ORNL/TM-9881

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Grout Testing and Characterization for Shallow-Land Burial Trenches at the Idaho National Engineering Laboratory

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OPERATED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
NTIS price codes—Printed Copy: A04 Microfiche A01

ORNL/TM-9881
Dist. Category UC-70B

Chemical Technology Division

GROUT TESTING AND CHARACTERIZATION FOR SHALLOW-LAND BURIAL TRENCHES AT THE IDAHO NATIONAL ENGINEERING LABORATORY

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Date of Issue - October 1986

Prepared by the
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under Contract No. DE-AC05-84OR21400

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EXECUTIVE SUMMARY

This investigation was conducted to test and define conditions for the use of grout to stabilize low-level and TRU waste in Idaho National Engineering Laboratory (INEL) shallow-land burial trenches. The types of grouts investigated were soil, ordinary particulate, fine particulate, and solution (or chemical) grouts. Soil grouts were found to be suitable for disposal in trenches or drums. Particulate grouts were found to be suitable to fill voids in closed-trench soil/waste matrices and to establish grout soil barriers around the trenches. The question concerning the suitability of chemical grouts in INEL soil has not been resolved. The recommended grout compositions listed in Table S.1 are based on results from phase separation, compressive strength, freeze/thaw, density, penetration resistance, hydraulic conductivity, apparent viscosity, gel strength, soil column, and other miscellaneous tests. The following is a list of performance requirements imposed on grout formulation studies:

Study	Requirement
• 7-d drainable water	0 vol %
• 28-d compressive strength	$\geq$50 psi, expected 200–800 psi
• Compressive strength after freeze/thaw	>200 psi
• Hydraulic conductivity	$\leq 1 \times 10^{-7}$ cm/s
• 10-min gel strength	$\leq 100\ lb_f/100\ ft^2$
• Shrinkage during curing	<1 vol %

With the exception of 10-min gel strength, all requirements were met satisfactorily for seven tested soil grout mixes (Table S.1). The 10-min gel strength tests were not attempted on the soil grouts because of the thickness of the mixes. The mixes exhibited the approximate thickness of conventional concrete that can be pumped.

The three ordinary particulate grouts listed in Table S.1 passed all requirement tests satisfactorily. Six additional ordinary particulate mixes were prepared from dry-solid blends containing 20 wt % type I,II cement. These grouts were not considered to be completely satisfactory,

primarily because of the softness exhibited after 18-d curing which disallowed hydraulic conductivity tests.

All three fine grout mixes that were tested (Table S.1) passed the requirement tests satisfactorily.

Table S.1. Summary of recommended grout compositions

Grout type	Uses	Type I,II Portland cement (wt %)	INEL soil (wt %)	Class C fly ash (wt %)	Bentonite clay (wt %)	Microfine cement (wt %)	Water-to-cement (weight ratio)	Additives
Soil	Open trench and drum disposals	22.5–38.5	30–40	10–20	–	–	0.67–1.00	a
Ordinary particulate	Fill large voids in closed trench soil/waste matrices	35–40	–	15–25	5	–	0.78–1.00	b
Fine particulate	Fill small voids; establish grout soil barrier around closed trenches	–	–	–	–	50–56	0.83–1.00	c

[a]0.2 to 0.8 wt % Dowell D-65 fluidizer.

[b]0.5 to 0.7 wt % Dowell D-65 fluidizer.

[c]0.02 wt % CFR-1 set retarder.

GROUT TESTING AND CHARACTERIZATION FOR SHALLOW-LAND BURIAL TRENCHES AT THE IDAHO NATIONAL ENGINEERING LABORATORY

O. K. Tallent, T. L. Sams, T. Tamura, T. T. Godsey, C. L. Francis, and E. W. McDaniel, Group Leader

ABSTRACT

An investigation was conducted to develop grout formulations suitable for in situ stabilization of low-level and transuranic (TRU) waste in shallow-land burial trenches at Idaho National Engineering Laboratory (INEL). The acceptabilities of soil, ordinary particulate, and fine particulate grouts were evaluated based on phase separation, compressive strength, freeze/thaw, penetration resistance, rheological, water permeability, column, and other tests. Soil grouts with soil-to-cement weight ratios from 0.91 to 1.60 were found to be suitable for open trench or drum disposal. Ordinary particulate grouts containing type I,II Portland cement, class C fly ash, bentonite, water, and a fluidizer were formulated to fill large voids within the soil/waste matrix of a closed shallow-land burial trench. Fine particulate grouts containing fine (mean particle size, 9.6 m) cement and water were formulated to fill smaller voids and to establish a grout-soil barrier to prevent water intrusion into the grouted waste trench. Solution, or chemical, grouts were evaluated as possible substitutes for the fine particulate grouts.

1. INTRODUCTION

Approximately 2.2 x 10 ft of transuranic (TRU) waste has been disposed of in shallow-land burial at the Idaho National Engineering Laboratory (INEL).[1] EG&G Idaho, Inc., prime operating contractor at INEL, has developed a long-range plan for buried TRU waste studies at INEL (EGG-2350). This plan details specific technology studies to be applied toward long-term management of the INEL TRU waste.[1] During FY 1985 and FY 1986, improved-confinement technologies have been investigated by EG&G Idaho Waste Technology Programs. The improved-confinement technology to be investigated is in situ grouting in an arid environment. Oak Ridge National Laboratory (ORNL) is providing technical support and consultation services to EG&G Idaho in the area of grout selection. ORNL is providing the rationale, laboratory comparative results of different grout formulations and grout chemicals with INEL soils, cost comparisons, and the final selection of the recommended grout formulations for INEL in-situ grouting test. This report deals specifically with grout formulation and selection.

A suite of grouts was selected for investigation by ORNL for application in an in-situ field test at the INEL. The suite of grouts consists of the following types:

1. ordinary particulate (cement, fly ash, bentonite);
2. soil;
3. fine (microfine) particulate;* and
4. solution.

These grouts are expected to perform different specific functions in the in-situ application to INEL buried TRU waste. A brief discussion of each is presented below.

1. The primary purpose of the ordinary particulate grout would be to fill large voids** within the soil/waste matrix. This procedure would reduce the cost of using relatively expensive fine or solution grouts in waste zones that require a void filler material rather than a grout that would fill the macroscopic spaces between individual soil grouts.
2. Soil grouts are a subgroup of particulate grouts, differing in that they employ soil or dirt as a component in the grout formulation. The INEL has several hundred thousand cubic feet of low-level radioactive contaminated soil produced by decontamination and decommissioning (D&D) activities; this soil may be utilized as a grout in filling voids in waste containers or pits. Thus, the feasibility of using INEL soil as a component in a grout formulation is being considered. The most desirable characteristic is that the soil grout have rheological properties which allow it to be applied in situ into closed waste trenches. However, if the soil grout could not be applied in situ in closed waste pits, it would have other applications (e.g., as a filler material in operational or open pits).

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*See Sect. 3.3 for explanation of microfine particulate.

**Large voids are defined as those spaces within the waste/soil matrix through which trench cover material can enter.

Hence, the following soil grout application characteristics hierarchy allows for the formulated rheology to match the various application options. The hierarchy is as follows:

(a) providing in-situ injection in closed waste pits,

(b) filling space around containers in open waste pits, and

(c) filling voids in waste containers prior to waste container emplacement in low-level-waste disposal pits.

3. The purpose of fine particulate grouts is to penetrate and fill all the accessible voids that the soil or particulate grouts could not penetrate. Furthermore, fine particulate grouts will be able to penetrate into the surrounding soil walls and backfill and establish a grout/soil barrier (grout curtain) to prevent any water intrusion into the grouted waste pit, especially from lateral water movement.

4. The purpose of the solution grouts is to serve as a backup option for the fine particulate grouts in the event that the latter group proves to be unsatisfactory. Because of the temporal and financial limitations of this project, the large number of solution grouts available were not considered for experimental evaluation. The singular advantage of most of these grouts over the various particulate-based formulations is their absence of suspended particulates. These solutions can penetrate into the smaller soil and rock pores that would normally clog if cement-based grouts were applied. The penetrability of solution grouts into geologic formations can approach that of water, the fluid from which TRU waste isolation is desired. The disadvantages of solution grouts include generally higher costs for materials, uncertainties regarding toxicity,[2] and the potential susceptibility to physicochemical and/or microbial degradation in the arid Idaho environment. Whether the environmental conditions at the Radioactive Waste Management Complex (RWMC) would pose significant deterrents to the performance of any, or all, solution grouts is, at present, unknown. Particular concerns include potential desiccation damage under the annual drying soil moisture regime and the more remote potential for freeze/thaw damage under harsh winter conditions. Empirical evaluation would be required; however, as mentioned above, financial constraints eliminate this approach.

Potential solution grouts for this demonstration would include sodium silicate, polyacrylamide, phenol-formaldehyde, urea-formaldehyde, chromium-lignosulfonates, polyacrylic acid, polyurethanes, and polyisocyanates. Detailed descriptions of these grouts, each of which is as diverse in its potential formulations and properties as the cement-based grouts, are far beyond the scope of this study. Such descriptions can be found elsewhere.[3]

Because it is difficult to characterize the soil, waste form, leachate properties (if any), and void matrix that exist in a given buried TRU waste pit at a given time, the grout(s) must perform over a broad range of arid environmental conditions. Grout selection for a specific site thus involves matching what is known about the trench backfill material, waste forms, leachate (if any), and void matrix with the properties of the candidate grout.[4]

Considering the need for long-term durability of any grout used to stabilize trenches containing TRU wastes, the inorganic systems offer durable grouts with minimal health hazard. The development and manufacture of ultrafine cement (see Sect. 3.3) in Japan[5] and its proven large-scale use in the United States[6,7] offer application in inorganic grouting of trenches that require reduction of infiltration and subsequent leaching by water.

