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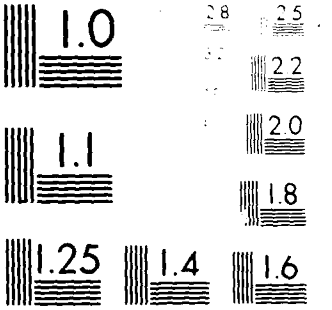

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GROUT FORMULATIONS FOR NUCLEAR WASTE ISOLATION

by

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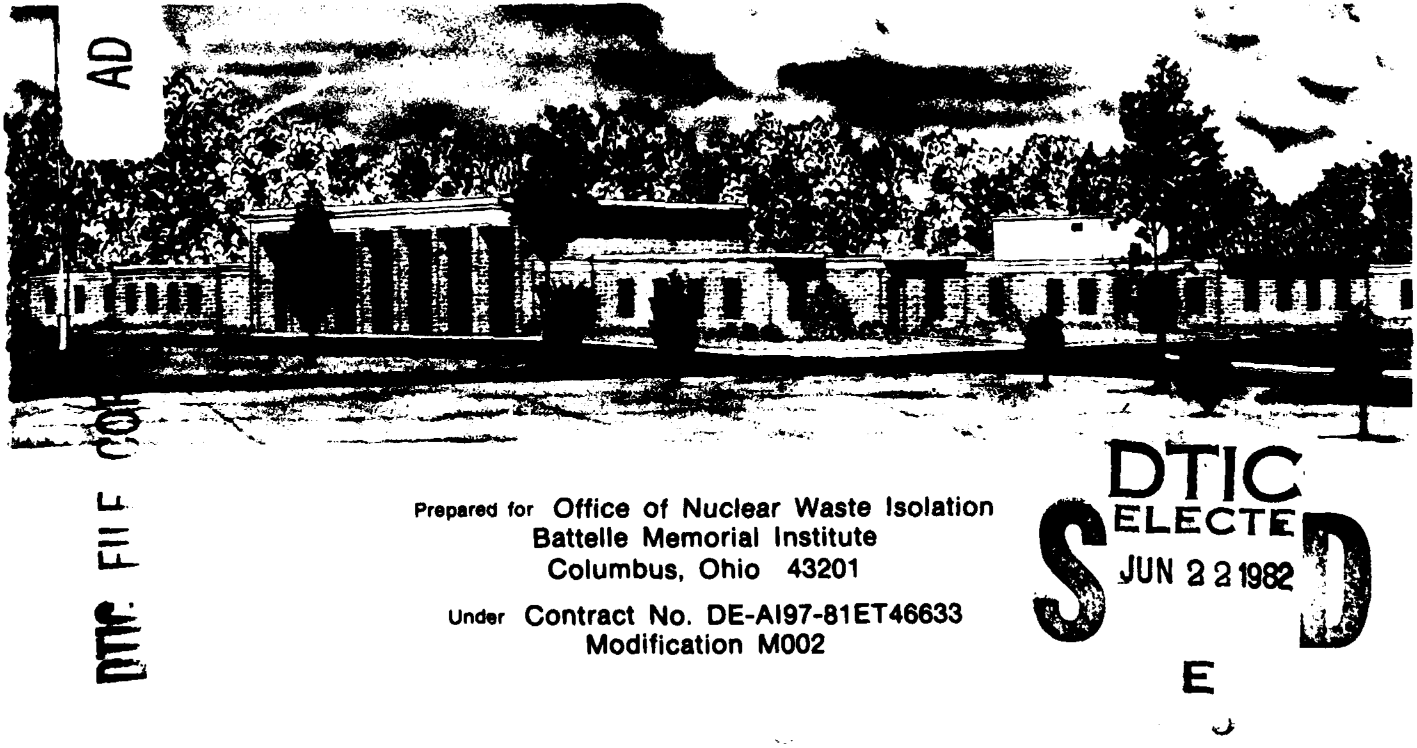
Structures Laboratory
U. S. Army Engineer Waterways Experiment Station
P. O. Box 631, Vicksburg, Miss. 39180

June 1982

Final Report



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Some of the considerations involved in the development of hydraulic cement-based grout mixtures intended for use in the isolation of nuclear waste stored underground are longevity, impermeability, uniformity, ease of placement, and relevance to previously used systems. Work done at the Structures Laboratory of the U. S. Army Engineer Waterways Experiment Station on this subject has indicated that apparently adequate systems can be based on well known ingredients. Information on field testing and long-term testing of these grouts has confirmed this indication. (Continued)

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20. ABSTRACT (Continued).

The following conclusions are indicated:

a) Selection of control parameters such as flow time, time of setting, compressive strength, and permeability for a candidate grout mixture should be criteria used in the formulation of a grout mixture.

b. Once quantitative levels of such parameters have been established, the hydraulic cement-based grout system has adequate latitude in its formulation to accommodate these needs.

c. While longevity in nonaggressive environments and compatibility with host rocks is known in general for such systems, data for specific mixtures under specific conditions must be developed.

A Standard Practice for Selecting Mixture Proportions for Hydraulic Cement-Based Materials Systems for Repository Sealing is presented as an Appendix.

PREFACE

This report was prepared for the U. S. Department of Energy under modification M002 to contract DE-AI97-81ET46633. The subject, "Grout Formulations," comes under FY 82 Task 3, "Properties of Rock and Grout." Mr. Floyd L. Burns, Office of Nuclear Waste Isolation (ONWI), Battelle Memorial Institute, Columbus, Ohio, was Project Manager for this project. Mr. Lynn Myers of ONWI took over as Project Manager on 1 February 1982.

