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DEVELOPMENT OF A SLURRY FILLING SYSTEM FOR MODIFIED IN SITU OIL SHALE MINING

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Rio Blanco Oil Shale Co.
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16. Abstract (Limit: 200 words) The modified in-situ process is one of the methods being considered for extracting oil from oil shale. One of the questions to be answered for this approach is the environmental consequence of underground retort abandonment. This report outlines a study which addresses the question in terms of a grouting solution. It covers the development of a spent shale ash based grout which will fulfill the environmental requirements in terms of strength, stability, and impermeability. In addition, it will allow the reinjection of up to 50 percent of material mined and surface retorted as part of this process.			
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FOREWORD

This report was prepared by Rio Blanco Oil Shale Company, Aurora, Colorado, under USBM Contract Number J0295046. The contract was initiated under the Minerals Environmental Program. It was administered under the technical direction of Spokane Research Center with David Nicholson acting as Technical Project Officer. Darlene Wilson was the contract administrator for the Bureau of Mines. This report is a summary of the work recently completed as a part of this contract during the period 9-28-79 to 6-30-82. This report was submitted by authors on 6-4-82.

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DEVELOPMENT OF A SLURRY FILLING SYSTEM FOR MODIFIED
IN-SITU OIL SHALE MINING

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SUMMARY

Rio Blanco Oil Shale Company (RBOSC) has developed two experimental small-scale retorts at Tract C-a in Rio Blanco County, Colorado. These retorts are intended to test the feasibility of the Modified In-Situ (MIS) method for commercial shale-oil production. This method employs underground chambers whose function is identical to that of conventional surface retorts. A fraction of each underground chamber is excavated leaving a pre-designed void. The remaining shale in the chamber is then rubblized and retorted in place.

The MIS method has some favorable technical and environmental features, principally that a reduced volume of shale would be extracted from the mine for surface retorting and subsequent disposal. However, a commercial-sized MIS operation would still produce a substantial volume of shale.

A commercial MIS operation will therefore be a combination of both surface retorting and underground retorting to maximize resource recovery. The former will generate large quantities of spent shale ash that will require disposal. The latter will result in underground chambers (abandoned MIS retorts) containing spent shale rubble of low strength and high porosity and permeability. The potential may be present for underground mine failure followed by differential surface subsidence, contamination of the ground water regime, or accumulation of hazardous gas in the abandoned retorts and its leakage into the mine.

A possible solution to minimize surface disposal of the ash and stabilize abandoned MIS retorts is grouting the retorts with a slurry containing ash as its major component. Both surface and underground disturbance would thereby be minimized.

To investigate that possible solution, RBOSC designed a four-phase research program to develop and ultimately field-test a grout slurry system for filling abandoned MIS retorts:

Phase I: Laboratory research, grout development and grouting tests on small-scale models of abandoned MIS retorts.

Phase II: Design and estimate the cost of a pilot grouting plant to grout a small-scale MIS retort at Tract C-a.

Phase III: Construct the pilot grouting plant designed in Phase II and grout a small-scale MIS retort at Tract C-a.

Phase IV: Evaluate the effectiveness of the retort grouting in Phase III and install a system for monitoring the long-term behavior of the grouted retort.

Phase I and Phase II research was conducted for the Bureau of Mines, U.S. Department of Interior, under Contract J0295046 (awarded to RBOSC on September 28, 1979) and is described in this report. The objectives of Phase II are self-explanatory. The objectives of Phase I were to:

1. Develop two satisfactory grouts containing the maximum amount of surface retorted ash possible in order to minimize disposal of spent shale on the surface.
2. Inject the grouts developed into small-scale models of abandoned MIS retorts to confirm that their use in a grout slurry system is operationally feasible.
3. Bind spent shale rubble into a coherent mass sufficiently strong to reduce potential underground mine failure and differential surface subsidence.
4. Render spent shale rubble virtually impermeable to minimize potential contamination of the ground water regime.
5. Decrease the porosity of spent shale rubble by sealing or filling its voids to reduce potential accumulation sites of hazardous gas or its leakage into the mine.

Results of Phase I research indicate that the objectives described above can be achieved.

Grout Development

Two satisfactory grouts were developed, each containing 850 grams ash/liter of grout (equivalent to 1,433 lb/yd³). A third satisfactory grout was also developed (for the trench test described below) that contained 907 g/l of ash (1,529 lb/yd³). At these concentrations, an estimated 40 to 50% of all ash generated by surface retorting can be injected into abandoned MIS retorts and thereby eliminated from surface disposal.

Grout Injection

Three grouts were successfully injected into small-scale models of abandoned MIS retorts. The first two grouts were used at Colorado School of Mines Research Institute (CSMRI) in two steel vessels, each containing about 4.7 m³ (6.1 yd³) of spent shale rubble. The third grout was used at Tract C-a in a trench test that contained about 62 m³ (81 yd³) of shale rubble (unretorted because no spent shale was available). The trench rubble was derived from experimental MIS retort development at Tract C-a and its size distribution was therefore representative of that in an actual MIS retort.

The primary objective of the trench test was to determine the horizontal distance of grout penetration into shale rubble from a grout injection hole. That was measured at 6.1 to 6.4 m (20 to 21 ft). However, this distance is considered minimal because operational constraints for the test limited grout injection pressure to about 1.4×10^5 Pa (15 to 20 psi).

UngROUTED Versus Grouted Rubble Properties

Spent shale rubble consists of a heterogeneous mass of individual shale fragments with unconfined compressive strengths ranging from 48 to 68×10^5 Pa (700 to 985 psi), parallel and perpendicular to bedding, respectively. The strength of individual fragments is highly dependent upon the degree of retorting with well retorted fragments exhibiting strengths as low as 16×10^5 Pa (230 psi). Some highly retorted fragments are very fragile, easily crumbled by hand and will probably crush under their own weight.

Under confined conditions, successful grouting converts the weak rubble into a competent engineering material capable of sustaining heavy loads without significant deformation or failure. Well grouted rubble has an undrained shear strength of 12×10^5 Pa (167 psi; 12 t/ft²), an initial tangent modulus of $4,788 \times 10^5$ Pa (69,445 psi; 5,000 t/ft²) and a secant modulus at 50% of peak stress of $1,149 \times 10^5$ Pa (16,667 psi; 1,200 t/ft²).

Permeability of spent shale rubble ranges from 0.1 to 60 cm/sec (0.1 to 62×10^6 ft/yr), similar to those of sandy to clean gravel deposits. Grouting substantially reduces these high permeability values to a range of 0.7 to 60×10^{-6} cm/sec (0.7 to 62 ft/yr) similar to that of silty clay.

Even though the rubble in the trench test was incompletely grouted (insufficient volume of grout available), rubble permeability was reduced by a factor of well over 1,000, strongly suggesting that even partial grouting of rubble is highly effective.

Porosity of spent shale rubble is significantly reduced by grouting. In one vessel at CSMRI, rubble porosity of 45% was reduced by a factor of 22.5 to 2% with the filling of 96% of the rubble voids. In the second vessel, rubble porosity of 40% was reduced by a factor of 2.7 to 15% with the filling of 62% of the voids. Reduction of rubble porosity in the trench test is unknown because part of the volume of injected grout leaked between the plastic liners of the trench. Full porosity reduction of the trench rubble was not possible anyway because of an insufficient supply of grout for complete grouting of the rubble.

The trench test indicated that initially injected grout first travels along the bottom and sides of the rubble mass adjacent to the rubble-liner interface. This phenomenon corresponds to one that is commonly observed in conventional alluvium grouting where grout initially migrates along a change in soil conditions (a gravel-clay boundary, for example) before seeking the interior of the alluvium mass. This implies that even partial grouting of an abandoned MIS retort may be effective in reducing both porosity and permeability of its contained mass by the development of a grouted rubble "skin" at the retort's periphery.

Additional Comments

A commercial MIS operation will produce large volumes of process waste water from both underground and surface retorting. Possible use of this poor

quality water in grout preparation might immobilize large volumes that otherwise could require extensive treatment prior to discharge or other use.

Lastly, the grouts developed in this research program were specifically designed to stabilize abandoned MIS retorts in an oil-shale mine. Rheological and mechanical characteristics of the grouts were established toward that goal. By varying additives and concentration, setting times can be decreased and strength increased. Ash-based grouts could therefore be adopted as hydraulic backfills in other mine oil shale mining methods.

1. INTRODUCTION

The Modified In-Situ method is considered by Rio Blanco Oil Shale Company as one of the alternatives for commercial shale-oil production at Tract C-a in Rio Blanco County, Colorado. In conjunction with this method, a considerable amount of shale will be mined in order to develop the underground retorts. This shale will be retorted at the surface to maximize resource recovery and large quantities of spent shale ash (SSA) will be generated. One possible way to dispose of the spent ash would be to inject it in the form of a grout into abandoned MIS retorts. This will prevent significant water flow through the retorts and also increase their stability.

1.1 Modified In-Situ Retorting

The RBOSC MIS method consists of removing about 20 to 40 percent of the oil shale from an underground retort by conventional mining methods and taking it to the surface for above-ground retorting. The remaining shale in the underground retort is rubblized. This rubblized material is then retorted as follows: the retort is ignited by lowering a burner into the retort from the surface; as the retort burns downward, the fluids flow to the base of the retort where they are collected and pumped to the surface.

One of the alternatives being considered for surface retorting is the Lurgi-Ruhrgas¹ (L-R) method. Fresh oil shale is fed into a screw mixer in which heat is supplied by hot spent ash. The kerogen in the fresh shale converts to gas and oil vapors, and these gases flow through a product gas cyclone to the oil recovery system. The cyclone removes some of the dust carried with the gas stream, and the remaining dust is removed in a heavy-oil dedusting process. All dust is combined and carried to the lift pipe where air picks it up along with the recirculating stream and the freshly retorted shale from the surge bin. This combined material is burned in the lift pipe and separator bin to provide the heat for retorting. The fine ash is blown out of the separator bin into the flue gas cyclone and the waste heat recovery system. The coarse ash drops into the screw mixer, where it provides heat for the retort, and the process continues.

About half of the fine ash is recovered in the flue gas cyclone and half by an electrostatic precipitator located in the waste heat recovery system. There are, therefore, two types of ash from the L-R process that will require disposal: one from the cyclone and the other from the electrostatic precipitator. Substantial quantities of both ash types will be generated by surface retorting in a commercial plant.

¹Reference to specific brands, equipment or trade names in this report is to facilitate understanding and does not imply endorsement by the Bureau of Mines.

1.2 Statement of the Problem and Research Objectives

After burning is complete, some cavities may exist at the top of an MIS retort. Backfilling such cavities is analagous to conventional backfilling of abandoned mines or the grouting of caverns in karstic formations. The spent shale in the retorts is expected to range from lumps to very fine powder, and its overall porosity will be in the range of 0.2 to 0.4 according to the amount of oil shale removed from the retort to obtain the desired void volume.

A previous laboratory study performed by Soletanche showed that the preparation of grouts from spent ash and water is feasible and that the grouts' rheological characteristics would be close to a classic bentonite-cement grout. Therefore, this work was considered similar to a basic alluvium grouting with a bentonite-cement grout; and the methodology to grout the MIS retorts was based on the technologies to grout alluvial soil.

However, some specific characteristics of the MIS process must be considered:

- The retort depth (several hundred feet of overburden).
- The hypothetical conditions of MIS retorts at this stage, such as the granulometric gradation of the spent rubble and the retort temperature. The latter was assumed close to 77°C (170°F) for this work.
- The chemical and physical characteristics of the spent ash.
- The magnitude of a commercial grouting operation for MIS retorts, which is many times larger than what is currently done for soil grouting.

The result of underground retorting will be chambers filled with spent shale rubble. In addition, processing mined material on the surface will produce large quantities of spent ash. If the MIS retorts are abandoned without treatment, there is the possibility of mine failure followed by surface subsidence, of ground water contamination, and the upper and lower aquifer water mixing through the MIS retorts. Furthermore, abandoned retorts could collect gases that might be hazardous.

This research program developed and tested grouts that would be injected into abandoned MIS retorts. These grouts were intended to seal the retorts adequately to prevent the leaching of contaminants from the in-situ spent shale; stabilize the retorts sufficiently to reduce the possibility of significant mine failure and surface settlement; and use spent ash from surface retorting as a base component in order to reduce waste disposal as much as possible.

The research program consisted of:

1. Grout development.
2. Grouting of laboratory scale models to measure the hydraulic and mechanical characteristics of a grouted rubble mass.

3. Grouting of a test trench filled with rubble to measure the radius of penetration of the grout.
4. Design of a pilot grouting plant and estimation of its cost.
5. Proposals for grouting a small-scale MIS retort and monitoring its performance after sealing.

Chapter 2 describes the extensive laboratory work done to develop hydraulic grouts from the spent shale ash. Chapters 3 and 4 report the results of the large scale testing of the grout in two vessels and field trench experiments. These sections are followed by a concluding chapter which discusses the results of the work. Appendices are included showing testing procedures and standards and detailed testing reports performed during the research work.

2. GROUT DEVELOPMENT

2.1 Goals

The laboratory testing program had two major goals. The first was to confirm the feasibility of grouts based on ash mixed with water. If feasibility was proven, the second goal was to select the two grouts most suitable for grouting rubble in the steel vessels and the trench.

As mentioned previously, earlier research work performed by Soletanche for RBOSC proved that spent ash could be used as the major component of grouts having mechanical and rheological characteristics similar to those of bentonite-cement grouts. In this program, target values were established for these characteristics. Tests were performed to select the two most promising grouts with characteristics as close as possible to the target values. The variable parameters in the program were the ash content and the use of different types and concentrations of additives.

2.2 Target Characteristics for the Grouts

Target characteristics were selected according to the goals to be achieved as well as by analogy to the characteristics of bentonite-cement grouts used for alluvial grouting. The results of the previous Soletanche research program on ash grout were also considered for this selection. Table 1 summarizes the grout characteristics chosen for this program.

2.2.1 Rheological Characteristics

The rheological characteristics are among the main parameters that control how effectively and extensively bentonite-cement grouts will penetrate an alluvium formation and, by analogy, spent shale rubble in an abandoned MIS retort. Other important criteria are the spacing of the grout holes, the

grouting pressure and the rate of pumping. The desired rheological values are:

- o Viscosity within the range of 45 to 55 Marsh seconds, to limit head losses during injection and to achieve a high radius of injection. However, the viscosity must not be too low and preclude a stable slurry.
- o A setting time of about two hours at 170°F, to obtain the maximum radius of penetration during injection. The grout must remain liquid as long as possible; however, a two hour set-time was difficult to achieve because of the high temperature.
- o A bleeding value* of 5% or less, to maximize the stability of the grout.

2.2.2 Mechanical Characteristics

In conjunction with the target rheological characteristics, the desired mechanical characteristics were:

- o Strength: highest strength possible, to limit underground mine failure and surface subsidence. The minimum target value of the unconfined compressive strength was set at 0.7 MPa (100 psi).
- o Permeability: permeability as low as possible, to limit the quantity of water flowing through the spent rubble. The target value selected was 1×10^{-6} cm/s.

2.2.3 Ash Content

The ash content of the grout was to be as high as possible to reduce surface disposal of ash generated from surface retorting of oil shale mined underground during the development of MIS retorts.

Table 1. Target Characteristics of the Grouts

Characteristic	Target
Ash content or ash/water ratio	as high as possible
Viscosity	45 to 55 Marsh seconds
Setting Time	2 hours at 77°C (170°F) (approximately)
Bleeding	5%
Unconfined Compressive Strength	7×10^5 Pa (100 psi)
Permeability	1×10^{-6} cm/s

*See Appendix A, page 112 for definition.

2.3 Outline of the Laboratory Program for Grout Development

The grout study was carried out in several steps from the delivery of the ash to the selection of the grout composition. These steps are described below.

The ash originated at the Lurgi Pilot Plant in Essen, West Germany, where a number of small-scale processing runs were made varying conditions such as retorting temperature and shale residence time. No individual run was of sufficient magnitude to supply the large quantities of ash required for the subject research program. It was therefore necessary to combine bulk ash samples from several runs. These samples were selected from runs considered most representative of a commercial-size Lurgi plant.

Previous research indicated that the cementitious properties of ash vary considerably according to the ash's retorting history. Bulk ash samples from the selected Lurgi runs not only had to be combined but thoroughly blended to assure a uniform ash material for subsequent steps in the grout development. This was necessary to eliminate the ash's cementitious properties as a variable and accurately determine the effect of ash content and additives in various grout mixtures.

The study made by Soletanche for RBOSC in 1978 proved that the mixing of only water and ash would not yield a satisfactory grout. The use of additive(s) was necessary to obtain the rheological characteristics required for a successful grouting operation. It was therefore necessary to select an optimum ash content to use for the selection of additives and additional testing.

Twelve additives were tested. The extent of the testing programs increased with the process of selection. Initially, only the rheological properties and the unconfined compressive strength were measured. Permeability tests were then run on six selected grout mixes. Then the two most satisfactory grouts were subjected to an extensive testing program that included long-term permeability, triaxial and consolidation tests.

2.4 Grout Testing

All tests, measurements and procedures for the grout preparation were standardized. Each sample of grout was cured in a water bath at a regulated temperature of 77°C (170°F) to simulate the anticipated temperature of an abandoned MIS retort.

Six characteristics were measured on every grout formulation: specific gravity, Marsh viscosity, dynamic viscosity, setting time, stability, bleeding and unconfined compressive strength. Permeability or triaxial and consolidation tests were performed only in the last phases of the grout selection process.

More completed details of the test procedures are described in Appendix A.

2.5 Test Results

2.5.1 Selection of "Best" Ash for #1 and #2 Blends

Ten different bulk samples of spent shale ash were collected either from the flue gas cyclone (FGC) or from the electrostatic precipitator (ESP) after retorting at the Lurgi Pilot Plant. The ash's properties varied depending on the retorting conditions in the plant when the ash was collected.

A series of preliminary tests were run on each ash sample to determine its selection or rejection for the blends. The ash samples available with the highest unconfined compressive strength were selected for each blend. (The ash used for the trench test had higher strength - Table 26.) The results of these tests are shown in Table 2. Large variations are evident in characteristics such as viscosity, setting time and more particularly, strength.

2.5.2 Characteristics of Ash Blend #1

The blending process is described in Appendix F. Six different bulk samples of ash had to be blended to obtain the quantity needed for grouting vessel #1.

2.5.2.1 Grain Size Distribution. Full deflocculation of the suspension of ash in water could not be obtained, even with an overdose of deflocculating agent; so the grain size analysis could not be performed by means of the usual hydrometer analysis. As a result, within the range of 10 to 40 microns the analysis was performed by means of a series of five cyclones. Table 3 shows the average results of two tests performed on the ash blend #1.

2.5.2.2 Blaine Fineness, Specific Surface. Blaine fineness tests were performed on two samples of ash blend #1. These tests are usually made on cement to determine its fineness. The Blaine fineness that is expressed by a specific surface (cm^2/g) is related to the strength. For cement, it is usually within the range of 2,500 to 3,500 cm^2/g . The average of two measurements made on ash blend #1 was 5,650 cm^2/g .

2.5.2.3 Specific Gravity. The average specific gravity of two ash blend samples was measured according to the standard ASTM C-188 "Specific Gravity of Hydraulic Cement." The average value was 2.71 g/cm^3 .

2.5.2.4 Microstructural Analyses of the Ash. Anhydrous ash mix was analyzed by X-ray diffraction, scanning electron microscopy and electron probe microanalysis.

Results of analyses show the presence of: calcite : Ca CO_3 , dolomite : $\text{Mg Ca (CO}_3)_2$, quartz : Si O_2 , feldspars and calcium sulphates (anhydrite and hemihydrate).

Table 2. Results of Tests on Grouts Prepared with 10 Samples of Ash

Sample #	1	2	3	4	5	6	7	8	9	10
Type of SSA	ESP 4	ESP 6	FGC 6	ESP 7	FGC 7	FGC 8	ESP 9	FGC 9	ESP 10	FGC 10
Amount, g/l	850	850	850	750	800	800	750	800	750	800
Marsh viscosity, sec.	120	67	106	44	120	72	69	57.2	135	120
Dynamic viscosity, cP	-	-	-	350	-	234	450	100	620	580
Casson viscosity, cP	-	-	-	200	-	182	324	80	450	440
Yield stress, Pa	-	-	-	1.61	-	0.20	1.64	0.38	0.56	0.95
Vane shear stress, Pa	-	-	-	4.4	35	1.4	7.9	0.54	10.6	35
Setting time, min.	10	15	25	35	5	10	20	40	35	5
Bleeding, %	+2	+2	0.5	+1	+1	0	+2	7	0	+2
Unconfined compressive strength, 10^5 Pa (psi):										
2 days	0.35 (5.08)	0.39 (5.66)	0.66 (9.6)	0.02 (0.29)	2.39 (34.7)	0.92 (13.3)	2.71 (39.3)	1.18 (17.1)	1.45 (21.0)	4.10 (59.5)
7 days	0.42 (6.09)	0.64 (9.28)	0.85 (12.3)	0.10 (1.45)	2.05 (29.7)	1.48 (21.5)	3.69 (53.5)	3.53 (51.2)	2.40 (34.8)	5.69 (82.5)
14 days	0.44 (6.38)	0.56 (8.12)	1.08 (15.7)	0.35 (5.08)	3.82 (55.4)	1.73 (25.1)	4.33 (62.8)	2.38 (34.5)	2.27 (32.9)	6.35 (92.1)
28 days	0.49 (7.11)	0.61 (8.85)	1.22 (17.7)	0.39 (5.66)	4.73 (68.6)	1.88 (27.3)	4.86 (70.5)	2.59 (37.6)	2.32 (33.6)	7.14 (104.0)

Table 3. Grain Size Distribution of Blend #1

Grain Size, μm	Weight Percent Ash Blend #1
Greater than 39	12.1
29 to 39	7.3
20 to 29	6.6
13 to 20	10.7
9.5 to 13	7.0
less than 9.5	56.3

Complementary analyses were performed on a slurry with 1150 g of ash per liter of grout. The grout was cured for 24 hours at room temperature and analyzed after dehydration. The analyses showed that the setting was mainly controlled by the formation of ettringite or hydrated calcium sulfoaluminate : C_3A , 3 Ca SO_4 , $32 \text{ H}_2\text{O}$ which can quickly crystallize.

2.5.3 Selection of Ash Content

Four grouts with varying ash contents (from 750 to 900 g/l) from blend #1 were prepared and tested to choose the optimum concentration to be used in future tests.

Table 4 lists the test results. These four grouts have very short setting times (5 minutes) and a tendency to swell. As shown on Figure 1, the viscosity increased quickly at 800 g/l and was much too high at 900 g/l. Figures 2 and 3 show respectively the evolutions of unconfined compressive strength versus time and the variations of unconfined compressive strength versus the ash content in the grouts.

The ash content of 850 g/l was selected for the following reasons:

- o The $5.13 \times 10^5 \text{ Pa}$ (74.4 psi) strength at 28 days, lower than the target value, would be improved by addition of the retarders (improvement caused by their peptizing or deflocculating effect). The unconfined compressive strength value for the 750 and 800 g/l ash content were too low, and it was quite satisfactory for the 900 g/l grout.
- o The Marsh viscosity of 66 seconds, higher than the target value, would be reduced by the effect of the additives. On the other hand, the Marsh viscosity of 120 seconds obtained from the 900 g/l grout was so high that this ash content was eliminated.

Table 4. Results of Tests on Four Grouts
Prepared with Blend #1 (Without Additives)

Sample #	13	14	15	16
Type of Ash Amount, g/l	Blend #1 750	Blend #1 800	Blend #1 850	Blend #1 900
Marsh viscosity, s	37.0	43.6	66.0	120
Dynamic viscosity, cP	112	185	340	940
Casson viscosity, cP	52	115	332	840
Yield stress, Pa	1.52	1.11	0.58	0.20
Vane shear stress, Pa	0.89	1.52	3.80	8.50
Setting time, min	5	5	5	5
Bleeding, %	+3	+4	+4	+3
Unconfined compressive strength, 10 ⁵ Pa (psi):				
2 days	1.35 (19.6)	1.83 (26.5)	3.03 (43.9)	3.93 (57.0)
7 days	1.84 (26.7)	2.52 (36.5)	3.55 (51.5)	4.69 (68.0)
14 days	2.00 (29.0)	2.81 (40.8)	3.62 (52.5)	4.75 (68.9)
28 days	2.46 (35.7)	3.20 (46.4)	5.13 (74.4)	6.83 (99.1)

2.5.4 Selection of Retarding and Fluidizing Agents

The next step was to select a suitable retarder and fluidizier. Thirty additives had been tested in the previous grout study for RBOSC. The most efficient ones were based on active components of lignosulfonate, sodium gluconate or gluconic acid, and liquid sodium silicate.

Twelve additives were tested in this program:

- 6 sodium silicates whose silica/sodium oxide ratio by weight ($\text{SiO}_2/\text{Na}_2\text{O}$) varied between 2.55 and 3.85
- 2 Lignosulfonates
- 1 Lignosulfonate and gluconate mix
- 2 Sodium gluconates
- 1 Gluconic acid

The characteristics of these additives are shown in Tables 5 and 6.

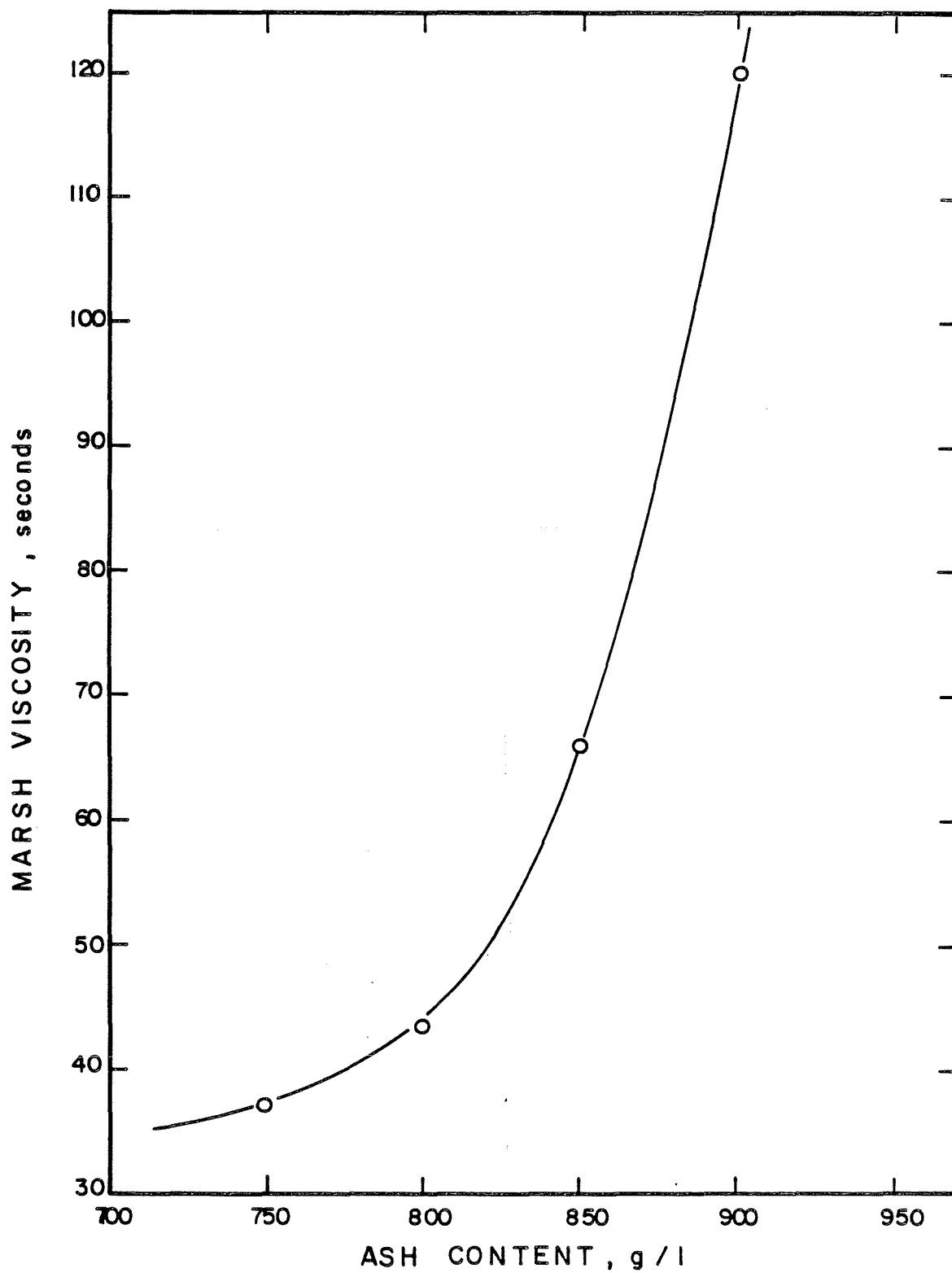


FIGURE 1 . - Marsh viscosity vs ASH blend#1 content

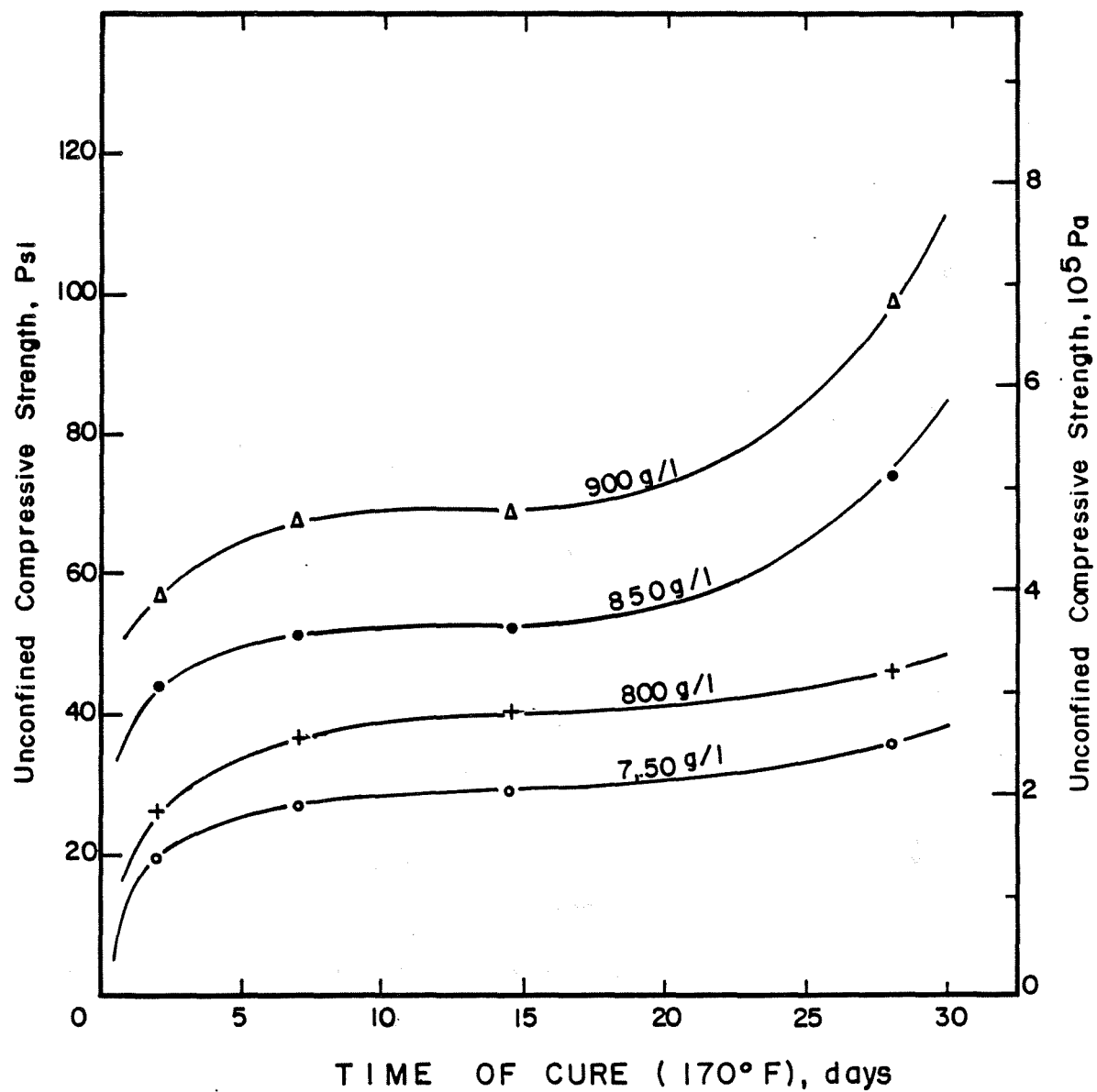


FIGURE 2 .— Unconfined compressive strength vs curing time for four ASH blend#1 contents

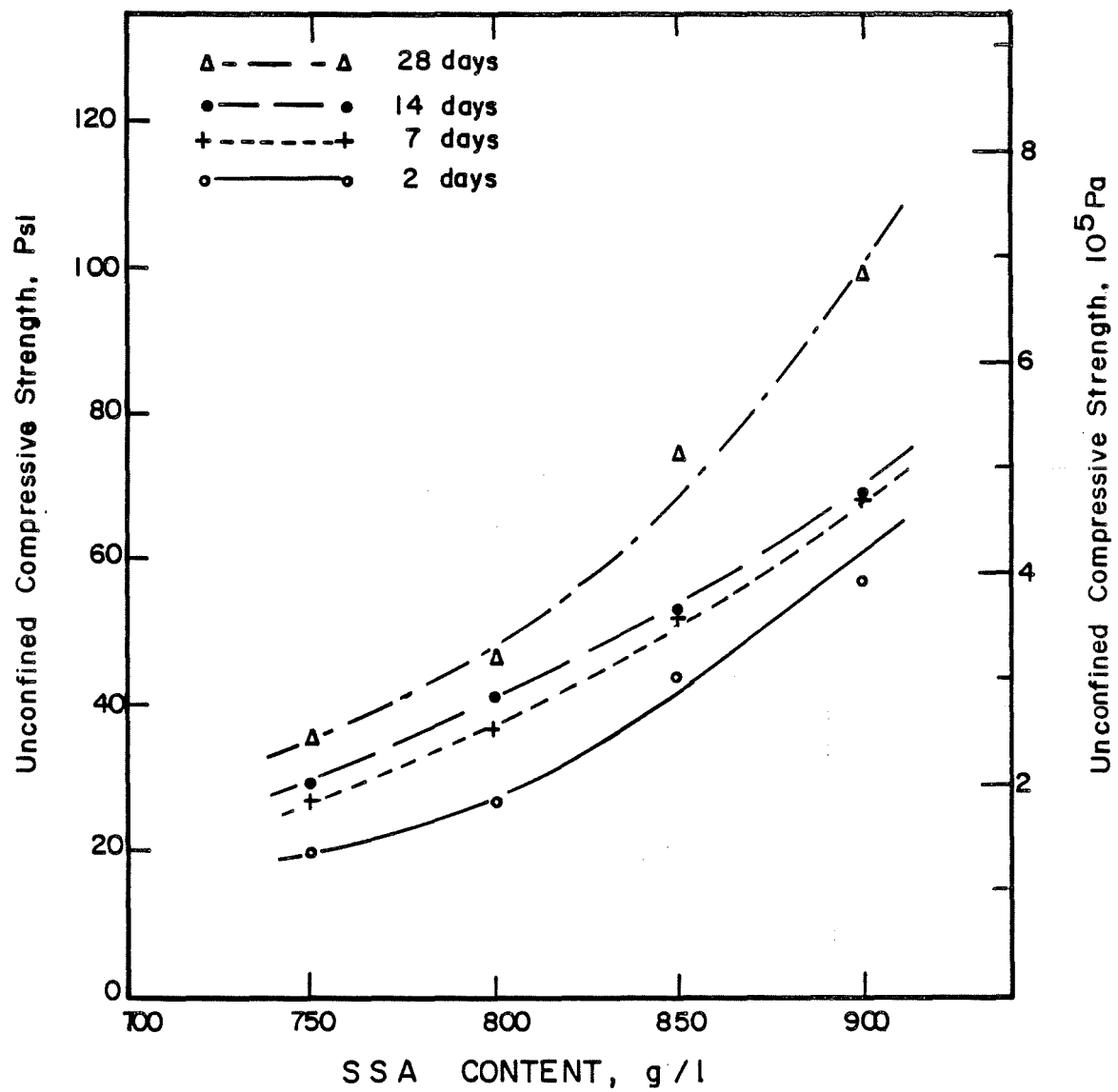


FIGURE 3 .- Unconfined compressive strength vs
ASH blend # 1 content

Table 5. List of Sodium Silicates

Type of Silicate	SiO ₂ /Na ₂ O by Weight	Specific Gravity g/cm ³	Baume°
M	2.58	1.51	49.3
N	3.22	1.39	41
SR 35	3.75	1.32	35
JW 25	2.55	1.41	42
40	3.22	1.40	41.5
34	3.85	1.31	34.5

Table 6. List of Chemical Retarders

Name	Main Active Component	Specific Gravity, g/cm ³
Orzan GL 50	Lignosulfonate	1.23
PDA 25R	Lignosulfonate	1.21
Daratard 17	(likely) lignosulfonate + gluconate	1.22
Protard	Na gluconate	1.17
Liquid gluconate 60	Na gluconate	1.32
Gluconic acid 50	Gluconic acid	1.23

2.5.4.1 Liquid Sodium Silicates. The six types of silicates were used at the same concentration of 10 cm³/l (samples #29 to 34). Table 7 shows all results of tests on six grouts containing those silicates (samples #29 to 34). The following observations can be made.

The grouts prepared with silicates of identical SiO₂/Na₂O ratios had very similar characteristics. Grouts 29 and 30 had ratios of 2.58 and 2.55; samples 31 and 32 had ratios of 3.22; and samples 33 and 34 had ratios of 3.75 and 3.85.

These three pairs of grouts showed the following characteristics:

- The Marsh viscosities of grouts with silicate ratios of 2.55 are lowest, whereas they are at their maximum with the grouts of silicate ratio 3.22;
- The setting time is longer when the silicate ratio is 2.55 than when it is higher;
- The unconfined compressive strengths of all the grouts are similar.

Table 7. Results of Tests on Grouts Prepared with Blend #1 and Sodium Silicates

Sample #	29	30	31	32	33	34	35	36
Type of SSA	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1
Amount, g/l	850	850	850	850	850	850	850	850
Additive 1: Type	Silicate M(2.58)	Silicate JW25(2.55)	Silicate N(3.22)	Silicate 40(3.22)	Silicate SR35(3.75)	Silicate 34(3.85)	Silicate M(2.58)	Silicate JW25(2.55)
Amount, ml/l	10	10	10	10	10	10	5	5
Marsh viscosity, sec.	99	92.5	134	132	119	119	80.5	84.5
Dynamic viscosity, cP	440	470	520	630	460	570	500	570
Casson viscosity, cP	360	390	500	560	450	530	460	500
Yield stress, Pa	0.64	0.45	0.23	0.13	0.20	0.31	0.26	0.38
Vane shear stress, Pa	8.23	7.1	8.7	2.85	7.6	6.5	4.75	4.9
Setting time, min.	60	60	50	55	40	55	40	25
Bleeding, %	0	0	+1	0	+0.5	+0.5	+1	+1.5
Unconfined compressive strength, 10^5 Pa (psi):								
2 days	4.00 (58.0)	3.76 (54.5)	4.12 (59.8)	3.79 (55.0)	3.38 (49.1)	3.77 (54.6)	3.83 (55.5)	3.34 (48.4)
7 days	4.76 (69.0)	5.34 (77.4)	5.52 (80.1)	5.17 (74.9)	4.62 (67.0)	4.74 (68.7)	4.33 (62.8)	3.72 (53.9)
14 days	5.62 (81.5)	5.66 (82.1)	6.30 (91.3)	6.06 (87.9)	5.38 (78.0)	5.54 (80.4)	5.03 (72.9)	4.34 (63.0)
28 days	5.76 (83.5)	5.80 (84.1)	6.16 (89.4)	6.16 (89.3)	5.47 (79.4)	5.57 (80.8)	5.42 (78.7)	4.77 (69.1)

Two other grouts were prepared with the best silicate ratio, 2.55, only the silicate was used in a lower concentration: 4 cm³/l (samples #35 and 36). For these samples the Marsh viscosities are around 80 seconds instead of 90, the setting times are shorter and the unconfined compressive strength slightly lower. Therefore, the lower concentration of silicate does not seem to improve the overall characteristics.

The bleedings and the unconfined compressive strengths were close to the target values. However, the viscosities were too high and the setting times too short. Sodium silicate could not achieve the target grout characteristics.

2.5.4.2 Chemical Retarders. Tables 8 and 9 show the test results with the various chemical retarders in various concentrations. The use of sodium gluconate (samples #42, 43 and 44) gave results closest to the targets.

2.5.4.3 Cement. For most grouts tested with chemical retarders, the unconfined compressive strengths were less than the target value of 100 psi. Two grouts were prepared with a small amount of Portland Type II cement to determine the minimum amount of cement that would take the grouts to the lower limit of 0.7 MPa (100 psi).

Table 9 shows that the addition of 25 and 50 g/l of cement (samples #46 and 47 respectively) yielded, with regard to the reference grout #15, a lower Marsh viscosity and a higher unconfined compressive strength. Even with a small addition of cement as low as 25 g/l, the strength increase was significant. For this reason, a last sample (#48) was prepared with the addition of both gluconate (1 ml/l) and cement (only 10 g/l). The viscosity, bleeding and unconfined compressive strength values were very encouraging; but the setting time was still too short.

2.5.5 Tests on Selected Grouts

The primary additive selected was sodium gluconate. Secondary additives, either silicate to increase the setting time or Portland Cement Type II to increase the unconfined compressive strength, were tested further.

The composition and characteristics of six selected grout formulations are given in Table 10 together with their test results. The first two samples were prepared with only one additive. Sample #49 contained gluconate and sample #50 Protard. The other four grouts contained two additives: #51, gluconate and cement, #52 and #54, gluconate and silicate (two concentrations), and #53 Protard and silicate.

As shown in Table 10, the Marsh viscosity was within the target range for the first three samples but was too high for the samples with silicate. The setting time was still 25 to 35 minutes too short for the first three samples; it was almost correct for samples #52 and #54 with gluconate and silicate. The bleeding value was within the target value for all the samples; samples #50 and #51 gave the best results with a bleeding value of only one percent. The unconfined compressive strengths were very close to each other at the same age except for sample #51 with cement, which had a noticeably higher strength.

Table 8. Results of Tests on Grouts Prepared with Blend #1 and Chemical Retarders

Sample #	15	37	38	39	40	41
Type of SSA	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1
Amount, g/l	850	850	850	850	850	850
Additive 1:						
Type	-	Orzan GL	PDA 25R	PDA 25R	Daratard	Daratard
Amount, ml/l	-	10	10	7	10	5
Marsh viscosity, sec.	66	47.2	45.2	48.3	46	41
Dynamic viscosity, cP	340	70	75	80	132	135
Casson viscosity, cP	332	40	46	45	42	66
Yield stress, Pa	0.58	0.21	0.20	0.33	0.37	0.80
Vane shear stress, Pa	3.8	0.16	0.10	0.16	0.16	0.32
Setting time, min	5	40	70	35	100	15
Bleeding, %	+4	+1.5	2	+2	2	+2
Unconfined compressive strength, 10^5 Pa (psi):						
2 days	3.03 (43.9)	1.24 (18.0)	1.02 (14.8)	2.07 (30.0)	too soft too soft	too soft too soft
7 days	3.55 (51.5)	2.78 (40.3)	3.62 (52.5)	3.18 (46.1)	0.36 (5.2)	2.60 (37.7)
14 days	3.62 (52.5)	3.12 (45.3)	3.99 (57.9)	3.07 (44.5)	1.93 (28.0)	2.82 (40.9)
28 days	5.13 (74.4)	3.60 (52.2)	4.67 (67.7)	3.60 (52.2)	2.39 (34.7)	2.73 (39.0)

Table 9. Results of Tests on Grouts Prepared with Blend #1 Chemical Retarders and Cement

Sample #	15	42	43	44	45	46	47	48
Type of SSA	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1
Amount, g/l	850	850	850	850	850	825	800	850
Additive 1:								
Type	-	Protard	Protard	Gluconate	Gluconic Acid	-	-	Gluconate
Amount, ml/l	-	5	2.5	1	1	-	-	1
Additive 2:								
Type	-	-	-	-	-	Cement	Cement	Cement
Amount, g/l	-	-	-	-	-	25	50	10
Marsh viscosity, sec.	66	51.7	47	51.5	53.6	61	52.3	50
Dynamic viscosity, cP	340	125	125	160	150	490	310	145
Casson viscosity, cP	332	68	68	84.5	79	480	360	85
Yield stress, Pa	0.58	0.83	0.80	1.00	1.00	0.20	0.15	0.90
Vane shear stress, Pa	3.8	0.57	0.16	0.47	0.47	3.65	2.7	0.47
Setting time, min.	5	135	110	100	100	10	10	85
Bleeding, %	+4	5	3.5	1.5	1	+3	+3.5	2
Unconfined compressive strength, 10 ⁵ Pa (psi):								
2 days	3.03 (43.9)	1.91 (27.7)	3.90 (56.6)	3.60 (52.1)	3.16 (45.8)	4.55 (66.0)	6.52 (94.5)	4.48 (64.9)
7 days	3.55 (51.5)	5.35 (77.7)	5.39 (78.2)	4.37 (63.4)	4.17 (60.4)	6.29 (91.2)	10.40 (150.8)	6.35 (92.1)
14 days	3.62 (52.5)	6.02 (87.3)	6.96 (100.9)	4.25 (61.7)	4.39 (63.7)	6.05 (87.7)	11.90 (172.6)	6.31 (91.2)
28 days	5.13 (74.4)	7.77 (112.0)	6.84 (99.2)	4.33 (62.7)	5.35 (77.6)	7.99 (115.9)	13.90 (201.6)	7.21 (104.6)

Table 10. Results of Tests on Six Selected Grouts

Sample #	49	50	51	52	53	54
Type of SSA	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1	Blend #1
Amount, g/l	850	850	850	850	850	850
Additive 1:						
Type	Gluconate	Protard	Gluconate	Gluconate	Protard	Gluconate
Amount, ml/l	1	2	1	1	2	1
Additive 2:						
Type	-	-	Cement Portland 2	Silicate PQ M(2.58)	Silicate PQ M(2.58)	Silicate PQ M(2.58)
Amount, g/l	-	-	10	5	5	7.5
Marsh viscosity, sec.	49.5	49.2	52	57.4	59	68
Dynamic viscosity, cP	140	140	160	235	225	305
Casson viscosity, cP	78	70	94	137	135	250
Yield stress, Pa	0.85	0.92	0.85	1.08	1.24	0.51
Vane shear stress, Pa	0.47	0.95	0.16	1.58	1.42	3.48
Setting time, min.	95	95	85	110	80	105
Bleeding, %	4	1	1	3.5	4	2
Unconfined compressive strength, 10^5 Pa (psi):						
2 days	3.50 (50.8)	2.80 (40.6)	5.02 (72.9)	3.40 (49.3)	2.61 (37.8)	3.12 (45.3)
7 days	4.77 (69.1)	3.89 (56.4)	7.24 (105.0)	5.26 (76.3)	4.36 (63.2)	5.43 (78.8)
14 days	5.65 (81.9)	4.72 (68.4)	8.04 (116.6)	6.12 (88.8)	5.88 (85.2)	5.88 (85.3)
28 days	6.10 (88.4)	5.49 (79.6)	8.02 (116.3)	6.68 (96.9)	6.43 (93.3)	6.36 (92.2)

Permeability tests were run on the six grouts after 14 and 28 days of curing at 170°F (see Appendix A for detailed test procedure).

One sample of every grout was tested over a period of 12 days after curing for 14 days. Table 11 shows that the average results are close to the target value. Four samples (#50, #52, #53, #54) had permeabilities close to each other in the range 10×10^{-7} cm/s, the other two samples (#49 and #51) showed a slightly lower permeability, especially #51 with 4.5×10^{-7} cm/s. The next series of permeability tests were performed on grout samples cured for 28 days.

The overall results were encouraging and rather close to each other, within the range 4.4 to 13.5×10^{-7} cm/s. The best results were obtained with grouts #49 and #51 which contained gluconate.

At this stage, it was concluded that grouts with only one additive should, if possible, be preferred because in a commercial project the preparation of a grout would be easier if it contained only one additive and the cost of the grout as well as the cost of the preparation plant would be lower.

2.5.6 Selection of Two Most Promising Grouts for Additional Tests

The two grouts judged the most promising were selected and subjected to further testing. The three grouts with silicate (samples #52, #53, and #54) were eliminated due to their high viscosities.

Table 11. Results of Permeability Tests on Six Selected Grouts

Sample #	Additives	Permeability, 10^{-7} cm/s				
		14 day samples	28 day samples			
			1st day	10th day	20th day	30th day
49	Gluconate	7.5	7.8*	4.9*	5.1*	4.7*
50	Protard	11.0	13.5*	7.2*	7.5*	7.3*
51	Gluconate cement	4.5	5.6*	4.7*	3.9*	4.4*
52	Gluconate silicate	9.0	7.7	6.2	5.6	6.0
53	Protard silicate	10.0	7.2	6.6	11.0	11.0
54	Gluconate silicate	10.0	--	--	--	--

*Average of measurements made on two samples.

The grout corresponding to sample #49 was selected over #50 because its permeability was slightly lower. The mix of sample #51 (gluconate + cement) was also chosen, even though it included two additives, because its mechanical characteristics were by far the best.

2.5.6.1 Results of Triaxial and Consolidation Tests. Consolidated drained triaxial tests were run on grouts #49 and #51. Sample dimensions and conditions are shown in Appendix C. Prior to testing, the samples were cured for 28 days. The tests were run at a constant rate of strain of 0.001 inch/min after being consolidated at 1.72 , 3.45 and 6.90×10^5 Pa (3.6, 7.2 and 14.4 kips/sq ft). The mohr-coulomb failure diagram confirms the higher value of the unconfined compressive strength shown in Table 10 for the mix with gluconate and cement (#51). A summary of the results is given in Table 12. The cement additive in grout #51 is probably responsible for the higher cohesion value.

One dimensional consolidation tests were also run on these sample mixes. The consolidation curves are in Appendix C. These results show that consolidation occurs slowly and that there is virtually no rebound on unloading and saturating the sample. They also confirm the higher strength of sample mix #51.

2.5.6.2 Long Term Permeability. To evaluate the long-term behavior of the spent ash grouts, the cured grouts used for the permeability tests were kept in the permeability cells for an additional 150 days.

These durability tests were performed on grouts #49, #50, #51 (two samples each) and #52 (one sample). Sample #53 was eliminated because it showed the highest permeability after 30 days (Table 11). For the permeability test, the gradient was maintained close to 50; i.e., a percolation pressure around 0.6×10^5 Pa (9 psi) through a 125 mm (5 in) high sample and a lateral pressure as high as 1.3×10^5 Pa (18 psi) (refer to Appendix A).

