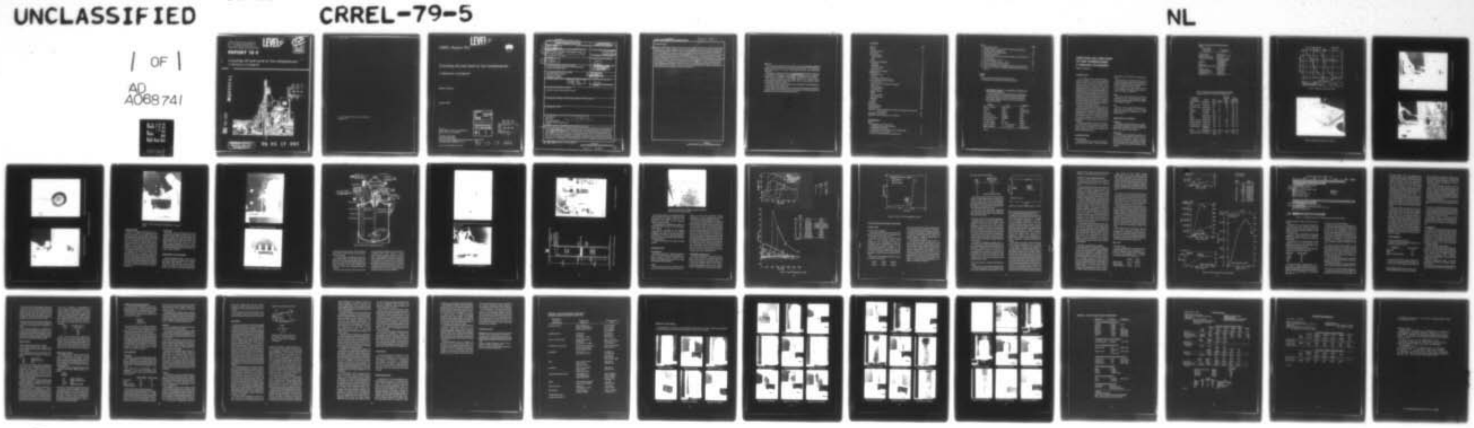
AD-A068 741 COLD REGIONS RESEARCH AND ENGINEERING LAB HANOVER NH F/G 11/2
GROUTING SILT AND SAND AT LOW TEMPERATURES, A LABORATORY INVEST--ETC(U)
MAR 79 R JOHNSON

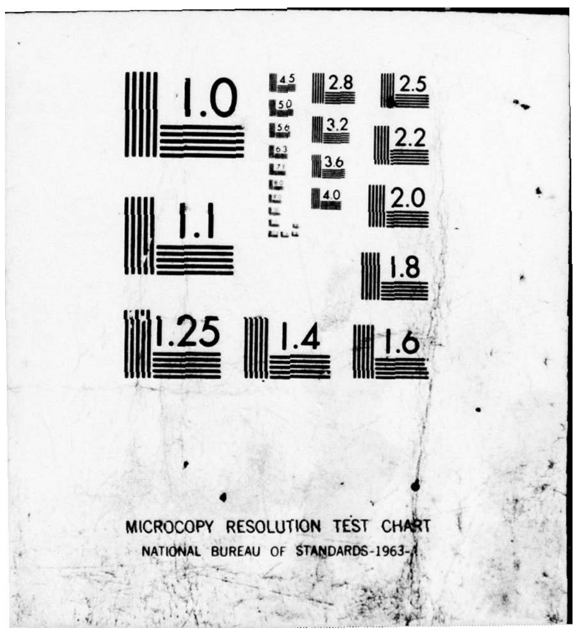
UNCLASSIFIED CRREL-79-5 NL

1 OF 1

AD
A068741



END
DATE
FILMED
7--79
DDC


1.0
1.1
1.25
1.4
1.6
1.8
2.0
2.2
2.5
2.8
3.2
3.6
4.0
MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

CRREL

LEVEL II

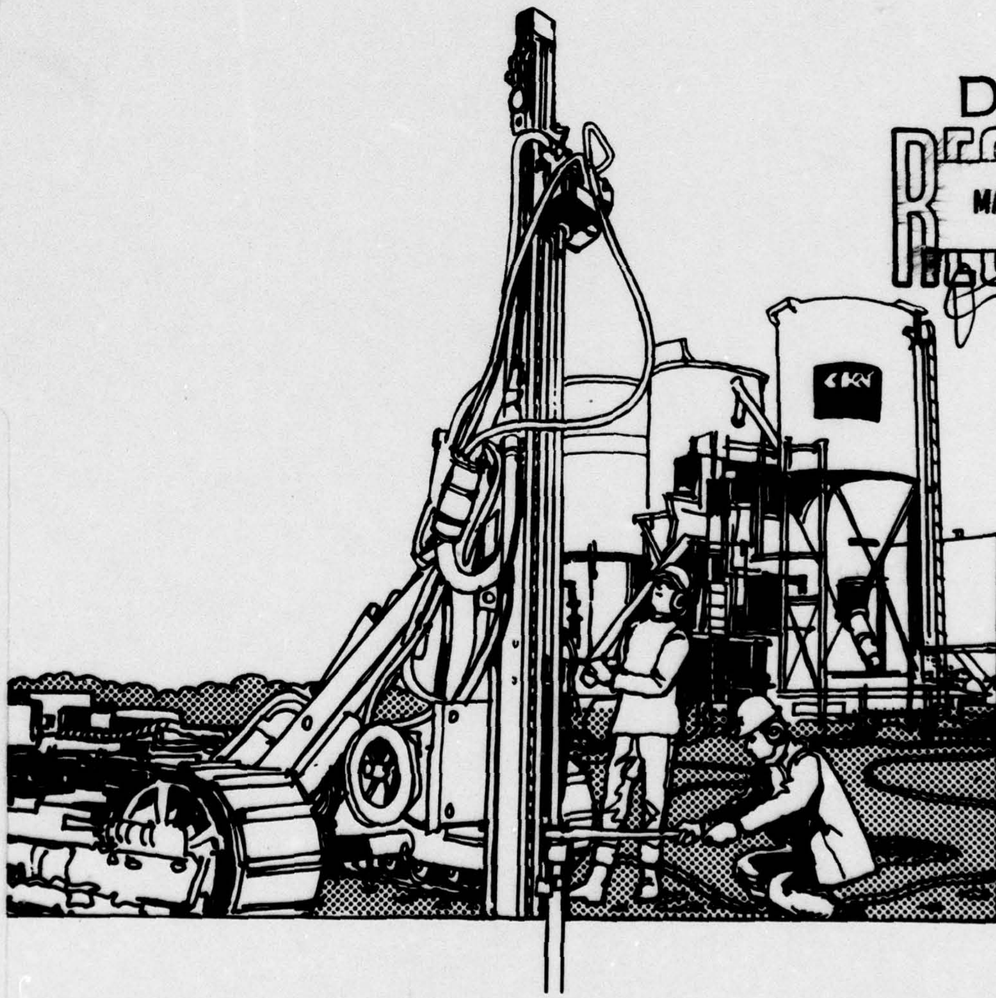
REPORT 79-5

Grouting silt and sand at low temperatures

A laboratory investigation

AD A068741

DDC FILE COPY



DDC
RECEIVED
MAY 18 1979
D

DISTRIBUTION STATEMENT A
Approved for public release;
Distribution Unlimited

79 05 17 001

Cover illustration courtesy of GKN Keller Ltd., Coventry, U.K.

LEVEL II

CRREL Report 79-5

Grouting silt and sand at low temperatures

A laboratory investigation

Robert Johnson

March 1979

ACCESSION for
NTIS White Section ☒
DDC Buff Section ☐
UNANNOUNCED ☐
JUSTIFICATION........
BY........
DISTRIBUTION/AVAILABILITY CODES
Dist. AVAIL. and/or SPECIAL
A

DDC
RECEIVED
MAY 18 1979
D

Prepared for
DIRECTORATE OF MILITARY PROGRAMS
OFFICE, CHIEF OF ENGINEERS
By
UNITED STATES ARMY
CORPS OF ENGINEERS
COLD REGIONS RESEARCH AND ENGINEERING LABORATORY
HANOVER, NEW HAMPSHIRE, U.S.A.

79 05 17 001

Approved for public release; distribution unlimited.

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER
CRREL [redacted]-79-5

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle)
GROUTING SILT AND SAND AT LOW TEMPERATURES, A Laboratory Investigation

5. TYPE OF REPORT & PERIOD COVERED
A2

6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s)
Robert Johnson

8. CONTRACT OR GRANT NUMBER(s)

9. PERFORMING ORGANIZATION NAME AND ADDRESS
U.S. Army Cold Regions Research and Engineering Laboratory
Hanover, New Hampshire 03755

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
DA Project 4A762730AT42
Task A2, Work Unit 001

11. CONTROLLING OFFICE NAME AND ADDRESS
Directorate of Military Programs
Office, Chief of Engineers
Washington, D.C. 20314

12. REPORT DATE
Marc[redacted]979

13. NUMBER OF PAGES
37

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)
39p.

15. SECURITY CLASS. (of this report)
Unclassified

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)
Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)
Chemical grouts
Grout slurry
Injection grouting of soil
Unconfined compressive strength
Viscosity

39 + or - 1F (3.88C)

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report presents data from an experimental program undertaken to develop information on proposed and existing chemical grout solutions to provide engineering properties in connection with grouting of soils in ambient temperatures of 39° ±1° F (3.88°C) and below. Twelve grout solutions were investigated, including organic chemicals, sodium silicates, cements, and clay (bentonite). Set or gel tests were performed on each chemical solution, in the center of which a thermocouple was placed for measuring the rates of heat of reaction, except those of the cements and the clay. These solutions consisted of the mixing portions of the chemicals and were placed into 4-in.-diam by approximately 6-in.-long cylinders at an ambient temperature of 39°F. The components of each chemical were also (over)

DD FORM 1473, 1 JAN 73 EDITION OF 1 NOV 65 IS OBSOLETE

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

037 100

(sq in. L39F)

20. Abstract (cont'd)

tempered to 39°F temperature before the mixing. Most of the solutions were tested at this temperature except those that did not seem to become too viscous to use as a grout material for injection into soils. Other set or gel tests were performed at approximately -26°F for one lignin based product; the results of all set-tested products are presented in terms of their dependence on temperature and time. Cylindrical samples of sand and silt soil, 2.75 in. diameter by 7 in. long, were then pressure injected at 20 lb/in.^{2} (39°F ambient), and the soil samples were allowed to set overnight, unless otherwise stated, before being soaked in a 39°F water-bath for 7 days to check the integrity of the injected samples. Samples that were not dissolved were taken from the 7-day water-bath and were then subjected to unconfined compressive strength tests. The stress-strain data obtained are presented. Seventy-two soil samples were used during the investigation. As the injection process progressed, some materials and soil type samples were eliminated as the ambient temperatures were lowered. Unconfined compressive strength tests revealed strengths of pressure-injected samples from 3 lb/in.^{2} to 1624 lb/in.^{2} The grout material that stressed to 1624 lb/in.^{2}, a urethane, was also successfully injected into a frozen sample in the final part of the program.

(3 lb/sq in.) to (1624 lb/sq in.)

PREFACE

This study was conducted and this report was prepared by Robert Johnson, formerly Research Civil Engineer, Geotechnical Research Branch, Experimental Engineering Division, U.S. Army Cold Regions Research and Engineering Laboratory.

The study was funded under DA Project 4A762730AT42, *Design, Construction and Operations Technology for Cold Regions;* Task A2, *Soils and Foundations Technology for Cold Regions;* Work Unit 001, *Design Criteria for Foundations.*

The technical reviewers of this report were Dr. R. Torence Martin, Massachusetts Institute of Technology, and Dr. Omar T. Farouki, Queen's University of Belfast, Northern Ireland.

The author wishes to thank Frederick Crory of CRREL for his professional advice and review of the manuscript.

The contents of this report are not to be used for advertising or promotional purposes. Citation of brand names does not constitute an official endorsement or approval of the use of such commercial products.

CONTENTS

	Page
Abstract	i
Preface	iii
Conversion factors	v
Introduction	1
Materials tested	1
Grout materials	1
Sand	1
Silt	1
Description of equipment	1
Coldroom	1
Water-bath	1
Cylindrical molds	6
Batch vessel	6
Preparation of soil samples	6
Methods of test	11
Penetration test	11
Set test	11
Unconfined compressive test	11
Tests of chemicals or grout solutions	13
Urethane resins	13
Injection of frozen sample with urethane	15
Epoxy resin	15
Urea-formaldehyde	18
Acrylamide gel	18
Sodium silicates	19
SIROC	19
Celite 55 Terraset	20
Chrome-lignin gel	20
Bentonite	20
Cements	20
Discussion	21
Conclusion	22
Recommendations	22
Literature cited	23
Appendix A. Grouts and chemical solutions, manufacturers and personnel contacted	25
Appendix B. Test samples	27
Appendix C. Grout or solution cost information	31

ILLUSTRATIONS

Figure	
1. Gradation curves of soils used	3
2. Water-bath maintained in coldroom	3
3. Assemblage of test mold	4
4. Table construction and placement of sample molds	6
5. Placement of scatter shield	7
6. Setup of injection apparatus,excluding soil	7

Figure		Page
7.	Binks pressure tank	8
8.	Sample being placed into pail of water before being injected	9
9.	Sample cylinder including soil	9
10.	Apparatus for unmolding samples	10
11.	Unmolding apparatus	10
12.	Cylinder molds with thermocouples centered in mixed chemical or grout solutions	11
13.	Set test temperature vs time	12
14.	Set test of urethane resin at −32.3°C	14
15.	Stress-strain graphs of tested samples	16
16.	Unconfined compressive strength bar graph of injected samples	17
17.	Mohr's Circle of stress for the highest strength gained by pressure injecting	21

TABLES

Table		
I.	Grout materials and chemical solutions tested	2
II.	Properties of various pressure injected soil samples	2

CONVERSION FACTORS: U.S. CUSTOMARY TO METRIC (SI) UNITS OF MEASUREMENT

These conversion factors include all the significant digits given in the conversion tables in the ASTM *Metric Practice Guide* (E 380), which has been approved for use by the Department of Defense. Converted values should be rounded to have the same precision as the original (see E 380).

Multiply	*By*	*To obtain*
gallon (U.S. liquid)	0.003785412	$meter^3$
centipoise	0.001*	pascal second
stokes	0.0001*	$meter^2/second$
$pound/inch^2$	6894.757	pascal
$pound\text{-}force/foot^2$	47.88026	pascal
$gram\text{-}force/centimeter^2$	98.06650*	pascal
$dyne/centimeter^2$	0.1*	pascal
ton (short, 2000 lb)	907.1847	kilogram
degrees Fahrenheit	$t_K = (t_{°F}+459.67)/1.8$	kelvins
degrees Fahrenheit	$t_{°C} = (t_{°F}-32)/1.8$	degrees Celsius

*Exact.

