WSRC-TR-99-00239, Rev. 1

Permeability of Consolidated Incinerator Facility Wastes Stabilized with Portland Cement

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WSRC-TR-99-00239-TL

September 21, 1999

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FROM: B. T. Butcher, 773-43A (x5-5810)

TRANSMITTAL OF REVISED CIF STABILIZED WASTE PERMEABILITY TESTING REPORT

Attached is the report entitled, "Permeability of Consolidated Incinerator Facility Wastes Stabilized with Portland Cement", WSRC-TR-99-00239, Revision 1, prepared by Bill Walker of SRTC. This report has been revised to incorporate reference to a procedure for conducting a percent saturation measurement that is important to interpreting results. In addition, this revision makes some minor changes to the tables and text. However, the conclusions of the report have not changed, namely, that CIF ashcrete was determined to meet the permeability and compressive strength requirements for disposal in E-Area.

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Revision 1
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KEY WORDS: Permeability
Waste Treatment
Portland Cement

RETENTION: Permanent

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PERMEABILITY OF CONSOLIDATED INCINERATOR FACILITY WASTES STABILIZED WITH PORTLAND CEMENT

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SUMMARY

The Consolidated Incinerator Facility (CIF) at the Savannah River Site (SRS) burns low-level radioactive wastes and mixed wastes as a method of treatment and volume reduction. The CIF generates secondary waste, which consists of ash and off-gas scrubber solution. Currently the ash is stabilized/solidified in the Ashcrete process. The scrubber solution (blowdown) is sent to the SRS Effluent Treatment Facility (ETF) for treatment as waste water. In the past, the scrubber solution was also stabilized/solidified in the Ashcrete process as blowcrete and will continue to be treated this way for listed waste burns and scrubber solutions that do not meet the Effluent Treatment Facility (ETF) Waste Acceptance Criteria (WAC).

The disposal plan for Ashcrete and special case blowcrete is to bury these containerized waste forms in shallow unlined trenches in E-Area. The WAC for intimately mixed cement-based waste forms intended for direct disposal specifies: 1) a minimum compressive strength of 500 psi, and 2) a saturated hydraulic conductivity, or permeability, of less than $1x10^{-8}$ cm/sec.

Simulated waste and actual CIF ash and scrubber solution were mixed in the laboratory and cast into forms for testing. The compressive strengths of the samples were approximated by using a concrete penetration instrument. The permeability was measured by two different methods, a falling head method conducted at the Savannah River Technical Center (SRTC) and a whole body centrifuge method conducted by UFA Ventures.

Results from this study indicate that Purex Ashcrete samples meet the WAC and are acceptable for direct disposal. Blowcrete samples met the compressive strength requirement of the E-Area WAC. Blowcrete samples made with Purex scrubber solution satisfy the permeability requirement for direct disposal when analyzed using the falling head method but are predicted to fail when tested by the centrifuge method.

The UFA centrifuge test method results in higher permeability values for the falling head test method based on the limited testing of this study. This discrepancy may be due to one or more of the following: 1) a greater degree of saturation may have been achieved by the UFA vendor, 2) pores may have been opened by the higher pressure used in the UFA method, 3) the epoxy used in the sample preparation (gluing desk in sample holder) for the ELE falling head method may have clogged surface pores and thereby decreased effective porosity. Either method of measuring permeability is acceptable for qualifying the CIF ashcrete and Blowcrete for E-Area disposal. Currently there is no vendor available to handle solid radioactive samples. If the centrifuge method is not available for testing then the correlation relating centrifuge permeabilities to falling head permeabilities could be used to guarantee permeability results are acceptable.

INTRODUCTION

Disposal plans for the CIF Ashcrete and Blowcrete depend on whether the waste burned in the incinerator is listed hazardous/mixed, characteristically hazardous/mixed, or radioactive. At the present time, SRS does not have an on-site disposal facility for listed hazardous/mixed waste even if the waste is treated and the resulting waste form passes the TCLP leaching requirements. An example of this type of waste/waste form is the Ashcrete and Blowcrete resulting from burning the M-Area Filter Paper Take-up Rolls, which carried the F006 listed waste code. Currently the drums of waste form generated in this campaign are stored in M-Area. Off-site disposal at Envirocare is a disposal option.