In addition to viscosity as a guide to penetrability, a relationship exists between the particle size of the grout components and the pore size of the soil matrix, which controls penetrability. For optimum results, grout particle size should not be greater than 10% of the soil grain size. We have investigated the use of a fine cement with a mean particle size of 9.6 μm, which is a much smaller particle size than for ordinary grouts. A finer-grind cement, generally referred to as ultrafine cement, is manufactured in Japan; however, it is not readily available in this country and, because of programmatic time restraints, was not available for this investigation.

2. GROUT CHARACTERISTICS AND PERFORMANCE CRITERIA REQUIREMENTS[1]

The characteristics listed below address the major performance-acceptance criteria parameters for grout selection for the "cold" field test at the INEL RWMC. Generic and specific acceptance characteristics are as follows:

1. Grout Emplacement Techniques. The grout emplacement techniques for in situ application in closed buried waste pits shall not fracture the top backfill material over the waste forms or disperse emplaced radionuclides into the environment or into the basalt layer under the top sediment layer of the burial ground.

   Soil grout application criteria require the grout to be sufficiently fluid to flow 30 ft horizontally and 30-ft free fall vertically without congealing. The soil grout rheology shall be able to flow around waste containers in open pits. It is also desirable for the grout to be injected in situ under pressure into accessible voids of a closed pit, as noted previously in the discussion of application hierarchy for soil grouts.

2. Hydraulic Conductivity. There shall be a minimum of two orders-of-magnitude reduction in hydraulic permeability or hydraulic conductivity in the host material; laboratory and field verification shall be as specified by ORNL.

3. Flow Characteristics. The grout in an in-situ application shall flow easily through small passages over short pathways (2 to 3 ft) without plugging these passages. Further chemical or fine-cement grout curtain emplacement shall require the grout to penetrate soil matrix pores without plugging. Mixing-formulation conditions for any grout formula must be specified by ORNL because the manner in which the various components are added can materially affect grout properties such as consistency and set time.

4. Grout Set and Cure Time. The grout set and cure time shall be established by specific application. However, in all cases, the grout shall exhibit 95% of the required properties within 28 d of injection, without further maintenance of the injected field. Some application will require a fast set time, such as within 4 h. This is true when

sealing off the basalt fractures from the top sediment layer. Set times are not critical with soil or particulate grout applications.

5. Unconfined Compressive Strength. The unconfined compressive strength shall be a minimum of 50 psi, with expected grout strengths in the range of 200 to 800 psi. The combined soil, waste material and form, and grout matrix shall support, without any subsidence, the weight of any RWMC operational vehicle that may traverse the trench during normal maintenance. This operation is performed without the presence of a trench cap.

6. Grout Shrinkage. The grout shrinkage shall be minimized. Less than 1% volume shrinkage is acceptable for the soil or particulate grout application. Chemical grout shrinkage via desiccation shall not promote subsidence in the waste pits and shall not affect the grout performance objectives. All grout formulations shall resist or preclude syneresis phenomena in unsaturated conditions in Idaho.

7. Phase Separation. Grouts shall not initiate a water front during phase separation in curing of the grout. No mobile water shall be generated by the curing or setting process.

8. Freeze/Thaw Deterioration. In the test phase, a determination will be made of the historical recorded temperature extremes at a 3-ft soil depth at the Soil Conservation Service Aberdeen Test Station. Thirty cycles will be initiated with the natural humidity of the soil at depth. No grout deterioration from this test shall be accepted.

9. Waste Container Distortion, or loss of integrity is acceptable as long as the grout fills the vacated void.

10. Vibrational Resistance. The grouted soil and waste forms shall be capable of withstanding vibration from nominal heavy equipment traffic over the trench.

11. Chemical and Particulate Grouts. Both chemical and particulate grouts shall be chemically and physically compatible with each other. During injection operations, the chemical grout will be applied in the same regions where particulate grouts have been applied. It is essential that these two formulations do not affect each other's set times or final characteristics.

3. GROUT MIX COMPOSITIONS

3.1 SOIL-GROUT

The compositions of the soil grout mixes that were tested in this study are shown in Table 1. The cement in the mixes acts as a binder. The ASTM class C fly ash component serves as an additional binder, as well as to improve flow properties of fresh-mix grout, to hold water, to minimize void space, and to form crystalline structures that act as a source of internal stabilization in the soil system.[4] The fluidizer also improves the flow properties of the freshly mixed grouts. Partial analyses of the type I,II Portland cement and the ASTM class C fly ash are shown, respectively, in Tables 2 and 3. The particle size distribution of the INEL soil is shown in Figs. 1 and 2. The soil had a surface area of 52.7 m /g, a real density of 2.75 g/cm , and a moisture content of 13.0 wt %. The soil surface area is a function of particle size and, thus, is significant to the soil grout penetrability. Real density is the density excluding all void volumes as determined by using an autopycnometer with helium gas. The compositions of the nine grout mixes listed in Table 1 were chosen based on results obtained from exploratory tests. Soil particles >7.0 mm in diameter (5 to 8 wt % of the soil) were sieved out of the soil before it was used.

Due to the cost of the cement, it is important that the soil-to-cement weight ratio be as great as possible, assuming other performance criteria are met. The soil-to-cement weight ratios in the tested mixes varied from 0.78 to 1.60, as shown in Table 1. Grouts with >40 wt % soil were not tested because of programmatic, financial, and time constraints. The mixing procedure consisted of adding the soils to the water in a Model N-50 Hobart Mixer within a period of 30 s at a low stirring rate, ~135 rpm, and then increasing the rate to 285 rpm for 30 s.

3.2 ORDINARY-PARTICULATE GROUTS

Tests were conducted with two ordinary-particulate grout series, denoted as Ordinary-Particulate Grout Series I and Ordinary-Particulate Grout Series II. The type I,II Portland cement and the ASTM class C fly ash used in these tests are the same as those used in the soil-grout mixes

Table 1. Soil-grout mix compositions

	Mix No.								
	1	2	3	4	5	6	7	8	9
Type I, II Portland cement, wt %	25	30	35	27.5	33	38.5	22.5	27	31.5
Water plus fluidizer, wt %	25	20	15	27.5	22	16.5	22.5	18	13.5
INEL soil, wt %	40	40	40	30	30	30	35	35	35
Fly ash (class C),[a] wt %	10	10	10	15	15	15	20	20	20
Fluidizer, wt %[b]	None	0.20	0.42	None	0.22	0.83	0.22	0.41	0.87
. . . .									
Water/cement weight ratio	1.0	0.67	0.43	1.0	0.67	0.43	1.0	0.67	0.43
Soil/cement weight ratio	1.60	1.33	1.14	1.09	0.91	0.78	1.55	1.30	1.11

[a]Purchased from Pozzolanic Northwest, Inc., of Mercer Island, Wash.

[b]Dowell D-65 fluidizer was used.

Table 2. Chemical composition of
type I,II Portland cement

Component or property	wt %
Silicon dioxide (SiO_2)	22.78
Aluminum oxide (Al_2O_3)	3.40
Ferric oxide (Fe_2O_3)	4.96
Magnesium oxide (MgO)	0.91
Sulfur trioxide (SO_3)	2.05
Loss on ignition	1.31
Insoluble residue	0.30
Total alkalies (as Na_2O)	0.40
Tricalcium silicate (Ca_3S)	57.70
Tricalcium aluminate (Ca_3A)	0.60
Fineness, Blaine	3640 cm^2/g

Table 3. Properties and composition of ASTM class C fly ash[a]

Property or component	
Surface area, m^2/g	1.98
Density, g/cm^3	2.47
Moisture, wt %	0.03
SiO_2, wt %	32.17
CaO, wt %	26.69
$Al_2O_3 + Fe_2O_3$	29.14
Na_2O, wt %	1.80
P_2O_5, wt %	1.40
TiO_2, wt %	1.51
Loss on ignition, wt %	0.75
MgO, wt %	5.00
SO_3, wt %	1.75
K_2O, wt %	0.43
C, wt %	0.52

[a]Purchased from Pozzolanic Northwest, Inc. of Mercer Island, Wash.

