DESIGN AND EVALUATION OF HYDRAULIC LIME GROUTS FOR *IN SITU* REATTACHMENT OF LIME PLASTER TO EARTHEN WALLS

Angelyn Bass

A THESIS

in

Historic Preservation

Presented to the Faculties of the University of Pennsylvania in
Partial Fulfillment of the Requirements for the Degree of

MASTER OF SCIENCE

1998

Supervisor
Frank G. Matero
Associate Professor of Architecture

Reader
Jeanne Marie Teutonico
Lecturer, Historic Preservation

Graduate Group Chair
Frank G. Matero
Associate Professor of Architecture

ACKNOWLEDGMENTS

Firstly, and above all, I would like to acknowledge Frank Matero, my graduate thesis advisor and Associate Professor of Architecture at the University of Pennsylvania. Not only did his professional expertise and enduring support guide me throughout this research, but his extraordinary energy and optimism, and commitment to advancing the field of conservation, has inspired and guided me well beyond the classroom.

I would also like to acknowledge Jeanne Marie Teutonico, whose initial research in the field of hydraulic lime grouts in the 1980s served as the model for this study, and whose critical review and perspective on this work was valued and greatly appreciated.

From the National Park Service, Jake Barrow of the Intermountain System Support Office in Santa Fe was instrumental in initiating the project and supporting practical application of this research in the field. My appreciation is also extended to Harry Myers, superintendent of Fort Union National Monument, for providing park resources and staff for the initial field work.

Finally, I must thank Robert Rivera, whose graphic expertise I sought throughout the preparation of this work, and whose artistry and companionship I will seek in many a future project.

TABLE OF CONTENTS

LIST OF TABLES

List of Figures

Abstract

In situ preservation of architectural plasters, stuccos, and renders in archaeological and ruined sites presents complex problems from the stand point of conservation. Often, architectural plasters and stuccos, once intended to serve as a continuous protective covering for the buildings structural system, are fragmentary, extremely fragile from years of weathering, and highly susceptible to deterioration from exposure. Since a majority of ruined sites are not roofed, designing treatments to preserve the plaster *in situ* must take into account high durability in exposed environments without excessive strength that could damage fragile original fabric. Conserving lime plasters in adobe ruins presents a particularly difficult problem because of the physico-chemical and mechanical differences in the historic adobe and lime plaster, and the necessity of using repair materials that are compatible with both.

In this study, a laboratory and field testing program was undertaken to design and evaluate lime, hydraulic lime, and clay-based grouts for the reattachment of lime plasters to earthen supports. In this first stage of research, the principle objective was to examine how various grout formulas performed in laboratory conditions as an adhesive and a light-weight void filler.

The laboratory experimental program consisted of first characterizing the historic adobe and lime plaster from Fort Union National Monument, the field site for this research. Following this, nineteen grout formulations were prepared and evaluated in a three phase testing program. Standard tests were employed to measure the critical properties of grout injectability, viscosity, unit weight, set time, shrinkage, splitting tensile strength, water vapor permeability, and adhesive bond strength. Of the initial 19 grout formulas tested, one mixture composed of (parts by weight) 1 part microspheres, 1 part sand, 2 parts hydraulic lime, and 1/10 parts acrylic emulsion in water was found to adequately meet the essential performance criteria.

Preface

For nearly a decade, the Architectural Conservation Laboratory at the University of Pennsylvania and the National Park Service have been involved in a collaborative research program to study materials and methods to preserve historic and prehistoric ruined sites and structures in the American Southwest. Among the many initiatives undertaken was a multi-phase research project to examine the materials, performance, and conservation of traditional surface finishes such as lime and mud plasters and stuccos. This thesis work was a first phase of research into using hydraulic lime grouts for *in situ* reattachment of surface finishes, and the springboard from which subsequent laboratory research and field testing could be launched.

The specific issue of reattaching lime plaster to adobe, and the interest in developing a grout for this purpose, began in 1991 when researchers from the University of Pennsylvania and staff of the National Park Service conducted a preliminary condition assessment of the extant historic plaster at Fort Union National Monument, a mid-nineteenth century adobe fort in New Mexico that retains a large portion of its original interior lime plasters *in situ*. Following the condition assessment that found the plasters to be fragmentary and actively deteriorating, a modest pilot plaster treatment program was undertaken using lime and hydraulic lime based grouts. Based on the promising results of the pilot treatments, and from encouraging research on hydraulic lime grouts conducted by ICCROM from 1979-83 (Ferragni et al. 1983, 1984), as well as from a practical realization that research on this topic could be applied to similar plaster detachment situations in many ruined sites in the Southwestern United States and Latin America, this thesis project was initiated.

It must be noted that the results of this thesis work are only preliminary. This first stage of research was intended to identify materials that were compatible and appropriate to use in a grout for reattaching lime plaster and adobe, and to observe how different combinations of materials performed under laboratory conditions. In the end, these objectives were met.

Since this first phase was exploratory in nature, there were certain shortcomings in the experimental program that should be mentioned. Firstly, though some of the standard testing methods employed proved useful and provided reproducible data, others were inadequate and produced questionable results. In Phase III for example, the bond strength in shear performance test conducted on grouted assemblies was essentially inconclusive regarding bond strength, but did provide some very interesting and valid results on the importance of prewetting porous materials prior to grouting. Also in Phase II, the splitting tensile strength test results are not highly reproducible due to the extreme sensitivity and high bias of the test to variations in the samples. One other drawback in the testing program was that often there were only enough samples to perform a test once, leaving insufficient data to statistically validate the results. Despite those factors, the laboratory experimental program did result in the confident selection of one grout formula that adequately met a prescribed set of optimal performance criteria, and the grout was subsequently tested in the field at Fort Union National Monument in 1993. Since then, Fort Union has embarked on an extensive plaster conservation program in the Mechanics Corral and other locations within the park using the grout.

It is hoped that long term monitoring of the field work and further laboratory testing into the adhesive properties and durability of hydraulic lime-based grouts will address many of the issues left unexplored in this initial phase of research.

1.0 Introduction

The philosophy and practice of conservation has recently evolved from one that tended to preserve cultural materials as objects with a prescribed value, to one that aims to preserve cultural materials as a resource with many values. The obligation now is to conserve not only the object, but also its information potential. This type of conservation approach allows for objects, even entire sites, to be preserved and studied within their cultural context.

The value of a cultural resource is not immutable and cannot be determined solely based on its physical attributes; human cognition and context are required as well (Lipe 1984, 2). Within the life span of an object, it can have many different values depending on the user's frame of reference. To ensure an object's or site's resource value for future use, some relationship to the original context must be preserved.

Preservation of original context in exposed architectural or archaeological sites is difficult, especially since most sites are directly exposed to the environment and highly susceptible to rapid deterioration. *In situ* conservation of architectural surface finishes is particularly complex because these elements are inherently fragile. In the past, it was common practice to remove "significant" architectural fabric such as painted plasters from archaeological sites for protection and display indoors, and to leave undecorated, or plain plasters unprotected, leading to their ruin and decay. An example of this was in the southwestern United States in the 1930's, where mud plaster murals found at the Hopi sites of Awatovi and Kawaik-a were detached from the mud walls, remounted on hardboard surfaces (Smith 1952, 33-52), and were placed on display and in storage at the Peabody Museum, Harvard University. Though these paintings were reported to be in good condition in 1987 (Silver 1987, 171) their detachment precludes any re-study of the artifact in its original context and restricts the variety of informational studies that can be carried out.

The emphasis now is to conserve what remains of such fragile elements, including both painted and plain plasters and stuccos, *in situ*. By preserving these in place, future contextual studies are preserved for both the near and long term future, and primary information on technology, chronology, and authenticity of a building remain evident. Equally valid, but on a more intuitive level, elements *in situ* also provide tangible and provocative clues to the past that can enrich the experience of a visitor to the site. Lipe puts it well:

> "Physically, cultural resources participate in both the past and present. Their authenticity is the basis for creating in the contemporary viewer the subjective knowledge that he has experienced a contact with the past that is direct and real, however incomplete the experience may be." (Lipe 1984, 4)

Experiencing cultural material *in situ* permits direct access and an interconnectedness with the resource that cannot be reproduced if it is lost to decay or removed to an isolated museum setting.

Since the tendency in the past was to remove painted plaster and stucco from its original support, conservation research and techniques fittingly focused more on detachment techniques such as *strappo* and *stacco*, and transfer of displaced fabric to a new support for storage or museum display, rather than on *in situ* treatment. Only recently has conservation research and practice emphasized stabilizing and reattaching plaster to its original support where possible, with the primary objective of saving it in its original context for the future.

In response to the need and responsibility to conserve cultural material *in situ*, the Architectural Conservation Laboratory at the University of Pennsylvania embarked on a multi-phased research program to study methods and techniques of conserving traditional surface finishes (i.e., plain and decorated lime and mud plaster and stucco) employed on masonry structures. This thesis is one part of that research initiative, and focuses on the reattachment of

historic lime plaster to adobe masonry walls by grouting. This work includes:

- a brief review of existing literature on *in situ* reattachment of plaster;
- results of analytical tests to characterize historic lime plaster and adobe from Fort Union National Monument, the project test site;
- and the design and evaluation of grouts to reattach lime plaster to adobe masonry walls.

1.1 Review of Published Literature

Reattachment of Plaster: Materials and Techniques

A review of conservation literature on *in situ* plaster reattachment revealed that numerous methods and materials have been used, from mechanical reattachment by pinning with steel pins and epoxy (Crosby 1980), to chemical consolidation and injection of adhesives. Some of the adhesive materials have included: natural water-soluble polymers or proteins such as calcium caseinate or lime casein (Mora, Mora, and Philippot 1984); acrylic resin dispersions with fluid coke (Phillips 1980, 1986); thermosetting synthetic resins such as epoxies (Crosby 1980), thermoplastic resins and emulsions e.g. vinyl acetate derived polymers such as poly(vinyl acetate) emulsion (Silver 1994); acrylic dispersions (Chiari 1980; Silver and Snodgrass 1993); and cementitious materials, such as lime, fluid hydraulic mortars or grouts (Ferragni et al. 1984) and plaster of paris (Agrawal 1984). A comprehensive review of the suitability of many of these materials for plaster reattachment is covered in Ferragni et al. 1984.

Most of the published research and field work on *in situ* plaster reattachment has focused largely on lime plasters on stone or brick masonry. Specific research and field experimentation on reattaching lime plasters to earthen supports is even more limited. Epoxies (Crosby 1980), polyvinyl alcohol (Rua, Rajer, and Mostacedo 1993) and polyvinyl acetate emulsions (Silver 1987, 1994) have all been used to reattach delaminating plasters from earthen walls; however, there has

been little coordinated effort to study the effects of these treatments or the viability of their use for large scale detachment conditions. Epoxy and polyester based solutions tend to be ill-favored for large scale repairs due to their tendency for high mechanical strength, hydrophobicity, brittleness, and uncertain performance in exposed and variable environmental conditions.

The problem of reattaching plaster is a difficult one given the complexities of having dissimilar or heterogeneous plaster-substrate systems such as lime plaster on adobe walls. Both lime plaster and adobe can have vastly different physico-chemical and physico-mechanical properties, which can even vary from wall to wall in the same room. For the grout to work successfully as an adhesive and a void filler, it must be flexible and responsive to the physical characteristics and mechanical properties of both adherends. As a result, our research in this area has focused on the design and initial performance evaluation of various hydraulic lime, hydrated lime or lime-clay based grout formulations, and low-pressure injection grouting techniques for reattachment and reintegration of lime plasters on earthen supports.

1.2 Grouting with Hydraulic Lime Based Mixtures

By far, the most comprehensive study and testing of grouts for the reattachment of lime or clay plasters and mosaics, and the model for this study, was undertaken at ICCROM from 1979-1983 (Ferragni et al. 1983, 1984). Their study began by researching materials to use as mortar for consolidating masonry (Peroni et al. 1982), and then led to the development of materials and methods of grouting for reattachment of plaster and tessera of mosaics. As part of the study, the ICCROM research team defined the ideal properties of grouts for reattachment and consolidation, as well as the difficulties met in grouting operations. They also gave specifications to use as guidelines for testing injectable mixtures in the conservation laboratory, and reviewed grouting materials used in the past for *in situ* plaster reattachment. After demonstrating how some of the

materials used previously, such as air-setting lime mixtures and thermosetting synthetic resins, were unreliable and even unsuitable materials for reattachment, they turned their attention to testing grout mixtures based on hydraulic lime binders, in particular, the Chaux Banche Lafarge hydraulic lime. They developed a model for designing, testing and evaluating grouts based on viscosity and injectability, setting time, mechanical strength, soluble salts, porosity, and shrinkage.

Based on results of the ICCROM testing program, hydraulic lime was selected as the preferred binder for injectable grout formulations, and was used by the ICCROM team in field experiments carried out at nine Italian sites in 1982-1983, including the House of Menander in Pompeii, where a hydraulic lime grout was used to stabilize the masonry core and reattach the murals to their tufaceous support (Mora et al. 1986), and in the Church of San Lorenzo in Rome, where the grout was used to consolidate a large detached area of a mural painting (Ferragni et al. 1984). English conservators also used the hydraulic lime grout to reattach early eighteenth century lime plasters on stone at the chapel at Cowdray Ruins (Ashurst 1984). According to published articles, hydraulic lime grouts have since been used to consolidate between layers of plaster on mural paintings in Thailand (Schwartzbaum 1986; Lujan 1991); and to consolidate masonry and reattach wall plaster and stucco on Roman Funerary Monuments in Carthage (Roby 1996), on the Mudejar Templete at the Royal Monastery in Caceres, Spain (Schnabel and Boornazian 1992), on two churches in Wachau, Austria (Hammer 1990), on the Sistine Chapel (Colalucci 1991), and on a Roman Fresco in Jerusalem (Cobau 1993).

In addition to using hydraulic lime for reattaching wall paintings or plasters, the ICCROM team also tested the grout as an adhesive for the consolidation and reattachment of wall mosaics at Torcello Cathedral, and the floor mosaic in Ostia (Ferragni et. al. 1984). Nearly a decade later, a similar grout was used by other conservators to repair the floor mosaics at the Building of the Nile in Zippori, Israel (Nardi 1996) and for the replacement of the Orpheus

Mosaic at Paphos, Cyprus (Kosinka 1991).

Hydraulic lime has also been used extensively to repair stone masonry that supports plaster and wall paintings. A hydraulic lime mixture was used at the Capitol Palace in Rome, to consolidate and fill large cracks in its peperino cornerstones. It was similarly applied to the damaged marble of the Arch of Septimus Severus (Nardi 1986). Essentially, hydraulic lime as an adhesive material and a grout has been used extensively for the last ten years to reattach and consolidate detached materials in a wide variety of situations.

1.2.1 Recent use of hydraulic lime in conservation

A consequence of the 1979-1983 ICCROM study was a renewed interest in using hydraulic lime and other lime-based materials in architectural conservation applications. Hydraulic lime is commonly used in continental Europe for construction and conservation purposes, but is rarely used in the United States, partially due to a preference for using hydrated lime and Portland cement (Boynton 1980, 454). According to Boynton, hydraulic lime lost favor in the US and even in Europe to cement due to its considerably lower compressive strength and slower setting time. Hydraulic lime also lost favor to Type S hydrated lime due to its lack of uniformity in performance, even within the same source, and reduced plasticity (Boynton 1980, 452). The variable nature of hydraulic limes was proven in a recent study on lime-based materials for use in repairing Hadrian's Wall as part of the Smeaton Project. Field tests demonstrated that hydraulic limes performed differently depending on the type of hydraulic lime utilized in the mix (Teutonico et. al 1994, 35).

The only known producer of hydrated hydraulic lime in the United States is the Riverton Corporation in Virginia. The Riverton Corporation has been producing hydrated hydraulic lime since the late 1920s, and uses it primarily as a component, along with Portland and other cements,

in their masonry cements. Little has been published regarding analysis and testing of the pure Riverton hydrated hydraulic lime for structural or conservation purposes. Riverton hydrated hydraulic lime has been used in the field as a grout to stabilize fractures in the sandstone masonry walls of the Universalist-Unitarian Church in Riverside, California (Twilley and Podany 1986) and at the Ohio State Capitol for all stone repairs. It was also used to repair fill losses in the nineteenth century limestone column in the convento at Mission San Jose, San Antonio, Texas (Brackin 1994) .

1.3 Grouting Adapted for Architectural Conservation

Grouting is the injection of a liquid binding material into a concealed area or void. The grout cures or sets into a gel form to fill voids and to strengthen weak areas. Grouting has been used for centuries to repair man-made structures such as masonry walls, bridges etc., and has also been used since the turn of the 20th century to consolidate and strengthen the soil foundations of large-scale civil engineering structures such as dams, tunnels and mines (Houlsby 1990, 271). The Middle English root of the word "grout" is "grut," meaning coarsely ground meal or porridge--"grut" being used to describe liquid mortars of similar consistency. Smeaton used the word "grut" in that context in his book about the construction of the Eddystone Lighthouse. (Houlsby 1990, 208).

Recently, low pressure, or gravity-feed grouting techniques have been modified from the civil engineering and geotechnical practice for use in architectural conservation as a method to stabilize and reinstate adhesion of weak or detached non-structural elements such as plasters on walls and tessera in mosaics. (Ferragni et al. 1983, 1984; Matero 1994).

1.3.1 Cementitious grouts

There is a vast array of grout types used in engineering practice, the most common being cementitious (aqueous suspension) grouts and chemical (solution) grouts. Cementitious grouts are the type that have been modified for use in conservation applications. Cementitious grouts are those that consist of inorganic binders such as cements or lime, fillers, usually admixtures, and water to form an aqueous suspension (Long 1990, 232). In the 1950's the US Army Corps of Engineers led a research initiative to study the behavior of cementitious grouts. It is from that focused effort that many of the basic principles and standardized tests for cementitious grouts were developed.

1.3.2 Shared aspects of grouting in engineering and conservation practice

In many ways, the properties and functions of grouts in engineering practice are far afield and even opposed to conservation principles and requirements[1]. Yet, there are some aspects of cementitious grouting, particularly with regard to the methodology of designing and preparing grout formulas, that are common to both fields. The following list briefly summarizes basic theoretical principles of grouting that apply broadly to all successful grouting practices:

- Grouting is a concealed treatment. The properties of the grout in both the liquid and solid states must be formulated and applied specifically to meet site conditions.
- Grouts must have fluid properties to allow for injection into voids, while retaining sufficiently stability to resist settling and displacement after injection (Littlejohn 1982,35).
- The properties of the grouts in the liquid state directly affect the performance of the grout in the cured state.

[1]Principally, the characteristics of an effective grout in engineering practice are maximum penetration into permeable materials to seal all voids (i.e. consolidation resulting in impermeability), high strength and permanence (Bowen 1981, 1).

- In the liquid state, grout particles must be separate from each other (no flocs or clumps of grains) and each active particle must be thoroughly wet. This chemically activates each particle, giving the full hydration necessary for strength and durability (Houlsby 1990, 24).
- All inert particles (fillers) should be thoroughly coated in the binding media, creating a uniform mixture throughout.
- Grout must have suitable setting time to insure stability and adhesion in the wet and semi-cured states.
- Optimal grout should achieve maximal volume to fill voids with minimal stress on the supporting material.
- Grouts must have little to no shrinkage to maintain maximum void filling potential.

2.0 Field Site: Fort Union National Monument

An important component of this research on *in situ* reattachment of lime plasters to adobe substrates was to apply the results to real field conditions. In 1991, a site assessment of the adobe ruins at Fort Union National Monument in New Mexico, followed by a condition survey and a modest plaster reattachment pilot program, led to the selection of this site for treatment.

This chapter includes a brief history of Fort Union and its preservation efforts, a summary of the condition of the historic plasters, and basic laboratory characterization of the historic lime plaster and adobe building materials.

2.1 History of Construction at Fort Union

Fort Union National Monument is located 100 miles northeast of Santa Fe along the historic Santa Fe Trail in Mora County, New Mexico. Three forts have existed on this site. The ruins of the Third Fort Union (adobe and stone ruins dating from 1863) are the most intact, and now constitute the largest adobe ruin in North America (Matero 1994).

The majority of the Third Fort buildings were built of masonry construction: adobe walls on sandstone foundations with brick fireboxes, chimney stacks, and copings. Most of the adobe structures were roofed flat and covered with tin-coated iron plates. As a general rule, most of the exteriors and interiors of the adobe buildings were plastered and stuccoed, and often painted.

Some of the adobe soil used for construction of the buildings may have been collected locally from a large triangular field of parallel furrows west of the depot (HBM 98). The stone used for the dressed and rough work foundations and walkways, a fine-grained Dakota formation sandstone, was quarried from the canyon walls less than two miles south of the fort. The use and production of lime for mortar and plaster, documented as early as 1851, is confirmed by the numerous lime kilns at the site.

Most of the Third Fort adobe buildings were covered with a protective exterior stucco and an interior plaster. Recipes reported in the military documents during the late 1860s and 1870s indicate that two different formulations were generally used at Fort Union[2]: exterior stuccoes consisted of 6 parts lime, 1 part gypsum, and 3 parts charcoal, sometimes with earth added; interior plasters were composed of lime, gypsum, and animal hair.

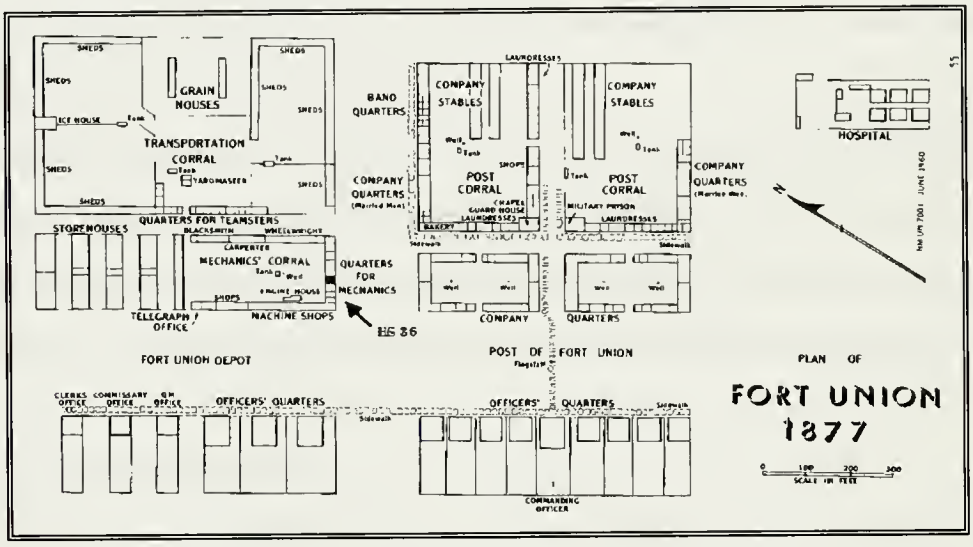

Figure 1. Plan of Fort Union in 1877. HS 36, known as the Mechanics Corral, was the area selected by the NPS for plaster conservation treatment. The darkened area is room 24, where one of the adobe samples analyzed in this testing program was taken. (from Utley, 1962)

Beginning in 1868, only five years after construction of the Third Fort began, inspection reports recorded the dire condition of the buildings, citing fallen plaster and exposed adobe on the interior and exterior walls. As construction failed, water seeped through the roof-wall junctures eroding the adobe core, which contributed to detachment and failure of plasters and stuccoes. Photographs of the 1870s and 1880s indicate that much of the original exterior stucco had fallen off by that time. By February of 1891, 28 years after it was built, Fort Union was abandoned.

---

[2] Materials and techniques for rough cast and plaster are found in the Fort Union building reports of June 30, 1873; June 30, 1874; March 29, 1875; and June 30, 1877.

2.2 Preservation at Fort Union

Beginning with the establishment of the park in 1954, experimental testing of then new chemical treatments and the eventual use of a wide variety of conservation approaches for the preservation of historic adobe and plaster occurred at Fort Union. Treatments to the plasters and stuccos included: structural stabilization with tension wires and steel plates (1956), lime and cement fills and plaster edging, and spraying of silicone water repellents on the plaster and adobe surfaces (c.1964-late 1970s) (Matero 1994).

Current preservation work at Fort Union addresses the preservation of the adobe ruins through a continuous program of cyclical maintenance involving traditional adobe capping and mudding. Extant plaster was preserved by maintaining the stability of the adobe wall on which it was attached, and by filling wide gaps along abrupt and broken plaster edges with mud.

2.3 Condition of Fort Union Plaster

The principal deterioration mechanism causing loss of adhesion and detachment of the Fort Union plasters was the infiltration of water behind the plaster. As water penetrated through cracks or along broken edges of plaster, it softened the adobe, causing it to lose cohesion and weaken the bond between the plaster and the supporting adobe wall. Eventually, the partially unsupported plaster deformed and became displaced, forming a void where loose debris could accumulate. The stress caused by this action progressively led to more cracking and eventual plaster loss. Secondary deterioration mechanisms were the intrinsic weakness of the bond between lime and adobe, made worse by poor quality of original construction achieved by untrained army personnel who knew little about durable adobe construction.

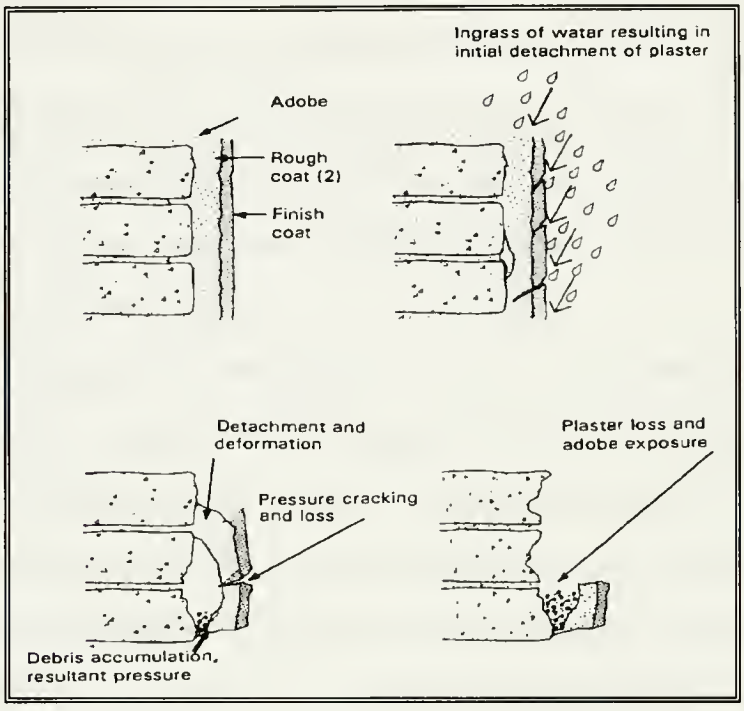

Figure 2. Schematic of plaster detachment processes at Fort Union NM. (from Matero 1995, 14; designed by Maribel Beas)

A condition survey conducted the National Park Service and the University of Pennsylvania in 1991 revealed that loss of adhesion was most prevalent along plaster edges where frequent and focused water action eroded the adobe and created a channel that undercut the plaster. Tapping on the surface and observation of significant deformation and bulges in the plaster surface indicated that detachment was widespread and not limited to only the edges. By comparing the plaster with 1960's photographs, it became obvious that plaster loss was progressing, and that in some cases, as much as 25% had been lost in just over 20 years (Matero 1994).

After the 1991 survey and assessment, it was decided that the principal objective of the plaster conservation program was to maintain stability of the supporting adobe walls, prevent water from infiltrating the walls, and secure the plasters *in situ* by grouting.

In June of 1991, a full graphic condition survey of the extant plasters and a modest pilot treatment program was undertaken at Fort Union by the Architectural Conservation Laboratory at the University of Pennsylvania and the National Park Service. The treatments took place in three areas[3] , and included grouting with various hydrated lime and hydraulic lime mixtures, as well as edging, compensation of losses, and cleaning. The performance of the test areas was monitored and assessed over the following year.

Approximately eight months after the initial intervention it was observed that the hydraulic lime mixtures were performing well. The areas stabilized with the hydraulic lime grout were well adhered. No new voids, cracks or bulges were detected. The plaster edges that had been filled with a hydraulic lime edging mix remained well attached to both the plaster and the adobe wall with no associated undercutting of the adobe. Though the results of tests using the Type S lime-based grout were equally impressive, it was assumed that hydraulic lime-based mixtures would be better suited for grouting deep voids where CO_2 may not be available in large enough quantities for lime carbonation to occur.

2.4 Characterization of Historic Fort Union Adobe and Plaster

Prior to selecting materials to make an adhesive grout, the historic adobe and historic lime plaster scratch from Fort Union were characterized. The objective of the analysis was to determine the components and basic properties of the adherends in order to select compatible grout ingredients.

