength test should be repeated using both treated and untreated stone samples with both coarse and fine grained textures. Composite samples for the frost resistance test and any other composite tests should always include both treated and untreated stone assemblies with both coarse and fine textures.

- 2. While the percent shrinkage of the grout was relatively minimal as indicated in the results for the drying shrinkage test, this shrinkage, combined with a higher modulus of elasticity characteristic of cementitious grouts, may be responsible for the detachment of the grout from the stone samples with restricted lateral movement in the shear bond strength molds during the curing process. Grout formulations with a lower modulus of elasticity (such as hydraulic lime grouts) should also be tested in the future and modifications may need to be made to the molds for the shear bond strength test. The base of the mold, which was built with plywood, may need to be replaced with a non-porous material such as acrylic to prevent excess moisture from being drawn out of the grout during the curing process.
- 3. It is recommended that cored samples of stone similar in composition and texture to that of the inverted cavity be prepared to determine the splitting tensile strength of the stone relative to Jahn M40 crack injection grout, and any other formulation that will be tested in the future. Both treated and untreated cores should be tested.
- 4. In addition to performing the vicat test based on the manufacturer's recommendations, it would also be useful to modify the temperature and relative humidity in order to simulate site conditions. The hot, arid climate may dramatically affect the setting time of the grout.

Chapter 18 Recommendations for Implementation

18.1 On-site Preparations for Grouting

18.1.1 Construction of a Shading Device

Due to the western orientation of the inverted cavity on the support rock, the suspended slab is subjected to diurnal extremes in temperature, particularly during the summer months. The construction of a shading device may be necessary to minimize thermal expansion and deformation of the rock during the curing process. Aluminized knitted shade fabric, a material commonly used for greenhouses and plant nurseries is able to deflect direct sunlight due to the crystalloid structure of the net filament, creating a cooler microclimate. The filament is coated with an anti-oxidation UV-resistant coating and the knitted structure allows the shading device to withstand high winds.⁹¹ A site survey will ultimately be required to determine the configuration and construction materials best suited for the shading device.

18.2 Preparations of the Support Rock Prior to Grouting

18.2.1 Flushing of the Cavity

Rock fragments, disaggregated material, and any other debris present in the cavities must be removed. Compressed air and a variety of hand tools may be used to achieve this. Water or alcohol may also be used to flush out loose material. Water will also be needed to dampen the cavity walls to prevent moisture from being sucked out of the

⁹¹ "Aluminet Thermal Screens," Ginegar Plastic Products Ltd., accessed April 4, 2014, http://www.ginegar.com/polysack-nets/aluminetmicroclimate-control.html.

grout and into the highly porous sandstone. Pre-wetting the cavity walls will also allow proper bonding between the stone and grout and improve the uniform flow of grout within the network of cavities. Caution should be taken not to introduce excess water into the cavities as residual moisture will dilute the grout in certain areas, leading to a non-uniform mixture. In addition, the process of flushing may mobilize soluble salts. Any holes or openings through which the grout will leak during injection must first be temporarily sealed with a non-staining easily removable product such as cyclododecane or plasticine until final set is reached. Following this, the temporary sealants must be removed and replaced by mortar suitable for filling cavities.⁹²

18.2.2 Temporary Facing at injection Holes and the Application of Clay Washes

In addition to consolidating the surface of the partially detached slab, additional precautions may be taken to prevent flaking and detachment of the case hardened surface in localized areas where the injection holes are drilled. In the case of the Mogao grottoes southeast of Dunhuang County in the Gansu Province of China, the mural paintings in cave 85 were stabilized with temporary facing prior to drilling injection holes.⁹³ At the Roman Necropolis of Hermopolis in Egypt, Tuna el-Gebel, decorative plaster was stabilized prior to treatment with Japanese tissue paper adhered with cellulose ether.⁹⁴ During the stabilization of the Early Phrygian Citadel Gate, the stone was covered with a clay wash (mud slurry) to protect the surface from grout which spilled during the injection process. The clay wash was then

⁹² Beril Bicer-Simsir and Leslie Rainer, Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces, (Los Angeles: Getty Conservation Institute, 2013) 2.

⁹³ Stephen Rickerby et al. "Implementation of Grouting and Salts-Reduction: Treatments of Cave 85 Wall Paintings," in Conservation of Ancient Sites on the Silk Road: The Second International Conference on the Conservation of Grotto Sites, Mogao Grottoes, Dunhuang, People's Republic of China, June 28-July 3, 2004, ed. Neville Agnew (Los Angeles: The Getty Conservation Institute, 2010) 482-483.

⁹⁴ "The Roman Necropolis of Hermupolis, Tuna el Gebel: Final Report on the Autumn 2012 Season," Landesmuseum Hannover, accessed, February 2, 2014, http://www.landesmuseum-hannover.niedersachsen.de.

removed with water after the injection process was complete.⁹⁵ Similar techniques may be used at the Holly Tower support rock to preserve the visual integrity of the western face.



Figure 53. Mural painting in cave 85 of the Mogao grottoes stabilized with temporary facing prior to grouting. Source: Stephen Rickerby et al. Los Angeles: The Getty Conservation Institute, 2010.

18.2.3 Spacing and Configuration of Injection Holes

Given the size of the cavity, injection holes may be drilled into the face of the stone at a downward angle to allow for even distribution of grout within the cavity. Air holes may also be drilled towards the top of the cavity to prevent the

⁹⁵ Kelly Wong, "Early Phrygian Citadel Gate 2006 Treatment Report," Architectural Conservation Laboratory, University of Pennsylvania, accessed May 1, 2014, http://www.conlab.org/acl/gordion/assets/pdfs/2006_FieldReport_Wong_small.pdf.

formation of air pockets.⁹⁶ The distance, depth, diameter and the configuration or pattern in which the holes are drilled across the face of the stone are critical to the success of the project.

A large hole diameter allows the grout to flow faster. This is especially important if there are large internal cavities which take longer to fill. The grout will first settle in the smaller crevices and begin to lose moisture. Therefore the more time that elapses before the cavity is filled reduces the flow of the grout which has thickened and no longer possesses its original rheological properties. Similarly, if there is a temporary loss of pressure during the injection process, grout will begin to adhere to the walls of the flow channels.⁹⁷

In terms of the configuration of the holes over the surface of the stone, which should be drilled just deep enough to penetrate the partially suspended slab, a staggered, diamond-shaped pattern generally allows for more even distribution of the grout than an orthogonal pattern. As the distance between the holes decreases, the distribution areas will overlap, providing even coverage. However, an excessive amount of holes may result in leakage.⁹⁸ In the case of the Holly Tower support rock, the friable nature of the suspended slab to be drilled through may drastically limit the number of holes which may be safely drilled without causing partial or full detachment of the slab. In this case, it may be feasible to drill fewer holes with larger diameters and increase the spacing in localized problematic areas.

⁹⁶ Beril Bicer-Simsir and Leslie Rainer, Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces, (Los Angeles: Getty Conservation Institute, 2013) 2.

 ⁹⁷ F. Van Rickstal, "Grout Injection of Masonry: Scientific Approach and Modeling," (PhD diss., Catholic University of Leuven, 2000): 67.
 ⁹⁸ Ibid, 68.

18.2.4 Temporary Stabilization of Slab

Stabilizing the partially detached slab prior to drilling the injection holes may prove to be the one of the most challenging aspects of this project. A structural engineer will ultimately determine the technique which presents the least risk of full detachment and failure of the slab. Of greatest concern is the impact of vibrations from drilling and the hydrostatic pressure introduced by the grout, which will exert outward-pushing forces on the slab. Due to the fact that pinning will not be used during this phase of the project, a temporary method of stabilization must be devised. In cave 85 in the Mogao grottoes, which was referenced earlier, a press was designed to support mural paintings on the ceiling after the grout was injected. This press not only stabilized the surface of the paintings against the increased gravitational pull due to the added weight of the grout, but the surface of the press was also layered with absorbent tissue which absorbed and contained the moisture from the grout, while drawing out soluble salts which were mobilized.

It seems that an external press such as the one described above may be adapted to resist lateral forces produced from the hydrostatic pressure of the grout. The design of a press with a degree of flexibility may prevent the development of concentrated stress points which would be introduced by supporting the slab from below with rigid struts.



Figure 54. Custom press in cave 85 of the Mogao grottoes. Source: Stephen Rickerby et. Al. Los Angeles: The Getty Conservation Institute, 2010.

18.3 Additional Measures to Maintain the Monolithic Integrity of the Support Rock

18.3.1 Sealing the Top of the Support Rock

To reduce water infiltration from the top of the support rock, it may be necessary to seal the surface with a mortar formulation which is compatible with Dakota sandstone and vapor permeable. Jahn M70 limestone/sandstone/brownstone repair mortar is a cementitious mineral-based mortar which does not contain latex, acrylic bonding agents or additives. It is vapor permeable and can be color-matched to minimize visual impact. Testing of M70 and other commercial or custom mortar formulations is recommended prior to implementation. Any intervention at the top of the support rock will require the removal of accumulated sediment at the base of the tower, as well as temporary stabilization measures. The interface of the lower courses of the tower with the top of the support rock must be examined to ensure that the mortar is applied in a way which does not trap water at the base of the tower walls.

18.3.2 Non-invasive Techniques for Void Detection

After the injection process is completed, it will be necessary to use non-invasive techniques both to ensure that the inverted cavity has been completely filled, and to regularly monitor the condition of the grout to detect cracking or other forms of deterioration. Ground penetrating radar (GPR) uses reflected and backscattered electromagnetic waves to locate and generate images of subsurface conditions based on variations in electrical properties. An electromagnetic pulse is applied to a surface with an antenna which moves at a constant speed. The pulse radiates in an elliptical conical pattern which is 90 degrees wide in the plane of the antenna and 60 degrees wide perpendicular

to the antenna. Variations in the dielectric constant (the degree to which a material concentrates electric flux) of the subsurface materials are recorded by the receiving antenna as reflected pulses.⁹⁹

The effectiveness of this technique depends on the dielectric constant and electrical conductivity of the material. The dielectric constant of rock for instance is determined by water saturation, mineralogy, porosity, frequency, inherent geometries and electrochemical interactions. The greater the variation in the dielectric constant between materials, the stronger the reflected pulse. The electrical conductivity of the material, which is determined by clay, mineral and water content, determines the depth of the pulse. The electromagnetic pulse is able to penetrate deeper into materials with lower conductivity.¹⁰⁰

In an article written by faculty members at the University of Napoli, infrared thermography, ultrasonics and electrictype geophysical methods were examined as methods for detecting defects in architectural structures. Infrared thermography, the first technique which was tested, converts thermal energy generated by materials in the infrared range into the visible portion of the electromagnetic spectrum. An image is created in which the colors or tones of grey correspond to specific energy levels. There are two types of infrared thermography used as non-invasive diagnostic tools. The first is referred to as pulse thermography (PT), which involves monitoring the surface temperature evolution of an object after it is stimulated with a heat pulse. The heat pulse travels below the surface via conduction and the infrared camera records the resultant temperature fluctuations on the surface. Subsurface discontinuities such as cracks inhibit the spread of thermal energy, resulting in temperature variations in localized