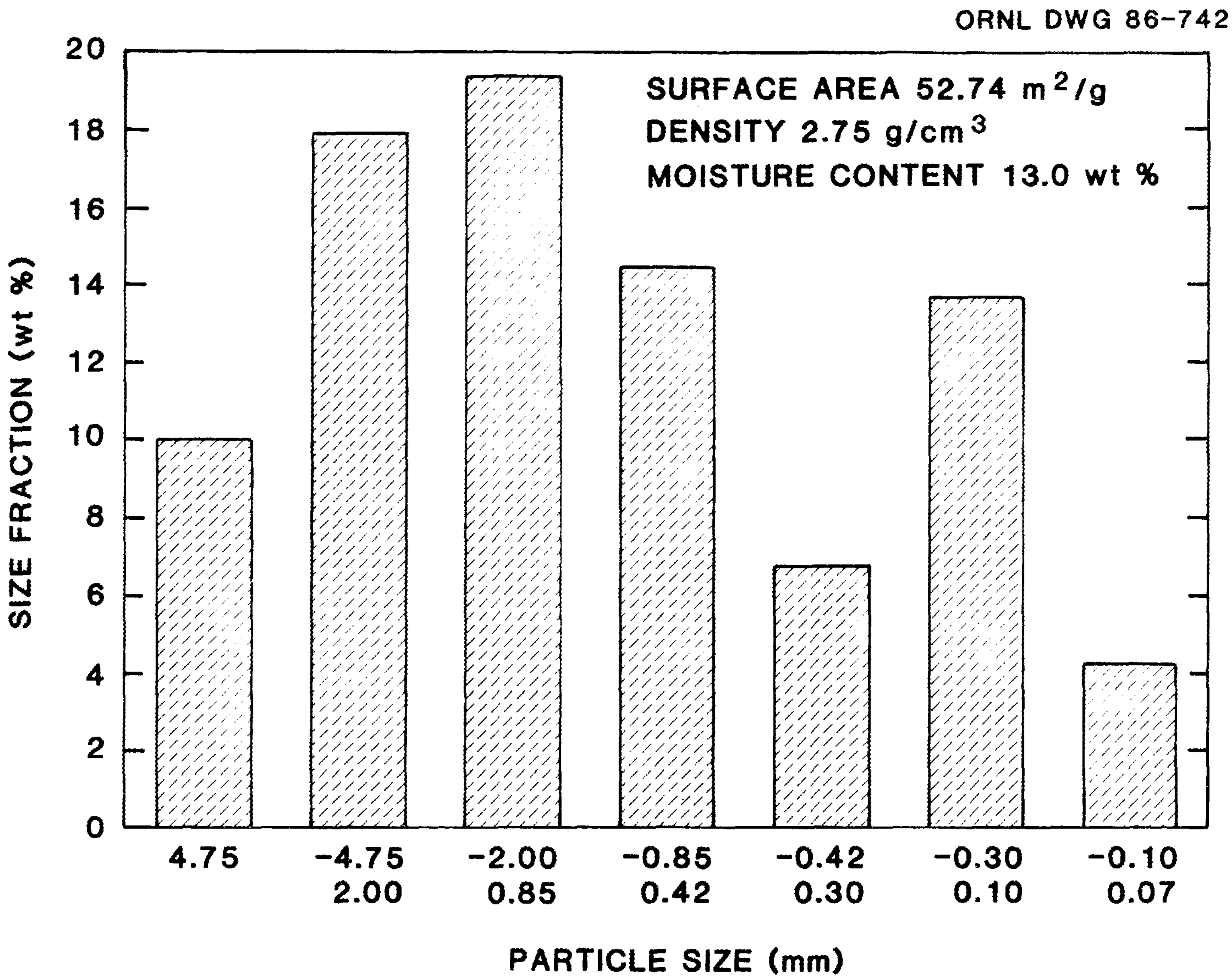

Fig. 1. Particle size distribution of soil from EG&G Idaho.

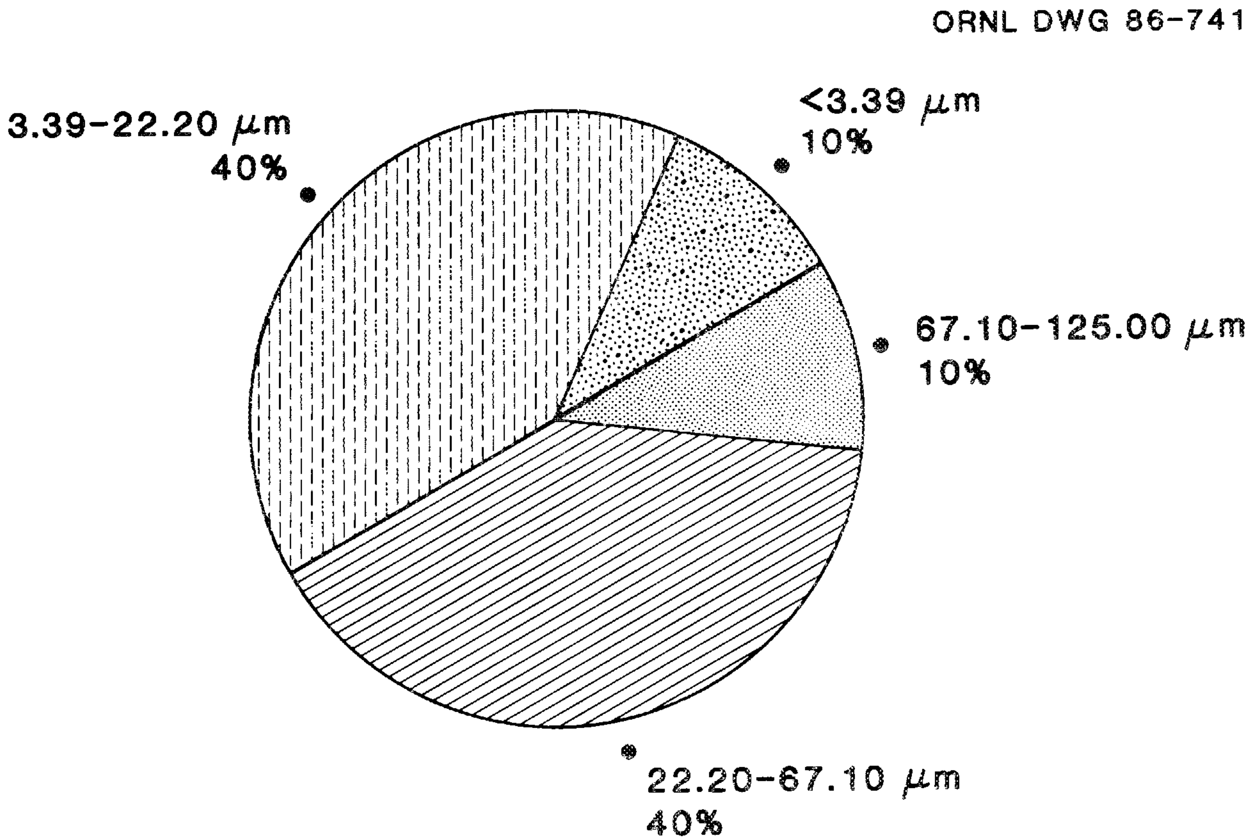

Fig. 2. Particle size distribution of E&G Idaho soil fines, consisting of 3.6 wt % of total soil sample.

(Sect. 3.1) and serve essentially the same purposes as in the soil-grout mixes. The Series II grouts were formulated to set faster and to develop greater compressive strength than the Series I grouts.

The three-dry-solid blend compositions used in the Ordinary-Particulate Grout Series I are shown in Table 4. Bentonite clay is included in blends 2 and 3 to increase the water-retention properties of grouts[8] prepared from these blends. Each of these three dry-solid blends was tumbled in a V blender for 1 h and then mixed with water at 12- and 14-lb/gal mix ratios, yielding a total of six mixes. The mixing procedure was the same as that for the soil grouts. The blend compositions and mix ratios were selected on the basis of the results from exploratory tests.

The compositions of the three mixes tested in Particulate-Grout Series II are shown in Table 5. The mixing procedure was the same as that used for the soil grouts, simulating low shear process or in-field mixing.[9] The reference procedure used for the mixing was ASTM C-192-81.[10]

3.3 MICROFINE-PARTICULATE GROUTS

The fine (or microfine) particulate grout composition and water-to-cement weight ratios selected for testing are listed in Table 6. The fine cement was obtained from Avanti International Co., Webster, Texas. Table 7 shows the physical properties and composition of the cement. The water and cement were mixed in the Hobart mixer for 30 s at the low stirring rate (140 ± 5 rpm) and then for 30 s at the high stirring rate (590 rpm) (see Sect. 3.2). A sugar-type set retarder, CFR-1, was included in the mix to prevent the grout from setting too rapidly.

Some confusion exists concerning the term "microfine" cement. Two finely ground cements from Japan are marketed in the United States. One is marketed by Avanti International Co. as "colloidal" cement but is also known as "microfine" cement. The other is marketed by Geochemical Corp. as "microfine" cement but is also known as "ultrafine" cement. Only the "colloidal" cement from Avanti was available at ORNL when the tests reported here were conducted; references to "microfine" or "fine" cement refer to this Avanti-supplied product. The Geochemical-supplied cement has a smaller size distribution and would presumably be more penetrating than the cement actually tested.

Table 4. Dry-solid blends used in Ordinary Particulate Grout Series I

Material	Blend 1 (wt %)	Blend 2 (wt %)	Blend 3 (wt %)
Type I,II Portland cement	20	20	20
ASTM class C fly ash[a]	80	75	70
Bentonite clay	0	5	10

[a]Purchased from Pozzolanic Northwest, Inc., of Mercer Island, Wash.

Table 5. Compositions of three mixes tested in Ordinary Particulate Grout Series II

Material	Mix 1	Mix 2	Mix 3
Type I,II Portland cement, wt %	35	40	45
Class C fly ash, wt %	25	20	15
Bentonite, wt %	5	5	5
H O + fluidizer, wt %	35	35	35
Fluidizer, wt %[a]	0.50	0.75	0.75
Water/cement weight ratio	1.00	0.87	0.78
Water/solids weight ratio	0.53	0.54	0.54

[a]Dowell D-65 fluidizer was used.

Table 6. Fine (or microfine) particulate grout compositions[a]

Mix ratio[b] (lb/gal)	Water/cement wt ratio
8	1.00
9	0.93
10	0.83

[a]Cement obtained from Avanti International Co., Webster, Texas.

[b]These mixes include 0.02 wt % CFR-1 set retarder.

Table 7. Physical properties and composition of fine (or microfine) cement[a]

Property or component	
Surface area, m^2/g	1.57
Density, g/cm^3	2.41
Mean particle diameter, μm	9.6
SiO_2, wt %	20.51
Na_2O, wt %	0.38
Moisture, wt %	0.38
Loss on ignition, wt %	2.41

[a]Obtained from Avanti International Co., Webster, Texas.

3.4 CHEMICAL, OR SOLUTION, GROUTS

Information available in the literature and discussed in Sect. 1 indicates that chemical, or solution, grouts would probably not be stable in INEL soil because of climatic conditions. Although these grouts were given a low priority due to time constraints, one column test was completed.

4. TEST METHODS, EXPLANATIONS, AND DATA USES

4.1 PHASE SEPARATION TEST (DRAINABLE WATER) METHOD

Phase separation, as previously noted, refers to a separate liquid phase (water) that collects at the top of freshly mixed grout. The volume of liquid is usually found to increase for a short period of time after the grout is mixed and then to decrease to zero with further cure time. The volume of the liquid layer is determined by a settling test in a 1-L plastic bottle. In the test, a known volume of freshly mixed grout, usually 500 mL, is poured into the bottle, which is capped and allowed to stand for time intervals up to 28 d. The phase separation, in vol %, is calculated as the volume of clear drainable surface liquid, hereafter referred to as "drainable water," divided by the total initial grout volume × 100. Since drainable water may contain trace waste substances, it is important that such water be adsorbed or otherwise contained inside the grout matrix after a reasonable cure time.