Both adobe and plaster samples from Fort Union were collected by Jake Barrow, Exhibit Specialist for the National Park Service, and sent to the Architectural Conservation Laboratory at the University of Pennsylvania for testing. Two types of Fort Union adobe were characterized: a

[3] Test Site 1: HS 29, Room 7, east wall; Test Site 2: HS 28, Room 3, southeast corner; Test Site 3: HS 28, Room 1, north wall.

historic adobe sample taken from a standing historic wall in the Mechanics Corral HS 36, Room 24 (hereinafter referred to as HS 36) and an adobe sample taken from the Boneyard, a refuse area that includes both discarded historic and modern adobes. It is uncertain if the Boneyard sample was historic or new adobe material. Analysis of the adobes included:

1. particle size distribution (ASTM D 422-63)
2. plasticity index and coefficient of activity (ASTM D 4318-84)
3. soluble salts- quantitative
4. organic material- quantitative
5. pH (ASTM D 4972-89)
6. determination of crystalline components by X-ray diffraction (XRD)

The historic plaster sample was taken from a fragment that had fallen to the ground in HS 36. Only the scratch coat, the portion that would have been attached to the adobe wall, was analyzed in most of the tests. Analysis of the plaster included:

1. carbonate content by acid dissolution and gravimetry
2. examination of stratigraphy by optical microscopy
3. identification of sulfates by microchemical spot testing
4. determination of crystalline components by X-ray diffraction (XRD)

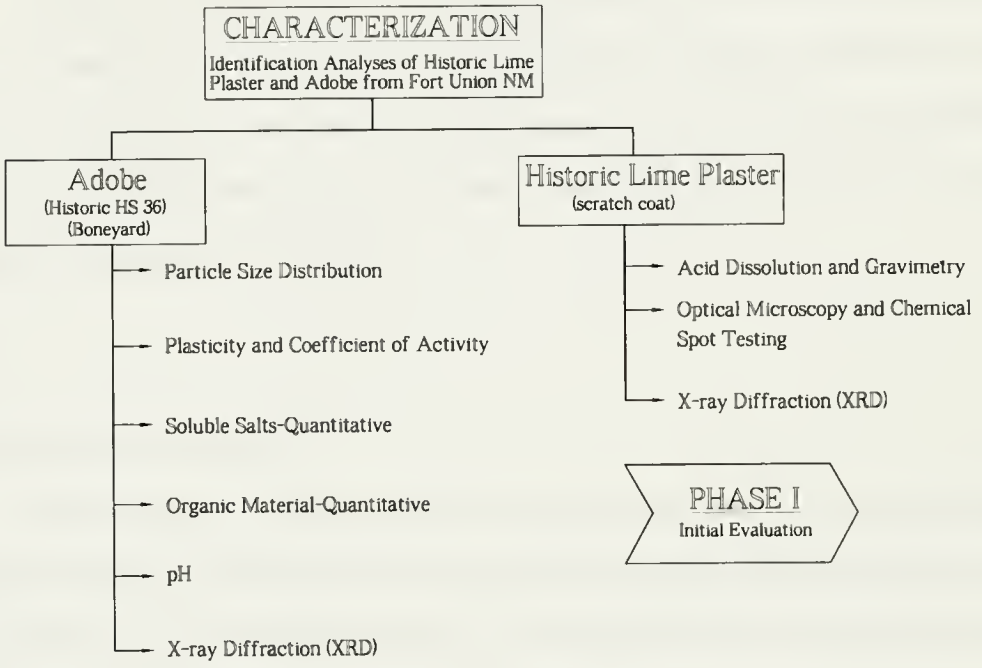

Figure 3. Flow chart of characterization tests conducted on Fort Union adobe and plaster samples

Whenever possible, laboratory experiments were conducted in accordance with ASTM standards, but often the tests were modified. In addition to ASTM, testing methods were also drawn from: A Laboratory Manual for Architectural Conservators by Jeanne Marie Teutonico, 1988; from courses in Advanced Architectural Conservation, run at the University of Pennsylvania Architectural Conservation Laboratory under the direction of Frank Matero and Dr. Alberto Tagle; and from standards established by the Italian NORMAL Committee, and Unesco RILEM.

2.4.1 Adobe characterization

Adobe is a composite material consisting essentially of soil and often organic matter. The physical properties or character and behavior of the adobe depend partially on the natural composition of the soil, in particular the clay mineralogy, and on the grain size distribution of sand, silt, and clay particles. The principal constituents of adobe are usually sand and silt in which clay minerals serve as the binder.

2.4.1.1 Particle size distribution

Test Procedures - Classifying soils by their particle size ratios of sand, silt and clay, and grouping soil types into categories that possess similar properties, was part of a larger system of soil classification developed by A. Casagrande in 1948 (Bell 1983). To determine the particle size distribution in the Fort Union adobe samples, sieving and sedimentation methods were used. Procedures for sieving and sedimentation were taken from A Laboratory Manual for Architectural Conservators by Jeanne Marie Teutonico, 1988[4].

In this procedure, an adobe sample was crushed using a mortar and pestle, weighed, and then soaked in a solution of Calgon (sodium hexametaphosphate) and distilled water. The Calgon acts as a deflocculating agent to disperse clay particles and to ensure that all particles settle individually. The sample was then sieved through a #200 (75μm mesh) screen to separate the coarse-grained particles from the finer silt and clay. The soil retained on the #200 sieve, particles greater than 75μm, was oven-dried, weighed, and sifted through a series of ASTM test sieves. Each sieve has successively smaller mesh sizes, which allows for particles larger than the mesh size to be retained, and particles smaller than the mesh size to pass through. The weight of the soil retained on each sieve was measured and calculated as a percentage of the whole sample. The

[4] Adapted from ASTM D 422-63.

following ASTM sieves were used:

Sieve Number	Diameter opening (mm)
4	4.75
8	2.36
16	1.18
30	600µm
50	300µm
100	150µm
200	75µm

Table 1. ASTM sieve sizes and diameter opening of the screen

The particle size distribution of the fine-grained fraction, silt and clay having a particle size smaller than 75µm, was determined by the sedimentation method. The fine fraction was placed in a glass sedimentation cylinder filled with deionized water, and allowed to settle over time. The specific gravity of the suspension at a particular depth was measured at regular intervals with a soil hydrometer . This test is based on Stokes law which states that the terminal velocity is proportional to the square of the particle diameter, or more simply, larger particles in suspension settle more quickly that small particles (Teutonico 1988, 83).

Results - Following are results of the particle size analysis of the historic HS 36 sample and the Boneyard sample. Percentages are based on ASTM particle size conventions[5] for clay, silt, sand, and gravel. Table 2 and Figure 4 summarize the particle size distribution for both the adobe

[5]Particle size breakdown based on ASTM D 422-63:
gravel: 4.75mm-76.2mm
sand: coarse 425µm-4.75mm
fine 75µm-425µm
silt: 2µm-75µm
clay: ≤2µm

samples tested. The sieve analysis is included in Appendix A.

Adobe Sample	% Gravel	% Sand	% Silt	% Clay
HS 36	1.9	62.0	24.8	11.2
Boneyard	0.2	55.1	27.4	17.3

Table 2. Adobe grain size distribution

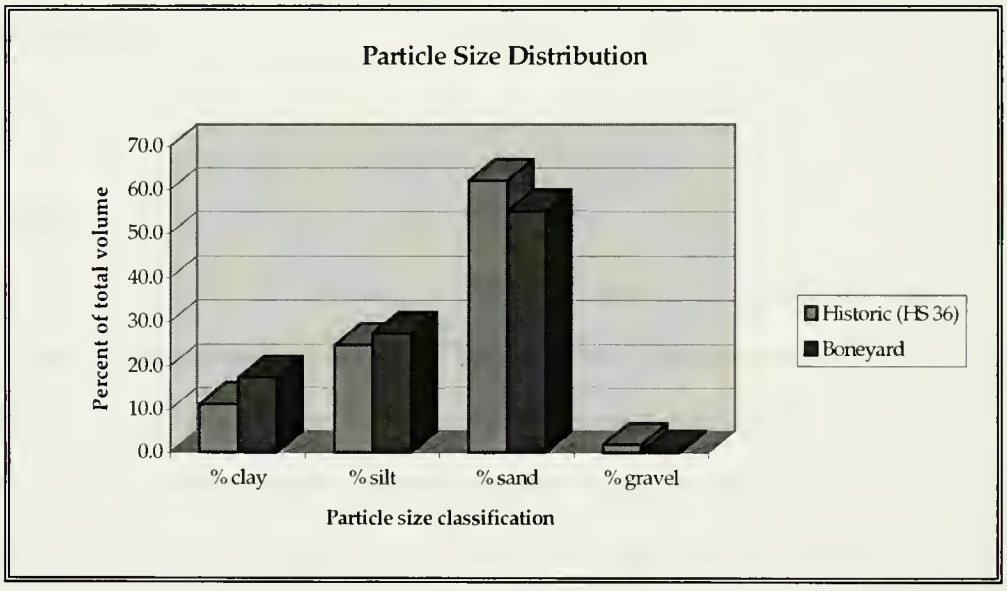

Figure 4. Adobe particle size distribution graph

Discussion - The results of the particle size analysis show that the HS 36 sample is composed of 62% sand, 25% silt, and 11% clay (weight percentages). The Boneyard sample consists of 55% sand, 27 % silt, and 17% clay (weight percentages). According to the Unified Soil Classification System (Wagner 1957), the soil in both adobe samples is considered coarse and is classified as a silty sand.

It is well known that particle size ratio affects the performance of adobe. Both the Fort Union adobe samples have relatively high silt and clay contents, nearly 36% by weight for the historic HS 36 sample, and 45% by weight for the Boneyard sample. High silt and clay adobes tend to be very cohesive and durable, but they also tend to shrink and have a higher coefficiency of expansion and lower porosity than high sand content adobes. In an exposed environment such as Fort Union, where the adobe structures are in ruins and exposed to direct sunlight, driving rain and snow, the adobe could undergo considerable expansion and contraction, even on a daily basis. Mechanical stress caused by such action may have been one of the factors contributing to plaster detachment.

2.4.1.2 Atterberg limits and plasticity index

Test Procedures - The Atterberg limits and plasticity index are values used to describe the limits and performance of a soil. Two of the seven limits of consistency defined by Dr. Albert Atterberg in 1932, the plastic limit and liquid limit, were used in this study. The plastic and liquid limit are the lower and upper limits, respectively, of the range of water content over which a soil exhibits plastic behavior (Craig 1992, 8). The plasticity index is determined by calculating the difference between the plastic limit and the liquid limit values of a soil. Plasticity describes the ability of a soil to undergo unrecoverable deformation at constant volume without cracking or crumbling, and is directly related to the amount and type of clay minerals and organic matter present (Craig 1992, 6). It is generally assumed that the higher the plasticity index, the greater the tendency of the soils to expand and contract during wetting and drying (Teutonico 1988, 6) and the lower the strength.

Test methods used to determine liquid and plastic limits for both Fort Union adobe samples were taken from A Laboratory Manual for Architectural Conservators by Jeanne Marie

Teutonico, 1988[6]. The liquid limit of a soil is the water content expressed as a percentage of the oven-dried soil at the boundary between the liquid and plastic states (Teutonico 1988, 102). The liquid limit was determined by using a Casagrande device, an apparatus consisting of a flat metal cup mounted on an edge pivot that holds a volume of wet soil paste that has been previously dry sieved through a 425μm sieve. The soil paste is grooved with a standard grooving tool, and the cup dropped repeatedly at a distance of 1cm until the two halves of soil gradually come together. The moisture content of the paste is determined by oven-drying, and expressed as a percentage of the weight of the oven-dried soil.

The plastic limit of a soil is defined as the water content expressed as a percentage of the mass of dry soil at the boundary between the plastic and semi-solid states (Teutonico 1988, 102). Plastic limit is determined by mixing dry soil sieved through a 425μm sieve with enough distilled water for it to become malleable enough to roll into threads A thread is rolled uniformly throughout until it is reduced to a diameter of 3mm. The procedure is repeated until the thread fails by breaking into pieces before reaching a diameter of 3mm. The failure point is considered the plastic limit. The moisture content of the threads, determined by oven-drying, is calculated and expressed as a percentage of the weight of the oven-dried soil.

[6] Adapted from ASTM D 4318-84, "Standard Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils.

Results- The plastic and liquid limits for the HS 36 sample are 21.41% and 34.98%, respectively; for the Boneyard sample, they are 13.32% and 31.40%. The plasticity index for each, calculated as the difference between the plastic and liquid limit values, ranges from 13.6 for the HS 36 sample, to 18.1 for the Boneyard sample. Since there is a mathematical relationship between liquid limit, plastic limit and plasticity index, it is also possible to assign a coefficient of activity value to the samples, as well as further characterize them in terms of their cohesiveness and expansiveness.

Coefficient of activity is a value that describes the degree of plasticity or the activity of the clay-sized fraction. It is determined by dividing the plasticity index by the amount of clay in the soil (Houben and Guillaud 1994, 59). Using this calculation, the coefficient of activity value for the HS 36 sample is 1.2, and the Boneyard sample is 1.0. Both values are considered to be in the range of "average activity" according to the following rating:

<0.75 = inactive
0.75-1.25 = average activity
1.25-2.0 = active
>2 = very active

Both samples are also considered to be medium cohesive (determined by dividing the plasticity index by the liquid limit) and to be medium expansive (determined by dividing the plasticity index by the quantity of clay). Levels of cohesion and expansion range from low to high. The numeric values that define each level are found in Houben and Guillaud 1994, 59.

Adobe Sample	Plastic Limit	Liquid Limit	Plasticity Index	Clay Content	Coefficient of Activity
HS 36	21.41%	34.98%	13.57%	11.25%	1.21
Boneyard	13.32%	31.40%	18.08%	17.31%	1.04

Table 3. Adobe plasticity index and coefficient of activity values

2.4.1.3 Quantitative analysis of soluble salts

Test Procedures - A simplified quantitative analysis of the historic adobe sample was undertaken to detect the presence of soluble salts. High percentages of water-soluble salts in either the historic adobe or the plaster could affect the performance of the grout. If soluble salts concentrate and crystallize at the adobe-grout-plaster interface, it can disrupt the bond between two systems and lead to grout failure and further detachment. Some of the most damaging ions are sulfates of sodium, potassium, and calcium.

The simple procedure used to determine the total percentage of soluble salts was based on gravimetry, and consisted of dispersing a weighed dry sample in deionized water, magnetic stirring for 30 minutes, collecting the filtrate, and weighing the sample again after drying (Houben and Guillaud 1994, 66). The difference in the weight between the samples is attributed to the amount of soluble salts.

Results - The test was conducted on three individual samples from HS 36. The average percentage of water-soluble salts detected is low. The lower the amount of soluble salts, the lower the risk of damage to the historic materials. Qualitative analysis of the soluble salts was not performed. Identification of the alkaline elements can be conducted by instrumental methods such as X-ray diffraction or atomic absorption spectrometry, or of the individual cations by microchemical spot tests. The test data is reported in Table 4.

Adobe Sample	Weight Before (g)	Weight After (g)	% Soluble Salts
HS 36-1	200.00	193.96	3.02%
HS 36-2	200.00	195.22	2.39%
HS 36-3	200.00	197.06	1.47%
			mean 2.29%

Table 4. Results of quantitative soluble salts test

2.4.1.4 Quantitative organic content

Test Procedures- Another material component of adobe is fibrous organic material such as straw or grass. Plant or animal fibers are often added to hinder cracking, accelerate drying, and to increase tensile strength (Houben and Guillaud 1994, 83).

The amount of organic material in the sample from HS 36 was determined by decomposing the organic compounds by dry ashing or oxidation. In this method, the sample was first lightly crushed with a mortar and pestle and then sieved through a #30 (600μm) sieve. The percent passing the sieve was divided into three smaller specimens and oven dried at a temperature of 105°C for six hours, allowed to cool in a dessicator, and then weighed. Weight loss was attributed to loss of water and CO_2 . The samples were then placed back in the oven at a higher temperature, 300°C, for eighteen hours, cooled in a dessicator, and weighed. The weight difference between the dried and the combusted sample was calculated, and the difference was attributed to combustion of organic matter (Shugar 1990, 301).

Results - Combustion of the three adobe samples from HS 36 resulted in a total weight loss of 5.99-5.95% from the original sample. Initial heating to 100°C indicates that some weight loss (0.53-0.52%) is due to water or other volatiles such as CO_2 within the organic fraction. The remainder of weight is attributed to combustion of organic matter. See Table 5.

Visual examination of the HS 36 sample prior to combustion revealed what appeared to be bits of dried grass or some type of dried vegetal matter.

Adobe Sample	Sample Weight Before (g)	Sample Weight After 105°C (g)	Sample Weight After 300°C (g)	Total % Weight Loss	% Weight Loss from Water and CO_2	% Weight Loss from Organic Material
HS 36-1	60.12	59.80	56.52	5.99%	0.53%	5.46%
HS 36-2	60.30	59.98	56.73	5.95%	0.53%	5.39%
HS 36-3	60.05	59.74	56.49	5.96%	0.52%	5.41%
					mean 0.53%	mean 5.42%

Table 5. Results of quantitative organic content analysis

2.4.1.5 pH

Test Procedures - pH is a measure of the hydrogen -ion concentration or activity in material, in this case, a soil solution. pH is measured in terms of acidity and alkalinity. An acid is a substance that yields hydrogen ions when dissolved in water. A base is a substance that yields hydroxyl ions in water (Shugar 1990). pH values range from 0-14, with 7 (concentration of H_30^+ and OH^- equal to 10^{-7}) representing neutrality, numbers less than 7 increasing acidity, and numbers greater than 7 increasing alkalinity.

pH is an indicator of the chemical properties of a soil (McLean 1982). pH measurements can be influenced by soluble salts, organic matter, and CO_2 content in the soil. pH can affect the stability of the clay minerals in the adobe: high pH can lead to the formation of stable clay minerals in suspension; low pH can promote clay flocculation. (Clifton et al. 1978, 10).

The pH level of both adobe samples from Fort Union were measured electrometrically with an Orion 91-50 pH meter. The pH meter measures the hydrogen ion concentration by immersing two electrodes into an aqueous soil solution. The two glass-calomel electrodes respond to the change in H_30^+-ion concentration (Shugar 1990).

To measure the pH of the soil, three 25g samples from each sample were sieved through a #30 (600μm) sieve and then mixed with distilled water (at 25°C). After 2 hours of soaking the electrodes of the pH meter were first standardized in a neutral yellow buffer solution and then placed in the soil solution. The pH of the solutions was read directly off the meter.

Results - The pH of the HS 36 sample is close to neutral 7.3. The Boneyard sample is slightly more alkaline at 9.96. The data is presented in Table 6.

Adobe Sample	pH	Adobe Sample	pH
HS 36-1	7.4	Boneyard-1	8.0
HS 36-2	7.3	Boneyard-2	7.9
HS 36-3	7.3	Boneyard-3	8.0
	mean 7.3		mean 7.96

Table 6. Adobe pH results

2.4.1.6 X-ray diffraction

Test Procedures - X-ray diffraction (XRD) is a method of instrumental analysis used to identify the mineralogical composition or crystallographic structure of materials. This method of analysis is especially useful for detecting minerals, inorganic pigments, metals, corrosion crusts etc., as long as they are crystalline and in measurable ranges >5%[7] of the sample. The objective of conducting XRD on the Fort Union adobe samples was to identify clay minerals in the adobe.

XRD is a technique where a powdered sample is irradiated with a beam of monochromatic x-rays, which are diffracted or reflected by the crystal planes (atomic layers) within the sample. The angle and the intensity of the diffracted X-rays are distinctive, and like

[7] from lecture notes on instrumental analysis given by Dr. Alberto Tagle, University of Pennsylvania, Historic Preservation, 1991.

fingerprints, are unique to that material. The position of the diffraction lines and their intensity are registered on a diffractogram, which are then compared to diffraction patterns of known samples. XRD also gives indirect information about chemical composition (Newman n.d.).

Results - XRD was conducted on both HS 36 and Boneyard adobe samples[8]. It revealed that the crystalline components of both adobes were quartz, feldspar, and calcite, with some illite and kaolinite clay[9]. The results are tentative with regard to the identification of the clay minerals. It was impossible to precisely identify the clay minerals because the degree range the reflection covered during scanning was too wide, from 6°-140°. The range of reflection should have been between 2°-40°, with the degree range from 2°-7° being the area on the diffractogram which is critical for the identification of the expandable clays. Furthermore, the samples were not prepared correctly. Proper preparation requires eliminating the non-clay minerals, orienting the clay minerals with their c-axis perpendicular to the slide, and using solvation techniques to swell clay to known positions[10].

Table 7 lists the minerals found in the order of concentration. Diffractograms are included in Appendix A.

Adobe Sample	Mineralogy	Clays
HS 36	quartz > feldspar > calcite > clays	illite > kaolinite
Boneyard	quartz > feldspar > calcite > clays	illite > kaolinite

Table 7. Adobe X-ray diffraction results

[8] XRD was conducted at the Laboratory of Research on the Structure of Matter at the University of Pennsylvania, using a Rigaku X-ray Diffractometer. Operation of the equipment was overseen by Elmer Anderson.

[9] According to Houben and Guillaud, illite is not particularly stable in contact with water and can swell. Kaolinite is generally stable. (Houben and Guillaud 1994, 27)

[10] XRD was conducted by the author. Diffractograms were interpreted by George Austin at the New Mexico Bureau of Mines and Mineral Resources. For further information on sample preparation see Austin and Leininger, 1976.

2.4.2 Plaster characterization

Plaster or stucco is a term used to describe the interior or exterior finish of a wall. Plaster and stucco protect a wall from exposure to wind, rain and other elements, and are often a form of surface decoration. Plasters are a composite material composed of various proportions of a binder, aggregates and fillers, and often additives. Common binders are clay, lime and gypsum.

At Fort Union, the interior plasters were recorded to be composed of lime, sand, gypsum and animal hair (Matero 1995, 12). Characterization of the plaster in the laboratory was conducted to verify the principal constituents.

2.4.2.1 Determination of calcium carbonate content by acid dissolution

Test Procedures - To determine the ratio of binder to aggregate, a simple acid dissolution method was used based on A Laboratory Manual for Architectural Conservators (Teutonico 1988). The results provided information on weight percentages of the acid soluble fraction (attributed to a calcareous binder), and the insoluble fractions, generally sand.

In the acid dissolution method, a 14% hydrochloric acid solution in water is used to dissolve the calcium carbonate from a weighed and crushed sample. The reaction that takes place is $2HCL + CaCO_3 \rightarrow CaCL_2 + CO_2 + H_2O$. Hydrochloric acid reacts with the carbonate to liberate CO_2. The insoluble carbonate is converted into a soluble chloride, which can be washed away with water. (Moncrieff and Weaver 1983). What remains of the sample is the insoluble fraction (aggregate and other fines). The weight of the insoluble material is subtracted from the total weight, and the difference is attributed to the amount of dissolved calcareous binder in the sample.

Following acid dissolution, the sand aggregate was sifted through a stack of ASTM sieves, ranging from sieve #8, with a mesh diameter of 2.36mm, to sieve #200, with an opening of 75μm. The weight of the soil retained on each sieve was measured and calculated as a percentage

of the whole sample.

Results - The results of the gravimetric analysis show that the binder to aggregate ratio of the sample is approximately 1.0:4.4 (w/w) with 31.74% coarse to medium sand; 66.80% fine sand, and 1.46% silt/clay[11]. Examination of the coarse fraction under a binocular microscope with normal reflected and polarized light at 30x revealed the sand to be sub-angular and composed primarily of quartz and calcite (as based on comparison with a particle atlas).

The plaster was not analyzed for clay silicates to determine if the binder was naturally hydraulic (e.g. a natural cement or hydraulic lime).

Results of the analysis are presented in Tables 8 and 9.

Weight of Powdered Plaster Sample (g)	Weight of Dry Sand After HCL (g)	Weight of Dry Fine Particles After HCL (g)	% Sand	% Fines	% Dissolved Binder
36.90	23.50	5.04	63.68	13.66	22.65

Table 8. Results of acid dissolution of plaster scratch coat

ASTM Sieve Size	Weight Retained (g)	% Retained	% Passing
4	0	0	100
8	14.5	5.08	94.92
16	37.7	13.12	81.80
30	38.6	13.54	68.26
50	132.3	46.42	21.84
100	48.6	17.05	4.79
200	9.5	3.33	1.46
dish	4.2	1.46	0
original weight of sample 285g			

Table 9. Sieve analysis of plaster scratch - insoluble fraction

[11] The results of the acid dissolution test to determine ratios of binder to aggregate can be imprecise due to limitations such as dissolution of calcareous aggregate, which could misrepresent the aggregate content, and because it cannot distinguish between clay minerals present as impurities and as silicates in hydraulic cements (Teutonico 1988).

2.4.2.2 Optical microscopy and chemical spot testing

Test Procedures and Results - Microscopic examination of a complete plaster sample from Fort Union in normal reflected light at 20x revealed the plaster system to have a multi-layered stratigraphy. The layer adjacent to the adobe, the scratch coat, was approximately 3.0cm thick and composed of lime and a coarse sand aggregate; the succeeding layer, designated as the brown coat, was approximately 0.75-1.0cm thick, had a much finer texture, and was composed of what appeared to be lime and a fine sand aggregate. This was then covered by two to three layers of limewash. No paint layer was detected on the sample, but it is known to exist on some plaster fragments *in situ* at Fort Union.

Microchemical spot tests were conducted on the plaster scratch coat to detect the presence of sulfates, in particular, calcium sulfate, also know as gypsum. Building records from Fort Union[12] state that gypsum was a component of the interior plaster and exterior stucco. To test for sulfates, a small portion of the plaster scratch coat was crushed into a powder with a mortar and pestle and then dissolved in distilled water. The solution was placed in a test tube and 2 drops of barium chloride ($BaCl_2$) were added to the solution. If sulfates are present, a white precipitate of barium sulfate ($BaSO_4$) should appear; in this case, no precipitate was observed, indicating that sulfates may not be present in this sample. The test was conducted twice, producing a negative result both times.

[12] June 30, 1873; June 30, 1874; March 29, 1875; and June 30, 1877

2.4.2.3 X-ray diffraction

The objective of conducting XRD on the historic Fort Union plaster scratch was to identify calcium silicates and aluminates, if any, suggesting the use of a hydraulic lime or natural cement. For a description of the XRD procedures see section 2.4.1.6.

Results - No valid results were produced regarding clay mineralogy. As with the XRD conducted on the adobe samples, the range for determining the clay fraction was not accurately scanned and reading of the silicate fraction was impossible.

The interpretation of the XRD conducted by George Austin indicated that at least one of the diffractogram peaks may be attributed to feldspar. Table 10 lists the minerals found in the order of concentration. The diffractogram is included in Appendix A.

Plaster Sample	Mineralogy	Clays
HS 36-scratch coat	quartz > feldspar > clays	trace illite and kaolinite

Table 10. Plaster X-ray diffraction results

2.4.3 Summary of analysis

Sample Type	Sample Location	Test Type	Test Designation	Results
Historic Adobe	HS 36	Particle Size Distribution - Sieve Analysis	ASTM D 422-63	1.93% Gravel 62.04% Sand
Adobe Historic	HS 36	Particle Size Distribution - Sedimentation	ASTM D 422-63	24.78% Silt 11.25% Clay
Adobe Historic	HS 36	Plasticity Index: Plastic and Liquid Limit	ASTM D 4318-84	13.57% 21.41% 34.98%
Adobe Historic	HS 36	Coefficient of Activity		1.21
Adobe Historic	HS 36	pH	ASTM D 4972-89	7.3
Adobe Historic	HS 36	Soluble Salts-Quantitative		2.29%
Adobe Historic	HS 36	Organic Content-Quantitative		5.42% organic 0.53% water
Adobe Historic	HS 36	X-ray Diffraction		Quartz>Feldspar>Calcite> Illite>Kaolinite
Adobe	Boneyard	Particle Size Distribution - Sieve Analysis	ASTM D 422-63	0.07% Gravel 55.26% Sand
Adobe	Boneyard	Particle Size Distribution - Sedimentation	ASTM D 422-63	27.35% Silt 17.31% Clay
Adobe	Boneyard	Plasticity Index: Plastic and Liquid Limit	ASTM D 4318-84 ASTM D 4318-84	18.08% 13.32% 31.40%
Adobe	Boneyard	Coefficient of Activity		1.04
Adobe	Boneyard	pH	ASTM D 4972-89	8.0
Adobe	Boneyard	X-ray Diffraction		quartz>feldspar>calcite> illite>kaolinite
Historic Plaster	HS 36	Determination of Calcium Carbonate Content by Acid Dissolution		1.0 : 4.4 (w/w) (binder: aggregate) 1.74% coarse sand 66.80% fine sand 1.46% silt/clay
Historic Plaster	HS 36	X-ray Diffraction		quartz>feldspar> trace illite and kaolinite

Table 11. Summary analysis of Fort Union historic adobe and plaster samples

Discussion - The historic adobe HS 36 is composed of 62% sand and 36% silt and clay; the Boneyard sample consists of 55 % sand and 45% silt and clay. Both have soils that are classified as coarse silty sand, with medium cohesiveness and medium expansiveness, and with a quantity of clay that places them within an "average activity" bracket. XRD analysis revealed the adobes to be composed of quartz and feldspar, with some illite and kaolinite clay. Determination of the clay mineralogy was not conclusive. Though determination of clay mineralogy is important for understanding adobe deterioration processes, the resulting "average" coefficient of activity values obtained for both samples indicate that the expansive clays may be in low to moderate quantity.

Both soluble salts content and organic content for the HS 36 sample are low, at 2.3% and 5.4%, respectively. Some of the organic material appears to be dry grasses or straw. The pH of HS 36 is close to neutral at 7.3. The Boneyard sample is slightly alkaline at 8.0.

According to the results of the analysis, there is no great difference between the composition of the HS 36 and the Boneyard samples. This can be interpreted two ways: the sample obtained from the Boneyard refuse area was composed of primarily historic material, or that the modern adobes have a composition similar to the historic fabric.

Microscopic analysis of the historic Fort Union plaster system revealed it to have a multi-layered stratigraphy consisting of a 3.0cm thick scratch coat, a 0.75-1.0cm thick brown coat, and a finish of 2-3 layers of limewash. Acid dissolution of the scratch coat indicated that it consisted of lime and sand at an approximate ratio of 1.0:4.4 (w/w). Though military records indicated that gypsum was an ingredient, microchemical spot tests for sulfates were negative.