The disposal plan for Ashcrete and Blowcrete generated from burning characteristically hazardous/mixed waste or low-level radioactive waste is to place the containerized waste forms in an unlined earthen trench in E-Area. The direct trench disposal is an SRS option for low-level, non hazardous (acceptable leachability for RCRA toxic metals) intimately mixed cement based waste forms. The WAC for direct trench disposal of cement-based waste forms includes an unconfined compressive strength requirement of greater than 500 psi and a permeability of less than $1\text{x}10^{-8}$ cm/sec.[1] The E-Area WAC is based on the contaminant transport modeling in the E-Area Disposal Facility Performance Assessment.[2]

Determination of permeability is difficult for samples which have low values. This is due, in part, to difficulty in:
1. measuring very small volumes of water flowing through an impermeable material
2. reducing the sample thickness to achieve flow in a shorter time. (Thin disks are fragile and in many case contain pores, aggregate, salt crystals, ash particles.)
3. achieving complete saturation.

In addition, there are no standard low permeable materials available from the National Institute of Standards and Testing (NIST) or from equipment vendors, which can be used to calibrate or verify measurements.

Consequently, two different permeability methods were applied to the CIF waste forms. Given the limited testing, results obtained by both instruments are considered valid since the techniques require different conditions for measuring permeability. For example, a force pressure of 10 psi was used in the falling head method whereas a gravitational force of up to 20,000 g was used in the centrifuge method. Both the hydraulic conductivity values and the samples themselves are affected by the magnitude of the forces applied during the measurement.

Waste Processing Technology (WPT) personnel at SRTC were requested to use an ELE Permeameter, which was available in the CIF Laboratory to perform the permeability measurements. In order to use this instrument, parts had to be ordered, the instrument set up and tested, and a procedure written. The procedure is included in Appendix 3. This instrument is similar to the one described for ASTM D2434-68. In ASTM D2434-68 a constant low pressure head (constant head test) is used to determine permeability of high permeability materials such as sand or gravel. The ELE instrument is a modified version of this method called a falling head test.

Duplicate samples made with non radioactive simulants were sent to UFA Ventures, Richland, WA for testing using a centrifuge method. An ASTM test method is being developed for the centrifuge method of measuring unsaturated and saturated permeability.

Compressive strength is usually performed by using a mechanical press to crush samples. The amount of force needed to cause the sample to fail is recorded and related to the area. In order to minimize the spread of contamination a method was developed to estimate compressive strength based on resistance to penetration with a penetrometer from Gilson Inc.

Characterization data of the Purex ash and blowdown used to make the samples are presented in Appendix 1.

EXPERIMENTAL PROCEDURE

Sample Preparation and Curing

Two non radioactive mixtures were prepared with Portland cement and a 10 and 30 wt % NaCl solution for the purpose of learning to use the ELE instrument and to obtain samples to send to UFA Ventures for permeability testing using the centrifuge method. These formulations were cast into several sample containers which were sealed for curing.

Three waste forms were also prepared in a radioactive hood using actual CIF blowdown (dilute and concentrated) and ash. Samples were cast in plastic containers and sealed for curing at ambient room temperature for a minimum of 28 days. No radioactive samples were sent for off-site testing.

All samples were mixed by hand stirring with a spatula in a plastic beaker for 10 minutes. Ingredients in the five formulations prepared for this study are listed in Table I. A sample of Purex ashcrete prepared in the Ashcrete Facility 6/9/99 was also tested. This sample was cured for 39 days in a sealed container prior to testing. The weight per cents of different components are shown in Table II.

The samples were cast as cylinders with a diameter of approximately 3 cm and heights varying from about 0.75 cm to 5 cm. The objective was to cast the samples to fit in the ELE sample holder. The thickness required for the sample depends to some extent on the permeability and inhomogeneity of the material. After demolding, the cylinders were cut into thinner disks if necessary. A hack saw blade was used for this manual operation. The flat sides of the sample disks were finished with a 60 and 240 grit abrasive paper.