 ⁹⁹ William Monaghan and Michael Trevits, "Application of Ground Penetrating Radar to Evaluate the Extent of Polyurethane Grout Infiltration for Mine Roof Control: A Case Study," National Institute for Occupational Safety and Health, accessed April 4, 2014, http://www.cdc.gov/niosh/mining/UserFiles/works/pdfs/aogprte.pdf.
 ¹⁰⁰ Ibid.

areas. The second type of infrared thermography known as lock-in thermography (LT) involves the use of thermal waves rather than pulses that generate a series of images which reveal temperature variations. ¹⁰¹

Another non-invasive technique known as ultrasonics involves the application of high-frequency (ultrasound) waves to solid materials. By exciting a piezo-electric crystal inside of a transducer which is in contact with the material, a high-voltage pulse of ultrasonic energy is able to generate longitudinal, shear and surface waves. Variations or discontinuities in a material which impede the transmission of energy are then recorded by the receiver probe, providing information about the condition of the subsurface materials.¹⁰²

Electric-type geophysical methods such as the self-potential method (SP) and the direct geoelectrical method (DCG) measure the electric potential drop of the electric charge configuration of subsurface materials by introducing a continuous electrical current using electrodes attached to the surface of a material.¹⁰³

The laboratory results indicated that infrared thermography was the fastest and most efficient method for detecting defects in cementitious materials. The lock-in thermography technique was able to reveal variations in the composition of the same or similar materials (i.e. original material vs. patching material applied in later restoration campaigns). It was also able to provide information about the location, size and characteristics of subsurface defects or discontinuities, but the maximum penetration depth of 15mm would be unsuitable for inspecting the grout

¹⁰¹ Carosena Meola et al, "Application of Infrared Thermography and Geophysical Methods for Defect Detection in Architectural Structures," *Engineering Failure Analysis* 12 (2005): 877-878.

¹⁰² Ibid, 882.

¹⁰³ Ibid, 886.

injected into the inverted cavity at the Holly Tower support rock. The pulse thermography technique (PT) allowed for the detection of defects as deep as 55mm but did not clearly indicate the interface between layered materials.¹⁰⁴

The ultrasonic technique was able to reveal structural discontinuities in thick materials but was unable to provide detailed information about the size and location of smaller defects in thin layers of cementitious materials. Similarly, electric-type geophysical methods were unable to provide accurate information about smaller defects near the surface of a material but revealed the location of both cracks and discontinuities throughout the entire structure and provided information about the porosity level of the materials.¹⁰⁵

18.3.3 Surficial and Sub-Surface Water Mitigation

The support rock is located in or near the path of the spring and the drainage from the mesa top, which implies that the base of the rock may be continuously or seasonally saturated with water. Upon inspection of the rock, a great degree of slabbing and detachment has occurred on the lower portion, particularly on the south face, which indicates the presence of rising damp. A long term conservation plan for Holly Tower must address the flow of surficial and ground water which is undercutting the rock. The abundance of rock fall at the base of the support rock may make the construction of swales untenable as a means for diverting surficial water, while the diversion of ground water may require methods deemed too invasive for the site. Further research must be done to identify less aggressive options which will not compromise the visual integrity of the site.

¹⁰⁴ Carosena Meola et al, "Application of Infrared Thermography and Geophysical Methods for Defect Detection in Architectural Structures," *Engineering Failure Analysis* 12 (2005): 890-891. ¹⁰⁵ Ibid, 891.

Chapter 19 Future Recommendations

While injection grouting may be the most viable method for addressing the inverted cavity on the west face of the support rock, more drastic measures may need to be taken in the future to maintain the monolithic quality of the support rock. Several anchoring techniques used on rock-hewn heritage sites may warrant further study in their applicability to the Holly Tower support rock. It should be noted that a temporary stabilization plan for the tower would have to be developed concurrently.

19.1 Cintec Harke Anchor System

The continued deterioration of the support rock may ultimately require more aggressive intervention such as pinning. The vibrations typically caused by drilling may result in crack propagation and slabbing in the fragile sandstone, destabilizing the tower.¹⁰⁶ The Cintec Harke anchor system is an injection grout anchor assembly which is suitable for fragile structural materials and may warrant further testing for use at the Holly Tower support rock.

The assembly consists of a stainless steel tube surrounded by a polyester textile sock. After the anchor is inserted into the hole, a low-alkali, sulfate-resisting cementitious grout referred to as Presstec is injected through the steel tube and overflows into the textile 'sock'. When the sock is filled, the residual moisture from the grout and the bonding

¹⁰⁶ Martin E. Weaver, "Reviewing Structural Conservation Measures for Heritage Resources in Rock," The Safeguard of the Rock-Hewn Churches of the Goreme Valley: Proceedings of an International Seminar, Ürgüp, Cappadocia, Turkey, 5-10 September, (Rome: ICCROM, 1995) 154.

agent pass through the sleeve, forming both mechanical and chemical bonds with the stone and conforming to the contours of cavities and holes.¹⁰⁷

By increasing the diameter of the textile sock, additional grout may be injected into the hole to disperse concentrated loads on weak or fragile parent material. Diamond-tipped coring bits reduce the vibration typically associated with core drilling.¹⁰⁸ All of these factors suggest that the Cintec Harke anchor system may address many of the structural concerns regarding the Holly Tower support rock.

19.2 Grouting with Compacted Sand and Pre-stressed Bolts

This technique, which was mentioned earlier in the literature review, is recommended in situations where the introduction of mortar or grout into the drilled holes would exacerbate the detachment of the rock. At the Dafosi Grotto in Xian, the capital of the Shaanxi Province, dynamic cracks in the sandstone cliffs which extended into the head of the eastern bodhisattva, threatened large scale detachment. Stainless steel or fiberglass bolts were prestressed with screw nuts and inserted into holes filled with compacted sand, which anchored the bolts with the use of friction.

¹⁰⁷ Martin E. Weaver, "Reviewing Structural Conservation Measures for Heritage Resources in Rock," The Safeguard of the Rock-Hewn Churches of the Goreme Valley: Proceedings of an International Seminar, Ürgüp, Cappadocia, Turkey, 5-10 September, (Rome: ICCROM, 1995) 157.

¹⁰⁸ Ibid, 158.

Such a technique could be adapted to the conditions at the Holly Tower. By using compacted sand with the same composition of the support rock, this chemically neutral system would be compatible with the native stone, creating a more seamless transition between the original and repair materials.¹⁰⁹

¹⁰⁹ Gerd Gudehus and Thomas Neidhart, "Geotechnical Stability Problems of the Dafosi Grotto," in Conservation of Ancient Sites on the Silk Road: International Conference on the Conservation of Grotto Sites, Mogao Grottoes at Dunhuang, October, 1993, ed. Neville Agnew (Los Angeles: The Getty Conservation Institute, 1997) 253-255.

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Appendix A – Terminology

Admixture: '...prescribed materials of water, aggregate, and cementitious materials that is added to a masonry mortar as an ingredient to improve one or more chemical or physical properties of the conventional masonry mortar.'¹¹⁰

Carbonation: 'Over longer time periods than required for cement hydration, the lime begins to adsorb carbon dioxide from the atmosphere, finally converting the binder back to calcium carbonate. The process is essentially one that produces an artificial limestone returning the product back to its approximate original form.'¹¹¹

Case hardened: 'formation of a hard, resilient crust on the surfaces of boulders and outcrops of soft, porous rock through the filling of voids with natural cement'¹¹²

Lime mortars (grouts): '..the principal component of the raw material is calcium hydroxide. Manufacturing processes are quite different than for Portland cement. A limestone of variable purity is "calcined" or brought to a temperature just high enough to drive off the carbon dioxide present in the original limestone. This temperature is much lower than the clinkering point and is typically less than 1000°C. The primary product is calcium oxide or free lime.'¹¹³

Hydraulic lime binders: 'These begin as "dirtier" limestones containing clay, quartz, and other minerals in smaller proportion. At the calcining temperature, the aluminum and silicon present in these other mineral phases will begin

¹¹⁰ ASTM C1180-10: Standard Terminology of Mortar and Grout for Unit Masonry, ASTM International, 2010.

¹¹¹ John J. Walsh, "Petrography: Distinguishing Natural Cement from Other Binders in Historical Masonry Construction Using Forensic Microscopy Techniques," *Journal of ASTM International*, Vol. 4, No. 1 (2007): 6.

 ¹¹² A. S. Goudie, Case hardening, in *The Dictionary of Physical Geography*, D. S. G. Thomas and A. S. Goudie eds., (Oxford: Blackwell, 2000), 73.
 ¹¹³ John J. Walsh, "Petrography: Distinguishing Natural Cement from Other Binders in Historical Masonry Construction Using Forensic Microscopy Techniques," *Journal of ASTM International*, Vol. 4, No. 1 (2007): 6.

to combine with the free lime to form hydraulic calcium aluminates and calcium silicates. These minerals may be identical to those present in true hydraulic cements.¹¹⁴

Injection grout: According to a Publication from the Getty Conservation Institute entitled *Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces,* 'Injection grouting is a method commonly used by conservators for filling voids and cracks and for reattaching plasters, wall paintings, and mosaics to architectural substrates. Injection grouting differs from structural grouting in the scale of implementation.'¹¹⁵ For the purposes of this report, however, injection grouting will include structural grouting to restore the monolithic quality of a rock formation or wall assembly.

Living stone: Rock in its original location through which groundwater or moisture is migrating.¹¹⁶

Mineral grout (It was difficult to locate definitions for this term, but based on the readings, the following

explanation will be used for the purposes of this report): This grout type contains 'combinations of ordinary Portland

cement in low quantities, together with materials usually present in masonry, such as lime and natural pozzolan.^{/117}

Mortar: A material composed of one or more inorganic binders, aggregates, water and admixtures used in masonry to provide for bedding, jointing and bonding of masonry units.¹¹⁸

¹¹⁸ RILEM TC167-COM (2005): Characterization of Old Mortars with Respect to their Repair, In: Groot C, Ashall G, Hughes JJ (eds.) state-of-the-art report, DOI:10.1617/2912143675.007.

¹¹⁴ Ibid, 7.

¹¹⁵ Beril Bicer-Simsir and Leslie Rainer, *Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces*, (Los Angeles: Getty Conservation Institute, 2013) 2.

¹¹⁶ Martin E. Weaver, "Reviewing Structural Conservation Measures for Heritage Resources in Rock," The Safeguard of the Rock-Hewn Churches of the Goreme Valley: Proceedings of an International Seminar, Ürgüp, Cappadocia, Turkey, 5-10 September, (Rome: ICCROM, 1995) 151.

¹¹⁷ E. Toumbakari et al., "Methodology for the Design of Injection Grouts for Consolidation of Ancient Masonry," Proceedings of the International RILEM Workshop oh Historic Mortars: Characteristics and Tests, (Paisley: RILEM, 1999) 395.

According to RILEM TC 203-RHM, "The requirements that each type of mortar must meet in service depend on its environmental exposure and its role in the masonry element that it is found within (e.g. issues such as historic authenticity, aesthetics, resistance to moisture ingress, structural integrity, and service life). Technical requirements such as adhesion, strength, elasticity, water and vapor transmittance, drying behaviour, thermal dilatation, ability to deal with salt contamination and freeze—thaw cycling, and its aesthetic properties can be quantified."¹¹⁹ The various functions or mortar are described as follows:

'Bedding mortar for setting units, adhesion, bearing load.