The report was prepared in the Structures Laboratory (SL) of the U. S. Army Engineer Waterways Experiment Station (WES) under the direction of Mrs. Katharine Mather, Project Leader. Mr. Bryant Mather was Chief of the SL; Mr. John M. Scanlon, Jr. was Chief of the Concrete Technology Division (CTD). Messrs. John A. Boa, Jr. and D. M. Walley of the Grouting Unit of the CTD provided technical assistance. Mr. A. D. Buck and Mrs. Mather prepared this report.

Colonel Tilford C. Creel, CE, was Commander and Director of WES, and Mr. F. R. Brown was Technical Director.

CONTENTS

CONVERSION FACTORS, INCH-POUND TO METRIC (SI) UNITS OF MEASUREMENT

Inch-pound units of measurement used in this report can be converted to metric (SI) units as follows:

Multiply	By	To Obtain
Fahrenheit degrees	5/9	Celsius degrees or Kelvins*
inches	25.4	millimetres
pounds (force) per square inch	6.894757	kilopascals
pounds (mass) per cubic foot	16.018463	kilograms per cubic metre
pounds (mass) per gallon (U. S. liquid)	80.51963	kilograms per cubic metre

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* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: $C = (5/9)(F - 32)$. To obtain Kelvin (K) readings, use: $K = (5/9)(F - 32) + 273.15$.

GROUT FORMULATIONS FOR NUCLEAR WASTE ISOLATION

INTRODUCTION

1. The basis for proportioning hydraulic cement-based grouts is closely related to the basis for selecting proportions for structural and mass concrete. Many papers have been written about grouting to reduce the ingress of water to foundations of hydraulic structures and to fill cavities below foundations of such structures. Hydraulic cement-based grouts for sealing boreholes, the use of pumped concrete and grouts in sealing and plugging tunnels, cementing casings in well bores and shafts, and stemming shafts, are aspects of concrete and grouting technology that have developed from four principal roots: (a) foundation grouting, (b) the control of water and part of the control of rock bursts in underground mining, (c) oil well cementing to cut off water and gases and to set casing, and (d) underground tests of nuclear weapons. Requirements for grouts used in connection with underground tests of nuclear weapons have been particularly critical since they have required both complex design considerations and complex highly efficient sealing to prevent the escape of radionuclides. Underground nuclear weapons tests have been games with very high stakes. Underground nuclear waste repositories may be considered also as games, for stakes just as high, that will have the added factor of duration through thousands to hundred thousands and millions of years.

2. Much of the technology developed in underground weapons tests will prove useful in mining and sealing underground nuclear waste repositories, but it is difficult to collect a scattered body of reports that were generally prepared in small editions with limited distribution. The Structures Laboratory (SL) of the U. S. Army Engineer Waterways Experiment Station (WES) has been closely involved in the grouting and concreting technology used in underground weapons testing and has continued to be involved in borehole plugging and in other uses of grout and concrete in connection with experiments related to nuclear waste isolation.

Since our involvement has been with laboratory proportioning and testing, field control of grouting and concreting, and later tests of specimens cured under simulated field conditions, we have an unusually complete view of these operations and the associated logistics. These include operating in remote areas without access roads or with limited access and with materials choices limited to those at the site and mixtures that could be batched, mixed, placed by pumping in tunnels, and later sealed by pressure grouting. All laboratory and field work involving grouts should keep the practical point in mind that the end result is to get a mixture into place that will serve its intended function for the intended service life and all efforts should be geared to this end. There is no substitute for practical experience in this area.

BACKGROUND

3. The use of hydraulic cement-based grout* in boreholes has been practiced for many years, especially in connection with development of oil and gas wells. Concern with similar material systems for use in filling and sealing openings in the earth to isolate nuclear waste that may be stored underground is much more recent. Such interest has intensified since about 1976.

4. The grout formulations reviewed in this report are those developed for use for isolation of nuclear waste.

5. The requirements applicable to such grouts include:

a. The ingredients to be used should be readily available.

b. Freshly mixed grouts should have the required degree of workability and should harden neither too soon nor too late, i.e., have a proper setting time. This means the grout can not be so viscous so it can not be pumped to where it is to be emplaced. Time of setting must be delayed, usually for about 4 hr after mixing, to permit moving the grout to its final destination.

c. Physical properties of the hardened grout should include low permeability to impede the movement of liquid, adequate compressive strength to make it solid, durability to assure long life, and compatibility with the host rock or rocks. Very high levels of strength may be contraindicated since such would lead to brittleness and reduced strain capacity whereas a low to moderate strength would mean a more plastic material of higher creep and greater strain capacity that is less likely to crack due to strain caused by stress. Ideally a compressive strength and hence a creep

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* The term "grout" means a mixture of pourable and pumpable consistency and fluidity. Hydraulic-cement based grouts may or may not include aggregate either fine aggregate only or both fine and coarse aggregate. Thus, grouts may be either (a) neat cement (or cementitious material blend) pastes, (b) mortars, or (c) concretes, of pourable consistencies. Most grouts used in underground applications include little or no coarse aggregate since neither the spaces to be filled nor the pumping equipment used permit use of such material. However, when larger volumes are to be filled, concrete pumping equipment could be used, cost could be reduced, and heat rise with consequent cooling contraction reduced, with benefit, by pumping grout mixtures containing coarse aggregate. Such mixtures have been used in tunnel plugging and other applications.

and strain capacity similar to that of the host rock would seem desirable. Since some ancient structures made of mortars or concretes based on use of hydraulic cements having similarities to present-day hydraulic cement-based systems still exist, it is generally known that durability should be satisfactory if the environment is not aggressive (especially acid) which the environments under consideration would normally not be. Other considerations of durability should be that the choice of materials would avoid the likelihood of an alkali-aggregate reaction or sulfate attack. Compatibility between host rock and hydraulic cement-based grout should not be a problem for most rock materials such as those that are currently being considered, i.e., rock salt, granite, basalt, or welded tuff.