The samples tested in the ELE permeameter were about 0.75 cm in thickness. The cylindrical samples tested by UFA Ventures, Inc. were 3-4 cm in height. Samples used to determine the resistance to penetration were cylinders about 5 cm in height. The penetration tests were performed on the flat top surface of these samples. A technique was developed to glue the disk-shaped samples into the ELE sample holder. An epoxy resin was used to obtain a secure leak-tight seal between the sample holder and the waste form. After the glued sample sets over night the samples are saturated by immersing in water and placing under house vacuum for 48 hours. The degree of saturation was not determined during these tests but is recommended for future determinations. A procedure IWT-OP-131 has been developed for determining the amount of saturation and will be used in future permeability determinations.[3]

Table I. Waste forms prepared for Permeability Testing.

Sample No.	Sample Descrip.	PC Wt (g)	Add. H_2O Wt (g)	Sim. 10 wt % NaCl (g)	Sim. 30 wt % NaCl (g)	Scrub. Soln 1 wt % (g)	Scrub. Soln. 40 wt % (g)	Dry Purex Ash (g)
1	Simulant With 10 Wt % NaCl	100	-	46	-	-	-	-
2	Simulant With 30 Wt % NaCl	100	-	-	59	-	-	-
3	Purex blowcrete With 1 wt % Solids	87	-	-	-	63	-	-
4	Purex blowcrete With 40 wt % Solids	97	-	-	-	-	60	-
5	Purex ashcrete	110	111	-	-	-	-	42
6	Plant Purex Ashcrete	225	146	-	-	-	-	66

Table I notes: 1) 1 wt % and 40 wt % scrubber solution refer to the concentration of total solids in the solution. 2) Initially the ash contained close to 25 weight % water. The ash amount is reported in this table on a dry basis and the ash water included in the additional water column. 3) Plant Purex Ashcrete Sample # 6 was plant sample # 99-CIF-0451 taken 6/9/99. Component amounts in the table are based upon the standard formulation used by the plant.

Table II. Waste Form Component Proportions.

Sample No.	Sample Descrip.	Portland Cement (wt %)	Water (wt %)	NaCl (wt %)	Purex Scrubber Solids (wt %)	Dry Purex Ash (wt %)	W/C
1	Simulant With 10 Wt % NaCl	68.4	28.4	3.2	-	-	0.415
2	Simulant With 30 Wt % NaCl	62.9	26.0	11.1	-	-	0.413
3	Purex blowcrete With 1 wt % Solids	58.0	41.6	-	0.4	-	0.717
4	Purex blowcrete With 40 wt % Solids	61.8	22.9	-	15.3	-	0.371
5	Purex ashcrete	41.8	42.2	-	-	16.0	1.009
6	Plant ashcrete	51.5	33.4	-	-	15.1	0.649

Table II notes: 1) Scrubber solids refer to total solids in the solution. 2) Initially the ash contained close to 25 weight % water. The ash amount is reported in this table on a dry basis and the ash water included in the water column. 3) W/C is water to cement ratio

ELE Falling Head Method

The falling head method of determining permeability is based on Darcy's Law. An ELE permeameter, Model K-670A, was used in these experiments. A picture of the permeameter apparatus is given in Figure 1.

The apparatus consists of a tank, tank manometer, inlet pressure gauge, outlet pressure gauge, pressure regulator, isolation valves, sample holder, two sample holder heads, connection tubing, and a sample head manometer. Appendix 3 contains the SRTC procedure covering details of operation of the apparatus.[4] A schematic of the experimental apparatus is given in Figure 2 and a summary of the procedure is given below.