Based on the results of the adobe and plaster characterization, which were conducted to determine compatible materials for the grout formulas, lime and a stable clay, kaolin, were included as binders in the grout formulas tested in Phase I.

3.0 Grout Components and Sample Preparation

3.1 Performance Criteria

The aim of grouting is to modify or restore properties or functions that have been lost in the original construction. At Fort Union, the grout must perform an adhesive function, where it fills the interstices and larger voids between the detached plaster and adobe wall. It must adhere to both surfaces and, after hardening, achieve a sufficiently durable interface to restore a modicum of cohesive strength to the standing walls.

By carefully selecting materials for the grout formulas based on compatibility and known properties and characteristics, and by modifying and experimenting with the component ratios in the laboratory, specific properties of the grout can be manipulated to meet desired performance criteria. The principal performance criteria used to design and evaluate the grout formulations in the study were:

1. ease of mixing and use
2. adequate viscosity in the liquid phase to fill voids by low-pressure injection
3. minimal segregation and stability in composition until set
4. reasonable setting time to resist displacement and allow proper cure
5. minimal shrinkage between the liquid and solid states
6. low weight
7. moderate strength within the range of the historic material
8. adequate water vapor permeability to prevent moisture accumulation
9. gap filling potential with good adhesive bonding to the adherend surfaces
10. low toxicity

Also important, but not evaluated, was the ability of the grouts to tolerate movement, known as modulus of elasticity, and their durability or weathering resistance.

3.2 Selection of Materials

The grout derives its character from the properties of the individual components, and from the interaction between them; therefore, choosing the proper components for the grouts must be soundly based on chemical, mechanical and physical compatibility of the ingredients with the original material, and an understanding of how they will interact together under predicted environmental conditions. Compatibility is fundamental since grouting is essentially an irreversible treatment. For this study, selection of the grout components was based on a basic understanding of the chemical and mechanical characteristics of the historic lime plaster and adobe from Fort Union as defined in Chapter 2 (see summary results).

In addition to choosing grout components based on compatibility, the grout formulations were also designed to be simple to reduce practical difficulties in site preparation and application. This entailed choosing materials that were readily available at a low cost, and that have a low toxicity. For this reason, the use of some additives such as air-entrainers or water reducing agents was avoided.

Selection of materials to use in the grout formulations was also guided by a basic understanding of the deterioration mechanisms affecting the *in situ* plasters at Fort Union. As previously mentioned, the principal deterioration mechanism was the ingress of liquid water between the plaster and the adobe wall, which contributed to failure of the adhesive bond between the two surfaces. A secondary deterioration mechanism was the inherent weakness of the bond between lime and adobe.

The raw materials selected for inclusion in the initial test program are grouped into three main categories: binders, fillers, and organic admixtures.

3.2.1 Binders

The active component in the grout is the binder, and the properties and performance of the grout in the cured state is largely determined by the binder. Three different binders, Riverton hydrated hydraulic lime, Type S lime, and Kaolin clay, were included in the testing program.

3.2.1.1 Riverton hydrated hydraulic lime

(Riverton Corporation, Riverton, VA).

The Riverton hydrated hydraulic lime is a calcium lime (36% by weight hydrated $Ca(OH_2)$ with "moderate"[13] hydraulic properties forming di-calcium silicate during hydrolysis. According to the Riverton Corporation, their hydrated hydraulic lime (HHL) meets the requirements of ASTM specification C 141-85 "Standard Specification for Hydraulic Hydrated Lime for Structural Purposes," having an average compressive strength of 700 psi at 28 days. EDS (energy dispersive spectrometry) conducted on a sample of pure Riverton hydrated hydraulic lime detected the following elements (in order of intensity): calcium, silicon, aluminum, magnesium, sulfur, potassium, and phosphorus, with traces of titanium and iron (Matero 1995, 98).

[13] It is uncertain how "moderate" is defined. The Riverton Corporation stated that "moderate" referred to its hydraulic strength compared to hydrated lime and Portland cement. There are numerous formal systems used to express the hydraulic value of a cementing material. One is the *hydraulic index*, that classifies hydraulic lime into two groups based on the ratio of silica plus alumina to the percentage of lime: "feebly hydraulic" has a hydraulic index ranging from 0.10 to 0.20; and "eminently hydraulic" has a hydraulic index ranging from 0.20 to 0.40 (Eckel 1922, 173). The higher the silica and alumina content, the greater the hydraulicity. The cement industry uses the *cementation index*, which like the hydraulic index, takes into account silica and alumina content, but also includes magnesia and iron oxide contained in the lime. The results are reported in values: "feebly hydraulic" contain products whose cementation index ranges from 0.70 to 0.30, and "eminently hydraulic" contain products whose cementation index ranges from 0.70 to 1.10 (Eckel 1922, 177). Michael Wingate uses an index loosely based on set times as follows: "feebly hydraulic" setting in 15-21 days; "moderately hydraulic" setting in 5-15 days, and "eminently hydraulic" as setting in 1-4 days. (Wingate 1988, 11). Wingate does not clearly state though under what conditions set time occurs, such as under water or in open air.

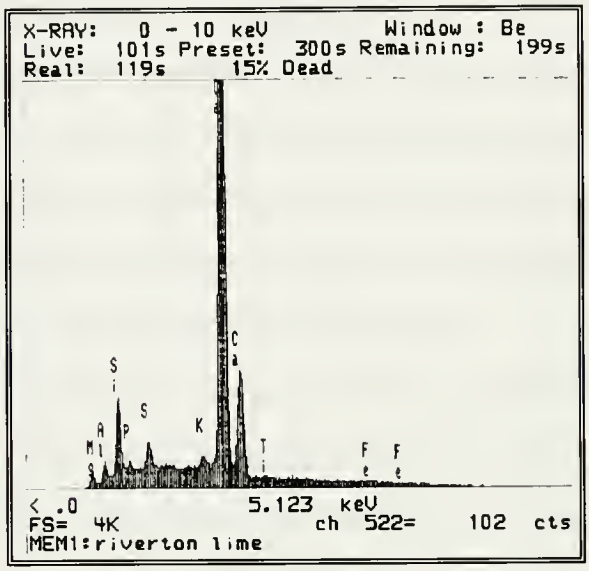

Figure 5. SEM/EDS spectrum of Riverton hydrated hydraulic lime (from Matero 1995, 98)

Natural hydraulic limes are produced from argillaceous (clay-rich) limestone containing silica, alumina, and sesquioxide of iron, as well as calcium and magnesium carbonates. They are distinct from non-hydraulic lime in their high percentage of calcium silicates, which give them their ability to set and harden in the presence of water, even under water, and to resist the washing action of water indefinitely once hardened (Mora 1984, 47). The chemical reaction of hydraulic lime is as follows: as temperatures reach 1000°C the lime reacts with clay rich in silica and alumina to form pozzolanic compounds, dicalcium silicate, monocalcium aluminate and calcium oxide. The product is treated with enough water to convert calcium oxide into calcium hydroxide, leaving dicalcium silicate and monocalcium aluminate to be anhydrous. When the resulting hydraulic lime is mixed with water, dicalcium silicate and monocalcium aluminate can

set harden in the presence of water (Collepardi 1990, 83).

The degree of hydraulic activity and the strength of the cementing agent is generally related to the proportion of silica, alumina, and lime in the raw material, and the manner in which they are combined (Eckel 1922, 173). Naturally hydraulic limes vary in their degree of hydraulicity and can even vary considerably from batch to batch. Variations are caused by impurities in the limestone and also from firing temperature and conditions of manufacture (The British Quarrying and Slag Federation Ltd. *Lime* In *Building*, 7).

Chemically, hydraulic limes are broadly classified as intermediate between hydrated lime and Portland, or natural cement (Boynton 1966, 311). The performance of hydraulic lime differs from Type S hydrated lime in that it has less plasticity (Boynton 1980, 452) and lower compressive strength. Hydraulic lime differs from cement in that it possesses considerable free lime, so that the product slakes in water, and it has considerably lower compressive strength and slower set time (Boynton 1980, 454).

Riverton hydrated hydraulic lime was selected for use as a possible binder based on its physico-chemical compatibility with historic Fort Union lime plaster, and for its properties of low shrinkage and moderate strength, as demonstrated by the 1991 pilot site treatments at Fort Union. A moderately low strength binder was desired to reduce stress on the historic fabric caused by differential movement. Furthermore, the hydraulicity of the lime, the ability to set and cure in the presence of water, was considered ideal for this type of outdoor application. Riverton hydrated hydraulic lime is referred to in the testing program as "HL".

3.2.1.2 Corson's Type S Miracle Lime

(Corson Lime Company, Plymouth Meeting, PA)

A hydrated dolomitic lime conforming to ASTM Standard C 207 "Standard Specification for Hydrated Lime for Masonry Purposes" for Type S Lime. It was selected for testing because of its predicted physico-chemical compatibility with the existing lime plaster, high water retentivity, and moderate strength.

Type S is used principally as a binder in mortars, stuccos, plasters and concrete. Type S hydrated lime was developed in the 1940s specifically for use in plastering (Boynton 1966, 407). The hydrated form is a dry powder obtained by hydrating quicklime with enough water to satisfy its chemical affinity, forming a hydroxide due to its chemically combined water (Boynton 1966, 193).

The lime cycle for non-hydraulic lime is as follows:

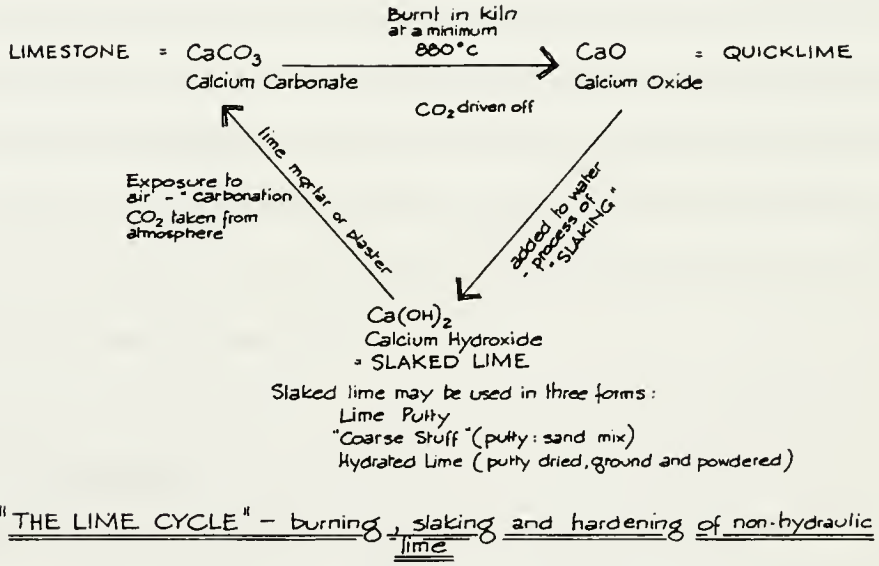

Figure 6. The lime cycle --burning, slaking, and setting of non-hydraulic lime (Ashurst 1988, 2)

Type S is an ASTM designation used to differentiate this type of hydrated lime from normal hydrated lime such as Type N, usually referred to as Mason's Hydrated Lime. Type S and Type N hydrated limes vary principally in their physical characteristics. Type S is suitable for structural purposes because it achieves high early plasticity and high water retentivity. It is more precisely milled than the Type N lime, and does not require as much soaking as Type N to achieve adequate plasticity. (Boynton 1966, 194). Both Type S and Type N can be either dolomitic or calcium lime, but they differ chemically in the percentages of unhydrated oxides--Type S has a maximum of 8% unhydrated oxide content, Type N has no specification. (Boynton 1966, 460). The Corson's Type S Miracle Lime is referred to in the testing program as "L".

3.2.1.3 Kaolin clay/Hydrite Flat D

(Dry Branch Kaolin Company, Dry Branch, Georgia)

Kaolin Clay is a hydrous aluminum silicate ($Al_2O_3 \bullet 2SiO_2 \bullet 2H_2O$) selected for testing based on its predicted physical and chemical compatibility with the clay-rich adobe substrate, low soluble salt content, and low chemical reactivity. Furthermore, it was chosen as a possible binder because of its small particle size. Clays are comprised of minute mineral particles smaller than 2μm which can easily be injected through a narrow gauge cannula, and which could theoretically penetrate the tiny interstices on the irregular surface of the adobe wall. Kaolin clay is referred to in the testing program as "C".

3.2.2 Fillers and aggregates

Fillers and aggregates are usually added to cementitious mixtures to reduce shrinkage, alter fluidity characteristics, control strength, and to reduce cost (Miltiadou n.d., 144). Two types of fillers, ceramic microspheres and fine quartz sand, were included in the grout formulas. Hereinafter, both microspheres and sand will usually be referred to as "fillers."

3.2.2.1 Ace-Crete white sand

(Ace-Crete Products, Inc., Syosset, New York)

Ace-Crete white sand is a sub-angular, white quartzitic sand that conforms to ASTM C 778-80a "Standard Specifications for Standard Sand". It was selected as a filler because of its small size and sub-angular shape, which relative to the spherical microspheres, has greater surface area for bonding. The sand has a particle size range of 100-400μm. The Ace-Crete white sand is referred to in the testing program as "S".

3.2.2.2 Zeelan Z-Light Spheres G-3500

(Zeelan Industries, St. Paul, MI)

Z-Light Spheres are hollow, inert microspheres composed of a silica-alumina ceramic alloy. With a particle size range of 10-350μm and a specific gravity of 0.65-0.75, they function as a broadly graded, lightweight filler. Their spherical shape, referred to in the product literature from Zeelan as "acting as miniature ball bearings" positively influence the workability of the mix by allowing flow without the need to greatly increase water. In addition, their light weight and wide particle size distribution give them the ability to stay well dispersed in the grout during the liquid phase. Z-Light Spheres are referred to in this testing program as "MS".

3.2.3 Organic admixtures

Additives are usually included in cementitious formulas to modify their performance. In this program, the choice of admixtures was limited to two acrylic emulsions, El Rey Superior 200 and Rhoplex E-330, added for the purpose of increasing bond strength of the cured grouts to the historic plaster and the historic adobe walls at Fort Union, and for general observation.

3.2.3.1 El Rey Superior 200

(El Rey Stucco Company, Inc. Albuquerque, N.M.)

El Rey Superior 200 is an acrylic emulsion used commercially in cementitious applications. It is an aqueous emulsion of a acrylic terpolymer methyl methacrylate, butyl acrylate and dimethylaminoethyl methacrylate[14]. It was chosen as an additive in the grout formulas to provide a "tackiness" to the grout and to the surface of the adherends, and for its purported ability to increase bond strength at the grout-plaster and grout-adobe interface[15]. This product was selected because it contains a defoaming agent essential for high velocity mixing.

Acrylic emulsions function by coalescent film formation. As the water evaporates, the discrete polymer spheres fuse into a continuous film (Lavelle 1986, 3). No chemical reaction takes place. For this reason, the samples with acrylic emulsion were not wet cured, otherwise the film would not adequately form. Once the film has formed it is not soluble in water, although it does soften and swell slightly when wet (Hartzler 1996, 16).

El Rey Superior 200 contains approximately 44±1% solids by weight in an alkaline water base. It has a pH of 9.5-10.0, a specific gravity of 1.06. Minimum film formation temperature is 10-12°C[16]. El Rey Superior 200 is referred to in this testing program as "El Rey".

3.2.3.2 Rhoplex E 330

(Rohm & Haas Company, Philadelphia, Pa.)

Rhoplex E 330 is an acrylic emulsion close in composition to El Rey Superior 200, but without a defoaming agent. It is also an aqueous dispersion of an acrylic polymer specifically designed for modifying Portland cement mixtures. It contains approximately 47% solids by

---

[14] Chemical composition provided by Charles Selwitz, Getty Conservation Institute, 1997.

[15] Charles Selwitz suggested that the nitrogenous amine group may contribute to increased adhesive properties.

[16] Material Safety Data Sheet, El Rey Superior Additive 200, Albuquerque, New Mexico

weight in an alkaline water base. It has a pH of 9.5-10.5, a specific gravity of 1.0-2.0[17]. According to Lavelle, Rhoplex E-330 increases the flexural and adhesive properties of cement, but decreases permeability. (Lavelle 1986, 18). It has a particle size ≤1.0µm.

Rhoplex E-330 was included in the testing program in only one formula, #07, the grout formula used in the 1991 Fort Union pilot plaster reattachment program.

[17] Material Safety Data Sheet, Rhoplex E-330 Emulsion (Philadelphia: Rohm and Haas, 1990).

3.3 Sample Preparation

3.3.1 Grout samples

Grout samples used in the testing program were prepared following general specifications from ASTM C 192-90a "Standard Practice for Making and Curing Concrete Specimens in the Laboratory", except in this case, the specimens were not moist cured. Moist curing, where free water is maintained on the sample surface at all times during curing, was not conducted since it would have affected the film forming capabilities of the acrylic emulsion.

3.3.1.1 Grout mixing

The fine particle-sized dry components, the lime, hydraulic lime and clay, were first passed through a No.140 sieve (passing particles <106μm) to reduce clumps, and blended together with the microspheres and sand. The dry ingredients were then mixed with water. The water to binder ratio used in the grout formulas was established by the minimum amount of water necessary to allow injection of the grout through a #12 gauge stainless steel veterinary cannula with a port diameter of ~4.0mm.

Once all the ingredients were combined, the grout formulations were mixed for 3 minutes in a Hamilton Beach high velocity (8,000 - 15,000 rpm) milk shake mixer, one minute at each of the three settings at low, medium and high. High velocity mixing is critical in achieving a high quality grout. Good workability ensures proper injectability through a syringe, with enough water retention to counter suction from porous building units and allow satisfactory hydration of the hydraulic lime. It also gives the grout compositional stability until it sets and cures. High speed mixing breaks down the clumps, allowing individual grains to be thoroughly wet and put into suspension, and also breaks down the size of the hydraulic lime particles, exposing new areas to water and activating the first phase of hydration (Houlsby 1990, 24- 25).

During the testing program it was found that the quality of the grout in the liquid state depended greatly on the type of mixer used. This was proven quite incidentally during the Phase II portion of the testing program when the milk shake mixer was unavailable, and was temporarily replaced with an ordinary kitchen blender that had much lower rpm's. It was found that the grouts mixed in the kitchen blender did not become as thixatropic, and tended to bleed, indicating that the components had segregated. When used with grout formulas that included the El Rey Superior 200 acrylic emulsion, the grouts foamed excessively in large bubbles at the surface that dispersed soon after mixing. When replaced with the high velocity milk shake mixer, the grouts that included the El Rey had tiny air bubbles that were stable and well dispersed within the grout matrix. These tiny air bubbles may create something similar to a Brownian movement effect that allow particles to stay in suspension, despite the mixture not being a true colloidal solution. When the high velocity milk shake mixer was used, it consistently produced a higher quality grout that was more thixatropic and stable.

In most cases, not enough grout could be prepared per batch to make a sufficient number of sample specimens, so multiple grout batches were made. Consistency and quality control between batches was maintained by standardizing mixing times and speeds, and by maintaining consistent water temperature and curing conditions when possible. By Phase II of the grout formula testing program, Marsh flow cone rates and specific gravity measurements by the Baroid Mud Balance were used to monitor batching.

3.3.1.2 Curing of molded grout specimens

After mixing, the grout was poured into molds specific to each test. For two of the six laboratory tests, cylindrical-shaped disks were used. The disk molds were made from rigid plastic tubing cut into rings, with an interior diameter of 69.8mm (2.75") and a height of

19.05mm (0.75"). Prior to filling, they were placed on a wax paper lined counter top and coated with a thin coat of greaseless lubricant to facilitate release of the specimens. The grout mixture was slowly poured into the forms from a narrow mouthed funnel until it overflowed. It was allowed to sit for a short time so that large air bubbles could rise to the surface. The excess was then removed with a spatula. Approximately 15-20 molds were made per batch.

The grout specimens were cured for a minimum of 28 days. Just after pouring, the specimens were protected under a damp cloth tent for 48 hours, after which time they were left to dry in an open laboratory environment. Temperature in the laboratory fluctuated between 18.7-24.5 °C and relative humidity between 30 -70%.

3.3.2 Adobe samples

For comparative purposes, adobe specimens were included in the water vapor transmission, splitting tensile strength, and bond strength tests in Phases II and III. Adobe obtained from the Boneyard was used to make the test specimens. The adobe was sieved through a #16 sieve (passing particles <1.18 diameter) to remove coarse particles and then re-plasticized with water and molded into either disks described above, or into rectangular blocks (8.9 x 8.9 x 2.54cm) for the bond strength test.

4.0 Experimental Program

The laboratory testing program was designed to examine and evaluate the characteristics and performance of various grout formulations in the laboratory for use in reattaching lime plasters to earthen walls at Fort Union National Monument, and to evaluate the broader applicability of using the grouts for *in situ* conservation of plasters in exposed earthen ruins.

As outlined in the previous chapter, the principal performance criteria used to evaluate the grout formulations were:

1. ease of mixing and use
2. adequate viscosity in the liquid phase to fill voids by low pressure injection
3. minimal segregation and stability in composition until set
4. reasonable setting time to resist displacement and allow proper cure
5. minimal shrinkage between the liquid and solid states
6. low weight
7. moderate strength within the range of the historic material
8. adequate water vapor permeability to prevent moisture accumulation
9. gap filling potential with good adhesive bonding to the adherend surfaces
10. low toxicity

To evaluate grout formulas for their performance in these categories, a three phase experimental program was designed. In Phase I, 19 potential grout formulations based on several types of binders and on varying ratios of binders to fillers were tested and qualitatively assessed in their wet and semi-cured states for the critical properties of injectability, unit weight and shrinkage. Depending on the results, formulas were either accepted or rejected from testing in Phase II.

In Phase II, 6 grout formulas were assessed for initial set time, and after a curing period of over 28 days, were measured for shrinkage, weight, splitting tensile strength, and water vapor transmission rates. From those results, one formula was chosen for evaluation in Phase III, where the grout was tested for its adhesive bond strength in shear to historic lime plaster and adobe specimens.

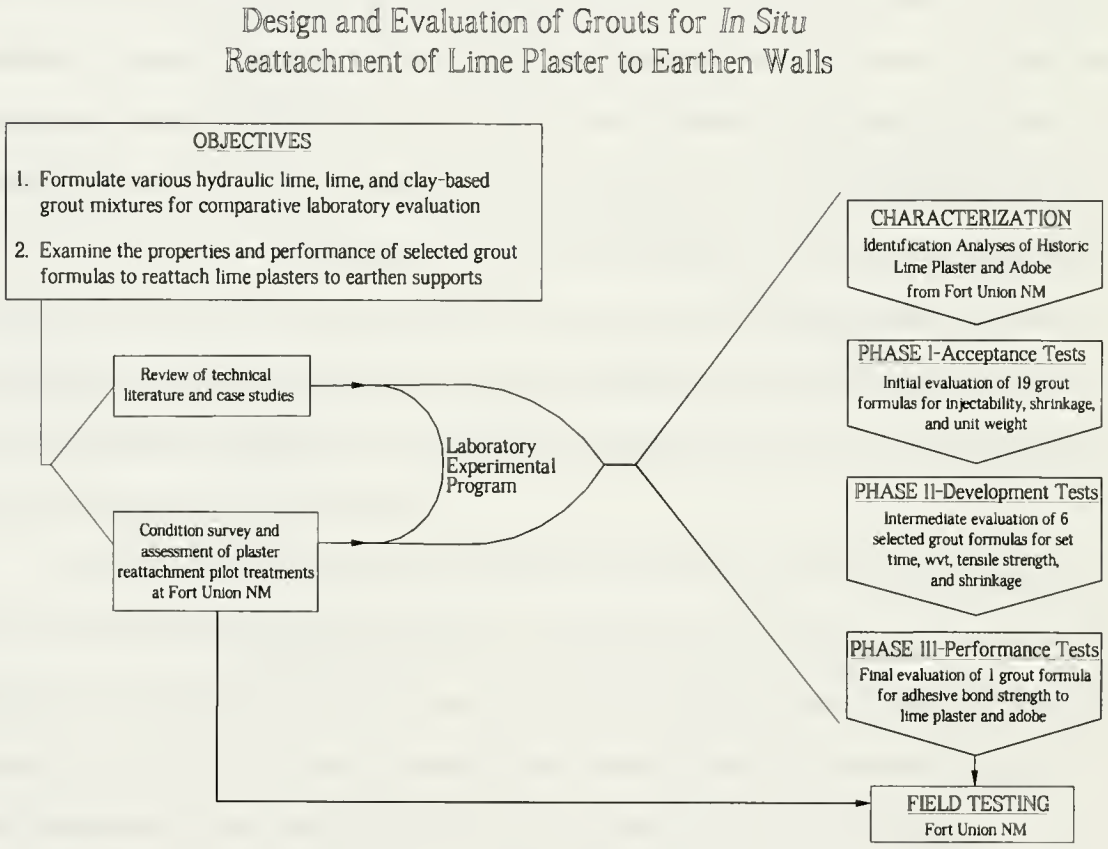

Figure 7. Experimental program activity flow chart

4.1 Phase I: Initial Evaluation

The objective of Phase I was to identify an initial group of grout mixtures that were injectable and stable in the liquid state, and lightweight, yet strong when cured. Any formula that showed segregation, shrinkage, cracking, or high weight was rejected; those that exhibited stability, low shrinkage and low weight were accepted for Phase II testing.

Nineteen formulas combining varying ratios by weight of binder, (kaolin clay, lime and hydraulic lime) filler, (ceramic microspheres and quartz sand) and water were mixed according to preparation protocols detailed in 3.3.1. After three minutes of high velocity mixing, the formulations were measured for specific gravity with a Baroid Mud Balance, poured into weighed, presoaked[18], unglazed ceramic garden saucers (2.54cm deep, 7.6cm diameter) and cured for 14 days. Ceramic saucers were used because the clay allowed for some moisture transmission. Specimens were cured for the first five days in an *ad hoc* moist curing chamber[19], and for the remaining time in the open laboratory environment having an average air temperature of 18.7-24.5 °C and a relative humidity from 30 -70%. There were three sample dishes for each of the 19 formulas, making a total of 57 samples.

After curing for one month, the specimens were visually assessed in their semi-cured state for shrinkage and cracking. Segregation, when observed in cross section, was noted, but not measured. After selecting the best of the grout formulas for testing in Phase II, their viscosity was measured with a Marsh flow cone. This first phase of testing took into consideration aspects 1-5 of the performance criteria: injectability, viscosity, segregation, shrinkage, and weight.

Only one formula included an acrylic emulsion additive, Rhoplex E-330. Though the objective in this phase was to test the performance of the materials without modifiers, this formula had been used in a 1991 field test at Fort Union and thus warranted evaluation in Phases

---

[18] The dishes were presoaked with water to reduce initial water loss from the grout.

[19] A sealed glass tank where the relative humidity on the interior was maintained at 86-94%.

I and II of the laboratory testing program.

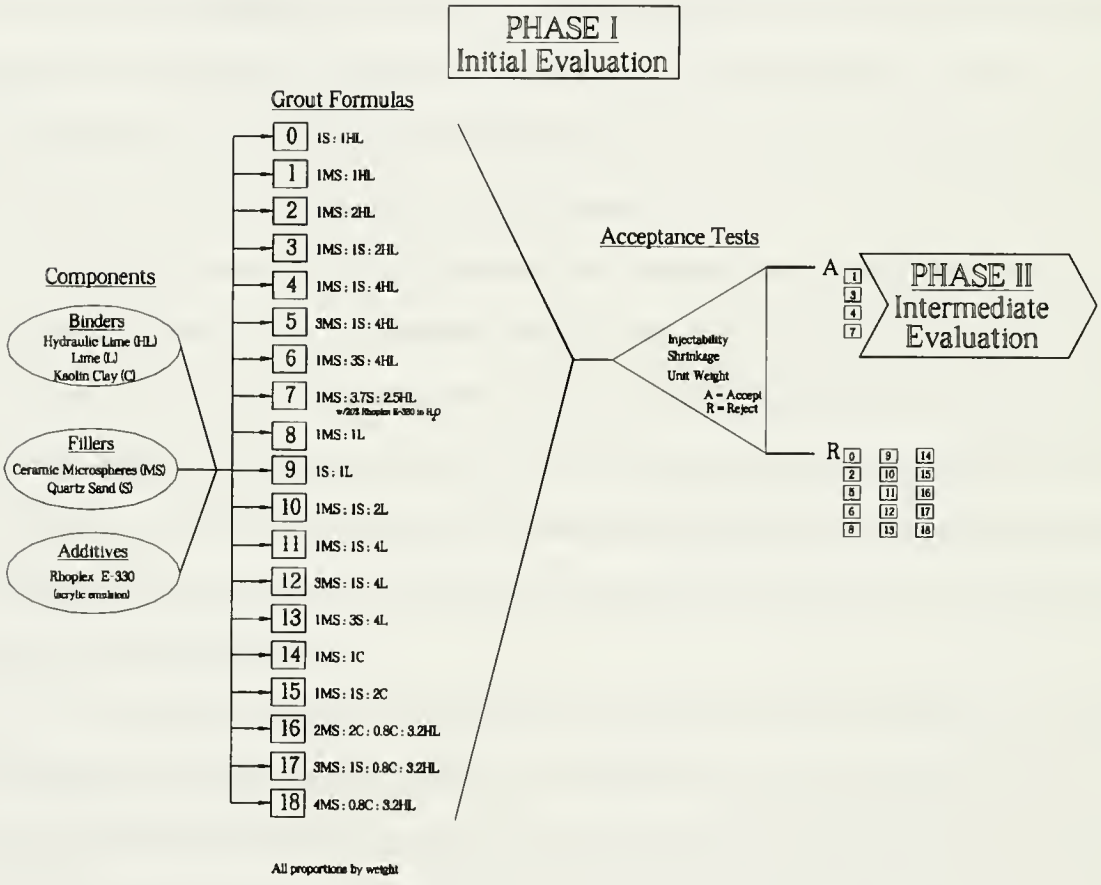

Figure 8. Phase I activity flow chart

4.1.1 Phase I testing program

4.1.1.1 Specific gravity

Specific gravity was measured to provide relative information on the unit weight of the grout mixtures. Specific gravity is defined as the "ratio of the density of a material to the density of some standard material."[20] In this case, the standard material was water at 21°C (70°F). Specific gravity is expressed by a number; since it is a ratio, it has no units (Shugar 1990, 396). In this testing program, specific gravity and Marsh flow cone values function as an index to maintain consistency and to control quality of the grout formulas.