Pointing mortar for water penetration protection and for aesthetic.

Exterior render water penetration protection, aesthetic covering.

Interior plaster aesthetic covering, a substrate for decoration.

Surface repair to replace and repair missing sections of masonry.

Grout material filling of cavities in masonry to improve monolithic behavior.

Flooring mortar supporting layer, levelling screed, a substrate for tiles and mosaics.

In-fill mortar filling mass between masonry faces (in certain types of masonry constructions) binding irregular masonry units that are part of the in-fill.¹²⁰

¹¹⁹ John J. Hughes, RILEM TC 203-RHM: Repair mortars for historic masonry, The role of mortar in masonry: an introduction to requirements for the design of repair mortars, RILEM Technical Committee, DOI 10.1617/s11527-012-9847-9.

¹²⁰ John J. Hughes, RILEM TC 203-RHM: Repair mortars for historic masonry, The role of mortar in masonry: an introduction to requirements for the design of repair mortars, RILEM Technical Committee, DOI 10.1617/s11527-012-9847-9.

Natural Cement: '... may be thought of as residing within the continuum between Portland cement and hydraulic lime. The composition of the raw materials is more similar to those of Portland cement containing higher proportions of silica and aluminum. These are the elements that when combined with calcium will form the hydraulic minerals. However, the burning temperatures for natural cements were closer to the calcining temperatures of limes and were never clinkered unless accidentally overburned.'¹²¹

Portland Cement: 'manufactured from ground limestone and shale and is formed through a process known as clinkering. This means the raw feed is brought to temperatures in excess of 1400°C, sufficient to cause a virtually complete reaction to hydraulic mineral species.'¹²²

Pozzolans: 'materials which, though not cementitious in themselves, contain

constituents which will combine with lime at ordinary temperatures in the presence of water to form stable insoluble compounds possessing cementing properties.¹²³

Quarry sap: groundwater present in newly quarried stone.¹²⁴

Rock Anchors: Steel reinforcement inserted into holes drilled perpendicular to faults which are filled with

cementitious or resin-based grouts.¹²⁵

¹²¹ John J. Walsh, "Petrography: Distinguishing Natural Cement from Other Binders in Historical Masonry Construction Using Forensic Microscopy Techniques," *Journal of ASTM International*, Vol. 4, No. 1 (2007): 8.

¹²² Ibid, 4.

¹²³ F.M. Lea, *The Chemistry of Cement and Concrete*, 3rd ed., (London: E. Arnold, 1970), In "Lime-based injection grouts for the conservation of Architectural Surfaces," Beril Bicer-Simsir et al, Reviews in Conservation, Vol. 10 (2009): 4.

¹²⁴ Christopher Gorse et al., *A Dictionary of Construction, Surveying and Civil Engineering*, (Oxford, Oxford University Press, 2013), accessed January 7, 2014, http://www.oxfordreference.com/view/10.1093/acref/9780199534463.001.0001/acref-9780199534463-e-5501.

Appendix B- Testing Matrix

| Test Matrix for Evalu. | ation of Grouting of Da | kota Sandstone at Holly To | wer Sup | port R | ck | | | multiply as required | |
|------------------------|-------------------------|----------------------------|---------|---------|---------|--------------------------|--|--|-------------------|
| Property Categories | Test | Stand ard | Grout | Stone P | ssembly | Period | Size/Quantity | Preparation / Equipment per sample | Samples Used |
| | | | | | | | | 250mL graduated glass cylinder reading to 10mL / | |
| | E xpansion and | ASTM C 940-10a | Yes | ΝA | NA | 1 day | 200mL of grout per sample | 50mL graduated glass cylinder reading to 1mL / | N/A |
| | Bleeding | | | | | | At least (2) samples per grout type | 10 mL syringe with cannula (# 20 or smaller) / | _ |
| | | | | | | | | thermometer / parafilm | |
| Working | | | | | | | | flow cone w/st.stl. discharge tube and non-corroding | |
| Properties | Flow | ASTM C 939-10 | Yes | N/A | N/A | 1 day | 1725mL per test | metal body / receiving container / ring stand / level / | N/A |
| | | | | | | | | stop watch / grout mixer | |
| | | | Í | T | t | | | / fiend values from the second state of the second se | CA1 CA3 |
| | Visual Shinkane | ASTM C 1148-92a (2008) / | Yes | N/A | NVA | 28 dave Intal | indiazed terra cotta saucers per drout typ | (5) undiazed terra cotta saireers / corried Milwankee drill w/ 0.850 RPM / | C4 3 C4 4 |
| | 0 | ASTM C 474-13 | | | | | 10 | balance / parailim / moist chamber / broad knife | C4.5 |
| | | | | | | | | st. stl. milkshake mixing cup / st. stl. milkshake mixing cup | |
| | | | | | | | | molds to create 1" x 1" x 6 ¼" prisms / gauge studs / tamper / | C1.1, C1.2, C1.3, |
| Properties during | Drying Shrinkage | ASTM C 1148-92a (2008) | Yes | Μ | N/A | 28 days total | Each sample to be 1"x1"x6 1/4" | leng th comparator / reference bar / moist chamber | C1.4, C1.5, C1.7 |
| Curing and Setting | | | | | | | | straight edged steel trowel | |
| | | | | ŀ | F | | Each sample to be 60mm top diam x | vicat apparatus / scale / 200-250mL glass graduates / straight edged steel trowel. | / |
| | Time of Setting by | ASTM C 953-10 / | Yes | NVA | N/A | 1 day | 70mm bottom diam x 40mm high | flat trowel with straight edged steel blade 100-150mm in length/ | N/A |
| | Vicat Needle | ASTM C 191-08 | | | | | At least (2) samples per grout type | (2) 40mm high x 70mm (inside bottom diameter) | |
| | | | | | | | | x 60mm (inside top diamater) conical ring molds / | |
| | | | | | | | | petroleum jelly / plumber's putty | |
| | | | | | | | | straight edged steel trowel | |
| | | | | | d | re-soak stone for 24 hrs | (2) 3" x 1 ½" x 1" thick pieces of | omposite molds w/ 1/2' spacers / funnel / electrical tape / pyrex dish / mineral oi | il A2.2 |
| | Shear Bond Strength | AS TM D 905-08(2013) | N/A | MA | Yes | prior to pouring grout | treated stone per assembly | oven / Instron testing intrument / curing chamber | A3.1 |
| Mechanical / | | reapproved | | | | min 28 days to cure + | extra sample to calibrate testing machine | | B2.3 |
| Hardened Properties | 6 | EN 196-1 (2005) | | | | 1 day testing | | | B3.1 |
| | | | | | | | | | B4.2 |
| | | | | | ľ | | | Instrom universal testing machine / min. 1/8" thk. X 1" wide balsa bearing strips | C2.1, C2.2, C2.3 |
| | Splitting Tensile | ASTM C496/C496M-11 | Yes | NA | NA | min 28 days to cure + | Each column to be 4" long x 2" diam | 4" long x 2" inside diameter molds / PVC pipe / plumber's putly / | C2.4, C2.5, C2.6 |
| | Strength | | | | | test on day 28 | (5) samples per grout type + | petroleum jelly / mineral oil / draffing tape / electrical tape / | C2.7, C2.8 |
| | | | | | | | extra to calibrate testing machine | 3/4" long x 2" inside diameter molds / stirring rods | |
| | | | | | | | | 1/4" thick acrylic sheet | |
| | | For grout/stone: | | | F | | | | B7.1, B7.2, B7.3, |
| Compatibili ty | Thermal | ASTM C 531-00 (2012) | Yes | Yes | NA | 2-3 days testing | Each sample to be 1"x1"x6 1/4" | gauge studs / epoxy / oven / length comparator / reference bar / scale | B8.1, B8.2, B8.3, |
| | Expansion | | | | | 0. | GROUT SAMPLES FROM DRY SHRINKAGE | | C1.1, C1.2, C1.3, |
| | | For stone: | | | | | TEST TO BE USED | | C1.4, C1.5, C1.6 |
| | | ASTM D4535-08 | | | | | | | |

¹²⁵ Martin E. Weaver, "Reviewing Structural Conservation Measures for Heritage Resources in Rock," The Safeguard of the Rock-Hewn Churches of the Goreme Valley: Proceedings of an International Seminar, Ürgüp, Cappadocia, Turkey, 5-10 September, (Rome: ICCROM, 1995) 154.

| List of Samples Prepare | ed for Tests | | | | | |
|--|---|---|--|---|--|-----------------------------------|
| | | | | | | |
| Shear Bond Strength Grout / Stone Samples | Frost Resistance Grout / Stone Sampled | Thermal Expansion / Water Immersion Stone Samples | Drying Shrinkage / Thermal Expansion / Water Immersion Grout Samples | Splitting Tensile Strength Grout Samples | Frost Resistance / Capillary Absorption / Drying Index Grout Samoles | Visual Shrinkage Grout Samples |
| A2.2 | A1.1/A1.2 | B7.1 | C1.1 | C2.1 | G.1 | C4.1 |
| A3.1 | B3.5/B3.6 | 87.2 | C1.2 | C2.2 | G.2 | C4.2 |
| B2.3 | B5.1/B5.2 | B7.3 | C1.3 | C2.3 | G.3 | C4.3 |
| B3.1 | | B8.1 | C1.4 | C2.4 | G3.4 | C4.4 |
| B4.2 | | B8.2 | C1.5 | C2.5 | G.5 | C4.5 |
| | | B8.3 | C1.6 | C2.6 | 0°E) | |
| | | | C1.7 | C2.7 | G3.7 | |
| | | | C1.8 | C2.8 | C3.8 | |
| | | | 6'1) | C2.9 | 6'E) | |
| | | | | C2.10 | C3.10 | |
| | | | | C2.11 | C3.11 | |
| | | | | C2.12 | | |
| | | | | C2.13 | | |
| | | | | C2.14 | | |
| | | | | C2.15 | | |
| One side treated | All sides treated | All sides treated | | | | |

Appendix C-List of Samples

| Jahn M40 | Unmodified | | | |
|-----------------------|--|--|---|-----------------------------|
| Grout Component | Weight of filter paper (grams) Mp | Weight of filter paper + grout (grams) Mt | Weight of grout (grams) Mt-Mp = Mg | Percentage of total mass |
| Microspheres | 4.39 | 4.64 | 0.25 | 0.79 |
| Fines (Clay + Binder) | 4.34 | 27.04 | 22.70 | 71.90 |
| Sand | 4.39 | 13.01 | 8.62 | 27.30 |
| | | | | |
| Total mass of grout | | | 31.57 | |
| | | | | |
| | | | | |