GROUTING SILT AND SAND AT LOW TEMPERATURES
A Laboratory Investigation

Robert Johnson

INTRODUCTION

In the northern states, including Alaska, a sizeable amount of construction engineering must be carried out under low temperature conditions because of the short summer season. Under these climatic conditions, unless grout materials produce required heat for grout setting properties, viscosity and setting problems of fluid and grout slurry materials will result. The need to exclude the application of heat, which is important in conserving energy, leads to the requirement for grouts to which heat will not have to be added before or during their use. The grout materials covered in this paper were tested for use in the stabilization of naturally or artificially thawed soils at low temperatures.

Previous to this paper, a literature search was conducted (Johnson 1977) to develop information on methods and grout materials for low temperature use. The literature search produced information on a variety of soil grout materials that could be used in warm climates, but it did not produce information on the effects of low temperatures on the properties of these grout materials.

The data reported here contain the results of tests conducted on grout materials at low temperatures and are preliminary to further laboratory testing as well as field testing in soils above the permafrost table.

MATERIALS TESTED

Grout materials

The grout materials and chemical solutions tested are listed in Table I. The soils used were sand and silt, as described in the following sections.

The base grout or chemical solution of successfully pressure-injected and set or gelled soil samples investigated, and the unconfined compressive strengths are listed in Table II.

The manufacturers of the materials used in this investigation, with addresses and points of contact, are listed in Appendix A.

Sand

Farrel sand. The sand used for these tests measured 5.6×10^{-3} cm/sec hydraulic conductivity (permeability) at 39°F (3.9°C). Its gradation curve is shown in Figure 1.

Silt

Jenks silt. The silt used for these tests is a sandy silt that measured 5.15×10^{-4} cm/sec permeability at 39°F. Its gradation curve is also shown in Figure 1.

DESCRIPTION OF EQUIPMENT

Coldroom

The equipment and materials were maintained and used in coldrooms in which the ambient temperatures were 39° ± 1°F, 33° ± 0.9°F, and 20° ± 1°F, unless otherwise stated.

Water-bath

A water-bath was maintained in the 39°F coldroom for soaking injected samples (Fig. 2). Samples were immersed in water for 7 days, unless otherwise stated, to determine whether the initial set product would dissolve or otherwise be affected by complete immersion of the samples.

Table I. Grout materials and chemical solutions tested.

Name and/or no. of grout or chemical solution	*Type of solution of system*
EP Systems and A-11	Urethane resin
XB-2403 Mud Lock	Hydrophilic urethane resin
Epotuf 37-130 and 37-052	Epoxy resin
Ancamine AD, LT and MCA	Curing agents (used with above epoxy resin)
Raylig-260 L	Chrome-lignin gel
AM-9	Acrylamide gel
Cyanaloc 62	Urea-formaldehyde
Hayward Baker Sodium Silicate	Sodium silicate
Siroc	Sodium silicate
Celtite 55 Terraset	Sodium silicate
Portland Cement Type I	Cement slurry
Portland Cement Type III	Cement slurry
Bentonite Clay	Bentonite slurry

Table II. Properties of various pressure injected soil samples.
(All samples were 100% water saturated except sample #I-66.)

Name and/or no. of grout or chemical solution	*Type of solution*	*Type of soil*	*Sample*	*Unconfined compressive strength (psi)*	*Ambient temperature (°C)*	*(°F)*
Epotuf	Epoxy resin	Sand	37	488	3.88	39
EP 65-92, 93 and 18	Urethane resin	Sand	35	305	3.88	39
EP 65-92, 93 and 18	Urethane resin	Silt	51	120	3.88	39
Celtite	Sodium silicate	Silt	53	15	3.88	39
Raylig-260 L	Chrome-lignin	Sand	26	8	3.88	39
AM-9	Acrylamide	Sand	7	8	3.88	39
Celtite	Sodium silicate	Sand	16	7	3.88	39
SIROC	Sodium silicate	Sand	20	3	3.88	39
EP 65-12, 17 and 18	Urethane resin	Sand	65	1624	0.55	33
EP 65-92, 93 and 18	Urethane resin	Sand	63	219	0.55	33
EP 65-92, 93 and 18	Urethane resin	Silt	61	164	0.55	33
XB-2403 Mud Lock	Hydrophilic urethane resin	Sand	69	115	0.55	33
AM-9	Acrylamide	Silt	70	51	0.55	33
EP 65-12, 17 and 18	Urethane resin	Frozen sand	I-66	306	−6.66	20

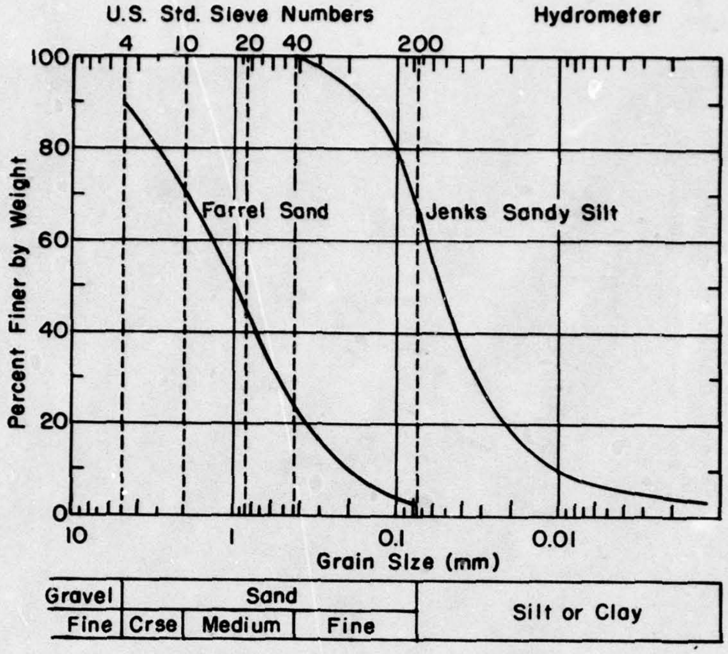


Figure 1. Gradation curves of soils used.

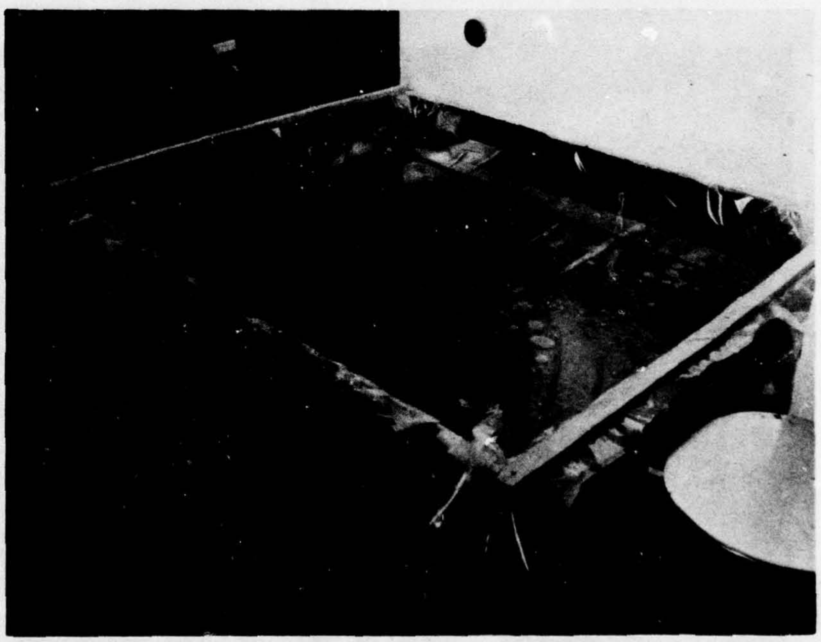
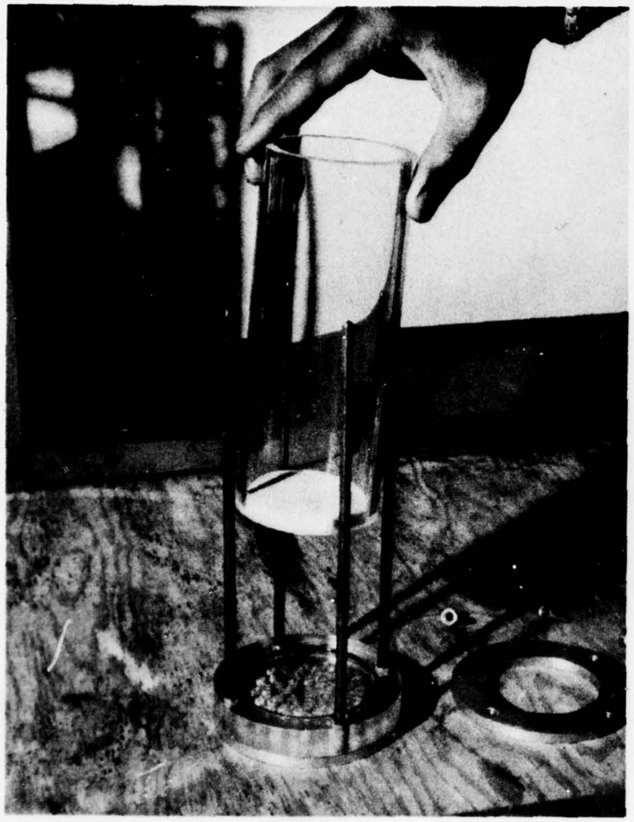
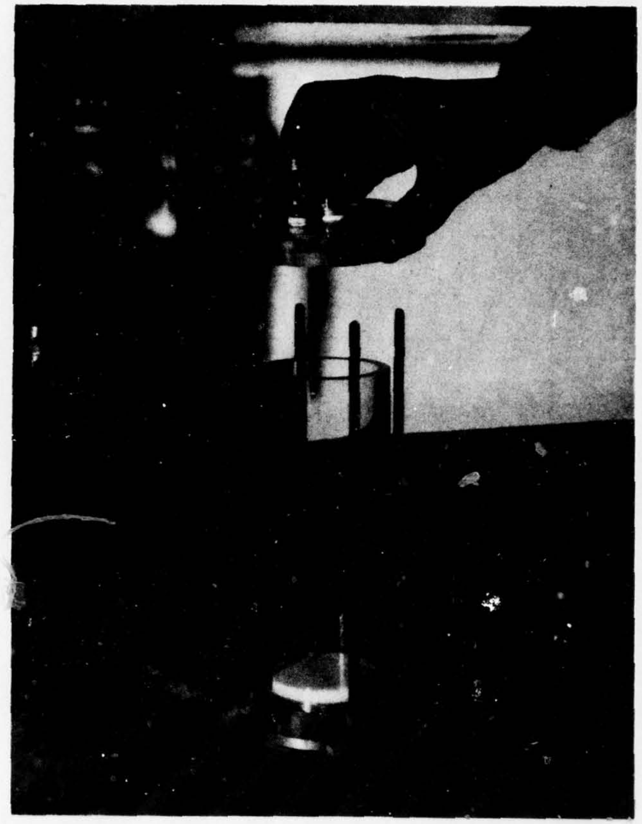


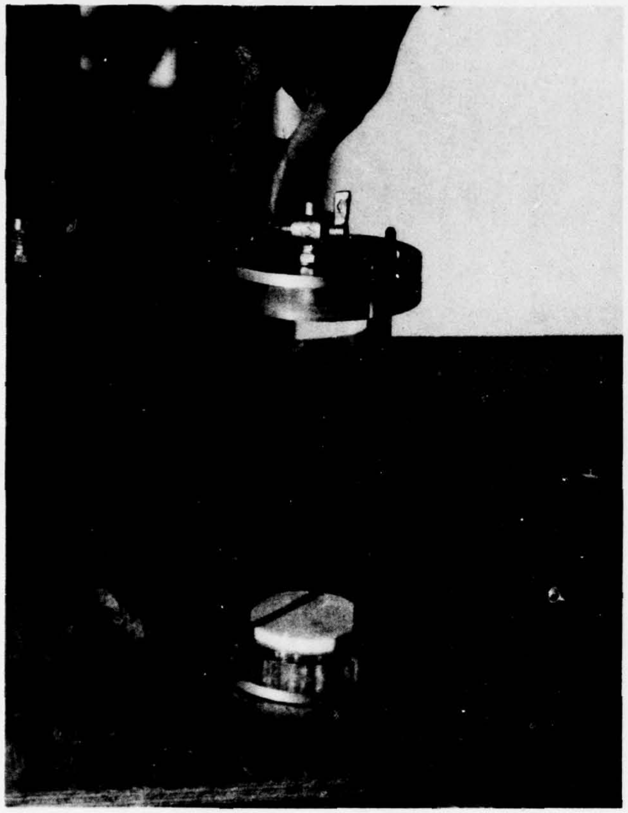
Figure 2. Water-bath maintained in coldroom.



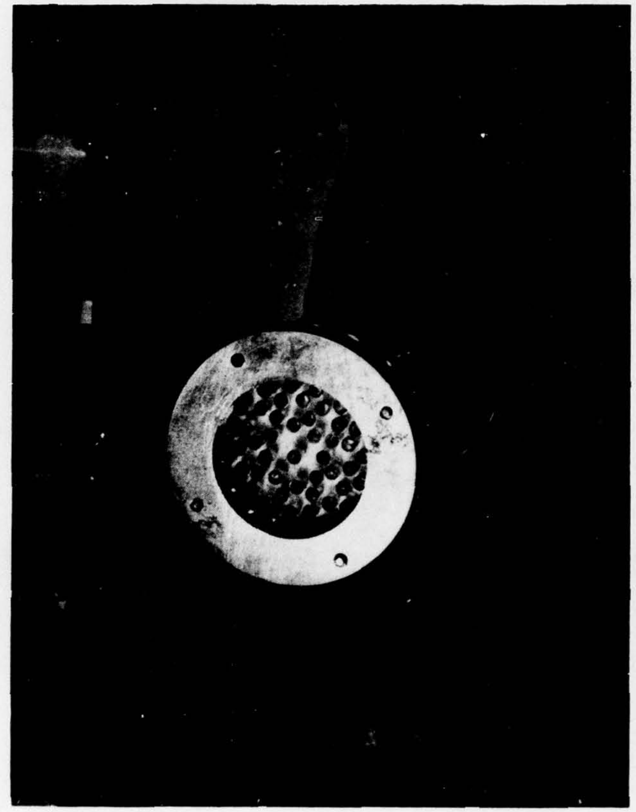
a. Injection cylinder center.



b. End caps, top and bottom.



c. Rings and clamps (wing nuts).



d. Bottom view of test cylinder with solution exit holes.

Figure 3. Assemblage of test mold.