Specific gravity of the grout mixtures was measured with a Baroid Mud Balance. The Baroid Mud Balance is a simple calibrated weighing scale commonly used in the drilling industry to measure mud density (Houlsby 1990, 95-96). The balance is sensitive to 0.01 g/cm^3; this is enough to detect even slight variations in a mix. The specific gravity of water at 21°C (70°F) was calibrated to be 1.0 on the mud balance[21]. In other words, liquids with a specific gravity less than 1 are lighter than water at 21°C, and those with a specific gravity greater than 1 are heavier than water at that temperature.

To measure specific gravity, the grout was poured into the Mud Balance cup just after mixing, and a weighted lid with a hole in it was placed on top. Excess grout was forced out through the hole until the lid was firmly seated on the rim. This assures a known amount of grout in the cup. The beam was leveled by moving the riding weight along the arm until the spirit balance indicated a horizontal level. The specific gravity reading was then read from the calibrated beam on the side of the rider.

---

[20] *McGraw-Hill Dictionary of Scientific and Technical Terms*. 1989. 4th ed. New York: McGraw-Hill, Inc. p. 1784.

[21] The density of water varies with temperature; therefore the temperature of water to which the specific gravity measurement is relative must be stated.

4.1.1.2 Shrinkage

Shrinkage was a critical factor in choosing a grout formula. For the grout to perform well as an adhesive and void filler, it must maintain its dimensional stability. Factors that influence grout shrinkage are: composition and ratio of the constituents including water; absorption of the water by binders and aggregates; reaction between the water and the lime; and temperature and humidity of the surrounding atmosphere during cure (Washa 1966, 190).

In this phase of testing, the level of acceptable or unacceptable shrinkage was determined empirically by examining the cured grout in the ceramic dishes in a semi-cured state after a fourteen day cure. Grouts that showed significant deformation, cracking, or marked lack of adhesion to the clay plates as a result of contraction were rejected.

In addition to visual assessment, the samples were weighed before and after cure in an attempt to quantify shrinkage as a function of total weight loss. Weight loss of the grouts can be attributed to loss of water by evaporation, absorption by aggregates, or reaction with the binder. Data and calculations for this are presented in Appendix A.

4.1.1.3 Segregation

In cementitious suspensions such as grouts, there is a tendency for solid particles to segregate and settle into layers depending on their size. For the cured grout to perform successfully, it must maintain a homogeneous matrix and the materials must not separate from each other. Failure can occur when the binder (hydraulic lime or hydrated lime) being finer than the filler or sand, rises to the surface with water, leaving a lime-rich surface where the binder is unbound by aggregate, and a lime-lean interior where the aggregate is unbound by lime. Furthermore, when larger particles settle to the bottom, bleeding, or the formation of a layer of water on the surface, usually occurs. Bleeding may give rise to laitence, a layer of weak, non-

durable material containing dilute calcium carbonate and fines from the aggregate (Ramachandran 1984, 16). Bleeding and rapid evaporation of surface water will leave voids, and will often result in some degree of setting shrinkage (Washa 1966 ,190).

Segregation was assessed visually by breaking the samples in half and looking at them in cross section. Formulas which showed considerable segregation were disqualified from further testing. Only formulas with a homogenous matrix were considered for inclusion in Phase II.

4.1.1.4 Viscosity

After assessment of the 19 grout formulas and selection of four grouts for testing in Phase II, the viscosity of the selected mixtures was measured with a Marsh Flow Cone following ASTM C 939-87 "Standard Test Method for Flow of Grout for Preplaced-Aggregate Concrete (Flow Cone Method), with modifications proposed by Deere (1982) and Houlsby (1990)[22]. Viscosity was measured primarily as a reference standard to maintain the consistency and quality of the grout throughout the experimental program and in the field.

Viscosity is defined by the ASCE Grouting Committee as the" internal fluid resistance of a substance which makes it resist a tendency to flow" (ASCE 1980). With a flow cone, viscosity of a fluid is indirectly measured as a rate (time required) for a known quantity of grout to flow through a graduated funnel with a standard diameter outlet. The rate is relative to the rate of water flowing through the same funnel. Though the values do not give a direct measure of viscosity, they can, if necessary, be correlated with viscometer readings to give an approximate value in centipoises. Deere claims that there is a good relationship, nearly straight-line in the

[22] The ASTM standard is designed for a US Army Corps of Engineers flow cone, rather than a Marsh Flow Cone (with an orifice diameter of 4.76mm at 50mm long). The Marsh Flow Cone was chosen over the US Army Corps of Engineers flow cone and others because the Marsh funnel has greater sensitivity and standardized procedures (Deere 1982, 287). With the Marsh Cone method, only part of the contents is discharged (Houlsby 1990, 98) as opposed to the ASTM method where the entire content of the cone is emptied.

range of interest of 35-50 seconds[for neat cement grout], between the viscosity in centipoises determined from a co-axial cylinder viscometer and the Marsh funnel viscosity (Deere 1982).

Measuring absolute viscosity of the grouts was not considered essential in this phase of testing. What was most important was that the grout be liquid enough to be injected under normal pressure through a hand-held syringe, but viscous enough keep the components suspended without segregating. If the fluidity of the grout is not appropriate, injection cannot be carried out properly, and the space between the two delaminating layers will not be completely filled. If the grout is too thick, it could accumulate near the injection point and block the passage of more material; if the grout is too thin, the components will segregate, and the solution will not cure or perform as designed.

To measure viscosity, the Marsh Flow was filled with 1,000ml[23] of grout. The time (to the nearest second) needed to pass 1 quart through the discharge orifice was recorded as the Marsh funnel viscosity value.

[23] Deere points out the of filling the funnel to the rim with 1,500ml of grout, otherwise an increase in time will be incurred (Deere 1982, 287). In this case, the cone was only filled with 1,000ml of grout because that is the maximum that could be blended at one time in the Hamilton Beach Mixer.

4.1.2 Phase I test results summary

Data

Grout Formula #	Grout Formula Ratio (weight)	% Shrinkage (weight)	Specific Gravity g/cm³	Accept/Reject (A/R)	Marsh Flow Cone 1,000ml efflux (min:sec)
0	1S : 1HL	7.2%	1.92	R	NA
1	**1MS : 1HL**	**5.4%**	**1.10**	**A**	**1:20**
2	1MS : 2HL	8.1%	1.33	R	NA
3	**1MS : 1S : 2HL**	**6.4%**	**1.49**	**A**	**1:21**
4	**1MS : 1S : 4HL**	**6.7%**	**1.65**	**A**	**1:26**
5	3MS : 1S : 4HL	5.1%	1.49	R	NA
6	1MS : 3S : 4HL	7.5%	1.88	R	NA
7	**1MS : 3.7S : 2.5HL : w/20% Rhoplex E-330 in H_20**	4.8%	**0.99**	**A**	**1:28**
8	1MS : 1L	5.0%	0.95	R	NA
9	1S : 1L	7.8%	1.87	R	NA
10	1MS : 1S : 2L	6.3%	1.52	R	NA
11	1MS : 1S : 4L	6.9%	1.41	R	NA
12	3MS : 1S : 4L	4.4%	1.18	R	NA
13	1MS : 3S : 4L	7.0%	1.68	R	NA
14	1MS : 1C	12.8%	1.05	R	NA
15	1MS : 1S : 2C	18.6%	1.15	R	NA
16	2MS : 2S : 0.8C : 3.2HL	12.5%	1.43	R	NA
17	3MS : 1S : 0.8C : 3.2HL	11.3%	1.38	R	NA
18	4MS : 0.8C : 3.2HL	10.10%	1.26	R	NA
-	Water @ 20°C±3°C	NA	1.0	NA	0:28.20

S- Quartz Sand HL- Hydrated Hydraulic Lime MS- Ceramic Microspheres L- Type S Lime C- Kaolin Clay

Table 12. Phase 1 summary test results

Phase I discussion

Most of the grout formulas exhibited some form of shrinkage cracking. Three principal cracking patterns were observed: *concentric cracking* -- long, continuous cracks on the surface and within the grout, that often spiraled out from the center; *perimeter cracking* -- where the grout contracted uniformly towards the center and detached from the rim of the clay dish; and *straight line cracks* -- narrow, short, straight cracks that formed on the surface just after initial set.

Figure 9. Phase I shrinkage test. Sample #15 with clay as the binder. Note extensive concentric cracking and shrinkage. At initial pour the grout was filled to the rim of the dish.

All five formulas that included kaolin clay, either as a single binder, or in combination with Type S or hydraulic lime, exhibited extreme shrinkage cracking and slumping. It was observed that the higher the clay content, the more severe the cracking and shrinkage. Based on these results, all formulas using kaolin clay were disqualified from further testing.

Of the 14 formulations that included Type S lime or hydraulic lime, four of the hydraulic lime samples were chosen for inclusion in Phase II. Although there was no significant difference in percentage shrinkage or unit weight between the Type S lime and the Riverton hydraulic lime, it was decided that hydraulic lime would nevertheless be better suited for full cure in the potentially damp cavity conditions between the earthen wall and plaster.

The Type S lime mixtures tended to form perimeter cracks near the rim of the dishes. In some cases, the grout had detached from the saucer and slumped toward the center. The cracking and separation at the rim was likely due to rapid drying and evaporation of water from the saucer.

The hydraulic lime mixtures tended to form thin, surficial concentric cracks that spiraled out from the center, and in most cases the grout remained firmly adhered to the sides of the ceramic dish. It was observed that the hydraulic lime mixtures tended to set faster than the Type S lime, which may have contributed to better bonding to the dish.

In consideration of the types and ratios of fillers, the grout formulas with a high sand to microsphere ratio did not perform as well as the formulas with more microspheres. The high sand grouts had higher unit weight and tended to bleed just after mixing. Examination of the cured samples #06, #07, #13, and #14 in cross section showed that the coarse fraction had segregated and settled to the bottom of the dishes. Grout formulas with a higher microspheres to sand ratio, or with microspheres alone, exhibited good lubricity and a thixatropic[24] tendency in the liquid state, and little to no segregation, low shrinkage, and low weight in the cured state.

Figure 10. Phase I shrinkage test dishes after 28 day cure

Only one formula in this phase, #07, included an acrylic emulsion additive, Rhoplex E-330. Its performance in this phase was fair despite foaming of the acrylic emulsion during mixing. Concentric shrinkage cracks were seen on the surface, and the grout slumped to the center of the dish. There was also some settling of the coarse aggregate.

Based on Phase I test results, four grout formulas were selected for Phase II: #01 (1MS:1HL); #03 (1MS:1S:2HL); #04 (1MS:1S:4HL); and #07 (1MS:3.7S:2.5HL:w/20% Rhoplex E-330 in H_2O).

[24] "thixatropy" - the phenomenon that some gels can liquefy if vibrated, e.g. by shaking, and re-set on standing." (Bowen 1981, 267).

4.2 Phase II: Intermediate Evaluation

The objective of Phase II was to assess the performance of six grout formulas, four from Phase I, and two additional formulas modified with an acrylic additive, and to choose one grout formula for final evaluation in Phase III. All grout formulas in Phase II used Riverton hydrated hydraulic lime as the binder, and microspheres and sand as the filler. All ratios are expressed by weight unless specified. For weight to volume conversions for Phase II grouts, see Appendix A.

Phase II of the experimental program included tests for initial set time, percent shrinkage, weight, splitting tensile strength and water vapor transmission. These tests take into consideration aspects of numbers 4-8 of the performance criteria listed in section 4.0: set time, shrinkage, weight, strength and permeability. The variables in the formulas were the ratio of hydrated hydraulic lime to filler, the ratio of microspheres to sand, and the inclusion of an acrylic additive. In the final stages of Phase II, SEM examinations were made of four formulas.

Grout formulas were mixed according to preparation protocols detailed in 3.3.1. After preparation, quality of the grout was checked by measuring specific gravity with a Baroid Mud Balance and viscosity with a Marsh Flow Cone. Specimens were molded according to testing procedures, and then cured 28 days in the open laboratory environment, having an average air temperature between 18.7-24.5 °C and a percent relative humidity from 30 -70%.

For two of the tests, water vapor transmission and splitting tensile strength, samples of the historic adobe were included to serve as a reference standard. Samples of historic lime plaster were not included because not enough material was available.

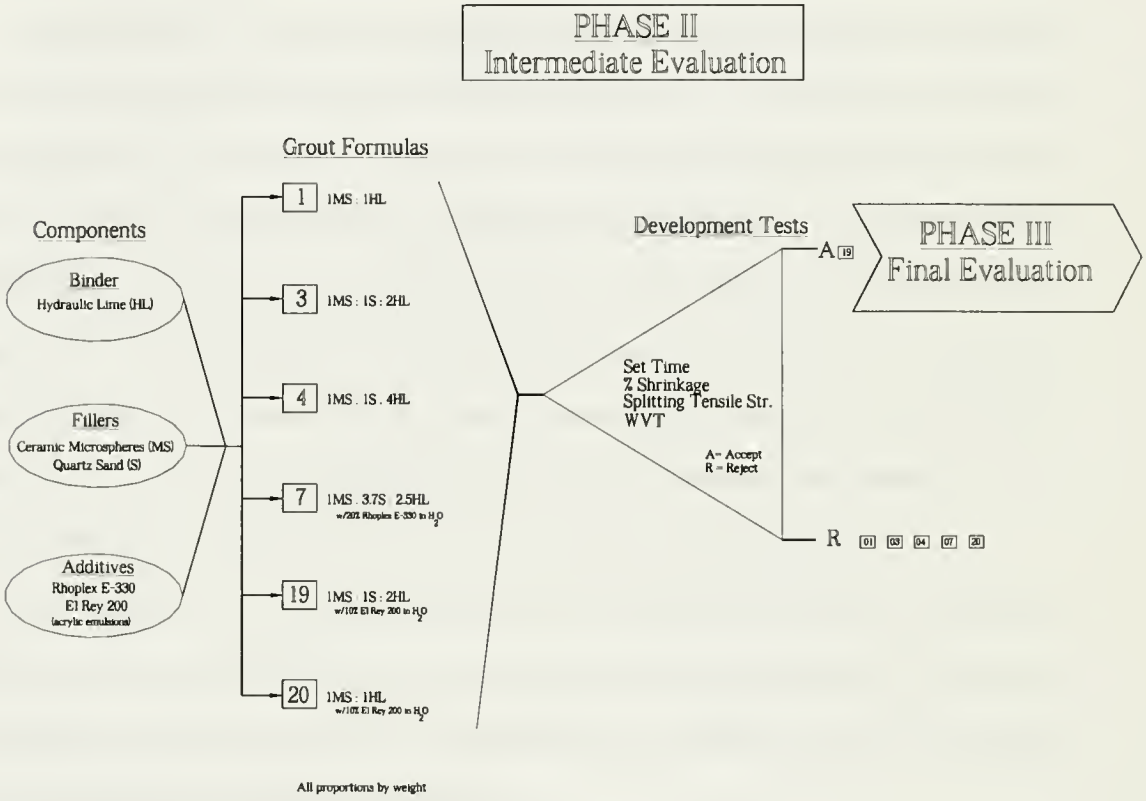

Figure 11. Phase II activity flow chart

4.2.1 Phase II testing program

4.2.1.1 Set time

Test procedures - For cementitious or lime-based grouts that undergo physical change as a result of water loss and chemical reaction with atmospheric CO_2, it is possible to evaluate and compare formulations in terms of their set time. It was important to formulate a grout that would have fast initial set and a slow final set. The advantage of a fast initial set is that the grout attains a stable physical structure with enough shear strength to resist settlement of the suspension and displacement of the loose fragment. A slow final set is necessary to allow for proper curing and formation of a stable bond between the grout and the adherends. In Phase II, initial set time was determined using a Vicat needle following ASTM C 191-77 "Standard Test Method for Time Setting of Hydraulic Cement by Vicat Needle." Final set time was not measured[25].

By the Vicat method, a known volume of grout is poured into a mold and subjected to indentation of a 1mm diameter needle over time. Initial set time was determined as the moment when the needle penetrated the grout to a maximum depth of 25mm. Each of the six grout formulas was measured for initial set time only once.

The tests were conducted in the Architectural Conservation Laboratory, where ambient room temperature averaged 21±4°C, and relative humidity fluctuated between 30-70%. It has been reported that nearly all grouts set more quickly at higher temperatures (Bell 1982, 95), but no tests were conducted to verify that characteristic. Since the grout will be used in the field where temperature and humidity can vary considerably depending on the weather, set time was measured under controlled laboratory conditions in order to set a standard by which performance was considered optimal.

Test results are reported in Table 13.

---

[25] Final set time was not measured due to time limitations and because only one mold was available for the testing apparatus.

Set time test results - Data

Grout Formula #	Grout Formula Ratio (weight)	Initial Set Time (hours)
01	1MS : 1HL	29
03	1MS : 1S : 2HL	35
04	1MS : 1S : 4HL	42
07	1MS:3.7S:2.5HL w/20% Rhoplex E-330 in H_2O	40
19	MS : 1S : 2HL w/ 10% El Rey in H_2O	32
20	1MS : 1HL w/ 10% El Rey in H_2O	25
MS- Ceramic Microspheres HL- Hydrated Hydraulic Lime S- Quartz Sand Rhoplex and El Rey- acrylic emulsion		

Table 13. Initial set time results- Phase II

Set time test results - Discussion

The results of the Vicat test indicated that the ratio of binder (hydraulic lime) to filler (microspheres and/or sand) influenced set time. It was observed that when hydraulic lime content was increased, initial set time decreased. This result was unexpected, as it was assumed that formulas with a higher hydraulic lime content would produce a faster initial set. Formula #04, which had double the amount of hydraulic lime by volume of formula #03, had a 16% faster set time. Similarly, formula #03, which had a higher hydraulic lime ratio by volume than #01, had a 17% faster set time.

Both formulas #19 and #20, which included the acrylic additive, showed a slightly faster initial set time when compared to #01 and #03 without the additive. These results indicate that the acrylic emulsion does influence set time, but how, and to what extend, is not known.

ASTM standard for cements dictates that cements should conform to an initial set time of no less than 45 minutes, and a final set time of no more than 8 hours (Ramachandran 1984, 15). The ICCROM testing program defined a reasonable *in situ* set time for hydraulic grouts as not to exceed 48 hours (Ferragni et al. 1984, 110). All six of the grout formulas fall within this acceptable range.

4.2.1.2 Percent shrinkage

Test procedures - Shrinkage was a critical property for evaluating the grouts. Minimal shrinkage is essential to ensure firm grout adhesion. To quantify volumetric shrinkage from a liquid to a cured state, grout samples were measured just after mixing, and then after 28 days using a test based on ASTM C 474-89 "Standard Test Method for Joint Treatment Materials for Gypsum Board Construction." This test was chosen over other shrinkage tests because it measures the total volume shrinkage of a sample, rather than linear shrinkage only along one axis. Grouts can shrink anisotropically, where shrinkage can vary along axes in different directions. This test was also used in previous ICCROM grout research (Ferragni et al. 1983, 1984).

Following ASTM C 474-89, volumetric shrinkage was determined by calculating the difference in specific gravity between grouts in their liquid and solid states. To determine the difference, the specific gravity of a known volume of liquid grout was measured just after preparation. After curing, the specific gravity of the solid grout was calculated as the difference in weight between the solid grout in air and in mineral spirits, divided by the specific gravity of the mineral spirits[26]. The formula used to determine volume change was [(A-B)/A] 100, where A is the average wet volume, and B is the average dried volume (ASTM C 474-89).

[26] Specific gravity of mineral spirits is 0.769 @ 20°C (Gordon, et. al 1972, 21).

Specimens for the test were prepared and cured as detailed in Section 3.3.1, but instead of curing the grout in PVC molds, 25ml of grout was injected and cured in lubricated aluminum dishes. The average thickness of the cured grout patties was approximately 5-7mm. ASTM specifies that the specimens be oven-dried at 38°C until they reached a constant weight; however, to better represent actual field conditions, the specimens were allowed to dry and cure naturally for at least 28 days in ambient laboratory conditions. Three specimens from each grout formula were used, making a total of 18 specimens tested. Test results are reported in Table 14 and Figure 13 (see Appendix A for calculations).

Percent shrinkage test results - Data

Grout Formula #	Grout Formula Ratio (weight)	% Shrinkage (volume)
01	1MS : 1HL	3.63
03	1MS : 1S : 2HL	4.02
04	1MS : 1S : 4HL	8.08
07	1MS:3.7S:2.5HL w/20% Rhoplex E-330 in H_2O	4.32
19	MS : 1S : 2HL w/ 10% El Rey in H_2O	2.98
20	1MS : 1HL w/ 10% El Rey in H_2O	2.98
MS- Ceramic Microspheres HL- Hydrated Hydraulic Lime S- Quartz Sand Rhoplex and El Rey- acrylic emulsion		

Table 14. Percent shrinkage test results - Phase II

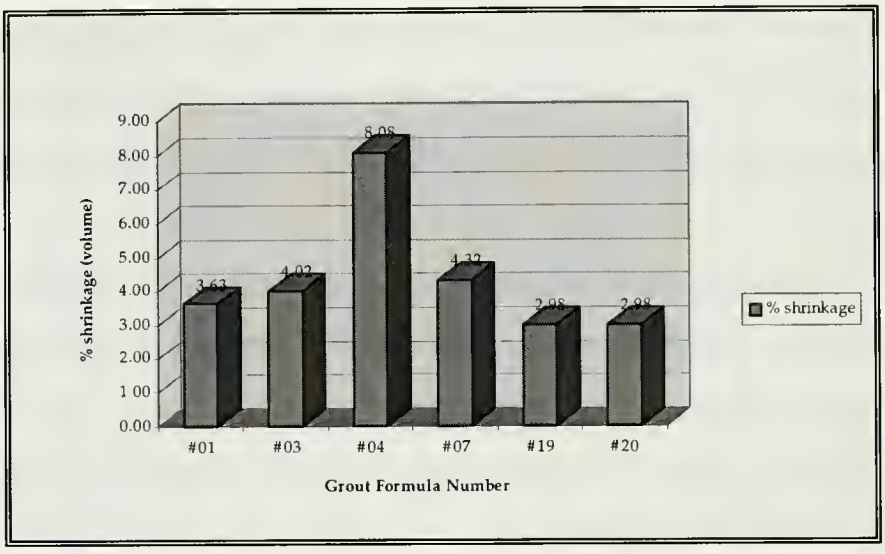

Figure 12. Graph of percent shrinkage test results

Percent shrinkage test results - Discussion

Of the six grout formulations tested, formula #04 had the highest percentage volume shrinkage at 8.08%; formulas #19 and #20 showed the least shrinkage at 2.98% each. Again, the ratio of filler to binder is a factor affecting grout performance. Test results indicated that the higher the ratio of microspheres and sand to hydraulic lime, the less the shrinkage. Similar results were obtained in Phase I shrinkage tests.

Formula #03, that had double the ratio of microspheres and sand to hydraulic lime compared to formula #04, had a 50% lower shrinkage rate than formula #04. This trend, though less extreme, is also seen when comparing formulas #01 and #03, where #01 with a higher filler ratio, had a 10% lower shrinkage rate than #03. In addition to filler to binder ratios, the types of filler also affect shrinkage. It was observed in Phase I, and confirmed in Phase II, that microspheres

alone as fillers produce grouts that shrink less than those made with a composite filler. This can be seen when comparing once again, formulas #01 and #03. Formula #01, composed of pure microspheres, had less shrinkage than formula #03, that included sand and microspheres. Also in Phase I, the pure microsphere formulas tended to shrink and crack less than similar formulas with sand.

Formula #07 had relatively high percent shrinkage at 4.32% . This may have resulted from the high sand content and segregation of the coarse particles and bleeding observed after mixing. Rapid evaporation of water on the surface can increase drying shrinkage (Washa 1996, 190).

The percent shrinkage value of formula #04 seems anomalous. Though it did have a considerably higher portion of hydraulic lime than the other formulas, the ~50% higher shrinkage value over the other formulas is extreme. Such high shrinkage was also not observed in Phase I shrinkage tests.

Formulas #19 and #20 that were amended with the acrylic additive, showed the same average percent shrinkage value, thought it was assumed that #19 would shrink a bit more because of its slightly lower filler to binder ratio. When compared to formulas #01 and #03, the same mixtures without the emulsion, formulas #19 and #20 had less shrinkage, in the range of 18-26%.

The ICCROM testing program recommended that volume shrinkage should not exceed 4.0% from a wet paste to fully cured condition (Ferragni et al. 1984, 110). Within this range formulas #01, #19, and #20 fall within acceptable limits at 3.63%, and 2.98 % (for both #19 and #20), respectively. Formulas #03, #04, and #07 exceed the acceptable limit.

4.2.1.3 Weight

Test procedures - The grout is intended to serve not only as an adhesive, but also as a void filler. Due to the large voids and wide gaps associated with plaster detachment at Fort Union, it was essential that the grout be lightweight during injection and after cure to prevent further displacement of unstable fragments. To assess relative weight, one specimen from each of the six grout formulas was weighed after the 28 day cure. Results are reported in Table 15.

Weight test results - Data

Grout Formula #	Grout Formula Ratio (weight)	Weight (g)
01	1MS : 1HL	70.0
03	1MS : 1S : 2HL	103.9
04	1MS : 1S : 4HL	107.8
07	1MS:3.7S:2.5HL w/20% Rhoplex E-330 in H_2O	38.3
19	MS : 1S : 2HL w/ 10% El Rey in H_2O	92.4
20	1MS : 1HL w/ 10% El Rey in H_2O	63.0
MS- Ceramic Microspheres HL- Hydrated Hydraulic Lime S- Quartz Sand Rhoplex and El Rey- acrylic emulsion		

Table 15. Weight test results - Phase II

Weight test results - Discussion

The results show that the weight of the grout after cure is directly proportional to the amount of sand in the formula; the higher the sand content, the higher the weight. There was also a difference in weight between the formulas that used the acrylic emulsion and those without. Formulas #19 and #20 with the acrylic emulsion were approximately 10% lighter than their

counterparts without the additive, #03 and #01. The lighter weight is probably due to air bubbles caused by foaming of the acrylic emulsion during mixing.

The result of #07 is most likely an error. The sand content was considerably higher than in the other formulas, and the result should have reflected a higher weight.

4.2.1.4 Splitting tensile strength

Test procedures - As an intermediary bonding agent between the adobe and plaster, the grout must have sufficient shear strength to withstand stress caused by differential movement of the adobe on one side, and the plaster on the other. At the same time, the grouts must also have low enough strength to fail under extreme stress without damaging the historic material. To evaluate the shear resistance of the six grouts, samples were tested using ASTM C 496-90, "Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens."

In this test, diametral compressive force is applied to each specimen from the top, and the plane on which the failure occurs is largely a response to uniform tensile stress. "It has been shown by mathematical analysis that a compressive load applied perpendicularly to the axis of a cylinder (loaded in compression on its side) in a diametral plane gives rise to a uniform tensile strength over that plane" (Wright 1955, 89).

Grouts specimens were made and cured according to procedures outlined in section 3.3.1. The specimen shape was a cylindrical disk having a diameter of 69.8mm (2.75") and a height of 19.05mm (0.75"). Three specimens from each of the six grout formulas were tested. As a reference, a set of Fort Union Boneyard adobe specimens was also included in the testing program. Historic plaster samples were not available. A total of 24 specimens were tested, 18 grout and 3 adobe.

The splitting tensile strength test was performed in the Materials Testing Laboratory at the University of Pennsylvania using a Instron Testing Machine 1331 with a Tinus Olsen Select Range Indicator. The machine has a universal type load that can be applied with a constant but adjustable rate of cross head movement. To conduct the test, a cylindrical disk of grout was set with its axis horizontal between the platens of the testing machine, balanced on the lower platen by wooden wedges approximately 1x⅛" in size. Force was applied to the specimen from above at a load rate of 1 inch per minute. The force required to fracture the specimen was measured and recorded, and then a mathematical determination of indirect tensile strength was calculated using the following formula:

Figure 13. Instron Testing Machine 1331 with a Tinus Olsen Select Range Indicator for testing splitting tensile strength. Note the grout disk loaded in the press, and broken grout samples on the table in the foreground

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

T = splitting tensile strength
P = maximum load applied indicated by the testing machine
l = length, in. (m) and
d = diameter, in. (m)

The formula used for calculating tensile strength is derived from mathematical analysis that assumes the grout specimens obey Hooke's law where strain is directly proportional to stress. However, Hooke's law does not hold true for cementitious materials. According to Wright, "the ratio of increase in stress to increase in strain decreases with rising stress and falls rapidly as the material approaches failure." (Wright 1955, 94). This fact tends to increase the load required

to cause failure in the specimen; therefore, the calculated splitting tensile strength value may be slightly higher than the true axial tensile strength.