6. Consideration of what would be required of borehole plugging materials has been reported;[1] these were general considerations that included recognition of the need for development of plugging techniques, sealing boreholes with plugs that will prevent movement of fluids and gases toward or through salt beds, and provide plugs that will maintain their integrity for time periods comparable to the life of the rock formations in which they are used. Gulick[1] stated that the only material currently available for plugging boreholes was high-quality cement.

7. A later report[2] provided the following specific data for cement grout plugs:

a. Permeability to water of not greater than 0.1 microdarcy when tested at 3 months.

b. Minimum unconfined compressive strength of 3500 psi when tested using a 2-in. cube at 3 months.

c. Workability and pumpability, a viscosity not greater than 100 poises, after 3 hr.

d. Young's modulus of elasticity not less than 2×10^6 psi.

While not quantitative, it also stated that grout should have physical and chemical compatibility with wall rock.

MATERIALS

8. Laboratory and field experience have indicated that a grout such as those desired for plugging and sealing will consist of hydraulic cement, either portland cement alone or portland cement and slag, with or without a pozzolan such as fly ash to contribute to immediate workability and long time stability, probably with an expansive additive to create enough overall permanent expansion to promote positive contact with the host rock, enough water to make the mixture workable, and possibly one or more chemical admixtures.

9. One of the major advantages of such materials is that they are readily and widely available commercially usually at quite acceptable levels of cost, especially as compared with alternate candidate materials.

RESULTS

10. All grout mixtures for which data are given were grouts for which control parameters were known either implicitly or had been specified before mixtures were made. These typically included those discussed previously.

11. In the period since 1950 the organization now known as the Grouting Unit of the Structures Laboratory of the U. S. Army Engineer Waterways Experiment Station has prepared* and supervised the emplacement of about 10,000 different hydraulic cement-based grout mixtures for many different purposes. Much of this work is unpublished but all of it is available for reference and is used as appropriate.

12. Based on discussions that started in 1975, the WES-SL Grouting Unit made a series of grout mixtures that were intended for consideration for use in borehole plugging associated with nuclear waste isolation. Boa[3] stated: "The major objective of this program was to develop one or more mixtures that can be pumped into boreholes, have a working time of 2 to 3 hr, and are of long-term durability. Also, the mixtures should have permeabilities not over a few microdarcies and should not be subject to attack by local groundwater." He reported that several hundred mixtures were proportioned, 59 of these were studed in some detail, and 5 of these were selected for detailed study. He reported[3] the mixture proportions, flow at different times, initial and final times of setting, and 28-day compressive strength for all 59 mixtures and additional data through 1 year for the 5 selected mixtures. The mixture proportions of these five grouts are shown in Table 1. Some of the physical data for them are shown in Table 2.

13. Gulick[1,4] has provided information on these five mixtures; he included[4] data through 2 years age for them.

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* Appendix A is a "Standard Practice for Selecting Mixture Proportions for Hydraulic-Cement Based Materials Systems for Repository Sealing. This recommended practice describes a procedure that incorporates as much of the art that is used by the Grouting Unit of the WES-SL as can be put down in written form.

14. Rhoderick and Buck[5] reported the results of petrographic examination of specimens from the same five mixtures at ages of about 4 years. They concluded that the phase composition and microstructure were proper for these mixtures and that the variables of starting materials had little overall effect on these properties. These five mixtures included variables of cement type (portland or expansive), use or nonuse of a natural pozzolan, and use or nonuse of salt. In general, their physical properties when freshly mixed and when hardened have been considered satisfactory.[1,3,4]

15. There has been limited use of grout mixtures in the field as part of the overall study of borehole plugging. The first use was when the ERDA-10 hole was plugged with four different grout mixtures in October 1977. Details of this were reported by Gulick.[6] The results of petrographic examination at different ages of surface-cast specimens of these grouts and of grout core retrieved from this hole along with some physical data were reported by Buck, Burkes, and Rhoderick.[7] As with the five laboratory grout mixtures,[5] the authors concluded that phase composition and microstructure were normal for such mixtures. In both cases, as the age of the specimens increased, cracking of them was noted. It was concluded that this was a laboratory phenomenon due to minor changes of temperature and moisture affecting the high cement content specimens and did not signify that such cracking would take place in an underground repository where the temperature and moisture content would be more constant. The constituents and proportions for the four ERDA-10 mixtures are shown in Table 3; compressive strengths are given in Table 4.

16. Another example of field use of a grout for this purpose was the two-stage plugging of a hole during the Bell Canyon Tests in New Mexico in September 1979 and February 1980.[8] In this work, a grout mixture, BCT-1-FF, that had been evaluated in the laboratory, was used. Details of this work and short-term physical data were published.[9] Additional testing of jobsite cast specimens in the laboratory has been underway for about 2 years but has not been reported. The compositions

of the laboratory and field versions of grout BCT-1-FF are given in Table 5; some physical data for the two plugs are shown in Table 6.