Appendix D – Gravimetric Analysis Data

| Appendix E – Particle Size Distribution |
|---|
|---|

| Jahn M40 | Sieve Analysis of M | icrospheres | | | | | |
|---------------------|---------------------|-------------|----------------------|------------------------------------|--------------------|--------------------|------------------|
| | | | | | | | |
| M _{cT} (g) | 6.73 | | | | | | |
| M _T (g) | 6.94 | | | | | | |
| M _{st} (g) | 0.21 | | | | | | |
| M _L % | | | | | | | |
| | | | | | | | |
| Sieve | Screen Size | Mcx | M _X | M _{sx} | %M _{sx} | %M _{rt} | %M _{pt} |
| Number | (μm) | (g) | (sample + container) | (M _X - M _X) | (M _{sx/} | Σ %M _{sx} | 100% - Mrt% |
| | | | (g) | (g) | /M _{st}) | (on or above) | |
| | | | | | *100% | | |
| 8 | 2360 | 6.98 | 6.98 | 0.00 | 0.00 | 0.00 | 100.00 |
| 16 | 1180 | 6.88 | 6.88 | 0.00 | 0.00 | 0.00 | 100.00 |
| 30 | 600 | 7.36 | 7.36 | 0.00 | 0.00 | 0.00 | 100.00 |
| 50 | 300 | 6.76 | 6.78 | 0.02 | 9.52 | 9.52 | 90.48 |
| 100 | 150 | 6.64 | 6.70 | 0.06 | 28.57 | 38.10 | 61.90 |
| 200 | 75 | 6.74 | 6.84 | 0.10 | 47.62 | 85.71 | 14.29 |
| pan | 1 | 7.36 | 7.39 | 0.03 | 14.29 | 100.00 | 0.00 |

| Jahn M40 | Sieve Analysis of Sa | nd | | | | | |
|---------------------|----------------------|-----------------|----------------------|------------------------------------|--------------------|--------------------|------------------|
| | | | | | | | |
| М _{ст} (g) | 6.88 | | | | | | |
| M _T (g) | 15.52 | | | | | | |
| M _{st} (g) | 8.64 | | | | | | |
| M _L % | | | | | | | |
| | | | | | | | |
| Sieve | Screen Size | M _{cx} | M _x | M _{sx} | %M _{sx} | %M _{rt} | %M _{pt} |
| Number | (μm) | (g) | (sample + container) | (M _x -M _{cx}) | (M _{sx /} | Σ %M _{sx} | 100% - Mrt% |
| | | | (g) | (g) | /M _{st}) | (on or above) | |
| | | | | | *100% | | |
| 8 | 2360 | 7.14 | 7.14 | 0 | 0.00 | 0.00 | 100.00 |
| 16 | 1180 | 7.12 | 7.12 | 0 | 0.00 | 0.00 | 100.00 |
| 30 | 600 | 6.26 | 6.30 | 0.04 | 0.46 | 0.46 | 99.54 |
| 50 | 300 | 6.51 | 9.40 | 2.89 | 33.45 | 33.91 | 66.09 |
| 100 | 150 | 6.42 | 10.98 | 4.56 | 52.78 | 86.69 | 13.31 |
| 200 | 75 | 6.88 | 7.75 | 0.87 | 10.07 | 96.76 | 3.24 |
| pan | 1 | 6.98 | 7.16 | 0.18 | 2.08 | 98.84 | 1.16 |

| Expansion and Bleeding Test Jahn M40 | | | | | | | | | | |
|---|---------|--------------|-------------|----------|-------|---------|-------------|--------------|---------------|-------|
| | | | | | | | | | | |
| Vo (mL): Volume of the sample at the beginning of the test | | Evnancion E | V | Mo v 100 | | Comb | inod Evnanc | ion (E (%)- | v ol/(ol/-40) | 19 |
| Vt (mL): Volume of the sample at set intervals | | глранзиин, г | 1. (U/) | | | | | | | 3 |
| Vg (mL): Volume of the grout portion of the sample at set intervals | | | | | | | | | | |
| Vw (mL): Volume of the decanted bleed water | | | | | | | | | | |
| | | a anglad | | 100 | | • | ined Direct | | 100 - 100 | |
| Ambient Temperature: 22.5°C | | pieeaing,p | (%)= (vr-vg | // X 0// | | | rinal bleed | ng, rb (%)=v | W/V0 X 100 | |
| Grout Temperature: 22.8°C | | | | | | | | | | |
| | | | | | | | | | | |
| | | | Test 1 | | | | | Test 2 | | |
| Hour:Min. | Vt (mL) | Vg (mL) | E (%) | CE (%) | B (%) | Vt (mL) | Vg (mL) | E (%) | CE (%) | B (%) |
| 91:0 | 200 | 199 | 9.0- | 0.0 | 0.6 | 201 | 200 | -0.6 | 0.0 | 0.6 |
| 05:0 | 200 | 199 | 9.0- | 0.0 | 0.6 | 201 | 200 | -0.6 | 0:0 | 0.6 |
| 0:45 | 200 | 199 | 9.0- | 0.0 | 0.6 | 201 | 200 | -0.6 | 0.0 | 0.6 |
| | | | | | | | | | | |
| | | | Test 1 | | | | | Test 2 | | |
| (Jm) OV | | | 200 | | | | | 201 | | |
| Vw (mL) | | | 0.5 | | | | | 0.5 | | |
| (%) EB | | | 0.2 | | | | | 0.2 | | |
| | | | | | | | | | | |

Appendix F – Bleeding and Expansion

Appendix G – Flow

| Flow Test Jahn Volume of grout a 2 Readings A/B we | M 40 Ind water used for all tests ere taken for each test teer commission 0 min + 1 | 1725 mL ± | :5mL | | | | | |
|--|--|-----------|-------------------|---------------------|---------------------------------|--------------------------------------|------------------------------------|--------------------------------------|
| Test Number | Ambient Temp. (°C) | RH (%) | Temp.of Water(°C) | Temp. of Grout (°C) | Rate of Efflux for Water (sec.) | Avg. Rate of Efflux for Water (sec.) | Rate of Efflux for Grout (seconds) | Ava. Rate of Efflux for Grout (sec.) |
| 1A | | | | | 3.8 | | 14.5 | |
| 18 | 0.12 | 77 | 19.0 | C.22 | 4.3 | 4.1 | 14.9 | 14./ |
| 2A | ר כר | 11 | 75.0 | 3 1 1 | 3.8 | 0.0 | 13.0 | 5 61 |
| 2B | 7.67 | 17 | 0.62 | C.#2 | 4.0 | <i>v</i> .c | 13.5 | C.C. |
| 3A | 0 6 6 | | 0 1 1 0 | 75.0 | 5.2 | 5 1 | 13.9 | 14.0 |
| 38 | 6.77 | 77 | 24.0 | 0.62 | 4.9 | 1.0 | 14.0 | 14.0 |
| 4A | 0 LL | | זנר | 016 | 4.0 | 11 | 14.1 | 0 61 |
| 4B | 0.77 | 77 | C.C2 | 24.0 | 4.1 | 4.1 | 13.4 | 0.61 |

| Drying Shrink | kage Test - Startin | g on Day 7 / | After Pourin | 6 | | | | | | | | | | | | |
|---------------|---------------------|--------------|--------------|-----|------------------|---------|---------|-----|------------------|---------|---------|-----|------------------|----------|--------|-----|
| | | Day 7 | | | | Day 1 | - | | | Day 21 | | | | Day 28 | | |
| Date | | 3/20/20 | 14 | | | 3/27/20 | 14 | | | 4/3/201 | 4 | | | 4/10/201 | 14 | |
| Sample # | le nath (0.00 | (ui 10(| Temp °C | RH% | lenath (0.00 | 01 in) | Temp °C | RH% | lenath (0.00 | (ul 10 | Temo °C | RH% | lenath (0.00 | 01 in) | Temp°C | RH% |
| | Initial Position | Min. | | | Initial Position | Min. | | | Initial Position | Min. | - | | Initial Position | Min. | - | |
| HT.C1.1 | 0.0190 | 0.0189 | 19.5 | 26 | 0.0162 | 0.0161 | 18.6 | 23 | 0.0156 | 0.0155 | 22.6 | 24 | 0.0151 | 0.0151 | 22.0 | 25 |
| HT.C1.2 | 0.0408 | 0.0408 | 19.5 | 26 | 0.0376 | 0.0376 | 18.7 | 23 | 0.0371 | 0.0368 | 22.7 | 24 | 0.0369 | 0.0367 | 22.0 | 25 |
| HT.C1.3 | 0.0578 | 0.0577 | 19.5 | 26 | 0.0551 | 0.0549 | 18.8 | 23 | 0.0539 | 0.0539 | 22.7 | 24 | 0.0538 | 0.0538 | 22.1 | 25 |
| HT.C1.4 | 0.0163 | 0.0163 | 19.5 | 26 | 0.0137 | 0.0137 | 18.8 | 23 | 0.0129 | 0.0129 | 22.8 | 24 | 0.0128 | 0.0128 | 22.1 | 25 |
| HT.C1.5 | 0.0159 | 0.0157 | 19.5 | 26 | 0.0127 | 0.0127 | 18.8 | 23 | 0.0119 | 0.0119 | 22.8 | 24 | 0.0119 | 0.0119 | 22.1 | 25 |
| HLC1.7 | 0.0282 | 0.0282 | 19.6 | 26 | 0.0258 | 0.0257 | 18.8 | 23 | 0.0249 | 0.0248 | 22.9 | 24 | 0.0248 | 0.0247 | 22.1 | 25 |

Appendix H – Drying Shrinkage

| Drying Sh | rinkage Test | | | | | | | | | | |
|------------------|---------------|---------------|-----------------|----------------|----------------------|---------------------|-------------------|-------------------|---------------|---------------|----------|
| Calculation | v | | | | | - | nominal dimension | | | | · |
| | Reference Bar | Reference Bar | Initial Reading | Initial Length | Reading after Curing | Length after Curing | Effective Gauge | Percent Shrinkage | Avg. Percent | Standard | Standard |
| Sample # | Length | Reading | (in.) | (in.) | (in.) | (in.) | Length (in.) | (%) | Shrinkage (%) | Deviation (%) | Error |
| | (in.) | (in.) | | Lı | | L | Lo | S | | | |
| ต.1 | 6.6200 | 0.0000 | 0.0189 | 6.6389 | 0.0151 | 6.6351 | 5 | 0.076 | | | |
| C1.2 | 6.6200 | 0.0000 | 0.0408 | 6.6608 | 0.0367 | 6.6567 | 5 | 0.082 | | | |
| C1.3 | 6.6200 | 0.0000 | 0.0577 | 6.6777 | 0.0538 | 6.6738 | 5 | 0.078 | 0.075 | 0.005 | 0000 |
| C1.4 | 6.6200 | 0.0000 | 0.0163 | 6.6363 | 0.0128 | 6.6328 | 5 | 0.070 | | 0000 | 700.0 |
| C1.5 | 6.6200 | 0.0000 | 0.0157 | 6.6357 | 0.0119 | 6.6319 | 5 | 0.076 | | | |
| CI.7 | 6.6200 | 0.0000 | 0.0282 | 6.6482 | 0.0247 | 6.6447 | 5 | 0.070 | | | |

| Vicat Test Jah | in M40 | | | | |
|----------------|----------------|-------------------------|-------------|----------------|-------------------------|
| | Sam | ple 1 | | San | nple 2 |
| Time (min.) | Time (hr:min.) | Penetration Depth (mm.) | Time (min.) | Time (hr:min.) | Penetration Depth (mm.) |
| 210 | 3:30 | 40 | 210 | 3:30 | 39.5 |
| 220 | 3:40 | 40 | 220 | 3:40 | 39.5 |
| 230 | 3:50 | 40 | 230 | 3:50 | 39.5 |
| 240 | 4:00 | 33 | 240 | 4:00 | 34.7 |
| 250 | 4:10 | 10.8 | 250 | 4:10 | 29.7 |
| 255 | 4:15 | 5.5 | 255 | 4:15 | 9.4 |
| 260 | 4:20 | 4.5 | 260 | 4:20 | 8 |
| 265 | 4:25 | 2.7 | 265 | 4:25 | 3.5 |
| 270 | 4:30 | 1.8 | 270 | 4:30 | 2.8 |
| 275 | 4:35 | 1.2 | 275 | 4:35 | 1.8 |
| 280 | 4:40 | 0.9 | 280 | 4:40 | 1.6 |
| 285 | 4:45 | 1.1 | 285 | 4:45 | 0.8 |
| 290 | 4:50 | 1 | 290 | 4:50 | 0.8 |
| 295 | 4:55 | 0.9 | 295 | 4:55 | 0.7 |
| 300 | 5:00 | 0.5 | 300 | 5:00 | 0.5 |
| 305 | 5:05 | 0.4 | 305 | 5:05 | 0.5 |
| 310 | 5:10 | 0.4 | 310 | 5:10 | 0.5 |
| 315 | 5:15 | 0.4 | 315 | 5:15 | 0.8 |
| 320 | 5:20 | 0.4 | 320 | 5:20 | 0.4 |