Figures 4 and 5 are graphs of permeability vs. time for samples #49 and #51, respectively. The permeability tests were stopped for 35 days, and during that time the grout samples swelled. After that, permeability values started at a higher level, but then they decreased to the previous levels within a few days. Afterwards, the permeability curve fluctuates, especially for #51. For grout #51, the results of both samples were very similar and the two curves show the same shapes. For grout #49, the results of both samples were so close to each other that the curves cannot be distinguished from one another. The permeability characteristics for the three other grouts were fairly similar.

Table 12. Results of Triaxial Shear Tests

Sample #	#49	#51
Angle of friction, degrees	39	30.5
Cohesion, 10^5 Pa (psi)	.58 (8.4)	1.9 (27.5)

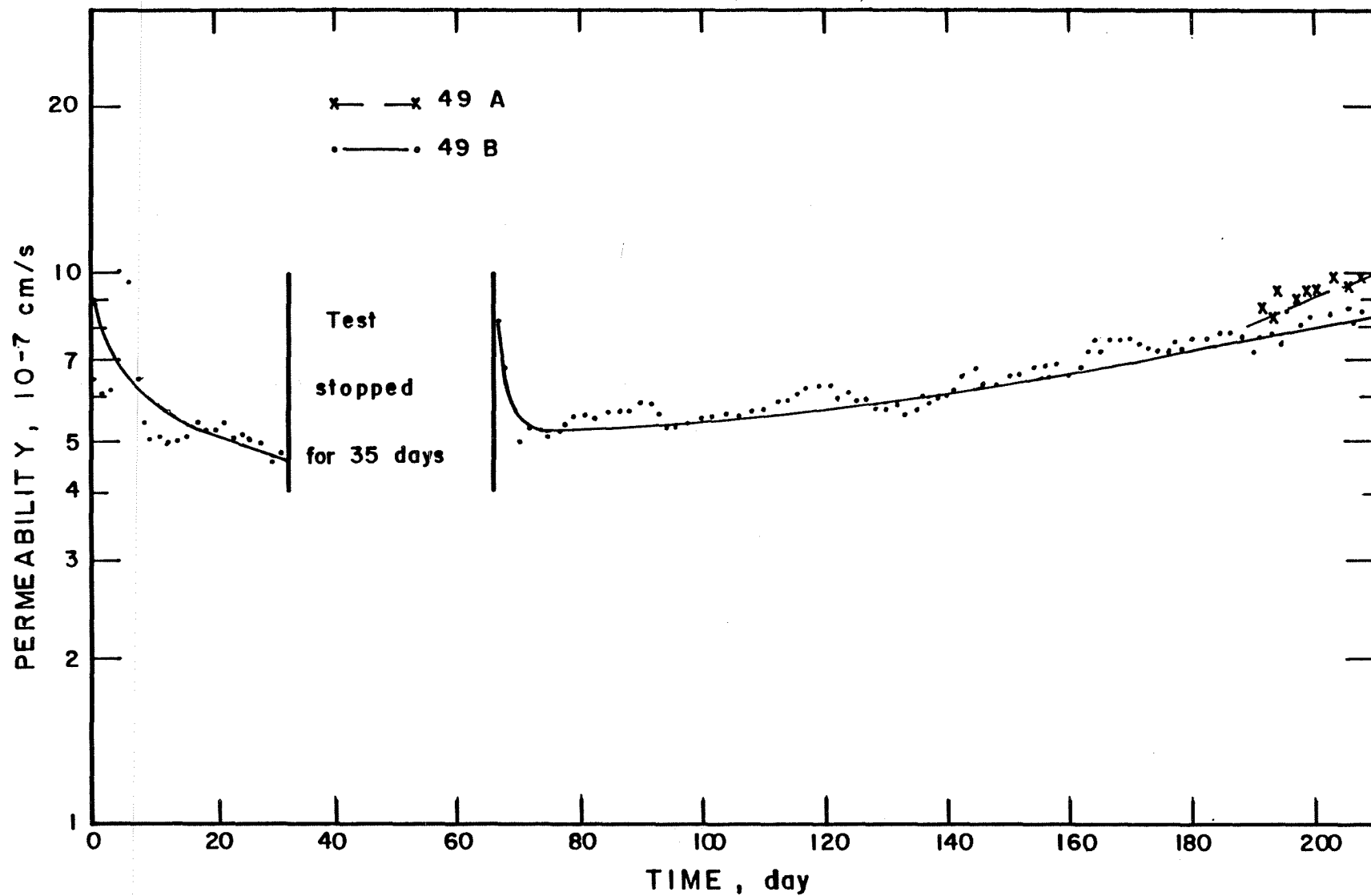


FIGURE 4.- Sample # 49. Permeability vs time

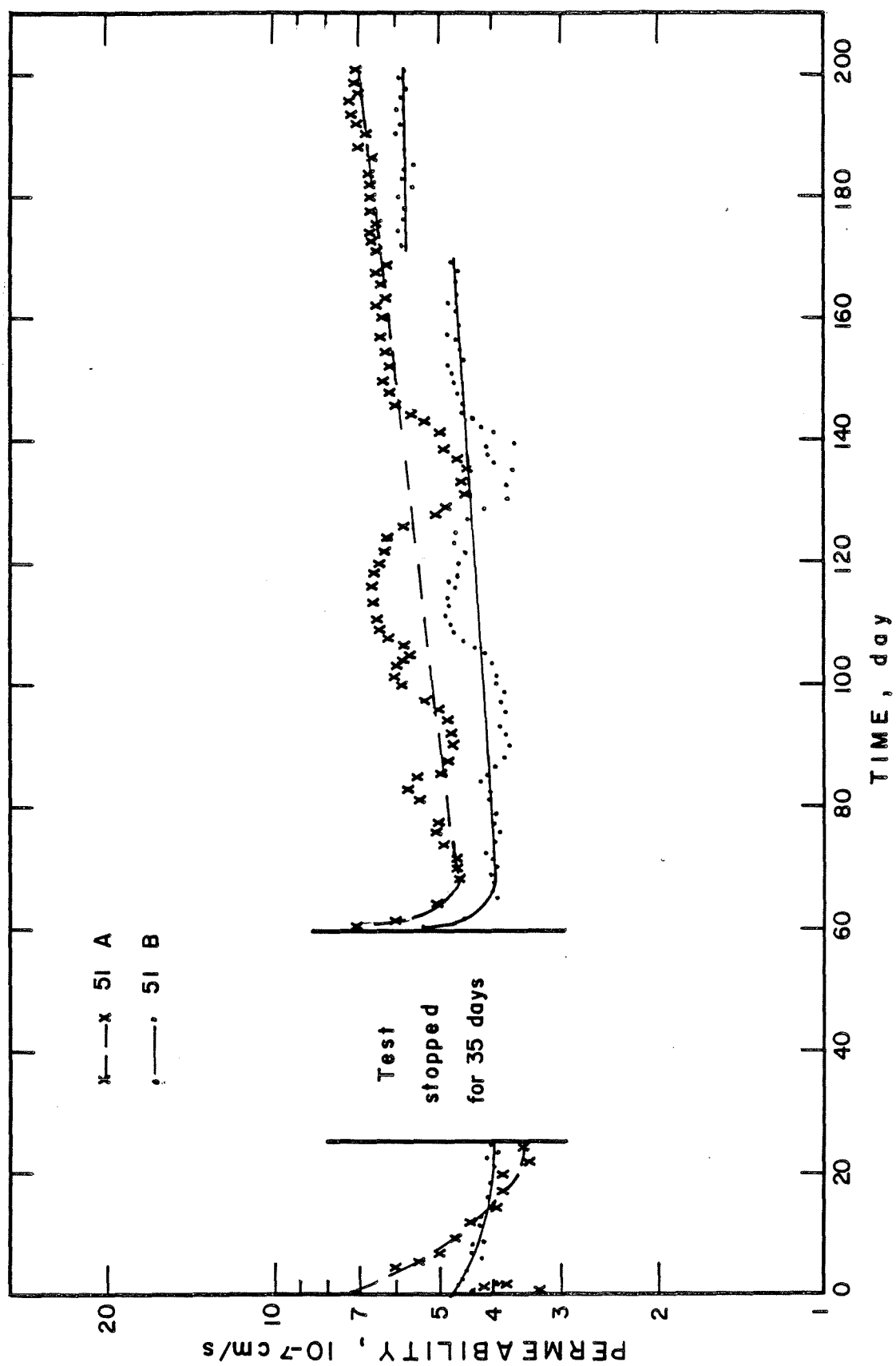


FIGURE 5 . -Sample # 51. Permeability vs time

It appeared that a very slow increase in permeability was taking place over time. This tendency is not necessarily due to a corrosion or deterioration of the grouts; it may be due to the indirect effect of the initial compression and decompression that could have led to microfissuration. It is possible that a secondary effect from silicate as well as Protard was taking place. Whatever the cause, the increase was very slow, even for a gradient as high as 50, whereas the real gradient in the retort would be less than one. Finally, long-term permeability tests confirmed the choice of grouts corresponding to samples #49 and #51 as the two most promising for grouting of the vessels.

2.5.7 Tests on Grouts Prepared with Blend #2

As explained previously, another ash blend (blend #2) was prepared in order to grout vessel #2 with the ash available at that time. The blending process was the same as the one used for blend #1 (refer to Appendix F for more details on producing various blends).

An additional series of grouts was prepared to check the characteristics of ash blend #2 and to find a formulation yielding characteristics similar to those of grout #51.

Seven grouts were prepared from blend #2, and they were cured and tested in the same way as the grouts from blend #1. Results are shown on Table 13.

The preliminary tests performed with the various types of ash (Table 2) showed that the ash added to blend #2 was weaker than the ash used for blend #1. Therefore, grouts made with blend #2 were expected to have overall lower mechanical characteristics. This was confirmed by the test results.

Compared to grouts from blend #1, grouts from blend #2 had weaker unconfined compressive strength, lower Marsh viscosities, and slightly longer setting times.

2.6 Characteristics of Grouts Selected for Vessel Grouting

Table 14 shows all the characteristics of both selected grouts and the target characteristics. The main differences between the two grouts were as follows:

- two different ash blends
- the addition of cement in sample #59
- a bleeding value of 4% for sample #49 instead of 1.5% for grout #59
- the unconfined compressive strength reached 7.7×10^5 Pa (112 psi) for sample #59 which was 1.65×10^5 Pa (24 psi) more than that of sample #49.

However, the rheological characteristics of the grouts were similar.

Table 13. Results of Tests on Grouts Prepared with Blend #2

Sample #	55	56	57	58	59	60	61
Type of SSA	Blend #2	Blend #2	Blend #2	Blend #2	Blend #2	Blend #2	Blend #2
Amount, g/l	850	850	850	850	850	850	850
Additive 1:							
Type	-	Gluconate	Gluconate	Gluconate	Gluconate	Gluconate	Gluconate
Amount, ml/l	-	1	1	1	1	1	1
Additive 2:							
Type	-	-	Cement Portland 2	Cement Portland 2	Cement Portland 2	Lime	Lime
Amount, g/l	-	-	10	20	15	20	40
Marsh viscosity, sec.	56.4	45.6	45.6	46.3	47	49	59
Dynamic viscosity, cP	460	135	135	135	145	200	300
Casson viscosity, cP	350	60	62	66	64	87.5	190
Yield stress, Pa	1.20	1.17	1.14	1.18	1.18	1.81	1.15
Vane shear stress, Pa	5.38	0.63	0.80	0.80	0.80	1.58	1.74
Setting time, min	5	100	95	115	100	30	25
Bleeding, %	+2	1.5	1.5	1	1.5	+4	+3
Unconfined compressive strength, 10 ⁵ Pa (psi):							
2 days	2.36 (34.2)	2.80 (40.6)	3.99 (57.8)	5.13 (74.3)	4.56 (66.2)	3.10 (44.9)	4.00 (58.1)
7 days	3.16 (45.8)	3.71 (53.7)	6.40 (92.9)	8.12 (117.8)	6.57 (95.3)	5.88 (85.2)	10.10 (146.5)
14 days	3.79 (55.0)	4.39 (63.7)	7.25 (105.1)	9.02 (130.8)	7.24 (105.0)	6.46 (93.7)	1) 1)
28 days	4.52 (65.5)	3.50 (50.7)	6.16 (89.4)	10.46 (151.7)	7.72 (112.0)	7.11 (103.1)	1) 1)

1) Broken when removed from the molds.

Table 14. Characteristics of Both Selected Grouts

Sample #	49	59	Target
Type of ash	Blend #1	Blend #2	As much as possible
Amount g/l	850	850	
Additive 1:			
Type	Gluconate	Gluconate	
Content, l/m ³	1	1	
Additive 2:			
Type		Cement	
Content, kg/m ³		15	
Marsh viscosity, sec	49.5	47	45-55
Dynamic viscosity, cP	140	145	
Casson viscosity, cP	78	64	
Yield stress, Pa	0.85	1.18	
Vane shear stress, Pa	0.47	0.80	
Setting time, min.	95	100	120
Bleeding, %	4	1.5	5
Permeability, cm/s x 10 ⁻⁷	5	4.5	10 ⁻⁶
Unconfined compressive strength, 10 ⁵ Pa (psi)			
2 day	3.5 (51)	4.6 (66)	
7 day	4.8 (69)	6.6 (95)	
14 day	5.6 (82)	7.2 (105)	
28 day	6.1 (88)	7.6 (112)	7.0 (100)

The only target value not achieved was the setting time, which was again 20 to 25 minutes short of the target. This characteristic of grouts prepared under laboratory conditions could be different from those of grouts prepared in commercial or pilot plants. Moreover, the characteristics of the grouted mass of rubble will depend greatly upon the technology and the quality of the grouting. These questions will be examined in Chapter 3 of this report "Tests on Laboratory Scale Models."

3. TESTS ON LABORATORY SCALE MODELS

3.1 General

The laboratory scale models were tested to check groutability of the selected grouts in a volume of simulated rubble and to define the strength and permeability of the grouted medium.

Two small scale models were designed that included simulation of the expected in-situ conditions of the retorted shale medium for temperature, rubble size distribution, and grouting pressure. Instead of the conventional groutability test molds normally used under laboratory conditions, large steel vessels were employed. The vessels were better adapted to the rubble size, and allowed several cubic meters of rubble to be pressure grouted.

3.2 Facilities and Test Preparation

Appendix F gives a complete description of the test facilities which are summarized below.

To minimize the effects of weather on equipment and instrumentation, the test program was conducted inside a pilot-plant building at CSMRI's facility in Golden, Colorado.

Two test vessels were designed and constructed. As shown in Figure 6 and Photo 1, each vessel was 3.4 m (11 ft) high, had a 1.5 m (5 ft) inner diameter, and weighed approximately 4,990 kg (11,000 lb) when empty. Each was equipped with a 1.9 cm (3/4 in) external steam coil that heated the vessel. The coil was helically wrapped around the vessel from top to bottom of the working height. The inside wall was equipped with 2.5 cm (1 in) baffles, as shown in Photo 2, spaced 0.3 m (1 ft) apart.

Each vessel was equipped with 33 access ports that were used for observation, grout injection, instrumentation, permeability testing or as pressure relief valves. Photo 2 shows the inside of the vessels including the lowest grouting pipes.

The grout mixing and injection plant, shown in Figure 7 and Photo 3, consisted of a grout mixer, a grout storage tank with an agitator, a feed tank and an injection pump.

The grout mixer (not shown) was a special unit developed by Soletanche. It consisted of a 170 l (45 gal) mixing bowl with a vertically mounted pump in the bottom of the bowl. When grout was prepared, the pump would circulate the mixture through the bowl and thoroughly mix the ash, additive and water. After preparation, the grout was transferred to the storage tank which was equipped with an agitator to prevent settling.

The injection pump was a pneumatically driven Moyno pump. The discharge of the pump was equipped with a recirculating line that allowed grout to be pumped back to the feed tank during periods of low or zero grout intake. The pump discharge was also equipped with a pressure gauge so that grouting pressure could be monitored during the operation.

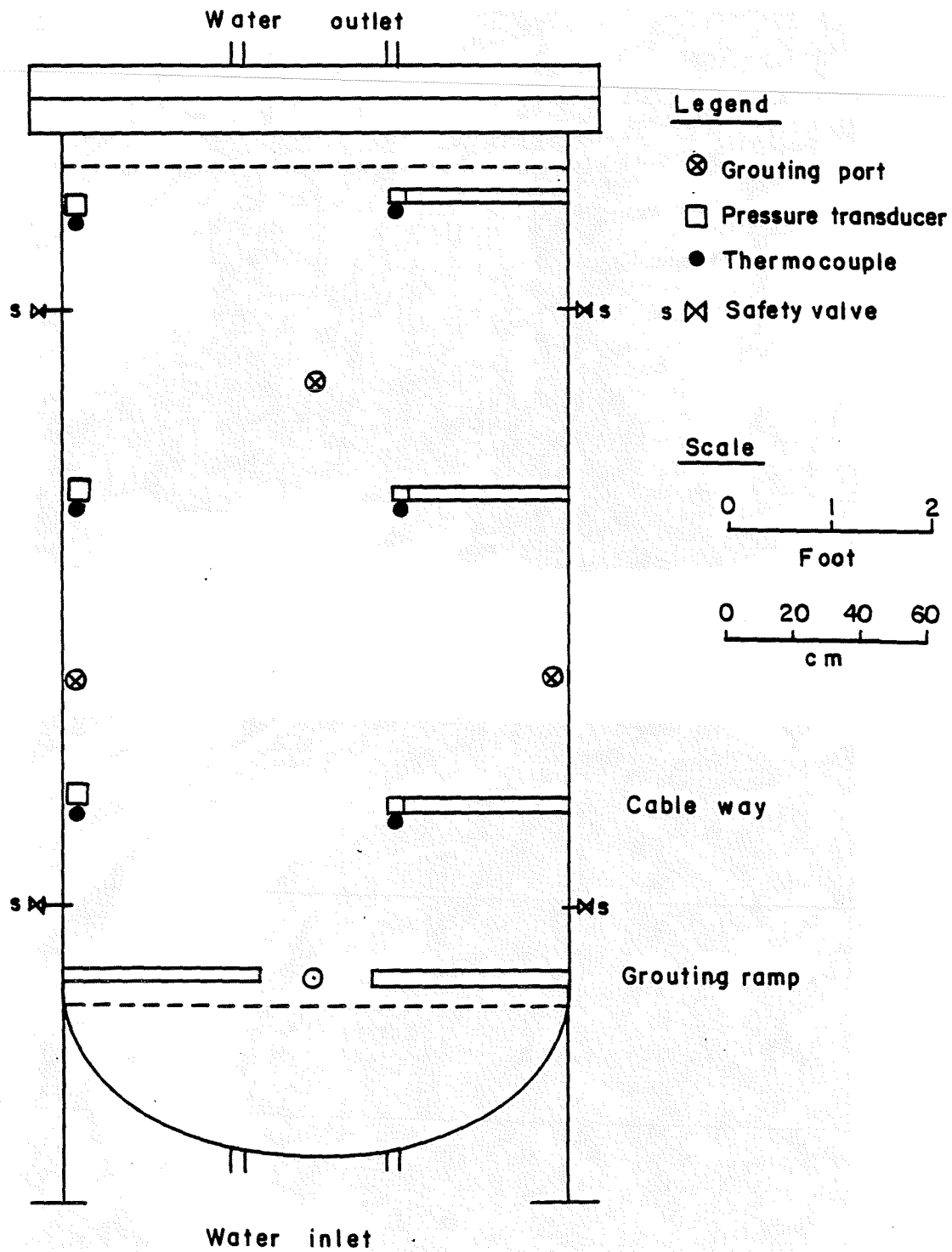


FIGURE 6 . — Vessel Schematic



PHOTO 1. Test Vessels and Platform

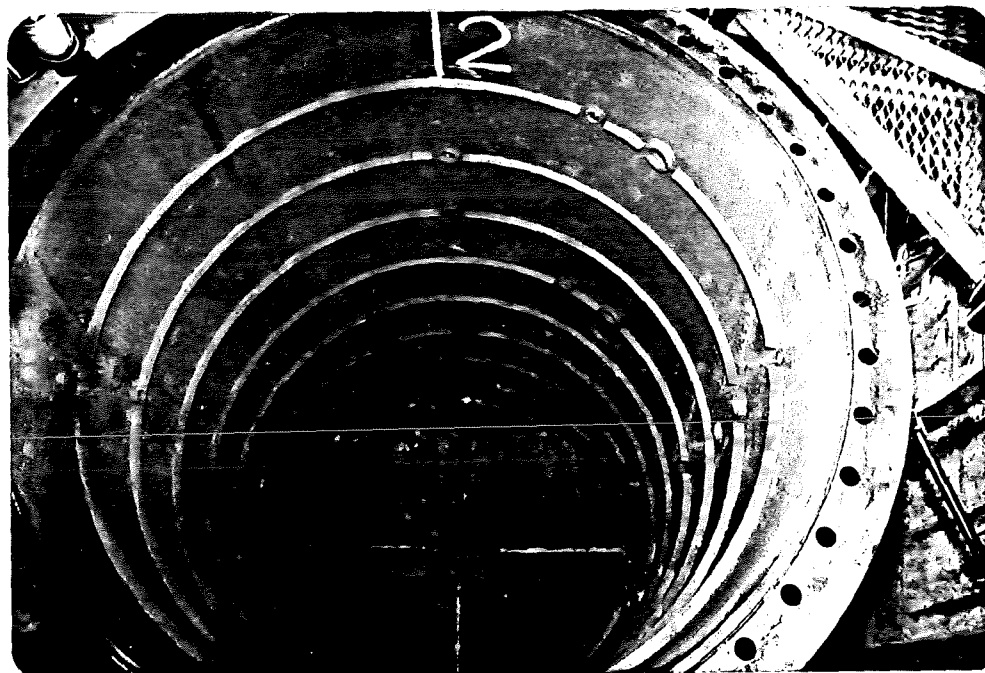


PHOTO 2. Grouting Pipes at Bottom of Vessel

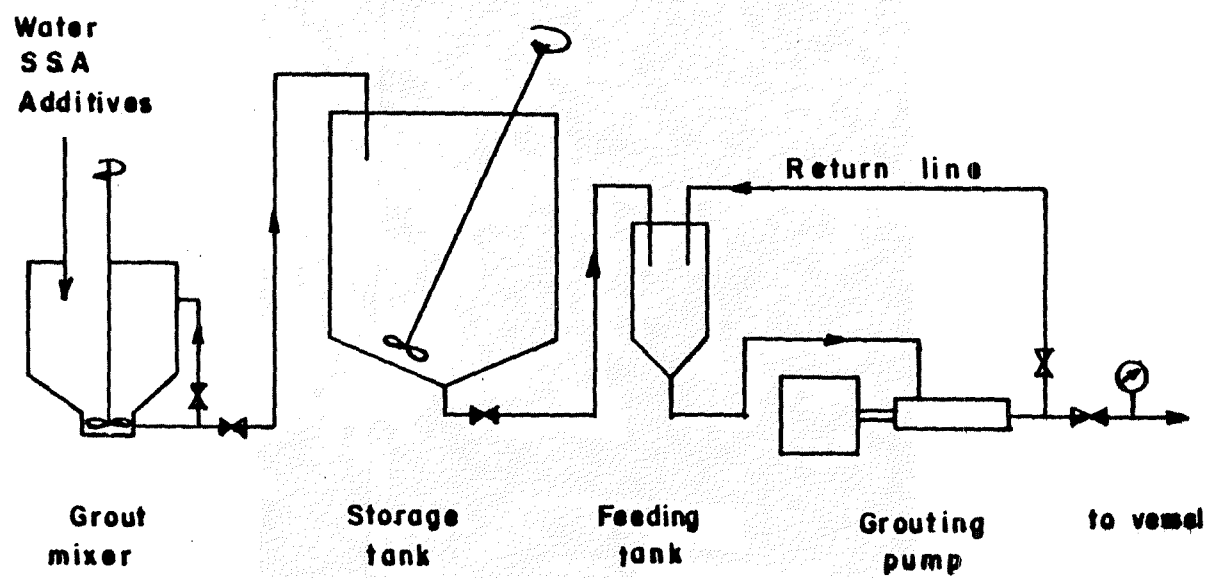


FIGURE 7. - Schematic of Grouting Plant

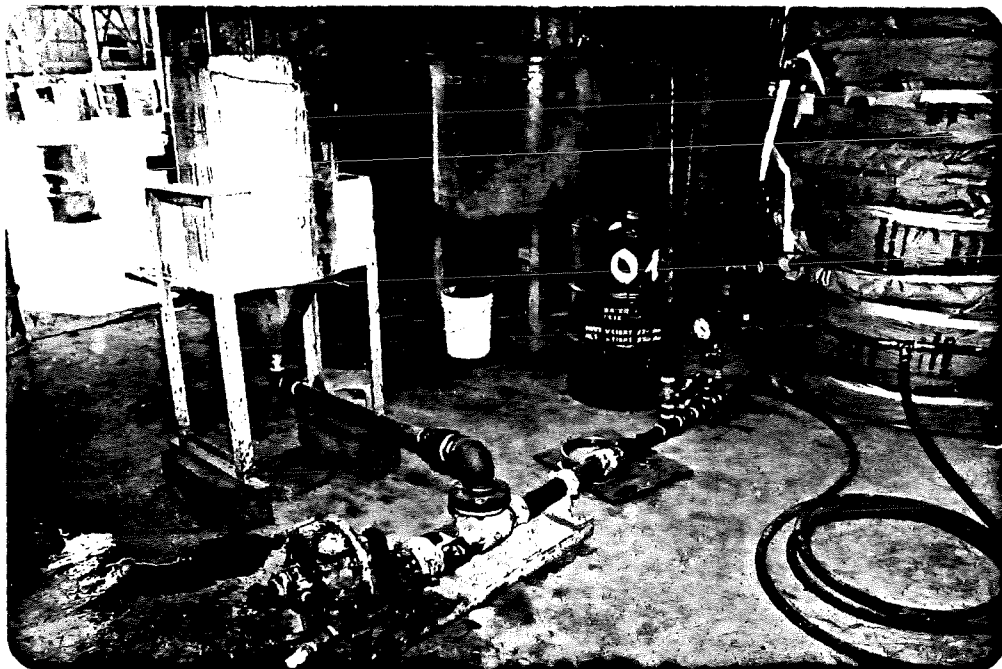


PHOTO 3. Grout Mixing and Injection Plant

3.3 Characteristics of Simulated Rubble

3.3.1 Origin of the Rubble

The spent shale rubble that was used to simulate the abandoned retort was produced at Laramie Energy Technology Center (LETC) by retorting oil shale from Federal Lease Tract C-a. The rubble was covered to minimize the effects of weathering during storage.

Before loading the vessels with spent shale rubble, certain physical characteristics of the rubble were measured, such as particle size distribution, specific gravity, water absorption and unconfined compressive strength.

3.3.2 Particle Size Distribution

Because of the large amount of handling that would be required to obtain a representative sample of the rubble and due to the brittle nature of the rubble, the particle size distribution was determined by screening the entire sample at 4 in, 1 in, $\frac{1}{2}$ in, and 6 Mesh (101.6, 25.4, 12.7, and 3.35 mm). Any material larger than 4 in (101.6 mm) was disregarded. The results of the screen size analysis are shown in Table 15.

3.3.3 Specific Gravity and Water Absorption

Six blocks of spent shale, representative the various degrees of retorting that would be found in full-scale MIS retorts, were collected before screening. Their color varied from light grey (highly retorted) to black (lightly retorted).

Specific gravity and water absorption measurements were performed on these samples according to ASTM C97, "Absorption and Bulk Specific Gravity of Natural Building Stone." The average, minimum and maximum values recorded in Table 16 are representative of the extremely wide range of results obtained due to the uneven retorting of the rubble pieces.

Table 15. Size Analysis of Spent Shale Rubble

Screen Size mm (in)	Percent passing (in weight)
101.6 (4)	100
25.4 (1)	73.5
12.7 ($\frac{1}{2}$)	56.4
3.35 (6 Mesh)	30.9

Table 16. Specific Gravity and Water Absorption
of Spent Oil Shale

	Minimum g/cm ³ (pcf)	Maximum g/cm ³ (pcf)	Average g/cm ³ (pcf)
Dry specific gravity	1.17 (72.89)	1.98 (123.35)	1.48 (92.20)
Soaked specific gravity	1.68 (104.66)	2.23 (138.92)	1.89 (117.74)
Water absorbed (% in weight of dry material)	7.2	48	30

The dry specific gravity depended on the percentage of non-retorted shale in the sample; the less retorted the sample, the greater its specific gravity. In fact, most spent shale blocks were entirely medium grey outside (well retorted) while a large fraction inside of the blocks was black (poorly retorted). Photos 4 and 5 show a spent shale block before and after it was sawed into 5 cm (2 in) cubes. No relationship could be found between the soaked specific gravity and the degree of retorting.

3.3.4 Unconfined Compressive Strength

The unconfined compressive strength of spent shale from the six selected blocks was measured according to ASTM C170, "Compressive Strength of Natural Building Stone." Approximately 2/3 of the 5 cm (2 in) cubes cut from the blocks broke during the sawing operations or were so fragile and cleavable that they could not be tested. The minimum, maximum, and average values that appear in Table 17 are therefore purely indicative of the order of magnitude of the unconfined compressive strength that can be expected from spent shale.

As might be expected, the unconfined compressive strengths were somewhat higher when the shale bedding was oriented perpendicular to the load. However, the strengths were extremely variable, even on the samples that could be crushed. The nature of the spent material is such that close to zero unconfined compressive strength can be expected on many fully retorted blocks.

Table 17. Unconfined Compressive Strength of
Spent Oil Shale

	Minimum 10 Pa ⁵ (psi)	Maximum 10 Pa ⁵ (psi)	Average 10 Pa ⁵ (psi)
Load perpendicular to bedding	32 (460)	110 (1600)	68 (985)
Load parallel to bedding	16 (230)	103 (1490)	48 (700)

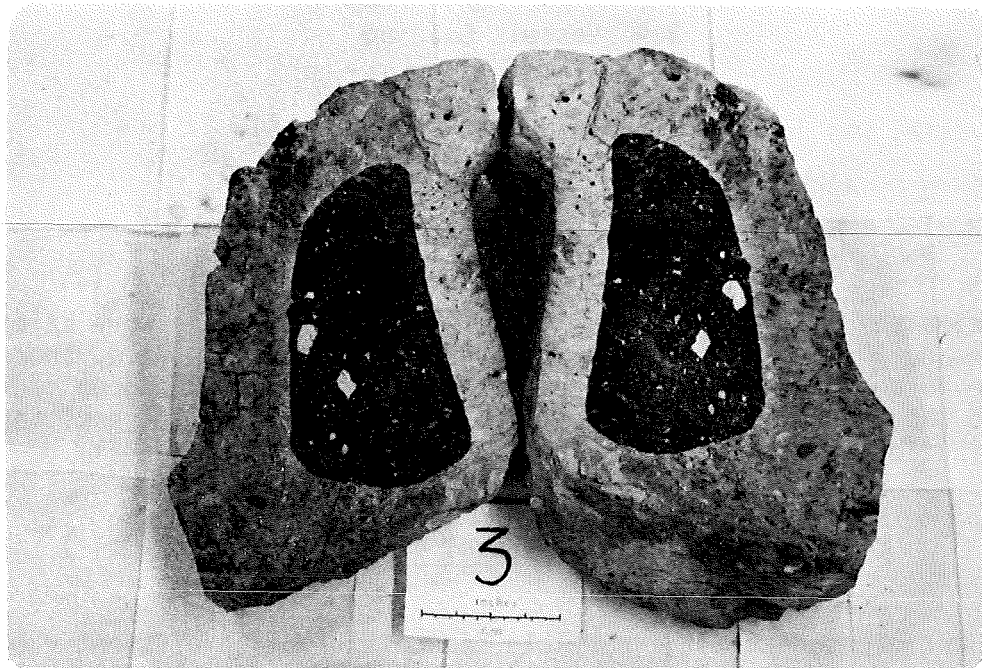


PHOTO 4. Block of Spent Oil Shale

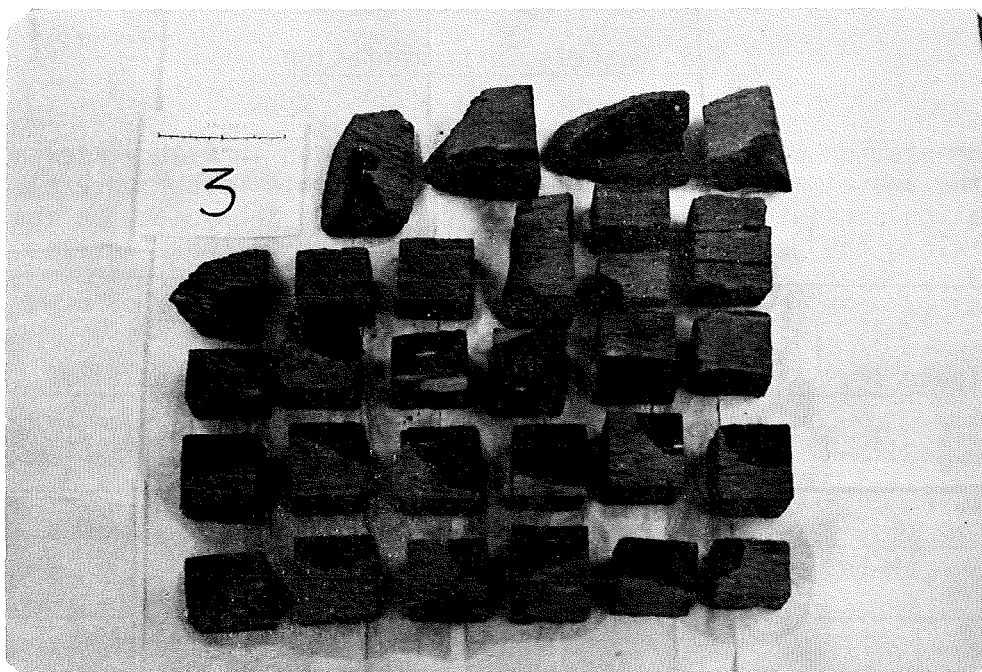


PHOTO 5. Cubic Samples Cut From the Spent Shale

3.4 Vessels Loading

Each vessel was loaded in the same manner (Photo 6).

3.4.1 Vessel #1

Approximately 5,100 kg (11,290 lb) of spent shale rubble were carefully placed and manually mixed in vessel #1. The finer particles, i.e. those passing the #6 Mesh (3.35 mm), were eliminated to simulate the open medium expected in the middle-height zone of actual MIS retorts. The resulting grain size distribution is shown on Table 18.

3.4.2 Vessel #2

It is anticipated that the particle size distribution of spent rubble might vary with depth in MIS retorts. Rubble dimensions could decrease with depth because spent shale may have a tendency to crush under its own weight. Therefore, the amount of fine particles was increased in vessel #2. Approximately 739 kg (1,630 lb) of material passing through the -6 Mesh (-3,350 μ m) was included in the rubble mix. Vessel #2 was loaded with approximately 5,294 kg (11,672 lb) of spent shale rubble. The particle size distribution is shown in Table 19.

3.4.3 Vessels Instrumentation

Instrumentation was installed at three elevations in each vessel at the same time the vessel was loaded with spent shale rubble (see Figure 6). The instrumentation consisted of pressure transducers and thermocouples installed inside and along the periphery of the vessels at each of these levels. This allowed:

Table 18. Size Analysis of Spent Shale Rubble in Vessel #1

Direct		Cummulative
Screen Product In (μ m)		Weight Passing %
	+4 (101,600)	--
-4 (101,600)	+1 (25,400)	100.0
-1 (25,400)	+ $\frac{1}{2}$ (12,700)	57.9
- $\frac{1}{2}$ (12,700)	+6M (3,350)	34.3
-6M (3,350)		--



PHOTO 6. Shale Rubble Being Loaded in Vessel

Table 19. Size Analysis of Spent Shale Rubble in Vessel #2

Direct		Cumulative
Screen Product In (μm)		Weight Passing %
	+4 (101,600)	--
-4 (101,600)	+1 (25,400)	100.0
-1 (25,400)	+ $\frac{1}{2}$ (12,700)	68.9
- $\frac{1}{2}$ (12,700)	+6M (3,350)	44.1
-6M (3,350)		14.0

- o control of the temperature inside the vessels prior to grouting and during curing of the grout, and
- o certainty that no abnormal phenomena were taking place during grouting operation such as preferential grout flow along the periphery of the vessels.

3.4.4 Boundary Conditions

The rubble mass was confined laterally by the walls of the vessels. A sand layer was placed above and below the rubble to obtain a uniform pressure distribution at the boundary for the permeability tests. A fabric filter was placed between the sand layers and the rubble to prevent migration of sand into the rubble during the permeability tests and to prevent penetration of grout into the sand during the grouting. The lids of the vessels were secured after installation of the top sand layer.

3.5 Test Procedures and Results

The test program consisted of three phases: tests before grouting to measure the porosity and permeability of the rubble mass, tests during grouting, and tests after grouting to determine the results of the grouting, i.e. the mechanical characteristics of the grouted rubble.

3.5.1 Tests Before Grouting

Two types of tests were performed on the vessels before grouting to measure rubble porosity and permeability.

3.5.1.1 Determination of Rubble Porosity Prior to Grouting. After the vessels were loaded with rubble, they were completely filled with a measured volume of water. The amount of water required to fill each vessel did not correspond to the volume of voids because some water was absorbed by the dry shale rubble.

For both vessels, the volume of water injected was greater than the amount drained. This difference between these volumes corresponds to the water volume absorbed by the dry rubble.

The final value of the porosity was taken as the average between the water injected and the amount of water drained off, i.e. 0.45 and 0.40 respectively for vessels #1 and #2. However, the uncertainty of these values is still high. The measurements are given in Table 20.

3.5.1.2 Rubble Permeability Prior to Grouting. Permeability tests were performed on each vessel according to the following procedure:

A constant flow of water was sent through the vessel from the bottom to the top and the flow rate accurately measured. Simultaneously, the head losses were measured by two transparent flexible tubings connected to the vessel on two ports three feet apart vertically. The difference of water levels between both tubings was proportional to the head losses in the three feet of rubble. The head losses measured between the two transparent tubings were so small (a few millimeters) that the results should be considered as a rough estimate.

Table 20. Rubble Porosity Prior to Grouting

	Vessel #1	Vessel #2
Bulk volume of rubble, liter (cu ft)	4,714 (166.5)	4,662 (164.6)
Volume of water injected, liter (cu ft)	2,305 (81.4)	2,282 (80.6)
Ratio with regard to bulk volume	0.489	0.489
Volume of water drained out, liter (cu ft)	1,921 (67.8)	1,460 (51.5)
Ratio with regard to bulk volume	0.407	0.317
Estimated Porosity	0.45	0.40

These various data allow the permeability coefficient K to be computed from the following equation:

$$K = \frac{Q}{GS t} \text{ cm/s}$$

where: G = gradient
 S = section area (cm²)
 t = time (s)
 Q = flow of water within the time t(cm³)

The results are given in Table 21.

3.5.2 Grouting of the Vessels

3.5.2.1 Grouting Procedure. Before starting the grouting operation, the vessels were insulated with fiberglass and steam heated until the total rubble mass and the water inside the vessel reached a temperature of 79°C (175°F). This simulated the anticipated temperature of abandoned MIS retorts. The heating was shut off during the grouting operation, turned on again after its completion and remained on during the curing period.

The grout for each vessel was mixed in 100 l (26.4 gal) batches according to the specifications determined in the grout development research. Approximately 300 to 500 l (79 to 132 gal) of grout were stored in the storage tank to provide an adequate supply to the pump feed tank, which was constantly agitated. The grout was transferred from the storage tank to the pump feed tank in 15 l (4 gal) amounts in order to monitor the volume of grout injected into each vessel. The specific gravity and viscosity of grout in the agitated storage tank were checked periodically.

Three stages of grouting were conducted for each vessel, one stage each day over a three-day period. Each stage was characterized by the following maximum grouting pressures:

- stage 1: 6.9 x 10⁵ Pa (100 psi)
- stage 2: 10.35 x 10⁵ Pa (150 psi)
- stage 3: 13.8 x 10⁵ Pa (200 psi)

The multi-stage grouting technique was selected to measure the grout intake at various maximum pressures and to compensate, during stages 2 and 3, for the eventual shrinkage or bleeding that could occur after stage 1.

Table 21. Rubble Permeability Prior to Grouting

	Vessel #1	Vessel #2
Permeability, cm/sec (10 ⁶ ft/yr)	40 to 60 (41 to 62)	8 to 10 (8.3 to 10.3)

For each stage, the ash grout was injected successively at three levels:

- at the bottom, through the four grouting ramps;
- at the middle, through two grouting ports on opposite sides of the vessel;
- at the top, through two grouting ports in opposite sides of the vessel.

Grouting continued at each port until grout intake ceased at the maximum stage pressure. The pressure was maintained for ten more minutes to make sure the grouting was completed.

3.5.2.2 Vessel #1 Grouting. The grout was mixed according to formula #49 (850 g/l of ash blend #1 and 1 ml of gluconate).

o Grouting Stage 1

Lower level - A total of 2,333 l (82.4 cu ft) of grout were injected through the lower level (four ramps). This represented 49.5% of the bulk volume of rubble. The grouting operation ran smoothly and lasted seven hours and fifteen minutes. The grout flow rate ranged between 255 and 400 l/h (9 and 14 cu ft/h) with an average of 310 l/h. The pressure was maintained close to 3.5×10^5 Pa (50 psi) most of the time and increased slowly up to 6.9×10^5 Pa (100 psi) during the last hour.

Middle and Upper Levels - Almost no grout was injected through the middle and upper levels (30 and 15 liters respectively; 1 and 0.5 cu ft), even at the maximum 6.9×10^5 Pa (100 psi) pressure.

The Marsh viscosity and specific gravity of the grout were controlled regularly. The Marsh viscosity ranged between 43 and 53 seconds and the specific gravity ranged between 1.52 and 1.54.

o Grouting Stage 2

The next day, the grouting was resumed after cleaning the grouting ports.

Lower Level - As soon as the grouting started, the pressure reached 6.2×10^5 Pa (90 psi) and then increased steadily to 10.3×10^5 Pa (150 psi) after 20 minutes. The pressure was maintained at 10.3×10^5 Pa (150 psi) for 10 more minutes. The volume of grout injected was close to 56 liters (2 cu ft).

Middle Level - For the two grouting ports, the pressure increased very quickly up to 10.3×10^5 Pa (150 psi). About 28 liters (1 cu ft) of grout were injected during the two 10-minute periods of grouting.

Upper Level - No grout intake occurred.

o Grouting Stage 3

The intake of grout during stage 3 through the three levels was equal to only 14 liters (0.5 cu ft).

During all three grouting stages, rheological characteristics of the grout were controlled regularly in the storage tank. The Marsh viscosity

fluctuated between 40 and 53 seconds and the specific gravity between 1.52 and 1.54. Grout samples were taken to test the grout's characteristics after curing for 28 days.

The remainder of grout from the 100 l (26.3 gal) batch for stage 3 was used to prepare the five samples for the direct shear test to be performed at the USBM laboratory in Spokane.

The grout was color-coded for each stage so that its travel pattern could be observed later when the grouted rubble was removed from the vessel. Fluorescein, detectable with ultraviolet light, was added to the stage 1 grout. Red and green cement dyes were used for stage 2 and 3 grouts, respectively.

3.5.2.3 Vessel #2 Grouting. The grout corresponded to grout #59 (ash blend #2, 1 l/m³ of gluconate and 15 kg/m³ of cement). It was expected that the lower rubble porosity would have a significant effect on the grouting operation.

o Grouting Stage 1

Lower Level - Grouting began fairly easily with a grout inflow rate of 425 l/h (15 cu ft/h) under a pressure as low as 1.4×10^5 Pa (20 psi). It then stabilized at about 340 l/h (12 cu ft/h) for three hours, during which the pressure increased steadily to 6.9×10^5 Pa (100 psi). The average pressure for the entire operation was about 2×10^5 Pa (30 psi). However only 1200 l (42.4 cu ft) were injected, i.e. 27.5% of the bulk volume of rubble.

Middle Level - During this step, the grouting pressure ranged between 2.75 to 3.5×10^5 Pa (40 to 50 psi) with a flow rate of 350 l/h (12 to 13 cu ft/h). Six hundred liters (21.2 cu ft) of grout were injected within an hour and a half (12.8% of the bulk volume).

Upper Level - The grout pressure increased steadily from 2 to 6.9×10^5 Pa (30 to 100 psi) with a flow rate of about 370 l/h (13 cu ft/h). Two hundred seventy liters (9.5 cu ft) were injected (5.76% of the bulk volume).

o Grouting Stages 2 and 3

No grout intake occurred during stages 2 and 3 even at the maximum pressures of 10.3 and 13.8×10^5 Pa (150 and 200 psi). This was probably caused by a fast hardening of stage 1 grout due to the vessel overheating.

The Marsh viscosity ranged from 40 to 47 seconds and the specific gravity from 1.52 to 1.53 g/cm³. Samples of grout were also collected for additional testing. For vessel #2, only the two first grouts were colored:

Stage 1: Red cement color
Stage 2: Green cement color

Table 22 summarizes the grouting data for both vessels.

During stage 1 grouting through the lower level, the percentage of grout injected into the apparent volume of rubble for each vessel was respectively: Vessel #1, 45.2%; Vessel #2, 27.5%. This means that the distance the grout penetrated the rubble in vessel #2 is smaller than it penetrated in vessel #1. This was compensated for by a larger volume of grout injected during the two last steps (middle and upper levels). This can be explained as follows:

The lateral boundary conditions in the vessels limited the possibility of lateral displacement of the rubble during grouting. Such displacements take place in the field and are necessary to allow the grout to travel from the grouting point. The first stage grouting of vessel #2 through the bottom port might have been more complete if such deformations could have occurred and if the grouting pressure were not limited (by design) to 6.9×10^5 Pa (100 psi). Such "displacement" was not necessary to grout vessel #1 due to higher porosity of its rubble.

Injecting no grout into vessel #2 during the last two stages might have led to lower quality grouting than in vessel #1. From these data, it was estimated that 5% to 10% of the theoretical volume was not injected.

It is interesting to note the differences between the volume of grout injected and the volume of water collected. The divergence in vessel #1 is 43 l (1.4 cu ft) of extra grout and 100 l (3.5 cu ft) in vessel #2. These differences are due in part to air being trapped in the rubble that was pushed upward by the grout or compressed by the pressure and also to grout that was absorbed by the rubble under the higher pressure.

For vessel #1, the volume of water collected during the grouting was larger than the volume of water injected into the dry rubble including the saturated water. This was likely caused by a phenomenon of grout filtration. When grout reached the top of the rubble at the contact of the filter cloth and filter sand, the ash was blocked against the filter while the water could flow through it until the cake of ash was thick enough to inhibit any further water filtration.

3.5.3 Tests on Samples Collected During Grouting

The samples of grout collected during grouting were tested to make sure their characteristics were in agreement with the results of the laboratory study. The results are given in Table 24. Note that samples #65 and #69 were cured at room temperature.

The unconfined compressive strength values after curing the samples for 28 days were usually higher than for the laboratory samples. This may be due to the difference in the preparation methods. In the lab, the grouts were prepared with a small stirrer in five liter batches whereas the grouts for the vessels were prepared with a powerful cement mixer. Some errors in the weighing of grout components may have occurred also since the scale used to prepare the vessel grouts was, of course, less accurate than the laboratory balance.

Table 22. Grouting Data

Grout Injected	Volume of Grout Injected, liter (cu ft)				Volume of Grout to Volume of Voids, %	
	Vessel #1		Vessel #2		Vessel #1	Vessel #2
Stage 1 (100 psi)						
Lower level	2,333	(82.4)	1,200	(45.2)	49.5	27.5
Middle level	28	(1.0)	600	(21.1)	0.6	12.8
Upper level	14	(0.5)	270	(6.8)	0.3	5.7
TOTAL	2,375	(83.9)	2,070	(73.1)	50.4	46.0
Stage 2 (150 psi)						
Lower level	56	(2.0)	0	(0)	1.2	0
Middle level	28	(1.0)	0	(0)	0.6	0
Upper level	0	(0)	0	(0)	0.0	0
TOTAL	84	(3.0)	0	(0)	1.8	0
Stage 3 (200 psi)						
3 levels combined	14	(0.5)	0	(0)	0.3	0
TOTAL FOR 3 STAGES	2,473	(87.4)	2,070	(73.1)	52.5	46.0
Water collected	2,430	(85.8)	1,970	(69.6)	51.5	42.3

3.5.2.4 Grouting Test Analysis. Results of the grouting tests were very encouraging. They are summarized in Table 23.

Table 23. Comparison of Volumes Involved in the Grouting Process

	Vessel #1		Vessel #2	
	liter	cu ft	liter	cu ft
Volumes of water,				
-Injected into dry rubble	2,350	81.4	2,282	80.6
-Drained out of saturated rubble	1,921	67.8	1,460	51.5
-Collected during the grouting	2,430	85.8	1,970	69.6
Volume of grout injected	2,473	87.4	2,070	73.1

Table 24. Results of Tests on Grouts Injected Into Both Vessels

Sample #	62	63	64	65	66	67	68	69
Origin	Vessel 1 Phase 1	Vessel 1 Phase 1	Vessel 1 Phase 3	Vessel 1 Phase 3	Vessel 2 Phase 1	Vessel 2 Phase 1	Vessel 2 Phase 3	Vessel 2 Phase 3
Type of SSA	Blend #1	Blend #1	Blend #1	Blend #1	Blend #2	Blend #2	Blend #2	Blend #2
Amount, g/l	850	850	850	850	850	850	850	850
Additive 1: Type	Gluconate	Gluconate	Gluconate	Gluconate	Gluconate	Gluconate	Gluconate	Gluconate
Amount, ml/l	1	1	1	1	1	1	1	1
Additive 2: Type	-	-	-	-	Cement Portland 2	Cement Portland 2	Cement Portland 2	Cement Portland 2
Amount, g/l	-	-	-	-	15	15	15	15
Color	Fluorescein	Fluorescein	Green	Green	Red	Red	-	-
Marsh viscosity, sec.	53	45	40	40	40	47	40	40
Specific gravity, g/cm ³	1.54	1.53	1.52	1.52	1.52	1.53	1.52	1.52
Unconfined compressive strength, 10 ⁵ Pa (psi): 7 days	- (-)	- (-)	- (-)	- (-)	5.18 (75.2)	6.28 (91.1)	6.16 (89.4)	2.39 (34.6)
14 days	- (-)	- (-)	- (-)	- (-)	7.94 (115.1)	7.42 (107.6)	6.88 (99.7)	3.96 (57.4)
28 days	8.19 (118.8)	6.60 (95.7)	6.76 (98.1)	6.69 (97.0)	8.32 (120.7)	8.54 (123.8)	7.92 (114.9)	6.40 (92.8)
Curing temperature, °F	170	170	170	70	170	170	170	70

3.5.4 Curing Period

After completion of all stages of grouting, the grouted mass was cured for 28 days. The curing temperatures were continuously measured by the thermocouples and recorded. They fluctuated between 79 and 85°C (175 and 185°F).