A disk of a cured waste form is cut and the diameter and thickness of the sample measured. The disk is glued with epoxy resin in a sample holder and allowed to set over night. The sample holder is fastened between two heads on the apparatus which have gasket seals. The difference between the sample buret and tank buret is measured with the tank open to the atmosphere and without pressure and recorded as h1. After buret measurements the tank buret valve is closed and the tank pressurized continuously with air at a known pressure set with a regulator. The inlet pressure and outlet pressure are recorded. Water is forced from a tank through the sample and into a buret at the sample head outlet. The time that it takes to push a known amount of water through the sample is recorded. After flow and time measurements the difference between the sample buret and tank buret is again measured with the tank open to the atmosphere and without pressure and recorded as h2.

The permeability is calculated using the following equation:

$K = (QxL)/(AxH)$ = Permeability (cm/sec) where
$h = (h1 + h2)/2$ (cms)
$V = V2 - V1$ (cm^3)
$T = T2 - T1$ (sec)
$Q = V/T$ = water flow rate (cm^3/sec)
L = length of the sample (cms)
A = area of sample (cm^2)
$H = (P_{in} - P_{out})x70.31 + h$ (cms)

h1 is the measured distance in centimeters between the top of the tank manometer level and the top of the sample holder manometer when the permeameter is open to the atmosphere before the analysis is performed. h2 is the measured distance in centimeters between the top of the tank manometer level and the top of the sample holder manometer when the permeameter is open to the atmosphere after the analysis is performed. V is the change in volume of the sample manometer (V2 – V1) from the beginning to the end of time interval , T = T2 - T1. P_{in} and P_{out} units are pounds per square inch (psi). The 70.31 is a factor to convert psi to centimeters in the H equation.