4.2 COMPRESSIVE-STRENGTH TEST METHOD

Compressive strength for waste grout development is significant in that it is a measure of the structural integrity that grouts are expected to exhibit after curing. Low compressive strength, allowing crushing, would result in increased grout surface area and the possibility of increased leaching. Thus, triplicate or quadruplicate compressive-strength tests were conducted on each grout.

The specimens for the compressive-strength tests were prepared by pouring freshly prepared grout into 2-in.^{3} stainless molds and allowing the molds to stand in a humidity cabinet at approximately 100% relative humidity at room temperature for up to 28 d. The 2-in. cubes were used in accordance with an American Standard For Testing and Materials procedure (ASTM C-109-80).[11] Crushing strengths of the grout cubes were then determined using a Model 60,000 Super "L" Tinius Olsen Testing Machine. The dimensions of the specimens were measured before they were crushed. It is important that the grouts have a compressive strength of $\geq$60 psi to prevent them from cracking and crumbling and thus exposing additional surface to possible leach water.

4.3 FREEZE/THAW TEST METHOD

The specimens for the freeze/thaw tests were prepared in the same manner as for the compressive-strength tests. The tests were conducted using a Model 16635 thermal test chamber, manufactured by Despatch Industries, with a programmable digital controller. The temperature was cycled 30 times between −25 and +60°C, with 50% relative humidity being maintained at the 60°C temperature. The time for each cycle was 4 h — 1 h at −25°C, 1 h to increase the temperature to 60°C, 1 h at 60°C, and 1 h to recycle back to −25°C. A compressive-strength test was conducted on each specimen after the freeze/thaw test. The reference procedure was ASTM C-666-84.[12]

4.4 PENETRATION-RESISTANCE TEST METHOD

Penetration resistance serves as a measure of set time. Penetration-resistance tests were conducted on grout specimens prepared by pouring freshly mixed grouts into 2-in. stainless steel molds. The specimens were allowed to cure in a humidity cabinet for various times, and then the grout penetration resistance was measured using an Acme penetrometer. The reference procedures were ASTM C-403-80 and ASTM C-803-82.

4.5 RHEOLOGICAL MEASUREMENT METHOD

For the purposes of this work, rheological measurements were conducted primarily to measure properties relating to flow properties of the grouts using a Fann direct reading viscometer, Model 35A. The data obtained are used to determine the fluid consistency index, K'; the flow behavior index, n'; the apparent viscosity; and the 10-min gel strength. Since the grouts are non-Newtonian fluids, the values of n' and K' allow the apparent viscosity, μ, to be calculated at any specific shear rate in the laminar flow region from

$$\mu = 47880 K' S_r^{n'-1} \quad , \qquad (1)$$

where the units are cP, $lb_f\ s^{n'}/ft^2$, and s^{-1}, respectively, for μ, K', and S_r the shear rate (n' is unitless).

4.5.1 Fluid Consistency Index, K', and Flow Behavior Index, n'

For non-Newtonian grouts, shear stress is dependent on shear rate and is represented by the Power Law model[13]

$$S_s = K'(S_r)^{n'} \quad , \tag{2}$$

where

S_s = shear stress, lb_f/ft^2,

K' = fluid consistency index, $lb_f s^{n'}/ft^2$,

S_r = shear rate, s^{-1}, and

n' = flow behavior index ($0<n'<1.0$), dimensionless.

Values of n' and K' are determined from the Power Law model for a given set of viscometer-shear-stress vs shear-rate data. An example plot of such data (for 8-lb/gal mix ratio microfine cement grout) with the slope of line, n', equal to 0.5185 and the intercept, K', equal to 0.006 is shown in Fig. 3. Values for n' and K' were determined for each mix except where indicated otherwise.

4.5.2 Density

The density of each freshly mixed grout was directly measured in lb/gal at room temperature using a Baroid mud balance.

4.5.3 Apparent Viscosity

Viscosity in a grout varies with shear rate. The apparent viscosity in these tests was measured at 511 s^{-1} (300 rpm on the Fann Viscometer), which is a common practice.[10]

4.5.4 Gel Strength (10-min)

The 10 min gel strength is a measure of the force required to restart the flow of grout in a pipe after the flow has been stopped for 10 min. The measurement was made in the Fann viscometer with the same grout sample after the other rheological measurements. The grout was allowed to stand in the viscometer for 10 min without stirring, after which the instrument was turned on with the shear rate set at 3.0 rpm. The 10-min gel strength in $lb_f/100\ ft^2$ was read directly from the viscometer at the maximum deflection on the shear stress scale.

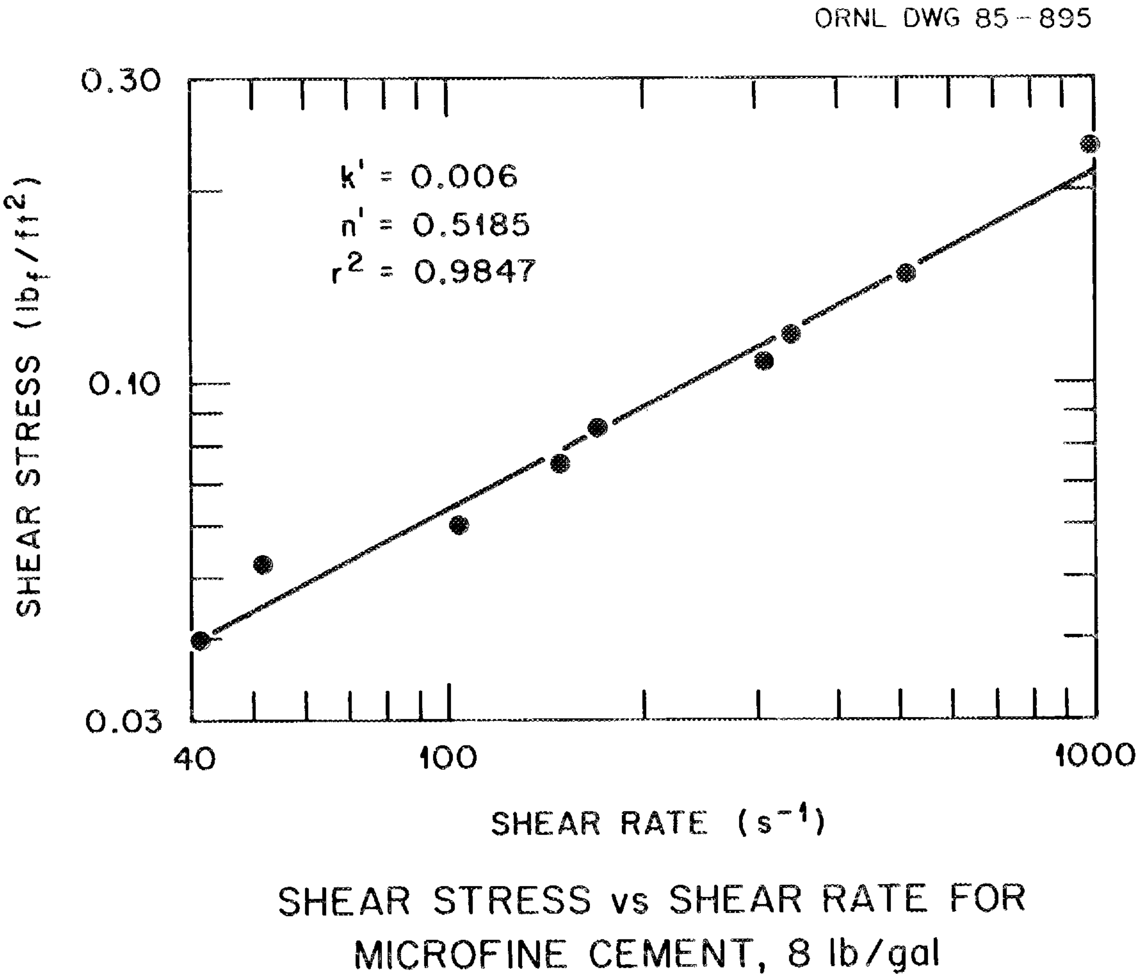

Fig. 3. Shear stress vs shear rate for microfine particulate grout, 8 lb/gal.

4.6 WATER-PERMEABILITY TEST METHOD

A schematic of the test apparatus used for measuring liquid permeability is shown in Fig. 4. One-inch-diameter grout samples cured for a minimum of 28 d were cut to length (approximately 3/4 in.) by a wet cutting wheel. The exact diameter and length of each specimen were measured with calipers. Each specimen was then rinsed with water and loaded into the sample chamber after the heavy-wall rubber tube was pulled back by a vacuum exerted through the Hassler opening (see Fig. 4). The line and inner cell chamber were then filled with water from the stainless steel liquid reservoir. A sealing pressure of approximately 300 psi was exerted on the sample by the heavy-wall rubber tubing as the result of water in the space between the outer walls of the Hassler cell and the rubber tubing. A differential water pressure of 2 to 4 atm was exerted across the sample. The flow rate was calculated by weighing the amount of water flowing through the sample per unit time on an electronic balance. The balance was enclosed in a shield with a wet paper towel inside to water saturate the surrounding air.

The permeability coefficient, K_a, was determined from the slope of a plot implementing Darcy's Law, which is expressed as:

$$K_a = \frac{\mu QL}{PA} , \qquad (3)$$

where

K_a = permeability coefficient, darcy;
μ = viscosity of the working fluid, cP;
Q = flow rate, cm^3/s;
L = length of the sample, cm;
P = pressure gradient $(P_1 - P_2)$, atm; and
A = cross section of the sample, cm^2.