17. A short-term study of specimens of the BCT-1-FF grout mixture under different environments (temperature, pressure, fresh water or brine) was reported;[10] it was concluded that hydration was normal for the temperature range investigated (78 to 153° F) and that effects caused by differences in pressure were not detectable by X-ray diffraction. There was no effect of the type of water storage.

18. A modification of the BCT-1-FF grout mixture was studied in detail through 1 year at the Pennsylvania State University and at the SL-WES.[11] Although the intent was to compare methodologies between the two laboratories, the results also showed that the grout had the proper physical properties, phase compositions, and microstructures for the materials used and ages covered.

19. In unpublished work at WES eight modifications of the BCT-1-FF grout mixture were made and tested through 1 year. The modifications incuded the use of three other cements including a shrinkage-compensating expansive cement, the use of two other fly ashes and one silica fume, different amounts of expansive additive, and different water contents. While two of the mixtures had excessive flow times (>20 sec) and one had significantly lower strength, their fresh and hardened properties were generally satisfactory or could be readily modified to be satisfactory. It was further concluded that while the BCT-1-FF grout mixture is as good as it needs to be by present criteria,[1,2] it would be possible to change it to modify certain properties if this became desirable.

DISCUSSION

20. This review of borehole plugging grouts has included approximately 67 laboratory mixtures whose proportions have been reported[3] or are known; 13 of these are being subjected to long-term physical testing and periodic petrographic examination. In addition, there have been two field experiments[6,8] where grouts were emplaced. In both cases, samples of these grouts are being subjected to long-term testing. In the case of ERDA-10,[6] this includes specimens recovered from the hole as core.

21. These various mixtures were made using a range of types of hydraulic cements including portland cements, self-stressing expansive cement, and shrinkage-compensating expansive cements. They include mixtures not containing pozzolan and others with different amounts of different pozzolans (usually fly ash), the use or nonuse of salt, different amounts of expansive additives, different amounts of chemical admixtures, and different amounts of water. Every important mixture component has been varied. In the original screening work[3] the water to cement or water to cementitious solids ratio ranged from 0.4 to higher than 1.0. In the ERDA-10 field work[6] water content was still high and was similar to the range just mentioned. In the Bell Canyon field work[8] and the more recent laboratory work[11] including some not yet published, water contents have been much lower with water to cementitious solids ratios of about 0.3 or slightly lower.

22. Extensive testing has shown that phase composition and microstructure are relatively independent of this range of materials and that each grout tends toward uniformity with the passage of time; in addition, they appear independent of the temperature and pressure extremes that are expected. While physical properties show a wide range, they tend to be as good or better as those tentative values that were listed earlier.

23. It has been repeatedly pointed out or inferred in the various reports that any or all of the properties of such grout mixtures can be modified in either direction if and as desired.

24. Mixture data and some physical data for 10 of the grouts are included in Tables 1 through 6 from published data. All of these are

being subjected to long-term study. These represent the best of those that have been studied, but it is also possible that one or more of those not listed would be equally suitable for borehole plugging in a specific case.

25. In a recent report Lankard and Burns[12] discussed in general terms many of the selection criteria that were mentioned in this report.

CONCLUSIONS

26. It is imperative that control parameters for a grout mixture such as flow time, setting time, compressive strength, permeability, or combinations of these or of other properties be specified before a grout mixture is formulated and used.

27. Once such parameters have been specified, it is possible to proportion a hydraulic cement-based grout mixture to meet the applicable requirements. This may be from experience or by trial mixtures or by both.

28. While longevity of cementitious mixtures in nonaggressive environments is known from history, satisfactory data involving specific mixtures in selected environments are still needed. Such testing is underway.

29. While compatibility, especially long-term compatibility, of cementitious mixtures with various rock types is also known, it is necessary to have such evidence for specific mixtures in contact with specific rocks in a selected environment or environments.

30. Satisfactory grout mixtures may be proportioned using the standard practice set forth in Appendix A.

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Table 1

Mixture Data for Five Grouts Selected for Long-Time Study*

Constituent	Unit	BPN-FA-SP-P	BPN-FA-BS-SP-P-1	BPN-CS-FA-1	BP-521-25MP	BPN-FA-BS-SP-P-1 (Ty III)
Cement, Type III	lb/ft^3					55.21
El Toro Chem Comp	lb/ft^3	61.12	55.21	62.02	43.54	--
ChemStress	lb/ft^3	--	--	9.00	9.00	
Fly Ash	lb/ft^3	20.56	18.58	16.76	12.40	18.58
Tufa (pozzolan)	lb/ft^3	--	--	--	9.84	
Fine Salt (dissolved)	lb/ft^3	--	11.43	--	--	11.43
Melment L10	lb/ft^3	1.63	1.48	1.73	2.10	1.48
Plastiment	oz/ft^3	2.60	2.94	3.02	2.76	2.94
Water	lb/ft^3	34.31	31.73	32.66	36.14	31.73
w/c+pozz	wt	0.42	0.43	0.37	0.48	0.43

* This was Table 2-1 from Reference 4; original data were from Reference 3.