Appendix I – Time of Setting by Vicat Needle

| | Stone Characterization | C = coarser grain size/higher porosity | $\mathbf{F} = \mathbf{finer} \ \mathbf{grain} \ \mathbf{size} \mathbf{Aower} \ \mathbf{porosity}$ | C | C | F | F | F |
|---------------------|------------------------|--|---|---------|---------|---------|---------|---------|
| | Standard | Error | | | | 14.09 | | |
| | Standard | Deviation | (psi) | | | 31.50 | | |
| | Avg. Shear Bond | Strength (psi) | Avg. fsb | | | 34.25 | | |
| | Shear Bond | Strength (psi) | fsb | 22:23 | 79.22 | 7.90 | 10.74 | 18.17 |
| | Breaking Load | (Ibs.) | F | 63.8260 | 80.9261 | 12.8143 | 16.4730 | 18.5408 |
| | Percentage of | Original Grouted Area | % | 40.5 | 34.8 | 53.4 | 53.7 | 33.8 |
| | Bond Line Area | (in. sq.) | AB | 1.156 | 1.022 | 1.623 | 1.534 | 1.021 |
| | Testing | Area (in. sq.) | Ao | 2.850 | 2.935 | 3.039 | 2.859 | 3.021 |
| | d Area Prior to | Width (in.) | W | 2.972 | 2.995 | 2.979 | 2.966 | 2.968 |
| וח סת בוווזרוו כפוי | Groute | Length (in.) | L | 0.959 | 0.980 | 1.020 | 0.964 | 1.018 |
| | | Sample | | A2.2 | A3.1 | B2.3 | B3.1 | B4.2 |

Appendix J – Shear Bond Strength











| Splitting Te | nsile Strength Test | | | | | | | |
|--------------------|---------------------|----------|------------------|----------------|--------|----------|--------------|---------------|
| Sample Meas | urements | | | | | | | |
| | Sample # | | Length (0.1 in.) | | | Diameter | r (0.01 in.) | |
| | | Length 1 | Length 2 | Average Length | Dia. 1 | Dia. 2 | Dia. 3 | Avg. Diameter |
| gth | HT.C2.1 | 4.042 | 4.043 | 4.04 | 2.066 | 2.066 | 2.067 | 2.07 |
| sile Stren | HT.C2.2 | 4.029 | 4.043 | 4.04 | 2.062 | 2.060 | 2.059 | 2.06 |
| tting Ten | HT.C2.3 | 4.045 | 4.05 | 4.05 | 2.075 | 2.077 | 2.078 | 2.08 |
| nples used for Spl | HT.C2.4 | 4.053 | 4.054 | 4.05 | 2.060 | 2.062 | 2.073 | 2.07 |
| | HT.C2.5 | 4.05 | 4.045 | 4.05 | 2.064 | 2.061 | 2.067 | 2.06 |
| Sai | HT.C2.6 | 4.021 | 4.026 | 4.02 | 2.036 | 2.040 | 2.040 | 2.04 |

Appendix K – Splitting Tensile Strength













| teadir | ngs Taken During | j Test Poforonco | Doforonco | | | ibeau alame2 | C 46 46 24 | 0°F | | | ibeor o lame 2 | 0+0+0 | Jo |
|--------|------------------|-------------------------|------------------|-------------|---------|------------------|------------|------------------------------------|-------------|---------|------------------|------------|------------------------------------|
| | Sample # | kererence bar length | kererence bar | | | oampie reau | ng at at 2 | 2 | | | Jampie reau | ng at at y | |
| | | (in.) | reading | Value (+/-) | Reading | Temperature (°C) | RH % | (W) Sample length with studs (in.) | Value (+/-) | Reading | Temperature (°C) | RH % | (W) Sample length with studs (in.) |
| | HT.C1.1 | 6.6200 | 0.0000 | (+) | 0.0103 | 24.1 | 32 | 6.6303 | (+) | 0.0148 | 25.5 | 24 | 6.6348 |
| | HT.C1.2 | 6.6200 | 00000 | (+) | 0.0320 | 24.1 | 32 | 6.6520 | (+) | 0.0367 | 25.5 | 24 | 6.6567 |
| 1nc | HT.C1.3 | 6.6200 | 0.0000 | (+) | 0.0483 | 24.1 | 32 | 6.6683 | (+) | 0.0533 | 25.5 | 24 | 6.6733 |
| פוס | HT.C1.4 | 6.6200 | 0.0000 | (+) | 0.0077 | 24.1 | 32 | 6.6277 | (+) | 0.0125 | 25.6 | 24 | 6.6325 |
| | HT.C1.5 | 6.6200 | 0.0000 | (+) | 1/200.0 | 24.2 | 32 | 6.6271 | (+) | 0.0120 | 25.6 | 24 | 6.6320 |
| | HT.C1.6 | 6.6200 | 0.0000 | (+) | 0.0658 | 24.2 | 32 | 6.6858 | (+) | 0.0706 | 25.6 | 24 | 6.6906 |
| | HT.B7.1 | 6.6200 | 0.0000 | (-) | 0.0455 | 24.3 | 32 | 6.5745 | (-) | 0.0397 | 25.6 | 24 | 6.5803 |
| | HT.B7.2 | 6.6200 | 0.0000 | (-) | 0.0531 | 24.2 | 32 | 6:5669 | (-) | 0.0475 | 25.6 | 24 | 6.5725 |
| əu | HT.B7.3 | 6.6200 | 0.0000 | (-) | 0.0485 | 24.3 | 32 | 6.5715 | (-) | 0.0425 | 25.6 | 24 | 6.5775 |
| 015 | HT.B8.1 | 6.6200 | 0.0000 | (-) | 0.0393 | 24.3 | 32 | 6.5807 | (-) | 0.0335 | 25.7 | 24 | 6.5865 |
| | HT.B8.2 | 6.6200 | 0.0000 | (-) | 0.0349 | 24.3 | 32 | 6.5851 | (-) | 0.0292 | 25.7 | 24 | 6.5908 |
| | HT.B8.3 | 6.6200 | 0.0000 | (-) | 0.0389 | 24.3 | 32 | 6.5811 | (-) | 0.0331 | 25.7 | 24 | 6.5869 |

Appendix L – Thermal Expansion

| Therma | il Expansion Te ions | st | | | | | | | |
|---------------|--------------------------------|---------------------------|----------------------------|-------------------|-------------|-------------|------------------------|---------------|-------------------------------|
| | | Sample length | Sample length | Length of 2 studs | Temperature | Temperature | Coefficient of thermal | Stud | Linear coefficient of thermal |
| | Sample # | with studs at 23°C / 73°F | with studs at 99°C / 210°F | at 23°C/ 73°F | change | change | expansion of studs | expansion (Y) | expansion of sample |
| | | (in.) | (in.) | (in.) | (J.) | (°F) | (in./in.°F) | (in.) | (in./in.°F) |
| | | M | 7 | Х | Т | Т | k | Y | C |
| | HT.C1.1 | 6.6303 | 6.6348 | 1.6260 | 76 | 137 | 8.90E-06 | 1.98E-03 | 3.67E-06 |
| | HT.C1.2 | 6.6520 | 6.6567 | 1.6260 | 92 | 137 | 8.90E-06 | 1.98E-03 | 3.95 E-06 |
| ţno | HT.C1.3 | 6.6683 | 6.6733 | 1.6260 | 92 | 137 | 8.90E-06 | 1.98E-03 | 4.37E-06 |
| ero | HT.C1.4 | 6.6277 | 6.6325 | 1.6260 | 76 | 137 | 8.90E-06 | 1.98E-03 | 4.11E-06 |
| | HT.C1.5 | 6.6271 | 6.6320 | 1.6260 | 92 | 137 | 8.90E-06 | 1.98E-03 | 4.26E-06 |
| | HT.C1.6 | 6.6858 | 6.6906 | 1.6260 | 76 | 137 | 8.90E-06 | 1.98E-03 | 4.06E-06 |
| | HT.B7.1 | 6.5745 | 6.5803 | 1.6260 | 92 | 137 | 8.90E-06 | 1.98E-03 | 5.63E-06 |
| | HT.B7.2 | 6.5669 | 6.5725 | 1.6260 | 92 | 137 | 8.90E-06 | 1.98E-03 | 5.34E-06 |
| əue | HT.B7.3 | 6.5715 | 6.5775 | 1.6260 | 92 | 137 | 8.90E-06 | 1.98E-03 | 5.93E-06 |
| ot2 | HT.B8.1 | 6.5807 | 6.5865 | 1.6260 | 92 | 137 | 8.90E-06 | 1.98E-03 | 5.62E-06 |
| | HT.B8.2 | 6.5851 | 6.5908 | 1.6260 | 76 | 137 | 8.90E-06 | 1.98E-03 | 5.47 E-06 |
| | HT.B8.3 | 6.5811 | 6.5869 | 1.6260 | 76 | 137 | 8.90E-06 | 1.98E-03 | 5.62E-06 |

JAHN M40 Crack Injection Grout

Jahn M40 is formulated to repair cracks and voids ranging in width from approximately 3/16" to 9/16" (5.0 mm to 15.0 mm) or larger using low pressure mechanical or gravity feed equipment. M40 is completely mineral based, contains no latex or acrylic bonding agents or additives, and is vapor permeable for compatibility with masonry substrates.