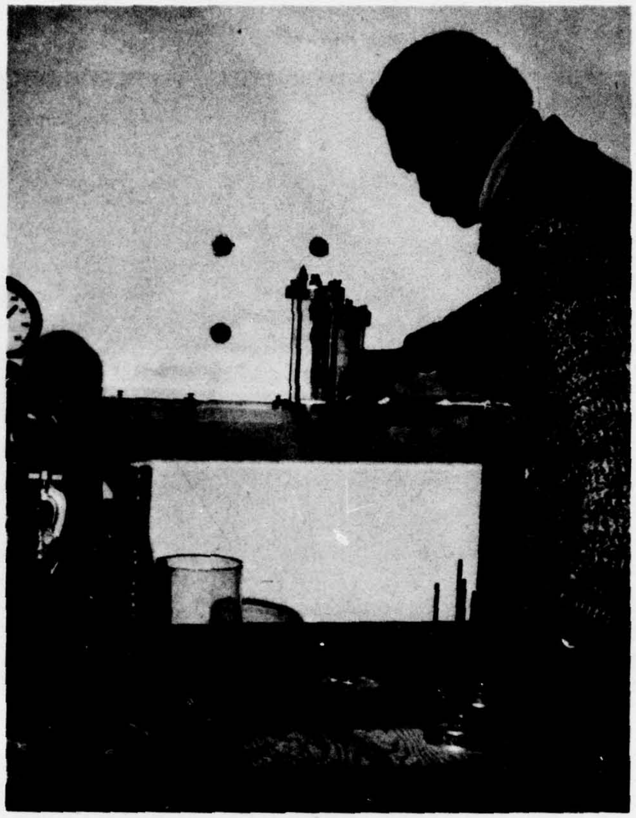


Figure 4. Table construction and placement of sample molds.

Cylindrical molds

Cast acrylic cylinders, approximately 3$^1/_4$ in. OD and 2$^{11}/_{16}$ in. ID by 8 in. long were used to mold the samples. The cylindrical molds were held together with threaded ready rods holding end caps and rings, fastened with wing nuts. The top of each mold was constructed so that a pressure injection line could be connected and fastened to the top center of each mold. The bottom of each cylindrical mold included exit holes for the pressurized grout solutions. The assemblage of the test molds is shown in Figure 3. Assembled test molds were placed into clamped positions on a specially constructed table that was made to hold the test molds while the soil samples were pressure injected (Fig. 4). After the sample mold was placed and clamped into position, to keep the sample from scattering if the injection pressure exceeded the strength of the mold, a shield was then placed over the mold (Fig. 5).

Batch vessel

The vessel used to pressure-inject the soil samples was a two-gallon standard-pressure top-outlet fluid tank with an air motor agitator and pressure regulator. The supply line pressure was approximately 105 psi and the tank safety valve pressure was 120 psi. The setup of the tank (batch vessel) is presented in Figure 6, and a combined diagrammatic sketch and data sheet of the manufacturer (Binks Manufacturing Company) is shown in Figure 7.

PREPARATION OF SOIL SAMPLES

The inside diameter surface of each of the cylinders in which the samples were prepared was coated with Simoniz® wax, by Texize Chemical Co. Then the Farrel sand was put into molds at 110 lb/ft^3 dry density and the Jenks sandy silt at 80 lb/ft^3 dry density before being

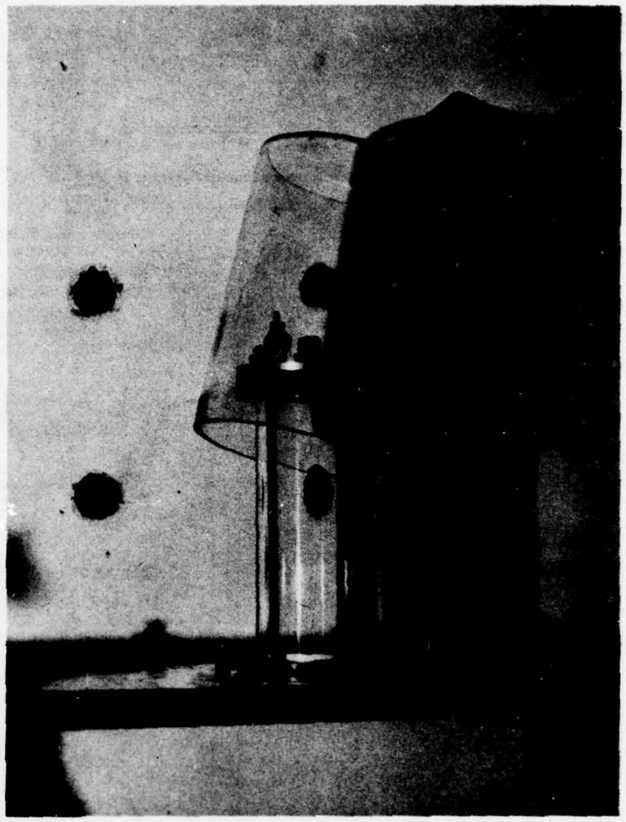


Figure 5. Placement of scatter shield.

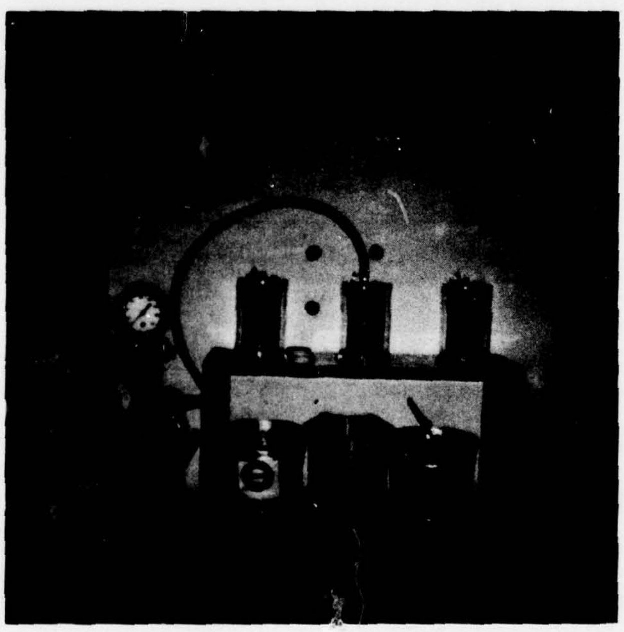


Figure 6. Setup of injection apparatus, excluding soil.

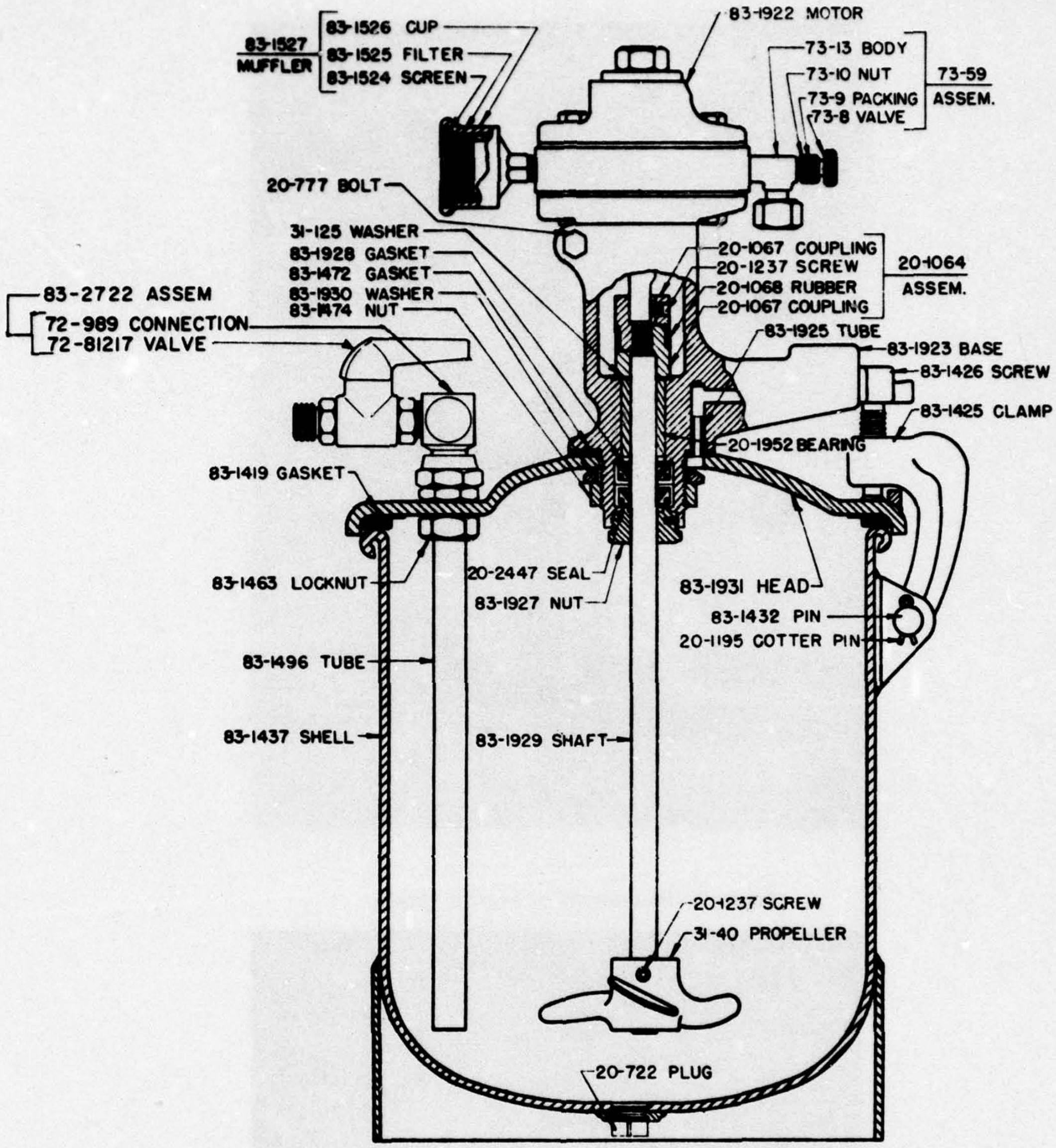


Figure 7. Binks (batch vessel) pressure tank.

saturated with distilled water. The dry densities were chosen as representative of frozen ground which has thawed.

All soil samples were prepared in the cylindrical molds without end caps and clamps. The weight of soil for the given dry density was placed by spoon into the mold. Then, the side of the mold was tapped until the soil settled to the chosen dry density and a designated length of cylinder.

The maximum saturation, before the samples were injected, was obtained by putting the soil cylindrical mold, which included a porous plastic (similar to porous stones) in its bottom section, containing the soil sample, into a pail of water at 39°F (3.9°C) or 33°F (0.5°C) and keeping the soil under a 1-in. head of water. A sample being placed in a pail of water is shown in Figure 8. The sample was kept immersed in the water overnight to allow as much air as possible to rise through or out of the soil. Samples ready for pressure injection are shown in Figure 9.

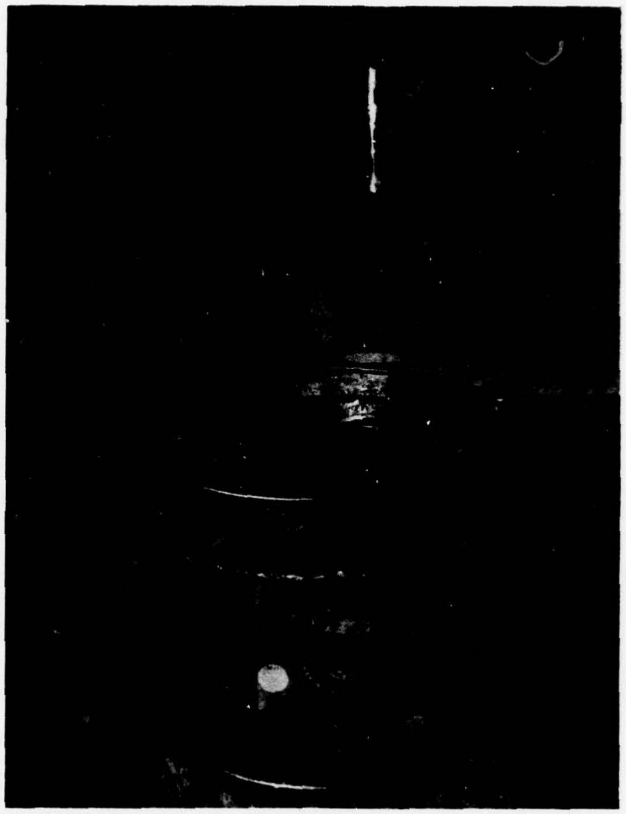


Figure 8. Sample being placed into pail of water before being injected.

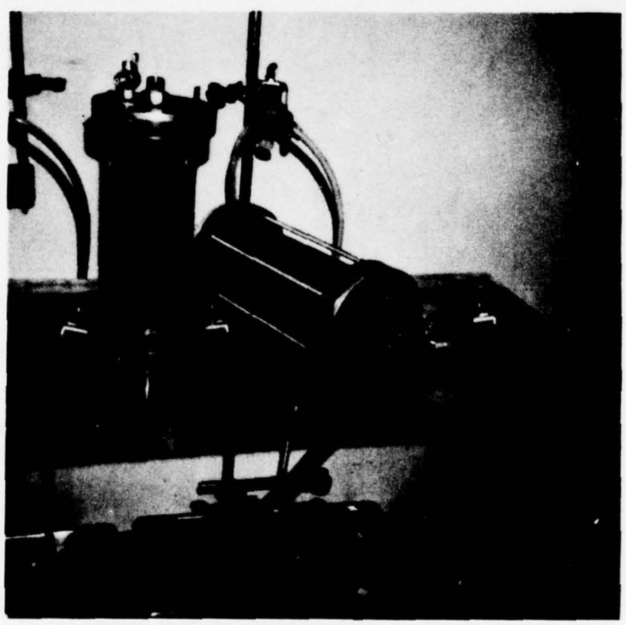


Figure 9. Sample cylinder including soil.

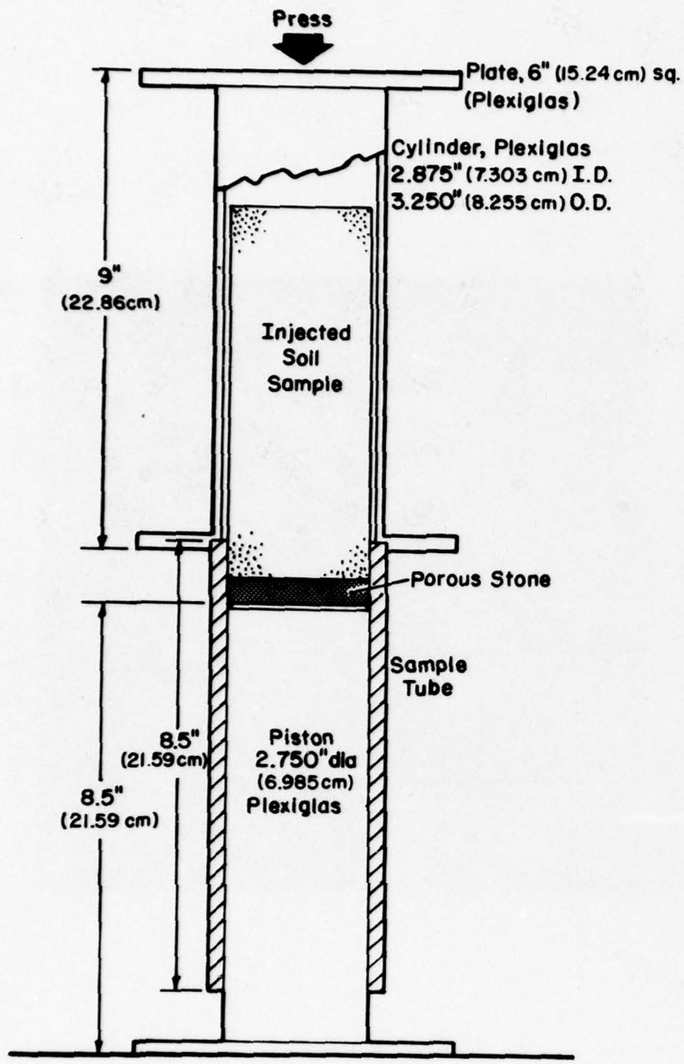


Figure 10. Apparatus for unmolding samples.