The heat was turned off after 28 days. The insulation was removed and the vessels were allowed to cool for three days prior to the permeability tests.

3.5.5 Permeability Tests

3.5.5.1 Test Procedure. Permeability tests were performed on the entire grouted mass of rubble in each vessel. Tap water was directly connected to the bottom ports of each vessel and circulated to the top. The tap water pressure was fairly uniform; any variation was within the range of 8.6 to 9.3 x 10⁵ Pa (125 to 135 psi). The corresponding hydraulic gradient was 35.5.

The outflowing water was collected from the top ports through flexible tubings into containers equipped with lids to prevent evaporation. The volume of water that percolated through each vessel was measured once or twice a day.

The water pressures at various levels in the vessel were measured during the entire test by means of the six pressure transducers mounted in each vessel (three along the wall and three in the middle). These data were recorded continuously on a multi-channel recorder. The variations of pressure versus the elevation in the vessel gave the gradient at various levels.

3.5.5.2 Permeability Test Results - Vessel #1. Unfortunately, the test had to be stopped for thirteen days to perform the preliminary tests and grouting of vessel #2. Figure 8 gives the variation of permeability versus time for vessel #1.

The curve shows a regular shape despite the 13-day interruption. The permeability decreased from 25 x 10⁻⁷ cm/s to 7 x 10⁻⁷ cm/s after 40 days and then stabilized around this value until the end of the test. The permeability of the grouted rubble in vessel #1 was only slightly higher than the permeability of the same grout (sample #49) in the lab test (7 versus 5 x 10⁻⁷ cm/s).

The six pressure transducers installed in each vessel (three along the wall and three in the middle) allowed the pressure at various levels to be measured throughout the permeability test. The data enabled gradients and hence permeabilities to be evaluated at various levels in the vessel. Figure 9 shows the locations of the six pressure transducers and the various gradients and permeabilities computed from the pressure data after testing for ten days. Large variations existed in the permeability values, depending on locations. With an average value of 36.5, the gradient varies from 7.6 in the middle to 175 in the top layer, whereas the permeability ranges from 50 to 2 x 10⁻⁷ cm/s for an average of 10 x 10⁻⁷ cm/s.

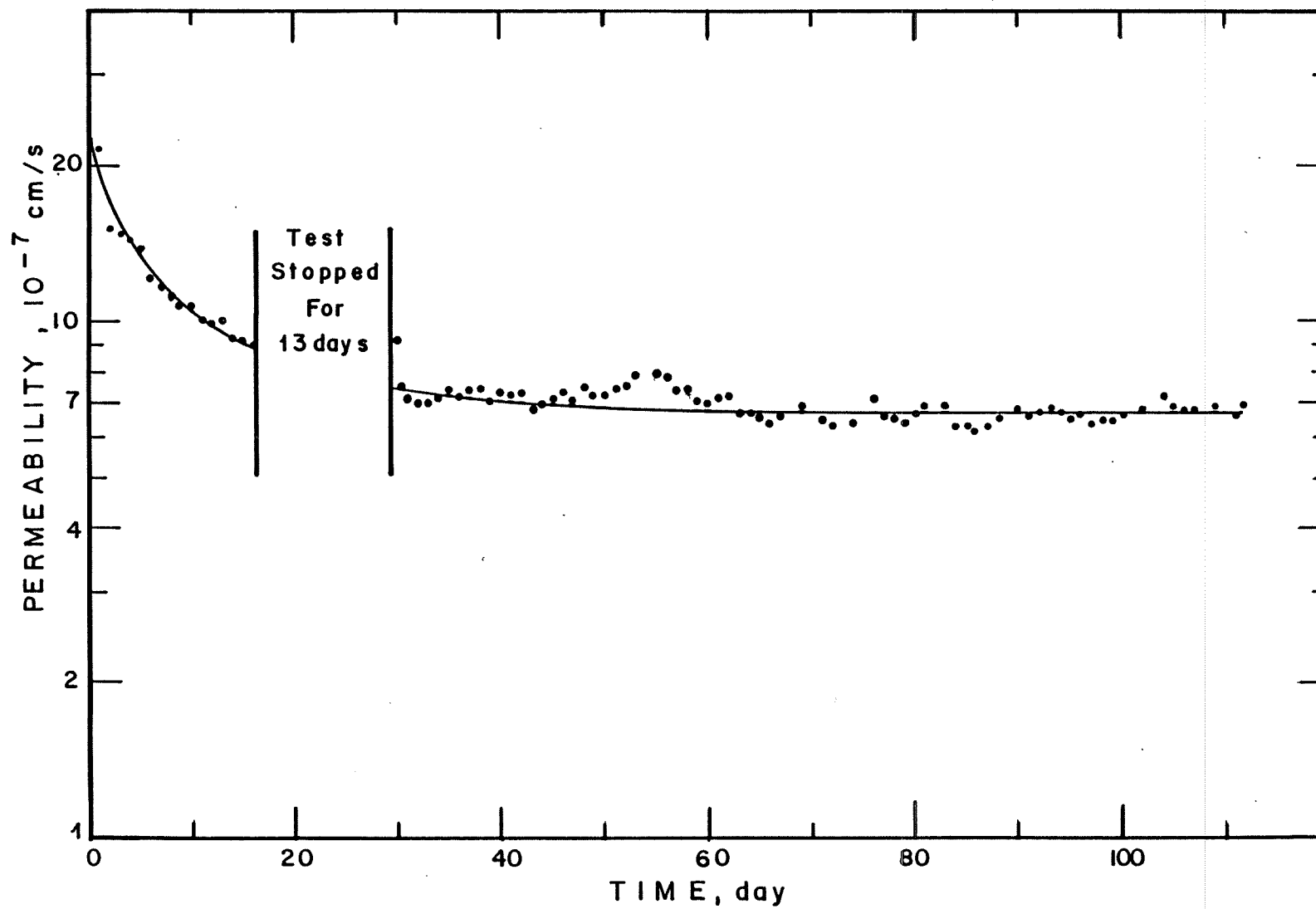


FIGURE 8. - Vessel # 1 . Permeability vs time

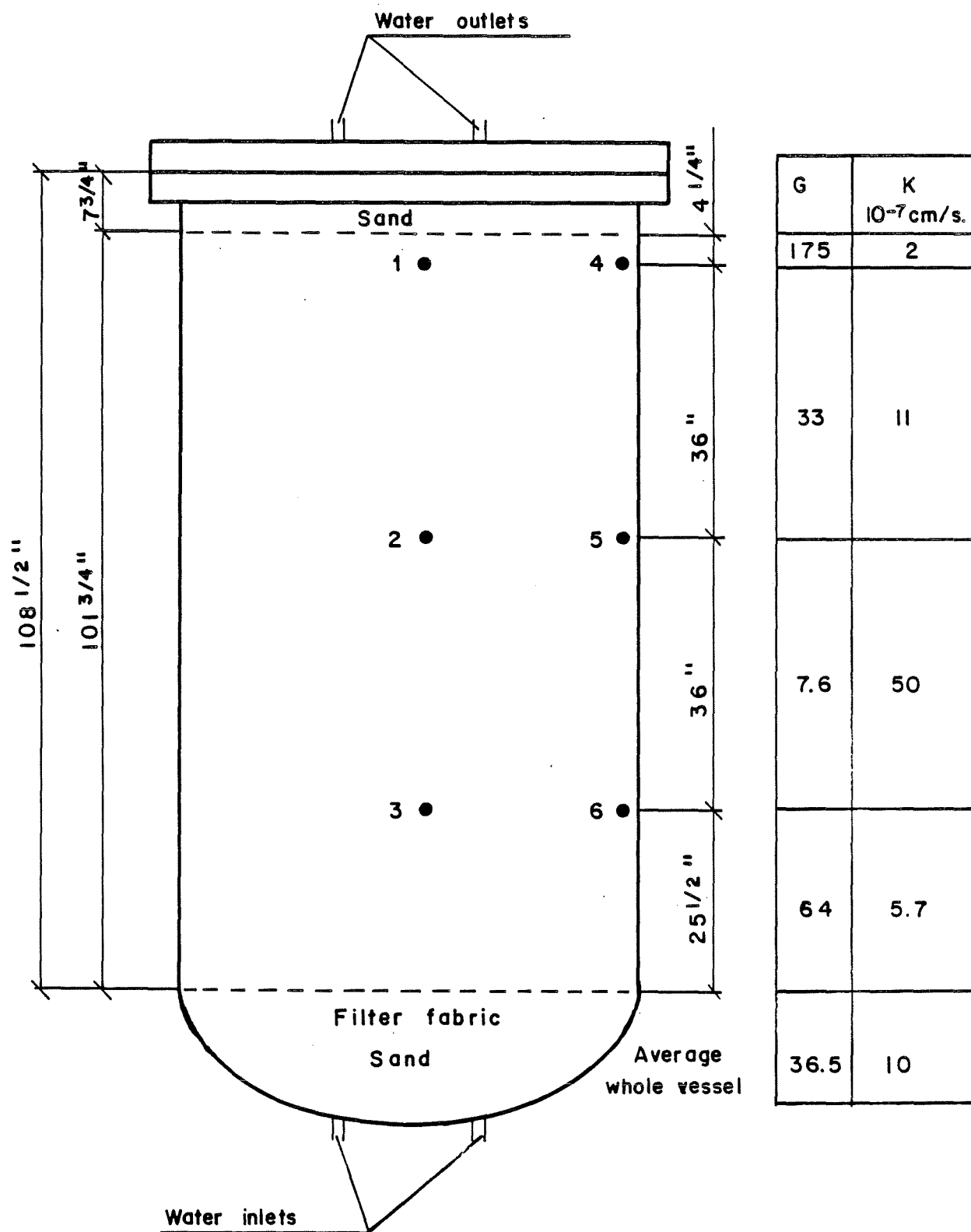


FIGURE 9 - Vessel #1. Pressure transducer location
Gradient and permeability distribution

Hence, the grouting appears to have been more effective at the top and bottom grouting ports of the vessel. This can be explained by the fact that the grouting of rubble was most efficient at the bottom where the grout was injected. As for the top zone, this may be the result of filtration process already discussed.

3.5.5.3 Permeability Test Results - Vessel #2. The evaluation of the overall permeability coefficient with time is shown in Figure 10. The test was stopped after 55 days. During this period, there was a continuous decrease of the permeability coefficient from 9.5×10^{-6} cm/s to 4×10^{-6} cm/s. This permeability is higher than that obtained for the laboratory mix (4×10^{-6} versus 5×10^{-7} cm/s measured on sample #51). This confirms that vessel #2 was incompletely grouted.

The variations of gradient and permeability after 13 days of testing, shown on Figure 11, are not as large as those for vessel #1. The grout intake during stage 1 from the middle and upper ports was larger for vessel #2 than for vessel #1. This resulted in a more "homogenous" grouting.

3.5.6 Coring and Pressuremeter Tests

After termination of the permeability tests, the lid, the upper layer of sand, and the filter fabric of each vessel were removed. Vertical corings were performed to the total depth of the vessels with a diamond bit concrete coring machine equipped with a 76 or 60 mm (3 or 2-3/8 in) diameter, 300 mm (one foot) working length, thin-walled sampler bit. The corings were intended to:

- provide vertical holes in the grouted rubble for pressuremeter tests; and
- obtain, if possible, undisturbed samples of the grouted rubble.

The pressuremeter tests were performed in each corehole with a Menard pressuremeter. In this test, a probe carrying an inflatable cell is set in the corehole. The variations in volume of the cell are measured for various cell pressures. They correspond to the deformations of the rubble mass. A pressuremetric modulus is then computed. These tests defined the deformability and pressuremetric modulus of the grouted mass. Three tests were carried out in each hole at three depths, three feet apart. Each test was performed as soon as a three-foot deep coring was made to limit the risk of decompression with time. Photo 7 shows a typical core.

3.5.6.1 Pressuremeter Test Results. Detailed results of the pressuremeter tests are presented in Appendix C. They indicate that the grouted rubble has considerable strength under confined conditions. It will sustain relatively heavy loading without significant deformation and has a shear strength of 12 t/ft² ($c=7$ t/ft², $\phi=20^\circ$). Table 25 is a summary of the test results.

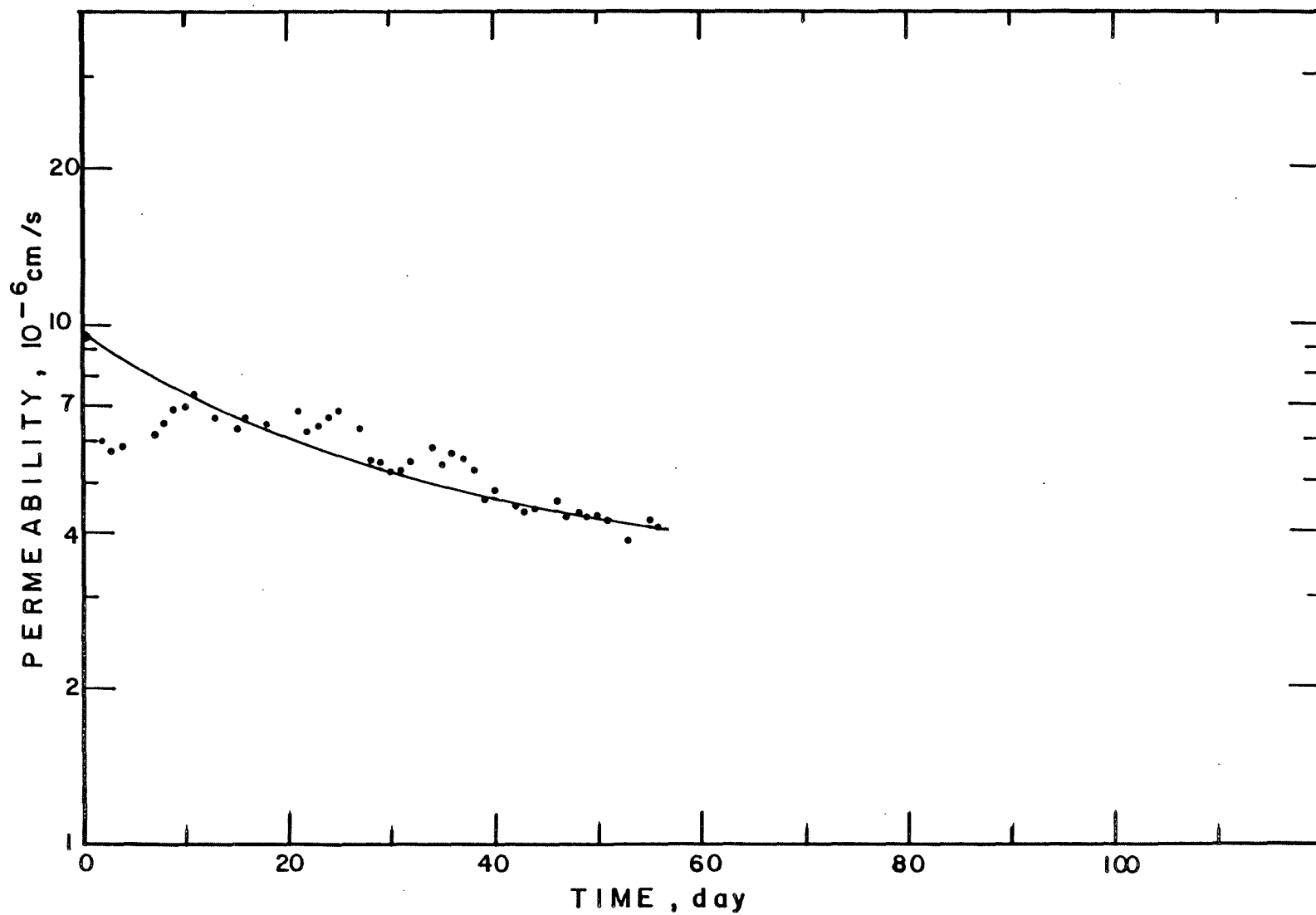


FIGURE 10.- Vessel # 2. Permeability vs time

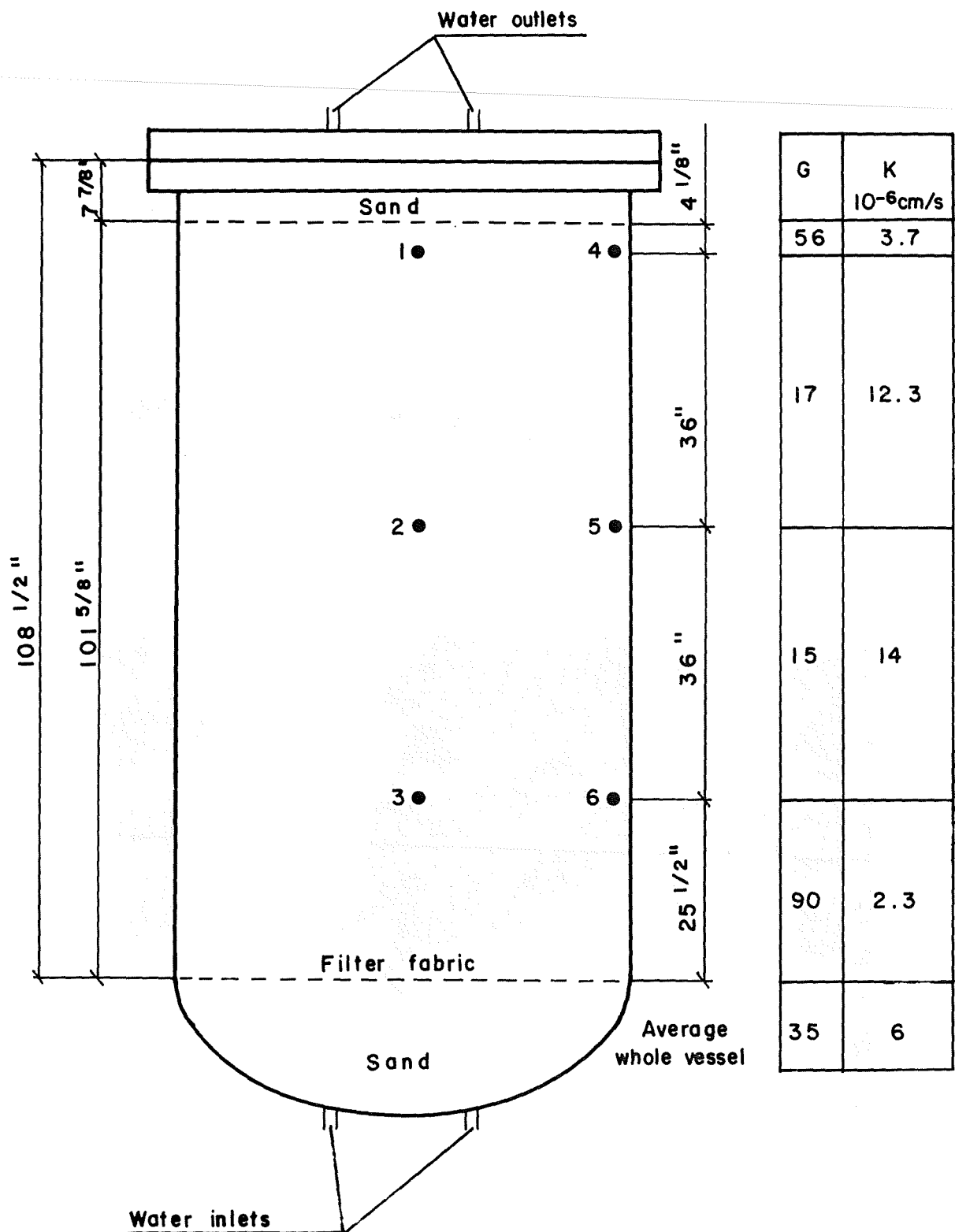


FIGURE 11 . — Vessel # 2 — Pressure transducers location .
Gradient and permeability distribution

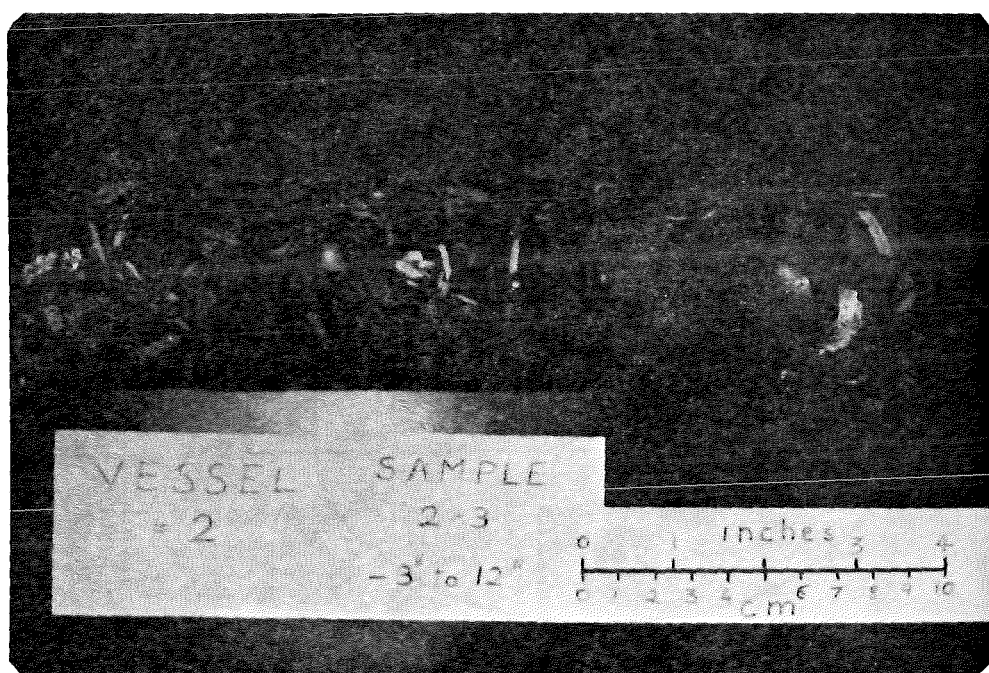


PHOTO 7. Core Sample

Table 25. Pressuremeter Tests Results

	Initial Modulus					
	Top		Middle		Bottom	
	psi	10 ⁵ Pa	psi	10 ⁵ Pa	psi	10 ⁵ Pa
Vessel #1 - corehole #2	20,126	1,388	11,455	756	17,893	1182
Vessel #2 - corehole #1	2,247	148	1,000	67	580	38
corehole #3	4,031	266	--	--	--	--

The results for vessel #1 showed that the center part of the vessel was not as extensively grouted as its upper and lower zones. Results for vessel #2 confirmed that the grouting was incomplete.

The "hypothetical" Mohr Coulomb failure envelopes of the grouted rubble are shown on Figure 12. They were derived from the pressuremeter tests.

3.5.7 Rubble Removal and Visual Observations

A jack hammer was required to break up the grouted rubble mass in each vessel. It was then removed in layers about one foot thick. The exposed surface of rubble in each layer was carefully cleaned with a brush and compressed air so that any visible grouting flaws could be detected. The distribution of the various colored grouts could then also be observed.

In general, the grouted mass in both vessels was fairly fragile; i.e., the rubble was not strongly bound together, and pieces of it could be easily split from the grout matrix. However, grout penetration into the rubble was good, especially in vessel #1.

3.5.7.1 Vessel #1. Because of the different cement dyes, the distribution of grouts from the various stages of injection were easily observed. The first stage grout represented 96% of the total volume of grout. The second stage grout (the volume of which was about 3.5% of the total volume) was found mainly along the wall of the vessel. The thickness of this grout layer varied from one to ten millimeters (1/32" to 1/2") and covered a quarter to a third of the wall surface. The third stage grout was present as only a very thin layer on the wall near a grouting port.

This demonstrated the beneficial action of a second or third grouting stage with a higher grouting pressure. The mass of rubble with a grout already set was compacted and consolidated by the newly injected grout (Photo 8). No cracks in the grouted rubble mass were observed.

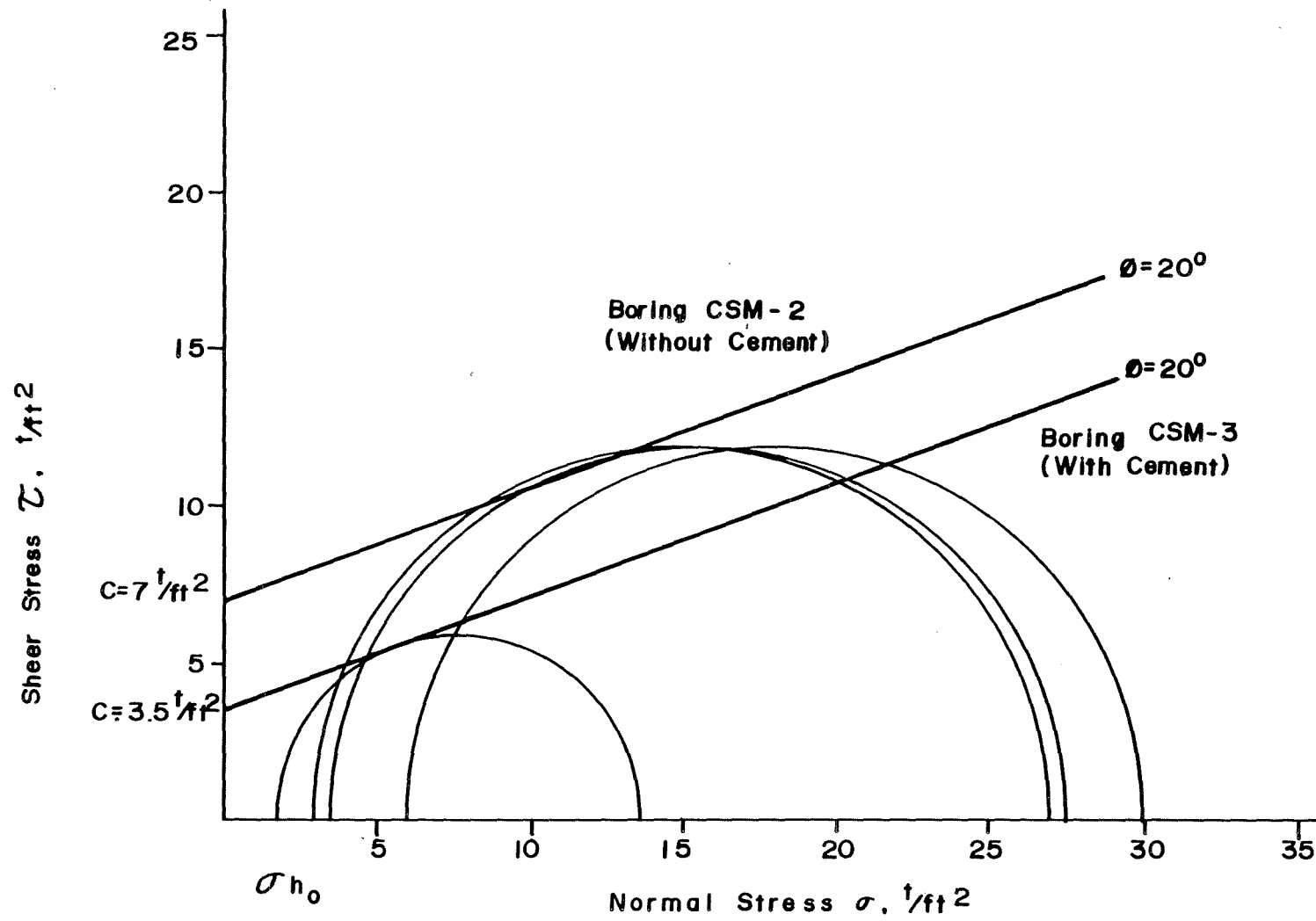


Figure 12. - Hypothetical Mohr Coulomb Failure Envelopes for grouted Rubble

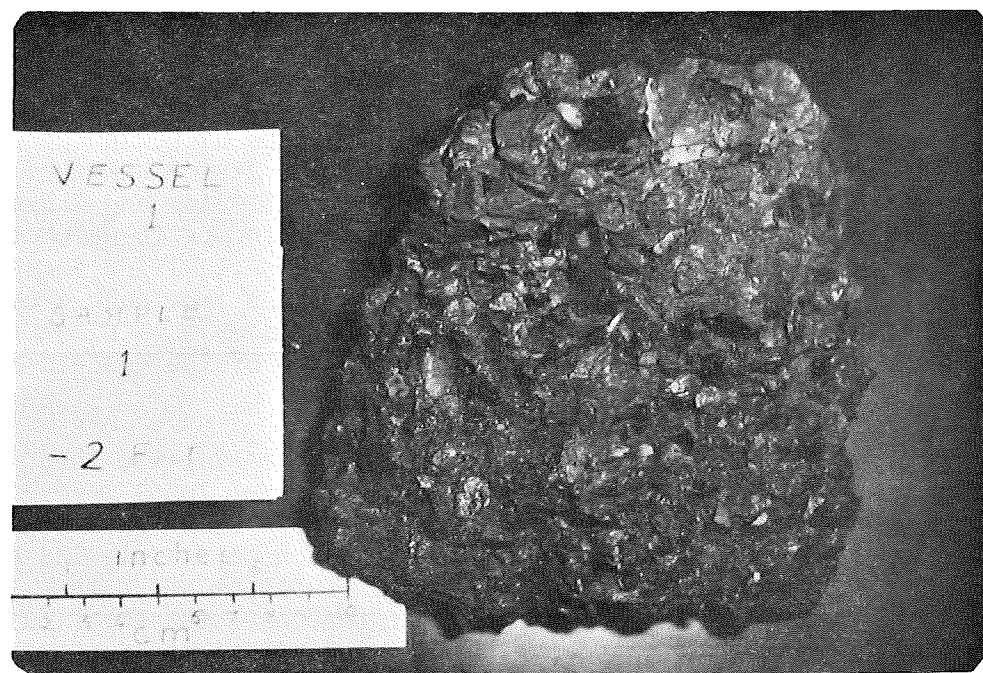
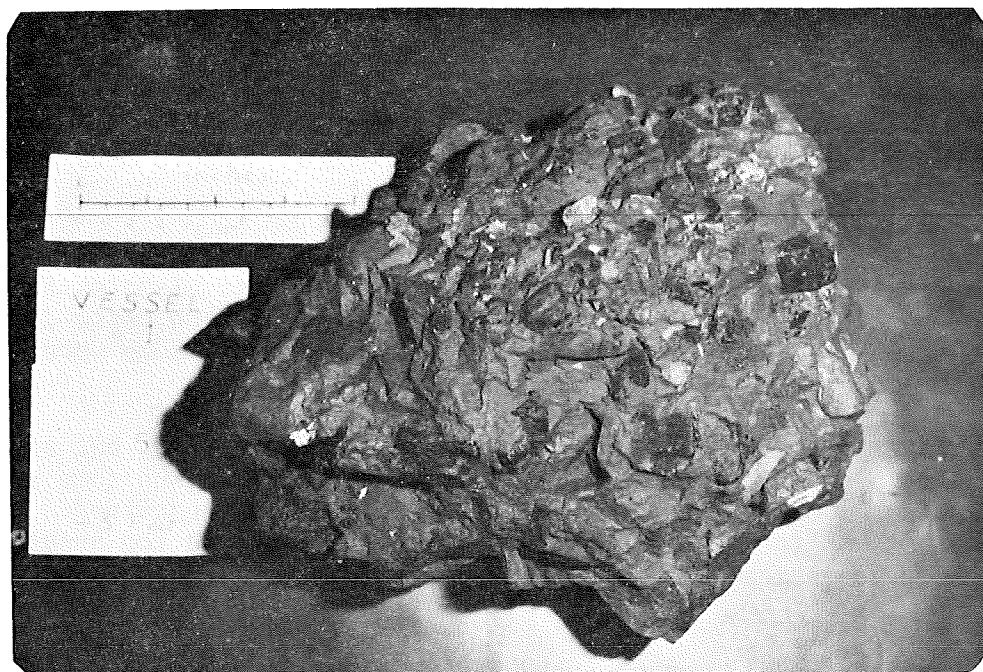


PHOTO 8. Samples of Grouted Rubble (Vessel #1)

3.5.7.2 Vessel #2. The distribution of the first stage grout, which was the only one injected, could be observed very clearly. No cracks were found, but some small pockets of ungrouted fine material were found. All these pockets were located where manual blending inadequately mixed the various rubble sizes. This resulted in pockets of fine rubble that were difficult to grout (Photo 9). Except for these small pockets, the overall rubble mass was well grouted.

3.5.7.3 Observations on Samples Removed From the Vessels. Samples from both vessels were obtained from the top, middle and bottom of each vessel, stored in separate marked barrels and protected from dehydration. Some were brought to the lab for more detailed examination with a magnifier.

No cracks or flaws were observed on the vessel #1 samples; but some zones on vessel #2 samples were found insufficiently grouted, leaving some small voids between small or fine rubble. Attempts to perform unconfined compression tests on these samples were unsuccessful because the grouted rubble was so fragile that it was not possible to cut well-shaped sample cubes.

Bulk specific gravity was measured on six samples from each vessel. The following are their average values.

Vessel #1: 1.87 g/cm³ (116.7 lb/cu ft)
Vessel #2: 1.91 g/cm³ (119.3 lb/cu ft)

3.5.7.4 Direct Shear Test Results on Large Samples. At the completion of the vessel grouting experiments, samples of rubble and grout were mixed and cast for direct shear testing at the Bureau of Mines, Spokane Research Center. To facilitate handling the samples, outer molds were made from a commercial tubular concrete form product that were 14 inches in diameter and 7-5/8 inches high. Two split-ring inserts were cut from the form product to match the upper and lower sections of the shear box. A third insert, 1-1/2 inches high covered the shear plane gap. Five samples were prepared from each grouted rubble used in the test vessels. In addition, a bulk sample of plain rubble was prepared for shear testing to compare the strength of the grouted and ungrouted rubble.

The test procedure was to remove the outer mold and fit the shear box over the sample now contained in the split rings. Normal loads were applied to consolidate the sample, the shear box raised and the shear gap ring removed. A slow shear load applied at about .015 inch per minute was applied until a peak shear stress was observed. Results of direct shear test series are shown in Figure 12-A. At the completion of the test, the shear failure plane was examined and photographed. A typical failure plane is shown in Photo 10. The large aggregate rubble particles are cleaved on the shear plane indicating good bonding and bedding of the grout. Samples were weighed and dried after completion of the shear test to obtain moisture content and density of the samples. Average sample moisture content on a dry solids by weight basis was 18.5 percent while dry density was 97.7 lbs per cu ft. Much of the contained moisture probably reflects hydrated water contained in the shale minerals rather than free pore water.

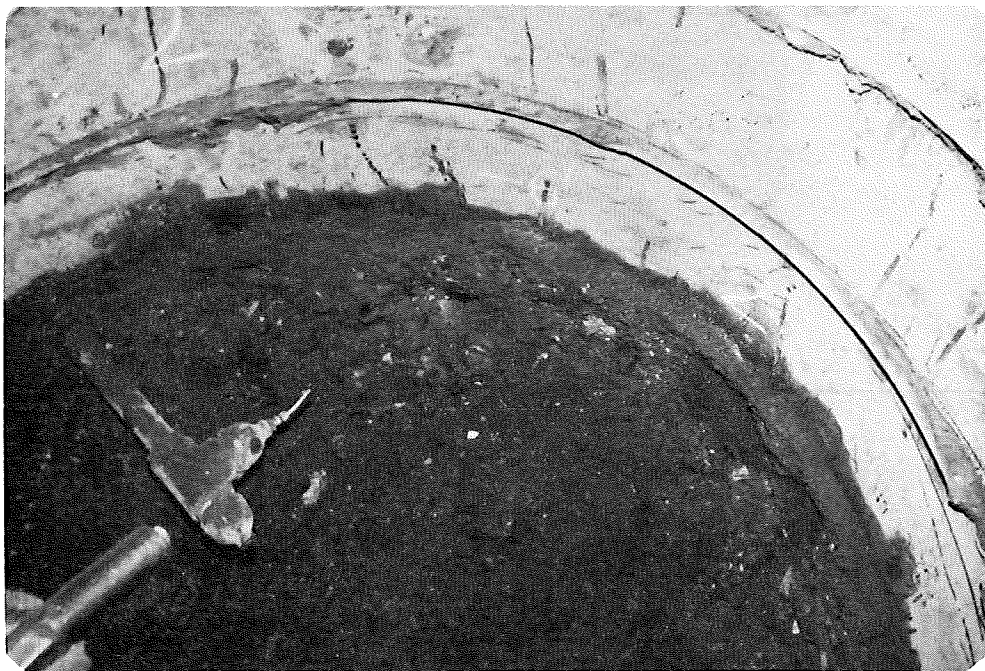


PHOTO 9. Visual Observation (Vessel #2)

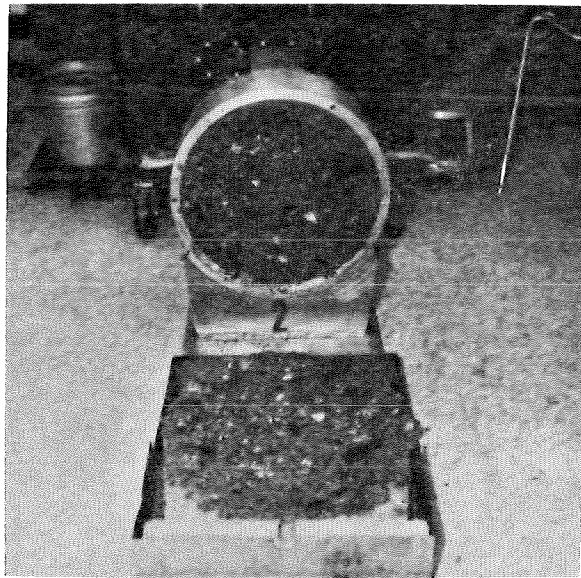
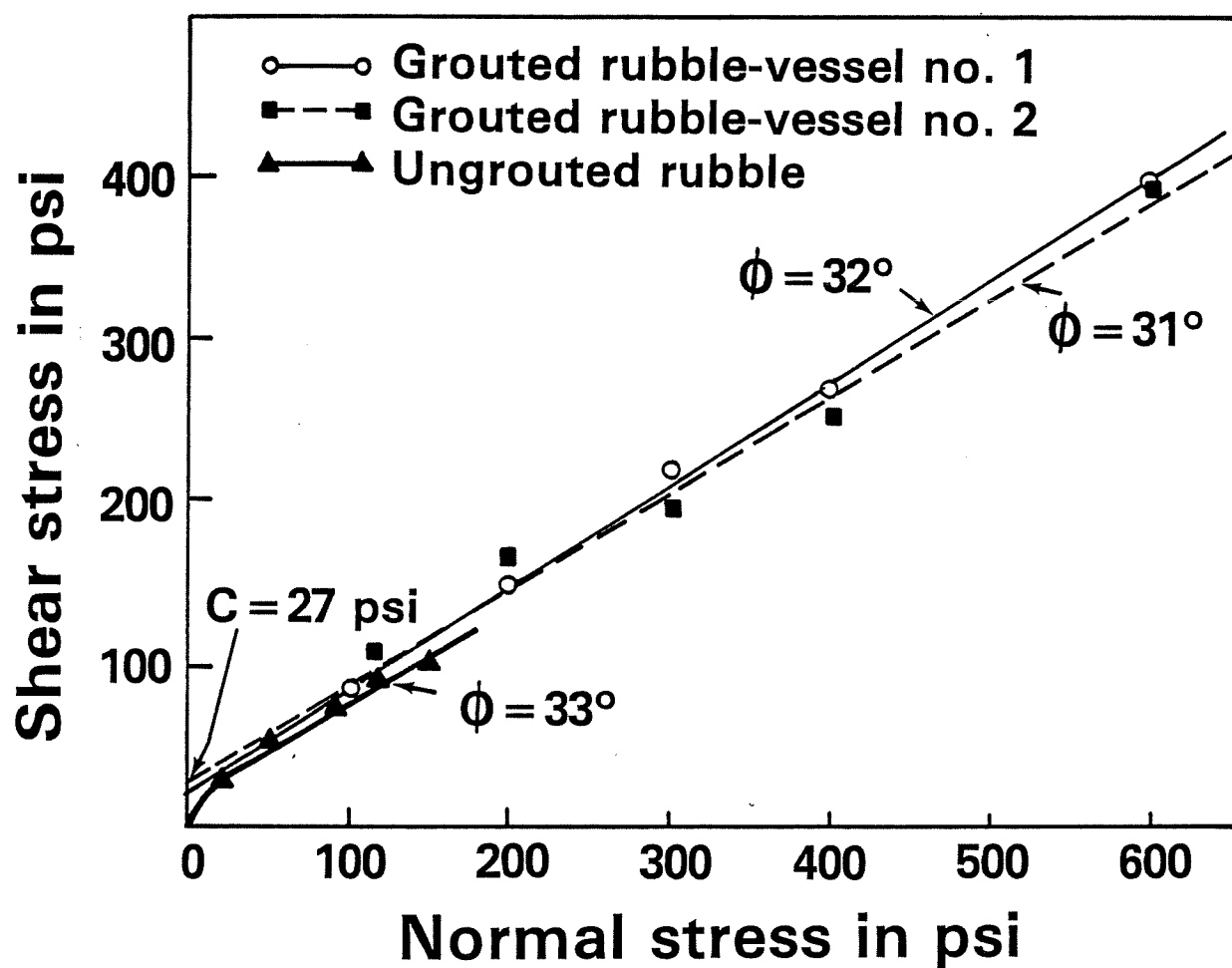


PHOTO 10. Typical Shear Surface of Grouted Rubble

Figure 12-A

Direct shear tests results



The direct shear tests compared favorably with the results of the unconfined compressive strength tests and triaxial tests performed on small samples of the ash grout.

4. TRENCH TEST

Previous stages of this research had demonstrated that a grout can be formulated using spent ash as a major component and that if the grout is successfully injected into a pilot scale retort, the grouted rubble will display the desired mechanical properties. To design a successful grouting system for abandoned MIS retorts, the radius of the grout's penetration into rubble had to be determined. This was the main objective of the trench test. Secondary goals were to check the operational performance of the grouting technique and the physical properties of the grouted rubble.

The trench test was performed at Tract C-a. It consisted of opening a trench, filling the bottom of the trench with rubble, burying the rubble, grouting the rubble through several injection pipes, and then, after a curing period, re-excavating the trench and visually inspecting the grout penetration.

4.1 Trench Construction

The trench was excavated in silty alluvium soil with a backhoe. Figure 13 shows the planned sections and dimensions of the trench. A ramp was built at one end to allow access to the bottom of the trench.

Raw shale was used as simulated rubble because no retorted oil shale was available on site. This shale was derived from experimental MIS retort development at Tract C-a, so its size distribution was representative of an actual small-scale MIS retort. This raw shale had characteristics different from those of spent shale, especially in the areas of strength and water absorption. However, it was believed that these differences would not significantly affect the radius of grout penetration.

A layer of rubble 1.5 m (5 ft) thick was laid at the bottom of the trench by means of a backhoe. This operation was done carefully to prevent excessive breakage of the rubble due to handling. The rubble included a large fraction of fine material.

To prevent the grout from flowing into the soil surrounding the trench, three liners were laid in the trench and then folded over the top of the rubble after it was set in place (Figure 13). The first two layers were polyethylene films 10 mils (0.25 mm) thick, to establish impermeability. The third liner was a non-woven polypropylene fabric to prevent the polyethylene film from being punctured by sharp pieces of rubble (Photo 11). Both polyethylene and polypropylene are chemically inert.

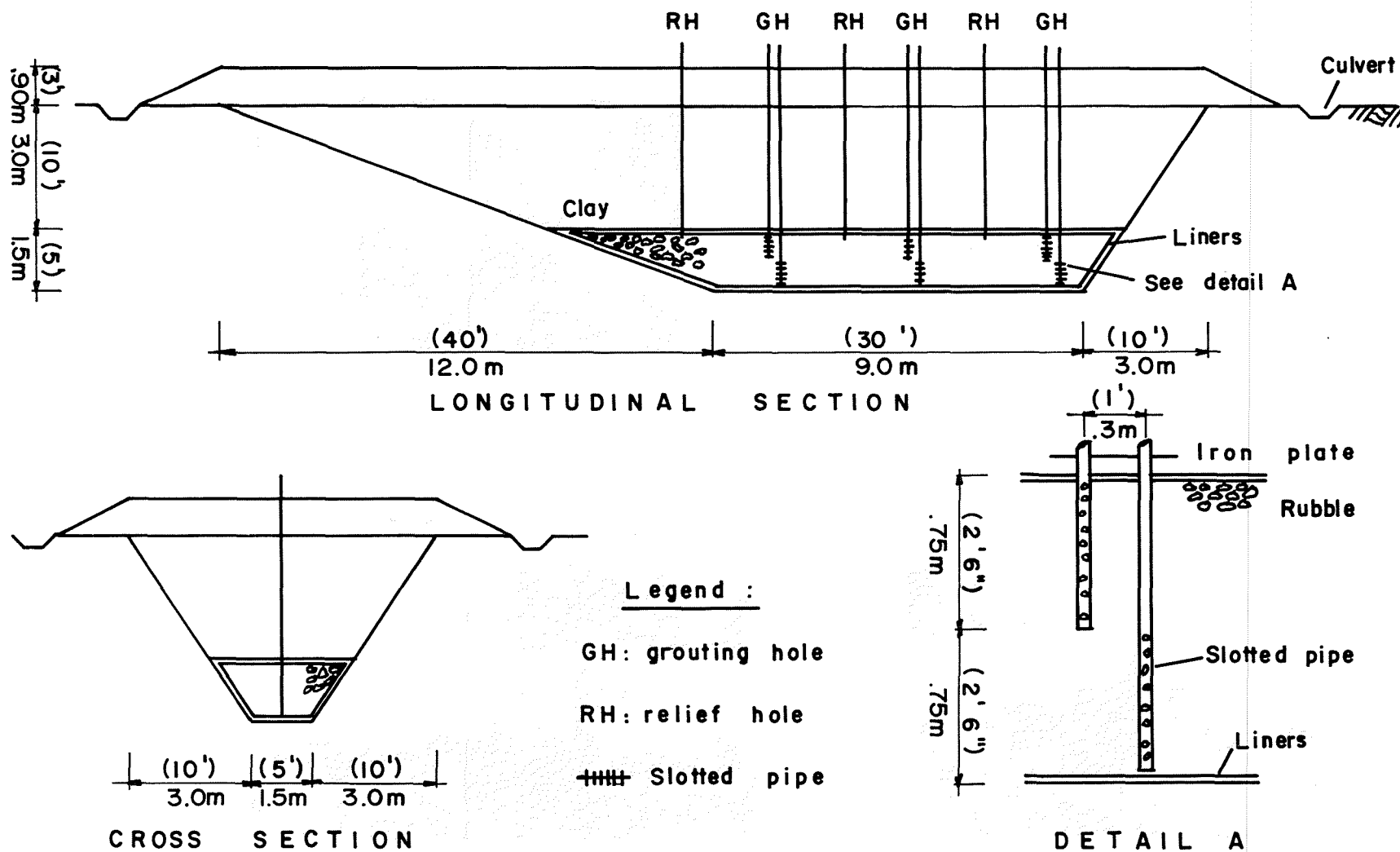


FIGURE 13 .- Schematic sketch of the trench

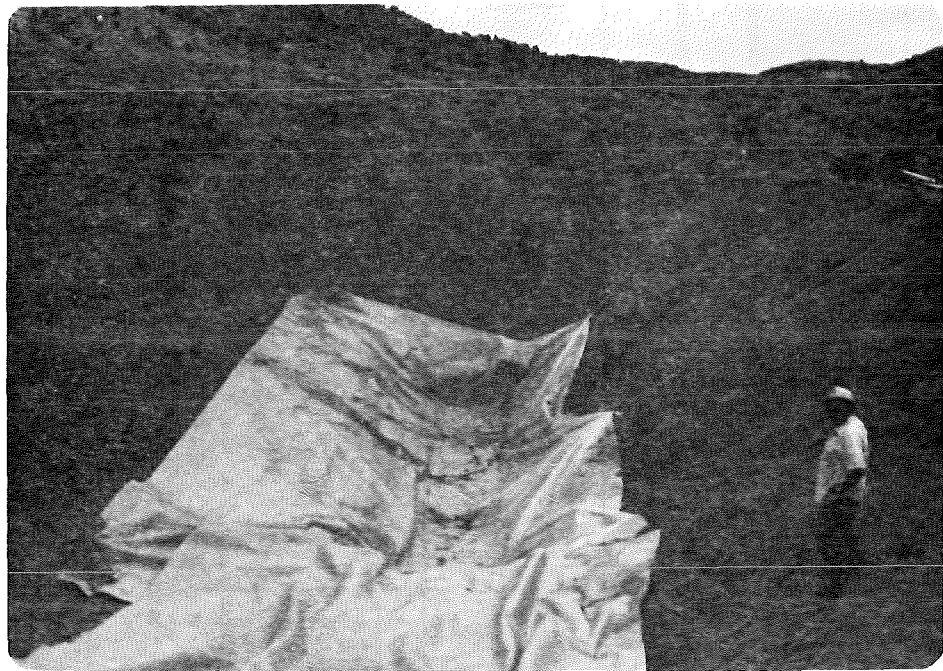
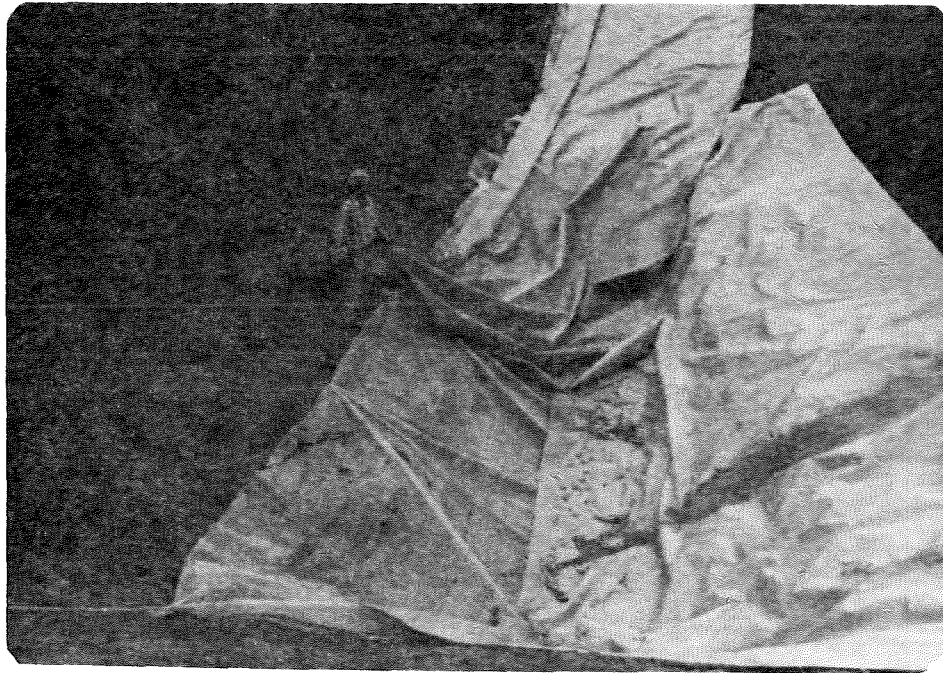


PHOTO 11. Placing the Liners in Position in the Trench

Two layers of clay each 0.3 m (1 ft) were then laid over the enclosed rubble and thoroughly compacted by means of a hand-driven vibrating roller (3 tons) to seal the layer of rubble. All the soil previously extracted from the trench was then laid over the two clay layers and compacted. Four additional 35-ton loads of soil were dumped over the trench to provide a blanket one-foot thick above the original ground level.