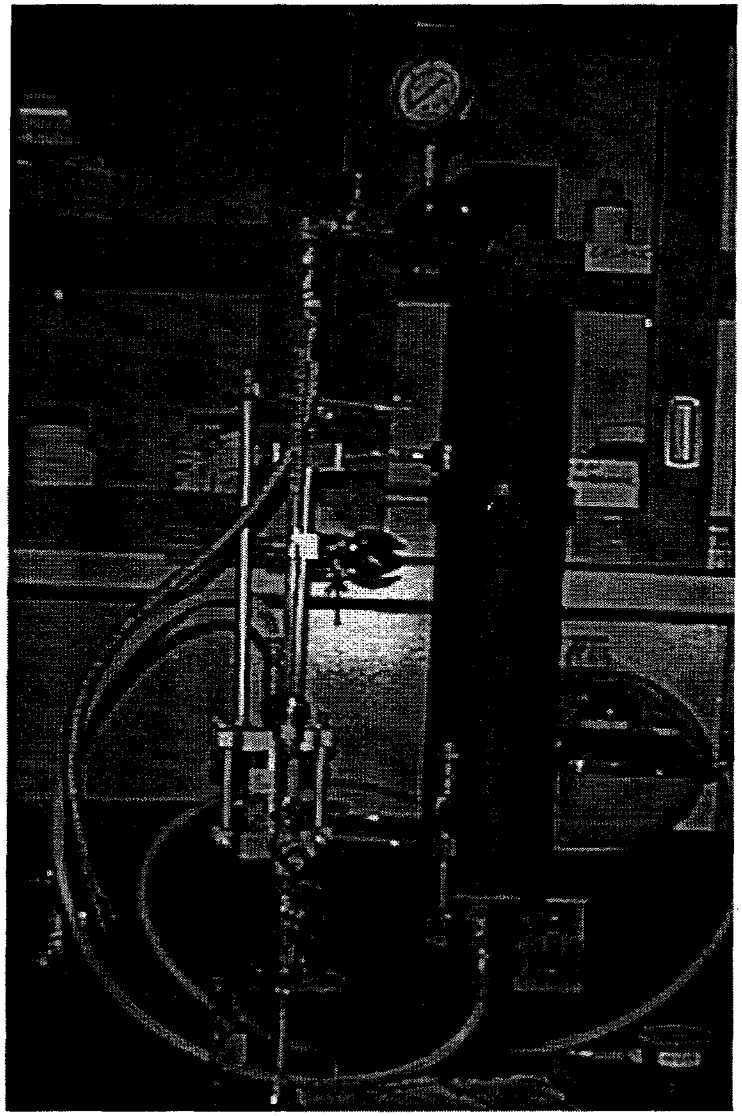


Figure 1. ELE Permeameter

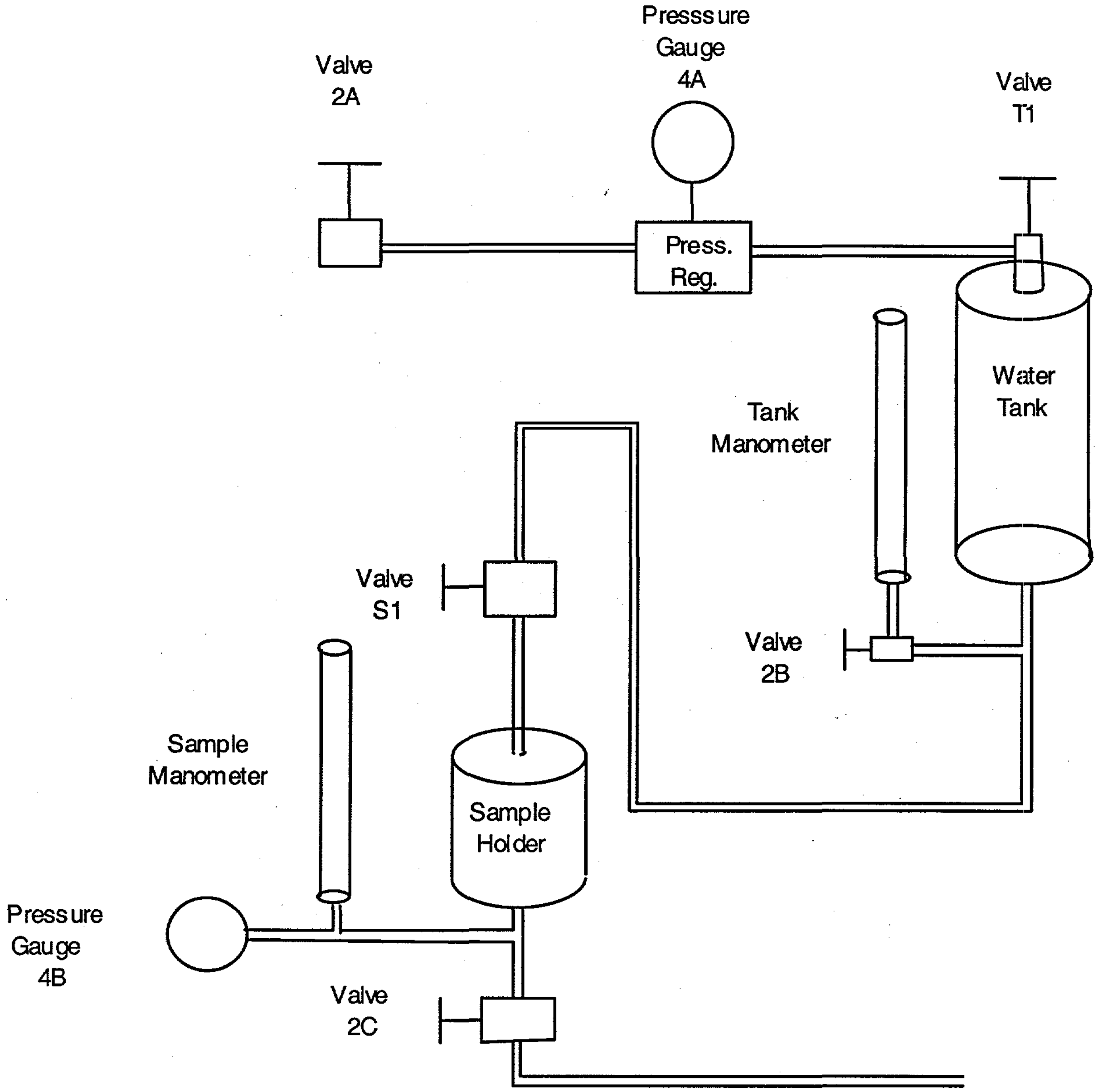


Figure 2. Permeameter Schematic

UFA Centrifuge Method

The permeability results using the Falling Head method were compared to results obtained by UFA Ventures with an open flow Centrifuge method on duplicate samples.