Table 2

Physical Data for Five Grouts Selected for Long-Time Study*

Mixture Designation	Unconfined Compressive Strength* 28-Days Age psi	Time of Setting hr Initial	Time of Setting hr Final	Flow, sec +5 min	Flow, sec +1 hr	Flow, sec +2 hr	Flow, sec +3 hr
BP-521-25MP**	5940	--	--	12.8	13.2	15.0	16.4
BPN-FA-BS-SP-P-1**	5380	28†	31	13.2	21.0	24.2	28.6
BPN-FA-BS-SP-P-1 (Type III)**	5060	~20††	~23	~15.0††	~25.0	~30.0	None
BPN-FA-SP-P**	7570	5-3/4	12-1/2	15.2	20.0	21.0	23.4
BPN-CS-FA-1**	8980	7	11-3/4	18.0	22.0	25.6	None

Note: Flow was determined at 65°-70° F. Test method consisted of time of efflux of 1725-ml grout through a 1/2-in. orifice from a cone-shaped container. Note, particularly, that in some instances the flow actually decreased with time. This is unusual and attributed to the water reducer used. Flow was determined on mixture mixed for 3 min. every 15 min.

Time of setting <u>Final</u> set was that time when specimen had hardened. <u>Initial</u> set was determined to be the time when a 1-mm needle ceased to pass a point 5 mm from the base of a test specimen.

Unconfined compressive strength was determined on 3- by 6-in. cylinders cut from a 3- by 20-in. cylinder and cured, sealed, at ~65° to 70° F. Strengths shown are at 28-days age; however, strengths will also be determined at 1-yr age and the specimens containing pozzolans can be expected to increase in strength, some more drastically than others.

* This is Table 2 in Reference 2.

** Five mixtures selected for further testing.

† These times can be decreased by decreasing retarder amount to obtain more realistic setting time for field use.

†† Estimated.

Table 3

ERDA-10 Grout Mixture Data*

	Units	Plug 1** 30% Salt	Plugs 2, 3** 36% Salt	Plug 4** Fresh Water
Cement, Class C(SR)	lb/ft^3	42.90	39.58	54.83
Fly ash	lb/ft^3	14.47	13.35	18.50
Salt gel (Attapulgite)	lb/ft^3	1.15	1.06	--
Bentonite gel	lb/ft^3	--	--	1.47
Salt, D44	lb/ft^3	10.77	14.15	--
Silica sand, D30	lb/ft^3	3.26	3.01	--
Dispersant, D45	lb/ft^3	0.06	0.05	0.29
Dispersant, D65	lb/ft^3	--	--	--
Calcium chloride (S1)	lb/ft^3	1.15	1.06	--
Water	lb/ft^3	36.6	39.3	36.0
Density	lb/ft^3	108.5	107.0	112.2
Density	lb/gal	14.5	14.3	15.0
Yield	ft^3/sack	1.5	1.7	1.2
Water content	gal/sack	6.6	7.8	5.2
Water/cement ratio		0.85	0.99	0.66
Water/cement and fly ash ratio		0.64	0.74	0.49
Thickening time	hr:min	4:35	7:45	5:05
Unconfined compressive strength				
24 hr	psi	712	420	1210
48 hr		1543	1032	1522
72 hr		1888	1275	2080

---

* This was Table 1 in Reference 6.

** Plug 1 cured at 128° F, 2445 psi; Plugs 2, 3 cured at 125° F, 2112 psi; Plug 4 cured at 80° F, 445 psi.

Table 4

Average Static Unconfined Compressive Strength (psi) for ERDA-10 Specimens*

	Age at Test				
	16 days	27 to 29 days	230 days	27 mo	39 mo
Plug 1					
Surface Cast	1770	1795	--	3910	4,400
No. of Tests	1	2		4	2
Cores	700	1735	2579	3270	--
No. of Tests	1	2	6	4	
Plug 2					
Surface Cast	--	2230	--	3870	4,070
No. of Tests		2		2	2
Plug 3					
Surface Cast	--	2220	--	3820	3,320
No. of Tests		2		4	2
Recirculated Return	--	2056	--	--	--
No. of Tests		8			
Plug 4					
Surface Cast	--	6333	--	6060	10,210
No. of Tests		2		4	2

---

* The data through 230 days were Table 3-5 in Reference 4. The 27- and 39-month data were from Reference 7.

Table 5

Data for Laboratory and Field BCT-1-FF Grout Mixtures*

Proportions (Wt %)	Lab	Field 9/26/79	Field 2/14/80
Class H Cement	52.2	53.1	52.7
Fly Ash	17.6	18.1	18.2
Expansive Additive	7.0	7.1	7.5
Dispersant (D65)	0.2	0.1	0.2
Defoamer (D47)	0.02	0.02	0.02
Water	23.0	21.6	21.4
Properties			
Water/Cementitious Ratio	0.30	0.28	0.27
Fluid Density, g/cm^3	1.98	2.11	2.11
Fluid Density, lb/gal	16.5	17.6	17.6

* This was Table 5 in Reference 8.

Table 6

Physical Properties of BCT-1-FF Grout Field Mixtures*

Unconfined Compressive Strength, MPa (psi)

Age/Days	9/26/79 Plug	2/14/80 Plug
1	--	33 (4,840)
3	--	58 (8,410)
7	--	73 (10,610)
14	75 (10,870)	74 (10,670)
21	--	78 (11,350)
28	--	79 (11,445)
56	93 (13,440)	
90	88 (12,750)	

Compressional Wave Velocity, m/s (ft/sec)

Age/Days	9/26/79 Plug	2/14/80 Plug
19	3,945 (12,945)	--
28	--	4,197 (13,770)
62	4,005 (13,140)	
99	4,149 (13,612)	

Dynamic Modulus, E 10^3 MPa (E x 10^6 psi)

Age/Days	9/26/79 Plug	2/14/80 Plug
19	16.3 (2.36)	
28	--	26.3 (3.81)
62	16.5 (2.40)	
99	16.8 (2.44)	

Density and Porosity at 105 Days-Age for 9/26/79 Plug

	Dry Density (g/cm^3)	Grain Density (g/cm^3)	Porosity %
3 x 13 Cylinder, Top	1.878	2.715	30.83
3 x 13 Cylinder, Bottom	1.724	2.668	35.38
Grout in Permeability Specimen:			
T1	1.908	2.709	29.57
T2	1.911	2.860	33.18
B2	1.910	2.803	31.86
B1	1.900	2.773	31.48

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* This was Table 6 in Reference 8.