Figure 11. Unmolding apparatus.

Figure 12. Cylinder molds with thermocouples centered in mixed chemical or grout solutions.

Many of the samples were injected with 20-psi injection pressure. However, at times the injection pressures were higher or lower, as is indicated in the sections on the respective chemical or grout solution.

The samples were unmolded by using the apparatus described in Figure 10 and photographed in Figure 11.

All samples remained in the ambient temperature of 39°F (3.9°C) or 33°F (0.5°C) throughout the period of sample preparation and testing to the end of the 7-day period, at which time they were trimmed for the unconfined compression test.

A table model blender was used for mixing samples.

METHODS OF TEST

Penetration test

Penetration tests (ASTM - C403) were performed on set test samples to measure hardness of setting samples. However, samples were measured for hardness only when the set or gel was not produced immediately.

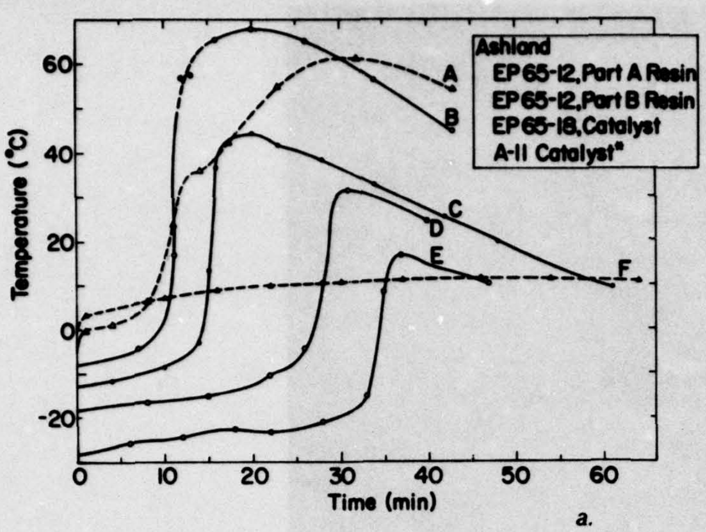
Set test

Set tests were performed to determine if the grout material would set or gel at 39°F (3.9°C). Further set tests were conducted at lower temperatures on the materials that showed potential after being pressure injected at 39°.

The set tests were performed in 4-in.-diam by approximately 6-in.-long cylindrical molds with thermocouples centered in the mixed chemical or grout solutions (Fig. 12). The thermocouples were wired to a data retrieval system which records or logs temperatures at controlled set intervals of 1 minute to 24 hours. In the tests conducted, the usual procedure was to initiate the test with the setting at 1-minute intervals, changing these gradually to 30-minute intervals as the liberation reaction heat began to fall slowly. For the chemical reactions that were not initiated until some preset time, data log setting time intervals were begun at every 1 minute and after a brief period were then reduced to every 5 minutes, until near reaction gel time, when 1-minute intervals were again selected.

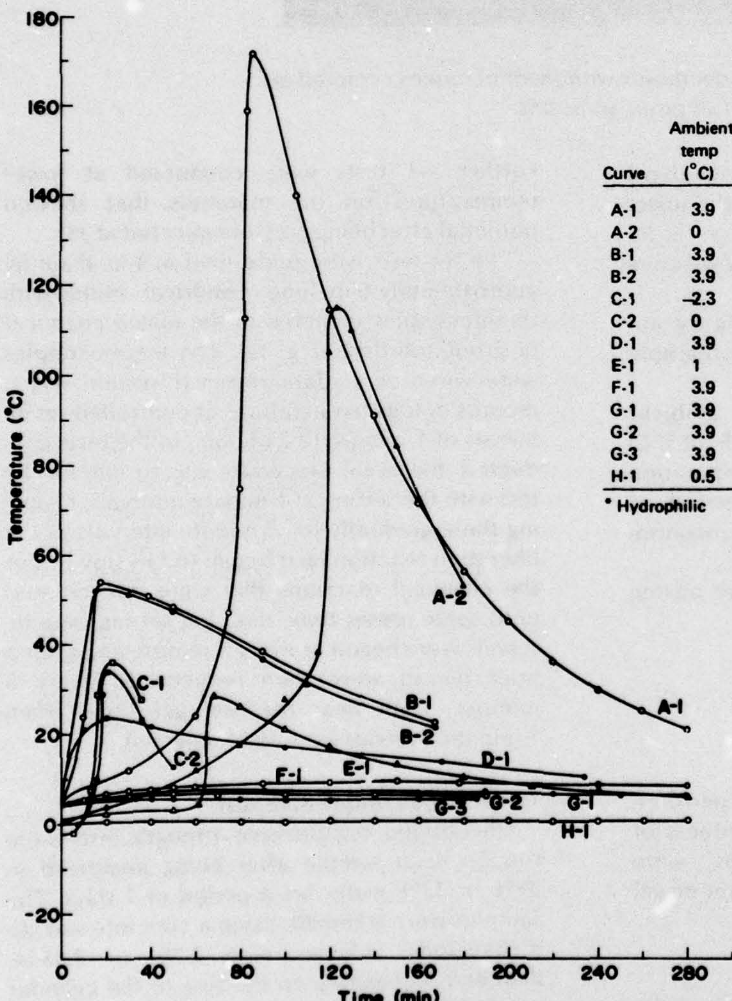
Unconfined compressive test

Unconfined compressive strength tests were run on each sample after being immersed in 39°F or 33°F water for a period of 7 days. The samples were trimmed, using a concrete saw, to a 6-in.-long cylinder with 2.75- or 2.85-in. diameter, depending on the size of the cylinder mold. All samples were strained at 0.1 cm/min.



Curve	Catalyst (%)	Ambient temp (°C)
A	0.54	– 1
B	1	– 7.6
C	1	–12.9
D	1	–18.0
E	4.1	–28.4
F*	1.5*	0.3

a.

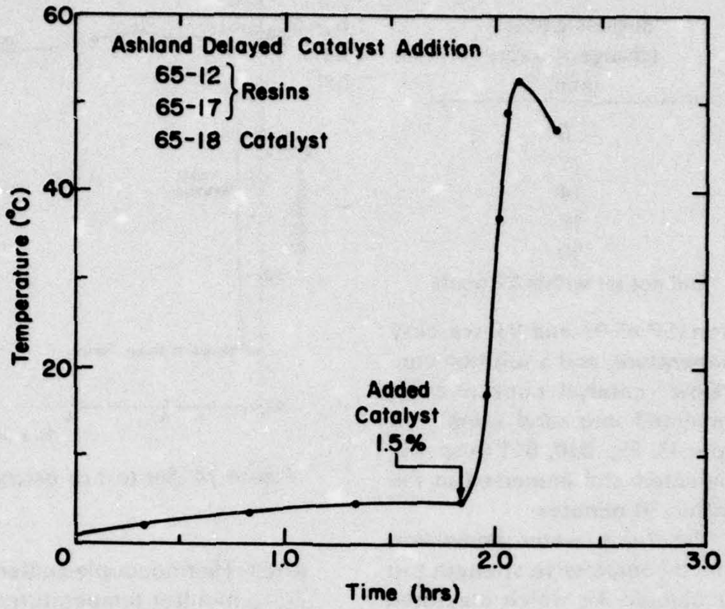


Curve	Ambient temp (°C)	Solution	Name
A-1	3.9	Epoxy Resin	EPOTUF
A-2	0	Epoxy Resin	EPOTUF
B-1	3.9	Urethane Resin	EP 65-92, 93 (1% EP 65-18)
B-2	3.9	Urethane Resin	EP 65-92, 93 (1% EP 65-94)
C-1	–2.3	Urethane Resin	EP 65-92, 93 (1.5% EP 65-18)
C-2	0	Urethane Resin	EP 65-92, 93 (1.5% EP 65-18)
D-1	3.9	Urethane Resin*	Mud Lock
E-1	1	Acrylamide	AM-9
F-1	3.9	Chrome-Lignin	Raylig 260-L
G-1	3.9	Sodium Silicate	Celtite
G-2	3.9	Sodium Silicate	Hayward Baker
G-3	3.9	Sodium Silicate	SIROC
H-1	0.5	Urea-Formaldehyde	Cyanaloc 62

* Hydrophilic

b.

Figure 13. Set test temperature vs time.



c.

Figure 13 (cont'd.). Set test temperature vs time.

TESTS OF CHEMICALS OR GROUT SOLUTIONS

Urethane resins

EP resin system (urethane)

This system includes urethane resins EP 65-92, EP 65-93, EP 65-12, EP 65-17, and catalysts EP 65-94 (fast), EP 65-18 (slow), and A-11.

According to the manufacturer, the EP resin system is a low viscosity urethane system suitable for soil consolidation in arctic environments, but may be obtained in more concentrated forms of higher viscosities. At the manufacturer-suggested percentage of 1% catalyst based on the mixed resin system, set tests were conducted at 39°F (3.9°C) ambient temperature using the formula below (see Fig. 13b).

This resin system (Ashland Chemical formula) by weight was as follows:

65-92	65-93	Catalyst
50 parts	50 parts	added %

Test results at 39°F (3.9°C) ambient temperature: Set test temperature graphs (Fig. 13b) show that with the slow catalyst the sample produced a maximum temperature of 128.8°F (53.8°C) in 20 minutes, and that with the fast catalyst the sample gained a maximum temperature of 126.1°F (52.3°C) in 18 minutes.

It was desired to obtain a longer time to initial set than 20 minutes, and because of the short periods of initial set or gel, further set tests were conducted using a simple cup test method and the slow catalyst; this consisted of set testing by adding various percentages of slow catalyst to 50-ml (cup) of the resin mix, and recording the set time. The time measurements began when the catalyst was added to the base solution and ended when the solution color began to change, indicating beginning of set, which occurred within seconds.

Data from this set are as follows:

Catalyst (%)	Beginning of set (change of color) (min)
1.5	8
1.25	11
1	14
¾	18
½	30
¼	Did not set within 27 hours

This resin solution (EP 65-92 and 93) waseasy to inject at this temperature, and a solution consisting of 1/2% "slow" catalyst concentration was successfully injected into sand sample 35 [see photo of sample 35, Fig. B10, B11 (App. B)]. This sample was injected and immersed in the 39°F water-bath within 30 minutes.

At the end of the 7-day water immersion period, an unconfined compressive strength test was conducted on sample 35, which measured 304.6 psi (21.93 ton/ft²).

Test results at 33°F (0.5°C) ambient temperature

Because of the ease at which sample 35 was injected (resulting in a solid set sample), it was desired to know how the lower ambient temperature would affect the set and viscosity by visual inspection. The simple cup method set test was performed using the above formula with 1.5% catalyst. The catalyst was increased to 1.5% to ensure a set at the lower temperature. The set time was the same as that at the 39°F ambient temperature and the viscosity appeared to be the same, indicating no significant temperature effect.

Unconfined compressive strength tests, at 33°F (0.5°C), measured 219 psi (15.76 tons/ft²) for sand sample 63 and 164 psi (11.90 tons/ft²) for silt sample 61. Problems were not encountered at this temperature. However, this lower soil temperature may have acted as a heat sink and prevented the catalyst from producing a high enough heat for the grout material to gain strength, results similar to those obtained with samples tested at 39°F.

Test results at below +32°F (0°C) ambient temperature

Further tests, at lower ambient temperatures, were conducted because the resin solution is not an aqueous solution that would freeze at 32°F

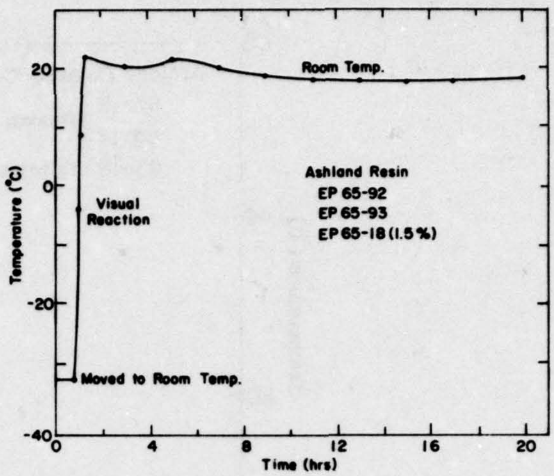


Figure 14. Set test of urethane resin at −32.3°C.

(0°C). Thermocouple-centered samples were used to monitor temperatures below +32°F.

Samples of base components EP65-92 and EP 65-93 with EP 65-94 and EP 65-18 ("fast" and "slow" catalysts) were placed in a −28.66°F (−33.7°C) ambient temperature. The "fast" catalyst immediately froze. None of the other components froze within an hour and ten minutes, at which time the EP 65-92 component temperature became stable at a thermocouple-measured temperature of −25.86°F (−32.3°C). Visual inspection showed that the components became only slightly more viscous at this temperature. However, the components including 1.5% catalyst at the stable temperature did not produce a reaction within the 50 minutes.

The sample was moved to room temperature (20°C) and the temperature rose to 23.18°F (−4.9°C) within 11 minutes. At this temperature, the color began to change (visual reaction), indicating start of set, but only the top portion of the sample reacted and set, resulting in approximately half of the sample remaining in liquid form (see Fig. 14). Perhaps settling of component materials of different specific gravities affected the blend of the mixture while sitting undisturbed over the 50-minute period. Because some of the components did not freeze at such low temperatures, further testing of the urethane resin system was considered desirable. A more concentrated system than was previously used, EP 65-12 and EP 65-17 (50-50 by volume) including the 65-18 "slow" catalyst, was further set tested. Results are presented in Figures 13a and 13c.

Injection of frozen sample with urethane

The resin system used for the frozen injected soil sample was EP 65-12 and EP 65-17 (50-50 by volume), including the 65-18 "slow" catalyst at ½%.

An oven-dried sand sample I-66 was molded at 110 lb/ft³ dry density, as were all other sand samples. The sample weighted 1255.7 g, was saturated to zero, and 198 g of water was added to saturate it. The sample was allowed to drain for 30 minutes, at the end of which time it weighed 104 g, consisting of water. It was then put into a −18.4°F (−28°C) ambient temperature, along with the resin system solutions (components), for five hours. The coldroom ambient temperature was then turned to −10°F (−23.3°C), which remained overnight. The following morning the coldroom ambient temperature was raised to 20°F (−6.7°C), and when the resin system solutions rose to 20°F (as measured by thermocouple), the solutions were mixed and pressure injection of the sample was successfully accomplished. The injected sample remained at the 20°F ambient temperature until the next morning after the injection.