Features and Benefits

- Single-Component: Easy to mix correctly, thereby improving quality control at the point of injection.
- Compatible Formulation: Compatibility of physical properties ensures that the grout and natural substrate react to the environment in the same way.
- Contains No Latex or Acrylic Bonding Agents: It protects the substrate by allowing salts, water vapor, and liquid water to reach the surface, preventing failure due to salt expansion or freeze/thaw cycles.
- Tenacious Adhesion: Strong bonding capabilities.
- Factory Controlled: No field chemistry resulting in product variation.
- Low Viscosity: Deep, thorough penetration.
- **Simple Application:** Can be manually or mechanically applied.
- Water Based: Environmentally and user safe. No solvent clean up or disposal problems.

Application Procedures

Wash the surface and interior of the crack using clean water to remove all dust, loose or deleterious material, which could prevent proper flow and/or adhesion thereby compromising the integrity of the cured injection grout.

Mixing

The mixing ratio is approximately 2 - 2 1/2 parts powder to 1 part water by volume. Mix by hand or mechanically, using a slow speed drill (400 - 600 RPM) equipped with a Jiffler-type mixing paddle. The material should be mixed for a minimum of three minutes, with continued agitation.



Injection Procedures

Immediately before injection, moisten interior of the crack by flushing with water. If the crack is allowed to dry out before the grout is injected, this step must be repeated. This is very important.

Transverse Cracks:

Drill a series of injection ports in the center of the crack. These ports should be drilled in a downward direction. Seal the crack with removable, non-staining clay, sealant, or caulk.

Inject grout into the lowest port and continue until it flows freely from this port and other ports at the same level. Seal ports using non-staining clay, sealant, or caulk and proceed in identical fashion until the crack is filled. **Clean up overflow immediately with clean water.**

Lateral Cracks (Delaminating Layers):

Drill a series of injection ports in a square configuration (90° angles) on the face of the substrate to create a "drill frame". Ports should be drilled in a downward direction. Wash the surface and interior of the crack using clean water to remove as much dust and loose material as possible. Any dust or debris remaining between the layers will impede the flow of the grout. If this is the case, more holes will be required to attempt to fill all hollow areas.

Inject grout into lower left port and proceed until it flows freely from this port and other ports at the same level. Seal ports using non-staining clay, sealant, or caulk. Inject grout into lower right port and proceed in identical fashion. The order of injection is lower left, lower right, upper left, and then upper right. Clean up overflow immediately with clean water.

Removal of Sealant

Let the grout dry (24 – 48 hours) and remove all sealant, caulk, or clay. After removing the sealant, repair the crack surface and injection holes with Jahn Mortar that matches the color and type of existing masonry.

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Clean Up

While injecting, continually check for grout runs and spills on the surface of the masonry, and clean the surface before the grout has time to set. This is normally done with a clean sponge and water, and may have to be done several times, rinsing the sponge repeatedly with clean water.

Remove uncured mortar from tools and equipment with water as soon as possible. Cured grout many only be removed chemically or mechanically.

Safety Requirements

It is recommended that safety goggles, gloves, and a dust mask equipped with P-2 filters (or equivalent) be worn for protection while mixing.

Limitations

- Do not apply Jahn Injection Grout to a frozen or exceedingly hot substrate. The applied grout must be protected from extreme heat, freezing, excessive wind, direct sunlight, and rain. Ambient temperature range should be 40° F to 90° F with low to average humidity.
- Do not add bonding agents to Jahn Injection Grout or use them as surface preparation materials.

Packaging

A two-gallon plastic pail contains approximately 18 lbs. of material. Coverage will vary depending on the type of substrate and the size of the crack.

A five-gallon plastic pail contains approximately 44 lbs. of material. Coverage will vary depending on the type of substrate and the size of the crack.

Storage And Shelf Life

Store material in a dry area away from direct sunlight. Ambient storage conditions should be in the range of 40° F to 90° F with low to average humidity. Average shelf life 10 years in original, unopened packaging.

Technical Data

Jahn M40 - Crack and Void Injection Grout

| LIQUID/PLASTIC PHASE | |
|-----------------------------|-------------------------|
| Volume mixed M40 in fluid | |
| oz. per lb. of dry material | 14.3 fl oz/lb (approx.) |
| | |
| HARDENED PHASE | |
| Compressive strength | 1500 to 4400 psi |
| Tensile bending strength | 290 to 730 psi |
| Tensile strength | 58 to 100 psi |
| Ratio in/3 water/lb of dry | |
| material | 5.3 to 6.0 fl. oz/lb. |
| Specific gravity | 1.3 (approx.) |

Warning

Not for internal consumption. Keep out of reach of children and animals. Consult Material Safety Data Sheet (MSDS) for specific information.

Notice: The information contained herein is based on our own research and the research of others, and it is provided solely as a service to help users. It is believed to be accurate to the best of our knowledge. However, no guarantee of its accuracy can be made, and it is not intended to serve as the basis for determining this product's suitability in any particular situation. For this reason, purchasers are responsible to make their own tests and assume all risks associated with using this product.

03/2014

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Jahn Restoration Mortar (Jahn M-Products) Material Safety Data Sheet

| Section I – Product and Company Identification | | | | | | |
|---|--|-------------------------------------|--|---------------------------|--|--|
| Trade Name | : Jahn Restorat | ion Morta | ar | Date Prepared: | | 3/1/2013 |
| Supplier: Address: | Cathedral Sto 7266 Park Cir Hanover, Mar | one Produ cle Drive vland 21(| ucts 9 076 U S A | Manufacturer: Address: | Cathedral Stone 7266 Park Circle Hanover, Maryla | Products e Drive and 21076, U. S. A. |
| Emergency I Telephone N Eax Number | Number: lumber: | Chen (410) | ntrec (800) 42 782-9150 782-9155 | 4-9300 Customer | Code: CDTS | |
| | Soctio | | omnosition | Information on | Ingradiants | |
| In one dia of M | Sectio | | omposition | | | |
| Silicon Dioxic | le (Quartz) (CA | S: 14808 | 8-60-7) | See table Z3 | 0.1 mg/m ³ Rdust; 9293 | VV7330000 |
| Non-Hazardo | ous Aggregate | | | N/A | N/A | 1000314NH |
| Tricalcium Si | licate (CAS: 12 | 168-85-3 |) | N/A | N/A | 1004122TS |
| Dicalcium Sil | icate | | | N/A | N/A | 1004278DS |
| Tricalcium Al | uminate (CAS: | 12042-78 | 3-3) | N/A | N/A | 1004124TA |
| Calcium Oxid | le (CAS: 1305-7 | 78-8) | | 5mg/m ³ | 2mg/m ³ | EW3100000 |
| Non-Hazardo | ous Ingredients: | Inorgani | c pigments | N/A | N/A | 10000314NH |
| | Sec | tion III | – Physical/ | Chemical Chara | cteristics | |
| Boiling Poin | t: N/A | | | Specific Gravity: | 1400/1700 kg/m3 | |
| Vapor Press | ure: N/A | | | Melting Point: N// | 4 | |
| Vapor Densi | ty (Air=1): N/A | | | Evaporation Rate | N/A | |
| Solubility in | water (20° C): | Negligib | le | Solubility in Otner | r Solvents: N/A | |
| Color: White to Pastel | | | | Udor: No Odor | D to | |
| Section IV – Fire and Explosion Hazard Data | | | | | | |
| Flash Point: N/A | | | | | | |
| Extinguishing Media: Media Suitable for Surrounding Fire (FP N). | | | | | | |
| Special Fire Fighting Procedure: Wear NIOSH / MSHA Approved SCBA & Full Protective Equip. (FF | | | | | Equip. (FP N). | |
| Unusual Fire | and Explosio | n Hazaro | ds: Not Releva | nt | | |
| 2 | | | Section V – | Reactivity Data | | |
| Stability: Y | es | | | Conditions to Avo | oid Stable (Stabi | lity): N/A |
| Incompatibil | ity (Materials t | to Avoid) |): N/A | | | |
| Hazardous | | | | Conditions to Avo | oid | Delevent |
| Polymerizati | on: NO | | | (nazardous Polyn | nerization): Not I | Relevant |

MSDS – Jahn Mortars (M-Products) Page 1 of 2

3/1/2013

Cathedral Stone Jahn Mortars Material Safety Data Sheet

| Section VI – Health Hazard Data |
|---|
| Primary Routes of Entry: Inhalation Ingestion |
| Health Hazard acute and Chronic: Eye and Skin Irritation, Removes Oil From Skin. |
| Other Potential Health Risks: None |
| Carciogenicity – NTP: Yes |
| |
| Explanation Carciogenicity: Not Relevant |
| Sians (Symptoms of Exposure: See Health Hazards |
| Medical Condition Aggravated by Exposure: None Specified by Manufacturer. |
| Contact with Eyes: |
| IMMEDIATELY FLUSH WITH POTABLE WATER FOR A MINIMUM OF 15 MINUTES, SEEK ASSISTANCE |
| FROM MD (FP N). INHALATION: REMOVE TO FRESH AIR. SUPPORT BREATHING (GIVE 0*2/ARTF |
| RESP) (FP N). INGESTION: CALL MD IMMEDIATELT (FP N). |
| Steps to be Taken in Case Material is Released or Spilled: Normal Clean Up. |
| |
| Neutralizing Agent: None Specified by Manufacturer. |
| Waste Disposal Method: If This Material as Provided by the Manufacturer, Becomes a Waste, It Doesn't |
| Meet the Criteria of a Hazardous Waste as Defined by the EPA Under Authority of the RCRA. Disposal |
| Must be in Accordance With Federal, State, or Local Regulations (FP N). |
| Precautions-Handling / Storage: Store Dry |
| Other Precautions: Avoid Contact Between Skin Surfaces and Wet Mortar, or Clothing Saturated With Wet |
| Mortar. Wash Clothing in Clean Water. |
| Normal Use : Mix With Water and Use Within 30 Minutes. Do Not Use Under 5°C (41°F). |
| Section VIII – Control Measures |
| Protective Gloves: Impervious Gloves Recommended. |
| Respiratory Protection: NIOSH / MSHA Approved Dust Respirator. |
| Ventilation: N/A |
| Eye Protection: Chemical Worker's Goggles. |
| Other Protective Equipment: None Specified by Manufacturer. |
| Work Hygienic Practices: None Specified by Manufacturer. |
| Suppl. Salety and Health Data: None Specified by Manufacturer. |
| Section IX – Label Data |
| Label Required: Yes |
| Label Status: G |
| Common Name: Cementitious Mortar |
| Special Hazard Precautions: Inhalation: Pulmonary Diseases. Dust Can Cause Inflammation of the Lining |
| Tissue of the Interior of the Nose and Inflammation of the Cornea. |
| Label Name: Cathedral Stone Products, Inc. |
| Label Street: 7266 Park Circle Drive |
| Label City: Hanover |
| Label State: Maryland |
| Label Zip Gode. 21070 |
| Section XX – Transportation |
| DOT Shipping: N/A DOT Hazard: N/A |
| Section XXI |
| Section AA |

Disclaimer:

Although reasonable care has been taken in preparation of this document, we extend no warrantees, and make no representations as to the accuracy or completeness of information Contained therein, and assume no responsibility regarding the suitability of this information for the user's intended purposes or for the consequences of its use. Each individual should make a determination as to the suitability of the information for his or her particular purpose.