Figure 14. Grout samples after failure from compressive loading

Splitting tensile strength test results - Data.

Grout Formula #	Grout Formula Ratio (weight)	Splitting Tensile Strength (psi)
01	1MS : 1HL	19.24 ±0.50
03	1MS : 1S : 2HL	26.86 ±3.07
04	1MS : 1S : 4HL	50.33 ±1.91
07	1MS:3.7S:2.5HL w/20% Rhoplex E-330 in H_2O	30.90 ±4.19
19	MS : 1S : 2HL w/ 10% EL REY in H_2O	31.32 ±1.03
20	1MS : 1HL w/ 10% EL REY in H_2O	25.93 ±1.88
NA	Fort Union Adobe (Boneyard)	60.0 ±1.70
MS- Ceramic Microspheres HL- Hydrated Hydraulic Lime S- Quartz Sand Rhoplex and El Rey- acrylic emulsion		

Table 16. Splitting tensile strength test results - Phase II

See Appendix A for calculations

Splitting tensile strength test results - Discussion

For the six grout formulas tested, the splitting tensile strength, expressed in psi (pounds per square inch), was less than the adobe at 60.0 ±1.70 psi[27]. Based on this information, it is safe to assume that all of theses grouts could be used at Fort Union without danger of causing mechanical damage to the adobe.

The results of splitting tensile strength of the grouts in order from strongest to weakest are: #01, #19, #07, #03, #20, #01. The ratio of binder to filler, and the type of filler and aggregates influenced the grout's strength. The test results show that an increase in hydraulic lime content, resulted in a higher splitting tensile strength. Similarly, an increase in sand content, also resulted in higher strength. Formula #04, with the highest binder content and a relatively high sand content, was the strongest grout at 50.33 ±1.91 psi, almost two times greater than formula #03 with half the amount of hydraulic lime. Conversely, formula #01, with the lowest binder ratio and only microspheres, and no sand, was the weakest grout at 19.24 ±0.50 psi. The reason the higher hydraulic lime ratio formulas have a higher strength is obvious, there is simply more of the binding media present. As for the influence of the filler type, the sand may impart strength to the grouts by nature of the grain's angular shape, which creates friction and resists movement. Microspheres alone produce weaker grouts because of their spherical shape and because their wide particle size distribution allows for tight packing of the spheres with less space available for binder.

When comparing formulas with and without the acrylic additive, #19 and #20 with the acrylic, showed a 16-26% increase in strength when compared to formulas #03 and #01 without it. The acrylic may increase strength by forming polymer lattices that bind particles and bridge gaps between microcracks.

---

[27] The tensile strength of the laboratory adobe samples may be slightly higher than the historic adobe at Fort Union. The laboratory samples were replastisized and molded into dense, compact samples.

During testing, it was observed that when compressive force was applied to the specimens, many of the them compressed and displaced considerably before they cracked or failed. Though displacement was not measured, this characteristic indicated that the grouts are somewhat flexible and could withstand certain stress before failure.

Though the results of this test were useful for comparative purposes within this study, the results are not highly reproducible. According to the literature (Wright 1955; Kesler 1966) this test is extremely sensitive to all aspects of specimen preparation and testing procedures, and many factors can interfere, or bias the results (such as irregularities in sample size and shape, variation caused during sample preparation, and uneven stress distribution under load due to imperfectly placed disks or variability in the loading rate). Due to its high bias and low reproducibility, this splitting tensile strength test was not considered satisfactory and should be replaced with a more practical and reliable test.

4.2.1.5 Water vapor transmission

Test procedures - As an interface between the lime plaster and the adobe support, the grout must allow for the transmission of water vapor through to either side. Deterioration mechanisms owing to condensation of trapped water vapor within masonry systems are a well-known phenomena. Obstruction of water vapor could not only compromise the adhesive properties of the grout, but it could exacerbate plaster detachment by causing moisture build up at the grout-adobe interface.

To determine rates of water vapor transmission, the fully cured grout samples and historic adobe specimens were subjected to ASTM E 96-80 "Standard Test Methods for Water Vapor Transmission of Materials" using the Water Method. This test was chosen because it had been shown in prior laboratory experiments (Jacob 1989; Beas 1991; Brackin 1994; Hartzler 1996) to be

well suited for measuring WVT rates of porous materials such as mortars, stone and adobe. The principal objective of the test was to compare the water vapor permeability of the grout samples to the adobe, and to determine to what extent inclusion of acrylic modifiers affected that rate.

Grout specimens were prepared and cured according to standards outlined in section 3.3.1. The grout and adobe specimens were molded in the same fashion as those used for the splitting tensile strength test. The sample disks, both grout and adobe, had an interior diameter of 69.8mm (2.75") and a height of 19.05mm (0.75"). Four samples of each grout and the Boneyard adobe were used in the test, making a total of 28 specimens: 24 grout and 4 adobes.

Using the standard water method, a grout or adobe specimen was sealed with paraffin over a beaker of distilled water and placed in a sealed, climate and humidity controlled glass chamber[28]. Three dish assemblies were made for each six grout types. One "dummy" dish assembly, made by sealing a sample over a dish without water, was made for each specimen type and served as a control. The dish assemblies were weighed daily on an electronic scale with a sensitivity of 0.01g and recorded and corrected using the dummy assemblies. After the dummy disk assemblies reached equilibrium (10 days), the test formally began and continued for 20 days.

Figure 15. Water vapor transmission testing chamber with grout and adobe samples under testing

[28] Temperature was maintained at 22°C±4°C and an RH of 47±5%. RH was controlled by filling the bottom of the glass chamber with an anhydrous calcium sulfate desiccant (Drierite) that was changed as needed (on the average of every 3-4 days). Temperature and humidity values were measured daily.

The change in weight of the dish assemblies resulting from passage of vapor through the specimen and into the atmosphere was measured to determine the rate of water vapor transmission. The greater the weight loss, the more water had passed through the sample and the greater the permeability of the specimen.

Results are reported in Table 17 and Figure 16. Calculations and graph of daily weight loss are presented in Appendix A.

Water vapor transmission test results - Data

Grout Formula #	Grout Formula Ratio (weight)	Water Vapor Transmission Rate ($g/h \cdot m^2$)
01	1MS : 1HL	8.31 ±0.03
03	1MS : 1S : 2HL	9.43 ±0.02
04	1MS : 1S : 4HL	8.52 ±0.74
07	1MS:2.7S:2.5HL w/20% Rhoplex E-330 in H_2O	13.69 ±0.02
19	MS : 1S : 2HL w/ 10% EL REY in H_2O	7.00 ±0.01
20	1MS : 1HL w/ 10% EL REY in H_2O	6.21 ±0.02
NA	Fort Union Adobe (Boneyard)	6.10 ±0.02
MS- Ceramic Microspheres HL- Hydrated Hydraulic Lime S- Quartz Sand Rhoplex and El Rey- acrylic emulsion		

Table 17. Water vapor transmission rate test results - Phase II

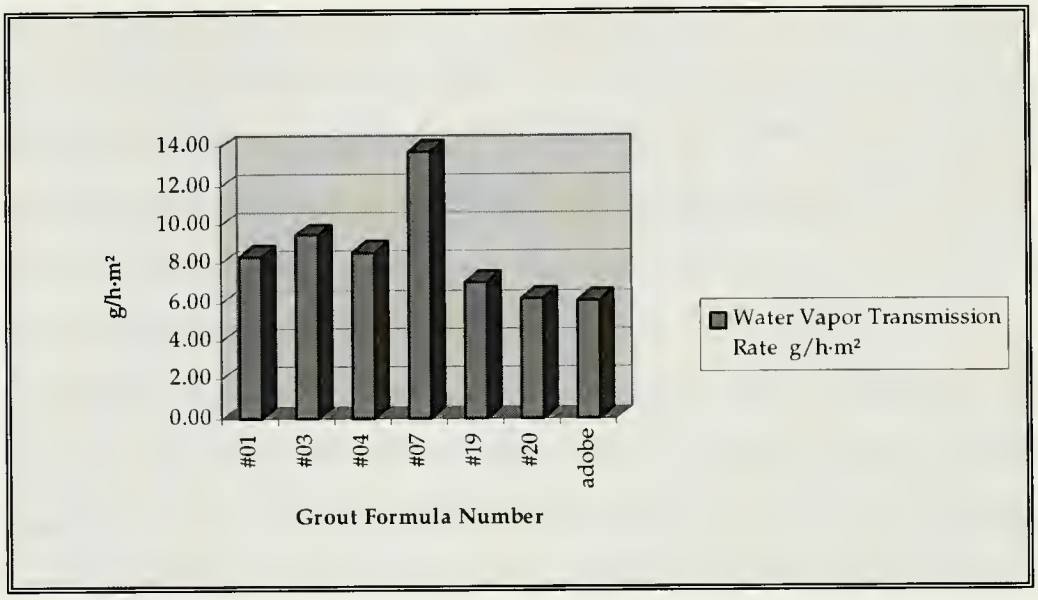

Figure 16. Graph of relative water vapor transmission rates

Water vapor transmission test results - Discussion

The results of the water vapor transmission test are presented in terms of g/h·m² , as a steady rate of water vapor flow in unit time though a unit area.

The WVT test results revealed the adobe to be the least permeable of all the specimens tested, at 6.10 ±0.02 g/h·m². As for the grouts relative to each other, the order from most to least permeable was as follows: #07 #,03, #04, #01, #19, #20. These results are interesting. The critical variable in this test was the addition of acrylic emulsion in three of the samples, which was expected to decrease their water vapor transmission rates. This proved true for formulas #19 and #20 that included a 10% aqueous solution of El Rey 200, and which showed a moderate decrease in permeability, approximately 34% for each, when compared to the same formulas without the additive, #03 and #01. Formula #07 though, which included the highest percent solution of acrylic emulsion, 20% Rhoplex E-330, had the highest rate of water vapor transmission or highest

permeability of all the grout samples. The high vapor transmission rate is likely due to the profusion of large air bubbles that resulted from the acrylic foaming during mixing. Formulas #19 and #20 also foamed during preparation and also have visible air bubbles, but they are far smaller in size and less concentrated than in the Rhoplex modified grout. It can be assumed that without the air bubbles caused by high velocity mixing, the amended grouts would have less permeability and an even lower water vapor transmission rate.

Based on the test results, it is difficult to make any assumptions on how binder to filler ratio or filler type affected permeability. There was only a slight variation in water vapor transmission rate between formulas #01, #03, and #04. Formula #04, which had a the highest hydraulic lime content, had a 10-13% higher WVT rate than the other two unamended grouts.

4.2.1.6 Scanning electron microscopy

Examination procedures

Following Phase II testing, four grout formulas, #01, 03, #19, and #20, were examined under high magnification at 20x, 150x, and 4,000x with a Scanning Electron Microscope (SEM). The SEM was ideal for observing grout's microstructure, in particular, the physical effects of the acrylic admixture. Appendix A contains most of the photomicrographs taken during the examination.

The four grout samples were examined on a JEOL 6400 Scanning Electron Microscope at the Laboratory for Research on the Structure of Matter, University of Pennsylvania[29]. The primary beam power was set at 1.0KV for the 150x magnifications and at 3.0KV for the 20x and the 4,000x magnification. With an SEM, an electron beam of high-energy electrons is focused on a sample, and the beam scans across it in parallel lines and interacts with the sample in what are called inelastic

[29] SEM operated by Xue Qin of the Laboratory for Research on the Structure of Matter, University of Pennsylvania. This specific research was funded by the National Science Foundation MRL Program under grant #DMR91-20668.

and elastic events. Inelastic events or scattering occurs when the beam transfers electrons to the specimen, generating a scanning electron photomicrograph and illustrating an secondary electron image of the sample. This image is similar to that given by a reflected light microscope, but at a considerably greater magnification. and with greater depth of field (Newman n.d., 6). The inelastic event also generates an energy dispersive X-ray spectrum which gives information on the elemental composition of the sample. EDS (energy dispersive spectrometry) was used to analyze the Riverton Hydrated Hydraulic Lime (Matero and Bass 1995, 98). See Appendix B for SEM photomicrographs.

SEM/EDS examination results

At magnifications of 20x and 150x the SEM photomicrographs clearly show the structure of the cured grouts to be homogeneous and uniform. One can clearly see the ceramic microspheres, and in some cases sand, tightly packed together and incorporated in the hydraulic lime matrix. The microspheres appeared to be broadly and well dispersed throughout the sample, with no obvious differential settlement of the heavier particles. For the most part, the individual microspheres and sand grains were thoroughly coated and surrounded by the hydraulic lime binder.

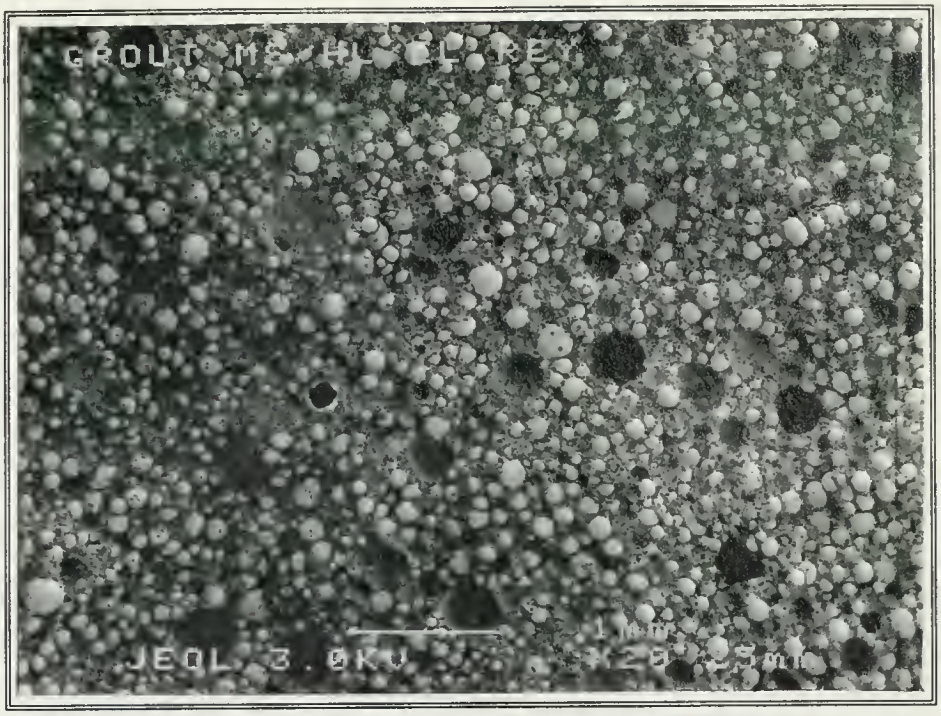

Figure 17. SEM photomicrograph of grout formula #20, showing a well dispersed matrix of ceramic microspheres and hydraulic lime. The vacuoles are a result of the El Rey acrylic additive foaming during mixing. White scale bar length is 1mm. (20x)

It was apparent that formulas #19 and #20, which included the acrylic emulsion additive, had a high percentage of entrained air visible as discreet vacuoles or pores. These small air bubbles dispersed throughout the hardened grout were produced in moderation by foaming of the acrylic during high speed mixing. There are advantages and disadvantages to these air bubbles. The advantages are that small air bubbles make the grout lighter by reducing the unit weight, increasing moisture transmission through the network of open pores, and may improve freeze-thaw capabilities by providing space for water to migrate and for ice crystals to grow without imposing stress. The disadvantage of air bubbles is that the resulting voids reduce the surface area available for bonding, and decrease the density, which can affect tensile and compressive strength.

At 4,000x magnification the hydraulic lime paste can be seen as a film on the microspheres. At this magnification, examination of samples #19 and #20 with the El Rey acrylic emulsion revealed a lattice of acicular needle-like forms extending from the surface of the particles. At first, it was assumed that these formations might be stringers of acrylic emulsion, but after discussion with Getty Conservation Institute scientists Carlos Navarro and Eric Hansen, it was decided that the needles are not amorphous strands of acrylic, but are instead crystalline, possibly formed as a result of the acrylic additive.

Figure 18. SEM photomicrograph of cured grout formula #20. Notice the acicular needles projecting from the surface of the hydraulic lime particles and the ceramic microsphere on the right. White scale bar measures 1 micron

Identification of the crystalline material, if it is indeed crystalline, was not conducted, but could be by using X-ray Diffraction. To answer how, and to what extent the addition of the acrylic additive affected crystal growth requires more detailed study. Many variables can alter crystal growth mechanisms in cements (extent of hydration, age, curing conditions, water to binder ratio (Lewin 1982, 121) and impurities in the mix[30]. It is assumed that the acrylic additive affected the mechanical rather than chemical character of the grout, since the acrylic sets by coalescent film formation, rather than by chemical reaction.

Visible evidence of the acrylic emulsion as stringers was not detected in the SEM photomicrographs. Bob Hartzler, who examined acrylic emulsion in adobe samples, found that a magnification of 5000x was best for detecting the characteristic polymeric stringers or the coating on solid particles (Hartzler 1996, 80). The highest magnification used in this SEM investigation was 4,000x.

[30] The presence of small amounts of impurities in solutions often cause marked changes in the shape of the crystal and its growth habit (Cabrera et al. 1958, 405).

4.2.2 Phase II test results summary

Data

Grout Formula #	Grout Formula Ratio (weight)*	Initial Set Time (hours)	Percent Shrinkage (volume)	Weight (g)	Splitting Tensile Strength (psi)	Water Vapor Transmission Rate ($g/m^{-2} \cdot h^{-}$)	Sp. Gravity (g/cm^3)	Marsh Cone 1,000ml (min:sec)
01	1MS : 1HL	29	3.73	70.0	19.24 ±0.50	8.31 ±0.03	1.10	1:20
03	1MS : 1S : 2HL	35	4.02	103.9	26.86 ±3.07	9.43 ±0.02	1.49	1:21
04	1MS : 1S : 4HL	42	8.08	107.8	50.33 ±1.91	8.52 ±0.74	1.65	1:26
07	1MS: 3.7S: 2.5HL: w/20% Rhoplex E-330 in H_2O	40	4.32	38.3	30.90 ±4.19	13.69 ±0.02	0.99	1:28
19	1MS : 1S : 2HL: w/10% El Rey in H_2O	32	2.98	92.4	31.32 ±1.03	7.00 ±0.01	1.33	1:23
20	1MS : 1HL w/10% El Rey in H_2O	25	2.98	63.0	25.93 ±1.88	6.21 ±0.02	1.02	1:19
NA	Fort Union Adobe (Boneyard)	NA	NA	NA	60.0 ±1.70	6.10 ±0.02	NA	NA

MS- Ceramic Microspheres HL- Hydrated Hydraulic Lime S- Quartz Sand Rhoplex and El Rey- acrylic emulsion

Table 18. Phase II summary test results

Phase II test results summary - Discussion

In Phase II, six grout formulas, composed of varying ratios of hydraulic lime, ceramic microspheres and sand, three of which were amended with an acrylic emulsion, were compared to each other based on their initial set time, volume shrinkage, weight, splitting tensile strength and water vapor transmission rate. Specific gravity and Marsh Flow Cone readings were taken periodically during mixing to monitor and maintain the grout's quality.

* See Appendix A for weight to volume conversions for the grout formulas

Results from Phase II tests revealed that variation in the ratio of the hydraulic lime to filler, and to a lesser extent, the ratio of microspheres to sand, affected the properties and performance of the grouts. Generally, the higher the ratio of hydraulic lime to filler, the higher the percent of volume shrinkage, and the higher the tensile strength, though the differences were slight. Formula #04, which had double the amount of hydraulic lime of formula #03, did have the highest tensile strength within acceptable limits, but it also exceeded the acceptable percent shrinkage level, which caused it to be rejected from Phase III testing.

In terms of fillers, the ratio of microspheres to sand made a difference in the cured grout's splitting tensile strength and weight. Using quartz sand as an aggregate resulted in grouts with higher splitting tensile strength, but at the expense of a having a considerably higher weight, as seen when comparing formulas #01 and #03. The formula with the highest sand content, #07 should have had the highest weight, but the value does not reflect this and is probably in error. When using microspheres alone, as seen in formulas #01 and #20, the grouts had a significantly lighter weight and slightly less shrinkage, but also lower tensile strength than the formulas with sand. Based on these results it was determined that a mix composed of a filler of primarily microspheres and a small amount of sand produced a stronger, lightweight grout.

Adding the acrylic emulsion to formulas #07, #19, and #20, affected the performance properties of the grouts, though not significantly. The formulas with the El Rey 200 additive, #19 and #20, had a slightly lower rate of water vapor transmission, and a modest increase in splitting tensile strength relative to formulas #01 and #03 without the additive. Between the two types of acrylic emulsion used, the El Rey performed best in the liquid state by causing a modest foaming effect during mixing. This foaming effect influenced the weight and workability of the grouts, causing them to be lighter and more thixatropic. Formula #07, which included the Rhoplex E 330 emulsion, foamed substantially during mixing, producing a cured grout full of large vacuoles. As a result, this formula also had the highest water vapor transmission rate.

Scanning Electron Microscopy of grout formulations, #01, 03, #19, and #20, revealed that all four mixtures had a well blended, homogeneous matrix, where individual solid particles were well coated by the hydraulic lime binder. Formulas #19 and #20 that included the El Rey 200 additive had a modest percentage of entrained air visible as discreet vacuoles, and a lattice of acicular needle-like forms extending from the surface of the larger particles, which could be crystalline. The crystalline microstructure as well as the acrylic emulsion polymer lattices (not visible in the SEM) may have resulted in increased splitting tensile strength of formulas #19 and #20. The air-entraining effect imparts additional light weight to the cured grout and may be advantageous in increasing the grout's freeze-thaw capabilities.

Based on the results of 6 grout formulations tested in Phase II, formula #19 was selected as the optimal grout for Phase III testing and for possible use in the field to readhere detached plaster. Compared with the other five formulations tested, the selected mixture exhibited the best combination of properties satisfying most of the performance criteria of ease of injectability without excessive water; adequate viscosity to fill gaps; minimal segregation in the liquid state; reasonable initial set time, low shrinkage, moderate tensile strength (one half that of the adobe), and a fair water vapor transmission rate relative to the other formulations and the adobe.

Formula #19 consisted of (parts by weight):

1 part microspheres
1 part fine quartz sand
2 parts hydraulic lime
4.0 parts (by volume) of a 10% solution of El Rey Superior 200 in water

parts by volume = 3.7MS : 1S : 3.9HL : 4.0 parts 10% solution of El Rey Superior 200 in water

4.3 Phase III: Final Evaluation

The objective of Phase III was to assess the adhesive capability of the selected grout in the laboratory under simulated conditions of use. The grout formula, MS : 1S : 2HL (by weight) with 10% El Rey in H_2O(v/v), was injected between historic Fort Union lime plaster and adobe samples and allowed to cure. Despite problems with the adobe-plaster half of the assemblies, the grout-plaster half was tested for adhesive bond strength in shear by compressive loading.

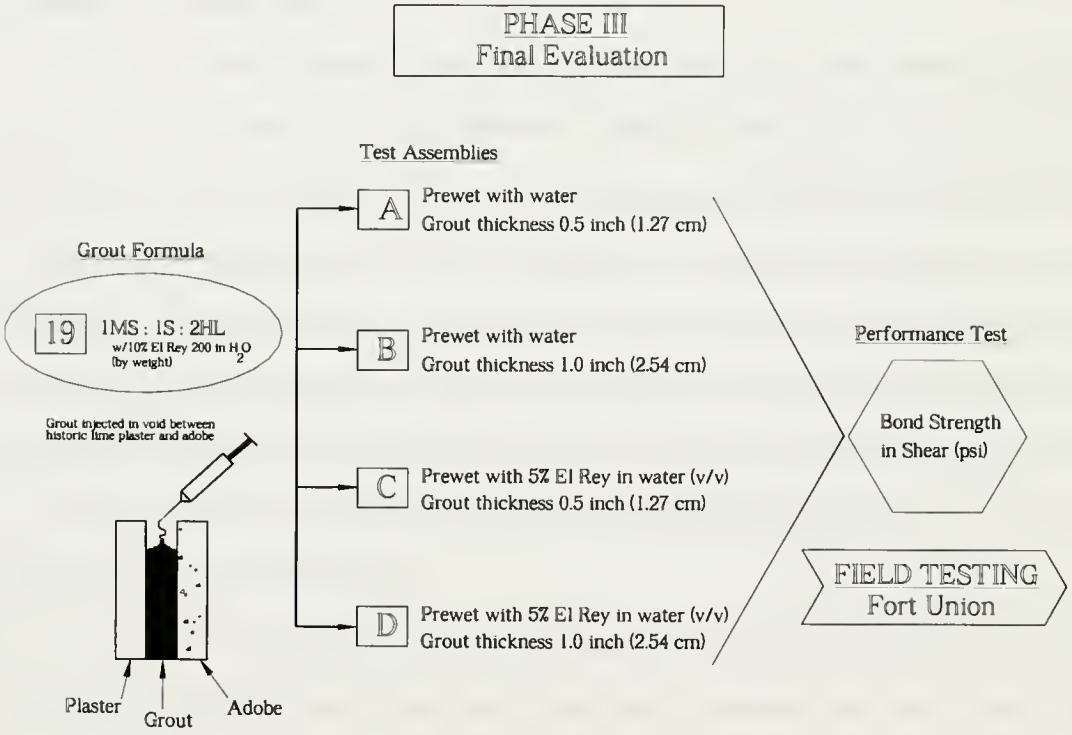

Figure 19. Phase III activity flow chart

4.3.1 Phase III testing program

4.3.1.1 Bond strength in shear

Test procedures - The adhesive bond strength of the grout to the historic lime plaster and the adobe from Fort Union was intended to be measured using ASTM D 905-89 "Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compressive Loading." The test was to be applied to assemblies, where the grout filled a measured void between the historic plaster and adobe specimens. Unfortunately, failure of the adobe-grout portion of the assemblies occurred prior to any mechanical testing; therefore, bond strength in shear was conducted only on the plaster-grout portion of the assemblages.

The proposed variables in this test were the width of the void between the adobe and plaster specimens (simulating actual detachment conditions at Fort Union), and the type of material used to prewet the adherends. The void space represented two conditions of detachment at 1/2" (1.27cm) and 1" (2.54cm). The preinjection materials were water, and 5% El Rey 200 in water (v/v). The plaster and adobe sides of the assemblies were each 8.9 x 8.9 x 2.54cm in size. The plaster samples were historic fabric from Fort Union[31] sized with a file. The adobe specimens were made from the Fort Union Boneyard sample. The adobe was sieved through a #16 screen (passing particles < 1.18mm) to remove coarse particles, and was then replastisized, molded into wood frames, and allowed to dry at room temperature.

To make the assemblies, the interior surfaces of the adobe and plaster were wet with water or with the 5% El Rey prior to grouting. Pre-wetting the adherends was an extremely important procedure to reduce moisture loss from the grout by absorption from the porous adherends. As was proven, excessive moisture loss from the grout can cause cracking and failure at the interface, and can detrimentally affect curing of the hydraulic lime. In addition to water, acrylic emulsion was

[31] Plaster samples were fragments that had fallen to the ground from a location that could not be determined, or that were damaged and could not be replaced.

considered as a prewetting agent for its purported capability to increase the bond strength of the grout (Mora et al. 1986; Ashurst 1984; Twilley and Podany 1986; Schnabel and Boornazian 1992). After prewetting, grout formula #19 was prepared following procedures outlined in section 3.3.1, injected into the assemblies in a natural vertical position, and left to cure for 28 days. Three assemblies were made for each of the four variables, making a total of 12 assemblies.

Approximately 5-7 days after preparation, all twelve of the assemblies detached at the adobe-grout interface. The bond between the grout and the adobe entirely failed. This was likely caused by the adobe component absorbing moisture too quickly from the grout, causing it to crack and separate from the adobe.

Since only one side of the assemblies was intact, the shear test as specified by ASTM D 905 was not suitable; nonetheless, a modified version of the test was used to measure the bond strength of the grout-plaster portion. Those portions were subjected to shear by compressive loading in a Soiltest Versa-Tester AP-1000. The grout portion of the assembly was fixed in place with a vise, and load from the Versa-Tester was applied from the top to the plaster portion. Results of this test were reported in terms of the load required (psi) for failure of the plaster-grout bond and location and type of failure. Three types of failure are reported (Horie 1987, 74-75.):

1. cohesive failure in the adhesive, where the adhesive material itself fails
2. adhesion failure, where the bond between the adhesive and the object fails along the interface
3. cohesive failure in the substrate where the object may break, leaving a small portion of the surface attached to the adhesive

4.3.2 Phase III test results summary

Data

Grout Formula MS : IS : 2HL: 1/10 El Rey in H_2O	Prewetting Agent	Void Width (")	Average Compressive Strength (psi)	Location and Type of Failure
A	H_2O	0.5	186 ±24.09	Cohesive failure within the grout
B	H_2O	1	148 ±12.01	Two samples w/ cohesive failure of the grout, one sample w/adhesion failure at the grout plaster interface
C	5% El Rey in H_2O	0.5	187 ±20.40	Cohesive failure within the grout
D	5% El Rey in H_2O	1	163 ±16.07	Cohesive failure within the grout

Table 19. Bond strength in shear test results - Phase III

Phase III test results - Discussion

The results of this final phase of testing were inconclusive regarding the bond strength of the grout to the adobe due to detachment of the materials prior to testing.