The unit most widely used to represent permeability is the darcy. This unit is defined as the permeability that results in a flow rate of 1 cm^3 per second of fluid with a viscosity of 1 cP through a 1-cm cube at a pressure differential of 1 atm. Thus,

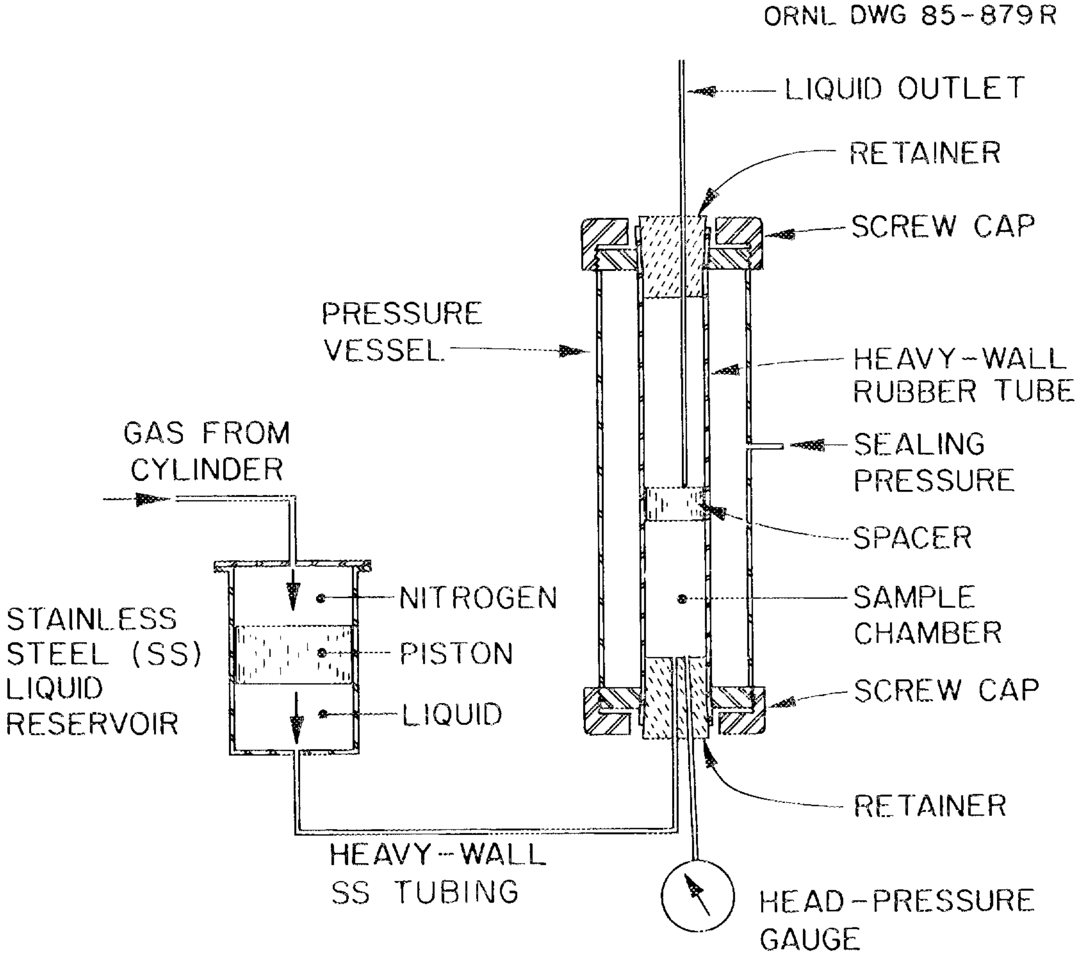

Fig. 4. Schematic of Hassler permeability cell.

$$1 \text{ darcy} = \frac{1\ (\text{cm}^3/\text{s}) \cdot 1\ (\text{cP})}{1\ (\text{cm}^2) \cdot 1\ (\text{atm/cm})} \qquad (4)$$

The permeability coefficients of the grout samples were determined from plots of $\mu Q/A$ vs P/L. Three points, at pressure gradients of 2, 3, and 4 atm were plotted, and the slope of the resulting straight line was taken as the permeability. In some instances, the permeability was so low that only one data point was obtained; the permeability is reported as a "$\leq$" value. An example plot, for soil grout mix 7, is shown in Fig. 5. It should be noted that permeabilities are related to hydraulic conductivity by the following equation,[14]

$$K_B = \frac{K_a d g}{\mu} , \qquad (5)$$

where

K_B = hydraulic conductivity, cm/s;

d = density of the fluid, g/cm^3;

g = acceleration due to gravity, cm/s^2; and

μ = viscosity, cP.

In effect, the permeability coefficient, K_a, can be multiplied by the factor 9.71×10^{-4} to obtain the hydraulic conductivity, K_B. Both K_a and K_B values are reported in appropriate tables in Sect. 5. The K_B value corresponds to the "falling head permeability" values as reported in the document, Soil Testing Results, Subcontract K-1801, Task Order No. 1, for EG&G Idaho, Inc. Low hydraulic conductivity is desirable to minimize the release of waste substances from the grouts via water solutions.

4.7 COLUMN AND OTHER MISCELLANEOUS TEST METHODS

The purpose of the columns and other miscellaneous tests was to obtain additional information and data pertaining to the flow properties of the grouts into the soil and the chemical and physical compatibility of the grouts with each other.

The column tests were conducted by injecting grouts under 15-psi air pressure through a perforated 2-in.-ID lance into a column of INEL soil. The column was constructed with a 30-in. length of 6.5-in.-ID methyl

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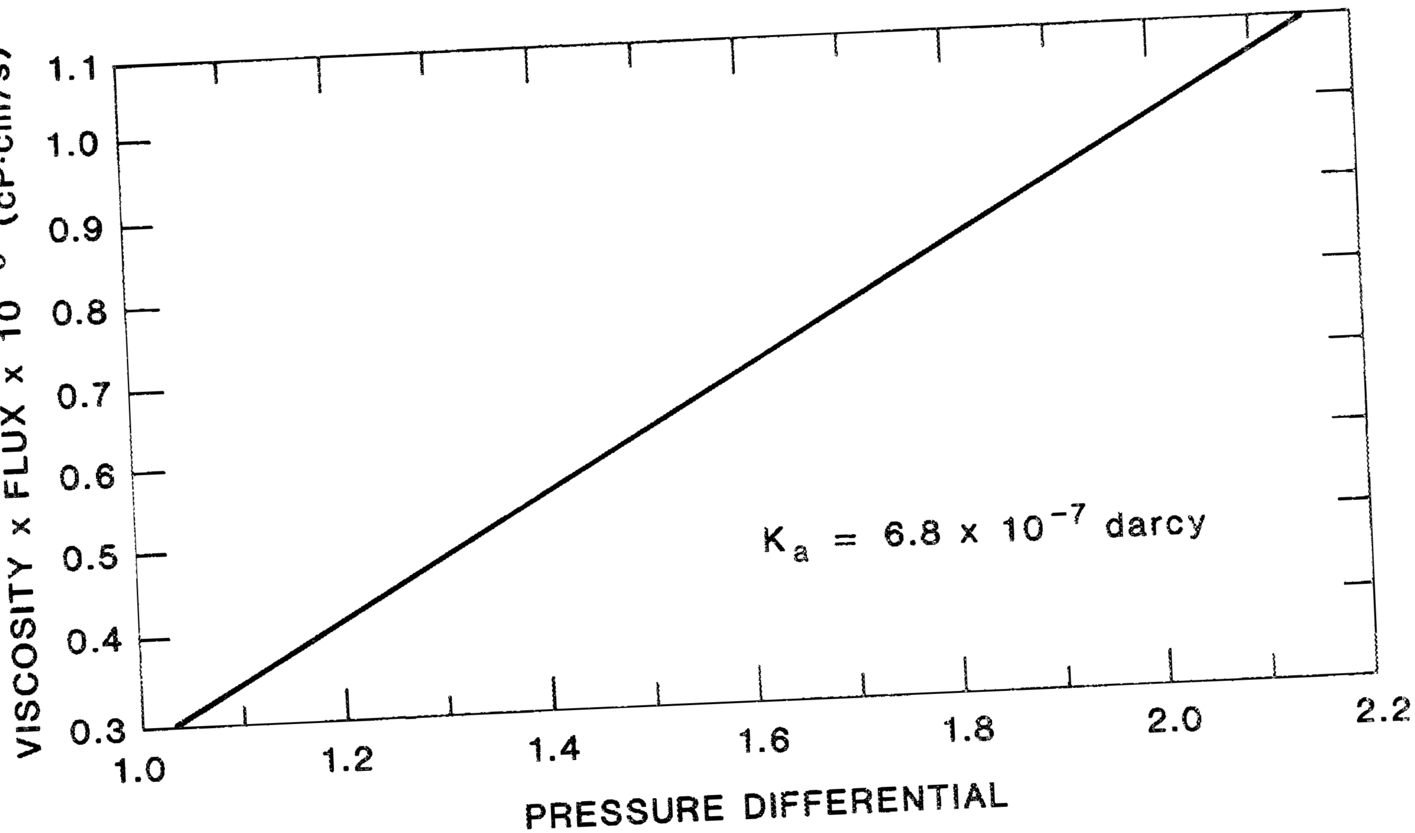

Fig. 5. Plot of viscosity times flux (~Q/A) vs pressure differential (ΔP/L) for soil grout mix 7.

methacrylate tubing packed with soil to a density of 1.10 g/cm^3 (69 lb/ft^3). Ordinary Particulate Grout Series II Mix 1 (Table 4) was injected into the soil in two 4-in. increments of depth starting at the bottom of the column. Fine particulate grout, 8-lb/gal mix ratio, was injected into a second column using the same procedure. A 20% sodium silicate chemical cement was injected into a third column. The results from the column tests cannot be directly extrapolated to actual grout placement in the field. These tests are intended only to show relative penetrations of grouts into disturbed INEL soil.

Other miscellaneous tests were conducted in which compressive-strength specimens were prepared so that the bottom halves of the specimens were poured (2-in. cube) using the above particulate grout and the top halves poured using the microfine-particulate grout. Compressive-strength tests were conducted on 15-d cured specimens. The specimens were also examined under a microscope to discern the extent to which the materials in the two grouts were diffused into each other. Similar tests were conducted to determine the effects, if any, on drainable water of pouring the freshly mixed grouts together.