MSDS – Jahn Mortars (M-Products) Page 2 of 2

3/1/2013



GUIDELINE FOR WRITING SPECIFICATIONS WHEN USING

Select Relevant Selections

Division 4 – Masonry

Part 1 – GENERAL

1.1 SUMMARY OF WORK

A. Furnish all labor, materials, tools, and equipment as necessary to stabilize or repair cracks and voids in masonry construction, using injection grouts, as shown on drawings and as specified herein.

1.2 SUBMITTALS

- A. Submit the following items in time to prevent a delay in work and to allow adequate time for review and resubmittals, if needed:
 - 1. Samples of all specified materials, product information and data, and Material Safety Data Sheets (MSDS).
 - 2. Certificates of Compliance as furnished by the Manufacturer, stating that all supplied materials are in conformance with the Manufacturer's published literature, and will meet or exceed the current specifications.
 - 3. Written verification that all specified materials will be used. Provide purchase orders, shipping tickets, receipts, etc. to prove that the specified materials were ordered and received.
- B. Materials shall not be ordered or work started before receiving written approval.

1.3 QUALITY ASSURANCE

A. Applicator Qualifications: Each applicator must have a record of successful historic masonry repair for at least five years.

1.4 DELIVERY, STORAGE, AND HANDLING

- A. Deliver and store material in original factory packaging, bearing identification of Manufacturer, product, and batch number. A Material Safety Data Sheet (MSDS) is to accompany all shipments.
- B. Deliver, store, and handle material so that it is protected from damage, extreme temperatures, and moisture in accordance with Manufacturer's product literature.
- C. Comply with Manufacturer's written specifications and recommendations for mixing, application, and curing of materials.

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D. Handle all products with appropriate precautions as outlined in the Manufacturer's product literature and Material Safety Data Sheets (MSDS).

1.5 PROJECT/SITE CONDITIONS

- A. Cold Weather Requirements: Do not perform specified work in air temperatures below 40° F, if substrate temperature is below 40° F, or if conditions are to be such within a 24-hour period.
- B. Hot Weather Requirements: Do not install material in temperatures exceeding 90° F. If, necessary, protect work area from direct sunlight, to prevent repair from drying out.

Part 2 – PRODUCTS

2.1 INJECTION ADHESIVE/GROUT

- A. Mineral-Based Jahn Injection Grouts distributed by Cathedral Stone[®] Products Inc., 7266 Park Circle Drive, Hanover, MD 21076; tel. (410) 782-9150; fax. (410) 782-9155; website: <u>www.cathedralstone.com</u> email: <u>info@cathedralstone.com</u>. are considered to conform to the requirements of this specification.
- B. Product Description:
 - 1. Jahn M30 Micro Injection Adhesive (for hairline cracks up to 3/16" or 5.0 mm) in width): Premixed cementitious injection grout that contains no corrosive constituents. The adhesive achieves extraordinary flow capacity, high penetration, and strong adhesion. Refer to product literature and technical data for material specifications.
 - 2. Jahn M40 Crack and Void Injection Grout (for cracks approximately 3/16" to 3/8" or 5.0 mm to 10.0 mm in width): Premixed cementitious injection grout that does not contain any acrylic, latex, or other synthetic polymer bonding agents or additives. The grout only needs to be mixed with clean water. The grout is vapor permeable, frost and salt resistant, shrink resistant, and is physically compatible with the substrate. Refer to product literature and technical data for material specifications.
- C. *Substitutions:* If proposed equal is submitted, laboratory testing shall be required to establish equivalent performance levels. An independent testing laboratory shall be utilized as determined by the Architect/Engineer and paid for by the submitting party.

Part 3 – EXECUTION

3.1 WORKMANSHIP

- A. All areas involved in the work shall be inspected by the Contractor to establish extent of work, access, and need for protection of surrounding construction, landscaping, etc. If conditions are not as expected, notify the Architect/Engineer immediately for direction. Do not proceed with work until unsatisfactory conditions are corrected.
- B. Grout workmanship should comply with all applicable recommendations of the Manufacturer's written specifications and requirements.
- C. Do not add any bonding agents, accelerators, or retarders to the grout.

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D. Discard all grout that has hardened or exceeded its allowable pot life after mixing. Provide separate, clearly labeled containers for discarded grout and remove material from the staging area as soon as practical.

3.2 PREPARATION

- A. *Transverse Cracks:* For cracks across the face of the masonry unit, drill a series of injection ports in the center of the crack. These ports should be drilled in a downward direction. Between the ports, the crack should be sealed with removable, non-staining clay or repaired with the appropriate Jahn Mortar.
- B. Lateral Cracks (Delaminating Layers) or Voids: Drill a series of injection ports in a square configuration (90° angles) on the face of the substrate to create a "drill frame". Ports should be drilled in a downward direction.
- C. Wash the surface and interior of the crack using clean water to remove all dust, loose or deleterious material, which could prevent proper flow and/or adhesion, compromising the integrity of the cured injection grout.

3.3 MIXING

- A. It is recommended that safety goggles, gloves, and a dust mask be worn for protection. Do not mix more material than can be used within approximately 30 minutes. Discard any mixed material that has been unused for 30 minutes or more.
- B. Jahn M30:
 - 1. The mixing ratio is approximately 2 to 5 parts powder to 1 part water by volume.
 - 2. Mix mechanically using a high-speed drill (3,000 RPM or higher) equipped with a Jiffler typemixing paddle. After mixing, the mortar should be poured into another clean container using a sieve. Continued agitation is necessary if the mortar is allowed to sit prior to use.
- C. Jahn M40:
 - 1. The mixing ratio is approximately $2 2 \frac{1}{2}$ parts powder to 1 part water by volume.
 - 2. Mix manually or mechanically, using a slow speed drill (400-600 RPM) equipped with a Jiffler type-mixing paddle. The material should be mixed for a minimum of three minutes, with continued agitation should the product be allowed to sit prior to use.

3.4 INJECTION PROCEDURE

- A. Wash the interior of the crack immediately before injection by flushing with clean water. If the crack is allowed to dry out before grout is injected, this step must be repeated.
- B. *Treatment of Transverse Cracks:* Inject grout into lowest port and continue until it flows freely from this port and other ports at the same level. Seal ports using non-staining clay, sealant, or caulk and proceed in identical fashion until the crack is filled. Clean up overflow immediately.
- C. Treatment of Lateral Cracks (Delaminating Layers) or Voids: Inject grout into lower left port and proceed until it flows freely from this port and other ports at the same level. Where necessary, insert threaded stainless steel dowels after some grout has been injected, agitate or tap several times to remove any voids or air pockets, and inject the remainder of the grout until port is full and grout flows

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freely from other ports at the same level. Seal ports using non-staining clay, sealant, or caulk. Inject grout into lower right port and proceed in identical fashion. The order of injection is lower left, lower right, upper left, then upper right. Clean up overflow immediately.

3.5 FINISHING

A. Remove plugs after 24 to 48 hours and repair the ports and the crack surface, if not previously performed, using an appropriate Jahn Mortar to match color and type of existing masonry.

3.6 CLEAN UP

- A. Remove uncured mortar from substrate before it dries using clean water and a rubber sponge. Cured mortar may only be removed chemically or mechanically.
- B. Remove uncured mortar from tools and equipment with water as soon as possible. Cured material may only be removed chemically or mechanically.

END OF SECTION

02/2012




Technical Information Sheet Article No. 0714

KSE 300 E

Elasticised stone strengthener on a silicic acid ethyl ester base. Gel deposit rate approx. 30%

Range of use

Remmers KSE 300 E is preferably used for friable, medium to coarsepored sandstone, certain volcanic rock (e.g. tuff) as well as weathered brick. It can also be used for strengthening historical renders and joints. Stone that has pronounced swelling and shrinking properties due to swelling capable clay minerals must be treated first with Funcosil Antihygro (Art. No. 0616) to reduce swelling. The stone should be examined in Remmers' laboratory.

Property profile

Remmers KSE 300 E, an elasticised stone strengthener, was developed in co-operation with Dr. E. Wendler (Munich) and a work group directed by Prof. Dr. J. Grobe (Münster) within the framework of a project called "Protecting Stone Surfaces through the Application of Elastic Silicic Acid Ester" which was sponsored by the German Federal Environment Foundation (Osnabrück).

Remmers KSE 300 E differs from conventional stone strengtheners by a

moderate E-modulus increase (stress-strain behaviour) while at the same time providing

Characteristic data of the product

Characteristic data of the product in the packaged state

Active ingredient content: Density at 20 °C: Colour: Odour: Catalyst system:

the reaction:

approx. 50% by mass 0.9 g/cm³ clear, slightly yellow typical neutral

Characteristic data of the product after application Deposited quantity of gel: approx. 300 g/l By-product caused by

ethanol (escapes)

 sufficient consolidation of the natural stone structure.

Remmers KSE 300 E reacts with the water or humidity stored in the pore system. During this reaction, amorphous and hydrous silicon dioxide linked through soft segments is deposited as a binder. The binder silica gel replaces the original binder lost through weathering.

The speed of the gel deposit reaction is highly dependent on temperature and humidity. Under normal conditions (20 °C, 50% relative humidity), the deposit of binder is concluded after approx. three weeks.

The most important property parameters of Remmers KSE 300 E stone strengthener are listed in the following:

- Gel deposit rate approx. 30 %
- Single component system reliable and easy to use
- Neutral catalyst
 High penetration depth, possible all the way down to the sound core of the stone material
- No by-products that damage the building
- High weather resistance and UV stability
- Partially strengthened natural stone can be worked over with Remmers Restoration Mortar.

Directions

Preliminary examination, setting up trial areas:

The following characteristic properties of the material should be determined (analysis of the state of the building):

- 1. Moisture content, content of damaging salts, hygroscopic water absorption
- 2. Absorbency, capillary water absorption
- Strength profile, depth of weathering, degree of hygroscopic swelling
- Application rate for each area, penetration depth of the stone strengthener, resulting strength profile
- 5. Establishment of working operations
- Set-up of a representative trial area which is necessary to see if there will be changes in colour and the correlation between laboratory results and the quantities and values achieved on the object.
- Execution of treatment and application rates should be controlled and documented.

Preparing the substrate:

Surfaces to be restored often show reduced absorption capacity due to a crust of soil or different types of "patina". The cleaning measures necessary to restore the original absorption behaviour should be as gentle as possible, e.g. by spraying with cold or warm water or by steam cleaning. In case of stubborn soil, the Rotec Low Pressure Blasting Device should be preferably used or Remmers cleaning products (see the respective Technical Information Sheets). In many cases the stone is already so friable that cleaning cannot take place without a sensitive loss of substance. To avoid this, prestrengthening with Remmers KSE 300 E or another suitable stone strengthener from the Remmers KSE family can be carried out prior to cleaning. The main strengthening measure is then carried out after the cleaned surface has dried.

In order to achieve complete saturation of the weathered zone of the stone with Remmers KSE 300 E, the surface to be treated must have reached compensation moisture balance, be absorbent and should not have been heated by the sun. When strengthening is carried out, the temperature of the stone strengthener as well as the temperature of the substrate and surrounding air should range between 8 °C and 25 °C. To avoid strong heating, use shading devices. The surfaces should be protected from sun, rain and wind before, during and after strengthening.