APPENDIX A: STANDARD PRACTICE FOR SELECTING MIXTURE PROPORTIONS FOR HYDRAULIC CEMENT-BASED MATERIALS SYSTEMS FOR REPOSITORY SEALING*

1. The materials systems, based on hydraulic cement as the binder, that will be used in repostiory sealing will necessarily be multicomponent and will be of infinitely variable proportions in order to permit use to the greatest extent possible of available low-cost ingredients and to optimize attainment of desirable levels of relevant properties of both the freshly mixed and hardened systems.

2. The process of materials selection for such materials systems and the applicable specification and acceptance testing systems is the subject of another manual in this series.

3. It is assumed that selection of the classes of materials and a selection among the relevant optinional specification requirements applicable to each class has been accomplished, that arrangements for procurement, inspection, testing, acceptance, and storage have been made.

4. This manual addresses the selection of proportions of the various materials for use in a repository sealing materials system. It will be assumed that the ingredients of the system are:

a. Portland cement or blended portland cement.

b. Pozzolan or ground glassy slag.

c. Mixing water or aqueous mixing solution (e.g. saturated brine when NaCl is used as aggregate).

d. Aggregate (fine and coarse or fine only; in some grouts no aggregate will be used).

e. Water-reducing chemical admixture.

f. Set-controlling admixture (usually a retarder; rarely an accelerator).

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* This appendix is based on and modified from the "ACI Standard Recommended Practice for Selecting Proportions for Normal and Heavyweight Concrete (ACI 211.1-77)" adopted in September 1977; also adopted by the Corps of Engineers as CRD-C 99-78 in the "Handbook for Concrete and Cement" published by the U. S. Army Engineer Waterways Experiment Station.

g. Air-entraining admixture.*

h. Expansion-producing admixture. May not be required if expansive cement is supplied under a.

Thus, we must deal typically with proportions of eight classes of materials.

5. The selection of mixture proportions involves a balance between reasonable economy and requirements for placeability, strength, durability, density, and impermeability. The required characteristics are governed by the use to which the sealing material will be put and by circumstances applicable to the conditions expected to be encountered at the time of placement. These are often, but not always, reflected in specifications for the job.

6. The ability to tailor mixture properties to job needs reflects technological developments which have taken place, for the most part, since the early 1900s. The use of the water-cement ratio as a tool for estimating strength was recognized about 1918. These two significant developments in the technology of the use of hydraulic-cement based materials have been augmented by extensive research and development in many related areas, including the use of admixtures to counteract possible deficiencies, develop special properties, or achieve economy. It is beyond the scope of this discussion to review the theories of proportioning which have provided the background and sound technical basis for the relatively simple methods of this manual.

7. Proportions calculated by any method must always be considered subject to revision on the basis of experience with trial batches.

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* Air-entraining admixtures are often not used in mixtures for repository sealing systems since the conditions that mandate the use of air-entrainment in many concrete mixtures typically do not apply to repository sealing; namely the need to resist freezing and thawing while critically saturated. However, while not mandated for this reason, many grout mixtures for subsurface use have, with benefit, been air-entrained. Among the benefits so obtained are: (1) Prevention or reduction of "bleeding" i.e. segregation by settlement with collection of water at the surface, (2) Increased workability, fluidity, pumpability due to the lubricating effect of the air bubbles, and (3) Reduced mass per unit volume. Thousands of cubic metres of "foam concrete" (= air-entrained grout of very high air content) have been used as thermal and sound insulation and as shock isolation backpacking around underground tunnels.

Depending on circumstances, the trial batches may be prepared in a laboratory or, perhaps preferably, as full-size field batches. The latter procedure, when feasible, avoids possible pitfalls of assuming that data from small batches mixed in a laboratory environment will predict performance under field conditions. Trial batch procedures and background testing are described in referenced documents.

8. Mixture proportions must be selected to provide necessary placeability, working time, strength, durability, and density for the particular application. Well established relationships governing these properties are discussed briefly below.

9. Placeability (including satisfactory pumping properties) encompasses traits loosely accumulated in the terms "workability" and "consistency." For the purpose of this discussion, workability is considered to be that property which determines capacity to be placed and self-levelling without harmful segregation. It embodies such concepts as moldability, cohesiveness, and compactability. It is affected by the grading, particle shape and proportions of aggregate, the amount of cement, the presence of entrained air, admixtures, and the consistency of the mixture. Procedures in this manual permit these factors to be taken into account to achieve satisfactory placeability economically.