Regarding the grout-plaster bond strength, all but 1 of the 12 samples fractured within the grout matrix and not at the grout-plaster interface. This indicated that the bond strength of the grout to the historic plaster is strong and compatible, and may even be stronger than the grout. The average force required to fracture the grout was 186 psi. This is very low relative to the historic plaster, which was tested as a reference, and failed under compressive load at an average of 530 psi. There was no significant difference between the types of failure displayed between the assemblies that were preinjected with water versus those preinjected with the acrylic emulsion. See Appendix A for further description of the test results.

Despite failure of the assemblies at the grout-adobe interface, the information provided by that occurrence alone was significant. It demonstrated that the method of grouting was also a variable in the grouts performance. Specifically, that prewetting adherends, especially adobe, was critical for maximizing bond strength. But, certain precautions must also be taken when prewetting adobe. Depending on the type of clay minerals present, water can cause the adobe to swell and expand. Mechanical stress caused by a swelling and shrinking action can break the adhesive bonds of the grout. It is recommended that research also be conducted into the use of either non-aqueous solutions or surfactants (surface active agents) as prewetting agents.

5.0 Experimental Program Conclusions

Prior to using the grout in the field, it was important to observe and test its behavior in the laboratory under ideal conditions. In this project, various grout formulas were analyzed and assessed for their performance in a three phase experimental program. Following is a summary of the test results.

5.1 Phase I

In Phase I, an initial group of 19 grout formulas were prepared, cured, and assessed for injectability, shrinkage and unit weight. Selection of grout components was based on characterization of the historic lime plaster and adobe from Fort Union, basic knowledge of the mechanisms of deterioration responsible for detachment, as well as from results of research on hydraulic lime grouts conducted by ICCROM from 1979-83 (Ferragni et al. 1983, 1984). The grout formulas were designed as simple combinations of Kaolin clay, Type S lime, and Riverton hydrated hydraulic lime binders, with varying ratios of Zeelan ceramic microspheres and fine white quartz sand. In this phase, an acrylic emulsion additive, Rhoplex E-330, was added to one formula because it had previously been used at Fort Union in a pilot reattachment treatment program.

Of the 19 initial grout formulas tested, four hydraulic lime based formulas were accepted for further testing in Phase II. The general results of Phase I test are summarized as follows:

- Kaolin clay, either as a single binder or in combination with Type S lime or hydraulic lime, exhibited extreme cracking and shrinking. The higher the clay content, the more severe the cracking and shrinkage. Kaolin clay as a binder was subsequently excluded from the testing program.

- Both the Type S lime and the Riverton hydrated hydraulic lime as grout binders performed well. Both had relatively low shrinkage compared to the Kaolin clay and low weight. There was concern about the ability of Type S lime to harden or carbonate in the large, possibly damp, stagnant cavity conditions that may exist between the lime plaster and adobe. For this reason, the Riverton hydrated hydraulic lime was chosen as the binder over Type S hydrated lime for the remainder of the testing program.

- In consideration of type and ratio of fillers and how they affect grout viscosity, shrinkage and weight, it was observed that microsphere alone, or in a high ratio to sand, performed better in all cases than the high sand content formulas. Sand increased unit weight and caused segregation of the coarse particles after mixing. It was also observed that high sand grouts required more water to be sufficiently fluid, and that they tended to clog when injected through the syringe. High microsphere formulas, on the other hand, had lighter unit weight, less shrinkage, and little obvious segregation of filler from the binder. Microspheres also contributed to ease of injectability by virtue of their spherical shape and act as miniature ball bearings that permit flow without the need to greatly increase water. Based on these findings, ceramic microspheres were used as the principal filler in the grout formulas. The decision was made to include low ratios of sand in some formulas for the purpose of testing its effect on the grout's strength.

5.2 Phase II

In Phase II, six grout formulas, composed of varying ratios of hydraulic lime, ceramic microspheres and sand, including three that were amended with an acrylic emulsion additive, were compared based on their initial set time, volume shrinkage, weight, splitting tensile strength and water vapor transmission rates. The results of Phase II testing are summarized as follows:

- It was observed that the properties and performance of the grouts in the cured state were largely determined by the binder, and to a lesser extent by the filler and aggregate. The test results indicated that the ratio of binder to filler influenced both shrinkage and tensile strength. Formula #04, which had the highest ratio of hydraulic lime (nearly double the amount by weight of most other formulas) had the highest tensile strength of all the samples, which still fell within an acceptable limit below the strength of the adobe, but it also had the highest percent shrinkage at 8.08%, which far exceeded the acceptable limit[32]. Primarily due to high shrinkage, formula #04 was rejected from further testing.

- Type and ratio of fillers played an important role in how the grout performed in the liquid state. It was observed in Phase I, and confirmed in Phase II, that microspheres imparted good workability and injectability to the grouts. The microspheres were well dispersed in the grout matrix and tended not to segregate from the other components. Good workability is extremely important in grouting, for if the grout is not properly injected, it is useless, and can cause damage.

  In the cured state, the inclusion of angular sand resulted in grouts with higher splitting tensile strength, but at the expense of a having a considerably higher weight. Contrariwise, formulas with a high microsphere ratio or with microspheres alone had a significantly lighter overall weight and shrinkage, but also lower tensile strength. Considering these results, it was determined that a combination of microspheres with a small amount of sand would produce a highly injectable, stable yet adequately strong lightweight grout.

[32] The ICCROM testing program recommended that volume shrinkage should not exceed 4.0% from a wet paste to fully cured condition (Ferragni et al. 1984, 110).

- The addition of acrylic emulsions in the grout formulas affected water vapor transmission and splitting tensile strength, though only slightly. Formulas with El Rey 200, #19 and #20, had a slightly lower rate of water vapor transmission, and a modest increase in splitting tensile strength relative to formulas #01 and #03 without the additive. Under SEM, it was observed that the acrylic modified grouts had a unique interlocking, needle-like microstructure that may be crystalline in nature. This phenomenon is not entirely understood and requires further investigation.

  Again, the acrylic emulsion influenced workability of the grout formulas. El Rey 200 increased the thixatropic character of the grout by producing an air entraining type of effect. Small air bubbles produced during high speed mixing increased stability of the solution and gave it a light weight. The vacuoles left by air bubbles could be clearly seen by SEM.

- Of the six grout formulas tested, one hydraulic lime-based formula composed of parts by weight 1MS : 1S : 2HL : 10% El Rey Superior 200 in water *(or parts by volume = 3.7MS : 1S : 3.9HL : 4.0 parts 10% solution of El Rey Superior 200)*, was chosen as a suitable grout for further testing in Phase III and for use in field tests to reattach lime plasters on earthen supports at Fort Union. This mixture met the essential performance criteria of injectability with low viscosity and minimal segregation, low shrinkage and weight, reasonable setting time, and adequate water vapor transmission. In a liquid state, the viscosity of the grout was high enough to be easily injected through a 12 gauge cannula, but viscous enough to stay where it was injected without dripping. The splitting tensile strength of the grout was determined to be adequate, having a cohesive strength nearly half that of the adobe sample tested.

  Using hydraulic lime as the binder offered the advantages of being a material that was chemically and physically compatible with the lime plaster, while offering the hydraulic properties necessary for successful grouting into potentially damp cavity conditions between

the earthen wall and plaster. The Riverton hydrated hydraulic lime is relatively easy to obtain and use, and offers an option to the existing European grouts employing hydraulic additions such as brick dust and fly ash.

5.3 Phase III

In Phase III, assemblies were used to test the bond strength of grout formula #19. The assemblies were made to simulate actual conditions of use, and were fabricated by injecting the grout between historic Fort Union lime plaster and adobe samples. The adherends were prewet either water or an acrylic emulsion to assess if, and to what extent, bond strength was influenced by the prewetting agent. After curing, the assemblies were to be tested for adhesive bond strength in shear by compressive loading, but the adobe-grout portion of the assemblies failed soon after preparation. Despite this setback, the test was modified to accommodate only the plaster-grout portion of the assemblies. The general results of Phase III are summarized as follows:

- It was demonstrated that when high compressive load was applied to the grout-plaster system, failure occurred in the grout portion of the assembly approximately 90% of the time. This indicates that bond strength of the grout to the historic plaster was strong, and that failure, should it occur, would likely happen in the grout. The average force required to fracture the grout was 186 psi.

- Failure of the bond between the grout and the adobe before testing was caused by the adobe absorbing water from the grout, which caused it to crack and fail. This demonstrated that prewetting adobe before grouting was critical, and the amount of aqueous prewetting agent used influences bond strength and ultimately grout performance.

5.4 Other comments

The importance of high speed mixing in grout preparation

High speed mixing is extremely important in producing a grout with excellent workability. If the fluid properties of the grout are not satisfactory, injection cannot be carried out properly, stability of the composition is at stake, and the treatment could end up a worthless fait accompli.

During the testing program it was found that the type of mixer used in the grout's preparation greatly influenced workability. When comparing grout formulas mixed in an ordinary kitchen blender to those mixed in the high velocity milk shake mixer, the high velocity mixing produced consistently higher quality grouts that were thixatropic and stable. SEM examination of four grout formulas, #01, #03, #19, and #20, all of which were prepared by high speed mixing, revealed a well dispersed, homogeneous matrix, where individual solid particles were thoroughly coated by the hydraulic lime binder. High speed mixing also resulted in the formation of numerous, diffuse, tiny air bubbles in the grouts that included the El Rey 200.

6.0 Field Testing

After completion of the experimental program, the grout was tested *in situ* to reattach fragments of historic lime plaster to adobe walls at Fort Union National Monument. The south end of the Mechanics' Corral (HS 36) was selected for treatment by the National Park Service due to the predominance of surviving plaster and the recognition that deterioration was active.

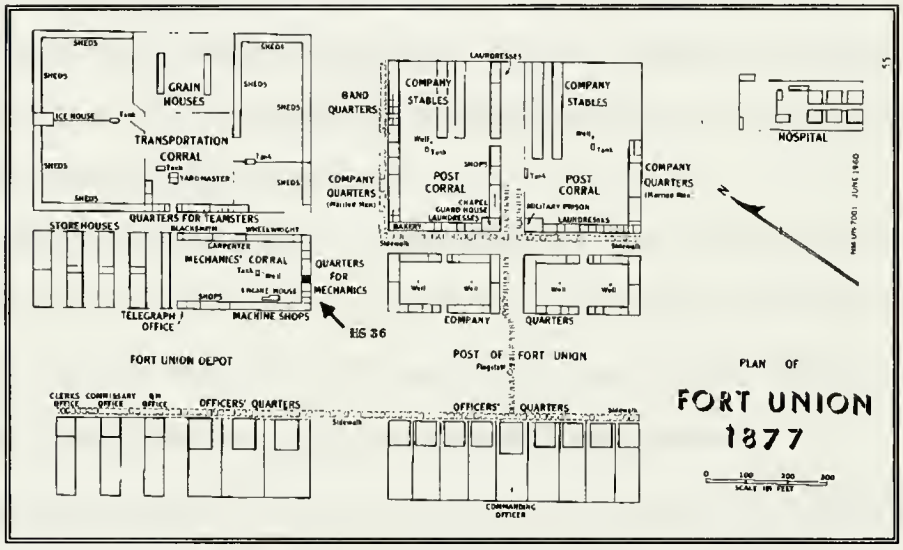

Figure 20. Plan of Fort Union from 1877. The treatment area was in HS 36, known as the Mechanics Corral.

In HS 36, an extensive plaster conservation program was undertaken from 1992-96 by the University of Pennsylvania under cooperative agreement with the National Park Service, as part of the University of Pennsylvania Conservation Field School. In addition to grouting, treatments included: documentation of the plasters before, during, and after treatment; emergency stabilization of fragile plasters with gauze and a water soluble adhesive; removal of previous cementitious repairs; edging and compensation to seal the fragments from the ingress of water; and aqueous cleaning. For further details on the Fort Union plaster conservation program, see Matero and Bass 1994. Following is a brief description of the grouting procedures.

6.1 Grouting Procedures

Most of the existing plaster fragments at Fort Union had previously been repaired by edging with both lime and cementitious mortars, keyed with iron nails, and massive wall capping of cement and wire mesh reinforcement. These edging, caps and surface fills were carefully removed by hand with small chisels and mallets to evaluate and gain access to the voids between the plaster and the adobe substrate. Debris, loose adobe, and organic matter was removed from the open and blind voids with compressed air, brushes, and small tools. Care was taken not to intensify detachment during this process.

The location of blind voids was determined by percussive sounding with small wooden mallets and designated on the surface with chalk. The majority of blind voids were located along existing cracks or holes. These were utilized as ports wherever possible. For blind voids with no access, small holes were drilled using a hand drill and a ⅛" masonry bit.

All voids were flushed with water to reduce premature drying of the grout, to clean out the voids, and to rehydrate clays in the adobe. Openings along the plaster edges, areas of surface loss, and cracks were temporarily dammed with clay or faced with tissue. Sticks were inserted at intervals along the damming for air release during grouting. These areas were then prewet by injection with a 5% El Rey 200 (acrylic emulsion) in water (v/v) to increase adhesion of the grout and to provide a gradient of compatibility between the adobe, grout and plaster.

A grout composed of parts by volume 3.9 parts hydraulic lime, 3.7 parts of ceramic microspheres, 1 part of fine sand and 4.0 parts 10% El Rey 200 in water (v/v) was used. Water was added to the dry ingredients and the mixture was blended for 3 minutes in a high velocity milk shake mixer (8,000-15,000 RPM) until it achieved a viscosity of 1:33 min:sec/1 qt. with a Marsh flow cone. The grout was then injected into voids through a 12 gauge steel cannula-tipped syringe always working from the bottom to the top. After injection, excess grout was immediately

removed from the surface and the treated area was protected from heavy rains and direct sunlight for at least the first 24 hours with polyethylene sheeting[33]. After the grout had time to set, all exposed plaster edges and surface holes and cracks were filled with a hydraulic lime mortar. This ensures the proper shedding of water off the fragment, and helps to preserve the plaster for the long-term.

6.2 Treatment Assessment

As of 1997, most of the plaster fragments in the Mechanics Corral have been reattached using the grout, and are reported to be in good condition[34]. The fragments are stable. There have been no new losses in the plasters since treatment and percussive tapping on the surface indicated that no new detachment has occurred.

Since 1993, and directly as a result of this experimental testing program, the grout has also been used to readhere lime plasters to adobe walls at Fort Davis National Historic Site in Fort Davis, Texas, and to readhere lime plaster to stone masonry walls at Mission San Jose in San Antonio, Texas, and at Mission San Juan Capistrano in San Juan Capistrano, California. A formal assessment of the grout's performance in the areas and at Fort Union is recommended.

---

[33] For optimal performance, grouting should be executed under weather conditions beneficial to proper drying and curing. Optimal temperature range for masonry work is between 40-80° F on a humid cloudy day. Grouting should not be implemented or cured in freezing temperatures, exposed to heavy rains, or left to cure too quickly by being unprotected on hot sunny days.

[34] This was based on an informal assessment by the author and from reports from Bob Hartzler and Anne Oliver, architectural conservators.

Recommendations for Future Research

To better understand the behavior of the grout and how it functions in the plaster-adobe system, more laboratory analysis and field testing is required. The following are recommendations for future research:

Analysis of individual components

1. Identification of the alkaline elements in the Riverton hydraulic lime would be interesting to determine if the grout has the potential to release damaging soluble salts or insoluble efflorescences that could cause further deterioration. Also valuable, would be research into calcium silicate hydration and lime carbonation in the hydraulic lime, and the affect those processes have on strength, durability and bond strength of the grouts.

2. Further testing and analysis of the acrylic emulsions is recommended to better understand their influence on the grouts formulas. Some questions include: Does the acrylic emulsion cause the formation of needle-like crystals seen in the SEM photomicrographs of grout formulas #19 and #20? If so, what is their crystalline composition, how are they formed, and how do they function? Does the acrylic emulsion act a protective colloid to retain water against the suction of the porous adherends? Does the acrylic emulsion increase adhesive bond strength of the grout when used as a prewetting agent?

Analysis of the properties and performance of individual grouts formulas

3. Grout strength should be re-tested with more precision and accuracy than was undertaken in this program. Both the splitting tensile strength test and the bond strength test must be reassessed and replaced with more suitable methods that offer better control and less bias.

4. The grouts should be tested for durability and weathering resistance, e.g. resistance to salt crystallization and freeze thaw.

5. Investigation into the pore structure and water absorption of the grouts is recommended to determine how water moves through the grout, if the pores distribute water and other elements to adjacent materials, and if the pores influence resistance to salt crystallization and freeze-thaw.

6. It would be also be informative to study the grouts under an ESEM (environmental electron scanning microscope) to observe how their micro-structure changes under various environmental conditions such as fluctuating humidity and freeze-thaw.

Performance of the grout in combination with other materials

7. The question of how the grouts perform in combination with other materials is outstanding. Important tests to be conducted on the grouts and on the adherends as a reference are modulus of elasticity and thermal coefficiency of expansion, and bond strength. Adhesive bond strength tests are vital to determine to what extent the grout can remain adhered to materials with vastly different chemical, physical, and mechanical properties.

8. The issue of grouting technique and prewetting porous adherends was only cursorily examined in this study. An interesting aspect to reconsider would be how certain prewetting agents affect bond strength between adobe and hydraulic lime-based grouts. Since adobe can swell and shrink considerably when wet with water, depending on the type and amount of clay minerals present, it would be interesting to investigate the viability of using non-aqueous solutions or surfactants (surface active agents) as prewetting agents.

In situ field testing

9. Lastly, assessment of the grouts performance in the field must be undertaken. Since 1997, the grout is known to have been used to reattach plaster at four sites (Fort Union NM, Fort Davis NHS, San Antonio Mission NHP, and Mission San Juan Capistrano). A follow-up assessment involving an inspection of the plaster surface and the edges, and other types of non-destructive examination methods (e.g. visual examination, tapping on the surface to detect for areas of detachment or instability) at each of these sites is recommended.

10. Since it is impossible to systematically and completely assess the performance of the grout without destructive sampling, it is recommended that facsimiles of the detached-grouted materials be fabricated *in situ* at new field sites. The facsimiles would replicate actual plaster detachment conditions and would be allowed to weather over time. By doing this, careful and thorough examination of the grouted system could be conducted, and the performance of the grouted system could be monitored and assessed without disturbing the historic fabric.

Appendix A. Experimental Program Data

CHARACTERIZATION - GRAIN SIZE DISTRIBUTION

Adobe Sample - HS 36			
ASTM Sieve Size	Weight Retained (g)	% Retained	% Passing
4	5.5	1.93	98.0
16	32.6	11.4	86.6
30	33.1	11.6	75.0
50	29.2	10.2	64.8
100	42.0	14.7	50.1
200	39.8	14.0	36.1
dish	102.6	36.0	0.1
original weight of sample 285g			

Table 20. HS 36 adobe sieve analysis

Adobe Sample - Boneyard			
ASTM Sieve Size	Weight Retained (g)	% Retained	% Passing
4	0.5	0.2	99.8
16	8.3	2.9	96.9
30	34.9	12.2	84.7
50	31.9	11.2	73.5
100	38.8	13.6	59.9
200	43.3	15.2	44.7
dish	127.4	44.7	0
original weight of sample 285g			

Table 21. Boneyard adobe sieve analysis

CHARACTERIZATION - X-RAY DIFFRACTION

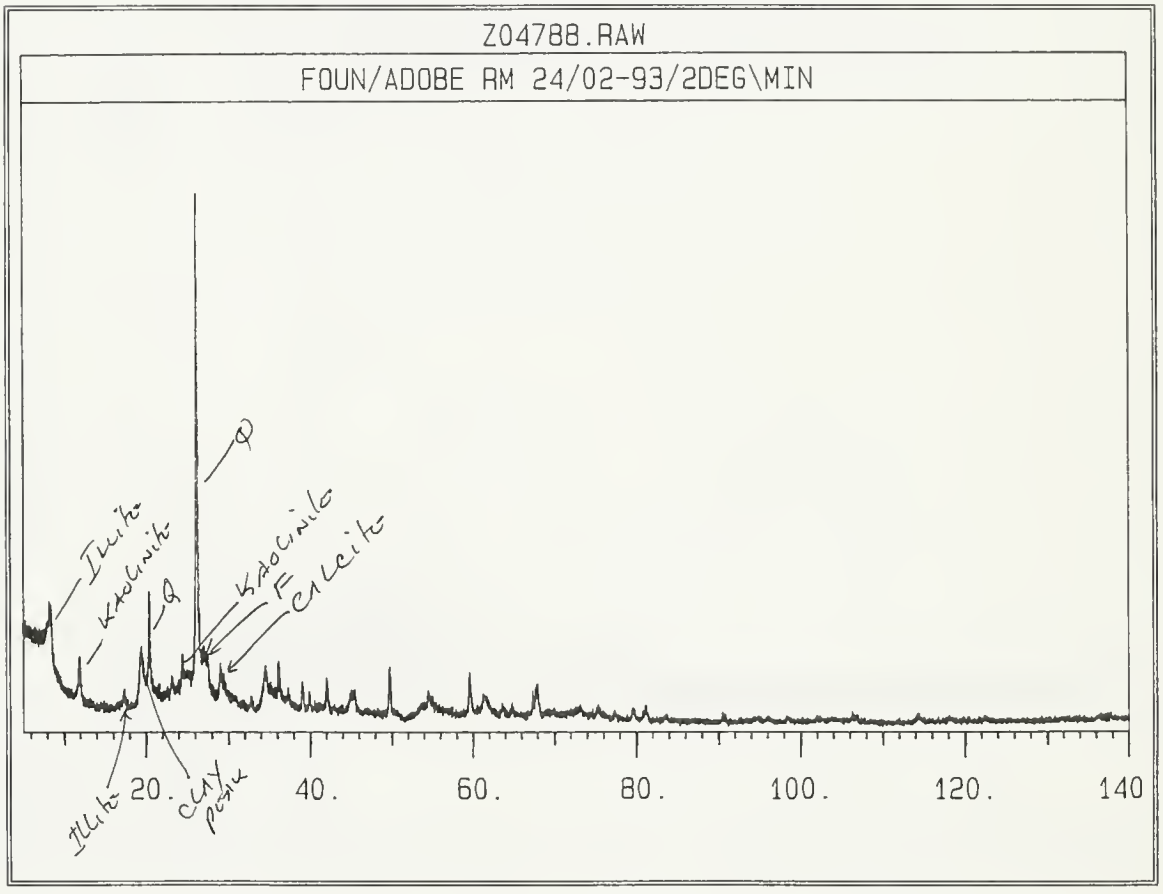

Figure 21. XRD diffractogram of adobe sample HS 36. Annotations by George Austin, New Mexico Bureau of Mines and Mineral Resources

CHARACTERIZATION - X-RAY DIFFRACTION

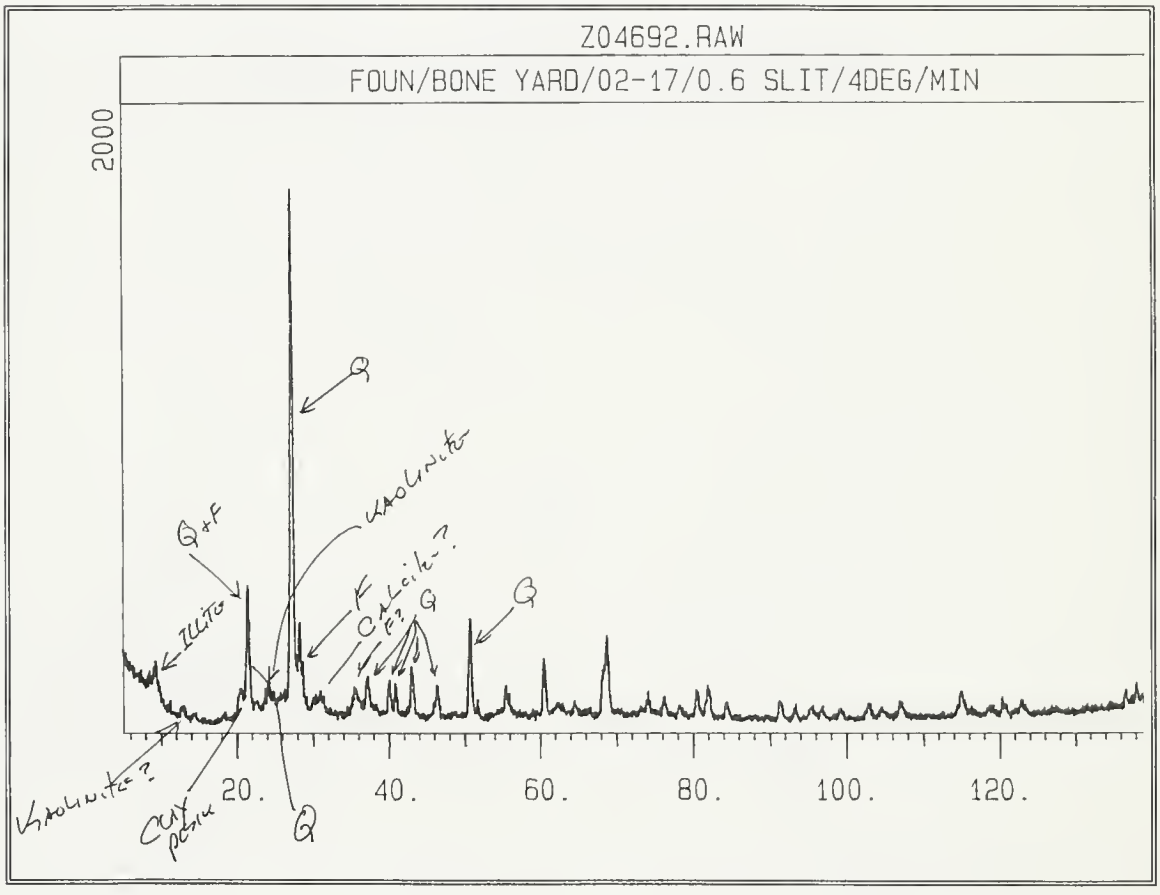

Figure 22. XRD diffractogram of the Boneyard adobe sample. Annotations by George Austin

CHARACTERIZATION - X-RAY DIFFRACTION

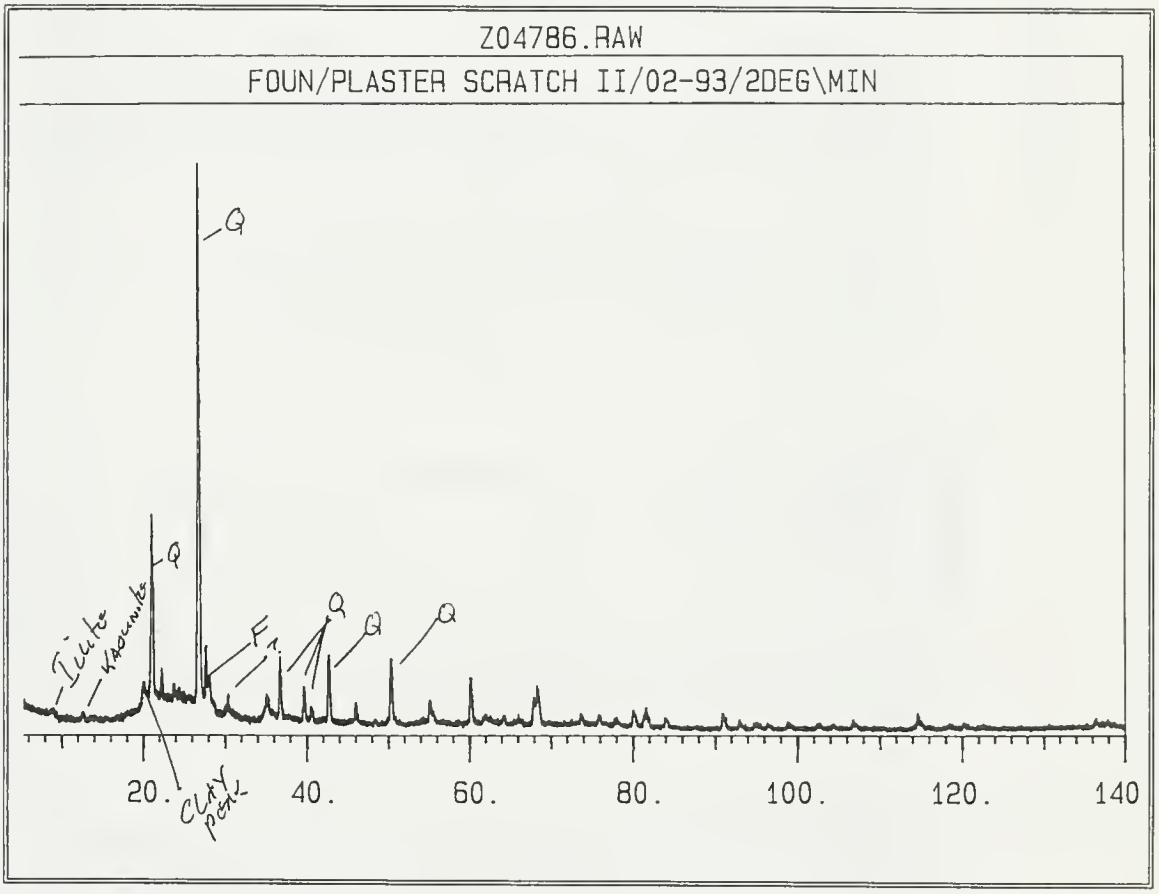

Figure 23. XRD diffractogram of plaster sample(scratch coat). Annotations by George Austin

PHASE I SHRINKAGE

Grout Formula #	Grout Formula Ratio	Wt. dry dish (g)	Wt. wet dish (g)	Wt. liquid grout + wet dish (g)	Wt. liquid grout - wet dish (g)	Wt. cured grout + dish (g)	Wt. cured grout - dry dish (g)	Wt. liquid grout - cured grout (g)	% Weight loss	Mean %
0	1S:1HL	133.7	152.4	282.0	129.6	253.9	120.2	9.4	7.3	
0	1S:1HL	130.8	149.6	278.4	128.8	250.31	119.51	9.3	7.2	
0	1S:1HL	139.3	150.8	280.1	129.3	259.31	120.01	9.3	7.2	7.2
1	1MS:1HL	139.0	151.2	216.6	65.4	200.9	61.8	3.6	5.4	
1	1MS:1HL	135.6	154.9	222.1	67.2	199.1	63.5	3.7	5.5	
1	1MS:1HL	136.9	152.7	221.4	68.7	201.9	65.0	3.7	5.4	5.4
2	1MS:2HL	139.7	155.8	230.8	75.0	208.6	68.9	6.1	8.1	
2	1MS:2HL	132.2	150.5	225.1	74.6	200.7	68.5	6.1	8.1	
2	1MS:2HL	137.5	152.1	227.7	75.6	207.0	69.5	6.1	8.1	8.1
3	1MS:1S:2HL	137.2	157.1	246.3	89.2	220.6	83.5	5.8	6.4	
3	1MS:1S:2HL	138.7	150.2	237.6	87.4	220.5	81.8	5.6	6.4	
3	1MS:1S:2HL	132.8	151.8	241.9	90.1	217.2	84.4	5.8	6.4	6.4
4	1MS:1S:4HL	134.7	155.2	241.1	85.9	214.9	80.2	5.7	6.6	
4	1MS:1S:4HL	135.3	156.4	240.6	84.2	213.9	78.6	5.6	6.7	
4	1MS:1S:4HL	133.4	149.3	235.0	85.7	213.3	79.9	5.8	6.7	6.7
5	3MS:1S:4HL	130.6	154.6	231.5	76.9	203.5	72.9	4.0	5.2	
5	3MS:1S:4HL	136.5	149.9	228.1	78.2	210.7	74.1	4.1	5.2	
5	3MS:1S:4HL	139.7	149.1	225.9	76.8	212.7	73.0	3.8	4.9	5.1
6	1MS:3S:4HL	138.4	153.3	259.0	105.7	236.2	97.8	7.9	7.5	
6	1MS:3S:4HL	135.9	156.3	261.5	105.2	233.2	97.3	7.9	7.5	
6	1MS:3S:4HL	135.0	158.2	262.6	104.4	231.6	96.6	7.8	7.5	7.5
7	1MS:3.7S:2.5HL	131.6	158.7	267.0	108.3	234.9	103.4	5.0	4.6	
7	1MS:3.7S:2.5HL	132.4	153.0	263.8	110.8	237.6	105.2	5.6	5.0	
7	1MS:3.7S:2.5HL	137.8	156.6	263.9	107.3	239.9	102.1	5.2	4.8	4.8
8	1MS:1L	135.3	157.4	216.4	59.1	191.3	56.1	3.0	5.1	
8	1MS:1L	137.5	148.1	205.6	57.6	192.2	54.7	2.8	4.9	
8	1MS:1L	136.2	149.3	207.9	58.6	191.9	55.7	2.9	5.0	5.0

S- Quartz Sand HL- Hydrated Hydraulic Lime MS- Ceramic Microspheres L- Type S Lime C- Kaolin Clay

Table 22. Shrinkage calculations (by weight) - Phase I

PHASE I SHRINKAGE (CONT.)