The next day, after the sample had been placed in the 39°F water-bath for four hours, it was surface-dried, tempered to room temperature, and an unconfined compression strength test was conducted [see sample in photos, Fig. B31, B32, B33 (App. B)]. The stress-strain curve and the results of the strength tests are given in Figures 15d and 16, respectively. As shown in Figure 16, this resin (EP 65-12 and 17 with EP 65-18 catalyst), injected at 20°F, measured 305.5 psi (22 tons/ft²).

XB-2403 Mud Lock (hydrophilic urethane)

According to the 3 M Company, XB-2403 is a liquid, water reactive polymer solution designed to react with saturated soil. In general, the application rate should be in proportion equal to the weight percentage of water in the soil. It is a brown liquid with 65 ± 2.0% solids by weight, at 8.7 ± 0.2 lb/gal. Acetone is a solvent of this material, and the material flash point is given as 0°F [Tag Closed Tester (Cup) — ASTM D56]. The XB-2403 solution is mixed with water, 50-50 by volume, before use. It was found in this investigation that attempting to pressure inject a soil sample using this formula was unsuccessful.

Test results at 39°F (3.9°C) ambient temperature. Mud Lock was tested for set at the 39°F ambient temperature using the 50-50 by volume (water-Mud Lock solution) formula. The product expanded and bulged, quickly set upon mixing, and was resilient to finger compressive pressure. However, upon cooling, the sample exhibited a shrinkage to less than the initial mixed volume. (See Fig. 13b, temperature-time curve D-1.)

Test results at 33°F (0.5°C) ambient temperature. Because of the complication of quick setting when using the Mud Lock 3M formula, previous attempts to pressure inject soil samples had failed. Therefore, it was decided to attempt an injection at the 33°F ambient temperature without mixing the Mud Lock with water.

This urethane resin solution reacts immediately upon mixing with water. Therefore, the only way a sample could be injected was by using the 100-part solution of XB-2403 Mud Lock. This solution injected into sand sample 69 successfully. However, the injection process forced the majority of the pore water ahead of the injection solution, leaving the soil grains moist for the water reactant hydrophilic urethane solution. The resin solution was injected under 20-psi pressure and moved through the sample with ease.

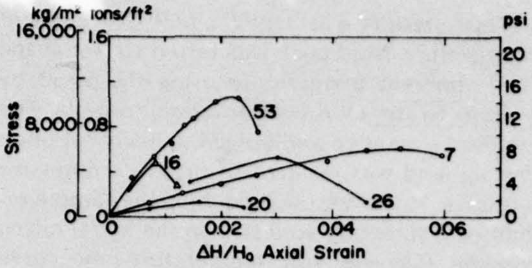
When sample 69 was unmolded, it appeared to be porous, which was evident by the emergence of small bubbles when it was immersed in the 33°F water-bath. At the end of the soaking period, the 7-day unconfined compression strength test measured 115 psi (8.28 tons/ft²); the stress-strain graph is shown in Figure 15b.

Epoxy resin

Epotuf resin system

The epoxy resin solutions are Epotuf 37-130 (a modified diglycidyl ether of bisphenol A — epoxy resin family) and Epotuf 37-052 (previously 37-149, a butyl glycidyl ether — aliphatic monepoxide family). The properties are:

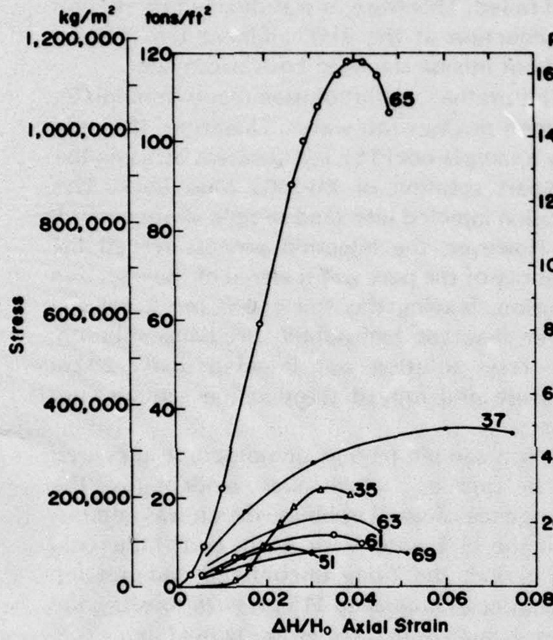
	37-130	37-052
Specific gravity	1.12-1.15	0.89-0.92
Density, lb/gal.	9.3-9.6	7.4-7.7
Epoxide equivalent	175-195	130-150



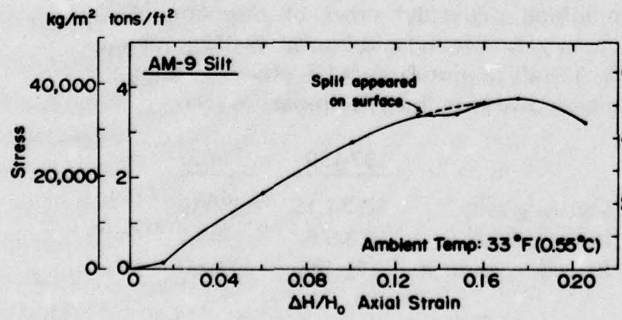
a.

A. Farrel, Silty Sand
B. Jenks, Sandy Silt

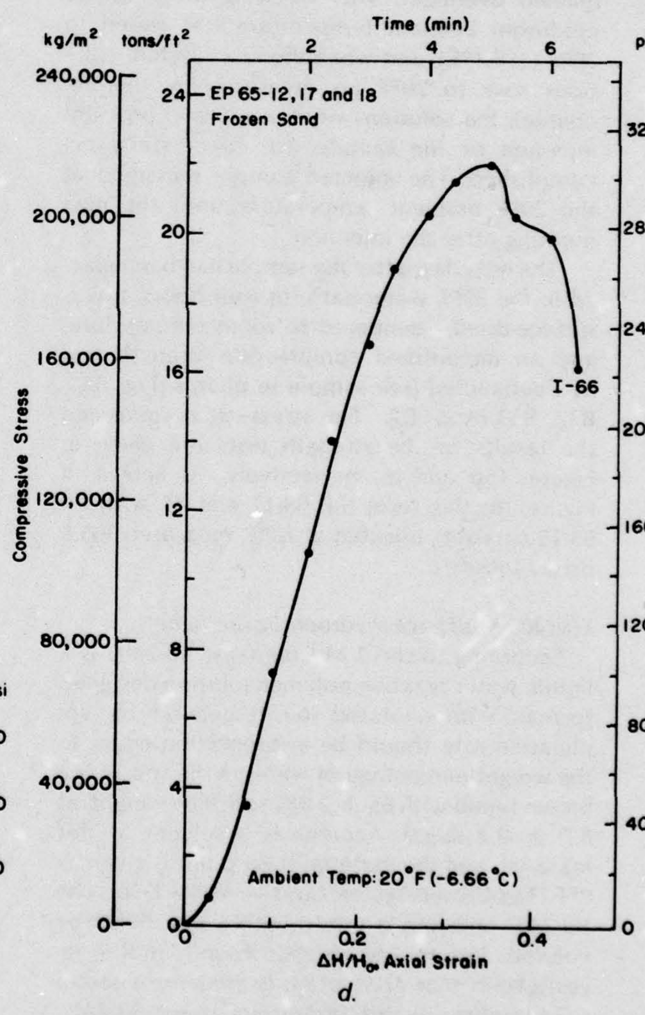
Sample and curve no.	Name	Ambient temp °F	Ambient temp °C	Type soil
7	AM-9	39	3.88	Sand
16	Celtite	39	3.88	Sand
20	Siroc	39	3.88	Sand
26	Raylig 260-L	39	3.88	Sand
35	EP	39	3.88	Sand
37	EPOTUF	39	3.88	Sand
51	EP	39	3.88	Silt
53	Celtite	39	3.88	Silt
61	EP	33	0.55	Silt
63	EP	33	0.55	Sand
65	EP	33	0.55	Sand
69	Mud Lock	33	0.55	Sand



b.



c.



d.

Figure 15. Stress-strain graphs of tested samples.

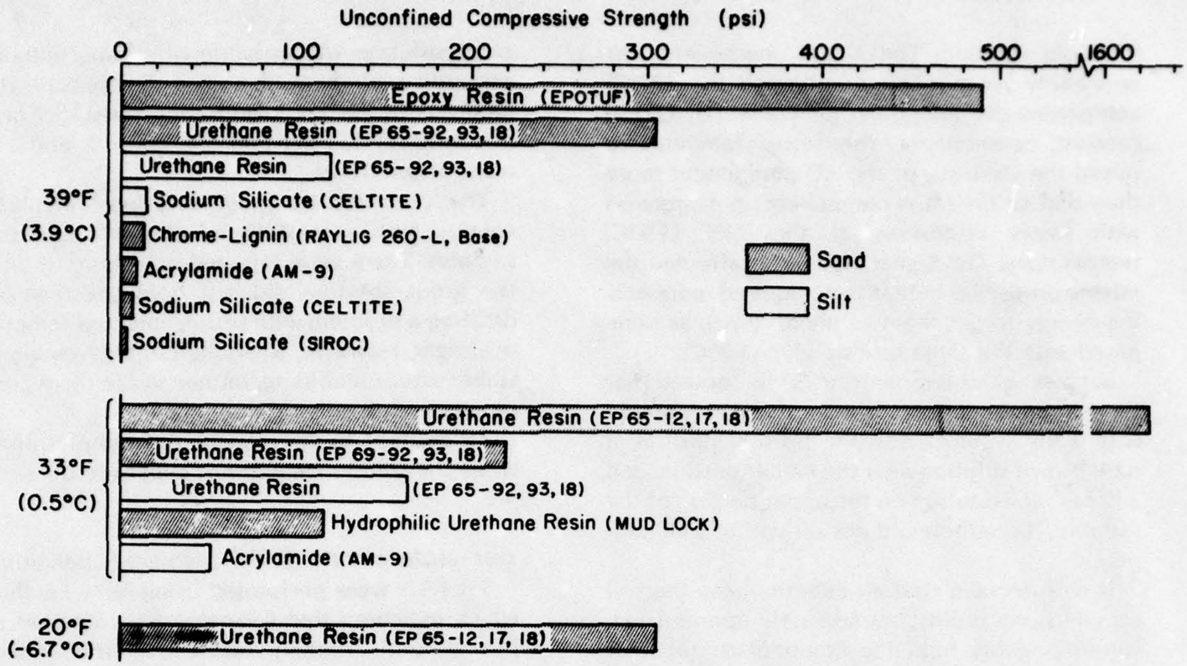


Figure 16. Unconfined compressive strength bar graph of injected samples.

Hardeners/reactants (used with above epoxy resins)

Ancamine AD is a curing agent for liquid and semi-solid epoxy resins, a blend of amines and hardening accelerators, and is capable of curing under cold damp conditions (Pacific Anchor data sheet CA/35).

Ancamine LT is an amine-based curing agent designed to operate down to -5°C, effective under water as well as in air (Pacific Anchor data sheet CA/24).

Ancamine MCA is a modified amine of high reactivity and cures are possible at low temperatures (down to 5°C) and under water (Pacific Anchor data sheet CA/37).

Epotuf was formulated as recommended by Pacific Anchor Chemical Corporation:

Component	Parts by weight
37-130	20
37-052	80
MCA	30
AD	30

However, the 37-052 component was a very low viscosity solution (approximately that of water) and was added to the 37-130 component (20% by weight) to lower the overall viscosity of the mix, as recommended by Reichhold Chemicals, Inc.

Test results at 39°F (3.9°C) ambient

During set tests, the Epotuf components mixed well at this ambient temperature and the heat of reaction rose immediately. Within five minutes, the sample temperature was 43.3°F (6.3°C), and within one hour the temperature was 75.2°F (24°C). Immediately following this, the sample began to liberate heat at a high rate and produced a temperature of 341.6°F (172°C) within the next 26 minutes. The temperature versus time graph is presented in Figure 13b, curve A-1.

Injecting Epotuf into sand sample 37, with 20-psi injection pressure, was without problem. The injected sample [see sample in Fig. B14 (App.B)] was not hard when unmolded the next morning. However, within 15 minutes of exposure to air, at the 39°F ambient temperature, the sample became hard.

At the end of the 7-day period, the sand sample had an unconfined compressive strength of 488.88 psi (35.2 tons/ft²).

An attempt to inject a silt sample failed when the resin solution would not penetrate the soil.

Test results at 33°F (0.5°C) ambient temperature.

Set tests were conducted at 33°F (0.5°C). This temperature did affect the mixing property of the resin solution. The 37-130 component was noticeably more viscous, although the 37-052 component did not appear to be affected. Of the catalyst components, the lower temperature raised the viscosity of the AD component more than that of the MCA component, as compared with lower viscosities at the 39°F (3.9°C) temperature. The higher viscosities affected the mixing properties in that they required more mixing energy to get them to blend (mix), as compared with the same task at 39°F (3.9°C).

Set test at this temperature (33°F) showed that the component parts of the total resin separated with a clear solution in the bottom portion, a dark brown solution near the center portion, and a light color solution on the upper portion of the sample. The sample did not set within a 48-hour period.

It was decided that an injection into the soil sample pores (voids), immediately upon mixing, would possibly hold the components together long enough for a set. However, the injection attempt failed when the material would not penetrate the sand at this temperature. Pressures from 20 psi to 60 psi were applied for more than 1½ hours, at which time the sample extruded a clear slightly viscous solution. A possible reason for separation of the components is that the lower viscosity material was passing through or around the sample. At the end of the attempted injection, it was found that a great portion of the remainder of resin solution mix had stayed on top of the sample.

Urea-formaldehyde

Cyanaloc 62 (urea-formaldehyde)

Cyanoloc 62 was formulated according to the following Cyanamid formula:

Component	Part by volume
Cyanaloc 62	50
Water	50
Sodium bisulphate ($NaHSO_4$)	by %*

Test results at 39°F (3.9°C) ambient temperature

The set test conducted with the above formulation and ½% catalyst revealed that this grout solution was capable of setting without any noticeable heat liberation. To measure the hardness of the set, penetration resistance was measured at 120 psi and 300 psi in 1 and 1½ days, respectively.

The Cyanaloc 62 grout solution with ½% catalyst was successfully injected into sand soil samples. There were injected sand samples that the grout solution did not hold together, indicating a problem with setting injected samples overnight. However, when the samples remained stable after unmolding, immersion in the water-bath would dissolve them.

An attempt to inject a silt soil sample failed when the solution would not penetrate the sample.

Test results at 33°F (0.5°C) ambient temperature

Set tests were performed using ½% catalyst which indicated that Cyanaloc 62 would set at the 33°F ambient temperature without liberating a high rate of heat (see Fig. 13b).

Attempting to inject a sand sample failed because the grout solution would not gel within the injected soil. However, the solution that remained above the soil portion of the sample did gel.

Acrylamide gel

According to Cyanamid AM-9 data booklet EN-4, AM-9 is a dry powder that readily dissolves in water to give a solution of low viscosity (about the same as water). This chemical grout is a mixture of two organic monomers—acrylamide and N, N'-methylene-bisacrylamide, in proportions which produce gels from dilute aqueous solutions by a polymerization-crosslinking reaction.