Application procedures:

An essential prerequisite for optimal strengthening is that the weathered zone is completely saturated all the way down to the sound core. To achieve this, Remmers KSE 300 E stone strengthener is applied to the building material in a flow coating, dipping and/or compress procedure. When using a flow coating procedure, smaller areas (if necessary, stone by stone) are treated, wet-on-wet, with KSE 300 E until the material is no longer absorbed. The procedure selected for application always depends on the task at hand. So-called "fast hydrolysis" is not recommended since this represents an uncontrolled influence on the gel formation reaction and therefore the success of the strengthening measure.

Notes

If necessary, treatment can be repeated 2-3 weeks after initial treatment. In this case as well, saturation of the complete weathered zone must be achieved. The application rate of Remmers KSE 300 E should be determined in the laboratory during preliminary examinations and on a trial surface and will depend not only on the absorbency of the substrate but also on the application procedure selected.

Follow up treatment:

To avoid a change in the colour of the surface caused by oversaturation with Remmers KSE 300 E, the stone surface should be washed off with a dry solvent (e.g. Thinner V 101) immediately after saturation has been achieved.

Application of stone substitution compounds, hydrophobizing impregnation agents and coats of paint:

Surfaces that have been strengthened with Remmers KSE 300 E can be worked over - after the deposit of gel has been concluded - with Remmers Restoration Mortar, Funcosil impregnation agents and/or products in the Remmers Silicone Resin Paint System. After application, the "silicic acid ester" chemical system leads to a temporary water repelling effect which disappears during the course of gel formation. If strengthened surfaces still show an annoying water repelling effect when restoration mortar is subsequently applied, this can be suppressed by wetting the surface with alcohol.

Adjoining surfaces:

Facade elements that should not come in contact with the impregnation agent such as, e.g. windows, varnished surfaces, glass and also plants should be protected by suitable measures (e.g. covered with plastic sheets).

Tools, cleaning

Depending on the task at hand, e.g. low pressure spraying equipment, airless equipment, hand sprayers. Tools and equipment must be clean and dry. After use and before longer pauses, they should be cleaned thoroughly with Thinner V 101. Once the stone strengthener has reacted, it can only be removed mechanically.

Packaging, application rate, shelf-life

Packaging:

5 I, 30 I and 200 I tin containers

Application rate:

The application rate of Remmers KSE 300 E depends to a considerable degree on the type and state of the substrate to be treated as well as the task at hand and the application technique used. The quantity required may vary accordingly and can range between 0.1 l/m² to several litres per m².

The rate should be determined in the laboratory during preliminary examination as well as on a trial surface.

Shelf-life:

At least 12 months stored cool but frost-free and dry in closed, original containers. Remmers KSE 300 E reacts with humidity/moisture, so close containers air-tight each time material is removed.

Safety, ecology, disposal

Further information on safety when transporting, storing and handling as well as disposal and ecology is found in the latest Safety Data Sheet. Personal protective equipment is required for spraying procedures. Use respiratory protection with a combination filter at least A/P2 (made by e.g. Dräger). For suitable protective gloves, see Safety Data Sheet. Wear closed work clothes.



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The statements above are compiled from our field of production and according to the latest technological developments and application techniques.

Since application and working are beyond our control, no liability of the producer can be derived from the contents of this information sheet. Any statements made beyond the contents of this information must be confirmed in writing by the producer.

In all cases, our general conditions of sale are valid. With the publication of this Technical Information Sheet all previous editions are no longer valid.



Technical Information Sheet

Funcosil[®] Antihygro

Art.No. 0616

Preservative that stops swelling in natural stone which has clayey binders to reduce hygroscopic swelling.

Product base:

Alkyl ammonium compound in aqueous solution.

Property profile:

Funcosil Antihygro clearly differs in the way it works from other stone preservatives. This is an agent that reduces the hygroscopic swelling value of natural stone by approx. 50% without essentially influencing water absorption and physicalmechanical behaviour of the stone. The effect of Funcosil Antihygro is based on blockage of swelling capable centres in the sheet silicates (clay minerals). According to the knowledge we have today, swelling and shrinking processes as the result of moisture penetration and drying are seen as the primary cause of damage in clayey sandstone, but also to some extent in brick and tuff stone material. These swelling processes can occur at average to high humidity. Protective treatment with Funcosil Antihygro, especially for highly swelling capable, clayey sandstone that has little resistance to weathering because of hygroscopic swelling, is an imperative.

| Characteristic data of the | product in the | packaged | state |
|----------------------------|----------------|----------|-------|
| | | | |

| Effective ingredient content: | 0.2 mol/l |
|-------------------------------|--------------------|
| Density at 20°C: | approx. 1.0 kg/l |
| pH value: | 6 ± 1 |
| Solvent: | water |
| Colour: | clear |
| Odour: | barely perceptible |
| | |

Range of use:

To be used for all porous, absorbent, cementitious building materials with a moderate to high swelling value.

Substrate conditions/Substrate pre-treatment:

A prerequisite for an optimal effect with Funcosil Antihygro is optimal penetration of the effective ingredients. To ensure this, the following points should be observed:

The surface to be treated may not have been hydrophobically treated (water repelling); for this reason, a

waiting period of 6 weeks must be observed if strengthening with silicic acid esters has been carried out.

- Alkalinity in the area of freshly filled joints may have a negative effect on the effectiveness of Funcosil Antihygro, a waiting period of at least 2 weeks must be observed after mortar consolidation measures have been carried out.
- Before applying the preservative, dirt and pollutant crusts, efflorescence and infestation with alga and moss must be removed from the surface by a suitable cleaning procedure. Cleaning opens capillaries and pores, allowing the impregnation agent to be absorbed.
- Cleaner residue (e.g. surface-active agents) must be thoroughly removed because it reduces penetration depth and therefore the effectiveness of Funcosil Antihygro.

Working instructions:

Funcosil Antihygro is to be applied in a pressureless flow coating procedure. A 30-50 cm long film of liquid on the building material surface indicates that a sufficient amount of material is being applied. If there is any difficulty wetting the building material with Funcosil Antihygro, the surface can be priorly sprayed lightly with water. During application the nozzle should be led along the facade horizontally without interruption. Dependent on the absorbency of the substrate, the process is repeated several times. As a rule, two applications are sufficient. Pressure and nozzle diameter are to be adjusted so that misting does not occur. To avoid missing areas, limited sections should be completely impregnated without interruption. For smaller, more complicated surfaces that do not allow a spray procedure, work can be carried out with a brush or, for ornamental elements, with cellulose compresses and/or a full saturation procedure. Freshly treated surfaces should be protected against driving rain and strong sunlight for at least 5 hours. For projects classified as historical monuments, we recommend preliminary examinations and trial surfaces. We would be pleased to give you advice.

Working temperature:

Treatment to reduce swelling can be executed at temperatures between 10°C and 25°C. The reaction may be delayed at working temperatures below 10°C.

Technical Information Sheet

Follow-up treatment:

Natural stone preservation can be executed in the Funcosil System with a measure package co-ordinated to the damage and weathering mechanism. To support the effect of Funcosil Antihygro, it makes sense in many cases to use a stone strengthener (Funcosil Stone Strengthener 300 and in some cases Funcosil Stone Strengthener 100 or Funcosil Stone Strengthener 510) and/or a hydrophobic impregnation (Funcosil SL, SNL, WS). Coloured coating of the surface can be carried out with the Funcosil Silicone Emulsion Paint System. Working details and specifications of the products named are found in the respective Technical Information Sheets.

Tools:

All non-corrosive, low pressure conveyor or spraying equipment, liquid pumps and especially the Funcosil MV2 Sprayer are suitable for working.

Packaging, application rate and storing:

Packaging: 5 I and 30 I plastic canisters

Application rate:

Shelf-life: At least 6 months in closed, original containers, stored cool but frost-free. Funcosil Antihygro reacts with oxygen, so close containers air-tight each time material is removed. Protect containers from strong sunlight.

Safety, ecology, disposal:

Further information concerning safety during transport, storage and handling as well as for disposal is found in the latest Safety Data Sheet.

The statements above are compiled from our field of production and according to the latest technological developments and application techniques. Since application and working are beyond our control, no liability of the producer can be derived from the contents of this information sheet.

Any statements made beyond the contents of this information must be confirmed in writing by the producer.

In all cases, our general conditions of sale are valid.

With the publication of this Technical Information Sheet all previous editions are no longer valid.

Index

| Α | | |
|---|--|--|
| | F | |
| additives, 33, 143 | fillers, 32 | |
| anisotropic, 10, 87, 130, 131 | flow, 15, 42, 72, 95, 96, 134, 166 | |
| В | frost resistance, xiii, 53, 56, 63, 132, 136 | |
| binder, 22, 31, 46, 47, 156 | Funcosil Antihygro, 38, 39, 53, 55, 56, 59, 82 | |
| C | G | |
| calcite, 10 | gauge studs, 56, 58, 62, 77, 97, 127 | |
| capillary absorption, 38, 132 | gravimetric analysis, 45, 46, 50 gravity grouting, 34, 97 | |
| case hardening, 1 | | |
| cementitious injection grout, 1 | Н | |
| consolidation, 1, 30, 31, 132, 136 | Holly Tower, ii, 1, 3, 10, 11, 12, 15, 16, 22, 23, 30, 33, | |
| D | 35, 36, 37, 38, 42, 53, 70, 84, 87, 104, 125, 131, | |
| drying shrinkage, 43, 76 | 132, 140, 141, 145, 146, 147, 148, 149, 152 | |
| | Hovenweep, 2, 4, 5, 132, 151 | |
| Ε | hydrated lime-based grouts, 31 | |
| expansion and bleeding, 42, 70, 94, 134 | hydraulic lime mortars, 28 | |

205

hydraulic lime-based grouts, 31

hydric expansion, 131

hydrostatic pressure, 107, 141, 142 I injection grouts, 29, 30 J Jahn M40 crack injection grout, 2, 32, 34, 35, 109, 135, 137 L length comparator, 56, 76, 77, 78, 87, 88, 91 lime-cement, 28 living stone, 23, 157 М microspheres, 32, 35, 45, 46, 47, 48, 49, 50, 51 modulus of elasticity, 32, 38, 103, 135, 137 Ν non-invasive techniques, 143 Р Portland cement, 22, 27, 29, 31, 156, 157, 159

potassium carbonate, 65, 66 pozzolans, 29, 32 Q quarry sap, 23 R Remmers KSE 300 E, 38, 39, 53, 59, 60, 125 rock bolting, 26 rock-hewn heritage sites, 23, 147 S sandstone, 1, 3, 5, 6, 9, 14, 15, 16, 26, 33, 36, 38, 42, 81, 87, 103, 104, 109, 124, 125, 130, 131, 135, 136, 139, 143, 147, 148 shear bond strength, 43, 63, 81, 107, 110, 135, 169 slabbing, 1, 30, 37, 146, 147 splitting tensile strength, 43, 61, 84, 115, 117, 124, 136, 150, 172

206

Т

۷

thermal expansion, 43, 62, 87, 126, 131, 136, 153,

176

thixotropic, 97

time of setting by vicat needle, 43, 78, 106, 135, 168

visual shrinkage, 43

W

water content, 33, 34, 144

water vapor transmission, 38, 133

wet density, x, 133