4.2 Grouting System

The grouting system is shown in Figure 13 and Photo 12. It consisted of 3 pairs of steel grouting pipes, 50 mm (2 in) in diameter, 3.3 m (11 ft) apart, and 3 steel relief pipes, 30 mm (1.2 in) in diameter.

The disposition of the grouting pipes was selected to permit two stage grouting from each point, as the allowable pressure was very low. The slotted parts of the pipes were surrounded with a thin plastic screen to prevent plugging by rubble particles. The pipes were capped at the bottom and equipped at the top with a valve. For each pair, the pipes were connected 30 cm (1 ft) apart, by means of a steel plate that extended about 15 cm (6 in) around the pipes and was located in the clay about 15 cm (6 in) above the rubble. This plate was intended to eliminate leakage along the pipes. As an additional precaution to seal the pipes above the rubble, temporary casings, 100 mm (4 in) in diameter and 300 cm (10 ft) long, were slipped over each pipe during its installation. After completion of the backfilling, the casings were removed and the annular space was cemented.

The relief pipes were installed between the grouting points as shown in Figure 13. They were slotted on the lower 15 cm (6 in), which corresponded to the upper 15 cm (6 in) of the rubble layer.

An open culvert was dug along the perimeter of the soil blanket to collect any waterspill (or grout) for transport to the waste water facilities.

4.3 Tests on Materials

4.3.1 Tests on Simulated Rubble

The bulk density and the porosity of the raw shale were estimated with the use of a 60 cm (2 ft) rigid steel cubic box (266 l or 8 cu ft). The box was first filled with uncompacted raw shale and weighed. Then the box was filled with water until no air bubbles escaped from the rubble and weighed again. These data allowed computation of both the dry and saturated bulk densities of the rubble as well as its porosity. Results are listed in Table 26.

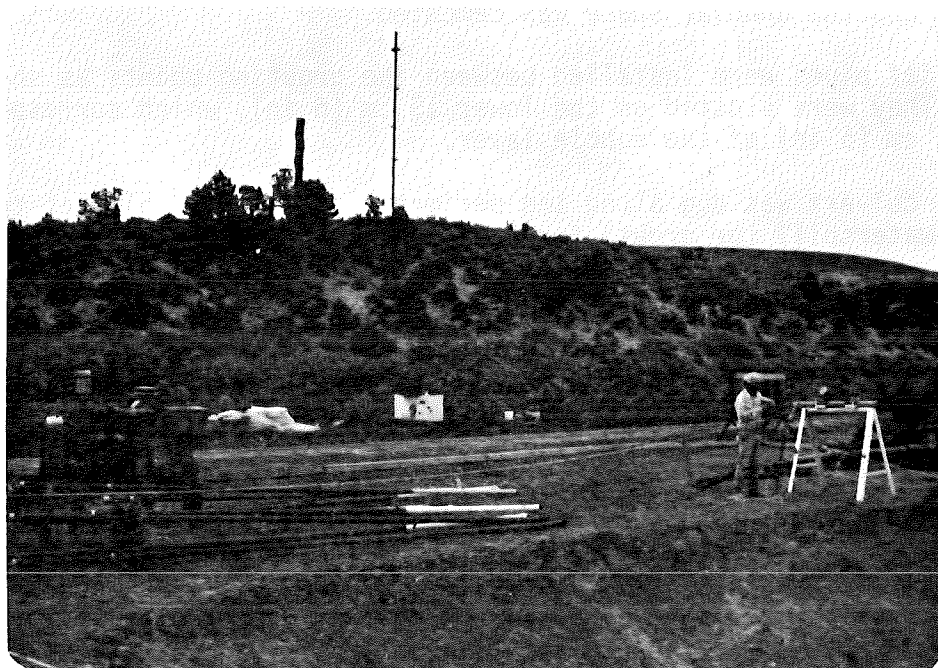
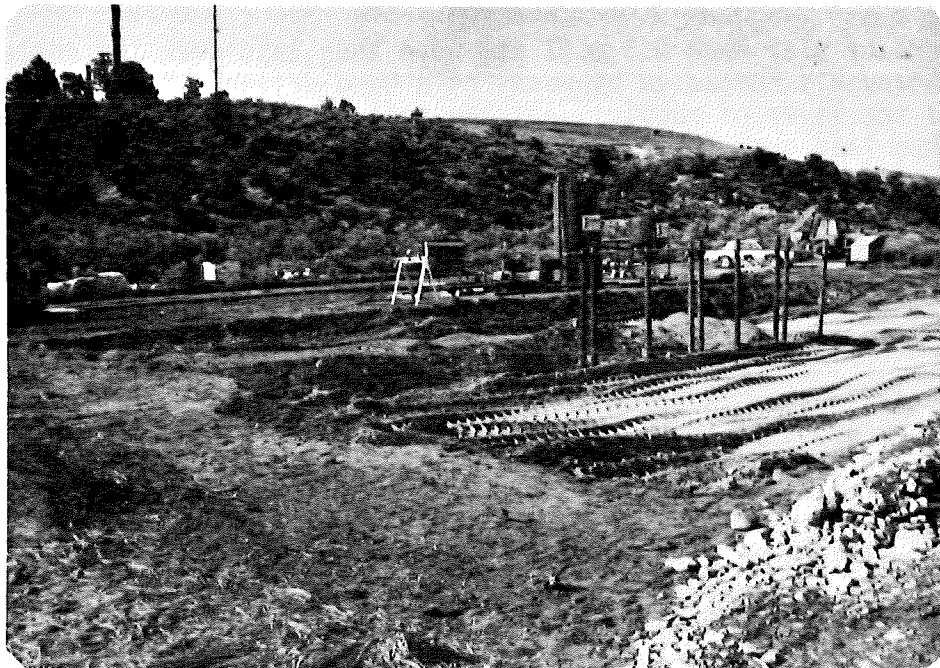


PHOTO 12. Instrumentation in the Trench

Table 26. Simulated Rubble Characteristics

Test	1	2	Average
Dry specific gravity			
kg/dm ³	1.566	1.588	1.562
lb/cu ft	97.76	97.26	97.51
Saturated specific gravity			
kg/dm ³	1.852	1.856	1.854
lb/cu ft	115.61	115.86	115.74
Porosity	28.6	29.8	29.2

NOTE: In the computation of porosity, the water absorbed by the raw shale was not taken into account for it was not known. However, it should be rather low for unspent shale. The porosity after saturation most likely ranges between 0.26 and 0.28.

4.3.2 Tests on Trench Blend

The grout formula selected after the lab study and the vessel tests was as follows:

Water	685 l	138.5 gal
Ash	850 kg	1433 lbs
Gluconate	<u>1 l</u>	<u>33.6 fl oz</u>
Total Volume	1 m ³	1 cu yd

and its main characteristics were:

Marsh viscosity		50 s
Bleeding		2 to 4%
Setting time	170°F	2 h
	70°F	4 h
Unconfined compressive strength (28 days at 170°F)		110 psi

The estimated volume of grout required to completely grout the trench rubble was 18 m³ (23.5 cu yd). A large quantity of ash (about 20 tons) was needed for this test. Upon receipt of the available ash, it was noted that the grain size distribution was substantially larger than that of the ash used in blends #1 and #2 for the vessel tests at CSMRI. Table 27 gives the grain size analysis of this new ash.

Table 27. Grain Size Analysis: Trench Ash

Grain Size		Weight Percent	
Mesh	mm	Incremental	Cumulative
8	2.382	3.0	3.0
28	0.589	16.6	19.6
42	0.351	5.7	25.3
65	0.208	5.2	30.5
115	0.124	5.9	36.4
325	0.045	4.5	40.9
<325	0.045	59.1	100.0

For comparison, the same analysis is shown graphically in Figure 14 along with the analysis of ash blend #1. The significant differences between the grain size distribution of blend #1 and this supplementary ash are apparent.

Such differences were not surprising because the Lurgi pilot plant was run under various conditions to optimize its oil production which resulted in varying ash properties. It was obvious that the new ash would not result in a satisfactory grout because the large ash particles (40% above 0.05 mm) would have quickly clogged the rubble voids and the grouting radius would have been significantly decreased. As no other ash was available, it was decided to process it as follows:

- screen the ash and use only the fraction finer than 35 Mesh (0.5 mm) for preparing the grout, and
- blend all the screened ash to obtain a homogeneous material.

Both processes were performed at the CSMRI facilities.

As a result of the screening process, the amount of ash was insufficient to completely grout the trench. It was nevertheless decided to proceed with the test because additional ash was not immediately available and the grouting operation could not be delayed.

NOTE: Some additional tests were also run on the trench ash prior to its processing. The results, shown on Tables 28 and 29, confirmed the impact of the retorting history on the grout characteristics. The unconfined compressive strengths of this ash were much larger than those of blends #1 and #2.

A quick series of tests, summarized in Table 30, were run on the trench blend to complete the grout design for the trench test.

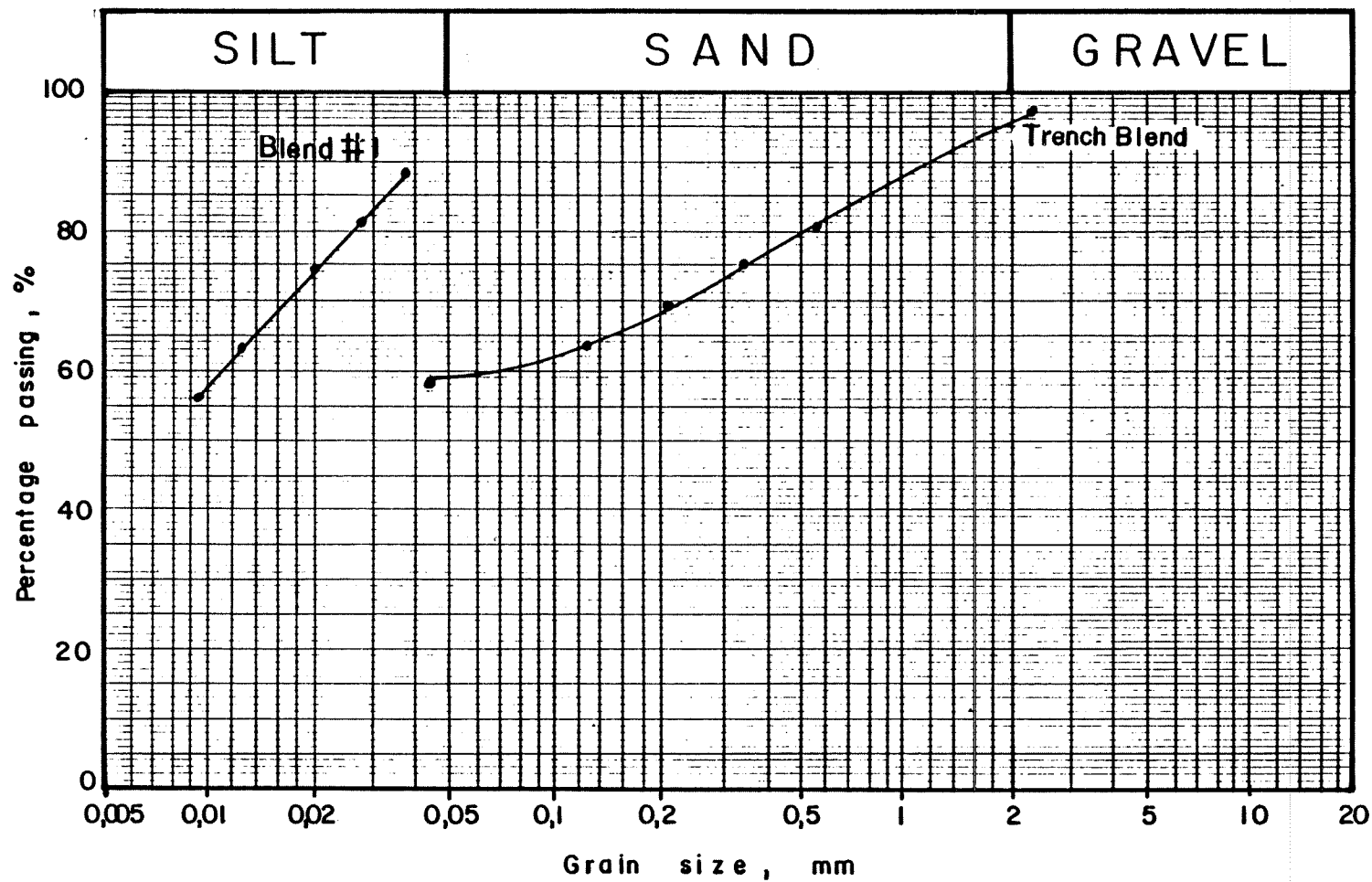


FIGURE 14.— Grain size analyses of #1 and Trench blends

Table 26. Results of Tests on Grouts Prepared with Samples of Trench Ash

Sample #	70	71	72	73	74	75
Type of SSA, Mesh	8	8	18	18	18	18
, mm	2.38	2.38	1	1	1	1
Amount, g/l	1140	1100	1000	950	950	1000
Additive 1:						
Type	-	Gluconate	Gluconate	Gluconate	Gluconate	Gluconate
Amount, g/l	-	1.3	1.3	1.3	1.3	2
Additive 2:						
Type	-	-	-	-	Cement	Cement
Amount, g/l					10	10
Marsh viscosity, s	120	122	59.5	44	44.4	60.4
Vane shear stress, Pa	3.5	3.5	1.0	0.2	0.5	0.87
Setting time, min						
(170° F)	5	50	45	50	65	65
Bleeding, % 170° F	+0.5	-2.5	-3	-5.5	-5.5	-8
70° F	-0.5	-7.0	-11	-11	-9	-11
Unconfined compressive strength, 10 ⁵ Pa (psi):						
2 days	10.7 (156)	17.8 (259)	14.7 (213)	13.2 (191)	17.2 (250)	21.0 (304)
7 days	17.5 (254)	28.6 (415)	24.7 (259)	23.5 (341)	30.2 (438)	37.0 (537)
14 days	21.8 (316)	33.7 (488)	26.1 (379)	24.6 (357)	32.5 (472)	39.4 (571)
28 days	24.0 (349)	30.6 (444)	35.5 (515)	29.9 (434)	36.6 (530)	40.8 (591)

Table 29. Results of Tests on Grouts Prepared with Samples of Trench Ash

Sample #	76	77	78	79	80	81
Type of SSA, Mesh	35	35	35	35	35	35
, mm	0.5	0.5	0.5	0.5	0.5	0.5
Amount, g/l	950	950	900	900	950	950
Additive 1:						
Type	Gluconate	Gluconate	Gluconate	-	-	-
Amount, g/l	1.5	2	1.5			
Marsh viscosity, s	47.3	54	41	42.5	59	60
Specific gravity, g/cm ³	1.60	1.59	1.56		1.61	1.59
Setting time (75° F)	24	24-48	24	24	24	24
Bleeding, %	-13	-11	-12	-8	-7	-5
Unconfined compressive strength, 10 ⁵ Pa (psi):						
2 days	too soft (too soft)	too soft (too soft)	too soft (too soft)	too soft (too soft)	too soft (too soft)	too soft (too soft)
7 days	0.72 (10.4)	too soft too soft	0.43 (6.3)	3.17 (46.0)	4.56 (65.8)	2.16 (31.3)
14 days	9.1 (132)	3.41 (49.5)	4.59 (66.6)	8.49 (123)	10.1 (145)	8.05 (116)
28 days	10.4 (151)	4.96 (72.0)	5.87 (85.2)	10.1 (147)	11.0 (160)	9.10 (132)

Table 30. Results of Tests on Grouts Prepared with Trench Blend

Sample #	Ash Content g/l	Gluconate ml/l	Marsh Viscosity sec.	Density	Setting Time
82	900	0	57	1.505	1h20
83	900	0.5	53	1.505	2h05
84	900	1.0	49	1.510	2h20
85	900	2.0	50	1.510	2h35

Mix #84 was selected because its setting time of 2 hours 20 minutes was the most appropriate. The trench test formulation is shown in Table 31 below.

4.3.3 Grout Formula and Control

At the trench test site, the Marsh viscosity as well as density of the field formulation were measured regularly and results are shown hereunder:

Marsh viscosity:	32.5 to 33 seconds
Density:	1.505 to 1.51 g/cm ³
Setting Time:	(10 to 15°C or 50 to 60°F) 2 h to 2h45
Bleeding:	4 to 6%

The Marsh viscosity was much lower than anticipated. This may be due to the different mixing process used in the field, which will be discussed later.

4.4 Tests on Trench Prior to Grouting

4.4.1 Volume of Rubble

Initial measurements of the trench dimensions gave a theoretical rubble volume equal to 55 m³ (73 cu yd). However, the installation of the rubble induced a bulging of the trench because the soil walls were very soft. The

Table 31. Grout Formula Used for Trench Grouting

Water	680 l	180 gal	138 gal
Ash	907 kg	2000 lb	1530 lb
Gluconate	<u>1 l</u>	<u>34 fl oz</u>	<u>26 fl oz</u>
Total Volume	1 m ³	1 m ³	1 cu yd

swelling was estimated to be about 12%. The corresponding bulk volume of rubble in the trench was close to 62 m^3 (82 cu yd). This would give a void volume close to 18 m^3 (23.5 cu yd) assuming a rubble porosity of 0.29.

4.4.2 Saturation of Rubble

A total of 55 m^3 (72 cu yd) of water was injected into the trench at a flow rate of $6 \text{ m}^3/\text{h}$ (7.5 cu yd/h). No water flowed out of the relief pipes because the excess water was absorbed by the surrounding soil (possibly along the edges of the plastic sheets). This operation induced further settlement of the soil below the rubble and cracks appeared on the surface.

4.4.3 Permeability Tests

Six permeability tests were carried out through the grouting pipes. The average of these six tests gave a permeability close to $1 \times 10^{-1} \text{ cm/s}$ (0.2 ft/min).

4.5 Grouting Plant

Figure 15 is a schematic diagram of the grouting plant installed near the trench. The high turbulence mixer had a 1,500 l (400 gal) capacity and was powered by a 35HP Mission Pump (Photo 13). Each 1 m^3 (265 gal) batch was prepared by mixing the water, ash, gluconate and a cement dye.

Average preparation time for a 1 m^3 batch was about 15 minutes. As soon as a batch was ready, it was sent to the storage tank. The grout was continuously circulated through one of two Moyno pumps. Compressed air was bubbled through the tank in order to prevent the ash particles from settling. The grout was pumped to the grouting point by one Moyno pump through the stone trap. The only limitations of grout intake were the absorption capacity of the rubble and the low grout-injection pressure.

A floating level indicator mounted on the storage tank allowed the volume and flow rate of injected grout to be measured.

4.6 Grouting Test

4.6.1 Planned Procedure

The grouting operation was planned to be performed in six phases, one for each grouting hole.

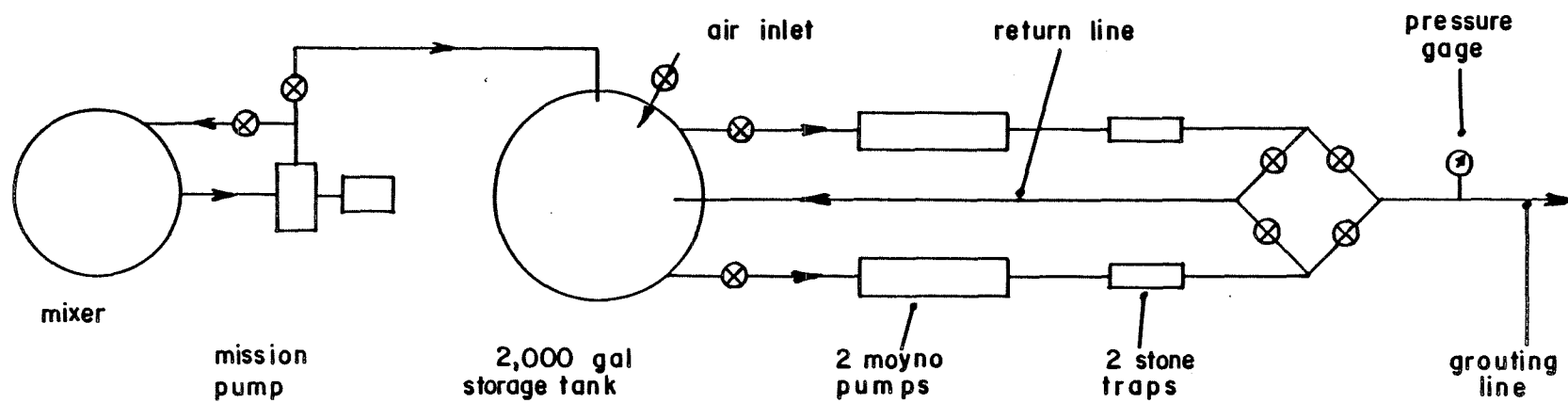


FIGURE 15 .— Schematic of grouting plant



PHOTO 13. Grouting Plant

4.6.1.1 Methodology. For each hole, the grouting methodology would be the same. Pressure at the top of the grouting hole would be carefully monitored by pressure gauges. Both grout pressure and flow rate would be controlled, according to the absorption capacity of the rubble, by adjusting the discharge valve on the return line and the rotation speed of the pump. Grout intake would be continuously monitored by checking levels in the storage tank at regular time intervals. All possible visual observations would be also made such as successive leakages out of the relief pipes and heaving or cracking of the working platform.

Water coming out of the relief pipes would be collected in a tank and measured as a second check of the void volume being filled by the grout. As soon as grout flowed out of a relief pipe, the corresponding valve would be closed.

Grouting would be continued until the pressure reached a maximum of 1 to 1.4×10^5 Pa (15 to 20 psi) even under a low flow rate. The grouting would then be transferred to the next grout hole.

4.6.1.2 Sequence of Grouting. The grouting operations were planned in the following sequence which can be followed on Figure 16.

Phase 1

Grout would be pumped into the bottom of the grouting hole #1 (GH1b); i.e., through the lower two and a half feet of slotted pipe. During this phase, the top valves of all other grouting holes would be closed and relief holes RH1, RH2, and RH3 would be open. The grout would not be colored in this phase.

Phase 2

Prior to phase 2 grouting, all grouting holes and relief holes (except GH1b) would be cleared out to prevent any remaining phase 1 grout from setting and plugging them.

The grout would be pumped into GH1t (grouting hole #1 top) corresponding to the upper two and a half feet of rubble layer. Again, the grout would not be colored.

Phases 3 to 6

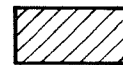
The grouting sequence would then resume as follows:

- phase 3 grouting GH3b with red grout.
- phase 4 grouting GH3t with red grout.
- phase 5 grouting GH2b with green grout.
- phase 6 grouting GH2t with green grout.

Legend

GH - Couple of grouting holes - 2" OD pipe

RH - Relief hole - 2" OD pipe

CH - Coring hole - $\emptyset$ 4" casing-coring-permeability testBH - Bore hole - $\emptyset$ 4" casing-boring-pressuremeter test

Simulated rubble

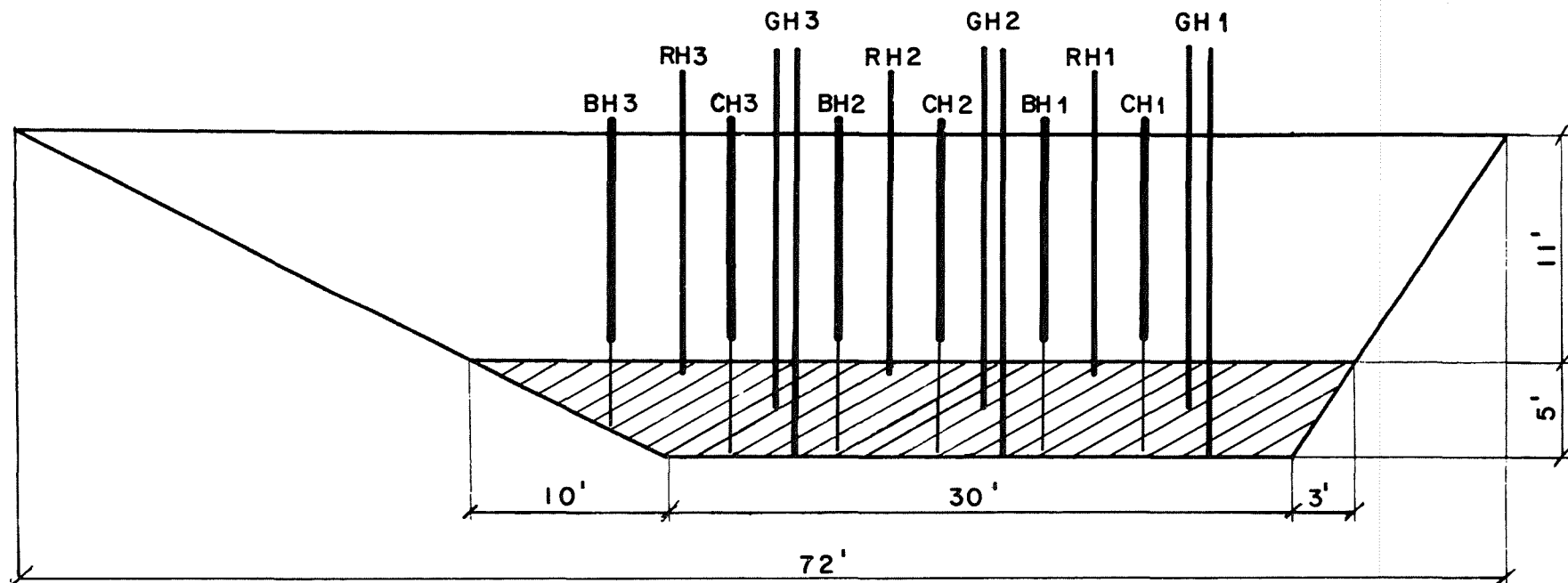


FIGURE 16. - Longitudinal trench section . Location of equipments and tests

4.6.1.3 Grouting Criteria. The pumping rate and the corresponding pressure were the only parameters that could be adjusted during the test.

At the beginning of grouting, the pump discharge rate would be fixed at the maximum value compatible with both the setting time of the grout and the maximum grouting pressure that would not hydraulically fracture the overburden. A maximum pumping rate of 4 m³/h (5.25 cu yd/h) was anticipated.

4.6.2 Trench Grouting

Phase 1 grouting started as soon as two 1 m³ (1.3 cu yd) batches of grout were ready in the storage tank. Grout was first injected into GH1b (grouting hole 1 bottom) at a steady rate close to 4 m³/hr (5.2 cu yd/h), the rate of grout production. Injection pressure started at 0.0 psi and after half an hour fluctuated around 3.4×10^4 Pa (5 psi). A total of 9 m³ (11.8 cu yd) of grout were injected in two and a half hours during which the pressure increased slowly to 6.9×10^4 Pa (10 psi).

Phase 2 grouting was then initiated at GH1t (grouting hole 1, top). An additional 3 m³ (3.9 cu yd) of grout were injected in 45 minutes at an average pressure of 1×10^5 Pa (15 psi).

The last two batches of grout were prepared with the addition of a red cement dye (25 lb/m³). Phase 3 and 4 grouting were attempted at GH3b and GH3t (grouting holes 3 bottom and top) but no grout intake occurred even at the maximum allowable pressure of 1 to 1.4×10^5 Pa (15 to 20 psi).

The grouting hose was then connected to GH2b (grouting hole 2 bottom) and Phase 5 grouting commenced. Within 35 minutes, 2 m³ (2.6 cu yd) of red grout were injected at an average pressure of 1×10^5 Pa (15 psi) until the grout storage tank was empty. The grouting operation was then ended for lack of ash to mix additional grout. Table 32 summarizes the main data of the trench grouting.

Table 32. Trench Grouting Summary

Grouting hole	Volume Injected		Time of Injection	Average Pressure	
	m ³	cu yd		psi	10 ⁵ Pa
1b	9	11.8	2h30	5-10	0.35-0.69
1t	3	3.9	0h45	15	1.0
2b	2	2.6	0h35	15	1.0
Total	14	18.3	3h50		

NOTE: No water displacement was noted except for the few last minutes of grouting through the GH2 top during the injection through GH2 bottom. About 50 l (13 gal) of water was recovered.

4.7 Curing

The grouted rubble in the trench was allowed to cure at a temperature of 10-16°C (50-60°F) for four weeks prior to the start of the post-grouting test program.

The rubble was not heated for the trench test because the heating cost would have been quite expensive. Consequently, the setting time of the grout was about twice what it was at 77°C (170°F). But the pumping rate, as previously mentioned, was much lower than it will be in actual MIS retorts due to the pressure limitation. At the minimum, this will compensate for the other factor.

4.8 Tests in the Trench After Grouting

Four types of tests were scheduled: coring, permeability, pressuremeter, and visual observation.

4.8.1 Coring

After the grouted rubble was cured, three corings were performed using a diamond core barrel, 44.45 mm (1-3/4") O.D., driven by an air powered drill. First, boreholes were made through the blanket about 2.4 to 2.7 m (8 to 9 feet) deep with a 127 mm (5") diameter rockbit and the boreholes were equipped with 102 mm (4") O.D. casings. Then the corings were performed down to the bottom of the rubble. They were located on the middle line of the trench as shown in Figure 16. No undisturbed core samples were recovered.

4.8.2 Permeability Tests

Permeability tests were performed in coreholes CH1, CH2, and CH3 (Figure 16). The results are given in Table 33.

The permeability of the rubble was decreased by a factor of over 1000 by grouting. The permeability of this grouted rubble is high compared to those measured in the lab samples or in the vessels. This was expected since the grouting was incomplete.

Table 33. Permeability Test Results After Grouting

Corehole	Permeability cm/s
CH1	1×10^{-5}
CH2	4×10^{-5}
CH3	6×10^{-5}

4.8.3 Pressuremeter Tests

Some problems were encountered in the pressuremeter tests. The bore holes were drilled with 63.5 mm (2-1/2") rockbit for a 60.3 mm (2-3/8") probe. The 1/8" difference in diameter is usually quite suitable. However, the coring was performed with compressed air which sometimes results in larger diameter holes. This actually occurred in two of the three coreholes. In these two coreholes, the difference in diameter was so large that the probe's membrane was almost fully inflated when it reached the borehole walls. The remaining expansion capability was too small to achieve a proper test.

However, a satisfactory test was performed in CH1. The probe was two feet below the top of rubble and the test was running well until the pressure reached 16×10^5 Pa (232 psi). Then the probe membrane burst. The initial elastic modulus (E) computed from the test data is 455×10^5 Pa or 6,597 psi. The shape of the curve and the premature bursting did not allow the limit pressure to be computed (Figure 17).

4.8.4 Visual Observations

After completion of the various tests on the grouted rubble, the overburden was stripped off. The rubble mass was first cleared on the top and the periphery with a backhoe and then by a compressed air stream.

The first observation was the presence of a thin layer of pure grout (without rubble) about 3 to 30 mm thick (1/8 to 2") between the layers of polyethylene that covered the entire top of the rubble. The grout, gray or red, obviously came from the grouting pipes at the level of the top plastic liner. The grout flowed upward along the pipe to the liners and then horizontally between both liners. Apparently, the grout filled a void that existed between the rubble and overlying clay. This void might have been caused by the settlement of the rubble after the rubble was filled with water.

The grouted rubble was cut into eight cross sections shown on Figure 18 (AA to HH). The first cross section was made on the side of the ramp (AA) with successive sections made toward the other end (HH). After each cut by the backhoe, the cross section was cleaned by compressed air so a clear observation of the grouted and non-grouted areas could be made. Figures 19 and 20 show schematic sketches of each cross section with grouted and ungrouted areas identified.

The bottom and the walls of the trench rubble were grouted over a thickness of at least one foot (except at section GG). As expected, the percentage of grouted area was greater where the cross sections were closer to GH1 through which 12 m^3 were injected. In sections AA and BB, the grout in the center was red grout. In several cross sections (CC, DD, and EE), red grout was found interlayered with gray grout. This shows that the red grout penetrated the first grout, which was already set.

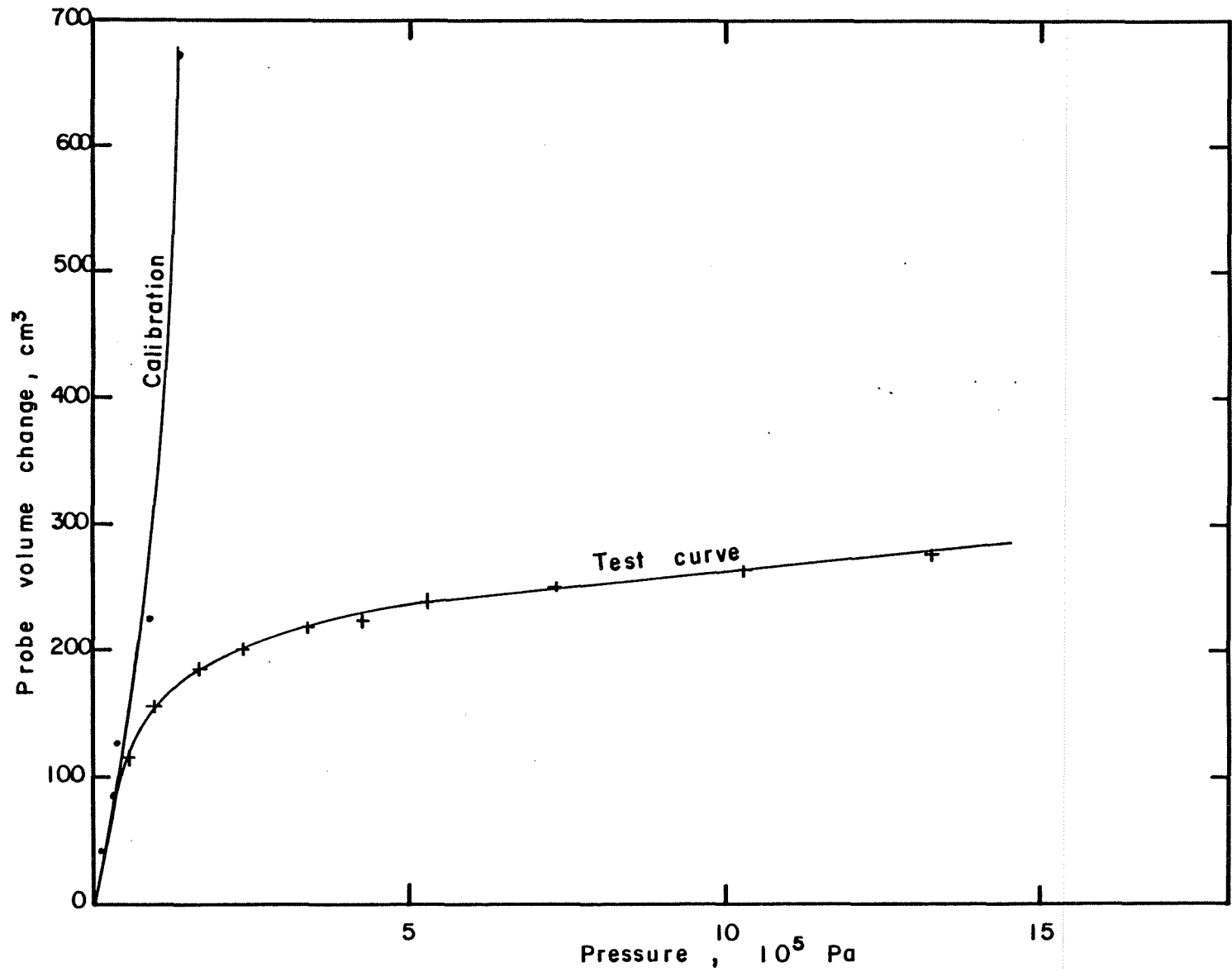
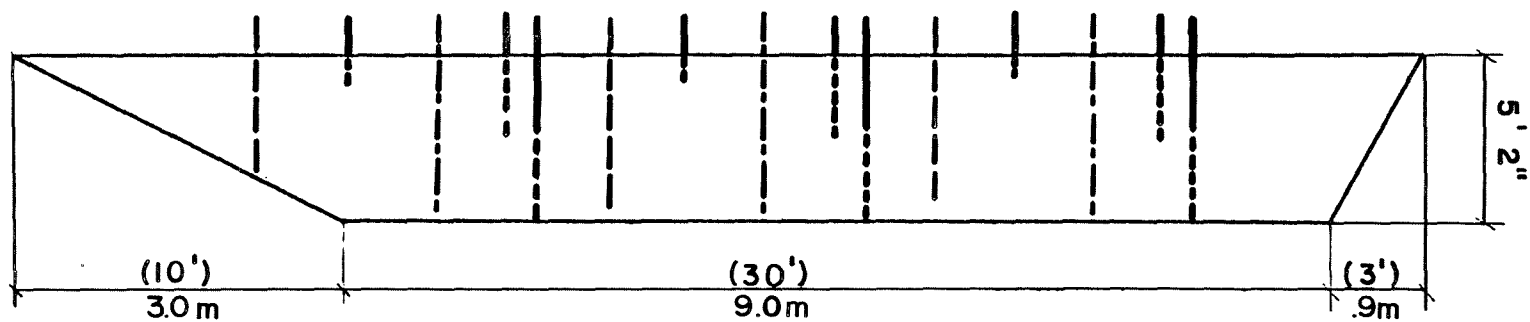


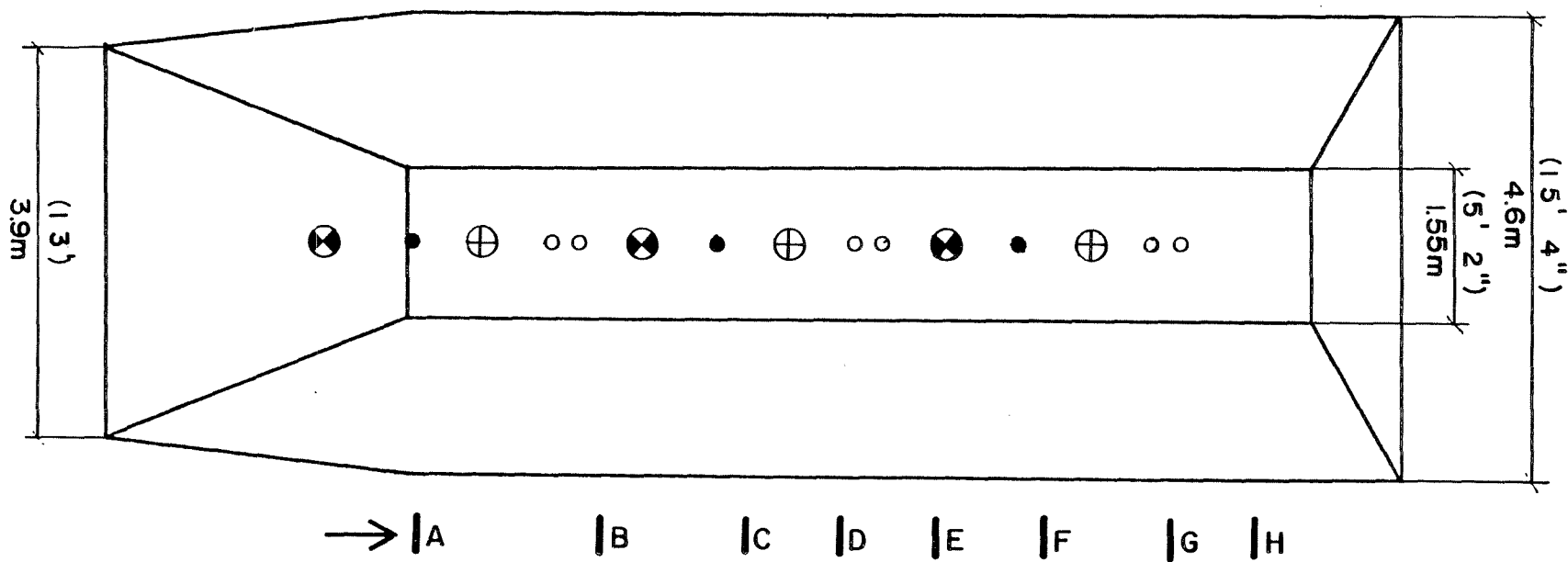
FIGURE 17 — Menard pressuremeter test - C H I



Legend :

- ⊗ — Bore hole - Pres. test
- ⊕ — Core hole - Perm. test
- Slotted 2" pipe

→ | A | B | C | D | E | F | G | H



→ | A | B | C | D | E | F | G | H

FIGURE 18.- Trench test- Location of equipments and tests

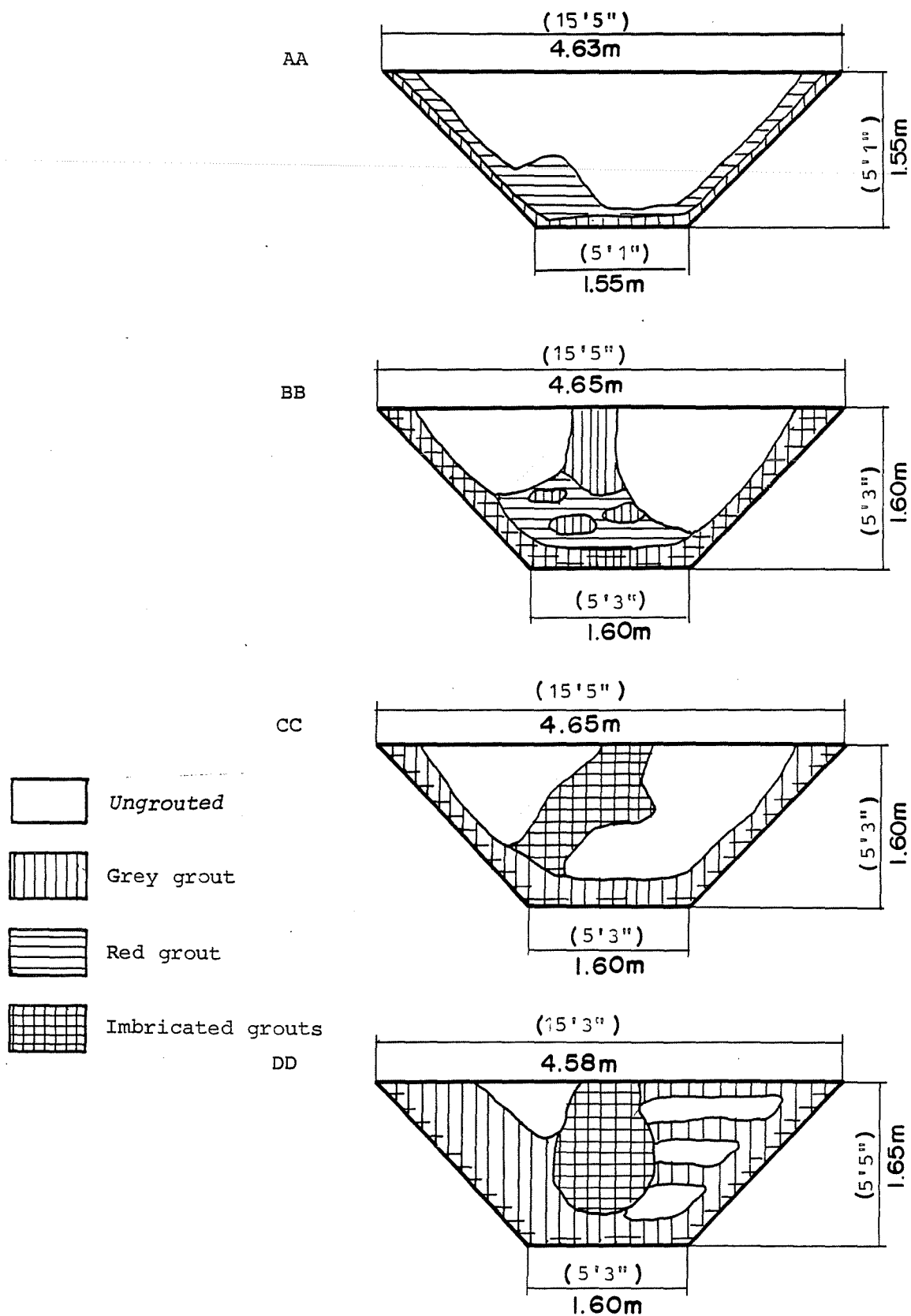


FIGURE 19 - Grouted trench - Cross sections

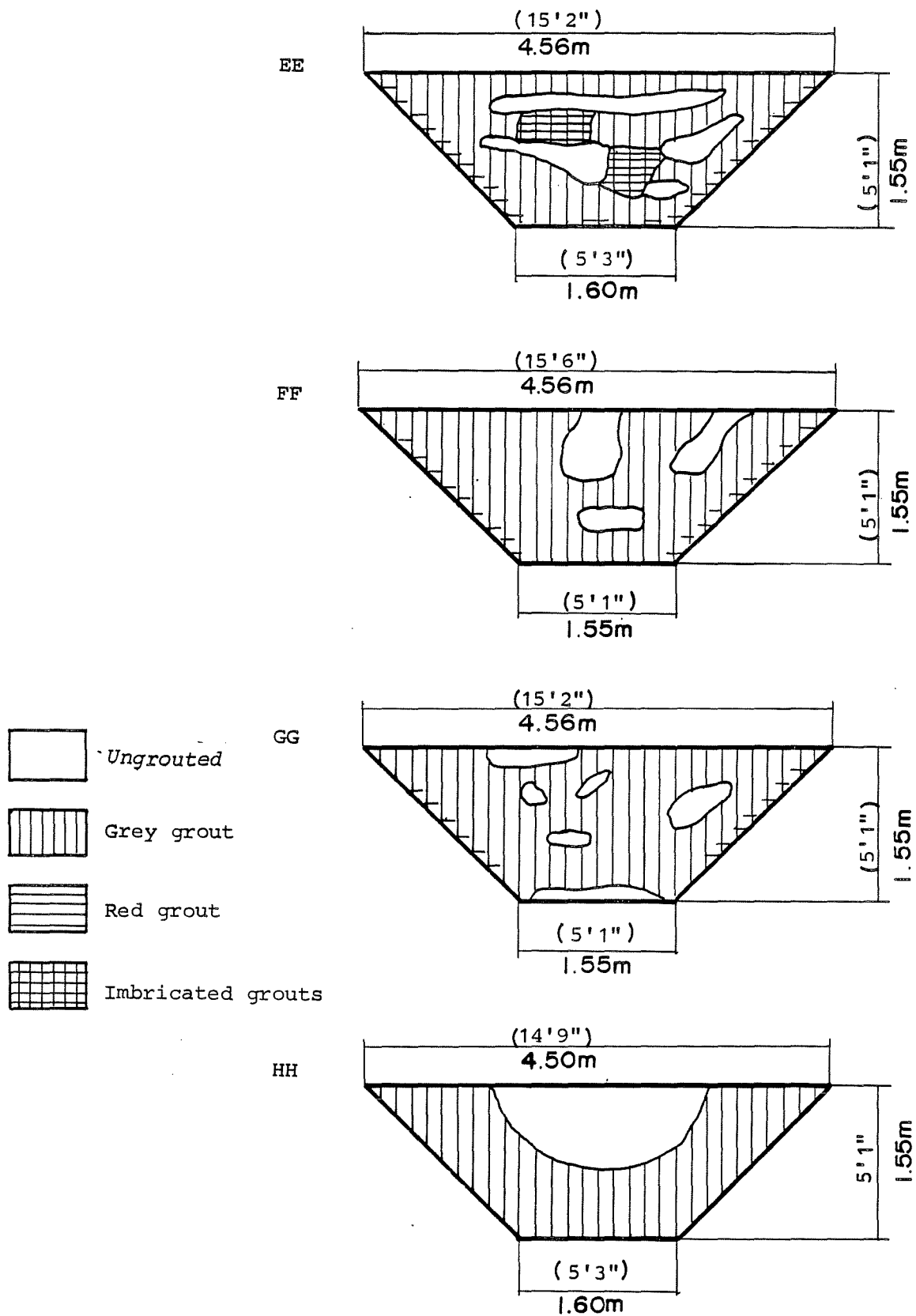


FIGURE 20 - Grouted trench - Cross sections

The first gray grout in the rubble was found 6.1 to 6.4 m (20 to 21 feet) from GH1. This distance was the maximum distance limit of grouting that was recorded in the trench. No cross section was found completely grouted. There remained at least some ungrouted pockets, mostly of fine rubble, which would have been more completely grouted if more grout had been available. In the last section, HH and beyond, a fairly large zone was not grouted. In cross section AA and beyond, most of the rubble was ungrouted due to the lack of grout. An analysis of the grout penetration is given in the next chapter.

Nowhere was the presence of grout observed outside the liners in the natural soil. The non-woven fabric efficiently filtered the grout and allowed only its water fraction to flow through. The volume of grouted rubble was evaluated at 70% of the total rubble volume which leads to an overall void volume of 20 m³ (26 cu yd). This represents the volume of grout that would have been required to achieve complete grouting.

The grout itself (red or gray) was homogeneous and quite satisfactory with a unconfined compressive strength in the range of 20 to 25 x 10⁵ Pa (300 to 360 psi). This value was far higher than the target strength of 7 x 10⁵ Pa (100 psi).

4.9 Trench Test Observations

In spite of several unforeseen problems, the primary objective of the test was accomplished; namely, to determine the horizontal distance of effective grout penetration through the rubble from an injection hole. The maximum distance found, 6.1 to 6.4 m (20-21 ft), is encouraging.

5. ANALYSIS OF LABORATORY AND TRENCH TESTS

The laboratory and trench tests showed that the impact that the MIS process might have on the environment can be limited.

5.1 Tests on Rubble Prior to Grouting

The unconfined compressive strength tests on completely spent rubble showed it to be a low-strength material. This rubble will probably crush under its own weight. Additional crushing may occur under the thermal stress to which it will be subjected in actual MIS retorts. This may result in the development of a large cavity at the top of the retort, rupture of the pillars between MIS retorts and eventual surface subsidence.

Prior to grouting, rubble porosities and permeabilities in both the vessels and the trench were quite high. The potential exists for ground water contamination or mixing of aquifer waters.

Furthermore, large quantities of water were absorbed by the spent rubble in the vessel tests. This confirmed that presaturation of actual MIS retorts with water is necessary before grouting. Otherwise, free water from the grout

would be absorbed by the hot spent rubble. This would totally change the rheological characteristics of the grout and substantially decrease grout penetration.

5.2 Grout Development

An extensive study of spent ash grouts was made. Eighty-five samples were tested. (See Appendix D for formulations.) These tests allowed for variations of major parameters such as types and contents of ash as well as additives. The results confirmed that:

- The retorting history of the spent ash is an important parameter for the development of its cementitious potential. (Its storage conditions may also be important.) This fact should not be overlooked in the development of the surface retorting system.
- Regardless of which type of ash was used, it was possible to develop a suitable grout. Only small variations of the ash content and additive content were necessary to obtain the target rheological and mechanical characteristics.
- The increased permeability with time on the laboratory samples should be further investigated to determine if this increase resulted from a microfissuration caused by swelling when the tests were stopped.