The AM-9 catalyst system follows:

Catalyst DMAPN (β-dimethylaminopropionitrile) is a liquid chemical used as an activator for the reaction. The density, between 32°F and 104°F, is approximately 7.1 lb/gal (0.196 g/cm^3).

Ammonium persulfate (AP), a granular material and oxidizing agent, is the initiator that triggers the reaction.

Potassium ferricyanide (KFe), a granular material, which behaves as an inhibitor, controls the reaction.

AM-9 (Acrylamide gel) was formulated to gel within 60 minutes at 39°F and 33°F (see App. C).

* The percentage of sodium bisulphate (catalyst) is added according to desired set time as a function of temperature.

Test results at 39°F (3.9°C) ambient temperature

The AM-9 set test indicated that this grout solution rose to 28.1°C once the preset gelling period occurred. This grout solution was successfully injected into sand sample 7 (see Fig. B2, App. B). The 7-day unconfined compression strength test measured 8 psi (0.61 tons/ft²).

Test results at 33°F (0.5°C) ambient temperature

Because of the low viscosity (near water) of AM-9, a silt soil sample was successfully injected.

The unconfined compressive strength test of the silt injected sample 70 produced 51.11 psi (3.68 tons/ft²) with a significant amount of deformation of the distortion and volumetric type as described by Lambe and Whitman (1969). [See sample 70 in Fig. B28, B29, B30 (App. B)].

Sodium silicates

Sodium silicate (Hayward Baker Formula)

The Hayward Baker's grout material is a sodium silicate with GELOC-3 reactant (ethyl acetate-amide).

As recommended by the manufacturer, this sodium silicate was formulated by volume as follows:

60%	Sodium silicate
8%	GELOC-3 (reactant)
32%	Water

Test results at 39°F (3.9°C) ambient temperature

Set tests were performed that showed a temperature gain of 44.06°F (6.7°C) in 103 minutes; this temperature remained for 50 minutes before beginning to cool to the 39°F ambient temperature within 15 hours. The set sample measured 300 psi penetration resistance after this 15-hour period.

Pressure injecting this sodium silicate grout presented problems. It displayed quick initial setting or gelling when attempts were made to inject the soil samples.

Further tests were conducted at the 39°F ambient temperature to determine if a lower-percentage catalyst would lengthen the time to initial set of the grout solution and provide a formula which could be used for pressure injection of soil samples. Therefore, 5, 6, and 7% catalyst formulations were made. The 7% catalyst produced a gel instantly; the 6% catalyst produced visual signs of gelling in approximately 15 minutes; and the 5% catalyst produced visual signs of gelling in approximately 1-1½ hours, contrary to previous test results using the 8% catalyst. The present samples remained at the 39°F ambient temperature for 3 days. Perhaps the component has a short shelf life that affects the control of setting. Penetration resistance measurements were then made. The results are as follows:

Catalyst (%)	Penetration resistance stress (psi)
5	280
6	450
7	470

Using a 5% catalyst, three samples were pressure injected. The 5% catalyst was chosen because time was needed to mix and inject the samples. The first sample (22) held together and was immersed in the water-bath for four days, before it was found dissolved. The other two samples did not seem to have set and fell apart.

SIROC (sodium silicate)

The SIROC grout is a sodium silicate system with chloride-amide components. Sodium silicate is the gel-forming material with the amide being the primary gel-producing reactant. The accelerators for controlling the rate of initial set for this system are chloride, aluminate, and bicarbonate.

The SIROC sodium silicate formula (as recommended by Mr. Peeler, SIROC chemist) is as follows:

Percent by weight	
11.65	SIROC #1 (Silicate)
9.5	SIROC #2 (Formamide)
0.156	Water
8.337	SIROC #3 (Chloride)

Test results at 39°F (3.9°C) ambient temperature

This grout solution produced a set or gel which appeared to be of a fine sponge texture and which would not offer any penetration resistance. See Figure 13b for temperature-vs-time graph of set test. However, a SIROC injected sand sample (20) did produce a 3-psi unconfined compressive strength.

Celtite 55 Terraset (Sodium Silicate)

According to the manufacturer, Celtite, Inc., the Terraset grout is a blend of organic esters designed to produce a stable gel, from sodium silicate solutions, down to the freezing point.

The Celtite, Inc. Terraset grout formula used was in parts by volume:

100 parts A
10 parts B
150 parts water

Test results at 39°F (3.9°C) ambient temperature

Set test produced no noticeable heat liberation, and the set product measured 120 psi penetration resistance at the end of 3 days.

Sand and silt samples 17 and 53 [sample 53 is shown in Fig. B17, B18 (App. B)], respectively, were successfully injected with the Celtite grout. The injected soil samples resulted in brittle, easily crumbling samples, but they did remain stable immersed in the 7-day water-bath. The unconfined compression test measured 7.22 psi (0.519 tons/ft²) for sample l6, and l4.58 psi (l.049 tons/ft²) for sample 53 (Table II).

Chrome-lignin gel

Raylig-260 L

Raylig-260 L, a neutralized sodium lignosulfonate, is formulated to a chrome-lignin using sodium dichromate and aluminum sulfate. The Raylig solids content (%) is formulated using the SD, AS, 250 L, and water (ITT Rayonier's data sheet).

The formation of chrome-lignin, using Raylig-260 L with sodium dichromate and aluminum sulfate, was for a 25% content Raylig solids solution:

	Parts by weight	Parts by volume
Raylig-260 L	50	40
Water	27.5	27
Aluminum sulfate	10.0	7.5
Sodium dichromate	12.5	8.7

Tests at 39°F (3.9°C) ambient temperature

Set test showed that components of this grout solution mixed rather well. However, a sludge formed that settled to the bottom of the mixing vessel. The low temperature noticeably increased the viscosity of the Raylig-260 L. The mix liberated a rate of heat to approximately 49.28°F (9.6°C) in 2½ hours after mixing. The test sample then gradually cooled, as shown in Figure 13b. After 24 hours, the penetration resistance measured 120 psi.

Sand samples 26 through 29 were successfully injected with this grout solution. The unconfined compression strength of sample 26 measured 8 psi (0.6 tons/ft²). The samples were moist in the center and bulged in shape under this test. [Sample 26 is shown in Fig. B8 and B9 (App. B).]

Bentonite

Four, six and eight percent bentonite contents were mixed with water, by weight.

The mixing was conducted by slowly adding or sprinkling the bentonite into agitated water, stirring in a table model mixing blender (Fig. 11). Each percentage of bentonite content was mixed for 10 minutes.

Each mix was allowed to set for 2 hours to hydrate or gel before being injected into the soil sample. While being agitated by the pressure vessel, to prevent settling, samples 38, 39 and 40 were injected with the 4%, 6% and 8% bentonite contents, respectively.

Sample 38 was unmolded, after 12 hours, but failed under its own body weight. Samples 39 and 40 were not unmolded because the bentonite slurry on top of each sample was still fluid. These samples were stored out of the water and in the molds at the 39°F ambient temperature. Unconfined compression strengths were not measured because the samples could not be cured in the same manner as the other samples, which set and were immersed in the water-bath.

Cements

The materials used were Portland cement, Type I (ordinary), and Portland cement, Type III (high early strength), for grout slurries. Ordinary Portland cement was used to make a grout slurry for samples 41, 42 and 43. The water cement ratios by weight for each sample were 1.7, 2.9 and 4.4, respectively.

All attempts to grout sand samples failed when the cement filtered out at the top of each sample. At the beginning of grout injecting, the cement fines would begin to move into the sample but would not move through the sample in a slurry (cement) at the above water/cement (w/c) ratios. Herndon and Lenahan (1976, p. 88) give w/c -2 to 5 initially in a holding tank, with more cement then added upon injection pressure feasibility. Another reference for w/c ratio is Department of the Army TM 5-818-6 (1970, p. 27 and 28).

Type III Portland cement was used at w/c ratios between 2 and 5. This cement, a finer grind, also failed to penetrate the sand samples.

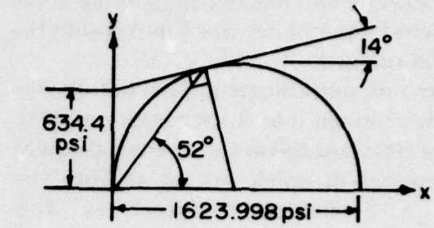
DISCUSSION

The tests conducted on sand and silt yielded strength values that can be compared with those of injected soil samples prepared, in this investigation, under the same conditions. Included in the tests were the chemicals that are intended not to add strength to the soils but to make them impermeable, for example, the acrylamide gel AM-9. However, tests of silt samples injected with the AM-9 gel gave higher unconfined compressive strength results than those of sand samples injected with the gel. This is shown in the stress-strain graphs of Figure 15a and 15c, and in Table II. These tests showed that a silt sample successfully injected with AM-9 gained a significant unconfined compressive strength, as evidenced by the 17.5% strain at 51.11 psi (3.67 tons/ft²) of the silt sample at 33°F versus the 8.472 psi (0.6 ton/ft²) sand sample at 39°F. Celtite 55 Terraset, brittle in nature, also gave higher strengths for the injected silt sample (14.583 psi -1.05 tons/ft²) in contrast with the injected sand sample (7.222 psi - 0.52 ton/ft²), both at 39°F (see Table II).

The Ashland Chemical Co. urethane resin (EP 65-12, 17 and 18) resulted in the product with the strongest set which maintained a low viscosity as the temperature lowered; and, according to visual inspection, all of the EP series injected soil samples were impervious (not porous). This product was successfully injected at 20°F and set. Set test data showed this urethane solution mixed at temperatures lower than 20°F and produced a set. This solution is capable of being mixed at -26°F, still fluid, and is capable of producing a set at approximately 17°F and below. This product shows promise for grouting and stabilizing fine-grain soils at lower temperatures. It has set at -4°F (-20°C) in a recent construction materials test program.

The results of the investigation on the unconfined compressive strength tests show that the injected urethane resin (Ashland Chemical Co.)

Sample 65 Urethane Resin, Sand



$$\theta = 52^\circ$$

$$45^\circ + \frac{\phi}{2} = 52^\circ$$

$$\therefore \phi = 14$$

Figure 17. Mohr's Circle of stress for the highest strength gained by pressure injecting (33°F ambient temperature).

produced the highest strength (1623.99 psi — 117.575 tons/ft², 33°F) and the injected epoxy resin (Reichhold Chemicals, Inc.) the second highest (448.88 psi — 32.319 tons/ft², 39°F) (see Fig. 16 and 17). However, the epoxy resin exhibited problems and a significant increase in viscosity as the temperature was lowered to 33°F, as opposed to the urethane resin that did not. The Mud Lock XB-2403, which is water reactive, maintained a low viscosity and was successfully injected into a sand sample at 33°F, resulting in an unconfined compressive strength of 115.22 psi (8.295 tons/ft²). Mud Lock, being injected into frozen soil, would perhaps remain stable until thaw, at which time it might consume water and react, becoming solid.

For soil injection purposes, the Mud Lock with water reacted too quickly for a one-solution system. It worked very well concentrated, when pressure injected into a 100% (approximate) water-saturated soil sample, by displacing the great majority of water and reacting with the moist soil. However, the set sample was porous.

The epoxy resin tested (Epotuf) gave a high unconfined compressive strength, but as the resin temperature was lowered, the viscosity of the solution increased noticeably, and at the 39°F set test, the heat of reaction increased the temperature to 341.6°F; this must be considered if the material heat would disturb or melt the normal balance of the substrate. The chrome-lignin, Raylig-260 L gel (ITT Rayonier, Inc.), became significantly viscous at the 33°F ambient temperature and was not sludge free at the 39°F ambient temperature, when mixed with the table model blender.

Among the sodium silicate grouts, Celtite was successfully injected into a silt sample at 39°F (3.9°C). The Hayward Baker Co. formula (sodium silicate) resulted in quick setting at both the 39°F and 33°F ambient temperatures. This would not allow the grout to be injected as a one-solution system. The SIROC sodium silicate grout would not set firmly enough to withstand any compression at the 39°F set test temperature but did result in a 3-psi (0.209 ton/ft^2) unconfined compressive strength in an injected sand.

Attempts to inject a cement slurry failed when the slurry would not penetrate the sand soil sample. The bentonite slurry was successfully injected into sand samples at 39°F (3.9°C); but the sample would have had to be dried for its unconfined compressive strength to be tested, and this would not have been consistent with other 7-day water-bath immersed samples.

A problem was encountered in getting the Cyanaloc 62 to set upon being injected into the soil sample at the 39°F ambient temperatures, but it did set during the set test. The set test produced results without noticeable heat liberation. This may indicate that Cyanaloc 62 can be placed into or adjacent to permafrost without melting it. Cyanaloc 62 did not produce successfully pressure injected set soil samples, even though an injected soil sample was firm enough to place into the water-bath. However, this sample might have set if it had remained out of the water-bath.

The acrylamide as well as sodium silicate grouts and urethane resins remained the least viscous of all the solutions as the material temperature was lowered, indicating potential as fine-grain soil (sands and silts) stabilizers at low temperatures. The acrylamide grout and the hydrophilic urethane are aqueous solutions and are limited to use in temperatures above freezing of their water portions. However, the urethanes, which are not aqueous and which are capable of remaining fluid at temperatures below 32°F, may show promise of having potential as soil stabilizers in lower temperatures. As previously stated, when the epoxy solution temperature was lowered, the viscosity of this material increased, presenting a problem of mixing and workability and decreasing the material's potential for pressure injecting soils as the temperature is lowered.

Figure 16 shows that the acrylamide, chrome-lignin and sodium silicates produced the lowest unconfined compressive strengths. However, in some projects where pressure-injection is used to stabilize soils, or where the materials can be used at low temperatures for other construction purposes, the strength parameter may not be the most important, for example, in grout curtain wall construction where impermeability of soil is needed, or in construction where dust prevention on dusty roads at temperatures 39°F and below is necessary.

The costs of specific grout materials and solutions are given in Appendix C. Material costs vary greatly. However, different catalyst concentrations (App. C) for the desired formula of grout solution chosen, affect the final material costs. The pressure injection process also requires the proper equipment to accomplish the various mixing procedures as well as the soil injection task, and this affects the in-place cost.

CONCLUSION

The Ashland urethane resin (EP65-12, 17 and 18) can be formulated to use in soils at low temperatures for stabilization by increasing the soil strength and making it impermeable (impermeable according to visual inspection). This resin can be applied to low temperature soils above freezing or to low temperature soils, with little or no ice content, that are below freezing.

RECOMMENDATIONS

1. *Laboratory test.* It is recommended that further research be carried out on urethanes such as the Ashland Chemical Co. resin to seek additional uses in cold environments (32°F and below) for constructing foundations, where concrete may not be feasibly used, and for construction patching. Information is needed on hydraulic conductivity and bonding strengths of urethanes used with other building materials, including concrete, steel, wood and glass. Information on thermal expansion of urethanes and the associated stresses in contrast to other

materials is also needed for structural purposes.