Grout Formula #	Grout Formula Ratio	Wt. dry dish (g)	Wt. wet dish (g)	Wt. liquid grout + wet dish (g)	Wt. liquid grout - wet dish (g)	Wt. cured grout + dish (g)	Wt. cured grout - dry dish (g)	Wt. liquid grout - cured grout (g)	% Weight loss	Mean %
9	1S:1L	131.9	155.2	252.4	97.2	221.2	89.3	7.9	8.2	
9	1S:1L	130.9	154.9	251.7	96.8	220.2	89.4	7.4	7.7	
9	1S:1L	136.9	157.7	252.0	94.3	224.0	87.1	7.2	7.6	**7.8**
10	1MS:1S:2L	136.0	149.2	228.2	79.0	210.1	74.1	4.9	6.2	
10	1MS:1S:2L	131.3	148.7	220.9	72.2	198.9	67.7	4.5	6.3	
10	1MS:1S:2L	133.1	156.8	229.7	73.0	201.5	68.4	4.6	6.3	**6.3**
11	1MS:1S:4L	134.4	154.3	220.3	66.0	195.91	61.5	4.5	6.8	
11	1MS:1S:4L	139.3	148.4	216.0	67.6	202.3	63.0	4.6	6.8	
11	1MS:1S:4L	138.6	147.7	217.4	69.7	203.5	64.9	4.8	6.9	**6.9**
12	3MS:1S:4L	139.9	158.6	222.7	64.1	201.1	61.2	2.8	4.4	
12	3MS:1S:4L	132.9	153.3	217.2	63.9	194.1	61.2	2.7	4.3	
12	3MS:1S:4L	139.1	156.7	220.5	63.9	200.1	61.0	2.8	4.4	**4.4**
13	1MS:3S:4L	134.9	158.5	236.7	78.2	207.5	72.7	5.6	7.1	
13	1MS:3S:4L	137.4	150.7	233.5	82.8	214.4	77.0	5.8	7.0	
13	1MS:3S:4L	130.7	151.9	232.3	80.3	205.6	74.8	5.5	6.9	**7.0**
14	1MS:1C	132.6	154.7	199.5	44.9	171.8	39.1	5.7	12.8	
14	1MS:1C	136.1	152.6	197.9	45.3	175.6	39.5	5.8	12.7	
14	1MS:1C	135.5	148.6	194.7	46.1	175.7	40.2	5.9	12.9	**12.8**
15	1MS:1S:2C	137.5	153.6	207.0	53.3	181.3	43.7	9.6	18.0	
15	1MS:1S:2C	136.6	152.2	204.8	52.7	179.6	43.0	9.6	18.3	
15	1MS:1S:2C	131.5	155.8	209.8	54.0	175.0	43.5	10.5	19.4	**18.6**
16	2MS:2S:0.8C:3.2HL	133.9	158.3	245.3	87.0	210.0	76.1	10.9	12.5	
16	2MS:2S:0.8C:3.2HL	138.7	147.5	230.4	82.9	211.2	72.5	10.4	12.6	
16	2MS:2S:0.8C:3.2HL	130.8	155.7	240.9	85.2	205.3	74.6	10.7	12.5	**12.5**
17	3MS:1S:0.8C:3.2HL	133.8	153.8	213.9	60.1	187.1	53.3	6.8	11.3	
17	3MS:1S:0.8C:3.2HL	137.1	151.6	214.1	62.4	192.4	55.3	7.1	11.4	
17	3MS:1S:0.8C:3.2HL	133.2	150.42	217.7	67.2	192.9	59.7	7.6	11.3	**11.3**
18	4MS:0.8C:3.2HL	139.7	148.78	209.1	60.3	194.0	54.3	6.1	10.1	
18	4MS:0.8C:3.2HL	138.9	150.42	211.9	61.5	194.1	55.2	6.3	10.3	
18	4MS:0.8C:3.2HL	131.7	152.8	214.6	61.8	187.3	55.6	6.2	10.0	**10.1**

S- Quartz Sand HL- Hydrated Hydraulic Lime MS- Ceramic Microspheres L- Type S Lime C- Kaolin Clay

PHASE II FORMULA STATISTICS

#01 1MS : 1HL
weight - 1 : 1
volume - 1.9 : 1
liquid:solids (v/v): 1.0 : 1.9
specific gravity (g/cm^3): 1.10
Marsh flow cone (min:sec): 1:20

#03 1MS : 1S : 2HL
weight - 1 : 1 : 2
volume - 3.7 : 1 : 3.9
liquid:solids (v/v): 1.0 : 2.1
specific gravity (g/cm^3): 1.49
Marsh flow cone (min:sec): 1:21

#04 1MS : 1S : 4HL
weight - 1 : 1 : 4
volume - 3.7 : 1 : 7.8
liquid:solids (v/v): 1.0 : 1.6
specific gravity (g/cm^3): 1.65
Marsh flow cone (min:sec): 1:26

#07 1MS : 3.7S : 2.5HL: 20% Rhoplex E-330 in H_2O (v/v)
weight - 1 : 3.7 : 2.5
volume - 1 : 1 : 2
liquid:solids (v/v): 1.0 : 1.8
specific gravity (g/cm^3): 0.99
Marsh flow cone (min:sec): 1:28

#19 1MS : 1S : 2HL : 10% El Rey in H_2O (v/v)
weight - 1 : 1 : 2
volume - 3.7 : 1 : 3.9
liquid:solids (v/v): 1.0 : 2.1
specific gravity (g/cm^3): 1.09
Marsh flow cone (min:sec): 1:23

#20 1MS : 1HL : 10% El Rey in H_2O (v/v)
weight - 1 : 1
volume - 1.9 : 1
liquid:solids (v/v): 1.0 : 1.9
specific gravity (g/cm^3): 0.96
Marsh flow cone (min:sec): 1:19

⇒ Approximate conversion from weight to volume:
S-sand @1.61g/ml **MS**-microspheres @ .43g/ml **HL**-hydrated hydraulic lime @ .82g/ml

⇒ H_2O added in 5ml increments until adequate viscosity was achieved to allow the material to pass fluidity through a 12 gauge cannula (~4.0mm diameter opening)

⇒ H_2O temperature @ 20°C±3°C

⇒ Marsh flow cone - efflux of 1,000ml

PHASE II PERCENT SHRINKAGE

Grout Formula #	Wt. In Air (g)	Wt. in Mineral Spirits (g)	Dw	vw(Ml)	vd	% Shrinkage
#01a	21.25	2.72	18.53	25.0	24.10	3.62
#01b	21.30	2.75	18.55	25.0	24.12	3.51
#01c	21.30	2.80	18.50	25.0	24.06	3.77
#01					**mean**	**3.63**
#03a	22.52	4.05	18.47	25.0	24.02	3.91
#03b	22.77	4.24	18.53	25.0	24.09	3.63
#03c	22.30	3.94	18.36	25.0	23.87	4.52
#03					**mean**	**4.02**
#04a	24.02	6.42	17.60	25.0	22.89	8.44
#04b	24.09	6.59	17.50	25.0	22.76	8.97
#04c	24.21	6.30	17.91	25.0	23.29	6.83
#04					**mean**	**8.08**
#07a	23.82	5.07	18.75	25.0	24.39	2.45
#07b	23.55	5.22	18.33	25.0	23.83	4.68
#07c	23.50	5.40	18.10	25.0	23.54	5.84
#07					**mean**	**4.32**
#19a	22.11	3.88	18.23	25.0	23.71	5.15
#19b	21.90	3.23	18.67	25.0	24.27	2.91
#19c	22.02	2.97	19.06	25.0	24.78	0.88
#19					**mean**	**2.98**
#20a	20.46	1.68	18.78	25.0	24.42	2.33
#20b	20.37	1.73	18.64	25.0	24.24	3.04
#20c	20.12	1.59	18.54	25.0	24.11	3.57
#20					**mean**	**2.98**

Table 23. Percent shrinkage calculations - Phase II

#01 1MS : 1HL; **#03** 1MS : 1S : 2HL; **#04** 1MS : 1S : 4HL; **#07** 1MS:3.7S:2.5HL w/20% Rhoplex E-330 in H_2O; **#19** 1MS : 1S : 2HL; w/ 10% EL Rey in H_2O; **#20** 1MS : 1HL w/ 10% EL Rey in H_2O (ratios by weight)

PHASE II SPLITTING TENSILE STRENGTH

Grout Formula #	Load Applied (psi)	Splitting Tensile Strength (psi)	Location and Type of Fracture
01-a	56.5	19.03	major jagged fracture along diametral plane; secondary, smaller fracture adjacent to upper end of fracture
01-b	58.8	19.81	single jagged fracture along diametral plane; chipped at upper and lower ends of fracture
01-c	56.0	18.86	single straight line fracture along diametral plane; chipped at lower end of fracture
#01	**mean**	**19.24**	**standard deviation** **±0.50**
03-a	82.5	27.79	single jagged fracture along diametral plane; chipped at upper and lower ends of fracture
03-b	82.1	27.66	single jagged fracture along diametral plane
03-c	66.6	22.44	two jagged fractures running the length of the diametral plane; third, smaller fracture adjacent to upper end of fracture
03-d	87.7	29.54	single jagged fracture along diametral plane; chipped at upper and lower ends of fracture
#03	**mean**	**26.86**	**standard deviation** **±3.07**
04-a	145.8	49.12	single curved fracture along diametral plane
04-b	157.4	53.02	two jagged fractures running the length of the diametral plane; chipped at upper end of fractures
04-c	144.9	48.81	single jagged fracture along diametral plane; chipped at upper and lower ends of fracture
04-d	149.5	50.36	major jagged fracture along diametral plane; secondary, smaller fracture adjacent to upper end of fracture
#04	**mean**	**50.33**	**standard deviation** **±1.91**
07-a	96.7	32.58	single jagged fracture along diametral plane; crushed at upper and lower ends of fracture
07-b	73.1	24.63	single jagged fracture along diametral plane; crushed at upper and lower ends of fracture
07-c	99.4	33.48	single jagged fracture along diametral plane; crushed at upper and lower ends of fracture
07-d	97.7	32.91	major jagged fracture along diametral plane; secondary, smaller fracture adjacent to lower end of fracture; chipped at upper end of fracture
#07	**mean**	**30.90**	**standard deviation** **±4.19**
19-a	94.5	31.83	single jagged fracture along diametral plane; slightly crushed at upper and lower ends of fracture
19-b	94.2	31.73	single jagged fracture along diametral plane; slightly crushed at upper and lower ends of fracture
19-c	88.4	29.78	single jagged fracture along diametral plane; slightly crushed at upper and lower ends of fracture
19-d	94.8	31.94	single jagged fracture along diametral plane; slightly crushed at upper and lower ends of fracture
#19	**mean**	**31.32**	**standard deviation** **±1.03**

Table 24. Splitting tensile strength data - Phase II

#01 1MS : 1HL; **#03** 1MS : 1S : 2HL; **#04** 1MS : 1S : 4HL; **#07** 1MS:3.7S:2.5HL w/20% Rhoplex E-330 in H_2O; **#19** 1MS : 1S : 2HL; w/ 10% EL Rey in H_2O; **#20** 1MS : 1HL w/ 10% EL Rey in H_2O (ratios by weight)

PHASE II SPLITTING TENSILE STRENGTH CONT.

Grout Formula #	Load Applied (psi)	Splitting Tensile Strength *(psi)	Location and Type of Fracture	
20-a	79.4	26.75	single straight line fracture along diametral plane; chipped at upper and lower ends of fracture	
20-b	79.4	26.75	single jagged fracture along diametral plane	
20-c	68.6	23.11	single jagged fracture along diametral plane; chipped at upper and lower ends of fracture	
20-d	80.5	27.12	single straight line fracture along diametral plane	
#20	**mean**	**25.93**	**standard deviation**	**±1.88**
adobe-a	174.3	58.72	small, single jagged fracture at center of diametral plane; severely crushed at upper and lower halves of the specimen	
adobe-b	178.2	60.03	small, single jagged fracture at center of diametral plane; severely crushed at upper and lower halves of the specimen	
adobe-c	174.7	58.85	small, single jagged fracture at center of diametral plane; severely crushed at upper and lower halves of the specimen	
adobe-d	185.2	62.39	small, single jagged fracture at center of diametral plane; severely crushed at upper and lower halves of the specimen	
adobe	**mean**	**60.00**	**standard deviation**	**±1.70**

* The following formula was used to calculate the splitting tensile strength of each specimen: $T=2P/\pi ld$

T=splitting tensile strength; **P**=maximum load applied indicated by the testing machine; **l**=length, in. (m); and **d**=diameter, in. (m)

#01 1MS : 1HL; **#03** 1MS : 1S : 2HL; **#04** 1MS : 1S : 4HL; **#07** 1MS:3.7S:2.5HL w/20% Rhoplex E-330 in H_2O; **#19** 1MS : 1S : 2HL; w/ 10% EL Rey in H_2O; **#20** 1MS : 1HL w/ 10% EL Rey in H_2O (ratios by weight)

PHASE II WATER VAPOR TRANSMISSION

Days elapsed	3	10	13	16	19	22	25	28	30	Average day 10-30	Standard Deviation
Temp °C	23.8	18.7	22.1	23.4	22.9	22.5	23.5	24.2	24.5	22.7	
RH	42.8	42.1	44.6	45.6	49.4	49.8	48.4	49.7	49.9	47.4	
#01	0.82	0.76	0.79	0.76	0.75	0.75	0.71	0.72	0.73	0.75	±0.032
#03	0.81	0.88	0.88	0.86	0.83	0.83	0.82	0.82	0.85	0.85	±0.025
#04	0.82	0.78	0.79	0.76	0.77	0.77	0.74	0.74	0.77	0.77	±0.740
#07	1.26	1.2	1.24	1.27	1.24	1.22	1.22	1.22	1.22	1.23	±0.020
#19	0.65	0.63	0.65	0.64	0.63	0.63	0.6	0.62	0.63	0.63	±0.014
#20	0.59	0.58	0.58	0.56	0.55	0.55	0.53	0.55	0.56	0.56	±0.018
adobe	0.57	0.59	0.58	0.55	0.54	0.54	0.53	0.52	0.53	0.55	±0.023

Table 25. Water vapor transmission data - corrected average weight loss (g/day) Phase II

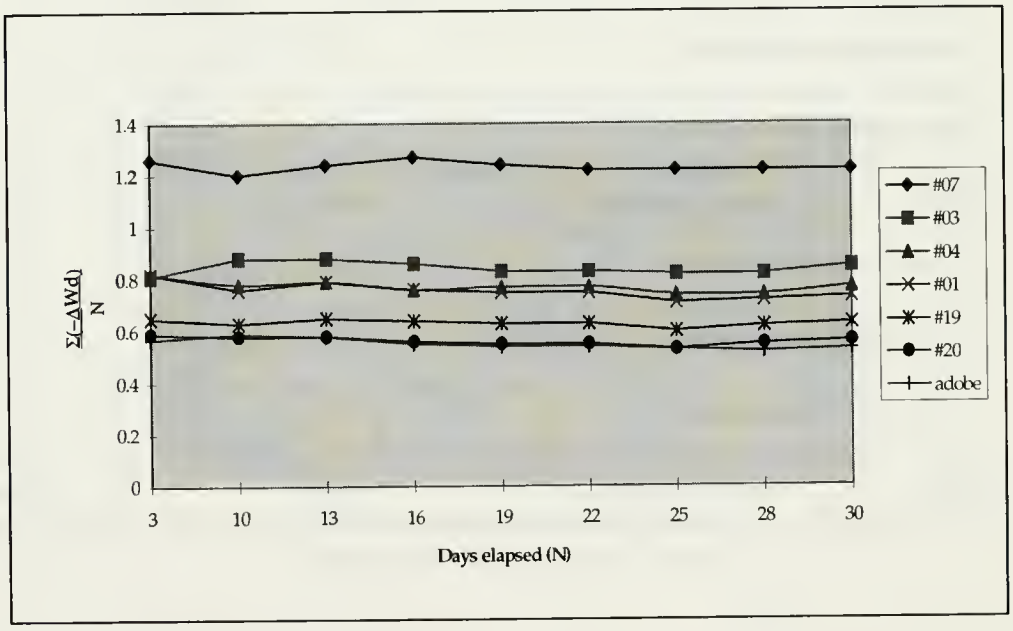

Figure 24. Graph of water vapor transmission data - corrected average weight loss (g/day) over a 30 day period

#01 1MS : 1HL; **#03** 1MS : 1S : 2HL; **#04** 1MS : 1S : 4HL; **#07** 1MS:3.7S:2.5HL w/20% Rhoplex E-330 in H_2O; **#19** 1MS : 1S : 2HL; w/ 10% EL Rey in H_2O; **#20** 1MS : 1HL w/ 10% EL Rey in H_2O (ratios by weight)

PHASE III. BOND STRENGTH

Grout-Plaster Assembly #	Prewetting Agent	Void Width (")	Load applied (psi)	Location and Type of Failure	
A-1	H_2O	0.5	214	Cohesive failure within the grout	
A-2	H_2O	0.5	175	Cohesive failure within the grout	
A-3	H_2O	0.5	170	Cohesive failure within the grout	
Mean A	**186 psi**			**Standard Deviation**	**±24.09**
B-1	H_2O	1	160	Adhesion failure at the grout plaster interface	
B-2	H_2O	1	149	Cohesive failure within the grout	
B-3	H_2O	1	136	Cohesive failure within the grout	
Mean B	**148 psi**			**Standard Deviation**	**±12.01**
C-1	5% El Rey in H_2O	0.5	170	Cohesive failure within the grout	
C-2	5% El Rey in H_2O	0.5	210	Cohesive failure within the grout	
C-3	5% El Rey in H_2O	0.5	183	Cohesive failure within the grout	
Mean C	**187 psi**			**Standard Deviation**	**±20.40**
D-1	5% El Rey in H_2O	1	145	Cohesive failure within the grout	
D-2	5% El Rey in H_2O	1	175	Cohesive failure within the grout	
D-3	5% El Rey in H_2O	1	170	Cohesive failure within the grout	
Mean D	**163 psi**			**Standard Deviation**	**±16.07**

Table 26. Bond strength test data - Phase III

Three types of failure are reported (Horie 1987, 74-75.):

1. cohesive failure in the adhesive, where the adhesive material itself fails
2. adhesion failure, where the bond between the adhesive and the object fails along the interface
3. cohesive failure in the substrate, where the object may break, leaving a small portion of the surface attached to the adhesive

Appendix B. SEM Photomicrographs

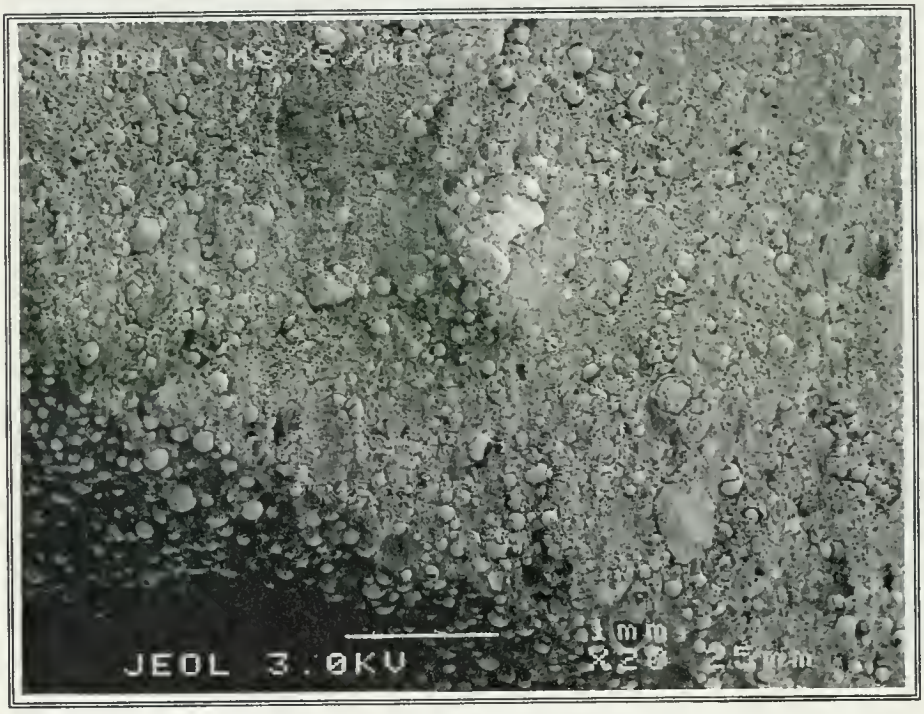

Figure 25. Grout formula #03 composed of 1MS:1S:1HL (w). The large, irregular sized particles interspersed between microspheres and hydraulic lime are sand grains. White scale bar equals 1 mm. (20x)

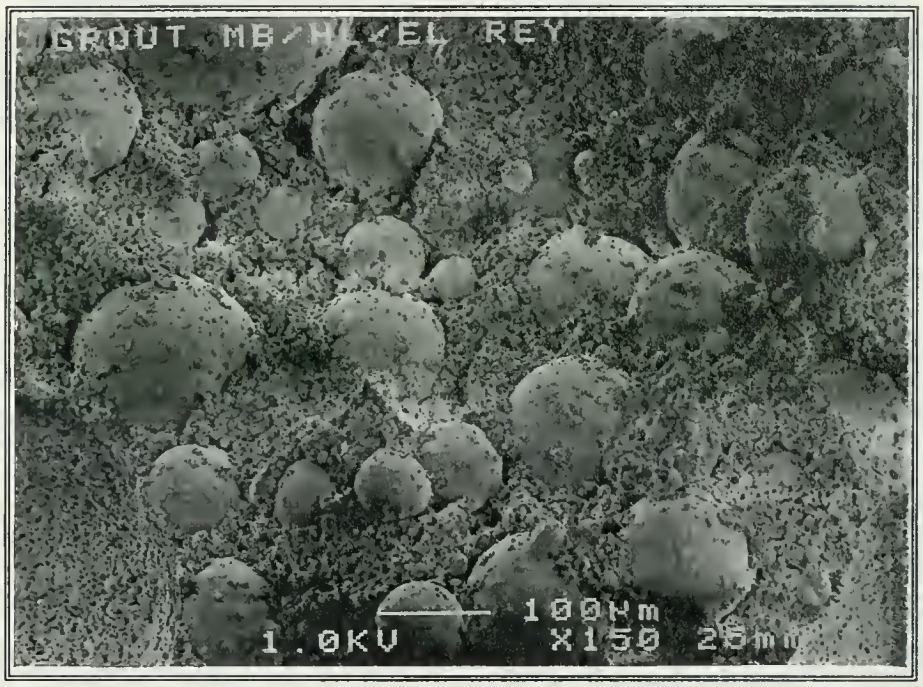

Figure 26. Grout formula # 20 (#01 amended with a 10% acrylic emulsion in H_2O) at 150x. Notice how the well dispersed individual microspheres are thoroughly surrounded by the hydraulic lime matrix.

Figure 27. Grout formula #01 composed of 1MS:1HL (w) at 4,000x. A large microsphere is seen on the right, and a smaller sphere on the left. Hydraulic lime fills the space in between. White scale bar measures 1 micron.

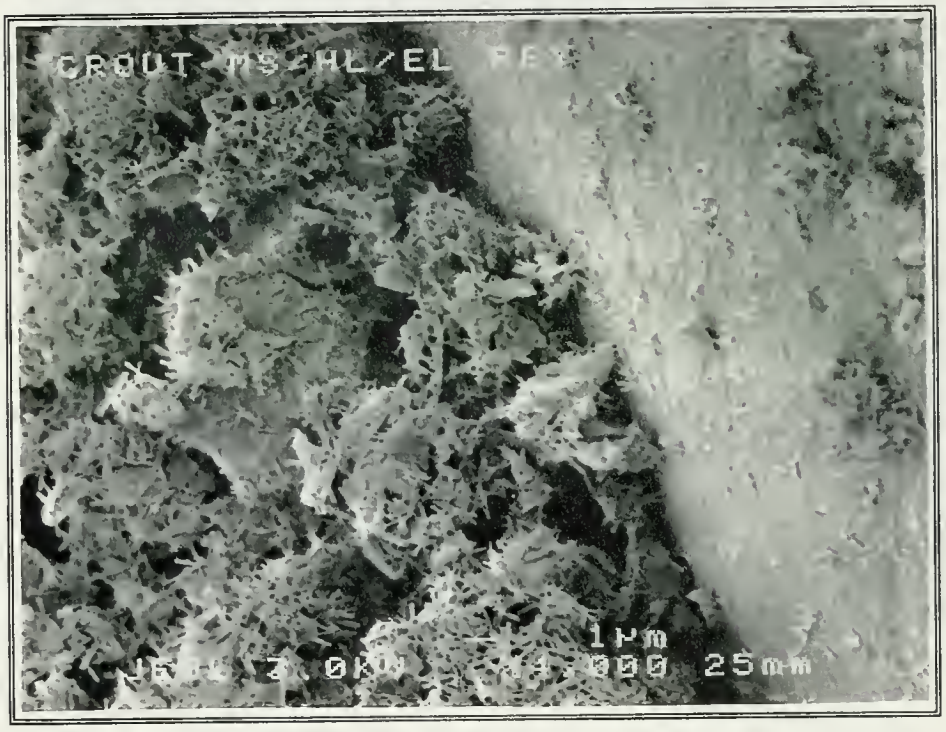

Figure 28. Grout formula # 20 (#01 amended with a 10% acrylic emulsion in H_2O) at 4,000x. The mass on the right is a microsphere; the adjacent platelets are hydraulic lime. Note acicular needle-like formations projecting from the particle surfaces. The needles may be crystalline and may have formed as a result of the acrylic additive.