5.3 Grouting Tests on Vessels and Trench

The grouting tests were also performed under various conditions to simulate the different porosities that may exist in actual MIS retorts. The rubble porosity varied between 0.29 and 0.45. Valuable information was obtained on the mechanical characteristics of the grouted rubble mass as well as the radius of grout penetration.

- Mechanical tests performed on the grouted rubble mass in the vessels showed that, when successfully grouted, it behaved as a competent engineering material under confined conditions, capable of sustaining heavy loads without significant deformation or failure (see Appendix C).
- The grouted mass from the vessels showed a decrease of permeability with time. The same tendency is observed in alluvium grouting. It is usually explained as a self plugging of the voids by fine particles when water travels through grouted alluvium.

Visual observations and measurements made on the grouted vessels and the trench are summarized in Table 34.

Table 34. Comparison of Grouting Data and Results
(Grouting Phase I) for Vessel and Trench

	Vessel #1	Vessel #2	Trench
Rubble porosity prior to grouting	.45	.40	.29
Rubble permeability prior to grouting, cm/sec	40 to 60	8 to 10	10^{-1}
Rubble permeability after grouting, cm/sec	7 to 25×10^{-7}	4 to 9.5×10^{-6}	1 to 6×10^{-5}
Observed grout penetration, m	3	1.5	6
Corresponding grouting parameters:			
Marsh viscosity, sec	45 to 55	40 to 50	33
Temperature, °C	77°	77°	10 to 15°
Setting time	95 min	100 min	165 min
% of voids grouted	96%	80 to 90%	?
Grouting pressure 10^5 Pa	6.9	6.9	1.4

The difference of grout intake as well as the different radii of penetration shown above can be explained by the different test conditions. The grouting of the rubble with the high porosity (.45) in vessel #1 was very successful because the voids between rubble were so large that the grout could easily flow through them even in a totally confined environment.

The incomplete grouting of vessel #2 was probably caused by:

- The grouting procedure itself. If no pressure limit (100 psi) had been set for the first phase of grouting, most of the voids probably would have been grouted in this first stage.
- The lateral boundary conditions. If lateral displacement of the rubble mass had been possible during the second and third stages of grouting under higher pressure, the grout would have created new paths by hydraulically fracturing the rubble mass. This would also have resulted in a more complete grouting. Boundary conditions of actual MIS retorts may allow such displacements under great pressure.

The grout intake during the trench test, which was conducted under very low pressure, was almost equivalent to the intake obtained on vessel #2 in the first stage; and the radius of penetration was in fact much larger than for vessel #2, although the porosity of the trench was smaller (.29 versus .40). This is believed to be caused by the combination of two factors:

- The boundary conditions for the trench test allowed deformation of the rubble mass during grouting; and
- The viscosity of the grout was much lower for the trench test (33 versus 46 Marsh seconds).

Both of these conditions resulted in smaller head losses than in the vessels.

The lower viscosity of the trench grout was most likely caused by the different mixing conditions. The characteristics of identical grout formulas mixed in the laboratory and in a field mixing plant often vary. First, the measuring of the components is not as accurate in the field as it is in a laboratory. But the major difference lies in the mixing process. The mixing energy of an adequate field-size mixer is much greater than that of a laboratory-size mixer. The grout is therefore more thoroughly mixed in the field. The grout mixed at the field plant actually had a much lower viscosity than the laboratory mix (33 versus 46 Marsh seconds) but it is important to note that its bleeding stayed within the target value. In future work, it may therefore be possible to increase the ash content of the grout and still obtain satisfactory characteristics.

The visual observations made in the trench test permit an analysis of the way the grout traveled within the rubble mass. Figure 18 shows the cross section and grout hole locations. Figures 19 and 20 show grout penetration at each cross section. An analysis of the cross sections shows that the grout traveled in the trench in two different ways:

- The initially injected grout traveled along the boundary of the rubble; i.e. between the rubble and the liner. This was observed on cross sections CC, BB, and AA where mostly first stage gray grout was observed. This corresponds to a well known phenomenon in alluvium grouting; i.e. if there is a change of soil conditions, such as a gravel-clay boundary, the grout will first travel along this boundary.
- The above described phenomenon was observed at every cross section except one (GG). At the remaining seven cross sections, a layer of grout was present along the base and sides of the rubble mass suggesting that even partial grouting of an abandoned MIS retort could reduce its permeability by the development of a grouted rubble "skin".
- The first phase gray grout was injected through the holes GH1b and GH1t. Its penetration in the center of the trench (observed on cross section GG through BB) corresponds mainly to a travel path inside the rubble mass. As the grout traveled further away from the grouting location, the grouting pressure decreased. This resulted in a decrease of the areas impregnated by the grout. The maximum distance of penetration was 6.4 m (21 ft).
- Cross section HH indicated that water was trapped in the top center zone of the rubble where no relief hole was placed. This trapped water probably prevented grout from traveling into that zone.
- The second stage red grout (observed in cross sections DD through AA) traveled through the rubble mass since the boundary zone was already

sealed by the first stage grouting. Its radius of grout penetration was 4.25 m (14 ft), which was a minimum value because this phase was stopped for lack of ash to prepare additional grout.

In conclusion, these tests confirmed that the basic principles used for alluvium grouting can be applied to abandoned MIS retorts, and that 40 to 50% of the ash generated from surface retorting could be injected into MIS retorts to stabilize them.

6. DESIGN OF THE PILOT GROUTING SYSTEM

Upon completion of Phase I, the next three phases were to design a pilot-scale grouting plant, prepare a plan to grout a small-scale MIS retort, evaluate the effectiveness of the retort grouting and design a program to monitor its long-term behavior. The following gives the results of the study which is completely outlined in Appendix E. It was assumed that MIS Retort Zero at Tract C-a will be used for this investigation.

6.1 Design of Pilot Grouting Plant (Phase II)

The pilot plant was designed to prepare and inject 20.9 m³ (25 yd³) of ash grout per hour using a grout formulation similar to the ones developed in Phase I. The pilot grouting plant can be divided into two distinct units, the mixing module and the grouting module.

The mixing module will be installed close to the surface retorting plant. A continuous mixing concept was selected over the batch system because it will keep pace with the required production of a commercial operation. The schematic of the mixing module is shown on Figure 21. Once mixed, the grout will be pumped to the retort where a grouting module will be installed. This module will consist of a storage tank with an agitator and three or four high capacity grouting pumps (Figure 22). Capital and operating costs for the pilot plant are listed in Appendix E.

6.2 Plant to Grout Pilot MIS Retort Zero (Phase III)

The drilling and grouting techniques were selected in view of the difficult conditions that are anticipated in Retort Zero; mainly, a small target area at a considerable depth, a cavity at the top of the retort, drilling through heterogeneous material and unstable zones, large variations in rubble size, and risk of drilling fluid loss.

Prior to grouting, the existent conditions in the retort will be determined by borings and water tests. The drilling and grouting procedure will be modified if necessary after this analysis.

The drilling and grouting of Retort Zero will be performed in two stages.

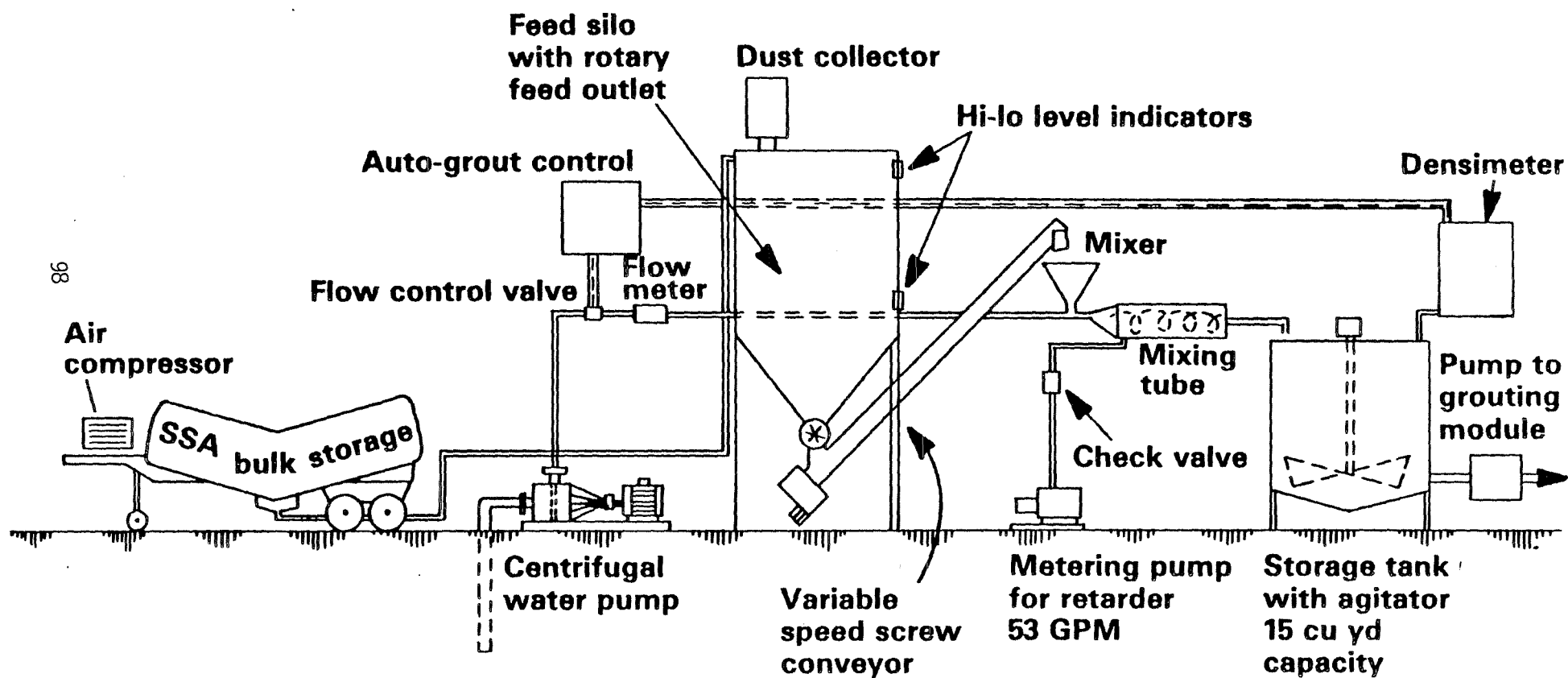
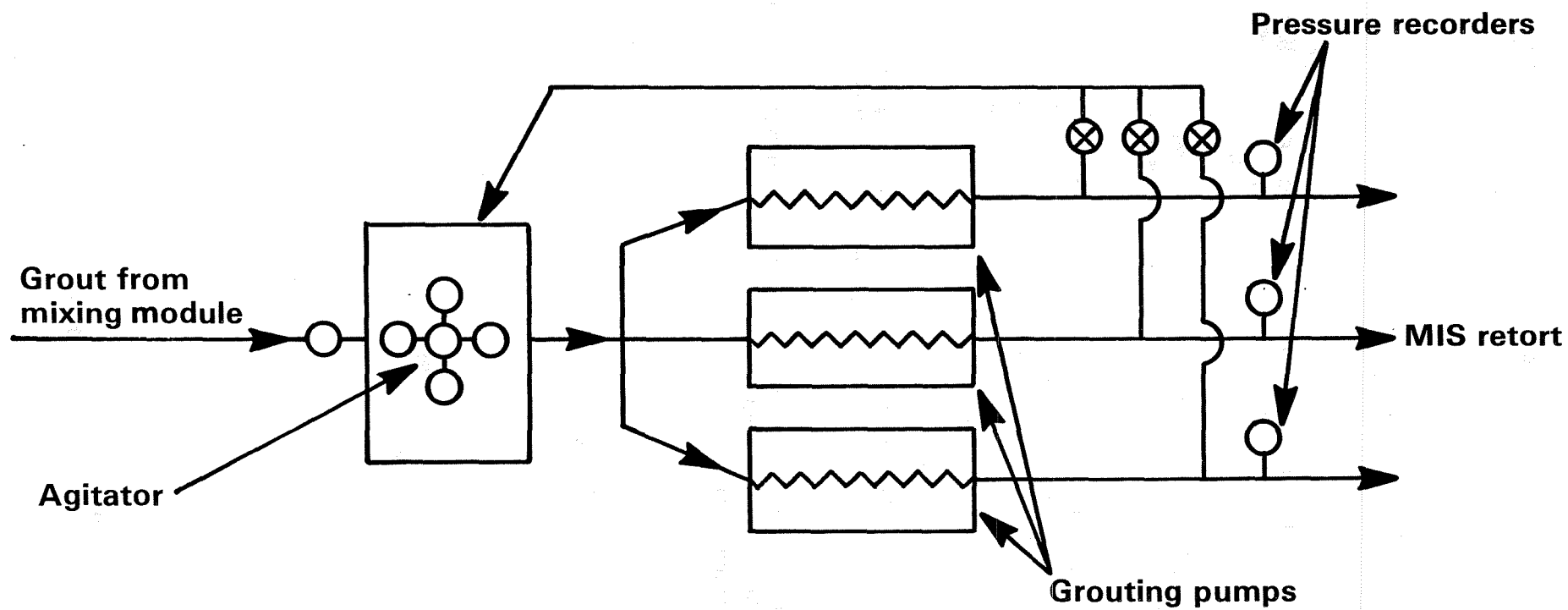


FIGURE 21. Schematic of the Mixing Module

Fig. 22
Schematic of the Grouting Pumps



Stage I will consist of grouting the cavity that will most likely exist at the top of the retort. This cavity will be filled by gravity through the retort blast holes already existing in the overburden. After allowing sufficient time for setting, a second phase of grouting at the top of the cavity will be performed to insure proper contact between the grout and the base of the overburden.

Once the cavity is filled, Stage II drilling using the same holes will proceed from top to bottom of the retort using bentonite fluid and grout alternately. When fluid losses are noticed, the drilling rods will be pulled up and grout will be pumped in until it comes out of the hole's annular space at the surface. Drilling then proceeds to the final depth.

Once each hole is completed and stabilized, it will be flushed with bentonite, the drilling equipment will be removed, and a steel sleeve pipe will be installed. The sleeve pipe will then be secured with grout.

Pressure grouting will then be performed through the sleeve pipes. If the total grout intake, including the cavity filling and pressure grouting, corresponds to 20% or more of the volume of the retort, the treatment will be considered successful. If this volume is less than 20%, additional grouting holes will be drilled using the same method.

6.3 Monitoring Program to Evaluate the Performance of Grouted Retort Zero (Phase IV)

The data collected during drilling and grouting of Retort Zero will be analyzed. The in-situ conditions and characteristics of the grouted retort will be thoroughly investigated by means of additional drilling, corings, and a series of permeability and crosshole tests.

A long-term monitoring system will be installed to measure or control any displacements inside and above the grouted retort and the hydrogeological conditions in and around it.

7. CONCLUSIONS

The objectives of this research program were to investigate the potential use of an ash-based grout to fill and stabilize MIS retorts; to design a pilot grouting plant and a program for grouting a small-scale MIS retort; and to monitor the grouted retort for some time.

7.1 Evaluation of Results of Laboratory Grout Development

Eighty-five grout samples were tested. Their formulations are listed in Appendix D. Many parameters of the grouts were varied, mostly the type of ash and additives used as well as their respective amounts in the grout mix. Most important, it was always possible to develop grouts from Lurgi ash, if not too coarse, that would fulfill the objectives of the program.

The grouts had to be satisfactory with respect to a number of criteria, specifically, viscosity, bleeding, setting time, groutability, strength, permeability and durability.

Target values were determined for these criteria assuming that the fundamentals of grouting alluvium would apply to grouting spent shale.

To take into account the high temperature that will exist in the abandoned retorts, all the grout samples prepared in the laboratory at room temperature were cured in water baths at a constant temperature of 77°C (170°F).

The first series of tests on ash grouts without additives confirmed the results of previous studies:

- They set and hardened by themselves.
- Their mechanical characteristics varied according to the ash used; this confirmed that such characteristics vary according to the retorting history and probably the conditions of storage.
- They were stable.
- Their rheological characteristics were not suitable to grout MIS retorts because their setting times were too short and viscosities too high. It was therefore necessary to find additive(s) that would lengthen the setting time, decrease the viscosity, and in some cases improve the mechanical characteristics of the grouts. Twelve additives were tested. Most of them yielded satisfactory results. Sodium Gluconate was selected because it was the most promising.

7.2 Evaluation of Results of Grouting Tests on Vessels

The next phase of the study consisted of defining:

- the groutability of spent rubble with the selected grouts,
- the mechanical properties of the grouted rubble mass.

First, two 5 m³ steel vessels were filled with spent rubble with different void ratios. They were grouted in three stages up to a maximum grouting pressure of 200 psi.

Practically all voids in vessel #1 (estimated rubble porosity .45) were grouted. Grouting of vessel #2, with a lower rubble porosity (.40) was estimated to be 80 to 90% complete.

Pressuremeter tests run in the vessels proved that well grouted rubble is a competent engineering material under confined conditions, capable of sustaining heavy loads without significant deformation or failure.

The values of the permeability coefficient with a gradient close to 36.5 were very encouraging. The "overall" coefficient of permeability for vessels

#1 and #2 were 7×10^{-7} and 9.5×10^{-6} cm/s, respectively. The permeabilities decreased with time.

The main conclusions of the vessel grouting tests were that:

- spent rubble can be grouted with ash grout;
- the resulting grouted rubble mass should have sufficient mechanical strength to decrease substantially any risk of large mine failure, surface subsidence or of ground water contamination; and
- grouting reduces the permeability of the spent shale from that of a sandy or clean gravel to that of a silty clay.

7.3 Evaluation of Results of Trench Test

It was then necessary to determine the radius of penetration of the grout into the rubble in order to design the grouting system of a small-scale MIS retort. This was done by means of a trench test.

A trench was excavated on Tract C-a. Its lower 1.5 m (5 ft) were backfilled with simulated rubble (raw shale, because spent rubble was not available); its upper part was then backfilled with a compacted impervious material. The simulated rubble was then grouted. The maximum grouting pressure was limited to 1.4×10^5 Pa (20 psi). This was necessary because of the thin confining overburden above the simulated rubble. This low pressure would tend to decrease the radius of grout penetration. On the other hand, the simulated rubble was not heated. Grouting under ambient temperature should increase the setting time and therefore increase the radius of grout penetration. It was estimated that the limit of pressure would have the most critical influence on the radius of penetration and the temperature parameter would compensate for the low pressure.

The visual observation of the trench allowed the measuring of a maximum radius of penetration of 6.4 m (21 ft) and a minimum of 4.25 m (14 ft).

7.4 Design of a Pilot Plant

A pilot plant was designed for mixing and grouting a small-scale retort. Two distinct modules were designed:

- A mixing module, which will be installed close to the source of ash (Surface Retorting Plant). Mixing of the grout will be continuous (instead of the batching system). Grout density will be monitored continuously. This information will be fed back to the control panel to adjust the mixing process as necessary.
- A grouting module, which will be installed at the grouting location.

The target capacity for the pilot plant was 20 to 25 cu yd/hr. The capital cost of the pilot plant was estimated at \$216,000 and the operating cost was estimated at \$18,500 per week.

7.5 Design of a Grouting System for a Small-Scale Retort

A grouting system was designed for Retort Zero at Tract C-a. The characteristics of the spent rubble in the retort as well as the ash available for the mix were assumed to be similar to those measured in the laboratory program.

It was concluded that the grouting operation should be performed from the ground surface via existing retort blast holes and not from the various existing mine shafts and galleries. Such shafts and galleries would not be easily accessible during a commercial MIS operation.

Grout holes will be spaced every 4.6 m (15 ft). The downstage grouting method will be used to stabilize the rubble as drilling proceeds. Once completed, the boreholes will be equipped with a special sleeve pipe and a high pressure upstage grouting of Retort Zero will be performed. The water required to make the grout could be obtained either from mine dewatering or from surface processing.

The retort in-situ conditions will be determined prior to grouting. This will allow a modification of the grouting technique and the grout characteristics if necessary.

The retort will be monitored after grouting to measure the effectiveness of the grouting program.

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Appendix A

TEST PROCEDURES AND STANDARDS

A.1 Preparation

The grouts are prepared in 10 liter (2.6 U.S. gallons) plastic utility buckets using an electric stirrer equipped with a propeller running at a speed of about 1,000 RPM. (Figure A-1)

The ash is poured into water¹ while stirring constantly (duration of pouring about 1 minute); stirring is continued for one minute after all the ash has been poured. Then additives, if any, are successively added and the mix is stirred for 2 more minutes.

The above description will yield approximately 5 liters (1.32 U.S. gallons) of grout. When larger quantities of grout are needed, a second 5 liter batch is prepared and both batches are then mixed together.

All operations are done at room temperature ($68 \pm 3^\circ\text{F}$, or $20 \pm 1.7^\circ\text{C}$).

The Marsh and Brookfield viscosities, as well as the bulk specific gravity, are measured immediately after preparation of the grout.

A.2 Molding and Curing of Grout Samples

After preparation, the 5 liter batch of grout is poured into:

- (i) two transparent plastic tumblers with end caps (diameter 2.5" [6.35 cm], height 4.5" [11.43 cm]) for two bleeding measurements.
- (ii) two plastic boxes with covers (0.5 liter capacity or 0.13 U.S. gallon) for setting time tests.
- (iii) one rectangular plastic box with cover (23 x 17 x 10 cm or 9 x 6.5 x 4 inches approx.). Twelve PVC tubes (I.D. 1-5/8" [41 mm], height 3-1/2" [9 cm]) lightly coated inside with grease and sunk into the grout, to be left in place while the grout sets and hardens and until the tests are performed. The cylindrical samples are removed from the tubes for unconfined compression testing after 2, 7, 14, and 28 days of curing. Three tests are run for each curing time.

All the molds described under (i), (ii), and (iii) are placed in a water bath where temperature is kept constant at 170°F (77°C). Whenever permeability tests, triaxial tests, and one dimensional consolidation tests are anticipated, an additional batch of grout is prepared and specific molds are filled with grout and cured in the water bath the same way.

¹Tap water was used for each grout preparation.



Figure A-1. Electric Stirrer

A.3 Bulk Specific Gravity of Fresh Grouts

The bulk specific gravity or density of the fresh grouts is measured according to the API Standard Procedure for Testing Drilling Fluids, by means of a drilling mud balance that gives the specific gravity directly.

A.4 Marsh Viscosity

Marsh viscosity is measured according to the API Standard Procedure for Testing Drilling Fluids, Section 2. The Marsh funnel is first filled with 1.5 liters of mud or grout. The Marsh viscosity is the outflow time of one U.S. quart (946 cm³) of fluid. A graduated cup is used as a receiver. It must be pointed out that the Marsh viscosity of water is 26 ± 0.5 Marsh seconds.

A.5 Dynamic Viscosity and Casson Viscosity

A.5.1 Elements of Rheology

Viscosity is the measure of fluid friction and may be considered as the internal friction resulting when a layer of fluid is made to move in relationship to another layer.

Dynamic viscosity was defined by Newton as: $N = \frac{F^1}{S}$

N = viscosity
F¹ = shear stress
S = rate of shear

The Newtonian fluids such as water have, at a given temperature, a viscosity that is independent of the rate of shear. But many fluids (non-Newtonian fluids), such as suspension grouts like ketchup, do not match this definition since viscosity decreases with the rate of shear. Those fluids are the pseudo-plastic or plastic fluids.

Moreover, in the case of plastic fluids or Binghamian fluids, a certain force must be applied to the material before any movement is produced. This force is called the yield value. Figure A-2 shows the curves for the three main types of fluids.

The suspension grouts, such as the ash grout, fall midway between the plastic and pseudo-plastic fluids since the curve of shear stress versus rate of shear usually shows a yield value but also a curved line (curve 4) instead of a straight line.

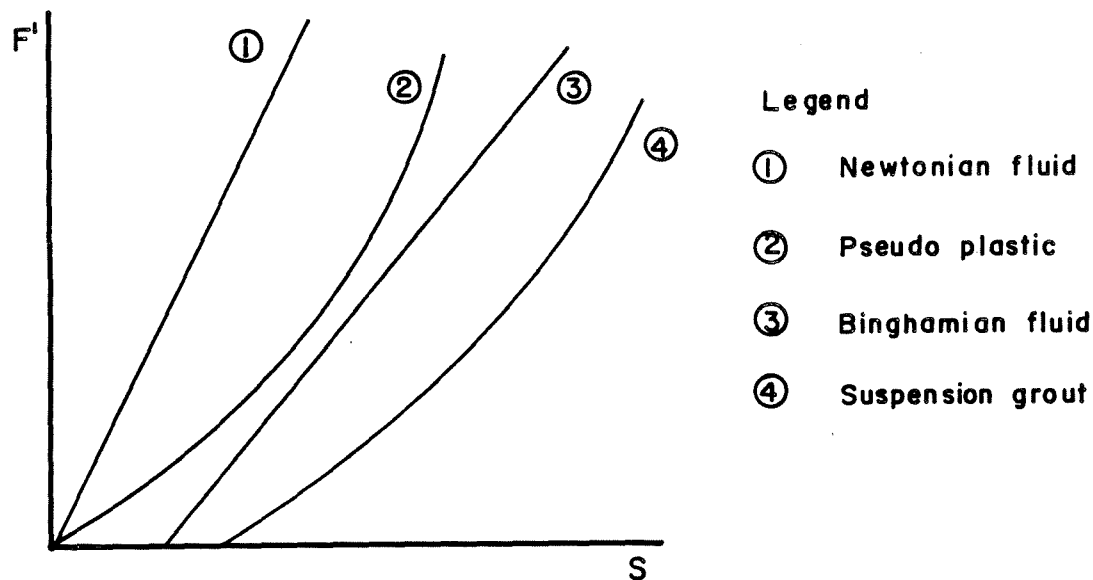


FIGURE A2 .— Typical rheological curves

A.5.2 Brookfield Synchro-lectric Viscometer

For all the grouts prepared in the lab, the curves of shear stress versus rate of shear are determined by means of a Brookfield synchro-lectric viscometer model LVT.

This viscometer rotates a cylinder in the fluid and measures the torque necessary to overcome the viscous resistance to the induced movement. This is accomplished by driving the immersed element, called a spindle, through a spring. The degree to which the spring is wound, indicated by the position of a pointer on the dial, is proportional to the viscosity of the fluid for any given speed and spindle.

The model LVT is an 8 speed viscometer (ranging from 0.3 to 60 RPM) which is provided with a set of four spindles.

For each grout the measurements are made with the most appropriate spindle with regard to the viscosity and at four different speeds (6, 12, 30, 60 RPM).

A.5.3 Viscosity Calculation

The interpretation of the curves in order to obtain a viscosity value can be made according to several methods. For this study two methods of calculation are used:

- The "standard method" which gives the dynamic viscosity,
- The Casson method that leads to the Casson viscosity.

A.5.3.1 Standard Method. This method consists of plotting the curve, dial reading versus rotation velocity, on a linear coordinates graph; then measuring the slope of the tangent to the curve at the point corresponding to the 60 RPM speed. The viscosity is proportional to the slope of this tangent; a factor depending on the spindle allows viscosity to be calculated in centipoises (cP).

A.5.3.2 Casson Method. This method is based on the following equation:

$$F^{1\frac{1}{2}} = K_0 + K_1 S^{\frac{1}{2}}$$

where:

F^1 = shear stress

S = rate of shear

This equation was specifically designed for the non-Binghamian fluids.

The plotting of dial reading (shear stress) versus speed RPM (shear rate) on square root dial reading-square root RPM coordinates graph, results in an approximately straight line. The Casson viscosity is determined by means of a special template that allows the slope to be measured. The latter is multiplied by a factor depending upon the spindle in order to obtain the viscosity in centipoises.

The intercept of the straight line with the shear rate axis allows calculation of the yield value by multiplying the intercept value by a coefficient. The yield value is expressed in Pascals (Pa) or psi.

As shown on the tables in this report, the Casson viscosity values are usually lower than viscosity calculated according to the "standard method".

A.6 Setting Time

Suspension grouts are considered set when shear stress reaches a predetermined value, usually 35 Pa (5×10^{-3} psi). At this value the grout is considered non-injectable since both viscosity and shear stress are too high for the grout to flow into the void spaces between the soil grains unless grouting pressure is increased to an extremely high level.

The shear stress measurements are made by means of the Brookfield viscometer equipped with a special four-blade vane. The measurements are taken at regular intervals of time (15 minutes), then the curve of shear stress versus time is plotted on a linear coordinates graph to determine the time when the shear stress reaches the value of 35 Pa.

A.7 Bleeding Stability

In a suspension grout, even the very fine grains of solids are attracted bottomward by gravity until the grout sets. Only viscosity and shear stress limit this movement.

This phenomenon leads to several results:

- Heterogeneous grout; i.e., the concentration of solid particles is higher at the bottom than at the top of grout samples, so that the overall strength of the sample is that of the top weaker part.
- At the same time that solid particles are moving downward, a layer of free water appears at the top of the sample. This is called bleeding, a phenomenon which could create problems, particularly in the case of imperviousness grouting since after treatment is completed, free spaces filled only with water may remain, resulting in a higher overall permeability. However, bleeding can be eliminated by using a multi-phase injection technique. Bleeding is measured in terms of the ratio of the volume of free water to the initial volume of grout and is usually expressed as a percentage.

Many years of experience in imperviousness grouting have shown that bleeding must not exceed 5% for the treatment to be effective. In fact, bleeding rate is an indication of the overall stability of a grout; at a rate of less than 5% the homogeneity of the grout is acceptable and compatible with an effective grouting treatment.

For this study, the samples used for bleeding measurement were cured in the water bath at 170°F; bleeding was measured after 24 hours of curing on two samples for every grout. The results given in the tables are the average of the two measurements.

A.8 Unconfined Compression Tests

Unconfined compressive strengths are measured according to ASTM standard D2166, "Unconfined Compressive Strength of Cohesive Soil," except that moisture content was not measured. The controlled strain procedure uses a strain rate of 2% per minute for every test.

These tests are performed for each grout at four different curing times (2, 7, 14, and 28 days) on at least three test samples for each time. All samples have a diameter very close to 1-5/8" (41 mm).

The samples are removed from the water bath 2 to 3 hours before testing in order to allow cooling. Before removal from the mold, both ends of each sample are trimmed so that each are flat and parallel.

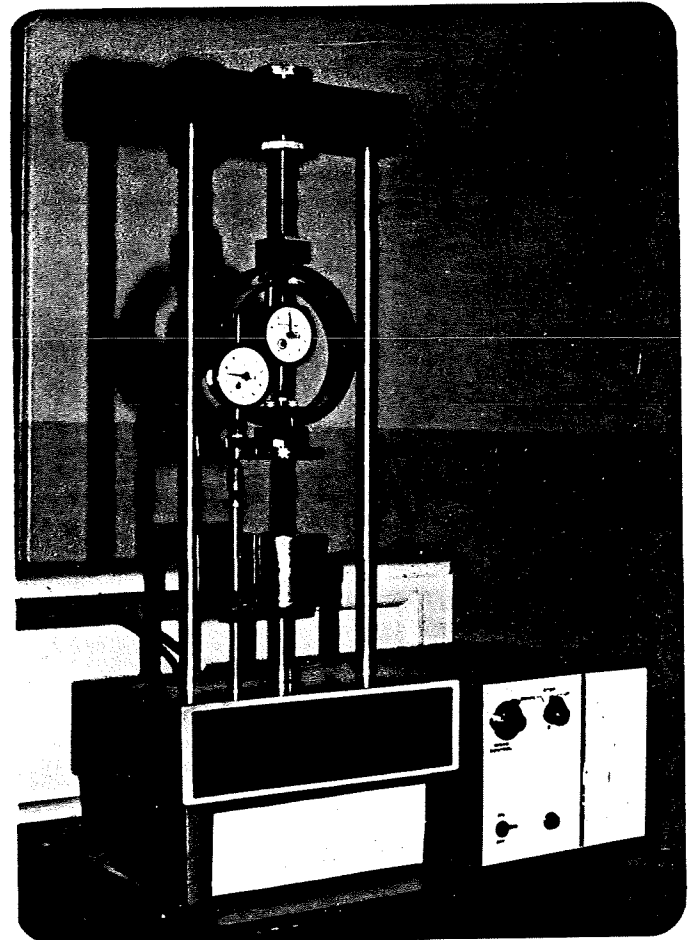
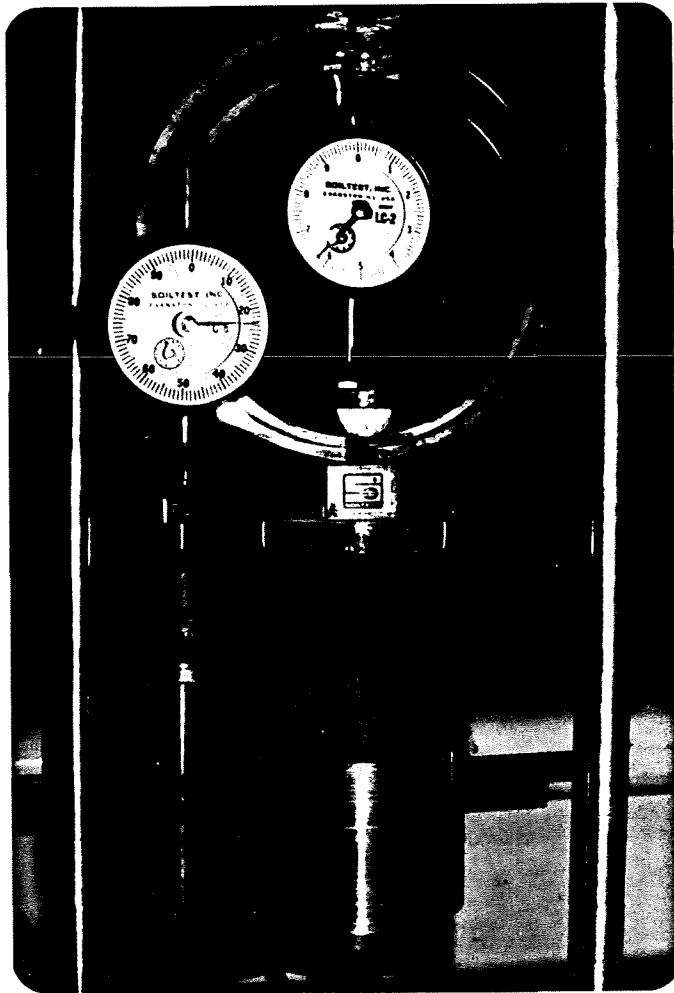


Figure A-3. Unconfined Compression Tests

A.9 Permeability Test

The grout permeability is measured by means of a permeability cell shown in Figure A-5. This is a lateral membrane cell type which works on the same basis as triaxial cells.

The grout test sample, 76 mm (3 inches) in diameter and 108 mm (4½ inches) high is placed inside a rubber sleeve which acts as an impermeable membrane between lateral pressure water and percolation water. The sample is put in a transparent plastic tube which is able to sustain a 5×10^5 Pa (72 psi) inside pressure. The unit is placed between two end plates which are secured together by means of three bolts.

A pressurized stainless steel bottle (Johnny) feeds the cell with percolation water, while another provides the lateral pressure water. Both Johnys are equipped with pressure regulators and pressure gauges. The percolation water is sent through the top end plate, permeates the grout sample and flows out of the cell through the bottom end plate. The volume of percolated water is carefully measured regularly.

The lateral pressure through the membrane prevents the percolation from flowing along the sample walls. In order to assure this, the lateral pressure must be maintained at least at 0.5×10^5 Pa (7 psi) higher than the percolation pressure.

The calculation of permeability coefficient K (cm/s) is made according to Darcy's Law:

$$K = \frac{Q}{SGt}$$

where:

Q = flow of water within the time t (cm³)
 t = time of test(s)
 S = section area of test sample (cm²)
 G = hydraulic gradient

A.10 Triaxial Shear Test

The consolidated drained triaxial tests were performed according to the standard AASHTO T234-74 "Strength Parameters of Soils by Triaxial Compression".

As the laboratory in CSMRI had no triaxial equipment, these tests were subcontracted by Woodward-Clyde (refer to Appendix C for more details).

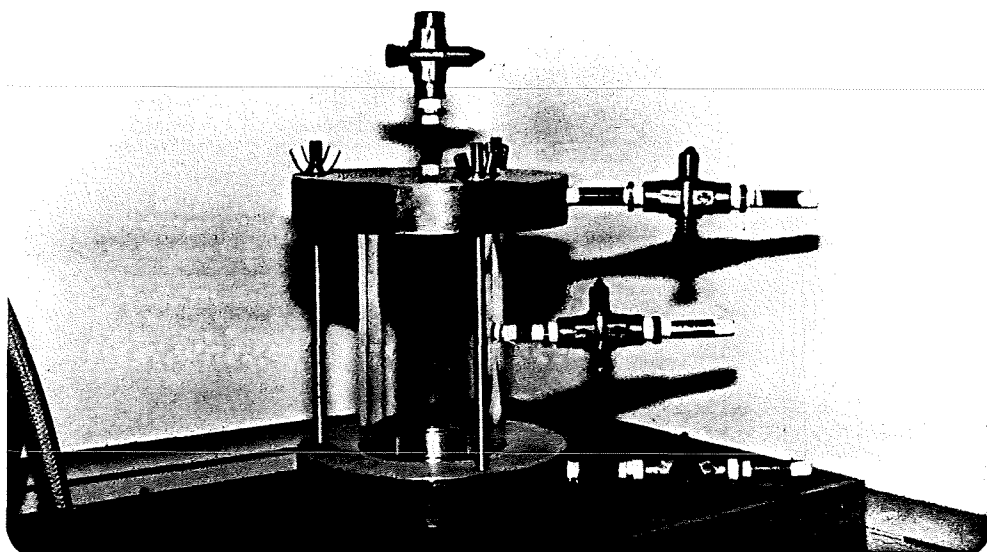
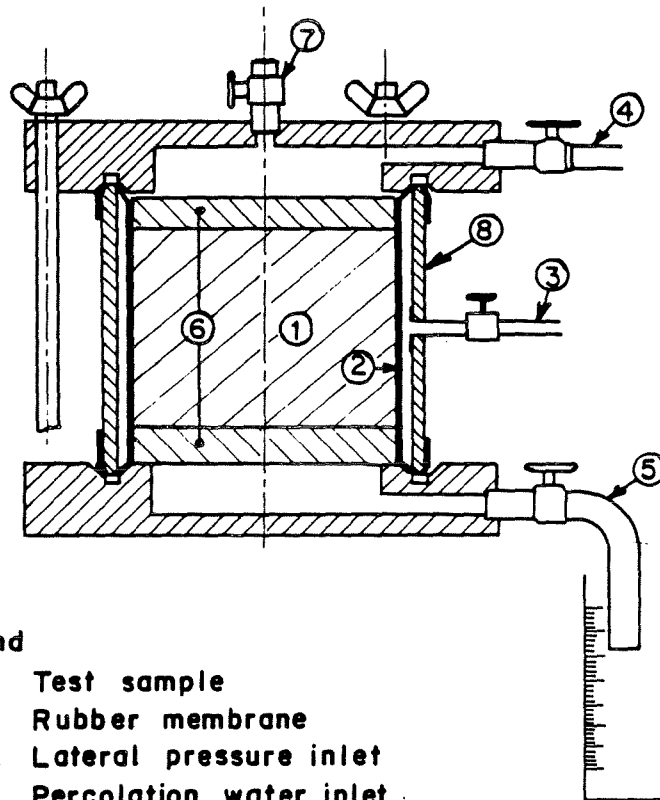


Figure A-4. Equipment for Permeability Test



Legend

- 1 . Test sample
- 2 . Rubber membrane
- 3 . Lateral pressure inlet
- 4 . Percolation water inlet
- 5 . Percolation water outlet
- 6 . Porous stones
- 7 . Blow-off cock
- 8 . Transparent plastic cylinder

FIGURE A5 .— Permeability cell

A.11 One Dimensional Consolidation Test

This test also called Oedometer test was performed according to the standard method of test ASTM D2435-70 and ASSHTO T216-74 "One Dimensional Consolidation Properties of Soils".

These tests were also carried out by the same subcontractor who performed the triaxial tests.

Appendix B

CONVERSION UNIT TABLES

B.1 Usual Units

Length

	m	cm	inch	foot
1 m	1	100	39.37	3.2808
1 cm	0.01	1	0.3937	0.0328
1 inch	0.0254	2.54	1	0.0833
1 foot	0.3044	30.48	12	1

Area

	m ²	cm ²	in ²	ft ²	yd ²
1 m ²	1	1x10 ⁴	1,550	10.764	1.1960
1 cm ²	1x10 ⁻⁴	1	0.1550	1.076x10 ⁻³	1.196x10 ⁻⁴
1 in ²	6.45x10 ⁻⁴	6.4515	1	0.00694	7.716x10 ⁻⁴
1 ft ²	0.0929	929.03	144	1	0.11111
1 yd ²	0.8361	8,361.3	1,296	9	1

Volume

	m ³	cm ³	in ³	ft ³	yd ³
1 m ³	1	1x10 ⁶	61,023.7	35.3147	1.30795
1 cm ³	1x10 ⁻⁶	1	0.061024	3.53x10 ⁻⁵	1.308x10 ⁻⁶
1 in ³	1.638x10 ⁻⁵	16.3871	1	5.787x10 ⁻⁴	2.143x10 ⁻⁵
1 ft ³	0.02832	28,316.8	1,728	1	0.037037
1 yd ³	0.76455	7.645x10 ⁵	46,656	27	1

Forces

	N	kgf	metric tons	lb	short tons
1 N	1	0.10197	1.020×10^{-4}	0.22481	1.124×10^{-4}
1 kgf	9.80665	1	0.001	2.20462	0.001102
1 met. ton	9,806.65	1,000	1	2,204.62	1.10231
1 lb	4.4482	0.45359	4.536×10^{-4}	1	5×10^{-4}
1 sh. ton	8,896.4	907.18	0.90718	2,000	1

Pressure - Strength

	10^5 Pa	bar	kgf/cm ²	psi	sh. ton/ft ²
1 $\times 10^5$ Pa	1	1	1.019716	14.5037	1.04427
1 bar	1	1	1.019716	14.5037	1.04427
1 kgf/cm ²	0.980665	0.980665	1	14.2234	1.02408
1 psi	0.068948	0.068948	0.070307	1	0.072
1 sh. ton/ft ²	0.957604	0.957600	0.97648	13.8889	1

B.2 Specific Units

Ash content

The amount of ash in the grout is expressed in grams per liter of grout (g/l) which is equivalent to kilograms per cubic meter (kg/m³).

g/l	lb/yd ³	$\frac{\text{water}}{\text{ash}}$ % by weight
100	168.5	963
700	1,179.9	105.8
800	1,348.4	88.0
850	1,432.7	80.6
900	1,517.0	74.1
950	1,601.3	68.2
1000	1,685.0	63.0

Additive concentration

g/l or kg/m ³	lbs/cu yard	cm ³ /l or l/m ³	gal/cu yard
1	1.6856	1	0.202
2	3.37	2	0.404
3	5.06	3	0.606
4	6.74	4	0.808
5	8.43	5	1.010
6	10.11	10	2.020
7	11.80	15	3.030
8	13.48	20	4.040
9	15.17	25	5.050
10	16.85		
15	25.28		
20	33.71		

Temperature

$$^{\circ}\text{F} = (^{\circ}\text{C} \times \frac{9}{5}) + 32$$

$$^{\circ}\text{C} = (^{\circ}\text{F} - 32) \times \frac{5}{9}$$

°C	0	20	60	70	80	90	100
°F	32	68	140	158	176	194	212

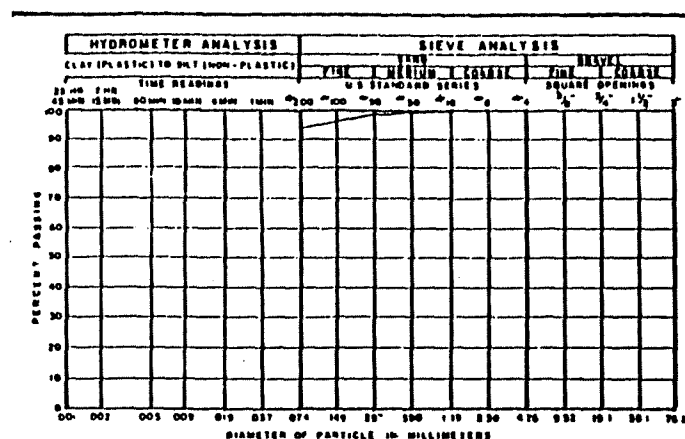
°F	0	100	150	160	170	180	190	200
°C	-18	37.8	65.6	17.1	76.7	82.2	87.8	93.3

Permeability

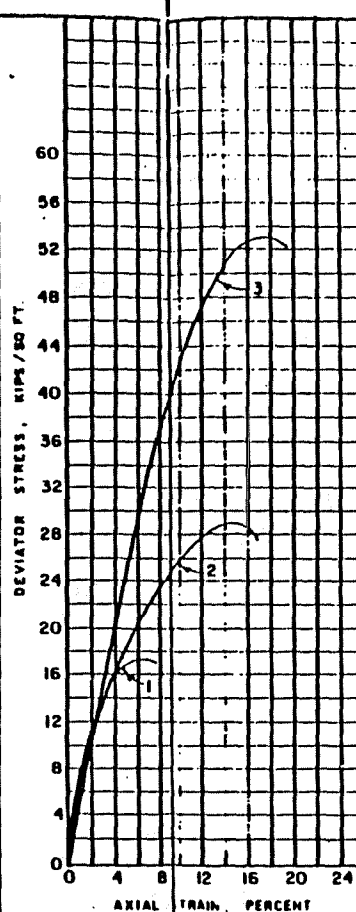
$$1 \text{ } 10^{-7} \text{ cm/s} = 0.10346 \text{ ft/year} = 1.241 \text{ inch/year}$$

Appendix C

TRIAXIAL, CONSOLIDATION AND PRESSUREMETER TEST RESULTS

SOIL CLASSIFICATION GROUTLL = % PI NON-PLASTIC % SPECIFIC GRAVITY 2.65

	TEST NO.	1			
		1	2	3	
INITIAL	WATER CONTENT, w_o %	71.5	70.4	71.5	
	DRY DENSITY LBS./CU. FT.	56.7	57.0	56.6	
	VOID RATIO, e_o	1.915	1.903	1.922	
BEFORE TEST	SATURATION, s_o %	98.9	98.0	98.6	
	WC AFTER SATURATION, w_s %				
	SATURATION, s %				
AT FAILURE	CONSOL. PRESSURE K/SQ. FT.	3.60	7.20	14.40	
	WC AFTER CONSOL., w_c %				
	VOID RATIO AFTER CONSOL., e_c				
AT FAILURE	MAJ. PRIN. STRESS, σ_1 K/SQ. FT.	21.25	36.18	67.33	
	MIN PRIN. STRESS, σ_{III} K/SQ. FT.	3.60	7.20	14.40	
	PORE PRESSURE, u K/SQ. FT.	0	0	0	
AT FAILURE	WATER CONTENT, w %	65.6	56.2	54.6	
	VOID RATIO, e				
SPECIMEN DIAMETER INCHES		1.64	1.64	1.64	
INITIAL HEIGHT INCHES		3.30	2.96	3.50	
TEST TIME TO FAILURE MINUTES		159	225	425	



TYPE OF TEST

- ☐ UNCONSOLIDATED
UNDRAINED
- ☐ CONSOLIDATED
UNDRAINED
- ☒ CONSOLIDATED
DRAINED

- ☒ CONSTANT RATE OF STRAIN
_____.001 INCHES/MINUTE
- ☐ CONSTANT RATE OF STRESS
_____. K/SQ. FT./MINUTE

TYPE OF SPECIMEN

- ☐ NATURAL _____ % OF ASTM
- ☒ REMOLDED OPTIMUM MOISTURE _____ %
- SAMPLE TYPE PREPARED BY CLIENT

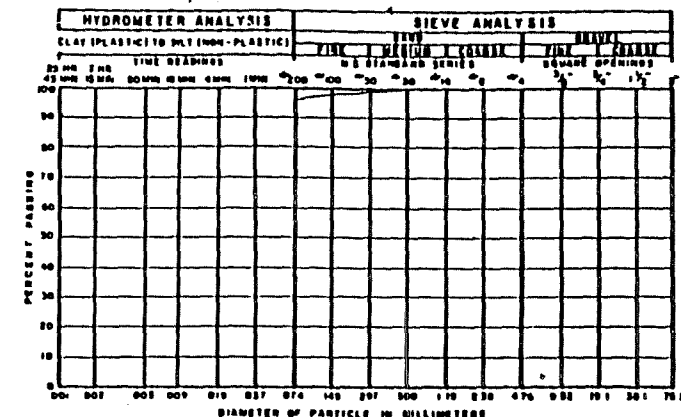
TRIAXIAL SHEAR TEST RESULT
EFFECTIVE STRESS TEST

Job No. 20307

Prepared By: D.J.P.

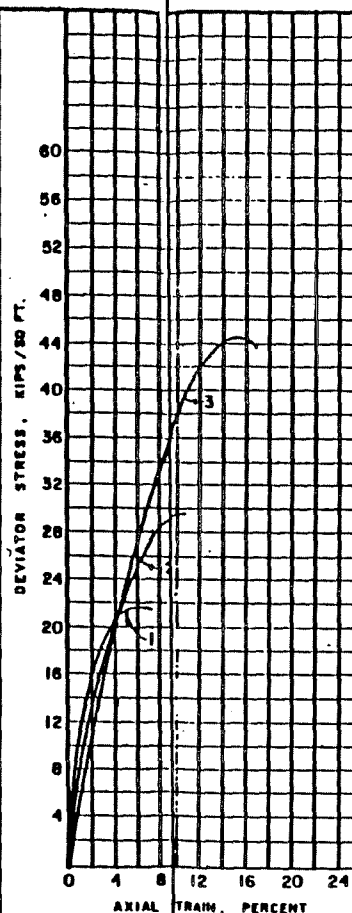
Date 1/22/81

FIG

SOIL CLASSIFICATION GROUT

.L = _____ % PI NON-PLASTIC % SPECIFIC GRAVITY 2.65

TEST NO.		1	2	3
INITIAL	WATER CONTENT, w_o %	71.2	71.2	71.3
	DRY DENSITY LBS./CU. FT.	57.2	57.3	56.7
	VOID RATIO, e_o	1.893	1.884	1.915
SATURATION	SATURATION, S_o %	99.7	100.0	98.7
	W.C. AFTER SATURATION, w_s %			
	SATURATION, S %			
CONSOLIDATION	CONSOL. PRESSURE K/SQ.FT.	3.60	7.20	14.40
	W.C. AFTER CONSOL., w_c %			
	VOID RATIO AFTER CONSOL., e_s			
STRESS	MAJ. PRIN. STRESS, σ'_1 K/SQ.FT.	25.33	36.88	58.81
	MIN. PRIN. STRESS, σ'_{III} K/SQ.FT.	3.60	7.20	14.40
	PORE PRESSURE, u K/SQ.FT.	0	0	0
FAILURE	WATER CONTENT, w_f %	62.2	57.6	49.8
	VOID RATIO, e_f			
SPECIMEN DIAMETER INCHES		1.64	1.64	1.64
INITIAL HEIGHT INCHES		3.43	2.85	2.45
TEST TIME TO FAILURE MINUTES		212	283	416



REMARKS : FAILURE CRITERIA: MAXIMUM STRESS DIFFERENCE.