The centrifuge method is effective because it allows the operator to set the variables in Darcy's Law. Darcy's Law states that the fluid flux equals the permeability times the fluid driving force. The driving force is fixed by imposing an acceleration on the sample through an adjustable rotation speed. The flux is fixed by setting the flow rate into the sample with an appropriate constant flow pump and dispersing the flow front evenly over the sample. Thus, the sample reaches the steady state permeability, which is dictated by that combined flux and driving force.

A UFA instrument consists of an ultracentrifuge with a constant, ultra-low flow pump that provides fluid to the sample surface through a rotating seal assembly and microdispersal system. The apparatus can reach accelerations of up to 20,000 g, temperatures can be adjusted from – 20 degrees to 150 degrees C. Effluent from the sample is collected in a transparent, volumetrically calibrated chamber at the bottom of the sample assembly. A diagram of the centrifuge internal parts is shown in Figure 3 and a picture of the instrument is shown in Figure 4.

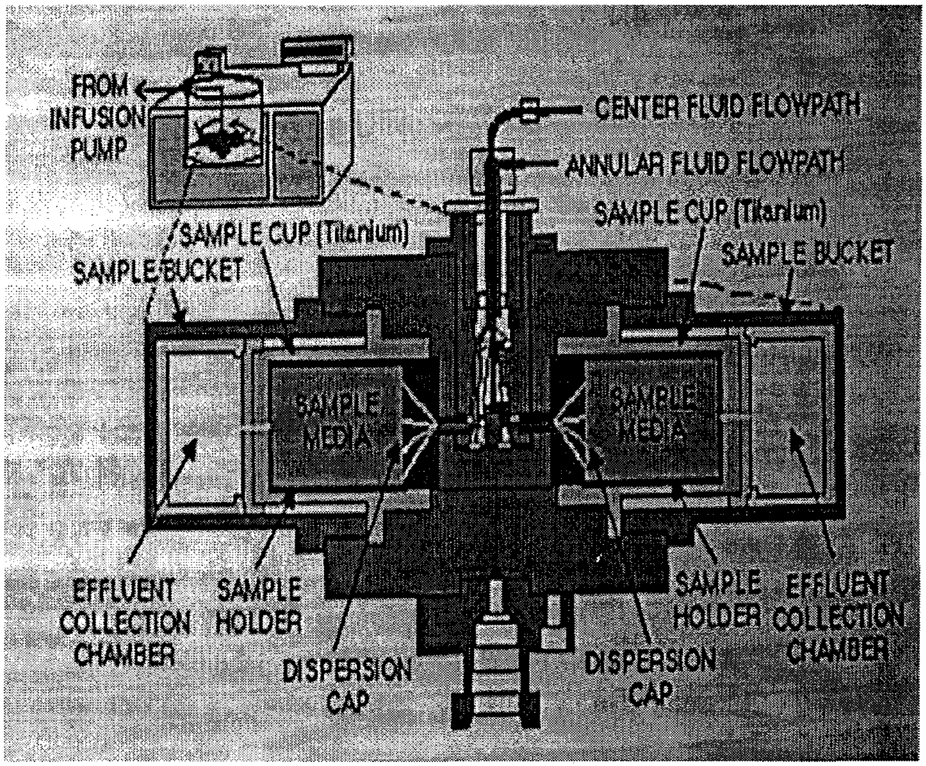


Figure 3. Centrifuge Internal Parts

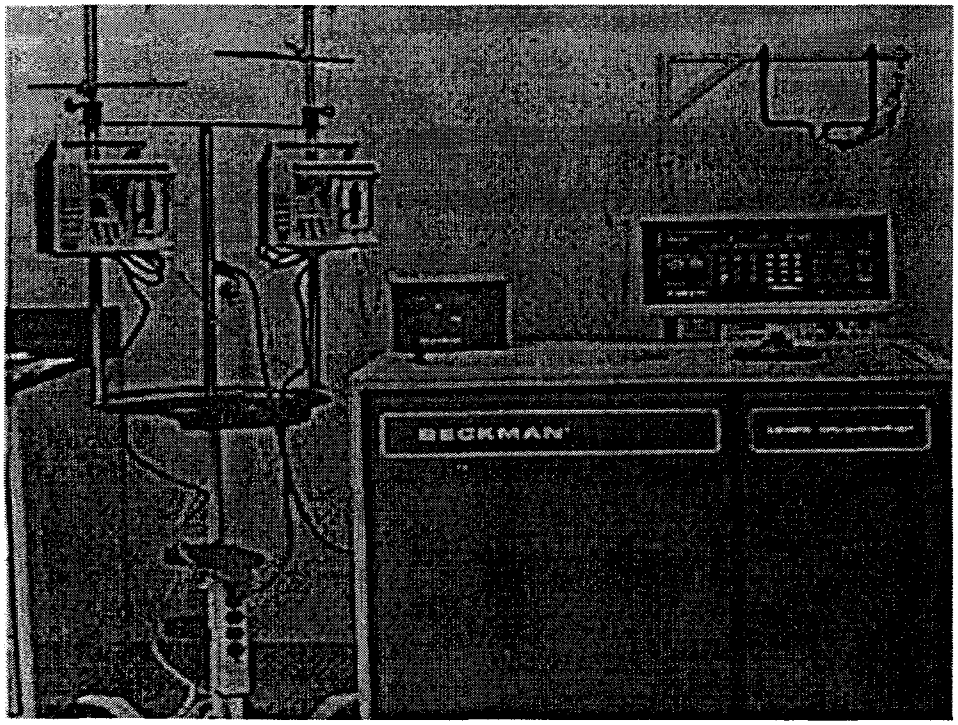


Figure 4. Picture of UFA Centrifuge

RESULTS

Permeability Results

Non radioactive simulant samples made with 10 and 30 wt % NaCl were used to establish a correlation between the falling head and whole body centrifuge permeability results. Results obtained by the centrifuge method were one order of magnitude higher (10x more permeable) than those obtained by the falling head method.