10. Consistency, loosely defined, is the wetness of the grout or concrete mixture. It is measured in terms of flow or slump - the shorter the flow time or the higher the slump, the wetter the mixture - and it affects the ease with which the mixture will flow during placement. It is related to but not synonymous with workability. In a properly proportioned mixture, the unit water content required to produce a given flow or slump will depend on several factors. Water requirement increases as aggregates become more angular and rough textured (but this disadvantage may be offset by improvements in other characteristics such as bond to cement paste). Required mixing water decreases as the maximum size of well graded aggregate is increased. It also decreases with the entrainment of air. Mixing water requirement will often be significantly reduced by high-range water reducers.

11. Strength. Strength is an important characteristic of hydraulic-cement based mixtures, but other characteristics such as low permeability and resistance to chemical alteration are often equally or more important. These may be related to strength in a general way but are also affected by factors not significantly associated with strength. For a given set of materials and conditions, strength is determined by the net quantity of water used per unit quantity of hydraulic-cement based binder. The net water content excludes water absorbed by the aggregates. Differences in strength for a given water-cement ratio may result from changes in: maximum size of aggregate; grading, surface texture, shape, strength, and modulus of elasticity of aggregate particles; differences in cement types and sources; air content; and the use of admixtures which affect the cement hydration process or develop cementitious properties themselves. To the extent that these effects are predictable in the general sense, they are taken into account in this manual. However, in view of their number and complexity, it should be obvious that accurate predictions of strength must be based on trial batches or experience with the materials to be used.

12. Durability. Any cement-based mixture after hardening must be able to endure those exposures which may deprive it of its serviceability - wetting and drying, heating and cooling, chemicals, and the like. Resistance to some of these may be enhanced by use of special ingredients: low-alkali cement, pozzolans, or selected aggregate to prevent harmful expansion due to the alkali-aggregate reaction which occurs in some areas when concrete is exposed in a moist environment: sulfate resisting cement or pozzolans for systems exposed to sulfates in solution. Use of a low water-cement ratio will prolong the life of the system by reducing the penetration of aggressive liquids.

13. Density. For certain applications materials may be used primarily for its mass characteristic. Examples of applications are counterweights on lift bridges, weights for sinking oil pipelines under water, shielding from radiation, and for insulation from sound. By using special aggregates, placeable mixtures of densities as high as 350 lb per cu ft can be obtained.

14. To the extent possible, selection of mixture proportions should be based on test data or experience with the materials actually to be used. Where such background is limited or not available, estimates given in this manual may be employed.

15. The following information for available materials will be useful:

a. Sieve analyses of fine and coarse aggregates.

b. Unit weight of coarse aggregate.

c. Bulk specific gravities and absorptions of aggregates.

d. Mixing water requirements of mixtures developed from experience with available aggregates.

e. Relationships between strength and water-cement ratio for available combinations of cement and aggregate.

16. The procedure for selection of mixture proportions given in this section is applicable to mixtures containing normal weight aggregate. Although the same basic data and procedures can be used in proportioning mixtures containing heavyweight aggregate, additional information will be needed.

17. Estimating the required batch weights for the mixture involves a sequence of logical, straightforward steps which, in effect, fit the characteristics of the available materials into a mixture suitable for the work. The question of suitability is frequently not left to the individual selecting the proportions. The job specifications may dictate some or all of the following:

a. Maximum water-cement ratio.

b. Minimum cement content.

c. Air content.

d. Slump.

e. Maximum size of aggregate.

f. Strength.

g. Requirements relating to such things as strength overdesign, admixtures, and special types of cement or aggregate.

18. Regardless of whether the mixture characteristics are prescribed by the specifications or are left to the individual selecting

the proportions, establishment of batch masses ("weights") per cubic metre of the mixture can best be accomplished in the following sequence:

19. Step 1. Choice of slump or flow. If slump or flow is not specified, a value appropriate for the work should be selected.

20. Step 2. Choice of maximum size of aggregate. Large maximum sizes of well graded aggregates have lower void content than smaller sizes. Hence, mixtures with the larger-sized aggregates require less mortar per unit volume. Generally, the maximum size of aggregate should be the largest that is economically available and consistent with dimensions of the space to be filled. In no event should the maximum size exceed one-fifth of the narrowest dimension of the space to be filled. These limitations are sometimes waived if workability and methods of consolidation are such that the mixture can be placed without honeycomb or void. When high strength is desired, best results may be obtained with reduced maximum sizes of aggregate since these produce higher strengths at a given water-cement ratio.

21. Step 3. Estimation of mixing water and air content. The quantity of water per unit volume of concrete or grout required to produce a given slump or flow is dependent on the maximum size, particle shape and grading of the aggregates, and on the amount of entrained air. It is not greatly affected by the quantity of cement. Depending on aggregate texture and shape, mixing water requirements may vary somewhat. Such differences in water demand are not necessarily reflected in strength since other compensating factors may be involved. For example, a rounded and an angular coarse aggregate, both well and similarly graded and of good quality, can be expected to produce concrete of about the same compressive strength for the same cement factor in spite of differences in water-cement ratio resulting from the different mixing water requirements. Particle shape per se is not an indicator that an aggregate will be either above or below average in its strength-producing capacity.

22. The use of normal amounts of air entrainment in mixtures with a specified strength near or about 5000 psi may not be possible due to the fact that each added percent of air lowers the maximum strength obtainable with a given combination of materials.

23. When trial batches are used to establish strength relationships or verify strength-producing capability of a mixture, the least favorable combination of mixing water and air content should be used. This is, the air content should be the maximum permitted or likely to occur, and the concrete should be gaged to the highest permissible slump. This will avoid developing an over-optimistic estimate of strength on the assumption that average rather than extreme conditions will prevail in the field.