SAMPLE NO. 2 AT DEPTH _____
COMPOSITE _____

TYPE OF TEST	TEST NO.	TEST DATE	TESTER	TEST RESULTS
1. Visual Inspection	1	10/10/2023	J. Smith	Pass
2. Tensile Test	2	10/10/2023	J. Smith	Pass
3. Impact Test	3	10/10/2023	J. Smith	Pass
4. Hardness Test	4	10/10/2023	J. Smith	Pass
5. Corrosion Test	5	10/10/2023	J. Smith	Pass
6. Fatigue Test	6	10/10/2023	J. Smith	Pass
7. Creep Test	7	10/10/2023	J. Smith	Pass
8. Fracture Test	8	10/10/2023	J. Smith	Pass
9. Torsion Test	9	10/10/2023	J. Smith	Pass
10. Compression Test	10	10/10/2023	J. Smith	Pass

☐	UNCONSOLIDATED UNDRAINED
☐	CONSOLIDATED UNDRAINED
☒	CONSOLIDATED DRAINED

☒ CONSTANT RATE OF STRAIN
_____ .001 _____ INCHES/MINUTE

☐ CONSTANT RATE OF STRESS
_____ K/SQ.FT./MINUTE

TYPE OF SPECIMEN

☐ NATURAL _____ % OF ASTM _____
☒ REMOLDED OPTIMUM MOISTURE: _____ %
 SAMPLE TYPE: _____ PREPARED BY CLIENT _____

TRIAxIAL SHEAR TEST RESULT.
EFFECTIVE STRESS TEST

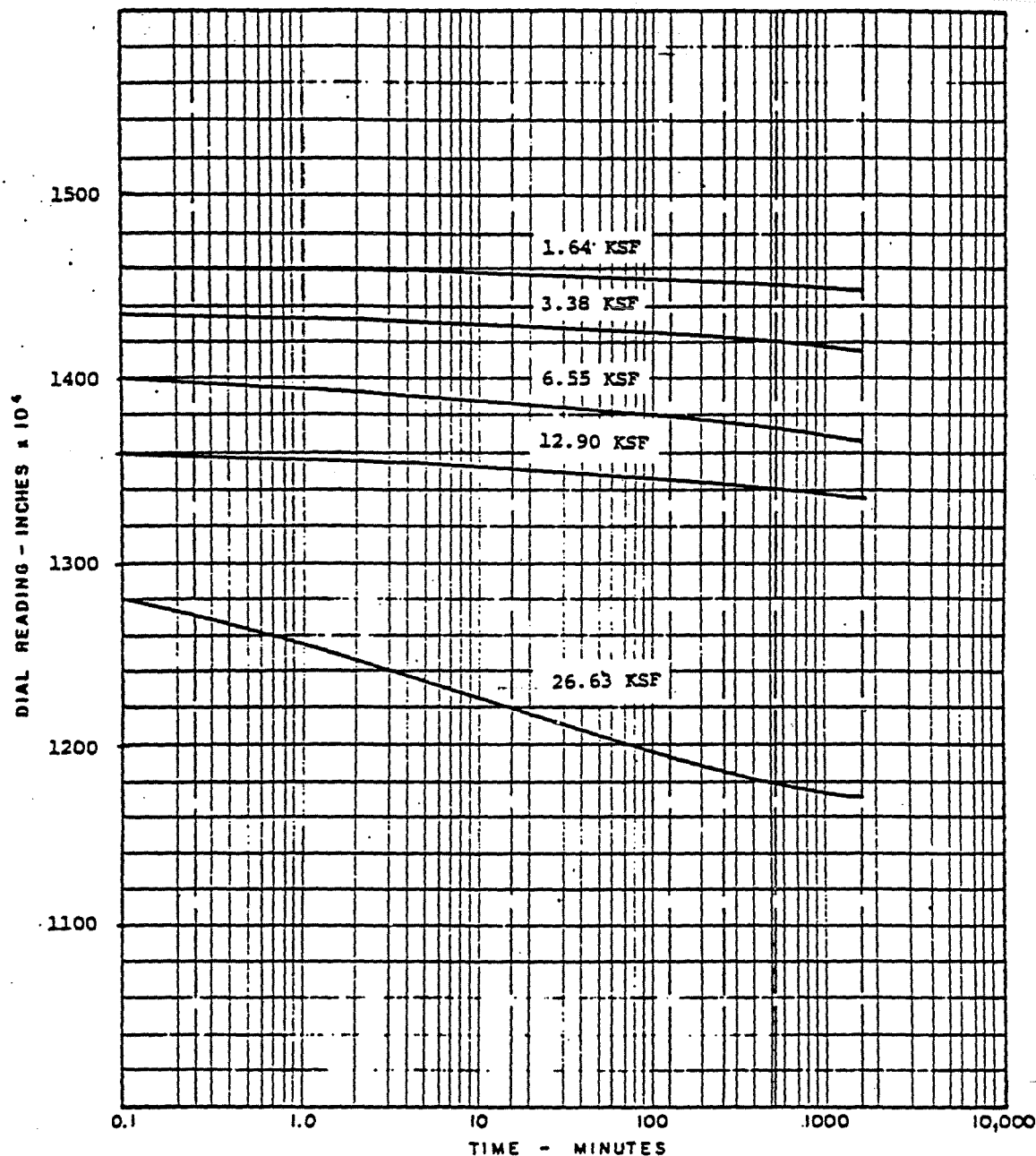
JOB NO 20307

Prepared by: D J.P

Date. 1/22/01

FIG. 9

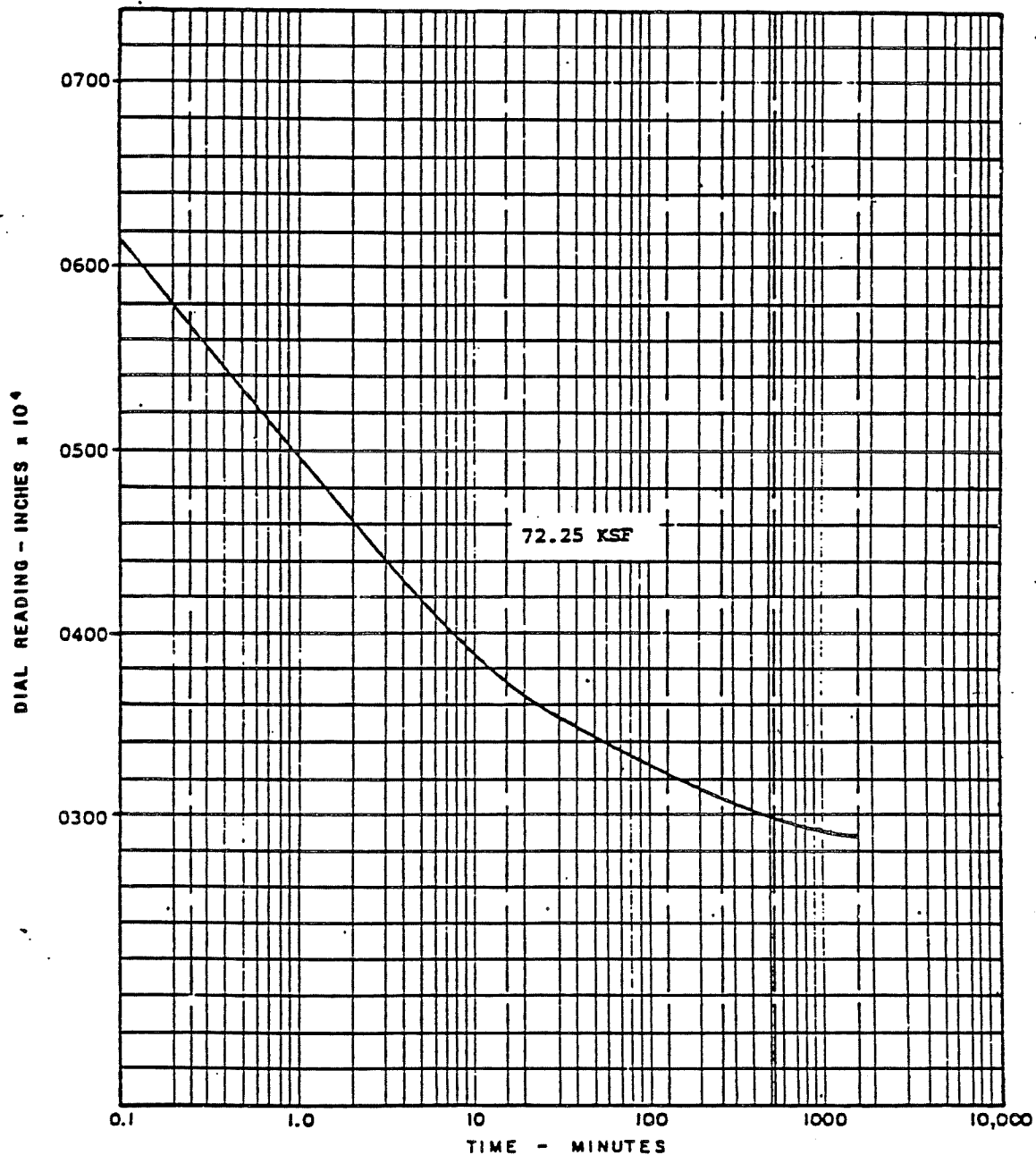
WOODWARD-CLYDE CONSULTANTS
CONSULTING ENGINEERS, GEOLOGISTS AND ENVIRONMENTAL SCIENTISTS
ROCKY MOUNTAIN REGION



SAMPLE FROM HOLE Sample No. 2 DEPTH _____
 DESCRIPTION Grout
 INITIAL DRY DENSITY 59.9 PCF MOISTURE 61.8 %

TIME-CONSOLIDATION CURVES

WOODWARD-CLYDE CONSULTANTS
CONSULTING ENGINEERS, GEOLOGISTS AND ENVIRONMENTAL SCIENTISTS
ROCKY MOUNTAIN REGION

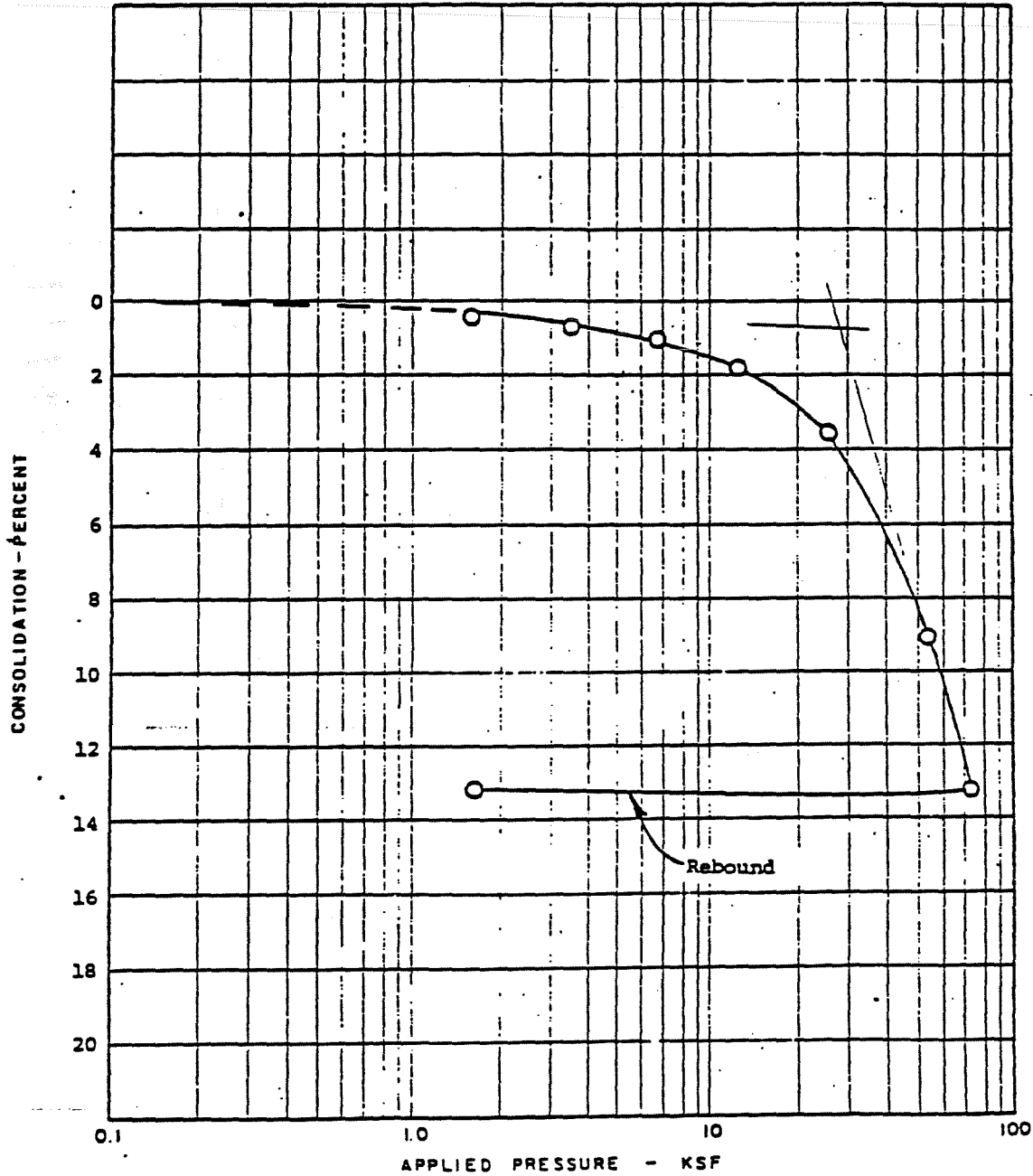


SAMPLE FROM HOLE Sample No. 1 DEPTH _____
DESCRIPTION Grout
INITIAL DRY DENSITY 55.5 PCF MOISTURE 63.5 %

TIME-CONSOLIDATION CURVES

JOB NO. 20307

WOODWARD CLYDE CONSULTANTS
CONSULTING ENGINEERS, GEOLOGISTS AND ENVIRONMENTAL SCIENTISTS
ROCKY MOUNTAIN REGION



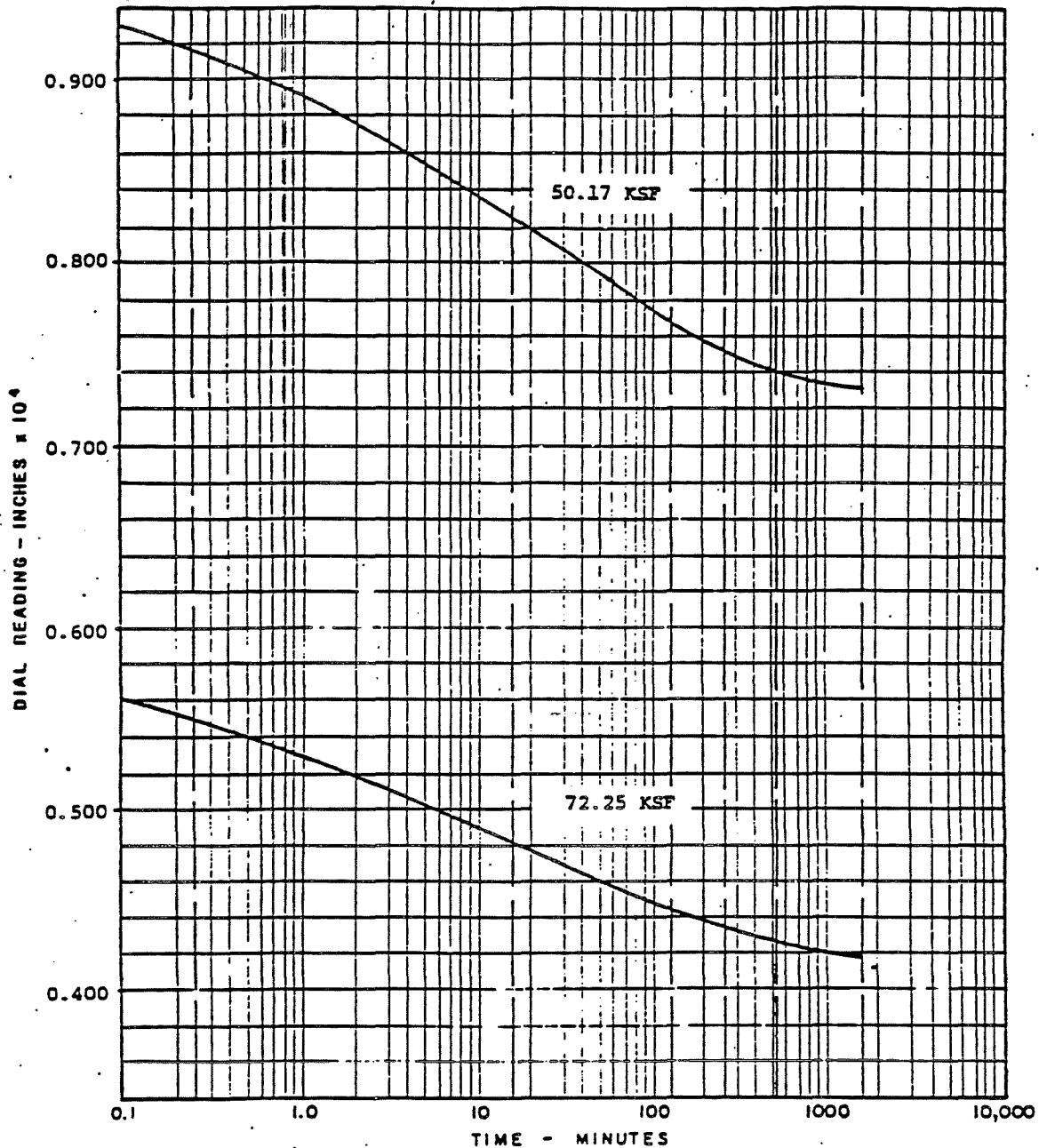
SAMPLE FROM HOLE Sample No. 2 DEPTH _____
DESCRIPTION Grout
INITIAL DRY DENSITY 59.9 PCF MOISTURE 61.8 %

SWELL - CONSOLIDATION TEST RESULTS

JOB NO. 20307

FIG.

WOODWARD-CLYDE CONSULTANTS
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ROCKY MOUNTAIN REGION



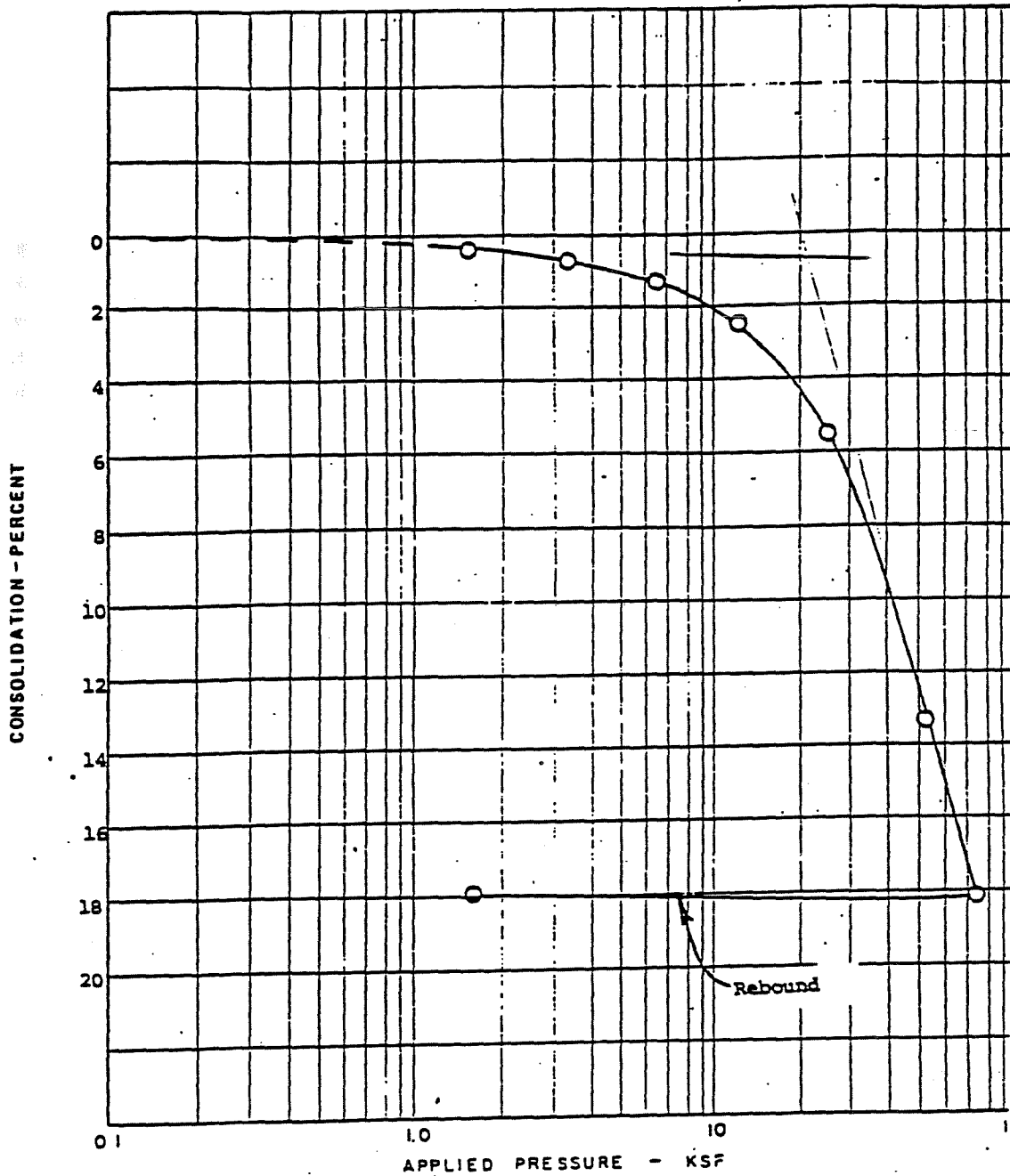
SAMPLE FROM HOLE Sample No. 2 DEPTH _____
DESCRIPTION Grout
INITIAL DRY DENSITY 59.9 PCF MOISTURE 61.8 %

TIME-CONSOLIDATION CURVES

JOB NO. 20307

FIG.

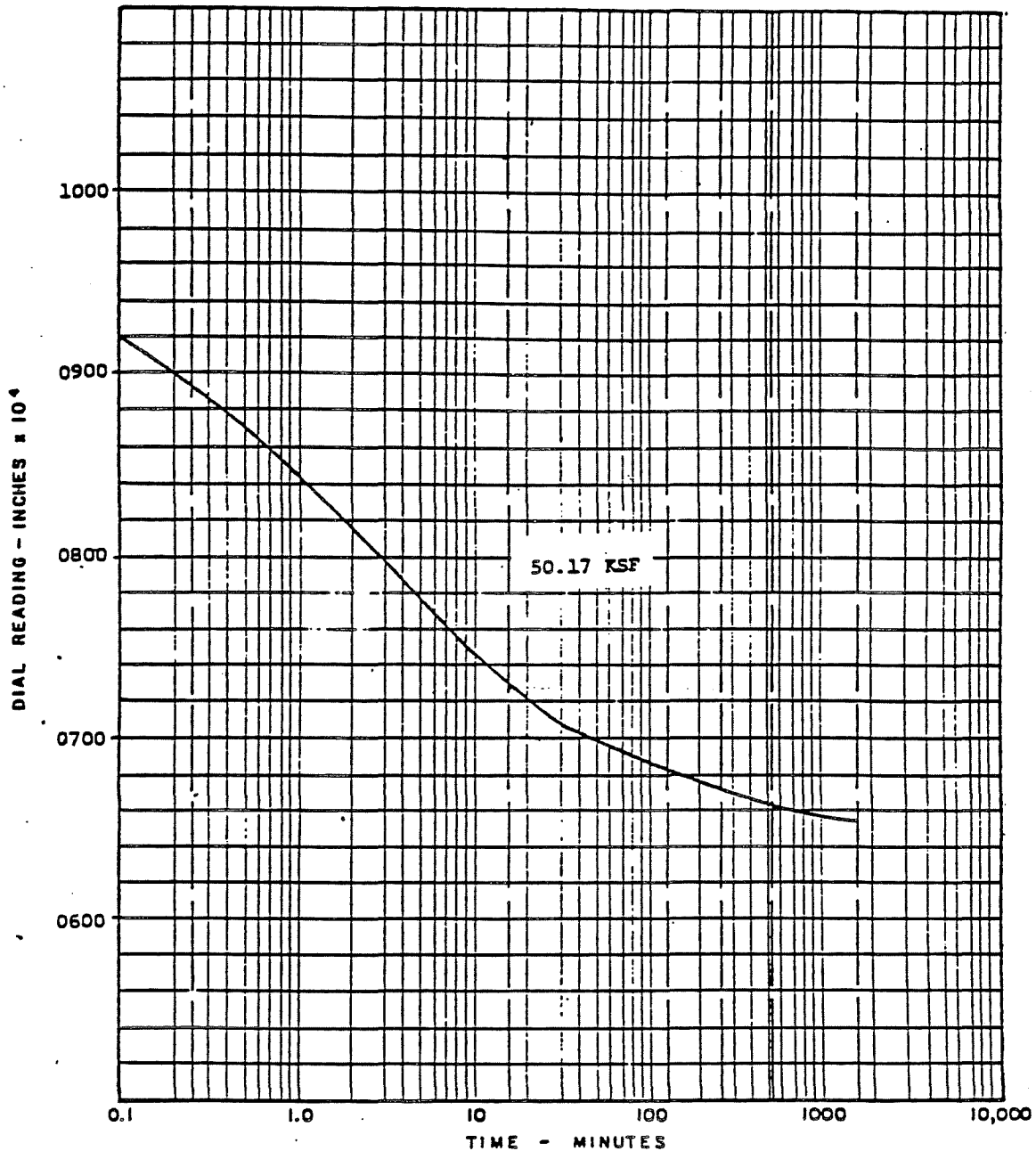
WOODWARD CLYDE CONSULTANTS
CONSULTING ENGINEERS, GEOLOGISTS AND ENVIRONMENTAL SCIENTISTS
ROCKY MOUNTAIN REGION



SAMPLE FROM HOLE Sample No. 1 DEPTH _____
DESCRIPTION Grout
INITIAL DRY DENSITY 55.5 PCF MOISTURE 63.5 %

SWELL - CONSOLIDATION TEST RESULTS

WOODWARD-CLYDE CONSULTANTS
CONSULTING ENGINEERS, GEOLOGISTS AND ENVIRONMENTAL SCIENTISTS
ROCKY MOUNTAIN REGION



SAMPLE FROM HOLE Sample No. 1 DEPTH _____
DESCRIPTION Grout
INITIAL DRY DENSITY 55.5 PCF MOISTURE 63.5 %

TIME-CONSOLIDATION CURVES

JOB NO. 20307

11 East Adams Street
Suite 1500
Chicago, Illinois 60603
312-939-1000
Telex 253875 (WOODWARD CGO)

Woodward-Clyde Consultants

REPORT

PRESSUREMETER TESTING

CSMRI OIL SHALE RESEARCH GOLDEN, COLORADO

Prepared for:

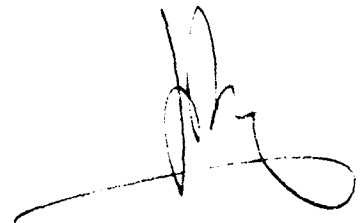
Dr Dominique Namy and Mr Paul Bissery
Soletanche and Rodio, Inc.
McLean, Virginia

By:



Richard R. Davidson, P.E.

and



Jean-Yves Perez, P.E.

13 February, 1981
81C201



1. INTRODUCTION

This report presents the results of seven pressuremeter tests run in two steel vessels filled with grouted rockfill at the Colorado School of Mines Research Institute (CSMRI). The pressuremeter testing was performed by Woodward-Clyde Consultants for Soletanche and Rodio, Inc. on January 13 and 14, 1981.

The rockfill samples were created to model spent oil shale rock after retorting had been completed. To increase strength and decrease permeability the spent oil shale was injected with a grout made from a by-product of the retorting process. Two types of grout were injected, one with a small percentage of cement added (0.3 percent by volume) and the other with no cement. The two steel test vessels were constructed 9 ft high and 5 ft in diameter to hold approximately 6 yd³ of material, see Fig. 1. The spent oil shale material was a well graded mix with particle sizes ranging from silt size to 4 inches. The grout was injected under a maximum pressure of 200 lb/in² at the top, middle and bottom of the vessel.

The two grouted samples were cured in the test vessels for 28 days at a temperature of 170°F to simulate adjacent in situ retorts. The first test sample which did not have cement added to the grout was thoroughly injected such that the entire sample was grouted. The second sample grouting was less complete as small portions of the rockfill were not infiltrated by the grout. At the time the pressuremeter tests were performed the first sample (without cement) was 5 months old and the second sample (with cement) was 3 months old.

The objective of the pressuremeter testing program was to determine the strength and compressibility properties of the grouted oil shale rockfill in the steel vessels. Our interpretations of the shear strength and elastic modulus parameters derived from the pressuremeter tests are presented and discussed in this report.

2. PRESSUREMETER TESTING PROGRAM

Pressuremeter tests were performed in three borings drilled vertically into the grouted rock samples from the top of the steel vessels. One boring was performed in the first sample (without cement) and two borings were drilled in the second sample (with cement) at the locations shown in Fig. 2. The WCC Menard GA pressuremeter system was used for the testing.

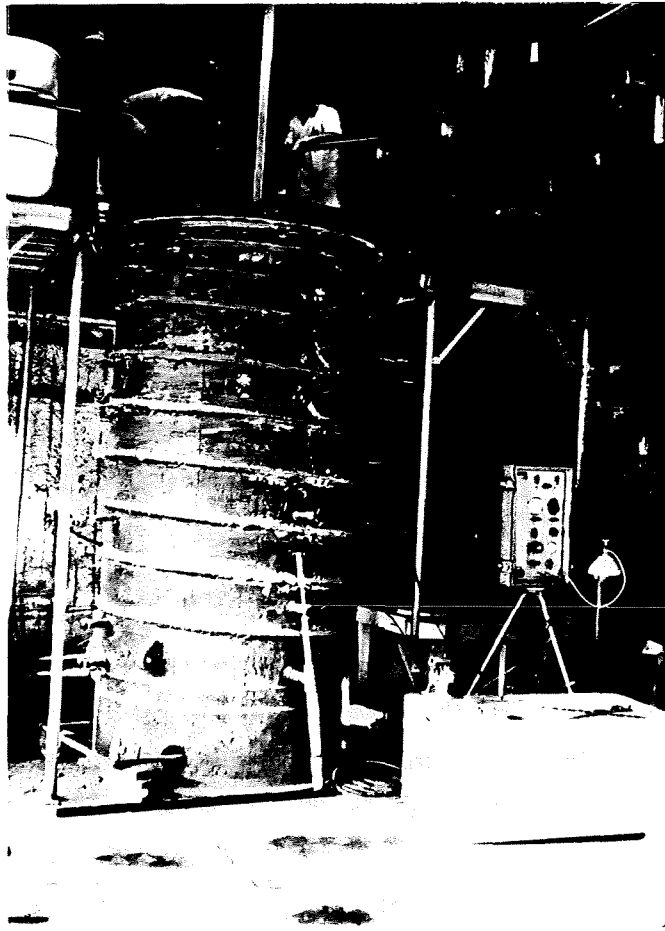


Fig. 1 GROUTED ROCKFILL VESSELS AT CSMRI

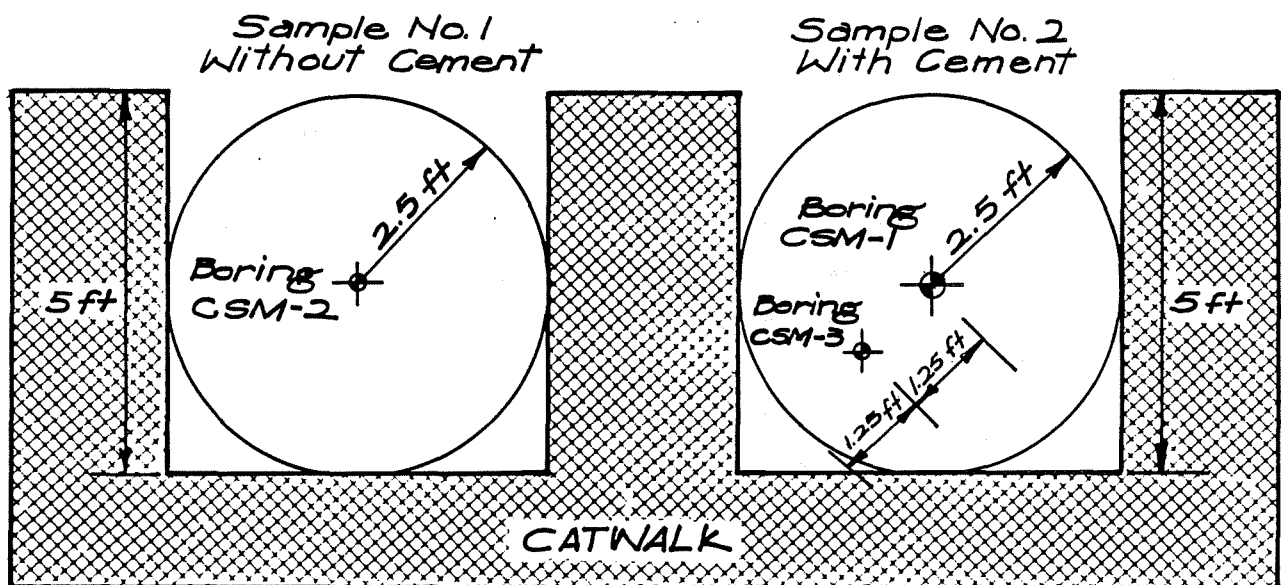


Fig. 2 BORING LOCATION PLAN

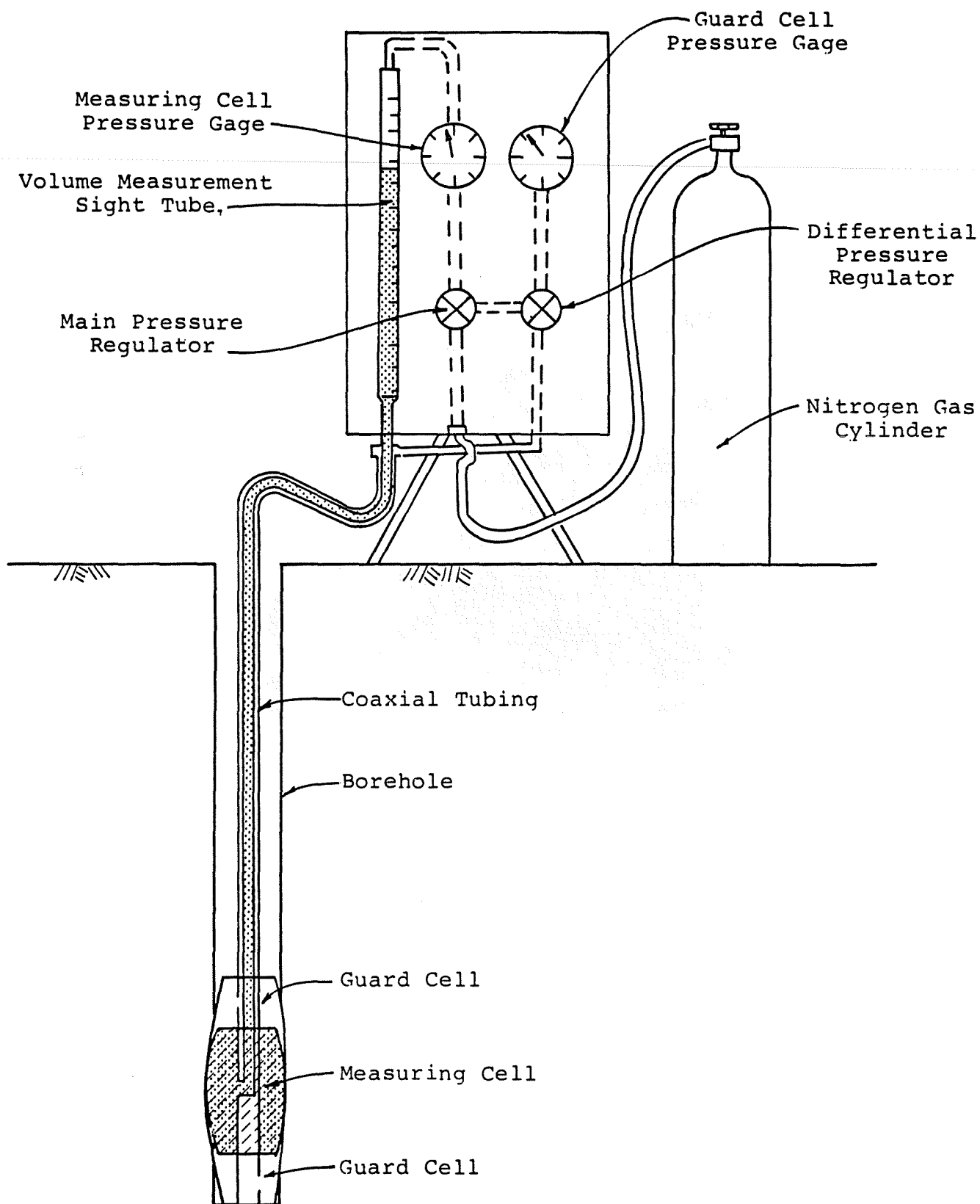
As shown by Figure 3, the pressuremeter apparatus consists of a probe, a volume measurement and pressure-control instrument, and a gas-supply tank. The cylindrical probe contains an expandable rubber membrane (measuring cell) and two contiguous independently expandable guard cells. The measurement system and the measurement cell was filled with water and the volume change induced in the measuring cell was measured by the sight tube volumeter. Expansion of the measuring cell was controlled by applying gas pressure from the gas-supply tank to the fluid column through a pressure regulator system. A differential pressure regulator maintained a constant differential pressure of one bar between the fluid pressure in the measuring cell and the gas pressure in the guard cell at all times during the test.

Immediately after the test hole was prepared, the probe was lowered to the designated testing depth. The test was performed from the catwalk above the rock vessels (see Fig. 4) by expanding the probe in equal pressure increments and measuring the change in volume of the measuring cell with time for each increment. For a given pressure, volume readings were taken at 15, 30, and 60 seconds.

Boring CSM-1 was drilled in the rock sample that was grouted with the cementitious grout (left chamber in Fig. 1) using a 3-inch diameter diamond bit concrete coring machine, see Fig. 5. The hole was drilled along the longitudinal centerline axis of the test vessel with a heavy stream of water to lubricate the coring action and wash the cuttings to the surface. The flushing water apparently eroded away the rockfill around the core barrel and disintegrated the sample within the barrel. As a result very little intact specimens were recovered from the core barrel and the resulting hole size significantly exceeded the size of the 2 7/8-inch diameter pressuremeter probe. The erosive action of the drilling water also disturbed the rockfill along the borehole walls. Three pressuremeter tests were performed in boring CSM-1, however their reliability is questionable because of the excessive hole size and drilling disturbance.

Boring CSM-2 was performed in the test sample that was grouted without cement. In boring CSM-2, the drilling procedure was changed to minimize disturbance to the borehole walls and maintain a borehole size the same as the pressuremeter probe. A smaller size core barrel with an outside diameter of 2 3/8-inch was utilized which was exactly the same diameter as a smaller 2 3/8-inch diameter pressuremeter probe.¹ For

¹A 2 3/8-inch diameter roller bit was also assembled as a back-up in case the coring operation caused excessive disturbance.



**MENARD GA PRESSUREMETER TEST
APPARATUS SCHEMATIC**

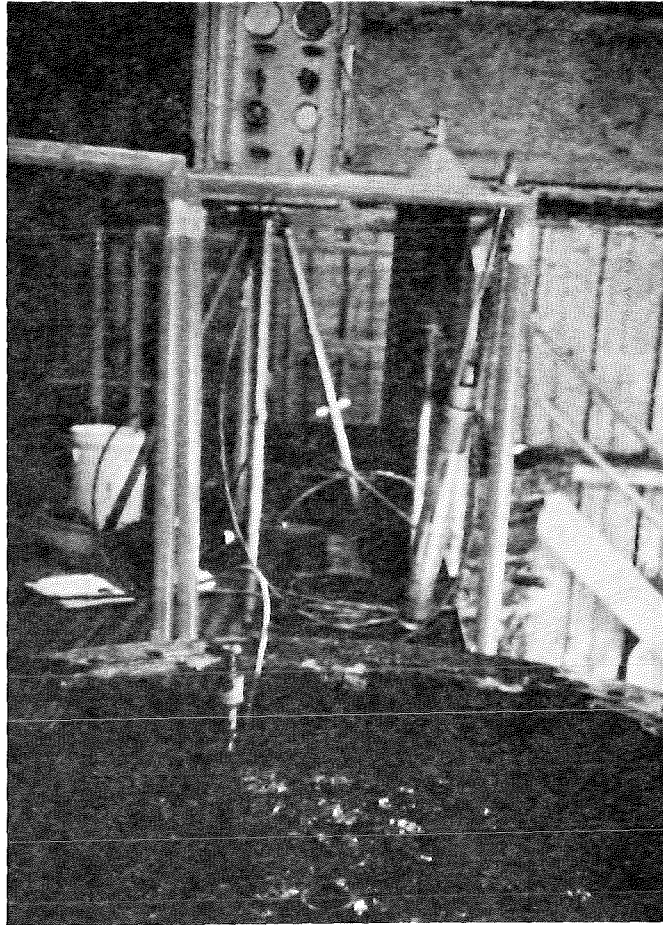


Fig. 4 PERFORMANCE OF PRESSUREMETER TEST
FROM CATWALK ABOVE GROUTED ROCKFILL
VESSEL

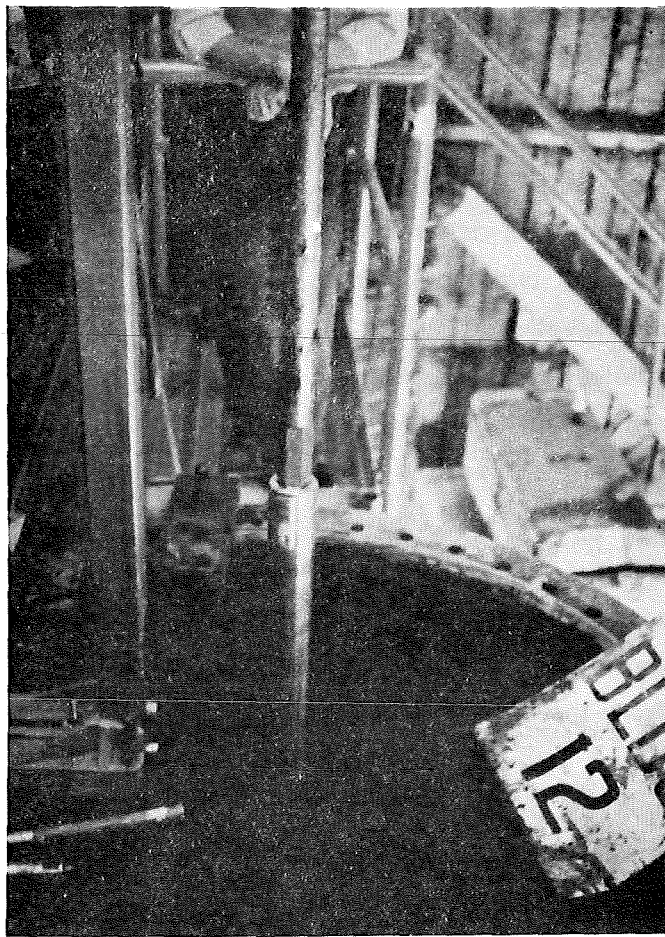


Fig. 5 DIAMOND BIT CONCRETE CORING MACHINE
USED TO DRILL PRESSUREMETER HOLE

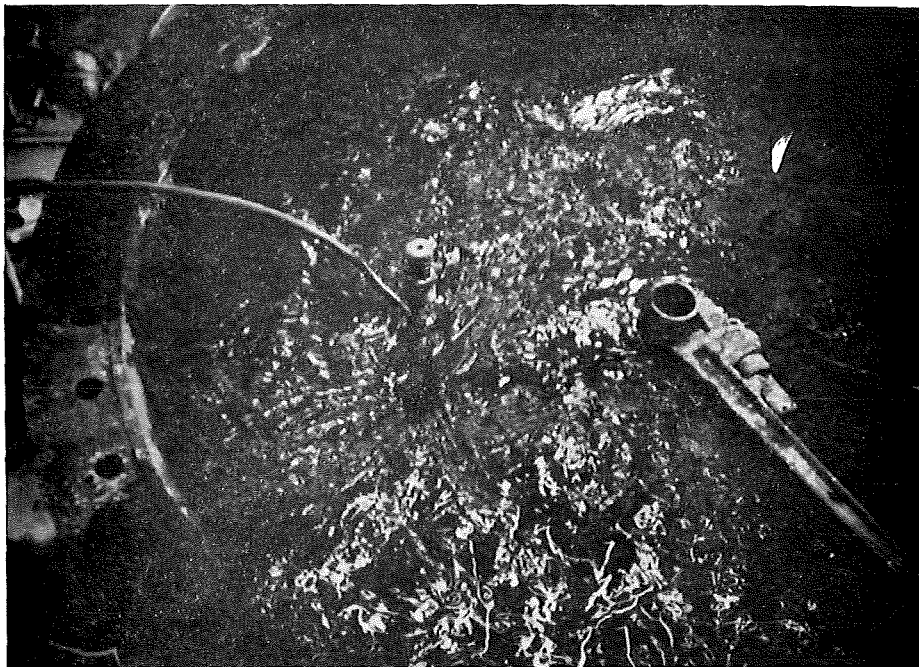


Fig. 6 LOCATION OF BORING CSM-3 BETWEEN THE
BOREHOLE FOR BORING CSM-1 AND THE WALL
OF THE VESSEL

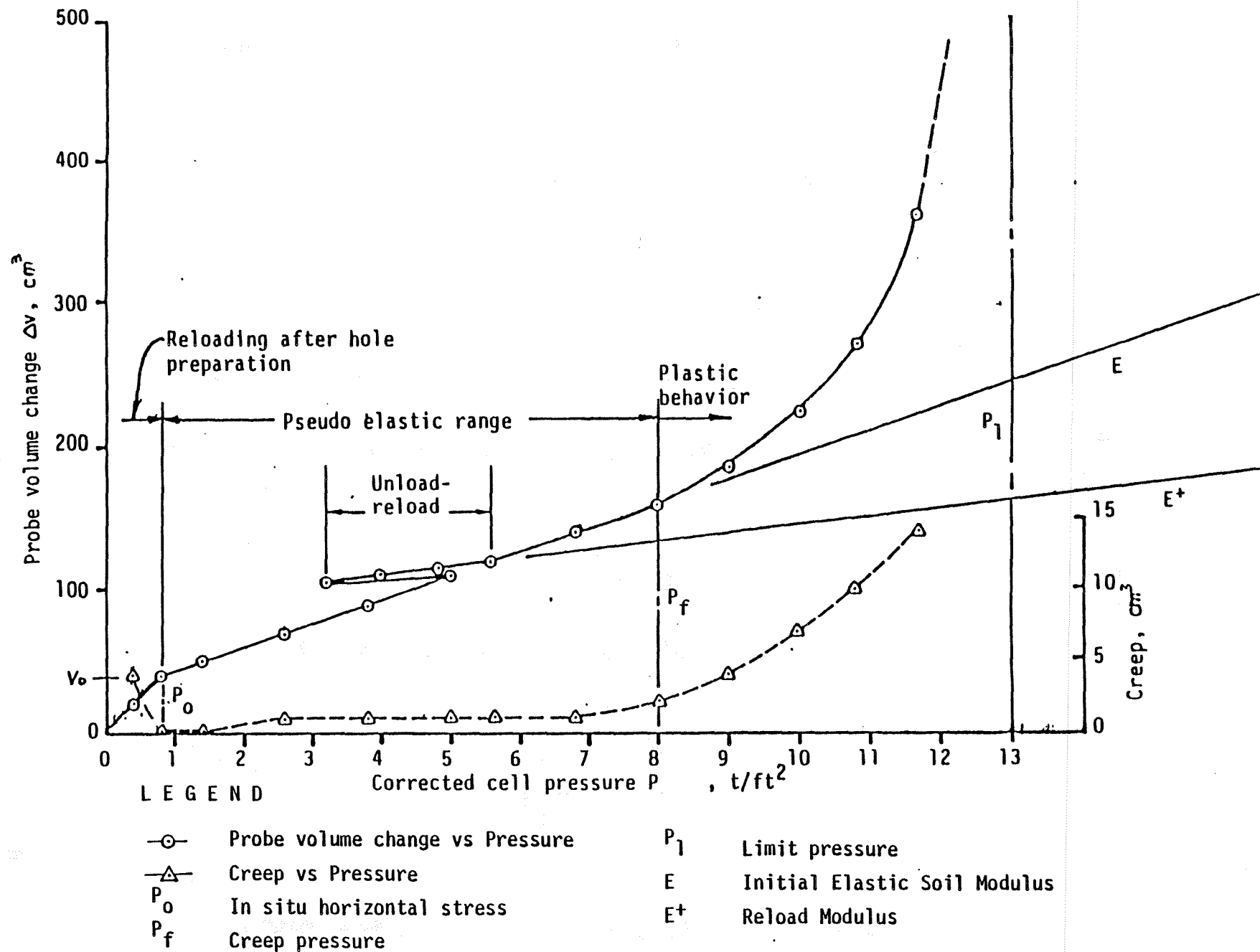
the first pressuremeter test no drilling fluid was used, but fluid was used for the other two test holes in this boring. Instead of drilling with water, a thick bentonite stabilizing fluid was used to stabilize the hole and lubricate the coring action. The flow of fluid was maintained at the minimum level required for smooth drilling without burning up the core barrel bit. Only 3 ft of hole was cored prior to each test to minimize the time between drilling and testing when the borehole walls are unloaded and can yield. The drilling procedure used in boring CSM-2 produced a tight hole with minimal disturbance to the borehole wall.

The excellent success of the bentonite coring procedure in boring CSM-2 stimulated a second boring to be performed in the cement grouted rockfill sample. Boring CSM-3 was drilled equidistant from the borehole for boring CSM-1 and the wall of the test vessel, as shown in Fig. 2 and 6. A 3-inch diameter rigid schedule 80 steel pipe was placed in the CSM-1 borehole to minimize stress relief in the area of the borehole. Only one pressuremeter test was performed in boring CSM-3 because only 3 ft of pipe was available to install in the open borehole (CSM-1). This test, however was of good quality because the hole was tight and borehole wall disturbance was minimal.