5. TEST RESULTS AND DISCUSSION

The performance criteria used to determine the acceptability of the grouts are summarized below:

Criterion	Requirement
7-d drainable water	0 vol %
28-d compressive strength	≥50 psi, expected 200–800 psi
Compressive strength after freeze/thaw	>60 psi
Hydraulic conductivity	$\leq 1 \times 10^{-7}$ cm/s
10-min gel strength	$\leq 100\ lb_f/100\ ft^2$
Shrinkage during curing	<1 vol %

5.1 SOIL-GROUT TEST RESULTS

The results of the soil-grout tests are shown in Table 8. Data for mixes 3 and 9 are excluded from the table because of the poor fluid consistency of these mixes. The drainable water was 0 vol % in 24 h for all of the other mixes listed in the table except mix 1, which was 0 vol % in 48 h. The 7-d compressive strength values for the grouts (Table 8) were greater than 1500 psi, well above the 50 psi that would be required after 28 d (28-d tests were not conducted due to program time constraints). No shrinkages of the specimens during curing were detected. The 28-d compressive strength values were >1800 psi for all the mixes after the freeze/thaw tests; thus, all the grouts listed in the table passed this test. The hydraulic conductivity of three of the grouts after 28 d of curing ranged from 2×10^{-9} to $\leq 1 \times 10^{-10}$ cm/s.

The fluid consistency of each of the soil grouts tested was so thick that meaningful rheological measurements could not be obtained using the Fann Direct Reading Viscometer. Because of the poor fluid consistency problem, all of the soil-grouts tests are not recommended for injection into closed shallow-land burial trenches without further investigation. The soil-grouts tested and listed in Table 8 are, however, suitable for use as a filler material in open or closed trenches, or for drum disposals. Mixes 1 and 7 would be preferred based on their soil-to-cement ratios of ~1.6. The thickness of the grouts appeared to be similar to that of

Table 8. Results of soil-grout tests

	Mix No.						
	1	2	4	5	6	7	8
24-h phase separation, vol % (drainable water)	[a]	0	0	0	0	0	0
7-d compressive strength, psi	1577 ± 25	3754 ± 141	1898 ± 37	3736 ± 158	6320 ± 235	2432 ± 106	4017 ± 325
28-d compressive strength after freeze/thaw, psi	1835	3933	2213	3955	8180	2843	6388
Density, lb/gal	15.6	15.9	15.2	16.5	17.3	15.9	16.7
24-h penetration resistance, psi	>8000	>8000	7600	>8000	>8000	>8000	>8000
Water permeability, darcy	2×10^{-6}	ND[c]	$\leq 1 \times 10^{-7}$	ND	ND	7×10^{-7}	ND
Hydraulic conductivity, cm/s	2×10^{-9}	ND	$\leq 1 \times 10^{-10}$	ND	ND	7×10^{-10}	ND

[a] 0 vol % after 48 h.
[b] Relationship between water permeability and hydraulic conductivity discussed in Sect. 4.6.
[c] ND = Not determined.

conventional concretes (e.g., concretes that would be poured in home patios). The pumping of such concretes has been reported[13] and discussed[14] in open literature.

5.2 RESULTS OF ORDINARY-PARTICULATE GROUT TESTS

Results of the Ordinary-Particulate Grout Series I tests for dry-solid blends 2, and 3 are shown, respectively, in Tables 9, 10, and 11. The vol % of drainable water (phase separation) was 0 after 24 h for all of the grouts except the 12-lb/gal blend 1 grout, which failed to go to 0 in 28 d. The 28-d compressive strength values and/or penetration resistance values for blends 1 and 2 (Tables 9 and 10) would normally be considered too low to be acceptable. Some relaxation of performance requirements would be required before these grouts could be used. The grouts prepared from blend 3 developed compressive strengths of ~235 and ~375 for the 12- and 14-lb/gal mixes, respectively. However, these grouts either did not pass or only marginally passed the 10-min gel strength test, which requires a 10-min gel strength of $\leq$100 $lb_f/100\ ft^2$. While none of the grouts in this series (Particulate-Grout Series I) appears to be completely acceptable, it is interesting to observe the effect of bentonite clay on the apparent viscosity and gel strength of the fresh grout mixes. Figure 6 shows that the apparent viscosity (at 300 rpm) increased from 15 to 35 cP for the 12-lb/gal mixes, and from 21 to 60 cP for the 14-lb/gal mixes, as the bentonite clay content in the dry-solid blends was increased from 0.0 to 10.0 wt %. Similarly, Fig. 7 reveals that the 10-min gel strength increased from 52 to 92 $lb_f/100\ ft^2$ for the 12-lb/gal mixes and from 77 to 152 $lb_f/100\ ft^2$ for the 14-lb/gal mixes as the bentonite content in the dry-solid blends was increased from 0.0 to 10.0 wt %.

Results of the Ordinary-Particulate Grout Series II tests are shown in Table 12. The three mixes in this series met the requirements of all tests. For each mix, the drainable water was 0 wt % in less than 48 h; the hydraulic conductivity was $\leq 10^{-7}$ cm/s;* the 28-d compressive strengths before and after the freeze/thaw tests were $\geq$3250 psi; and the 10-min gel

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*The hydraulic conductivity of mix 3 was not determined due to a defective sample; however, the hydraulic conductivity should have been similar to that of the other two mixes.

Table 9. Test results for Ordinary-Particulate Grout Series I, blend 1

Mix ratio, lb/gal	12	14
28-d phase separation, vol %	0.2	0[a]
28-d penetration resistance, psi	188	356
Water permeability, darcy	ND	ND
28-d compressive strength, psi	46, 52, 64	56, 78, 69
28-d compressive strength after freeze/thaw, psi	300	450
Apparent viscosity at 300 rpm, cP	15	21
10-min gel strength, $lb_f/100\ ft^2$	52 ± 4	77 ± 8
Density, lb/gal	13.2	13.8
Fluid consistency index (K'), $lb_f \cdot s^{n'}/ft^2$	0.0055	0.0112
Flow behavior index, n'	0.53	0.47

[a]0 vol % after 24 h.

[b]ND = not determined.

Table 10. Test results for Ordinary-Particulate Grout
Series I, blend 2

Mix ratio, lb/gal	12	14
28-d phase separation, vol %	0	0[a]
28-d penetration resistance, psi	40	88
Water permeability, darcy	ND	ND
28-d compressive strength, psi	266, 232, 161	377, 312, 313
28-d compressive strength after freeze/thaw, psi	206	185
Apparent viscosity at 300 rpm, cP	22	35
10-min gel strength, $lb_f/100\ ft^2$	82 ± 8	87 ± 11
Density, lb/gal	13.2	13.8
Fluid consistency index (K'), $lb_f \cdot s^{n'}/ft^2$	0.0111	0.0240
Flow behavior index, n'	0.48	0.43

[a]0 vol % after 2 h.

[b]ND = not determined.

Table 11. Test results for Ordinary-Particulate Grout Series I, blend 3

Mix ratio, lb/gal	12	14
28-d phase separation, vol %	0	0
28-d penetration resistance, psi)	1720	>4000
Water permeability, darcy	ND	ND
28-d compressive strength, psi	234, 233, 237	417, 368, 345
28-d compressive strength after freeze/thaw, psi	472	842
Apparent viscosity at 300 rpm, cP	35	60
10-min gel strength, $lb_f/100\ ft^2$	92 ± 8	152 ± 28
Density, lb/gal	13.3	13.8
Fluid consistency index, (K'), $lb_f \cdot s^{n'}/ft^2$	0.0257	0.0641
Flow behavior index, n'	0.42	0.36

[a]ND = not determined.

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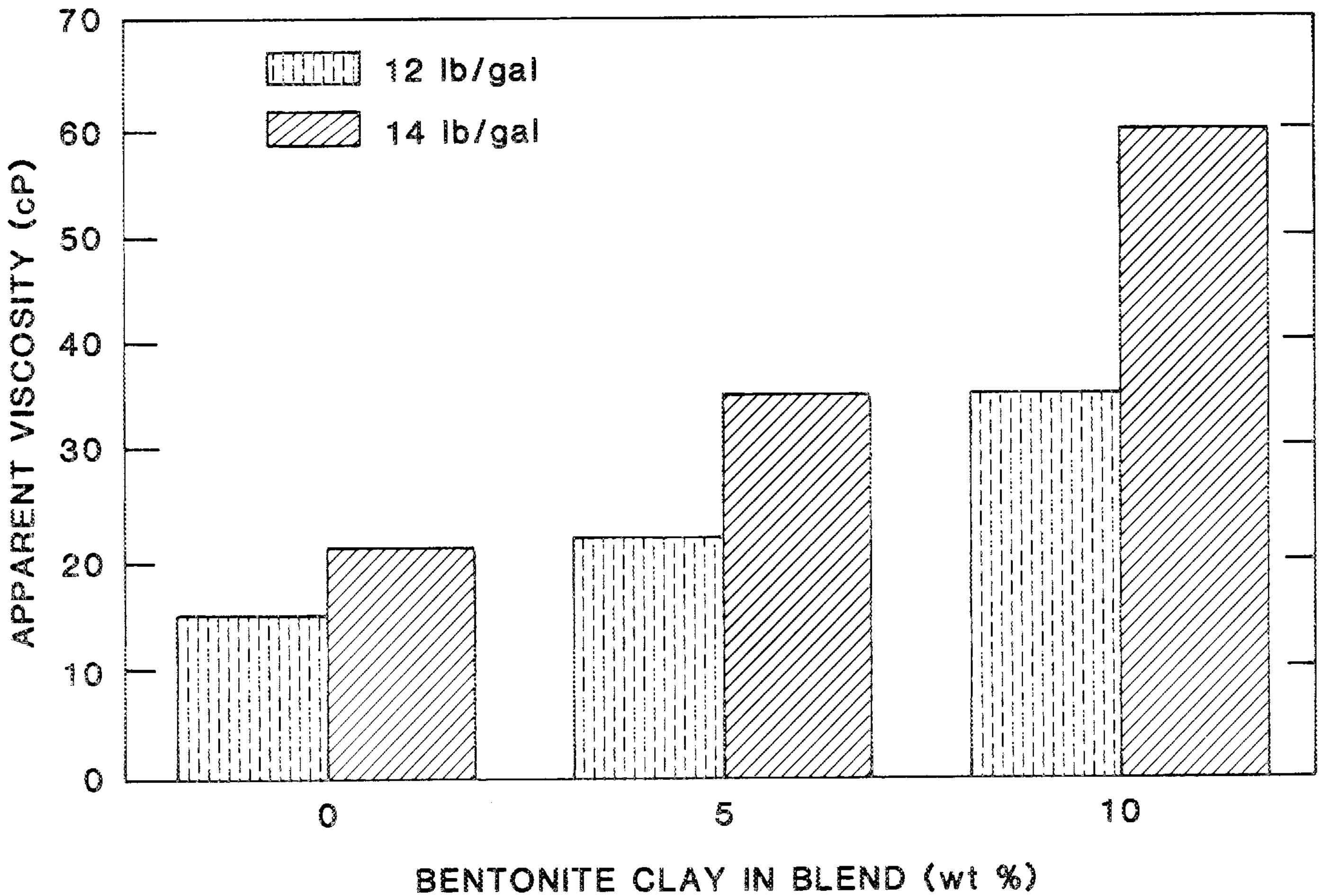

Fig. 6. Effect of dry-solids blend bentonite clay content on apparent viscosities of Particulate Grout Series I grouts.