The hydrophilic urethane Mud Lock XB-2403, which is water reactant, should be tested for soil stabilization by mixing water and soil down to the freezing point of the water portion. Further, Mud Lock should be investigated for its use as an injection grout for various percentage-saturated frozen soils samples. The samples should then be allowed to thaw to create the water for reacting. Depending on the shelf life of this material, it may have stabilization potential for injections in-situ previous to thaw.

Acrylamide AM-9 should be further tested for structural foundation purposes in low temperatures where water exists continuously around foundations, including those subject to vibratory loads. The silt AM-9 sample exhibited great resilience when examined by hand and should be tested for this property using injected silt or fine-grained soils with a variety of loading conditions.

Urea-formaldehyde such as Cyanaloc 62, which did set at a low temperature (39°F) with the lowest heat liberation rate, should be investigated for further low temperature set information as well as unconfined compressive strength of the material. This grout may show promise for other pressure injection purposes (cavity and jacking) or placement on permafrost for structural purposes, without melting or disturbing the normal balance of the substrate.

2. *Field evaluation.* An in-situ test site of sufficient size and proper soil conditions should be chosen so that an adequate number of samples can be produced to validate the laboratory test. This test site should be in a field environment where grouting can be performed with the ambient temperatures at 39°F and below. This would allow an in-depth evaluation of grouting procedures and materials.

LITERATURE CITED

Department of the Army Technical Manual (1970) Grouting methods and equipment. DA TM 5-818-6.

Herndon, J. and T. Lenahan (1976) Grouting in soils. Federal Highway Administration, Offices of Research and Development. Report No. FHWA-RD-76-26, p. 88, p. 285.

Johnson, R. (1977) Grouting of soils in cold environments. CRREL Special Report 77-42.

Lambe, T.W. and R.V. Whitman (1969) *Soil mechanics.* New York: John Wiley and Sons, Inc.

APPENDIX A. GROUTS AND CHEMICAL SOLUTIONS, MANUFACTURERS AND PERSONNEL CONTACTED

Name and/or no. of grout or chemical solution	*Company firm or manufacturer*	*Personnel contacted (1977)*
EP Systems and A-11	Ashland Chemical Co. Division of Ashland Oil Inc. Columbus, Ohio 43216	Dr. Robert Shafer Dr. G.L. Linden John Lundberg (614) 889-3333
XB-2403 Mud Lock	3 M Company 3 M Center St. Paul, Minn. 55101	John Simpson Paul Denuccio (612) 733-1110
Epotuf 37-130 and 37-052	Reichhold Chemicals, Inc. RCI Building White Plains, N.Y. 10602	Scott C. Raswyck Andover, Mass. 01810 (617) 475-6600
Ancamine AD, LT and MCA	Pacific Anchor Chemical Corp. (PVO International Inc.) Richmond, Calif. 94804	Colin G. Hull, President (415) 529-1020
Raylig-260 L	ITT Rayonier, Inc. New York, N.Y. 10016	E.K. Millette (212) 687-7880 F.W. Herrick (206) 426-4461 Shelton, Wash. 98584
AM-9	American Cyanamid Co. Berdan Avenue Wayne, N.J. 07470	Roger Nowell (201) 831-1234
Cyanaloc 62	American Cyanamid Co. Berdan Avenue Wayne, N.H. 04740	Roger Nowell (201) 831-1234
Hayward Baker Sodium Silicate	Hayward Baker Co. 1875 Mayfield Road Odenton, MD 21113	Wallace H. Barker (301) 551-8200 or (301) 621-9400 Washington, D.C.
SIROC	Raymond International Inc. Cherry Hill, N.J. 08034	E.R. Colle (609) 667-3323
Celtite 55 Terraset	Celtite, Inc. Cleveland, Ohio 44133	Tony Plaisted (216) 237-3232
Bentonite Clay	Federal Bentonite Aurora, Ill. 60538	Robert F. Waterloo (312) 896-4142
Portland cement Type I		
Portland cement Type III		

APPENDIX B. TEST SAMPLES

The samples shown in this appendix are listed as *before, during,* and *after* unconfined compressive strength testing. The samples designated as *before* testing had been soaked for 7 days in the water-bath.

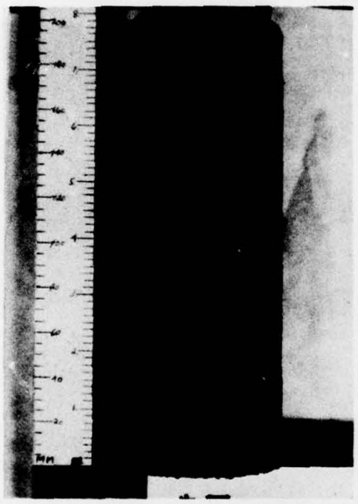


Figure B1. No. 7 before.

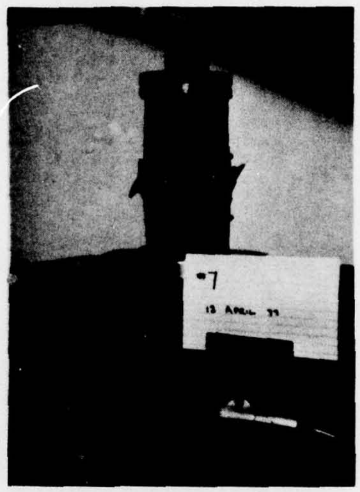


Figure B2. No. 7 after.

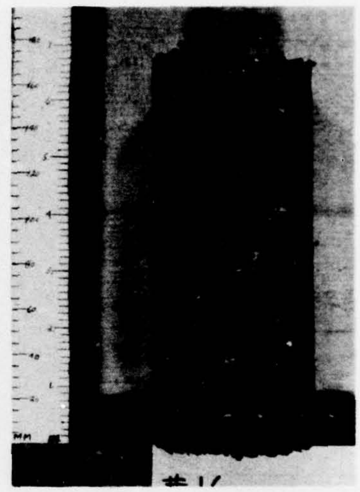


Figure B3. No. 16 before.

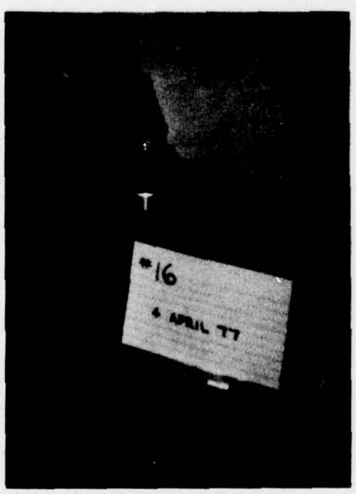


Figure B4. No. 16 after.

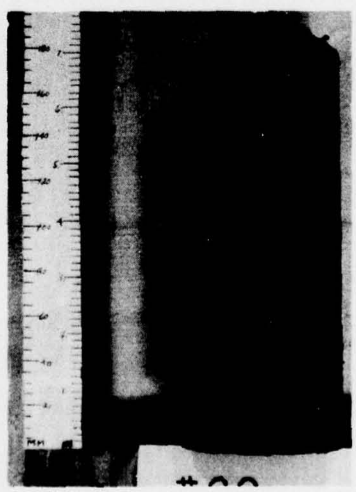


Figure B5. No. 20 before.

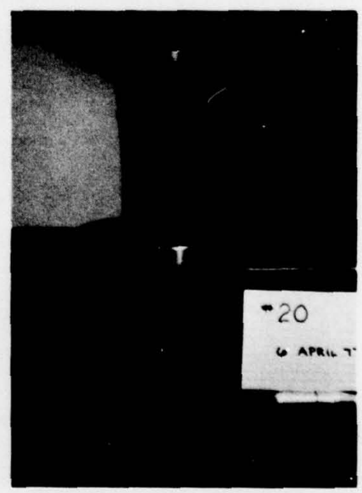


Figure B6. No. 20 during.

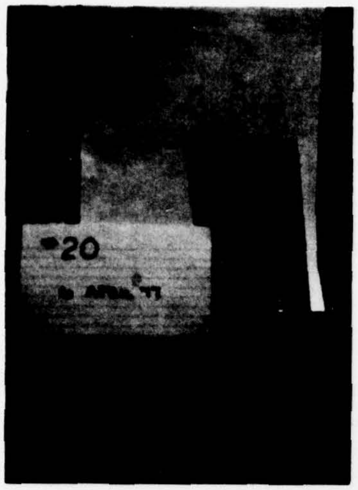


Figure B7. No. 20 after.

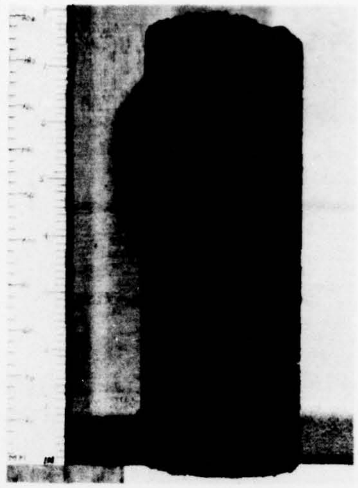


Figure B8. No. 26 before.

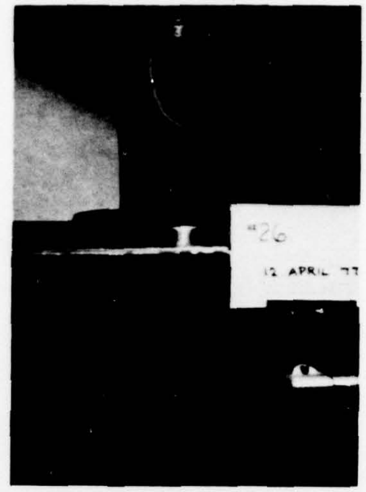


Figure B9. No. 26 after.

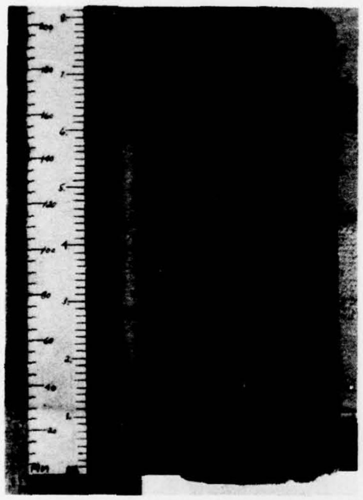


Figure B10. No. 35 before.

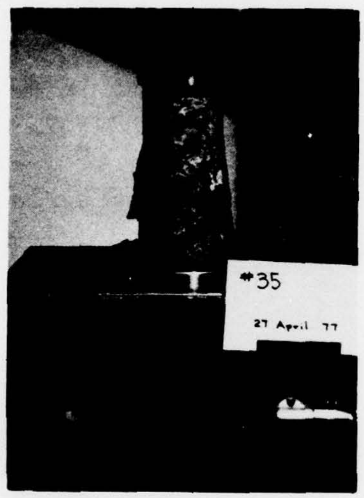


Figure B11. No. 35 after.

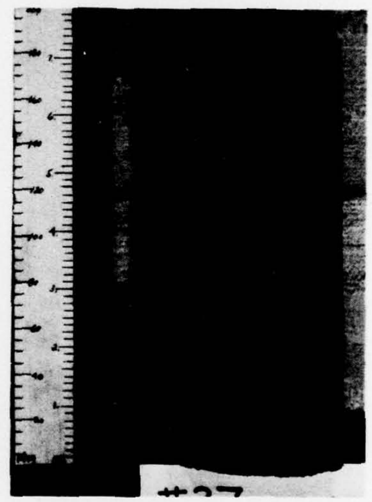


Figure B12. No. 37 before.

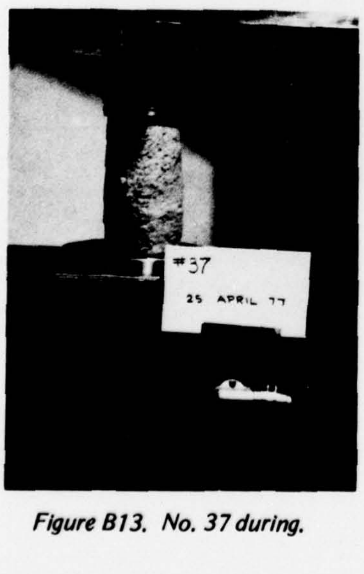


Figure B13. No. 37 during.

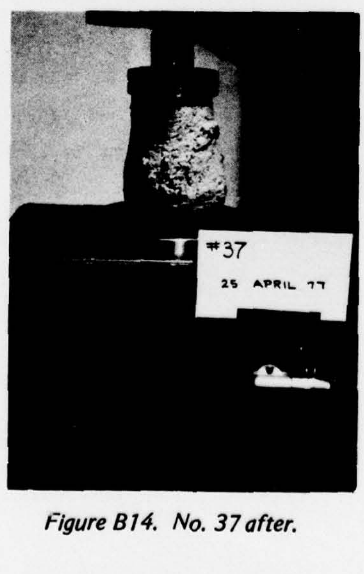


Figure B14. No. 37 after.

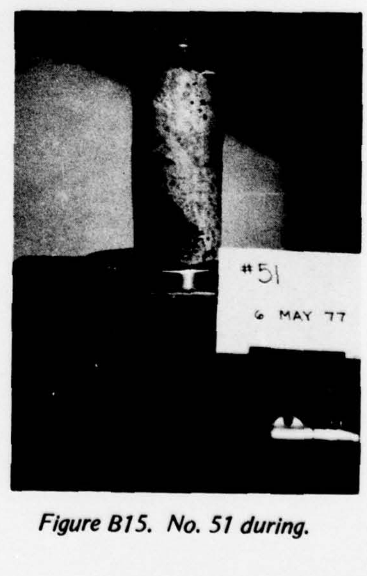


Figure B15. No. 51 during.

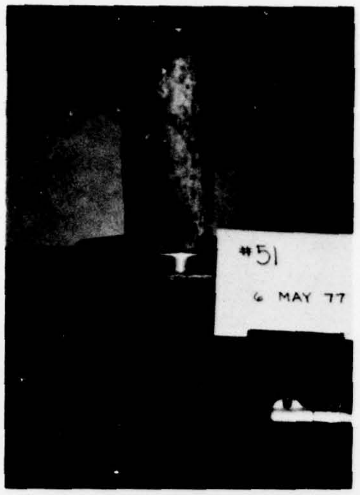


Figure B16. No. 51 after.

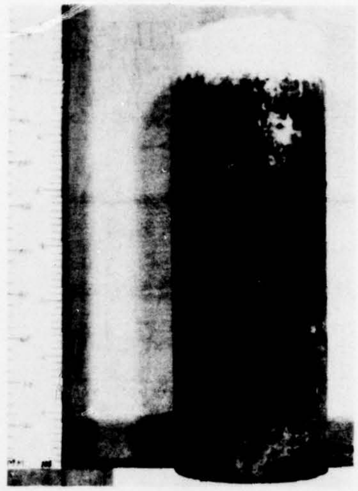


Figure B17. No. 53 before.

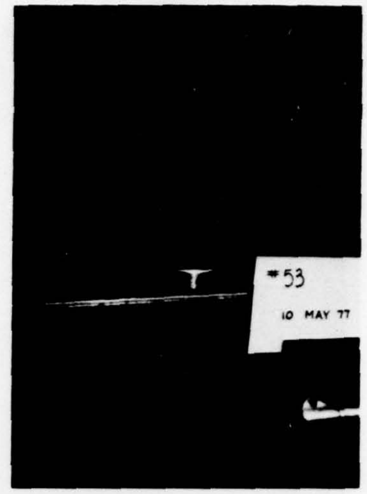


Figure B18. No. 53 after.