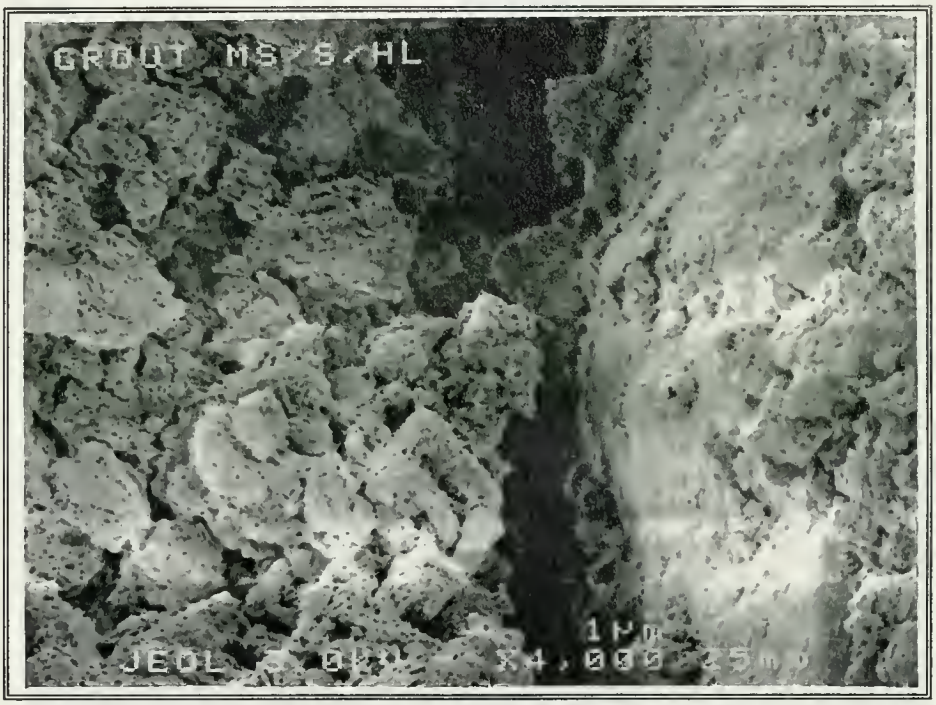

Figure 29. Grout formula #03 composed of 1MS:1S:1HL (w). Hydraulic lime can be seen coating the surface of the microsphere, but in this case, it does not appear to be well bonded to the sphere. White scale bar equals 1 micron. (4,000x)

Figure 30. Grout formula #19 composed of 1MS:1S:1HL with a 10% aqueous acrylic emulsion at 4,000x. Again, the needle like projections appear in the acrylic modified grout. The hydraulic lime appears to be well attached to the microsphere or sand grain on the right.

Bibliography

Agnew, Neville. 1987 "Adobe preservation: report on a three month research project at the Getty Conservation Institute." (on file at the Getty Conservation Institute).

Agrawal, O.P. and Kamal K Jain. 1984 "Problems of conservation of wall paintings in India." In *International Symposium on the Conservation and Restoration of Cultural Property: Conservation and Restoration of Mural Paintings (I) November 17-21, 1983, Tokyo, Japan*, ed. Y. Emoto and S. Miura, 31-39. Japan: Tokyo National Research Institute of Cultural Properties.

ASCE Grouting Committee. 1980. "Preliminary glossary of terms relating to grouting." In *ASCE Journal of Geotechnical Division*. Volume 106 (Gt7): 803-815.

Ashurst, John and Nicola Malnic. 1984. "Cleaning and consolidation of the chapel plaster at Cowdray House Ruins." London: Research and Technical Advisory Service, Historic Buildings and Monuments Commission (report on file at ICCROM).

Ashurst, John. 1983. *Mortars Plasters and Renders in Conservation: A Basic Guide*. London: Ecclesiastical Architects' and Surveyors' Association.

Ashurst, John and Nicola. 1988. *Practical Building Conservation: English Heritage Technical Handbook. Volume 3 Mortars Plasters and Renders*. Hants England: Gower Technical Press Ltd.

Ashurst, John and Francis G. Dimes (eds). 1990. *Conservation of Building and Decorative Stone, Volume 2*. London: Butterworth-Heinemann Ltd.

Atzeni, Cirillo, Maria C. Grazia, Luigi Massidda, and Ulrico Sanna. 1996. "The use of 'stabilized earth' in the conservation of megalithic monuments." In *Conservation and Management of Archaeological Sites*, Volume 1: 161-168.

Austin, George. 1990. "Adobe and related building materials in New Mexico, USA." In *6th International Conference on the Conservation of Earthen Architecture, Adobe 90 Preprints*, 417-423. Los Angeles: Getty Conservation Institute.

Austin, G.S. and R.K. Leininger. 1976. "The effect of heat-treated sedimented mixed-layer illite-smectite as related to quantitative clay mineral identifications." In *Journal of Sedimentary Petrology* Volume 46, No.1

Baker, Wallace Hayward (ed). 1982. *Proceedings of the Conference on Grouting in Geotechnical Engineering*. New York: American Society of Civil Engineers.

Barcellona, S., U. Santamaria, E. Borrelli,. and M. Laurenzi Tabasso. 1993. "Evaluation of injection grouting for structural strengthening of ancient buildings." In *Conservation of Stone and Other Materials, Volume 2: Prevention and Treatment, June 29-July 1, 1993, Paris*, ed., M.-J. Thiel. London: E & FN Spon.

Baronio, G. and L Binda. 1987. "Study of the interface between binder and aggregates, plaster and wall in ancient lime mortars and plasters." In *Proceedings of the Fourth North American Masonry Conference, Los Angeles, August 1987*: 66-1 -13.

Beas, Maria Isabel. 1991. Traditional architectural renders on earthen surfaces" Thesis; Graduate Program in Historic Preservation, University of Pennsylvania.

Bell, F.G. (ed) 1982. *Methods of Treatment of Unstable Ground*. London: Newnes-Butterworths.

Bell, F.G. 1983. *Engineering Properties of Soils and Rocks*. 2nd ed. London: Butterworths.

Binda, Luigia, Mario Berra, Giulia Baronio, and Alberto Fontana. 1990. "Repair of masonry by injection technique: effectiveness, bond and durability problems." In *Proceedings from the International Technical Conference on Structural Conservation of Stone Masonry, Athens, 1989.* Rome: ICCROM.

Biscontin, Guido and Gianna Riva. 1981. "Study about water vapor permeability in stone materials after protective treatment." In *The Conservation of Stone II, Preprints of the Contributions to the International Symposium, Bologna, 27-30 October 1981, Part B: Treatment.* ed. Raffaella Rossi-Manaresi.

Böttger, K.G. and D. Knöfel. 1993. "Development and testing of injection mortars of high sulfate resistance." In *Conservation of Stone and Other Materials, Volume 2: Prevention and Treatments, June 29-July 1, 1993, Paris,* ed., M.-J. Thiel. London: E & FN Spon.

Bowen, Robert. 1981. *Grouting in Engineering Practice.* 2nd ed. London: Applied Science Publishers, Ltd.

Boynton, Robert S. 1966. *Chemistry and Technology of Lime and Limestone.* New York: Interscience Publishers.

Brackin, Anne E. 1994. "A comparative study of the effects of applying acrylics and silanes in sequence and in mixture, with a case study of the column in the convento of Mission San Jose y San Miguel de Aguayo, Texas." Thesis: Graduate Program in Historic Preservation, University of Pennsylvania.

Cabrera, N. and D.A. Vermilyea. 1958. "The growth of crystals from solution." In *The Growth and Perfection of Crystals. Proceedings of an International Conference on Crystal Growth, Cooperstown, New York, 27-28 August, 1958:* 393-412.

Chiari, Giacomo 1980. "Treatment of adobe friezes in Peru." In *Third International Symposium on Mudbrick (Adobe) Preservation, Ankara, ICOM- ICOMOS, Turkish National Committees, 29 Sept.-4 Oct., 1980*: 39-45.

Clifton, James R. 1977. *Preservation of Historic Adobe Structures - A Status Report.* Washington, DC:, National Bureau of Standards.

Clifton, James R., Brown, Paul Wencil, and Robbins, Carl R. 1978. *Methods for Characterizing Adobe Building Materials.* Washington, DC: Institute for Applied Technology, National Bureau of Standards.

Cobau, Andreina Contanzi. 1993. "*In situ* consolidation of a roman fresco near Ein Yael, Jerusalem." In *Preprints of the ICOM Tenth Triennial Meeting, Washington, DC, USA, 22-27 August 1993.* Paris: ICOM Committee for Conservation.

Coffman, Richard, Neville Agnew, George Austin, and Eric Doehne. 1990. "Adobe mineralogy: characterization of adobes from around the world." In *6th International Conference on the Conservation of Earthen Architecture, Adobe 90 Preprints.* Los Angeles: Getty Conservation Institute.

Colalucci, Gianluigi. 1991. "The frescos of Michelangelo on the vault of the Sistine Chapel: original technique and conservation." In *The Conservation of Wall Paintings: Proceedings of a Symposium Organized by the Courtald Institute of Art and the Getty Conservation Institute, London, July 13-16 1987,* ed., Sharon Cather, 67-76. Los Angeles: J. Paul Getty Trust.

Collepardi, M.1990. "Degredation and restoration of masonry walls of historical buildings." In *Materials and Structures,* ed. M. Fickelson. France: Rilem.

Craig, R.F. 1992. *Soil Mechanics.* 5th ed. London: Chapman and Hall.

Crosby, A. 1980. "Conservation of painted lime plaster on mud brick walls at Tumacacori National Monument, USA." In *Third International Symposium on Mudbrick (Adobe) Preservation, Ankara, ICOM-ICOMOS, Turkish National Committees, 29 Sept.-4 Oct., 1980*: 59-73.

Dean, John A (ed) 1985. *Lange's Handbook of Chemistry*. 13th ed. New York: McGraw-Hill Book Company.

Deere, Don U. 1982. "Cement-bentonite grouting for dams." In *Proceedings of the Conference on Grouting in Geotechnical Engineering*, ed. Wallace Hayward Baker. New York: American Society of Civil Engineers.

Dolar-Mantuani, Ludmila. 1983. *Handbook of Concrete Aggregates*. New Jersey: Noyes Publications.

Dupas, M. and A.E. Charola. 1982. "L'analyse des mortiers et enduits des peintures murales et des batiments anciens." In *Mortars, Cements and Grouts Used in the Conservation of Historic Buildings, Proceedings of the Symposium, November 3-6, 1981*, 281-296. Rome: ICCROM.

Eckel, Edwin C. 1922. *Cements, Limes and Plasters: Their Materials, Manufacture, and Properties*. 2nd ed. New York: John Wiley & Sons, Inc.

Ferragni, D., M. Forti, J. Malliet, P. Mora, J.M. Teutonico and G. Torraca. 1983. "*In situ* consolidation of wall and floor mosaics by means of injection grouting techniques." In *Mosaics No. 3, Conservation In Situ, Aquileia, 1983*. Rome: ICCROM.

Ferragni, D., M. Forti, J. Malliet, P. Mora, J.M. Teutonico and G. Torraca. 1984. "Injection grouting of mural paintings and mosaics." In *Adhesives and Consolidants*, eds. N.S. Brommelle, E.M. Pye, P Smith, and G. Thomson, 110-116. Paris: IIC.

Gächter, R. and H. Müller (eds). 1990. *Plastics Additives Handbook*, 537-547. New York: Oxford University Press.

Gordon, Arnold J. and Richard A. Ford. 1972. *The Chemists' Campanion: A Handbook of Practical Data, Techniques, and References*. New York: John Wiley & Sons, Inc.

Hammer, Ivo. 1990. "Conservation of historical renderings and cooperation with artisans: two pilot projects in the Wachau, Austria." In *Superfici dell' Architettura: le Finiture: ATTI del Convegno di Studi Bressanone, 26-29 Guigni 1990*, ed. Guido Biscontin and Stefano Volpin, 357-366. Padova, Italy: Libreria Progetto Editore Padova.

Hartzler, Robert, Lyle. 1996. "A program of investigation and laboratory research of acrylic modified earthen mortar used at three prehistoric puebloan sites." Thesis; Graduate Program in Historic Preservation, University of Pennsylvania.

Hartzler, Robert. 1996. Acrylic-Modified Earthen Mortar: a program of investigation and laboratory research into acrylic modified earthen mortar used at three prehistoric puebloan sites." Intermountain Cultural Resource Center, Professional Paper No. 61.

Horie, C.V. 1987. *Materials for Conservation*. London: Butterworths.

Houben, Hugo and Hubert Guillaud. 1994. *Earth Construction*. London: Intermediate Technology Publications.

Houlsby, A. Clive. 1990. *Construction and Design of Cement Grouting*. New York: John Wiley & Sons, Inc.

Jacob, Judith and Norman R. Weiss. 1989. "Laboratory measurement of water vapor transmission rates of masonry mortars and paints." In *Bulletin of the Association for Preservation Technology (APT)*, Volume 21, No. 3-4.

Jedrzejewska, Hanna. 1982. "Ancient mortars as criterion in analyses of old architecture." In *Mortars, Cements and Grouts Used in the Conservation of Historic Buildings, Proceedings of the Symposium, November 3-6, 1981*, 311-330. Rome: ICCROM.

Jedrzejewska, Hanna. 1960. "Old mortars in Poland: a new method of investigation." In *Studies in Conservation*, Volume 5, No. 4: 132-138.

Kesler, C.E. 1966. "Strength." In *Significance of Tests and Properties of Concrete and Concrete Making Materials*, ASTM Technical Publication No. 169-A, 144-188. Philadelphia PA: American Society for Testing and Materials.

Kosinka, Jan. 1991. "Reinstallation of the mosaic." In *The Conservation of the Orpheus Mosaic at Paphos, Cyprus*, ed. Nicholas Stanley Price, 29-35. Los Angeles: The J. Paul Getty Trust.

Lavelle, Joseph A. 1986. "Acrylic latex modified Portland cement," Paper presented at the American Concrete Institute Fall Convention, Baltimore, Maryland.

Lawrence, S.J., and H.T. Cao. 1988. "Microstructure of the interface between brick and mortar." In *Proceedings of the Eighth International Brick/Block Masonry Conference, Dublin*, Volume 1, 194-204.

Leonard, M.W. and J.A. Dempsey. 1963. "Clays for clay grouting." In *Grouts and Drilling Muds in Engineering Practice*, 119-126. London: Butterworths.

Lewin, S.Z. 1982. "X-ray diffraction and scanning electron microscope analysis of conventional mortars." In *Mortars, Cements and Grouts Used in the Conservation of Historic Buildings, Proceedings of the Symposium, November 3-6*, 1981, 101-132. Rome: ICCROM.

Lipe, William D. 1984. "Chapter 1: Value and meaning in cultural resources," In *Approaches to the Archaeological Heritage: A Comparative Study of World Cultural Resource Management Systems*. Cambridge: Cambridge University Press.

Littlejohn, G.S. 1982. "Design of cement based grouts," In *Proceedings of the Conference on Grouting in Geotechnical Engineering*, ed. Wallace Hayward Baker. New York: American Society of Civil Engineers.

Long, W.B. 1990. "Chapter 15: Grouting" In *The Maintenance of Brick And Stone Masonry Structures*, ed. A. Maurice Sowden. New York: Van Nostrand Reinhold.

Lujan, Rodolfo. 1991. Conservation of mural paintings and external stuccos, Pagan, Union of Myanmar: ICCROM mission progress report, July 2-30". (on file at ICCROM).

Majewski, L.J. n.d. "The conservation of wall paintings in archaeological excavations." In *Colt Archaeological Institute*, Monograph series 3: 24-43.

Ma, Jiayu. 1995. "The Leshan Grand Buddha: investigations of traditional and alternative materials for repairs." In *Methods of Evaluating Products for the Conservation of Porous Building Materials: International Colloquium, Rome, 19-21 June 1995*. Rome: ICCROM.

Mariachi, Roberto. 1996 "Does your vapor mask fit? " In *Site Hazards*. Nevada: Canta Todo Press.

Matero, Frank G. 1995. "A programme for the conservation of architectural plasters in earthen ruins in the American Southwest: Fort Union National Monument, USA," In *Conservation and Management of Archaeological Sites*, Volume 1, No. 1:.5-24.

Matero, Frank G. and Angelyn Bass. 1995. "Design and evaluation of hydraulic lime grouts for the reattachment of lime plasters on earthen walls." In *Conservation and Management of Archaeological Sites*, Volume 1, No. 2:.97-108.

Matero, Frank G. and Angelyn Bass 1994. "Orphans of the storm: the preservation of architectural plasters in earthen ruins." In *CRM*, Volume 17, No. 4.

McCraig, Iain. 1992. "The Smeaton Project: factors affecting the properties of lime-based mortars for use in the repair and conservation of historic buildings." In *Lime and Other Alternative Cements*, eds. Neville Hill, Stafford Holmes, and David Mather, 237-244. London: Intermediate Technology Publications Ltd.

McGraw-Hill Dictionary of Scientific and Technical Terms. 1989. 4th ed. New York: McGraw-Hill, Inc.

Mclean, E.O. 1982. "Chapter 12: soil pH and lime requirement" In *Methods of Soil Analysis, Part 2. Chemical and Microbiological Properties*. Agronomy Monograph No. 9, 2nd ed. Wisconsin: Asa-Sssa.

Miltiadou, Androniki E. n.d. Grouting as a Method for the Repair of Masonry Monuments. Thesis; Graduate Program in Conservation Studies, University of York.

Miltiadou, Androniki E. 1991. *Etude des Coulis Hydrauliques: Pour la Reparation et le renforcement des Structures et des Monuments Historiques en Maconnerie*. Paris: Laboratoire Central des Pontes et Chausses.

Mora P., L. Mora, P. Philippot. 1984. *Conservation of Wall Paintings*. London: Butterworths.

Mora, Laura and Paolo, Giorgio Torraca, and Virginia Anne Bonito. 1986. "A coordinated methodology for the treatment and study of the peristyle garden wall of the House of Menander, Pompeii: an interim report." In *Case Studies in the Conservation of Stone and Wall Paintings, Preprints of the IIC Conference, Bologna, 21-26 September, 1986*, eds. N.S. Brommelle and Perry Smith, 38-43. London: The International Institute for Conservation of Historic and Artistic Works.

Nardi, Roberto. 1986. "Conservation of the Arch of Septimus Severus: work in progress," In *Case Studies in the Conservation of Stone and Wall Paintings, Preprints of the IIC Conference, Bologna, 21-26 September, 1986*, eds. N.S. Brommelle and Perry Smith, 38-43. London: The International Institute for Conservation of Historic and Artistic Works.

Nardi, Roberto. 1996. "Zippori, Israel: the conservation of the mosaics of the building of the Nile," In *Archaeological Conservation and its Consequences: Preprints of the Contributions to the Copenhagen Congress, 26-30 August* 1996, ed. Ashok Roy and Perry Smith, 127-132. London: The International Institute for Conservation of Historic and Artistic Works.

National Concrete Masonry Association. 1992. "Investigation of test methods for evaluating grout bond strength to concrete masonry units." Herndon, Virginia: National Concrete Masonry Association.

Newey, Charles, Ruth Boff, Vincent Daniels, Michael Pascoe, and Norman Tennant. 1992. *Science For Conservators, Book 3, Adhesives and Coatings*. London: Routledge.

Newman R. and G. Ferrell. n.d. "Summary of some important instrumental techniques." Paper on file at the Architectural Conservation Laboratory, University of Pennsylvania.

Penelis, G., M. Karaveziroglou, and J. Papayianni. 1989. "Grouts for repairing and strengthening old masonry structures." In *Structural Repair and Maintenance of Historic* Buildings, ed C.A. Brebbia, 179-188. Birkhauser: Computational Mechanics Publications.

Penelis, G., J. Papayianni, and M. Karaveziroglou. 1989. "Pozzolanic mortars for repair of masonry structures." In *Structural Repair and Maintenance of Historic* Buildings, ed C.A. Brebbia, 161-169. Birkhauser: Computational Mechanics Publications.

Peroni, S., C. Tersigni, G. Torraca, M. Cerea, M. Forti, F. Guidobaldi, P. Rossi-Doria, A. De Rege, D. Picchi, F.J. Pietrafitta, and G. Benedetti. 1982. "Lime-based mortars for the repair of ancient masonry and possible substitutes." In *Mortars, Cements and Grouts Used in the Conservation of Historic Buildings, Proceedings of the Symposium, November 3-6, 1981*, 63-99 Rome: ICCROM.

Phillips, M.W. 1980. "Adhesives for the reattachment of loose plaster." In *Apt Bulletin* XII: 37-63.

Phillips, M.W. 1986. "Experiences in the use of acrylic plaster adhesives." In *Case Studies in the Conservation of Stone and Wall Paintings, Preprints of the IIC Conference, Bologna, 21-26 September, 1986.* London: The International Institute for Conservation of Historic and Artistic Works.

Puertas, F., M.T. Blanco-Varela, S. Martínez, F. Acción, and G. Alvarez. "Methodology of analysis of stones and mortars in monuments." In *Proceedings of the Seventh International Congress on Deterioration and Conservation of Stone, Lisbon, Portugal, 15-18 June 1992*, eds. J. Delgado Rodrigues, Fernando Henriques, and F. Telmo Jeremias, 763-770. Lisbon: Laboratorio Nacional de Engenharia Civil.

Ramachandran, V.S. and R.F. Feldman. 1984. "Chapter 1: Cement science." In *Concrete Admixtures Handbook.* New Jersey: Noyes Publications.

Research And Technical Advisory Service. *Grouting: ancient monuments technical note # 14.* Great Britain: Department of the Environment, Directorate of Ancient Monuments.

Ritchie, A.G.B. 1965 "The rheology of cement grout." in *Cement and Lime Manufacture*: January.

Roby, Thomas, C 1996. "Site conservation during excavation: stabilization and consolidation of roman funerary monuments in Carthage." In *Archaeological Conservation and its Consequences: Preprints of the Contributions to the Copenhagen Congress, 26-30 August 1996*, eds. Ashok Roy and Perry Smith, 149-152. London: The International Institute for Conservation of Historic and Artistic Works.

Roby, Thomas, C 1995. "Site conservation during excavation: treatment of masonry, wall plaster and floor mosaic remains of a Byzantine church In Petra, Jordan." In *Conservation and Management of Archaeological Sites*. Volume 1, No 1: 43-57.

Roby, Thomas, C. 1993. "Consolidation of a floor mosaic during the excavation of a Byzantine church in Petra, Jordan." In *Proceedings of the Fifth Conference of the International Committee for the Conservation of Mosaics, Faro E Conimbriga, 1993*, 1-19.

Rua, C. Rajer, A. and N. Mostacedo. 1993. "A case study of the conservation/restoration of the seventeenth century adobe church at Carabuco, Boliva." In *Seventh International Conference on the Study and Conservation of Earthen Architecture, Lisbon, 1993*, 205-209. Lisbon: DGEMN.

Schäfer, J. and H.K. Hilsdorf. "Ancient and new lime mortars-the correlation between their composition, structure, and properties." In *Conservation of Stone and Other Materials, Volume 1: Causes of Disorders and Diagnosis, June 29-July 1, 1993, Paris*, ed., M.-J. Thiel. London: E & FN Spon.

Schnabel, Lorraine and Glenn Boornazian. 1992. "The conservation of the mudejar templete at the Royal Monastery of Our Lady of Guadelupe, Extremedura (Caceres), Spain." In *Conservation of the Iberian and Latin American Cultural Heritage, Preprints of the Contributions to the Madrid Congress, 9-12 September, 1992*, eds. H.W.M. Hodges, John S. Mills and Perry Smith, 140-144. London: The International Institute for Conservation of Historic and Artistic Works.

Schwartzbaum, Paul M, Ippolito Massari, Giovanna Pignatelli, and Carlo Giantomassi.1986 "Approaches to the conservation of mural paintings in underground structures." In *International Symposium on the Conservation and Restoration of Cultural Property: Conservation and Restoration of Mural Paintings (I) November 17-21, 1983, Tokyo, Japan*, eds. Y. Emoto and S. Miura, 41-56. Japan: Tokyo National Research Institute of Cultural Properties.

Schwartzbaum, Paul M., Wannipa Na Songkhla, and Ippolito Massari. "The conservation of mural paintings in Thailand," In *Case Studies in the Conservation of Stone and Wall Paintings, Preprints of the IIC Conference, Bologna, 21-26 September, 1986*, eds. N.S. Brommelle and Perry Smith, 90-95. London: The International Institute for Conservation of Historic and Artistic Works.

Shugar, Gershon J. and Jack T Ballinger. 1990. *Chemical Technicians' Ready Reference Handbook.* 3rd ed. New York: McGraw-Hill, Inc..

Sickels, Lauren-Brooke. 1982. "Organics vs. synthetics." In *Mortars, Cements and Grouts Used in the Conservation of Historic Buildings, Proceedings of the Symposium, November 3-6, 1981.* 25-52. Rome: ICCROM.

Silver, Constance S. 1987. Architectural Finishes of the Prehistoric Southwest: A Study of the Cultural Resource and Prospects for its Conservation. Thesis; Graduate Program In Historic Preservation, Columbia University.

Silver, Constance S.1990. "Analyses and Conservation of Pueblo Architectural Finishes in the American Southwest." In *6th International Conference on the Conservation of Earthen Architecture, Adobe 90 Preprints,* 176-181. Los Angeles: Getty Conservation Institute.

Silver, Constance S and Joel Snodgrass. 1993. "A program for the conservation of prehistoric mural paintings on mud renderings on the American Southwest.," In *Seventh International Conference on the Study and Conservation of Earthen Architecture, Lisbon, 1993,* 205-209. Lisbon: DGEMN.

Smith, Watson. 1952. *Kiva Mural Decorations at Awatovi and Kawaika-a, with a Survey of Other Wall Paintings in the Southwest.* Papers of the Peabody Museum of American Archaeology and Ethnology, Vol. XXXVII. Cambridge: Harvard University Press.

Stewart, John and Moore, James. 1982. "Chemical techniques of mortar analysis." In *Mortars, Cements and Grouts Used in the Conservation of Historic Buildings, Proceedings of the Symposium, November 3-6, 1981.* 297-310. Rome: ICCROM.

Sumanov, L., R. Staniseva, V. Trajkoska, D. Danilovski, and V. Sendova. 1995. "Lime mortar and lime based mixtures for conservation, restoration, repair, and strengthening of Byzantine churches (9th-14th century) in Republic of Macedonia." In *Methods of Evaluating Products for the Conservation of Porous Building Materials* In *Monuments: International Colloquium, Rome 19-21 June 1995.* Rome: ICCROM.

Teutonico, Jeanne Marie. 1988. *Arc A Laboratory Manual For Conservators.* Rome: ICCROM.

Teutonico, Jeanne Marie, Iain McCaig, Colin Burns, and John Ashurst. 1994. "The Smeaton Project: factors affecting the properties of lime-based mortars." In *Bulletin of the Association for Preservation Technology (APT),* Volume 25, No. 3-4.

The British Quarrying and Slag Federation Ltd. *Lime* In *Building.* London: The British Quarrying and Slag Federation Ltd.

Tomazevic, P.W., T. Velechovsky, V Apih. 1991. "The strengthening of stone masonry walls with grouting," In *Structural Repair and Maintenance of Historic Buildings II, Volume II: Dynamics, Stabilization and Restoration, Proceedings of the Second International Conference, Seville, Spain, 14-16 May 1991,* eds. C.A. Brebbia, J Dominguez, and F. Escrig, 215-225. Southhampton Boston: Computational Mechanics Publications.

Trautwine, John C. 1937. *The Civil Engineer's Reference-Book.* London: Chapman & Hall, Ltd.

Twilley, John and Jerry C. Podany. 1986 "Deterioration of Arizona red sandstone and refinement of methods for its treatment." In *International Symposium on the Conservation and Restoration of Cultural Property: Conservation and Restoration of Mural Paintings (I) November 17-21, 1983, Tokyo, Japan*, eds. Y. Emoto and S. Miura, 41-56. Japan: Tokyo National Research Institute of Cultural Properties.

Utley, Robert M. 1962. *Fort Union National Monument.* Historical Handbook No. 35. Washington, D.C.: National Park Service, U.S. Department of the Interior.

Van Nostrand's Scientific Encyclopedia. 1988. (7th ed.) ed. Douglas M Consindine. New York: Van Nostrand Reinhold.

Wagner, A.A. 1957. *Proceedings of the Fourth International Conference SMFE, London,* Volume 1. London: Butterworth and Co.

Washa, G.W. 1966. "Volume Changes." In *Significance of Tests and Properties of Concrete and Concrete Making Materials,* ASTM Technical Publication No. 169-A, 189-201. Philadelphia PA: American Society for Testing and Materials.

Wingate, Michael. 1988. "An introduction to building limes," In *SPAB News,* Volume 9, No. 2.

Wingate, Michael. 1992. "Standards for building limes," In *Lime and Other Alternative Cements,* eds Neville Hill, Stafford Holmes, and David Mather, 229-236. London: Intermediate Technology Publications Ltd.

Wisser, Stefan, and Karin Kraus. 1988. "Composition and properties of historic lime mortars." In *Proceedings of the Sixth International Congress on Deterioration and Conservation of Stone, Torun, 12-14, September 1988.* Nicholas Copernicus University Press.

Wright, P.J.F. 1955. "Comments on an indirect tensile test on concrete cylinders." In *Magazine of Concrete Research,* Volume 7, No 20.

Zepnik, R. and W. Schönbrodt-Rühl. 1989. "Application of cement grouting to historic buildings." In *Structural Repair and Maintenance of Historic Buildings,* ed C.A. Brebbia, 171-178. Birkhauser: Computational Mechanics Publications.

Index

R

S

T

V

W