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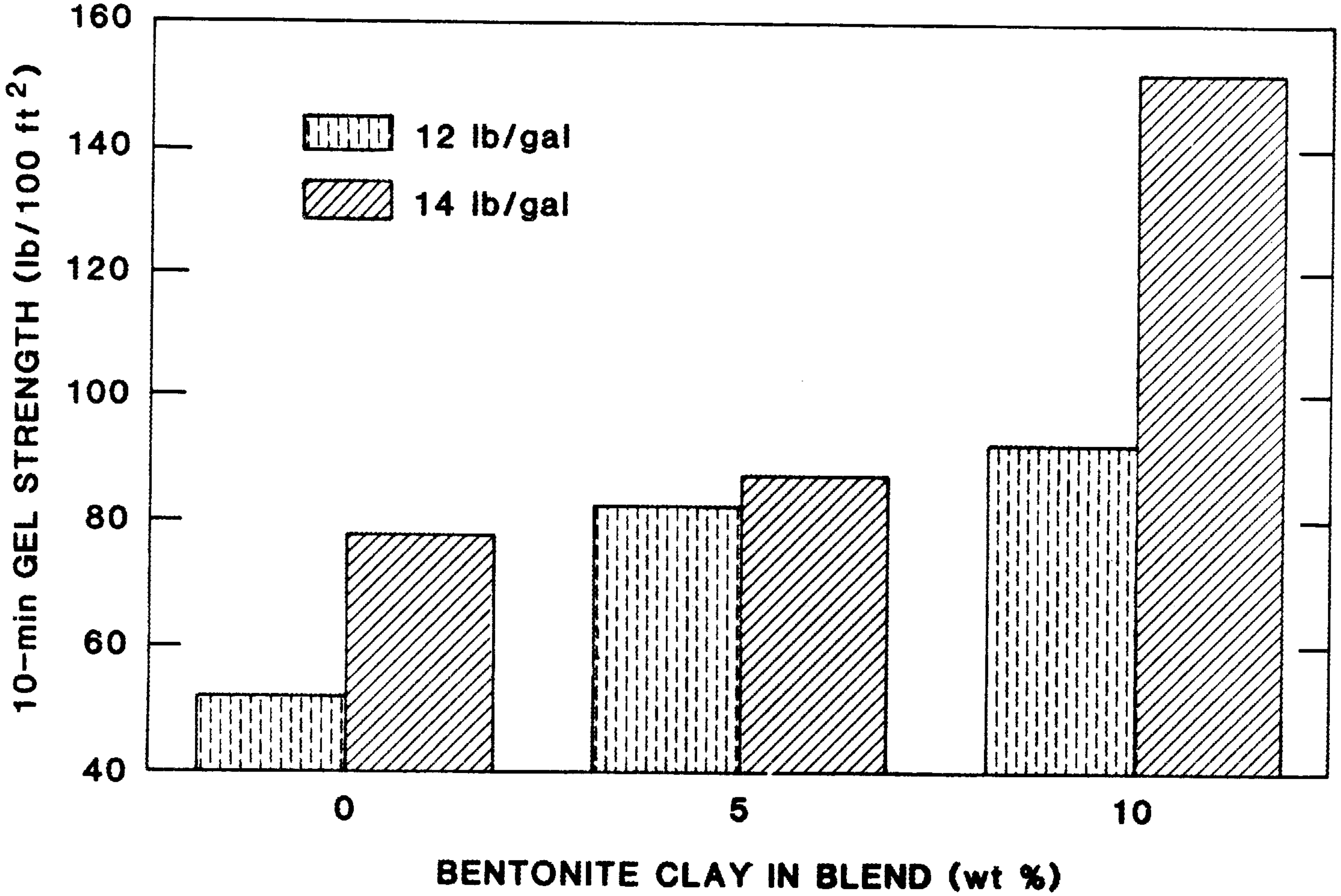

Fig. 7. Effect of dry-solids blend bentonite content on 10-min strengths of Particulate Grout Series I grouts.

Table 12. Results of Ordinary-Particulate Grout Series II tests

	Mix No.		
	1	2	3
5-d phase separation, vol %	0[a]	0	0
48-h penetration resistance, psi	6080	>8000	>8000
Water permeability, darcy	9.7×10^{-7}	6.6×10^{-7}	ND
Hydraulic conductivity,[b] cm/s	9.4×10^{-10}	6.4×10^{-10}	ND
28-d compressive strength, psi	3439 ± 259	3407 ± 248	4160 ± 250
28-d compressive strength after freeze/thaw, psi	3250	3770	4135
Apparent viscosity at 300 rpm, cP	32	33	36
10-min gel strength, $lb_f/100\ ft^2$	42.5 ± 4.9	39 ± 7.1	40.5 ± 7.8
Density, lb/gal	14.5	14.6	14.7
Fluid consistency index, (K'), $lb_f \cdot s^{n'}/ft^2$	0.0017	0.001	0.0009
Flow behavior index, n'	0.84	0.93	0.95

[a]0 vol % after 48 h.

[b]Relationship between water permeability and hydraulic conductivity discussed in Sect. 4.6.

[c]ND = not determined due to defective sample.

strengths were $\leq$43 $lb_f/100\ ft^2$. Shrinkage during curing was <1%. Improved setting (penetration resistance) properties of the Series II grouts over the Series I grouts result from the lower water-to-cement ratios in the former series. The water-to-cement ratio in the Series II grouts ranged from 0.78 to 1.00 as compared with ratios of 3.33 and 2.86 for the respective 12- and 14-lb/gal mix ratio grouts in Series I. The addition of the fluidizer also improved the rheological properties of the Series II grouts. The greatest apparent viscosity of the mixes at 300 rpm was 36 cP, about the same as the viscosity of light machine oil at 38°C. Results of the column tests and miscellaneous other tests are reported in Sect. 5.4.

5.3 RESULTS OF FINE-PARTICULATE GROUT TESTS

Results of the fine-particulate grout tests for 8-, 9-, and 10-lb/gal ratios (Table 6) are shown in Table 13. The three mixes met requirements for all tests. In each test, the drainable water was 0 wt % in less than 48 h; the hydraulic conductivity was $\leq 10^{-7}$ cm/s, the 28-d compressive strengths before and after freeze/thaw tests were $\geq$1415 psi; the freeze/thaw tests did not significantly change the compressive strengths; and the 10-min gel strengths were $\leq$83 $lb_f/100\ ft^2$. No shrinkages of the specimens during curing were detected. The lowest apparent viscosity (at 300 rpm) was 15 cP, which is less than the viscosity of the ethylene glycol at 20°C; the greatest apparent viscosity was 36 cP (at 300 rpm), approximately the same as the viscosity of light machine oil at 38°C. Results of the column and miscellaneous other tests are reported in Sect. 5.4.

5.4 RESULTS OF THE COLUMN AND OTHER MISCELLANEOUS TESTS

A photograph of a column containing 14,651 g of INEL soil in a volume of 13,284 cm^3 is shown on the left in Fig. 8. Based on a soil density of 2.75 g/cm^3, it is calculated that the initial void volume in the column was 60%, or 7970 cm^3. As it appears in the photograph, the column has had 2975 cm^3 of Ordinary Particulate Grout Series II Mix 1 injected into the soil, filling 37% of the void volume. The procedure (Sect. 4.8) did not crack the soil in the top part of the column. Comparable results were obtained with fine-particulate grout injected into a column of INEL soil, shown on the right in Fig. 8. As was expected, the microfine-particulate

Table 13. Results of fine-particulate grout tests

	Mix ratio (lb/gal)		
	8	9	10
24-h phase separation, vol %	0[a]	0	0
24-h penetration resistance, psi	2320	4480	7600
48-h penetration resistance, psi	6160	ND[b]	ND
Water permeability, darcy	9.5×10^{-6}	1.3×10^{-5}	8.6×10^{-7}
Hydraulic conductivity,[c] cm/s	9.3×10^{-9}	1.2×10^{-8}	8.3×10^{-10}
28-d compressive strength, psi	1978, 1925	2345, 2420 2418	2775, 2870 2858
28-d compressive strength after freeze/thaw, psi	1415	1905	2810
Apparent viscosity at 300 rpm, cP	15	22	36
10-min gel strength, $lb_f/100\ ft^2$	53.75 ± 10.8	55.75 ± 10.7	82.25 ± 27.9
Density, lb/gal	12.5	12.9	13.2
Fluid consistency index, (K'), $lb_f \cdot s^{n'}/ft^2$	0.0062	0.0113	0.0169
Flow behavior index, n'	0.51	0.48	0.49

[a]0 vol % after 48 h.

[b]ND = not determined.

[c]Relationship between water permeability and hydraulic conductivity discussed in Sect. 4.6.