Figure B19. No. 61 before.

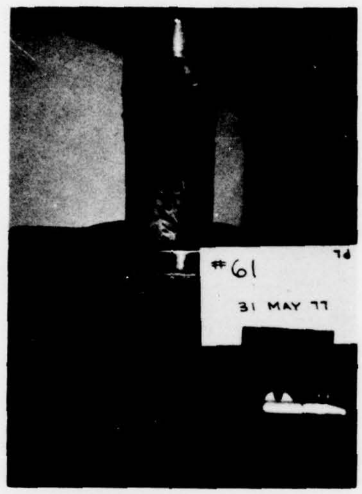


Figure B20. No. 61 after.

Figure B21. No. 63 before.

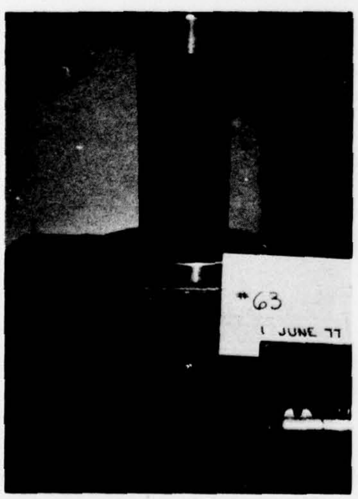


Figure B22. No. 63 during.

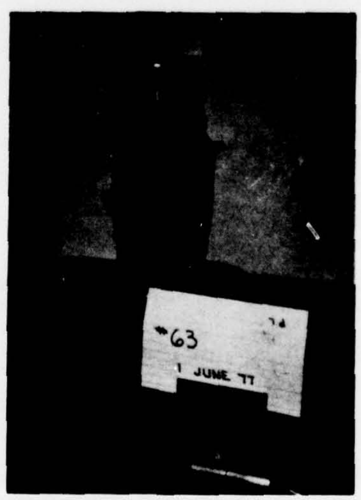


Figure B23. No. 63 after.

Figure B24. No. 65 before.

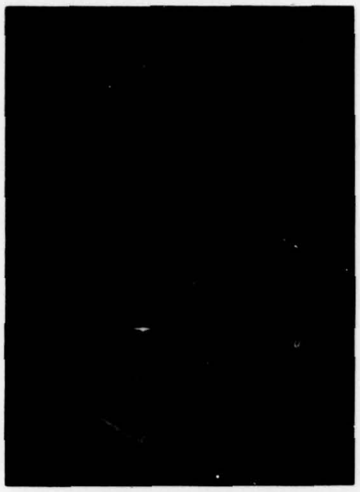


Figure B25. No. 65 during.

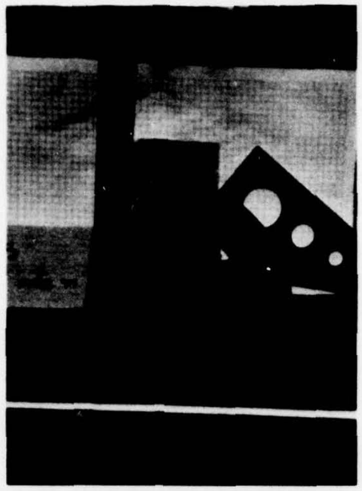


Figure B26. No. 65 after.

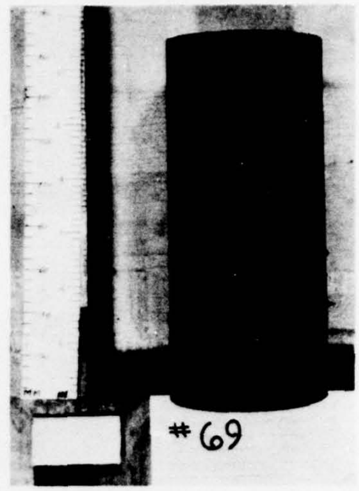


Figure B27. No. 69 before.

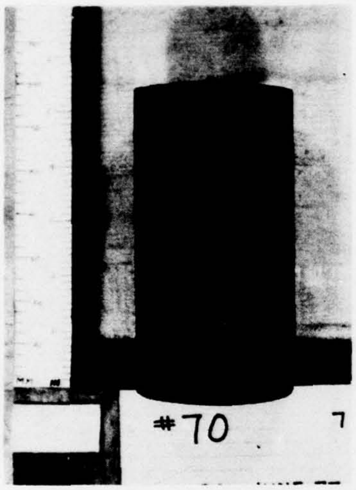


Figure B28. No. 70 before.

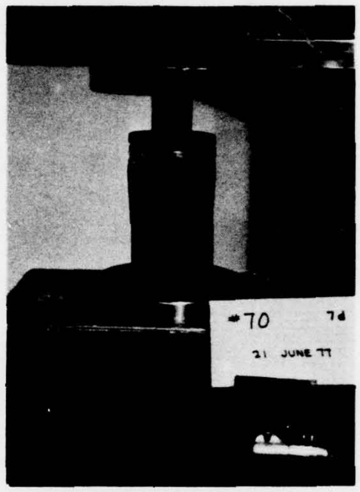


Figure 29. No. 70 during.

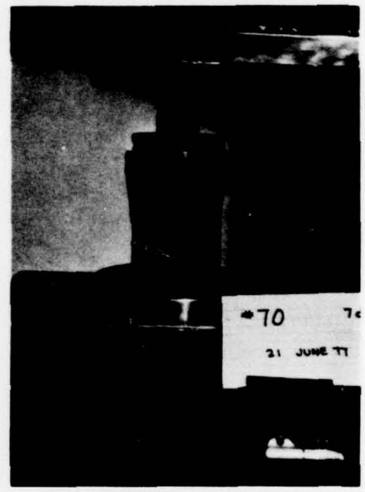


Figure B30. No. 70 after.

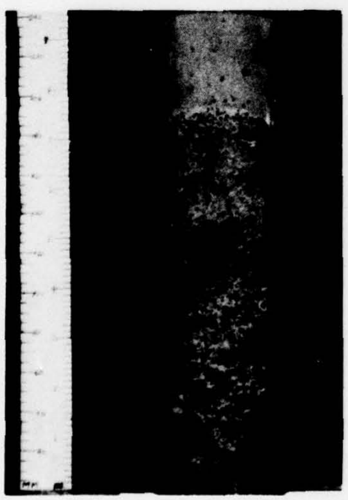


Figure B31. No. I-66 before.

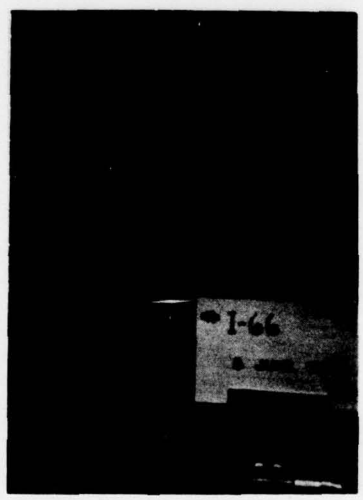


Figure B32. No. I-66 during.

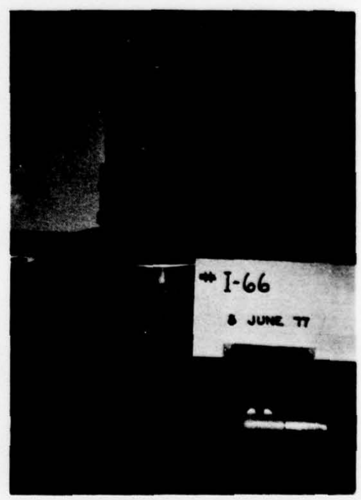


Figure B33. No. I-66 after.

APPENDIX C. GROUT OR SOLUTION COST INFORMATION

Solution no.	*Price/pound*	*Flash point*
Urethane resin (Ashland Chemical Co.)		
EP 65-92	$ 0.4968	105°F
EP 65-93	0.5393	
EP 65-12	0.7060	130°F
EP 65-17	0.5195	120°F (PM)
EP 65-18*	4.7237	276°F (PM)
EP 65-94*	7.0350	198°F (PM)
A-11*	1.5217	130°F (TCC)
Hydrophilic urethane resin (3M Company)		
XB-2403 Mud Lock	$13.00/gal.	0°F (TCC)
Epoxy resin (Reichhold Chemical Inc.)		
Epotuf 37-130	$ 0.78 (truck load)	200°F (TCC)
Epotuf 37-052	1.08 (truck load)	130°F (TCC)
Hardners/Reactant (Pacific Anchor Chemical Corp.)		
Ancamine AD	$ 1.60	180°F (TCC)
Ancamine LT	1.40	213°F (TCC)
Ancamine MCA	1.70	230°F (TCC)
SIROC (Raymond International)		
Silicate	$ 0.93/gal.	
Amide	4.74/gal.	
Chloride	0.30/10 lb. bag	
Celtite 55 Terraset (Celtite Inc.)		
Part A	$ 0.91/gal.†	281°F (TCC)
Part B	0.91/gal.†	
Lignosulfonate (ITT Rayonier Inc.)		
Raylig-260 L	$20.00/short ton F.O.B. Hoquiam, Wash.	

* Catalyst
† *Price from Paul Rausch*
(PM) = Pensky - Martens Closed Tester (ASTM D93)
(TCC) = Tag Closed Tester (Cup) (ASTM D56)

Effective February 1, 1976

AM-9* chemical grout

Delivery: F.O.B. Linden, New Jersey.
Terms: Net 30 days to app'vd. credit or cash with order.
Freight: Collect.
Shipping Points: Plant – Warners, New Jersey
Warehouses – Rosemont, Ill. (Add $0.05/lb.)
Tracers shipped from Linden sample department only.

Packing: Non-returnable containers.
Repacking: Not allowed.
Technical Information: AM-9 Technical Bulletin.
Manufactured: AM-9 Warners, New Jersey.

INDIVIDUAL SHIPMENTS: PRICES PER POUND

	Product Code	*40,000 lbs. and Over*	*20,000 lbs. to 39,950 lbs.*	*10,000 lbs. to 19,950 lbs.*	*2,000 lbs. to 9,950 lbs.*	*1,000 lbs. to 1,950 lbs.*	*950 lbs. or Less*
Bag Quantity		*800*	*400-799*	*399-200*	*199-40*	*39-20*	*19-1*
AM-9 Chemical Grout	94104-04	$0.80	$0.91	$1.02	$1.08	$1.29	$1.46
AM-9 Plus Chemical Grout	94104-12	0.90	1.01	1.12	1.18	1.39	1.56
50 lbs. Net							

	Product Code	*3,750 lbs. & Over*	*1,500 Lbs. to 3,375 lbs.*	*375 lbs. to 1,125 lbs.*
Drum Quantity		*10-up*	*4-9*	*1-3*
Catalyst DMAPN 375 lbs. Net	93802-01	$0.90	$0.95	$1.00

	Product Code	*3,500 lbs. & Over*	*1,050 lbs. to 3,465 lbs.*	*350 lbs. to 1,015 lbs.*	*210 lbs. to 315 lbs.*	*35 lbs. to 175 lbs.*
Pail Quantity		*100-Up*	*30-99*	*10-29*	*6-9*	*1-5*
35 lbs. Net	93802-04	$1.05	$1.10	$1.15	$1.25	$1.35

	Product Code	*2,000 lbs. & Over*	*1,000 lbs. to 1,950 Lbs.*	*500 lbs. to 950 lbs.*	*200 lbs. to 450 lbs.*	*150 lbs. or Less*
Drum Quantity		*40-Up*	*20-39*	*10-19*	*4-9*	*1-3*
Ammonium Persulfate	95055-04	$0.55	$0.60	$0.65	$0.75	$0.85
50 lbs. Net						

		Prices Per Pound Any Quantity
Potassium Ferricyanide (KFE)	95198-01	$1.35
Blue Tracer	42202-01	3.50
Violet Tracer	42701-03	3.50
Yellow Tracer	41812-01	4.25
Red Tracer	42601-04	6.00

	POUNDS Net	*POUNDS* Tare	*POUNDS* Gross	*TYPE*
AM-9	50	1	51	Polyethylene Laminated Bags
DMAPN	35	5	40	5 gallon pail
DMAPN	375	50	425	50 gallon black iron drum
AP	50	4	54	6 gallon fibre pak
KFE	1	–	–	bags

*Trademark

CYANALOC® 62 chemical grout

Effective Date: June 7, 1976

Delivery: F.O.B. Wallingford, Conn.
Terms: Net 30 days to app'vd. credit or cash with order
Freight: Collect.
Packing: Cyanaloc 50 gal. non-returnable steel drums
Net – 575 lbs., Tare – 41 lbs., Gross – 616 lbs.

Pounds Per Gallon: 10.4
50 Gallon Fibre Pak: Sodium Bisulfate – Net 400 lb.
Tare – 28 lb. Gross – 428 lb.

		INDIVIDUAL SHIPMENTS PRICES PER POUND					
	Product Code	*Tank Cars or Tank Trucks*	*Minimum 24,150 lbs. and over*	*10,350 lbs. to 23,575 lbs.*	*5,175 lbs. to 9,775 lbs.*	*2,300 lbs. to 4,600 lbs.*	*1,725 lbs. or Less*
Drum Quantity			*42 - Up*	*18 - 41*	*9 - 17*	*4 - 8*	*1 - 3*
Selling Price $/lb.	26202-01	$0.19	$0.23	$0.25	$0.27	$0.29	$0.31
Selling Price $/gal.		1.97	2.39	2.60	2.81	3.01	3.22

		INDIVIDUAL SHIPMENTS PRICES PER POUND				
	Product Code	*3,200 lbs. 3,200 lbs. to*	*2,400 lbs. to 2,800 lbs.*	*1,600 lbs. to 2,800 lbs.*	*800 lbs. to 1,200 lbs.*	*400 lbs.*
Drum Quantity		*8 - Up*	*6 - 7*	*4 - 5*	*2 - 3*	*1*
Sodium Bisulfate	95184-01	$0.19	$0.21	$0.23	$0.25	$0.28

® trademark

A facsimile catalog card in Library of Congress MARC format is reproduced below.

Johnson, Robert
Grouting silt and sand at low temperatures, A laboratory investigation / by Robert Johnson. Hanover, N.H.: U.S. Cold Regions Research and Engineering Laboratory; Springfield, Va.: available from National Technical Information Service, 1979.
v, 38 p., illus., 27 cm. (CRREL Report 79-5.)
Prepared for Directorate of Military Programs, Office, Chief of Engineers under DA Project 4A762730AT42.
Bibliography: p. 23.
1. Chemical grouts. 2. Grout slurry. 3. Injection grouting of soil. 4. Unconfined compressive strength. 5. Viscosity. I. United States. Army. Corps of Engineers. II. Army Cold Regions Research and Engineering Laboratory, Hanover, N.H. III. Series: CRREL Report 79-5.

☆U.S. GOVERNMENT PRINTING OFFICE: 1979 – A-1093/365