3. PRESSUREMETER TEST RESULTS

The results of the pressuremeter tests are presented as a plot of probe volume change versus pressure. A typical pressuremeter test plot is shown in Fig. 7. The pressure volume change curve is typically made up three components: the initial reloading portion - as the probe expands through the borehole, meets the borehole walls and restresses the soil back to its in situ condition; the linear pseudo-elastic portion; and the plastic portion where the soil exhibits substantial nonlinear deformation. An unload-reload cycle may also be included in the pressuremeter test. Pressuremeter test curves from borings CSM-1, 2 and 3 are presented in Appendix A.

Geotechnical parameters interpreted from the pressuremeter test curves are summarized in Table 1. These property interpretations are discussed below.



TYPICAL PRESSUREMETER TEST PLOT

Fig. 7

TABLE 1

SUMMARY OF PRESSUREMETER TEST RESULTS

PROJECT CSMRI Oil Shale Research LOCATION Golden, Colorado NO. 8/C201BORING CSM-1, 2, 3

Boring / Depth ft	σ_{ho} t/ft ²	u t/ft ²	$\bar{\sigma}_{ho}$ t/ft ²	$\bar{\sigma}_v$ t/ft ²	K_0	σ_{hmax} t/ft ²	$\bar{\sigma}_{hmax}$ t/ft ²	OCR	E t/ft ²	E^+ t/ft ²	$\bar{c}$ t/ft ²	C t/ft ²	ϕ °	P_L t/ft ²	Comments
CSM-1 / 2 *	5.8	0	5.8	.14	41	12	12	2.1	155	296	1.3	—	—	14	Cored with water, disturbance
CSM-1 / 4.5 *	.78	0	.78	.32	2.5	3.1	3.1	4.0	68.5	—	1.0	—	—	7	Cored with water, disturbance
CSM-1 / 7.5 *	1.88	0	1.88	.53	3.6	—	—	—	39.7	—	—	—	—	—	Cored with water, excessive hole size
CSM-2 ** / 2	2.8	0	2.8	.14	20	15.7	15.7	5.6	1450	4300	12.0	7.3	20	72	Drilled without mud
CSM-2 ** / 5	3.5	0	3.5	.35	12	19.8	19.8	5.7	790	4300	12.0	7.1	20	72	Drilled with mud
CSM-2 ** / 8	5.9	0	5.9	.56	10	21	21	3.5	1234	5670	12.0	6.5	20	80	Drilled with mud
CSM-3 / 2 *	1.7	0	1.7	.14	12	9.6	9.6	5.5	278	1780 3000	6.0	3.5	20	39	Drilled with mud
Notes:															
* Sample No. 2 (With Cement) ** Sample No. 1 (Without Cement)															

Stress Conditions

The horizontal stress σ_{ho} in the grouted rockfill can be interpreted as the start of the pseudo-elastic portion of the curve following the procedure discussed by Davidson (1979). The measured horizontal stresses of up to 6 t/ft^2 are not too surprising in light of the injection pressures of up to 14 t/ft^2 used for grouting the sample. Some of this grouting pressure was probably locked into the structure of the grouted mass. Similar locked-in horizontal stresses were measured on chemically grouted alluvial sands at Locks and Dam No. 26 near St. Louis, Missouri, see Perez et al (1981). The high overconsolidation ratio OCR values of 3 to 6 were also probably caused by locked-in grouting stress.

Compressibility

Two elastic moduli can be interpreted from the pressuremeter curve, an initial modulus E calculated from the slope of the pseudo-elastic portion of the curve, and a reload modulus E^+ calculated from the unload-reload curve. Research by Merritt et al (1979), Davidson and Perez (1980), Denby et al (1981), and Davidson and Bhushan (1981) has shown that for a wide variety of materials the initial pressuremeter modulus from a good quality test corresponds to the secant modulus at either 50 percent of the peak stress, failure or somewhere between the two. This secant modulus is measured in a good quality laboratory consolidated triaxial test on an undisturbed sample. This research has also indicated that the pressuremeter reload modulus corresponds to a good quality triaxial test initial tangent modulus.

The initial pressuremeter modulus value is strongly dependent on the level of disturbance experienced during hole preparation. For poor quality pressuremeter tests the initial modulus can be grossly underpredicted as was the case for the pressuremeter tests in boring CSM-1. Even in good quality pressuremeter tests, the borehole unloading during hole preparation is not usually totally elastic which thereby reduces the initial pressuremeter modulus below the initial tangent modulus value that would be measured in a triaxial test.

The modulus values from borings CSM-2 and 3 appear to be reliable because the level of hole disturbance was minimal. The initial tangent modulus of the grouted rockfill in the second vessel (no cement in grout) ranged from 4300 to 5600 t/ft^2 , and the secant modulus at 50 to 100 percent of the peak stress varied from 800 to 1450 t/ft^2 . The pressuremeter test result from boring CSM-3 indicates a lower initial tangent modulus

ranging from 1780 to 3000 t/ft² and lower secant modulus of 300 t/ft². It is difficult to assess whether the pressuremeter test in boring CSM-3 was influenced by the proximity of the borehole for boring CSM-1. This factor may have been insignificant because the borehole was filled with the tightly fitting rigid steel pipe. The effect of the adjacent borehole may have been balanced by the proximity of the steel walls of the test vessel which might have stiffened the response of the rockfill to pressuremeter expansion. Of probably more significance was the lower grouting effectiveness in this vessel than the vessel for boring CSM-2. The absence of grout in some portions of the fill may have increased the compressibility of the rockfill.

Shear Strength

The undrained shear strength of the grouted rockfill can be estimated from the pressuremeter limit pressure which is the asymptotic pressure on the curve where unlimited volumetric strain occurs without further increase in pressure. This limit pressure or ultimate stress is related to undrained shear strength τ by a theoretical relationship developed by Gibson and Anderson (1961). The grout reduces the permeability of this material to that of a cohesive material like the original shale rock, and this low permeability results in an undrained response to the rapid loading of the pressuremeter. Although the Gibson and Anderson procedure is not strictly valid for materials with a significant frictional component, an equivalent shear strength τ can be estimated for the level of confining stress which the sample experienced during the test.

The disturbed pressuremeter tests from boring CSM-1 yielded grossly underpredicted shear strengths. The pressuremeter results from boring CSM-2 indicate that the grouted oil shale has a shear strength τ of 12 t/ft² under the confined conditions which existed in the test vessel. The pressuremeter test result from boring CSM-3 yielded a lower shear strength of 6 t/ft². This lower shear strength is probably influenced by the same factors as the modulus values discussed above. A hypothetical Mohr Coulomb failure envelope has been prepared in Fig. 8 by using the measured horizontal stress as the confining stress σ_{3c} and the shear strength τ calculated from the pressuremeter tests. By selecting the angle of internal friction $\phi = 20^\circ$, a failure envelope can be constructed which yields a cohesion intercept $c = 7$ t/ft² for the sample without cement and $c = 3.5$ t/ft² for the sample with cement. A similar approach was used at Locks and Dam No. 26 on chemically grouted alluvial sand with good success. These values should be verified by laboratory triaxial testing, however sampling difficulties could cause disturbance which would result in substantially lower strength results.

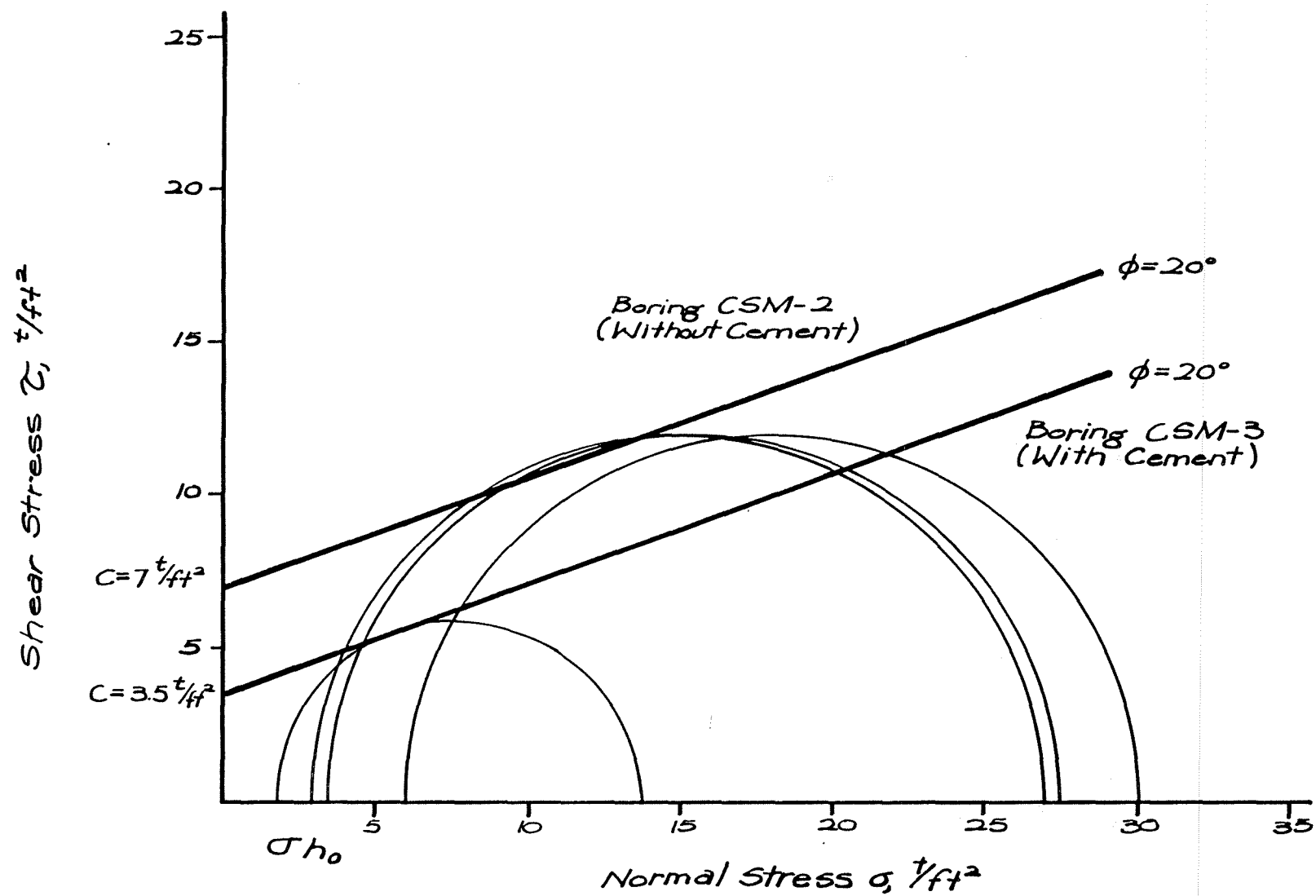


Fig. 8 Hypothetical Mohr Coulomb Failure Envelopes
Derived from Pressuremeter Results

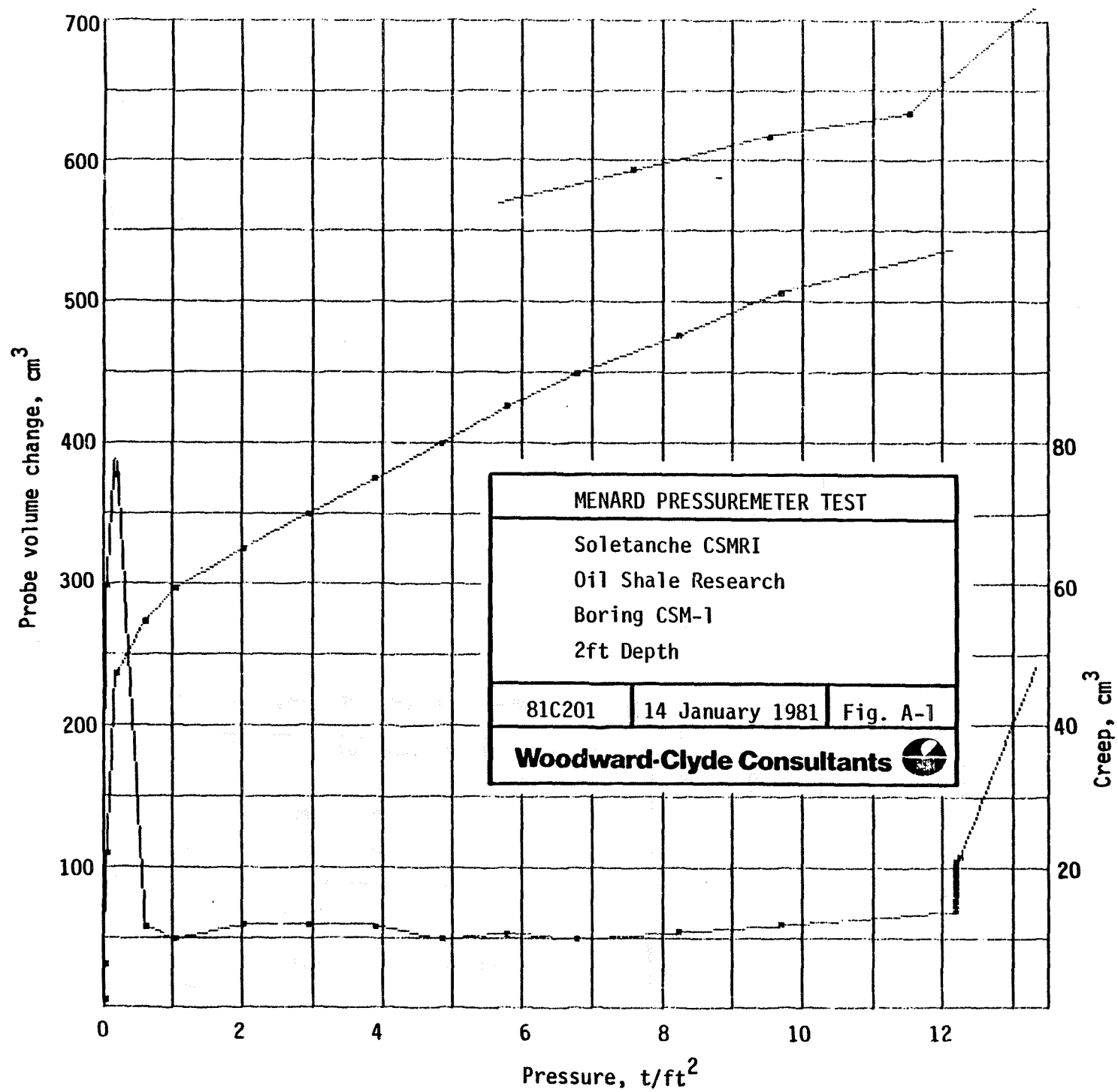
4. CONCLUSIONS

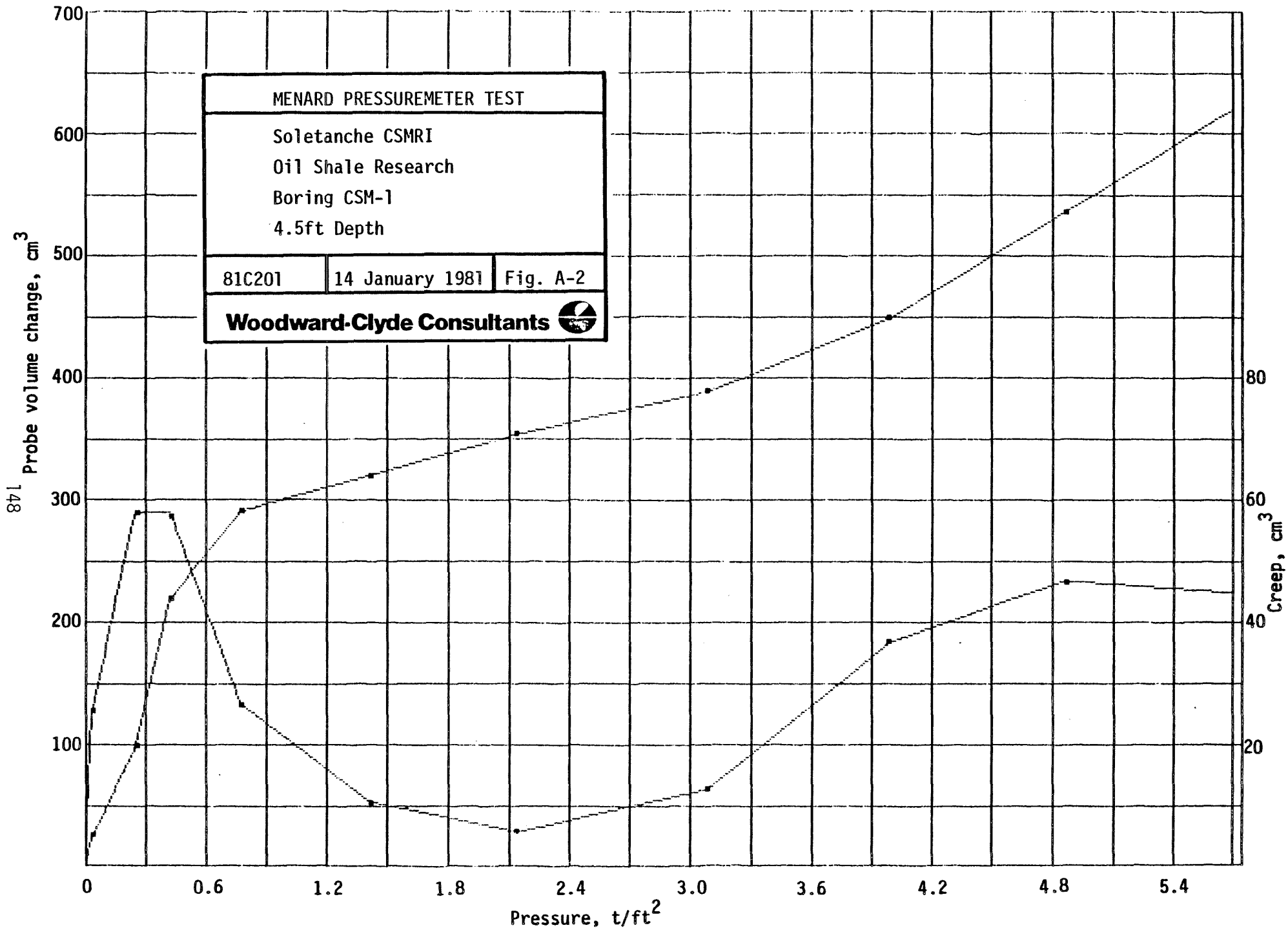
The pressuremeter test results indicate that the grouted spent oil shale is a competent engineering material under confined conditions. This material is capable of sustaining relatively heavy loading without significant deformation or failure. Thoroughly grouted oil shale has an undrained shear strength τ of 12 t/ft^2 ($c = 7 \text{ t/ft}^2$, $\phi = 20^\circ$), initial tangent modulus of 5000 t/ft^2 and secant modulus at 50 percent of the peak stress of 1200 t/ft^2 . Adding a small percentage of cement does not add any appreciable strength to the grouted rockfill, in fact it may have had some influence on the less effective grout penetration in the second vessel. This less complete grouting results in lower strength and compressibility properties.

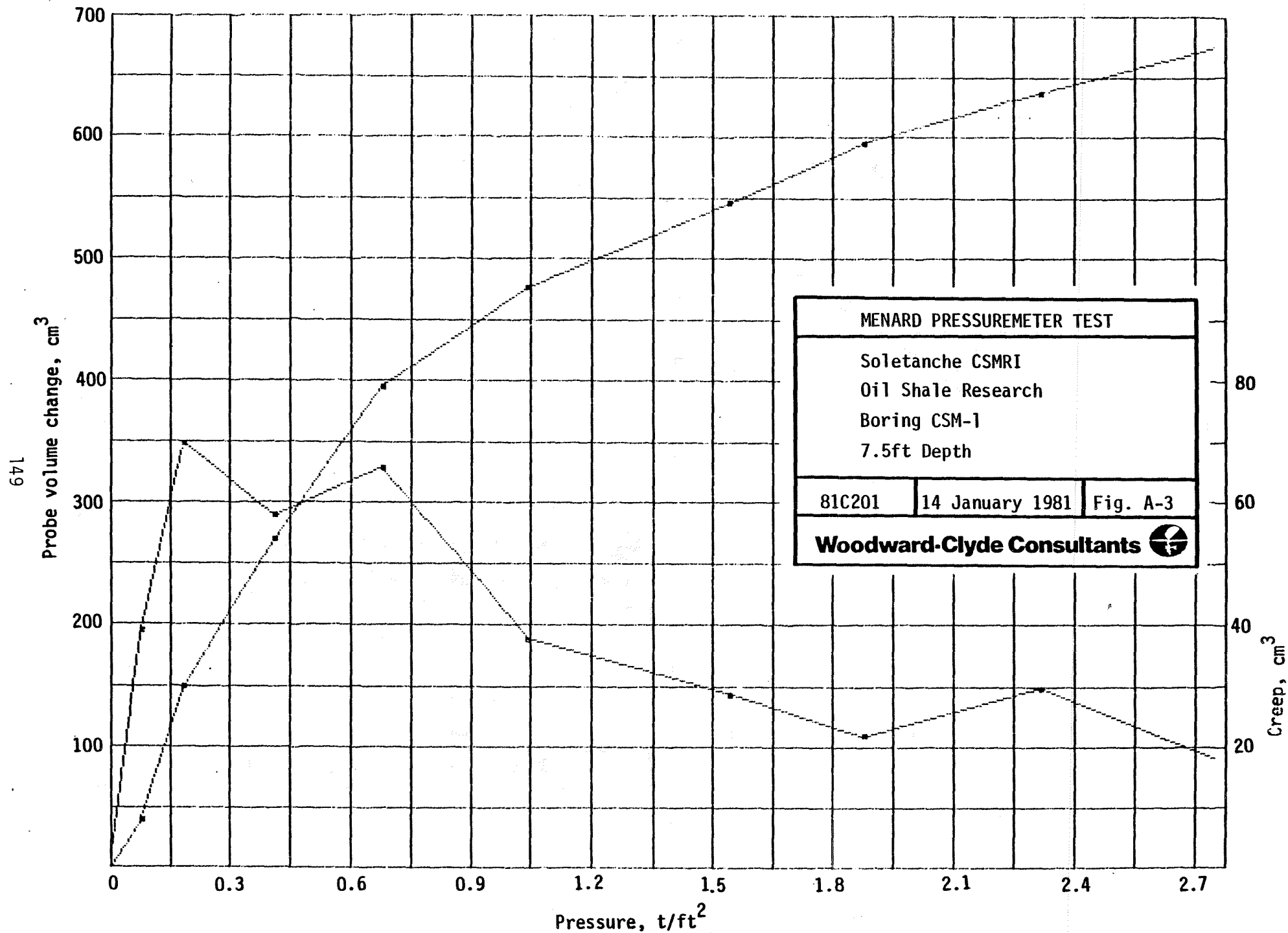
5. REFERENCES

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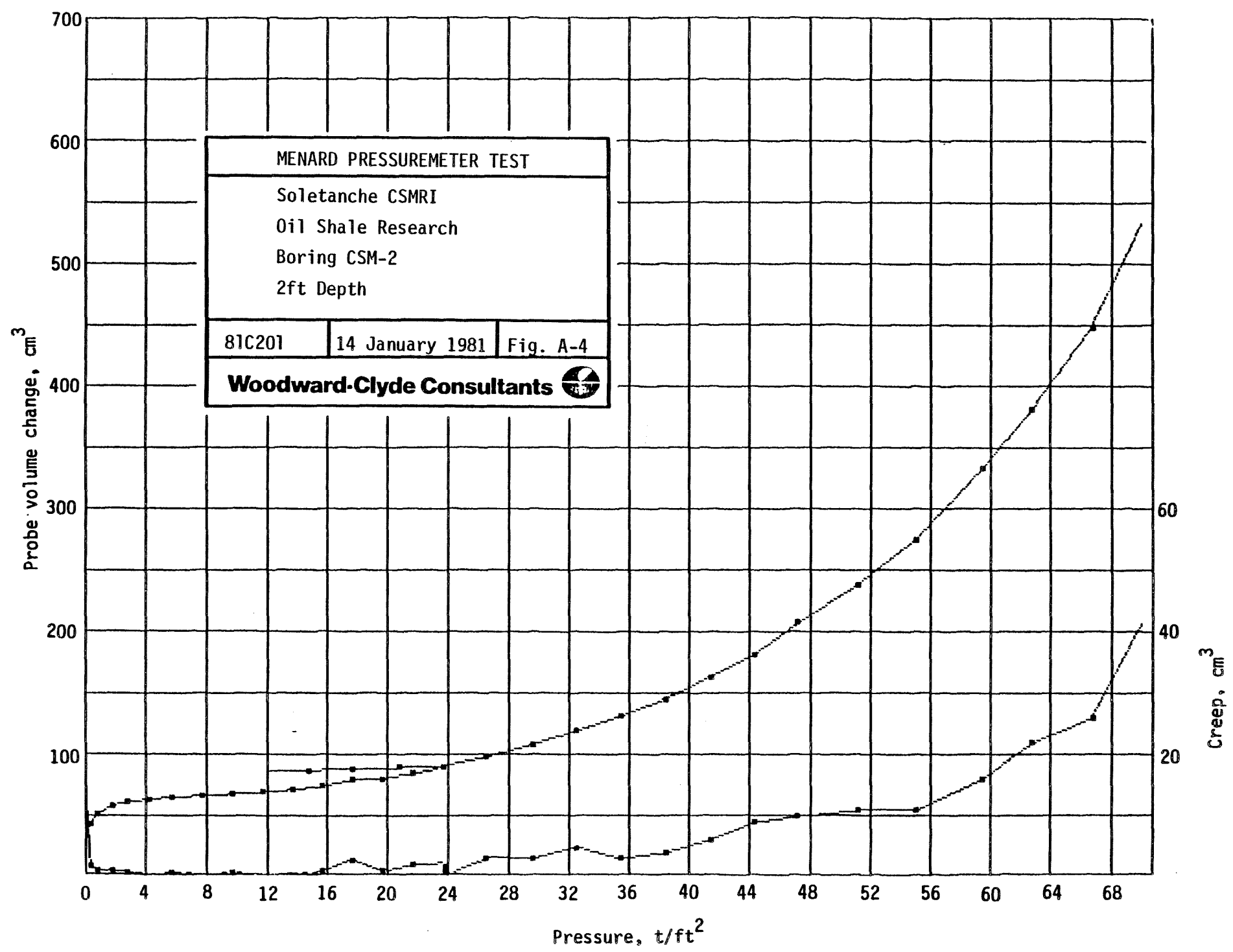
APPENDIX A
PRESSUREMETER TEST CURVES



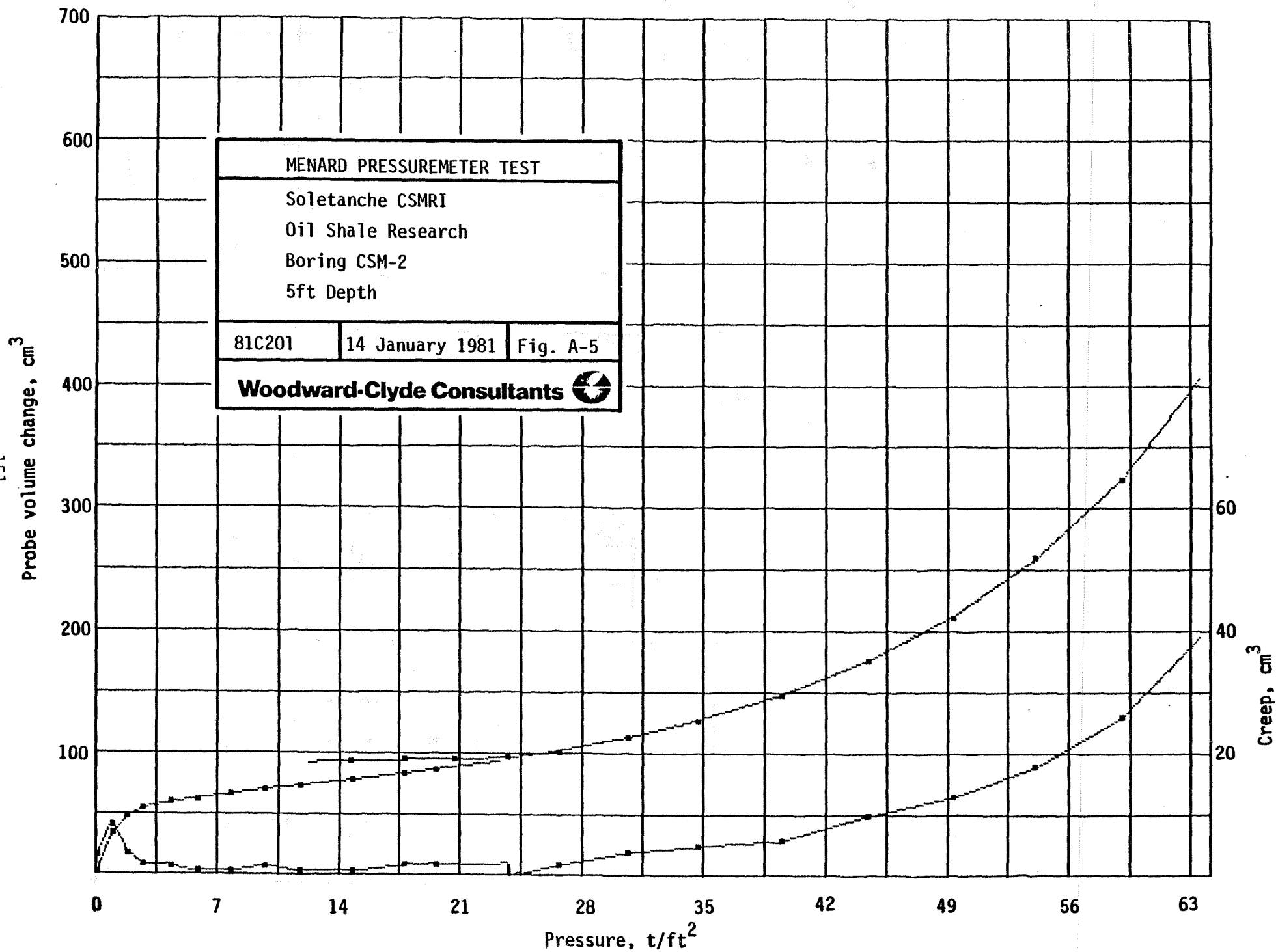


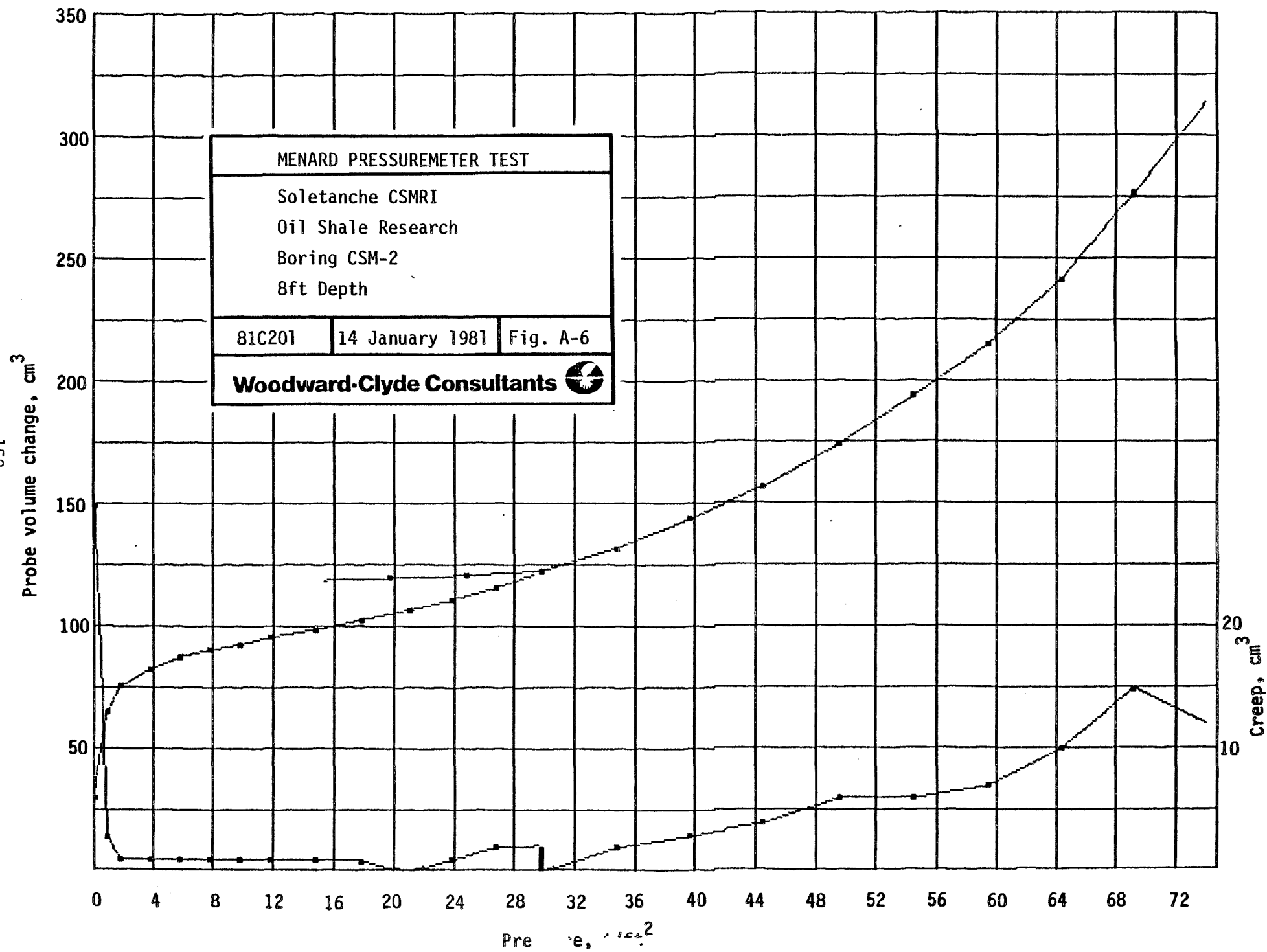


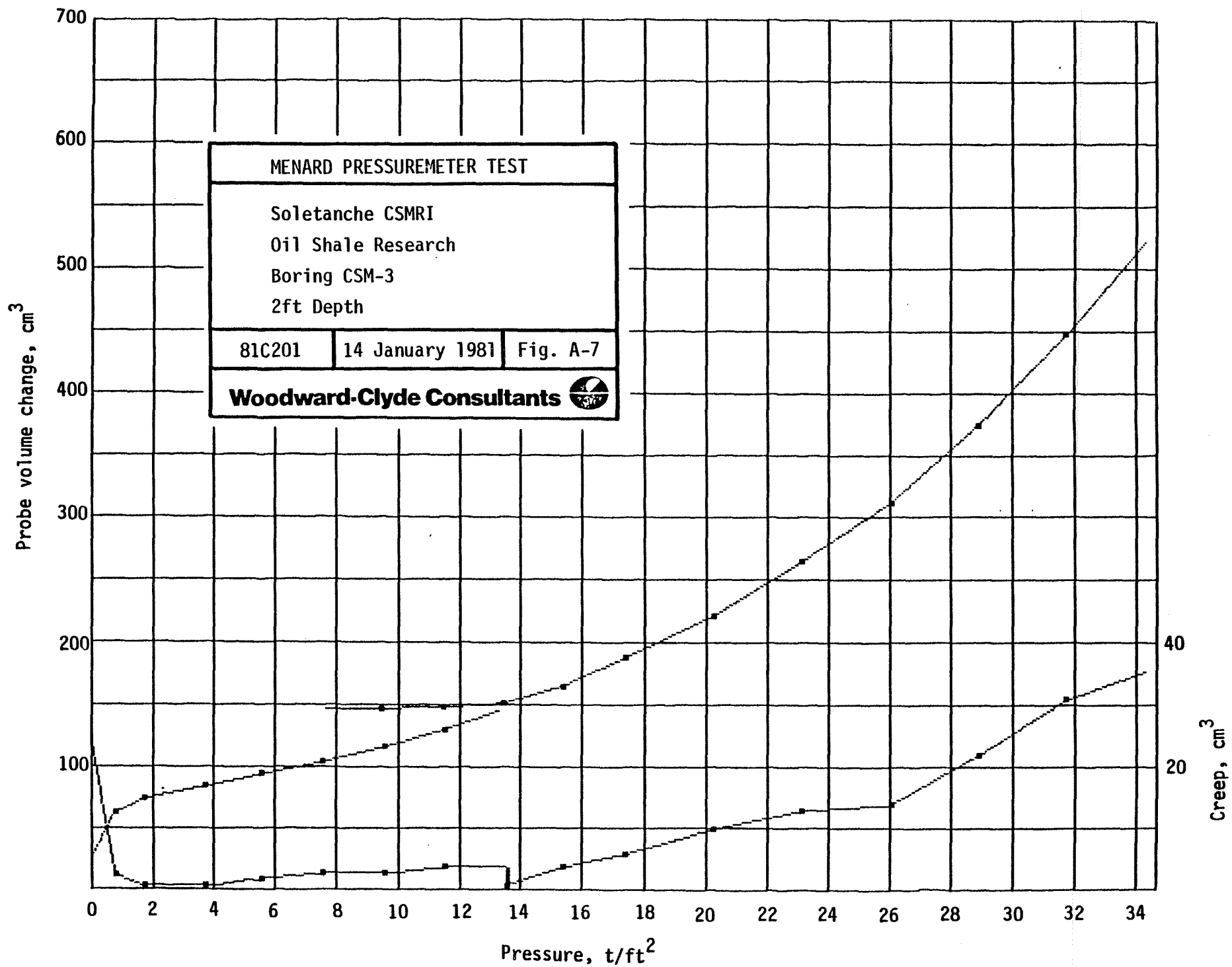
150



151







Appendix D

GROUT FORMULATIONS AND EXAMPLES OF UNCONFINED COMPRESSIVE STRENGTH CURVES

GROUT FORMULATIONS

Grout #	SSA		Additive 1		Additive 2	
	Type	Amount, g/l	Type	Amount, ml/l	Type	Amount, g/l
1	ESP 4	850				
2	ESP 6	850				
3	FGC 6	850				
4	ESP 7	750				
5	FGC 7	800				
6	FGC 8	800				
7	ESP 9	750				
8	FGC 9	800				
9	ESP 10	750				
10	FGC 10	800				
11	Various blend	750				
12	Various blend	800				
13	Blend #1	750				
14	Blend #1	800				
15	Blend #1	850				
16	Blend #1	900				
17	Various blend	750	PDA 25R	10		
18	Various blend	800	Protard	10		
19	Various blend	800	Daratard	10		
20	Various blend	800	Organ GL	10		
21	Various blend	800	Gluconate	1		
22	Various blend	800	Gluconic acid	1		
23	Various blend	800	Silicate M	20		
24	Various blend	800	Silicate JW 25	10		
25	Various blend	800	Silicate N	10		
26	Various blend	800	Silicate 40	10		
27	Various blend	800	Silicate SR 35	10		
28	Various blend	800	Silicate 34	10		
29	Blend #1	850	Silicate M	10		
30	Blend #1	850	Silicate JW 25	10		
31	Blend #1	850	Silicate N	10		
32	Blend #1	850	Silicate 40	10		
33	Blend #1	850	Silicate SR 35	10		
34	Blend #1	850	Silicate 34	10		
35	Blend #1	850	Silicate M	5		
36	Blend #1	850	Silicate JW 25	5		
37	Blend #1	850	Orzan GL	10		
38	Blend #1	850	PDA 25R	10		
39	Blend #1	850	PDA 25R	7		
40	Blend #1	850	Daratard	10		
41	Blend #1	850	Daratard	5		
42	Blend #1	850	Protard	5		
43	Blend #1	850	Protard	2.5		
44	Blend #1	850	Gluconate	1		
45	Blend #1	850	Gluconic acid	1		

GROUT FORMULATIONS (Continued)

Grout #	SSA		Additive 1		Additive 2	
	Type	Amount, g/l	Type	Amount, ml/l	Type	Amount, g/l
46	Blend #1	825			Cement	25
47	Blend #1	800			Cement	50
48	Blend #1	850	Gluconate	1	Cement	10
49	Blend #1	850	Gluconate	1		
50	Blend #1	850	Protard	2		
51	Blend #1	850	Gluconate	1	Cement	10
52	Blend #1	850	Gluconate	1	Silicate M	5
53	Blend #1	850	Protard	2	Silicate M	5
54	Blend #1	850	Gluconate	1	Silicate M	7.5
55	Blend #2	850				
56	Blend #2	850	Gluconate	1		
57	Blend #2	850	Gluconate	1	Cement	10
58	Blend #2	850	Gluconate	1	Cement	20
59	Blend #2	850	Gluconate	1	Cement	15
60	Blend #2	850	Gluconate	1	Lime	20
61	Blend #2	850	Gluconate	1	Lime	40
62	Blend #1	850	Gluconate	1		
63	Blend #1	850	Gluconate	1		
64	Blend #1	850	Gluconate	1		
65	Blend #1	850	Gluconate	1		
66	Blend #2	850	Gluconate	1	Cement	15
67	Blend #2	850	Gluconate	1	Cement	15
68	Blend #2	850	Gluconate	1	Cement	15
69	Blend #2	850	Gluconate	1	Cement	15
70	Trench SSA	1140				
71	Trench SSA	1100	Gluconate	1.3		
72	Trench SSA	1000	Gluconate	1.3		
73	Trench SSA	950	Gluconate	1.3		
74	Trench SSA	950	Gluconate	1.3	Cement	10
75	Trench SSA	1000	Gluconate	2	Cement	10
76	Trench SSA	950	Gluconate	1.5		
77	Trench SSA	950	Gluconate	2		
78	Trench SSA	900	Gluconate	1.5		
79	Trench SSA	900				
80	Trench SSA	950				
81	Trench SSA	950				
82	Trench Blend	900				
83	Trench Blend	900	Gluconate	0.5		
84	Trench Blend	900	Gluconate	1		
85	Trench Blend	900	Gluconate	2		

ESP: ash from electrostatic precipitator, Lurgi test run number.

FGC: ash from flue gas cyclone, Lurgi test run number.

Blend #1: for grouting of vessel 1 at CSMRI.

Blend #2: for grouting of vessel 2 at CSMRI.

Trench SSA: ash available for trench test at Tract C-a.

Trench Blend: for grouting of trench.

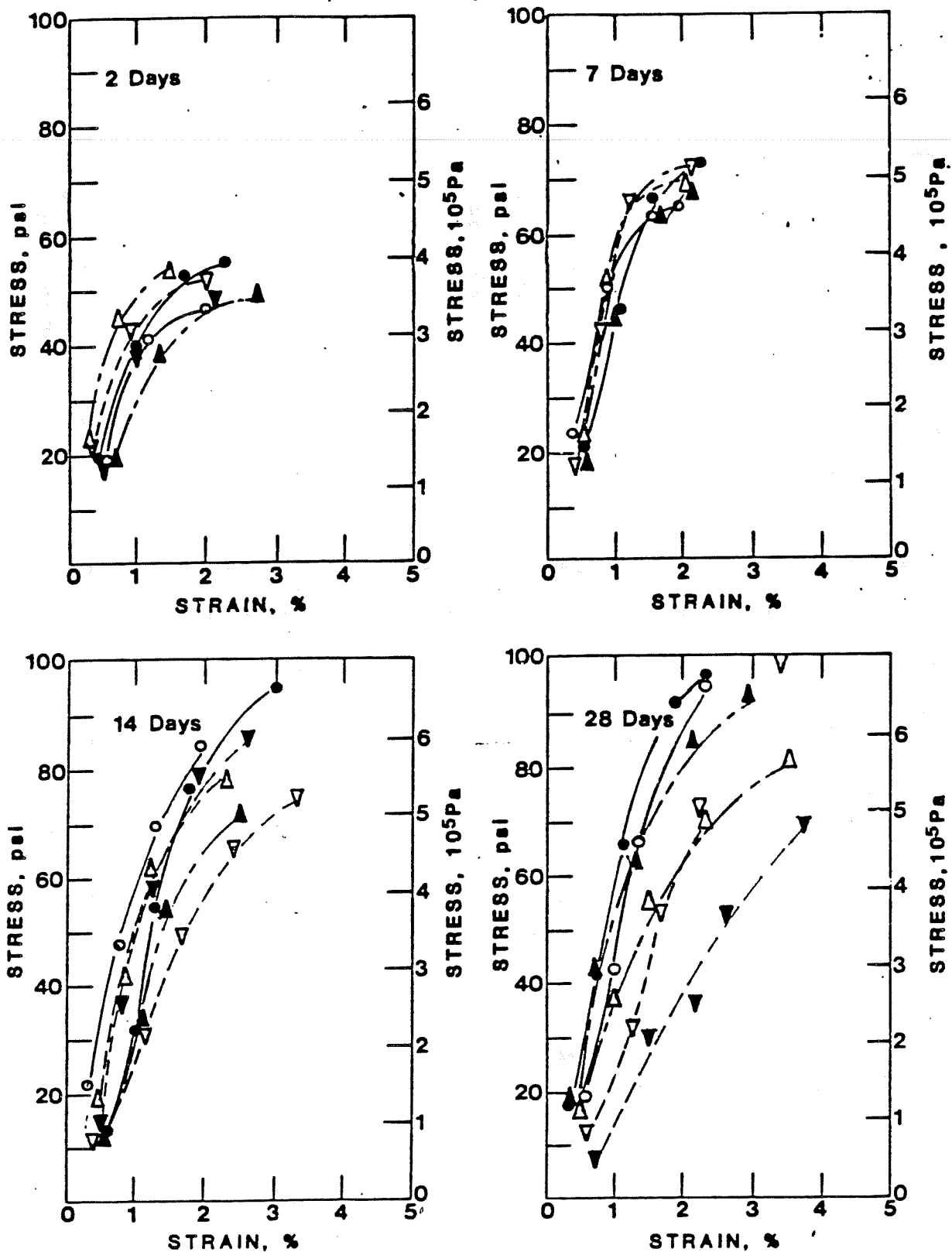


FIGURE D 1 .- Unconfined compressive strength. Grout # 49 (Blend #1) Several series of tests are shown for this grout. Different symbols signify different samples.

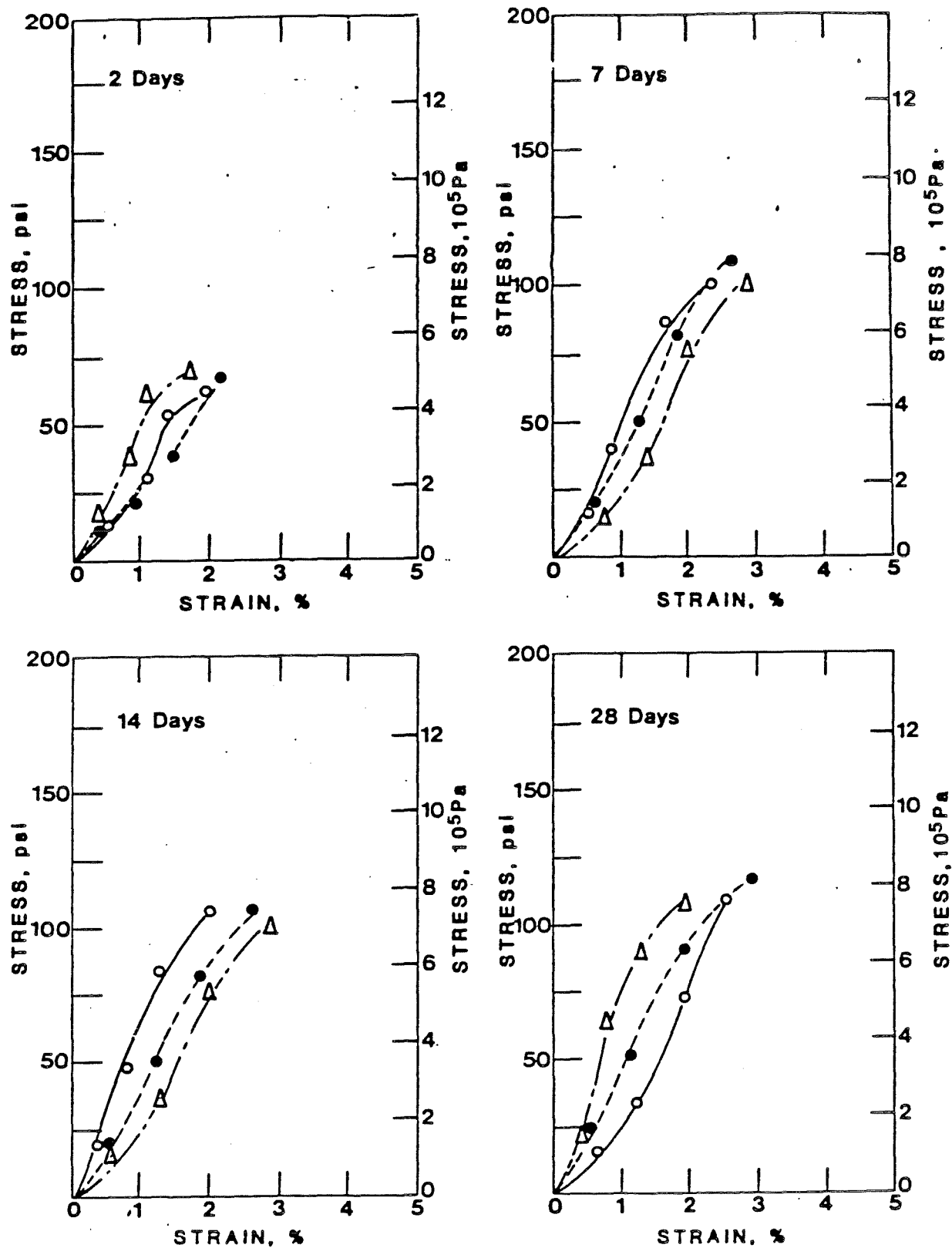


FIGURE D 2 .- Unconfined compressive strength. Grout #59 (Blend #2) Several series of tests are shown for this grout. Different symbols signify different samples.

Appendix E

PHASE III & IV OUTLINE
DESIGN OF THE PILOT GROUTING SYSTEM

E.1 Design of the Pilot Grouting System

The previous work showed that:

- it is possible to develop a suitable grout with ash as a major component to fulfill the objectives of the study.
- laboratory and trench scale models of rubble could be grouted with such a grout.
- the grounded rubble mass had the mechanical properties required to decrease surface subsidence and reduce the risk of contamination of the ground water.

The objectives in this section are to:

- design a pilot scale grouting plant.
- prepare a plan to grout a MIS pilot retort.
- design a monitoring program to evaluate the performance of the grouted pilot retort.

The assumption is made that MIS pilot Retort Zero at Federal Lease Tract C-a will be used for this investigation. The problem of filling such a MIS retort is very different from that encountered in conventional mining operations where large cavities or shafts are hydraulically backfilled with sands or mine tailings to prevent long-term subsidence after extraction of minerals.