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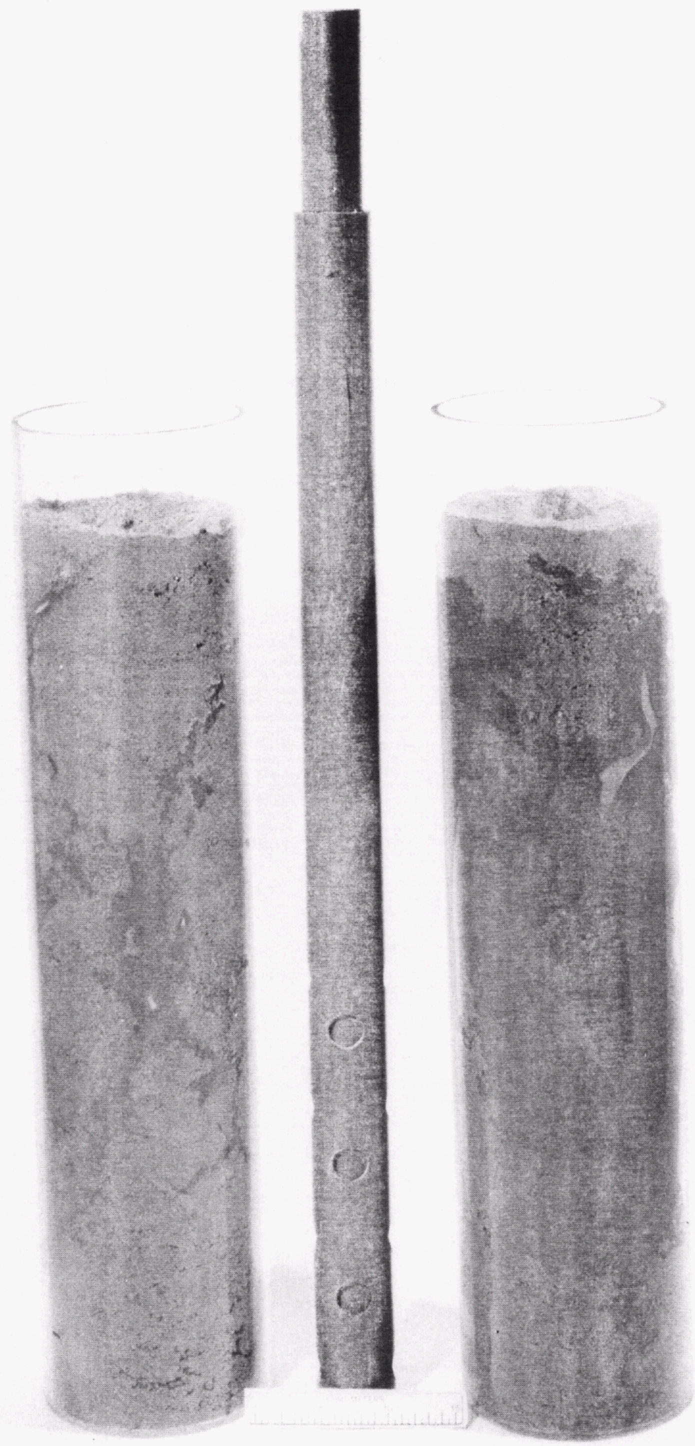

Fig. 8. Column tests with INEL soil.

grout filled a larger fraction, 52%, of the soil void volume. The lance used to inject the grout into the soil columns is shown between the columns in the figure. Samples taken from a similarly prepared soil column with ordinary-particulate grout are shown in Fig. 9. The dark-solid material in the sample is grout with the lighter-colored, more-porous material being the soil. In a chemical-cement column test similar to the above tests, 48% of the soil void volume was filled.

Inspection of specimens prepared with 8-lb/gal fine-grout mix poured over mix 1 of Ordinary Particulate Grout Series II showed satisfactory bonding at the boundary between the mixes. Considerable diffusion of the microfine grout into the other grout appeared to have occurred. No change in the phase separation properties of the combined mixes was observed. The 15-d compressive strength of the mixes was 1868 psi.

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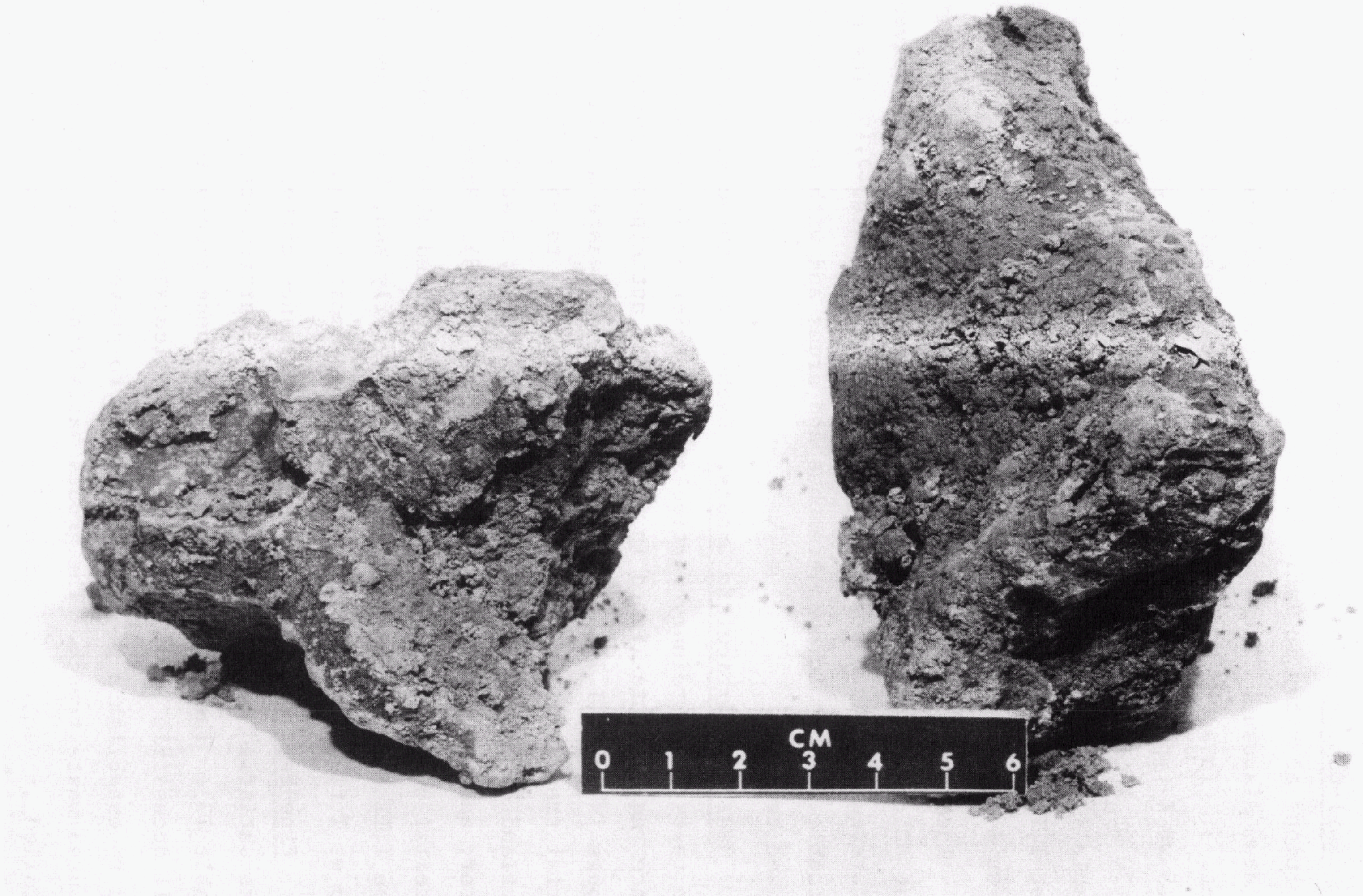

Fig. 9. Samples of soil with injected ordinary particulate grout.

6. CONCLUSIONS AND RECOMMENDATIONS

Soil-Grouts. The seven soil-grouts listed in Table 8, with compositions as shown in Table 1, are suitable formulations for open-trench or drum disposal. These grouts satisfy the performance criteria listed in Sect. 5. In addition to water, the grouts contained from 22.5 to 38.5 wt % type I,II Portland cement; from 30 to 40 wt % INEL soil; from 10 to 20 wt % class C fly ash; and from 0.00 to 0.083 wt % fluidizer (Table 1). The most economical grout was mix 1, which contained 25 wt % type I,II Portland cement, 25 wt % water, 40 wt % INEL/RWMC soil, and 10 wt % class C fly ash.

The thickness of each soil-grout mixture appeared to be similar to that of conventional concretes. The pumping properties of conventional concretes have been reported in the open literature.[15,16] The grouts are suitable for use in open trenches and drums and with proper pumps[15,16] in closed trenches.

Ordinary-Particulate Grouts. Each of the three mixes investigated in the tests with Ordinary Particulate Grout Series II satisfied the performance requirements listed in Sect. 5. The anticipated purpose of these grouts is to fill large voids within the soil/waste matrix in shallow-land burial trenches. The grouts contained from 35 to 45 wt % type I,II Portland cement; 15 to 25 wt % class C fly ash; 5 wt % bentonite; 35 wt % water; and 0.50 to 0.75 wt % fluidizer (Table 5). From the standpoint of material costs, mix 1 (Table 5), which contained 35 wt % water, 35 wt % type I,II Portland cement, 25 wt % class C fly ash, 5 wt % bentonite, and 0.5 wt % fluidizer (in the water) was the most economical.

Fine-Particulate Grouts. The performance criteria listed in Sect. 5 were satisfied by each of three microfine-particulate grout mixes tested (Table 13). The anticipated purpose of these grouts is to penetrate and fill accessible voids that ordinary-particulate grouts do not penetrate and to establish a grout/soil barrier to prevent water intrusion into the grouted waste trench. The grouts satisfying the performance criteria detailed in Sect. 5 contained fine (microfine) cement (Table 7) mixed with water at 8-, 9-, and 10-lb/gal mix ratios and 0.02 wt % CFR-1 sugar set retarder.

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