24. Step 4. Selection of water-cement ratio. The required water-cement ratio is determined not only by strength requirements but also by factors such as durability and pumping properties. Since different aggregates and cements generally produce different strengths at the same water-cement ratio, it is highly desirable to have or develop the relationship between strength and water-cement ratio for the materials actually to be used. The average strength selected must, of course, exceed the specified strength by a sufficient margin to keep the number of low tests within specified limits.

25. Step 5. Calculation of cement content. The amount of cement per unit volume of mixture is fixed by the determinations made in Steps 3 and 4 above. The required cement is equal to the estimated mixing water content (Step 3) divided by the water-cement ratio (Step 4). If, however, the specification includes a separate minimum limit on cement in addition to requirements for strength and durability, the mixture must be based on whichever criterion leads to the larger amount of cement.

26. The use of pozzolanic or chemical admixtures will affect properties of both the fresh and hardened concrete.

27. Step 6. Estimation of coarse aggregate content. Aggregates of essentially the same maximum size and grading will produce mixtures of satisfactory workability when a given volume of coarse aggregate, on a dry-rodded basis, is used per unit volume of concrete. For equal workability, the volume of coarse aggregate in a unit volume of mixture is dependent only on its maximum size and the fineness modulus of the fine aggregate. Differences in the amount of mortar required for workability

with different aggregates, due to differences in particle shape and grading, are compensated for automatically by differences in dry-rodded void content.

28. The volume of aggregate, in cubic feet, on a dry-rodded basis, for a cubic yard of mixture is equal to the volume fraction of coarse aggregate in the mixture multiplied by 27. This volume is converted to dry weight of coarse aggregate required in a cubic yard of concrete by multiplying it by the dry-rodded weight per cubic foot of the coarse aggregate.

29. For more workable mixtures, especially when placement is by pump, it may be desirable to reduce the coarse aggregate content by about 10 percent. However, caution must be exercised to assure that the resulting slump, water-cement ratio, and strength properties of the concrete are consistent with the recommendations made previously and meet applicable project specification requirements.

30. Step 7. Estimation of fine aggregate content. At completion of Step 6, all ingredients of the concrete have been estimated except the admixtures and fine aggregate. The fine aggregate quantity is determined by difference. Either of two procedures may be employed: the "weight" method or the "absolute volume" method.

31. Weight method. If the weight of the mixture per unit volume is assumed or can be estimated from experience, the required weight of fine aggregate is simply the difference between the weight of fresh mixture and the total weight of the other ingredients. Often the unit weight of the mixture is known with reasonable accuracy from previous experience with the materials. Even if the estimate of concrete weight per cubic yard is rough, mixture proportions will be sufficiently accurate to permit easy adjustment on the basis of trial batches as will be shown in the examples.

32. If a theoretically exact calculation of fresh weight per cubic yard is desired, the following formula can be used:

$$U = 16.85G_a(100 - A) + C(1 - G_a/G_c) - W(G_a - 1)$$

where

U = weight of fresh mixture per cubic yard, lb

G_a = weighted average specific gravity of combined fine and coarse aggregate, bulk SSD

G_c = specific gravity of cement (generally 3.15)

A = air content, percent

W = mixing water requirement, lb per cu yd

C = cement requirement, lb per cu yd

33. Absolute volume method. A more exact procedure for calculating the required amount of fine aggregate involves the use of volumes displaced by the ingredients. In this case, the total volume displaced by the known ingredients - water, air, cement, and coarse aggregate - is subtracted from the unit volume of mixture to obtain the required volume of fine aggregate. The volume occupied in the mixture by any ingredient is equal to its weight divided by the density of that material (the latter being the product of the unit weight of water and the specific gravity of the material).

34. Step 8. Adjustments for aggregate moisture. The aggregate quantities actually to be weighed out for the mixture must allow for moisture in the aggregates. Generally, the aggregates will be moist and their dry weights should be increased by the percentage of water they contain, both absorbed and surface. The mixing water added to the batch must be reduced by an amount equal to the free moisture contributed by the aggregate - i.e., total moisture minus absorption.

35. Step 9. Trial batch adjustments. The calculated mixture proportions should be checked by means of trial batches prepared and tested in accordance with applicable standardized testing methods or full-sized field batches. Only sufficient water should be used to produce the required slump or flow regardless of the amount assumed in selecting the trial proportions. The mixture should be checked for unit weight and yield (ASTM C 138) and for air content (ASTM C 138, C173, or C 231). It should also be carefully observed for proper workability, freedom from segregation, and finishing properties. Appropriate adjustments should be made in the proportions for subsequent batches in accordance with the following procedure.

36. Re-estimate the required mixing water per cubic yard by multiplying the net mixing water content of the trial batch by 27 and dividing the product by the yield of the trial batch in cubic feet. If the slump of the trial batch was not correct, increase or decrease the re-estimated amount of water by 10 lb for each required increase or decrease of 1 in. in slump.

37. If the desired air content was not achieved, re-estimate the air-entraining admixtures content required for proper air content and reduce or increase the mixing water content by 5 lb for each 1 percent by which the air content is to be increased or decreased from that of the previous trial batch.

38. If estimated weight per cubic yard of the fresh mixture is the basis for proportioning, re-estimate that weight by multiplying the unit weight in pounds per cubic foot of the trial batch by 27 and reducing or increasing the result by the anticipated percentage increase or decrease in air content of the adjusted batch from the first trial batch.

39. Calculate new batch weights starting with Step 4, modifying the volume of coarse aggregate if necessary to provide proper workability.

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