The main differences concern:

- The goal is not only to prevent subsidence but also to render the MIS retort virtually impermeable.
- The physical conditions existing in the MIS retort prior to grouting. Except for the top part of the MIS retort where some large cavities may exist, it will mostly consist of a column of various sized rubble. In order to perform a successful grout filling operation, the slurry will have to travel through the voids of the skeleton matrix of the rubblized materials. To do so, the slurry requires some very definite rheological characteristics. This is not the case for conventional hydraulic fill.
- The slurry developed in Phase I has some cementitious characteristics, picks up some mechanical strength through some chemical process, retains most of its water and becomes relatively impervious with time.

Some of the expected technical and experimental difficulties are:

- Determination of the in-situ conditions of Retort Zero prior to grouting, e.g.:
 - temperature
 - rubble size distribution and its likely variation with depth

- hydrogeological conditions inside and along the perimeter of the retort.
- Lowering of the retort temperature prior to grouting if necessary.
- Saturation of spent rubble prior to grouting.
- Installation of a suitable grouting system as well as a satisfactory monitoring system complicated by:
 - (i) the depth of the retort (672 feet of overburden to top Retort Zero)
 - (ii) its small size (30' x 30' x 166' high)
 - (iii) difficulty in obtaining undisturbed samples for laboratory testing due to the non-homogeneity of the grouted rubble mass.

E.1.1 Main Parameters for the Design

E.1.1.1 In-Situ Conditions of MIS Pilot Retort Zero. One of the most important unknowns to date is the exact in-situ conditions of Retort Zero.

The following data were obtained during the rubblization of the retorts:

o Cavity

The cavity at the top of the retort, if any, will be limited to a few feet.

o Size distribution

The maximum size of the rubble is four feet in diameter. The average size of the rubble is 2" to 3" in diameter.

o Temperature

It is assumed the retort will have cooled down to a maximum temperature of 170°F prior to grouting. The cooling will be achieved:

-either naturally, i.e. the lapse of time between retorting and grouting will be sufficient to allow for the cooling;

-or artificially, through circulation of water in the retort prior to grouting.

o The "dryness" of the rubble

The rubble will be dry after retorting. It will be necessary to saturate it prior to grouting in order to limit any changes of rheological characteristics of the grout through the process of absorption of its free water by the rubble.

Table E-1 is a summary of the basic data relevant to Retort Zero.

Table E-1

Retort Zero
Dimensions

Sides length	30 ft x 30 ft
Perimeter	120 ft
Depth of top of retort	672 ft
Depth of bottom of retort	838 ft
Height of retort	166 ft

NOTE: It is assumed that the above data, obtained prior to retorting, will not be significantly altered in the retorting and cooling process. This is one of the reasons why it is suggested to investigate the exact in-situ conditions of Retort Zero prior to grouting. This will allow the following:

- Adjust the grout characteristics to the in-situ conditions.
- Select the appropriate grouting and drilling procedures.
- Mainly evaluate at a later stage the "improvements" resulting from the grouting.

E.1.1.2 Ash Characteristics. The ash used for the phase III and IV study will most likely be produced by a Lurgi pilot plant. It was assumed that their characteristics should not be very different from those found in this study. They will be determined in the initial stage of the phase III study.

E.1.1.3 Grout Characteristics. A small laboratory program will be necessary to determine the suitable grout mix with the new source of ash. It is believed that it will be very similar to the mix developed in this study. Therefore, the same mix parameters were considered for the design of the grouting plant:

Ash content	850 kg/M ³	
Additive #1	1 l/M ³	(Gluconate)
Water	685 l/M ³	

TOTAL	1 cubic meter	
Setting time:	at normal temperatures	5 hours
	at 170°F	100 min

E.1.1.4 Radius of Penetration. The radius of penetration observed in the trench test was about twenty feet. The different parameters in the pilot grouting test which will affect this radius the greatest are the following:

- Grouting Pressure: a much higher pressure can be achieved when grouting Retort Zero than in the trench test.

-Temperature: the temperature will be close to 170°F versus the 60° to 80°F in the trench test.

-The size distribution of the rubble and void ratio may be different from those in the trench test. Furthermore, these two parameters are likely to vary with depth in the retort.

It is assumed in the following that the fact that significantly higher grouting pressure can be obtained will largely offset the "negative" effect of the higher temperature and/or of a smaller void ratio.

A minimum radius of penetration of the grout of 15 ft was therefore considered a safe hypothesis.

E.1.1.5 Scale and Quantities Involved. The volume of future commercial retorts may be approximately one million cy. The scale factor of Retort Zero is close to 1/200.

The maximum void volume of a commercial size retort should be close to 400,000 cy. It is expected that the equivalent of one retort should be grouted every month or a grouting rate close to 500 cy per hour if a continuous grouting operation is assumed. In order to achieve such a rate, a minimum of three retorts will be grouted at the same time and at different locations. This amounts to a grouting rate of 166 cy per hour and per retort. To apply a scale factor to this figure for the pilot plant design would be meaningless. On the other hand, if the temperature is similar in the pilot retort as in a commercial size retort, the flow of grout must be such that it has traveled as far away as possible before it starts to set; i.e., in a 100 min period of time. This is believed to be the main parameter to consider for determining the capacity of the pilot grouting test.

Assuming a grouting radius of 15 ft and that grouting will proceed by successive phases of five feet height maximum, the volume of grout to place in 100 min will be 39 cy/100 min or approximately 23 cy/hour. The pilot plant was therefore designed to mix grout 20 to 25 cy/hour. This should allow the grout to travel as far as possible from the grouting hole before it sets.

E.1.2 Pilot Grouting Plant

E.1.2.1 Concepts and Design. In a commercial operation, mixing will occur at one location, possibly close to the Lurgi plant. From there the grout will be pumped to the retorts being grouted where the grouting module will be installed. Therefore, the pilot grouting plant can be divided into two distinct units; the mixing module and the grouting module.

E.1.2.1.1 The Mixing Module. Two basic mixing concepts exist: the batching system and the continuous mixing system. The continuous mixing concept was selected because it will keep pace with the required production of a commercial operation.

The main components of the mixing module are listed below:

- One (or several) ash bulk storage unit(s).
- One 30 ton feed silo containing the ash.
- One variable speed screw conveyor to meter and transfer the ash into the mixer.
- One centrifugal high pressure pump with a turbine type meter to measure the water flow.
- One mixing hopper.
- One (or two) additive storage units with metering pumps.
- One mixing tube to homogenize the grout.
- One storage tank for the grout with an electrically driven agitator.
- A density meter to control the density of the grout and feed back the information to a control panel for automatic or manual adjustment.

The water flow to the hopper will be regulated by the flow control valve. The ash material will be fed from the silo to the screw conveyor by a rotary valve. The conveyor will measure and transfer the ash into the mixing hopper at a preselected rate. The slurry output will be adjusted by increasing or decreasing the speed of the screw conveyor as well as the flow of water. Ash material and water will be mixed in the hopper. The mixture will flow into the storage tank via the mixing tube. The additives will be fed in the mixing tube.

E.1.2.1.2 Grouting Module. Installed at the grouting location, each module will consist of a storage tank with an agitator and three or four high capacity grouting pumps. Meters will measure the grout flow per pump. The pressure will also be controlled.

E.1.2.2 Cost Evaluation. The cost breakdown of the pilot grouting plant is listed in Table E-2 (1982 dollars). Capital cost including plant components, final design and set up was estimated at approximately \$200,000. Operating cost was estimated at \$18,500 per shift week. A shift week is defined as 40 hours per week. The plant was designed to produce grout at a rate of 20 to 25 cy/hour.

E.1.2.3 Grouting System in Retort. Different techniques are used by specialty contractors for grouting alluvium or rock. The choice depends upon many factors, including:

- o The result(s) to be achieved; for instance, consolidation, impermeability, etc.
- o The type of formations involved and their geotechnical characteristics.
- o The conditions under which the work must be performed such as from a gallery, a shaft or the ground surface.

The grout is injected into the formations to be treated through grout holes. Four basic methods exist. They can be combined if necessary (see Figure E-2).

Table E-2. Capital and Operating Costs of Pilot Grouting Plant

Capital Cost:

Components Item Description	# Item	Unit Price	Extension	Total Cost
<u>Missing Module</u>				
Ash Bulk Storage Unit	2	6,000	12,000	
30 Ton Feed Silo	1	6,000	6,000	
Dust Collector	2	800	1,600	
Rotary Valve to feed Ash	1	1,725	1,720	
Speed Variator (for rotary valve)	1	1,600	1,600	
Screw Conveyor	1	3,700	3,700	
Centrifugal Water Pump	1	3,500	3,500	
Flow Control Valve and Command	1	700	700	
Mixer	1	1,200	1,200	
Metering Pump	1	700	700	
Regulating Valve	1	250	250	
Mixing Tube	1	3,300	3,300	
Densimeter	1	8,000	8,000	
Frame and Skid	1	2,000	2,000	
Miscellaneous (pipe, electrical and control panels . . .)			10,000	
Storage Tank and Agitator	1	3,500	3,500	
Dispatching pump	1	4,800	4,800	
Total Mixing Modulus				<u>\$64,570</u>
<u>Grouting Module</u>				
Storage Tank and Agitator	1	4,800	4,800	
High Capacity Grouting Pump	3	20,000	60,000	
Flow Meter	3	3,500	10,500	
Pressure Recorders	3	500	1,500	
Frame and Skid	1	2,000	2,000	
Miscellaneous (pipe, electrical and control panels . . .)			8,000	
Total Grouting Module				<u>\$86,800</u>
<u>Other Costs</u>				
Final Design		15,000		
Site Preparation (leveling, concrete slabs)		10,000		
Installation of electrical power supply		15,000		
Set up of Mixing and Grouting Module		25,000		
Total Other Costs				<u>\$65,000</u>
<u>Total Capital Cost</u>				<u>\$216,370</u> (1982)
<u>Operating Cost: per shift week</u>				<u>\$18,500</u> (1982)

Method a) Downstage Grouting.

The drilling and grouting are performed concurrently by downstaging. A given length is drilled, then grouted after removal of the drilling rod. Then the cycle is repeated for the next lower section. After each injection, it is necessary to drill through the solidified grout which obstructs the borehole.

NOTE: In some cases the grouting is performed through the drilling rod and tool.

Method b) Upstage Grouting.

The borehole is first drilled to its total depth, then injection proceeds from the bottom to the top.

NOTE: In some cases, grouting is performed through the drilling rods or the casing as they are removed.

Method c) Upstage Grouting; use of a "slit" pipe.

The borehole is first drilled to its total depth. The drilling rods are removed and the hole stabilized with a slurry or cased. A solid grout pipe is installed and sealed in the hole with a low strength grout. As grouting proceeds upward, the solid pipe is slit open.

Method d) Upstage Grouting; use of a sleeve pipe.

With this technique, a special perforated pipe, called a sleeve pipe ("Tube a manchette", originally patented by Soletanche) is sealed in the borehole with a low strength grout. As with the previous technique, this prevents the grout from moving upwards between the grout pipe and the periphery of the borehole. The pipe is perforated every foot to two feet. The perforations are covered by rubber sleeves (the "manchettes") which act as one-way valves during grouting. Grouting is carried out by means of a single or double packer (Figure E-1).

NOTE: To grout by methods c or d it is necessary to hydraulically fracture the grout sealing the pipes.

The most important conditions to consider for the selection of the drilling and grouting methodology are outlined below:

- o The goal. To render the retort impervious can be achieved either by an overall impregnation of the rubblized matrix by the grout, or by the formation of a series of interconnected grouted planes. The end result is the juxtaposition of a series of rubble masses sealed at their boundaries. Both end results are shown schematically on Figure E-3.

It is necessary to impregnate the entire skeleton matrix to increase the in-situ mechanical characteristics of the rubble mass. A spacing of the grout holes of 15 ft should allow achievement of this goal.

- o The "formations" through which drilling will proceed.

-The overburden and shale above the retort are of no concern since the blast and retort ignition holes could be used again if necessary.

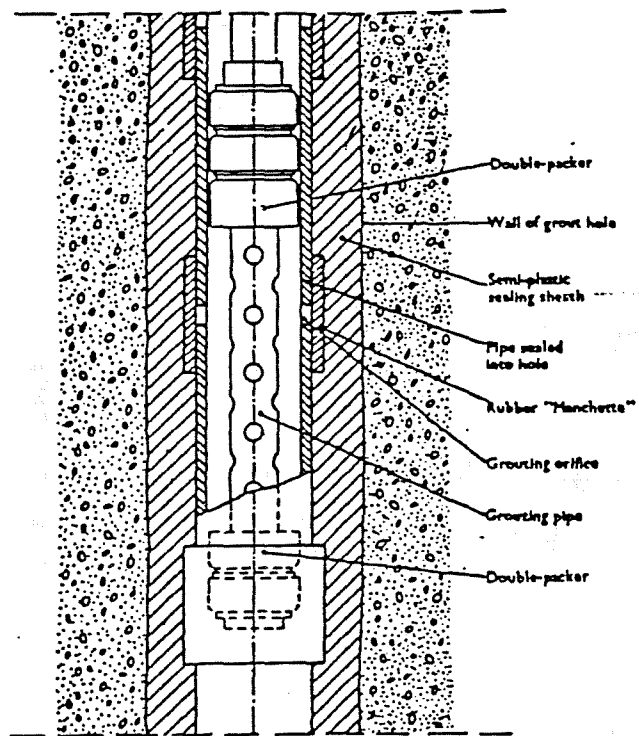
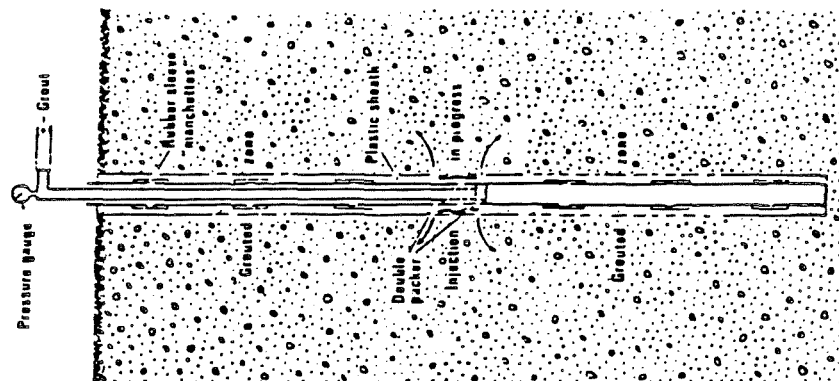
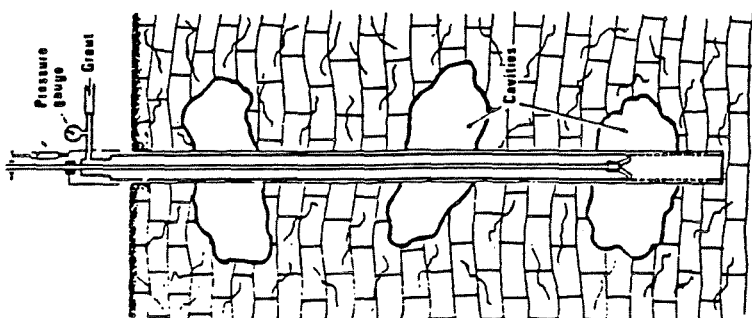


FIGURE E-1 - "Tube-a-Manchette" (sleeve pipe) Vertical Section



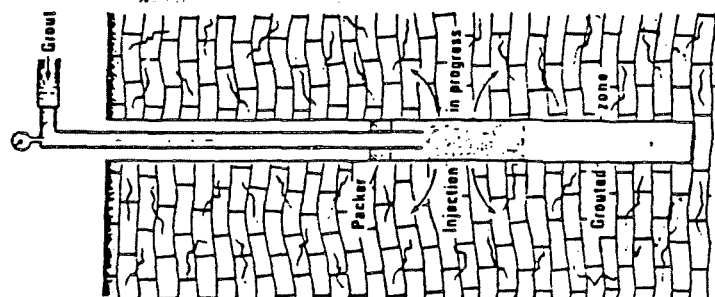
METHOD D

SLEEVE PIPE GROUTING USUALLY USED IN ALLUVIUM FORMATION. IT ALLOWS FOR ADDITIONAL TREATMENT WHERE NECESSARY.



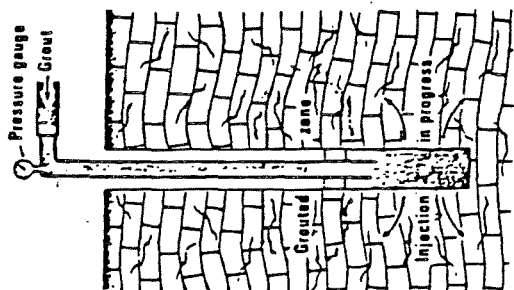
METHOD C

UPSTAGE GROUTING THROUGH A SOLID PIPE WHICH IS SLIT OPEN AS GROUTING PROCEEDS UPWARD.



METHOD B

UPSTAGE GROUTING (USED IN ROCK FORMATIONS)

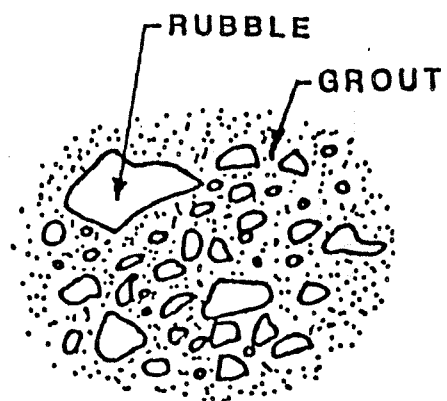


METHOD A

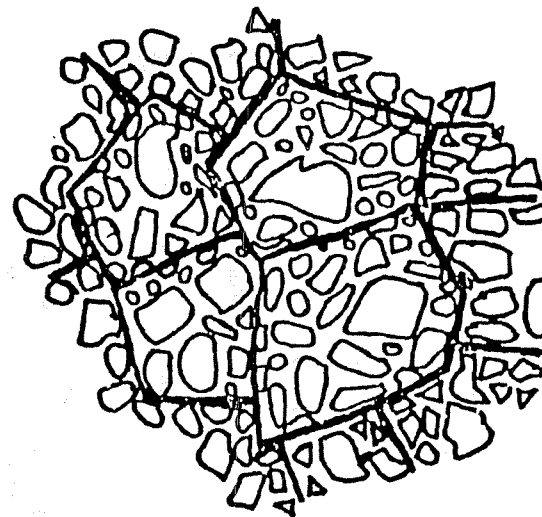
DOWNSTAGE GROUTING (USED IN ROCK FORMATIONS)

BASIC GROUTING METHODS

FIGURE E-2



Overall Impregnation of the
rubble matrix by the grout



Formation of a series of
interconnected grouted planes

FIGURE E-3 - Two Different Epd Results of Grouting

-Most difficulties will be encountered when drilling through the rubblized retort because:

- i) Localized cavities are likely to exist.
- ii) The rubble will probably be unstable.
- iii) The rubble size will vary between several cubic feet to very fine material.
- iv) The material will be heterogeneous as the strength of well retorted rubble will be much lower than that of the partially retorted rubble.
- v) Losses of drilling fluid will occur frequently.
- vi) The rubble temperature may be very high locally.
- vii) Retort Zero is quite deep and the target area is very small.
- o There exists a network of mine shafts and galleries connected to the retort. Some bulkheads have been or will be built to isolate the retort. They are designed to withstand a maximum pressure of 100 psi.
- o The rubble material will be quite dry after being burned and will have a tendency to absorb water from the grout unless it is saturated prior to grouting. It will therefore be necessary to saturate the spent shale.

Two general approaches were considered. The grouting can be done from the ground surface or from the various shafts and access galleries to the retort.

The operation from the ground surface seems preferable, for the following reasons:

- o Vertical drill holes will be easier to realize in the unstable rubble material than inclined holes. This is true whatever drilling method is used.
- o Sleeve pipes and packers will be easier to install in a vertical borehole. (The choice of the sleeve pipe method seems the most appropriate as will be seen in the next paragraph.)
- o It is believed that the existing retort blast and ignition holes can be used if grouting proceeds from the ground surface. They also establish the limits of the retort.
- o The grouting pattern in drilling from access or instrumentation galleries will be complex.
- o The grouting lines will have to be installed in the access shaft and galleries also if work proceeds from the galleries. This might interfere with normal mining activities.

o Grouting from shafts and galleries would be slower since:

- Work conditions are more severe and confined.
- The equipment will be less powerful.
- More hole collapses are likely to occur.

Rotary drilling with mud is believed to be the most appropriate approach in view of the in-situ conditions described above. The downstage grouting method will be used to grout the unstable zones as drilling proceeds. Once completed, the boreholes will be equipped with a special pipe and a thorough pressure grouting of Retort Zero will be performed.

E.1.3 Proposal for Phases III and IV

E.1.3.1 Objectives of Phase III and IV. Phases III and IV were defined as follows:

Phase III will include:

- the construction on Tract C-a of the pilot scale grouting system designed in Phase II.
- the determination of the "conditions" of Retort Zero prior to grouting.
- grouting of Retort Zero (including experimentation to select the most appropriate grouting procedure.

Phase IV will consist of:

- a field investigation program to measure the effectiveness of grouting and assess the mechanical characteristics of the grouted rubble. Permeability tests will also be run.
- the installation of a system to monitor the long-term behavior of the grouted retort.

E.1.3.2 Statement of Solution. The general testing procedure for Phase III will be as follows:

- The in-situ conditions of Retort Zero will be determined by borings and water tests.
- These conditions will be analyzed and the final rheological requirements of the grout will be selected as well as the grouted procedure.
- The ash will be obtained from Rio Blanco.
- A small laboratory program on the ash will be run. Any necessary adjustment to the grout mix will be made.
- The grout mixing plant will be installed.

Drilling and grouting of Retort Zero will be performed in two stages.

Stage I will consist of grouting the cavity which most likely will exist at the top of the retort. This cavity will be filled by gravity through the blast holes already existing in the overburden. After allowing sufficient time for setting, a second phase of grouting on top of the cavity will be performed to ensure proper contact between the grout and the base of the overburden.

Once this filling is achieved, the Stage II drilling of the same holes from the top down to the bottom of the retort will proceed using bentonite fluid and grout alternately. When fluid losses are noticed, the drilling rods will be pulled up and grout will be pumped in until it comes out of the hole annular space at ground elevation.

Once the hole is completed and stabilized, the hole will be flushed with bentonite, the drilling equipment will be removed and a steel sleeve pipe will be installed. The sleeve pipe will then be sealed with the grout.

Then pressure grouting will be carried out through the sleeve pipes, according to the procedure explained in the following paragraph. If the total grout take, cavity filling and pressure grouting corresponds to 20% or more of the volume of the retort, the treatment will be considered satisfactory. If this volume is less than 20%, additional grouting holes will be drilled using the same method on the retort periphery.

The grouting itself will proceed as follows:

An inflatable double packer will be lowered at the deepest sleeve level of the grouting hole. Once the packer is inflated, grout will be sent through the grouting pipe and forced out through its perforations. It will expand in the sleeve zone limited by the inflated packers, flow through the rubber sleeve, make its way through the cemented annular space between sleeve pipe and rubble as pressure will reach a certain level and then flow into the retort material to be grouted. The grouting pressure at the ground elevation will be recorded.

Once a stage at one sleeve is complete, the packer will be moved up to the next sleeve. The grouting will be carried out sleeve by sleeve on a given stretch (100 feet) before being performed in another hole.

After a 100-foot retort zone is completed, a zone overlapping the first is then grouted in the same manner as described above.

The grouting pressure to be expected is difficult to anticipate at this time. The existing height between the ground elevation and the grouted zone is already developing a high pressure which will be only partly balanced by the head losses. The pipe diameter might have to be adjusted to these parameters.

Appendix F

CSMRI REPORT ON VESSEL GROUTING

FACILITIES

Building

To minimize the effects of weather on the equipment and instrumentation, the test program was conducted inside one of the pilot-plant buildings located at CSMRI's facility in Golden, Colorado. The building was selected based on the availability of several features that would be required during the course of the program. These features included a 20-ft (6.1-m) ceiling that would provide ample space to position a coring device atop the test vessels and necessary utilities, such as electricity, water, steam, and compressed air.

Two significant additions to the building were necessary to conduct the program. A platform was erected to provide ready access to the tops of the test vessels. In addition, a boom and electric hoist, shown in Figure 1, were installed that allowed 55-gal (208-l) barrels of spent shale to be lowered and emptied in each vessel with a minimum of shale breakage.

Vessels

Two test vessels were designed and constructed according to the requirements specified in Division 1 of Section VIII of the ASME Boiler and Pressure Vessel Code. Shown in Figure 2, each vessel was 11 ft (3.4 m) in overall height, had a 5-ft (1.5-m) I.D., and weighed approximately 11,000 lb (4,990 kg) when empty. The working height of each vessel was approximately 8½ ft (2.6 m). In this space, the spent shale was loaded and grouted inside each vessel. The working height extended from 23 in. (58.4 cm) above the floor to approximately 6 in. (15.2 cm) below the top of each vessel.

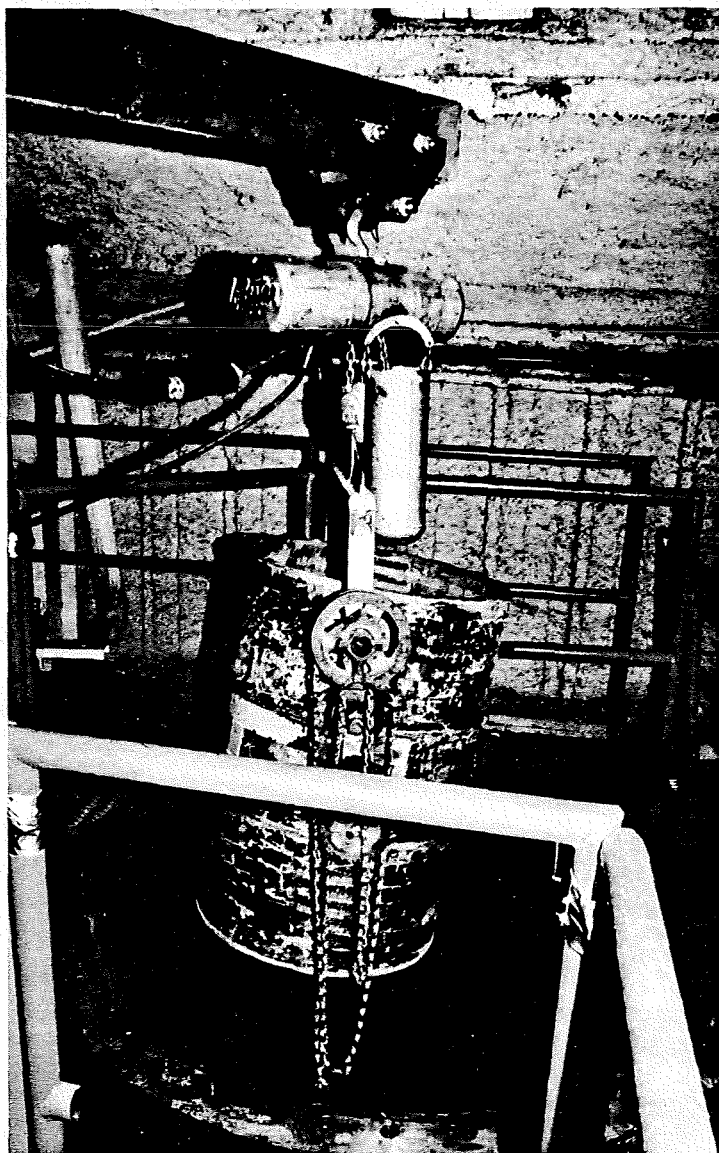


FIGURE 1

Boom and Electric Hoist for Barrel Handling

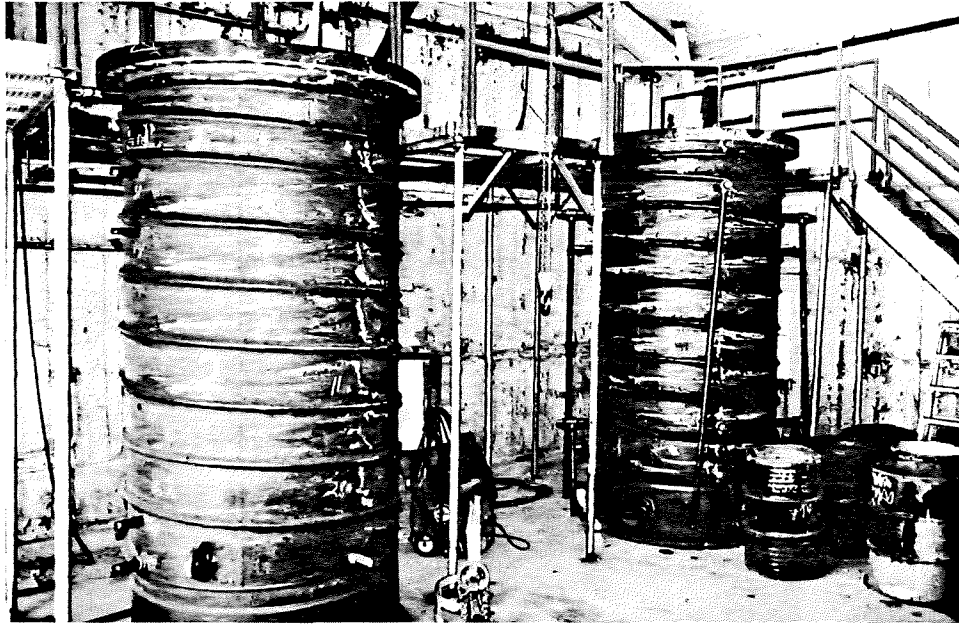


FIGURE 2

Test Vessels and Platform

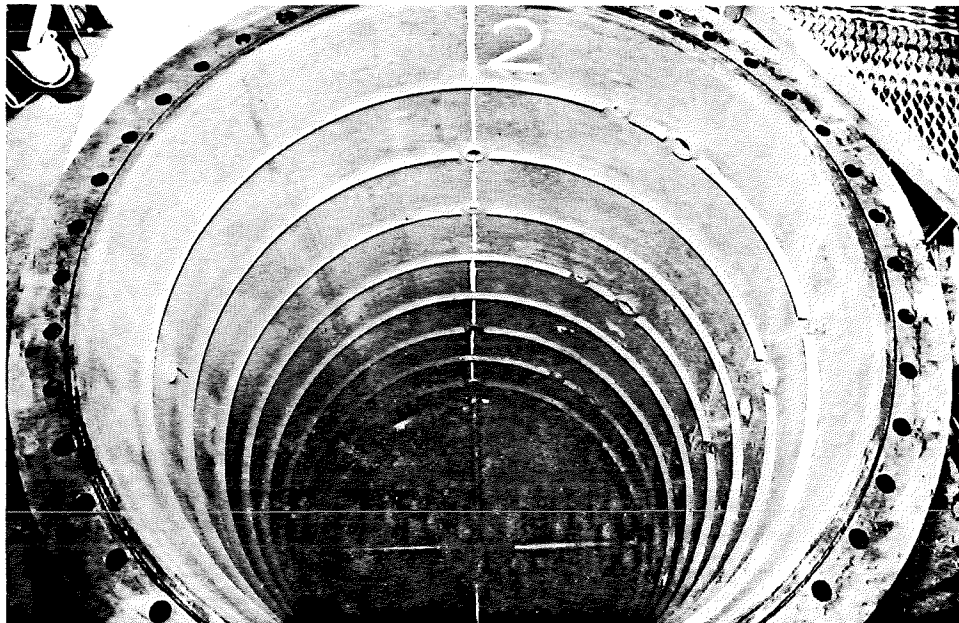


FIGURE 3

Grouting Ramps at Bottom of Test Vessel

Each vessel was equipped with a 3/4-in (1.9-cm) external steam coil that allowed the vessel to be heated. The coil helically wrapped the vessel from the top to the bottom of the working height. The coil wraps were spaced approximately 1 ft (0.3 m) apart.

The inside wall of each vessel was equipped with 1-in (2.5-cm) baffles that were 1/4 in.(0.6 cm) thick, as shown in Figure 3. The baffles were also spaced 1 ft (0.3 m) apart.

Each vessel was equipped with 33 access ports that were used for observation, grout injection, instrumentation, or permeability testing. The most significant amount of grouting was performed using the lowest 4 grouting ports. These 2-in.(5.1-cm) ports were located 3 in.(7.6 cm) from the bottom of the working height and were equally spaced around the perimeter of each vessel. Grouting "ramps," shown in Figure 3, were attached at each of these ports to insure that the center portion of the models were as efficiently grouted as the areas near the walls. Each grouting ramp consisted of a 24-in.(61-cm) section of 1 1/2-in.(3.8-cm) I.D. pipe with four 3/8-in.(1.0-cm) holes equally spaced along one side of the pipe. The ramps were installed horizontally into each vessel and were adjusted so that the holes were pointing downward. The interior end of each ramp was sealed so that all grout was forced down into the model through the 3/8-in.(1.0-cm) holes.

Eight 1-in.(2.5-cm) grouting ports were located 39 and 75 in. (99 and 191 cm) from the bottom of the working height. Four ports were on each level and were equally spaced around the vessel perimeter. Only two of the smaller grouting ports were used at each level. Ports on the same level were on opposite sides of the vessel and the pair

at the 75-in. (191-cm) level were rotated 90° relative to the pair at the 39-in. (99-cm) level. These grouting ports were not equipped with ramps and were used only when there was no more grout uptake at the four large grouting ports.

Nine instrumentation ports were located 24, 60, and 96 in. (61, 152, and 244 cm) from the bottom of the working height. A 2-in. (5.1-cm) cableway port, a 1-in. (2.5-cm) thermocouple port, and a $\frac{1}{4}$ -in. (0.6-cm) pressure transducer port were located at each of these levels.

Eight $1\frac{1}{2}$ -in. (3.8-cm) relief valve ports were located on each vessel as shown in Figure 4. Four ports were equally spaced around the vessel perimeter 12 in. (31 cm) from the bottom of the working height, and four ports were similarly located 84 in. (213 cm) from the bottom of the working height.

Two 1-in. (2.5-cm) ports were located in the dished bottom head, and two 1-in. (2.5-cm) ports were located in the flat lid to facilitate permeability testing of the grouted rubble.

Grouting Plant

The grout mixing and injection plant, shown in Figure 5, was comprised of a grout mixer, an agitated grout storage tank, an injection pump feed tank, and an injection pump.

The grout mixer (not shown) was a special, high turbulence unit developed by Sol-Expert International and consisted of a 45-gal (170-l) mixing bowl with a vertically mounted pump in the bottom of the bowl. When the grout was being prepared, the pump would circulate the mixture

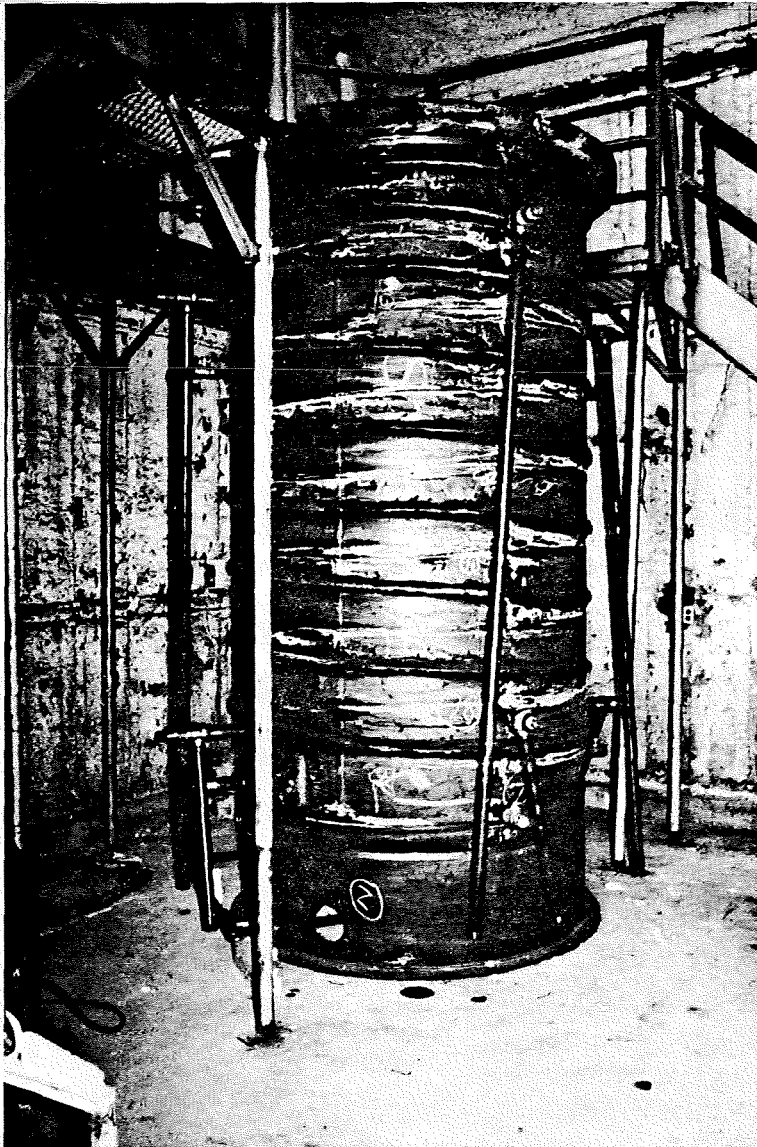


FIGURE 4

Relief Valves on Test Vessel

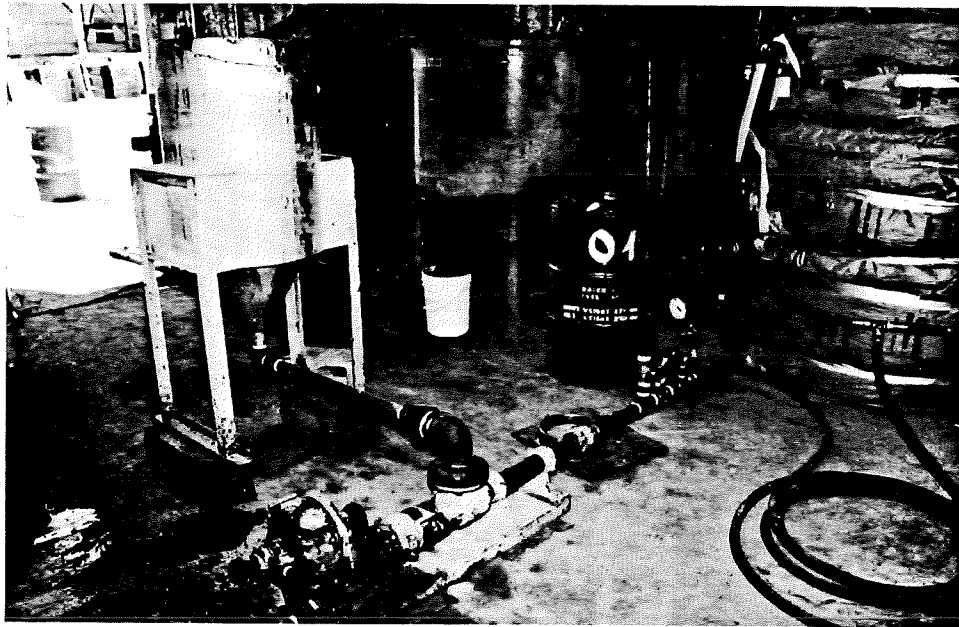


FIGURE 5

Grout Mixing and Injection Plant

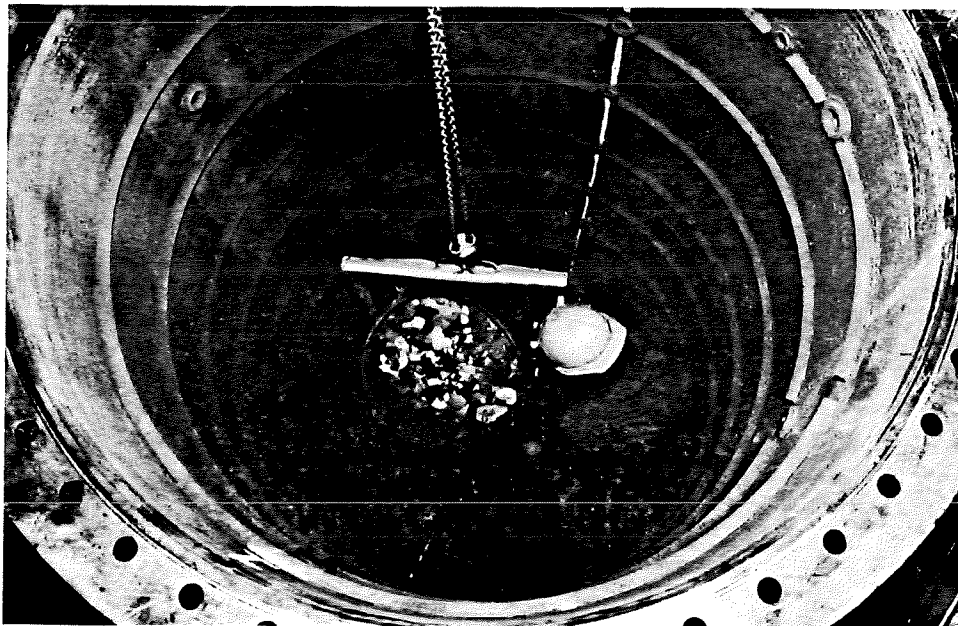


FIGURE 6

Shale Rubble Being Loaded in Test Vessel

through the bowl and thoroughly mix the SSA and water. After the grout was prepared, it was transferred to a 280-gal (1,060-l) storage vessel. This vessel was equipped with a mixer to prevent any settling that might occur.

The injection pump was a pneumatically driven Moyno pump with the high pressure and low flow characteristics that were necessary to properly control the grouting operation. The discharge of the pump was equipped with a recirculating line that allowed grout to be pumped back to the 50-gal (189-l) feed tank during periods of low or zero grout uptake. The pump discharge was also equipped with a pressure gauge so that the grouting pressure would be monitored during the operation.

As mentioned previously, the greatest amount of grout uptake was through the lowest four grouting ports. So that grout uptake would be approximately the same at each of these ports, a grouting "harness" was fabricated that permitted grouting at all four ports simultaneously. Because the "harness" was used, the grouting pressure at each port was the same and, therefore, grout uptake at each of the ports was approximately the same.

The temperature and pressure at six locations within each vessel were continuously monitored and recorded on a Leeds & Northrup Speedomax 250 Series Multipoint Recorder.

PROCEDURES

Rubble Preparation

The spent shale rubble that was used for this program was produced

by retorting raw oil shale from Federal Lease Tract C-a in the 150-ton (136-tonne) N-T-U retort at the Laramie Energy Technology Center (LETC) in Laramie, Wyoming. Approximately 41,880 lb (19,000 kg) of this material was transported from LETC to CSMRI on February 13, 1980. The rubble was covered to minimize the effects of weathering.

Before the rubble could be loaded into the test vessels, its particle size distribution was determined. Because of the large amount of handling that would be required to obtain a representative sample of the rubble and due to the friable nature of the rubble, it was decided to determine the particle size distribution by screening the entire sample at 4 in., 1 in., $\frac{1}{2}$ in., and 6 mesh (101,600, 25,400, 12,700 and 3,350 μm). Any material that was larger than 4 in. (101,600 μm) was made to pass a 4-in. (101,600- μm) screen. The results of the screen size analysis are shown in Table 1.

TABLE 1

Screen Size Analysis of Spent Shale Rubble

Direct		Cumulative	
Screen Product In. (μm)	Weight %	In. (μm)	Weight %
Head (calculated)	100.0		
+4 (101,600)	--	--	--
-4 (101,600) +1 (25,400)	26.5	4 (101,600)	100.0
-1 (25,400) + $\frac{1}{2}$ (12,700)	17.1	1 (25,400)	73.5
- $\frac{1}{2}$ (12,700) +6M (3,350)	25.5	$\frac{1}{2}$ (12,700)	56.4
-6M (3,350)	30.9	6M (3,350)	30.9

Each size fraction was barreled separately and appropriately marked.

Spent Shale Ash Blending

Approximately 10 different samples of spent shale ash (SSA) were received from Standard Oil Company (Indiana). To produce a uniform

test sample large enough to conduct the test program, several of the most promising SSA types were blended. Blending was accomplished by using two dry commodity transport trailers. The SSA was loaded into one of the two trailers, and the bottom discharge lines were connected. Each trailer had three compartments, each with an isolation valve that was used to control the rate of discharge from the bottom of that compartment. The isolation valves for all three compartments were opened on the second trailer. The first trailer was then pressurized, and the SSA was pneumatically transferred to the second trailer. The second trailer was then pressurized, and the SSA was transferred back to the first. The SSA was transferred from one trailer to another four times. After the blending operation was concluded, the SSA was transferred back into barrels.

Approximately 8,000 lb (3,630 kg) of blended SSA was produced during this operation. However, more SSA than expected was used to grout the first test vessel, and a second blending operation had to be conducted. Approximately 5,210 lb (2,360 kg) of SSA was blended in the second operation.

Vessel Loading and Instrumentation

Each test vessel was loaded with spent shale rubble according to the same procedure. Approximately 15 in. (38 cm) of sand was placed in the bottom of each vessel to minimize boundary effects during grouting and permeability testing. Wire screen and fabric filters were used to cover the two access ports in the bottom head of each test vessel to prevent loss of sand through these ports. A fabric filter was also placed between the sand layer and the spent shale rubble to prevent migration of sand into the void spaces between shale particles.

The interface between the bottom sand layer and the spent shale rubble was the bottom boundary of the working height.

The boom and electric hoist were used to lower 55-gal (208-l) barrels of spent shale into the test vessels. As shown in Figure 6, the barrels were emptied as close to the bottom as possible to minimize breakage of the spent shale particles. Each barrel contained a discrete size fraction, and these fractions were manually mixed in the vessels.

Approximately 11,290 lb (5,120 kg) of spent shale rubble with the particle size distribution shown in Table 2 was loaded into Vessel 1. As indicated, no -6 mesh (-3,350 μm) fines were included in Vessel 1. These were excluded to obtain a void ratio similar to that expected in an actual MIS oil shale retort. The void ratio in Vessel 1 based on grout uptake was 0.53.

TABLE 2

Screen Size Analysis of Spent Shale Rubble in Vessel 1

Direct		Cumulative	
Screen Product In. (μm)	Weight %	In. (μm)	Weight %
Head (calculated)	100.0		
+4 (101,600)	--	--	--
-4 (101,600) +1 (25,400)	42.1	4 (101,600)	100.0
-1 (25,400) + $\frac{1}{2}$ (12,700)	23.6	1 (25,400)	57.9
- $\frac{1}{2}$ (12,700) +6M (3,350)	34.3	$\frac{1}{2}$ (12,700)	34.3
-6M (3,350)	--	6M (3,350)	--

Vessel 2 was loaded with approximately 11,672-lb (5,294-kg) of spent shale rubble with the particle size distribution shown in Table 3.

TABLE 3

Screen Size Analysis of Spent Shale Rubble in Vessel 2

Direct		Cumulative	
Screen Product In. (μ m)	Weight %	In. (μ m)	Weight %
Head (calculated)	100.0		
+4 (101,600)	--	--	--
-4 (101,600) +1 (25,400)	31.1	4 (101,600)	100.0
-1 (25,400) + $\frac{1}{2}$ (12,700)	24.8	1 (25,400)	68.9
- $\frac{1}{2}$ (12,700) +6M (3,350)	30.1	$\frac{1}{2}$ (12,700)	44.1
-6M (3,350)	14.0	6M (3,350)	14.0

Because the exclusion of the -6 mesh (-3,350 μ m) fraction from Vessel 1 resulted in a void ratio that was greater than desired, approximately 1,630 lb (739 kg) of this material was included in Vessel 2. The resulting void ratio in Vessel 2 was 0.44 based on grout uptake.

Interior instrumentation was installed in each vessel concurrently with the loading of the spent shale rubble. Instrumentation cableways were installed 24 , 60 , and 96 in. (61, 152 , and 244 cm) from the bottom of the working height. Each cableway was 20 in. (51 cm) long and allowed a pressure transducer and a thermocouple to be installed inside each vessel at the desired height. A pressure transducer and thermocouple were also installed at the vessel wall at the same heights as the interior instrumentation cableways. A more detailed description of the pressure transducers is provided in the Appendix, Exhibit 1.

After the spent shale had been loaded to within approximately 6 in. (15 cm) of the vessel lip, a second fabric filter and sand layer were installed. This top sand layer filled the vessel to the lip and served the same purpose as the bottom sand layer. The flat lid was set in place and secured with 44 1 $\frac{1}{4}$ -in. (3.2-cm) bolts.

Void Ratio Determination

After the vessel was loaded with rubble, it was filled with a known quantity of water. Because the water occupied the same space that would eventually be filled with grout, the volume of water that was necessary to fill the vessel provided an estimate of the volume of grout that would be required during the grouting operation. As a check, the spent shale rubble was saturated with water, refilled to the same level that was present before saturation, and then allowed to drain. The amount of water that drained from the vessel was measured and compared to that necessary to fill the vessel. An estimated void ratio was determined in each case.

After the void ratio had been estimated, the permeability of the ungrouted mass was determined by measuring the flow of water at a constant pressure through the bed of spent shale.

The results of all tests that were performed prior to grouting are discussed elsewhere in this document. Following these tests, the vessels were pressure tested for leaks, insulated, and heated to approximately 185°F.

Grouting and Curing

The grout for each vessel was mixed according to specifications that were determined in the grout development phase of the program. Grout was mixed in 26.4-gal (100-l) batches and was stored in the agitated storage tank. Approximately 79 to 132 gal (300 to 500 l) of grout were kept on hand in the storage tank to provide an adequate supply to the grouting pump feed tank. The grout was transferred from the storage tank to the pump feed tank in 4-gal (15-l) lots. In this

way, the volume of grout that was injected into the vessel could be monitored. The specific gravity and viscosity of the grout in the agitated storage tank were checked periodically to maintain proper quality control.

Three stages of grouting were conducted for each vessel over a period of three days. The first stage of grouting was characterized by a maximum grouting pressure of 100 psig (790,000 Pa), the second stage was characterized by a maximum pressure of 150 psig (1,135,550 Pa), and the third stage was characterized by a maximum pressure of 200 psig (1,480,290 Pa). In addition, the grout for each stage was of a different color than other stages so that when the cured, grouted mass was removed from the test vessels, the progress of the grout during each stage would be noted. Each stage of grouting was initiated at the lowest four grouting ports. Grouting was continued at these ports until there was no more grout uptake. The grouting line was then connected to a mid-level grouting port on one side of the vessel followed by the other port on the same level. The grouting line was then moved to the upper grouting ports, and grouting was conducted in the same manner. At each port, grouting continued until grout uptake ceased. The two ports in the vessel lid remained open during the entire grouting operation to provide an outlet for the water that was in the vessel at the beginning of the operation.

Following completion of all stages of grouting, the mass was cured for 28 days at 175° to 185°F (79° to 85°C). At the end of the curing period, permeability tests and strength tests were performed on the grouted mass to determine the effectiveness of the operation.

After all testing was completed, the grouted mass was removed from the vessel. Samples were obtained from the top, middle, and bottom of each vessel for visual inspection. The remainder of the mass was disposed of.