An Ashcrete sample made at SRTC and a sample collected from the plant Ashcrete Facility met permeability requirements when tested by the falling head method. The 10x correlation was used to estimate that Ashcrete centrifuge method results would also meet permeability requirements. Blowcrete made with 1 and 40 wt % scrubber solution from burning Purex waste passes permeability requirements when analyzed using the falling head method. The 10x correlation used to estimate the centrifuge method results predicts these samples would not meet permeability requirements.

Permeability test results are summarized in Table III:

Table III. Waste Form Permeabilities

Sample Number	Sample Description	Falling Head Permeability (cm/s)	E-Area WAC	Centrifuge Permeability (cm/s)	E-Area WAC
1	Simulant with 10 wt % NaCl solution	$<1.7\times10^{-10}$	Pass	1.7×10^{-9}	Pass
2	Simulant with 30 wt % NaCl solution	2.9×10^{-9}	Pass	3.1×10^{-8}	Fail
3	Purex blow crete with 1 wt % solids solution	2.0×10^{-9}	Pass	*	Projected Failure
4	Purex blow crete with 40 wt % solids solution	8.5×10^{-9}	Pass	*	Projected Failure
5	Purex ashcrete	$<4.2\times10^{-10}$	Pass	*	Projected Pass
6	CIF Plant Purex ashcrete	$<7.4\times10^{-10}$	Pass	*	Projected Pass

* Not performed because vendor does not have license for handling radioactive material.

Compressive Strength

Compressive strengths of the first five samples were greater than 700 psi (the upper limit of the Gilson penetrometer) using the penetrometer method.

CONCLUSIONS

All samples prepared in the laboratory met the E- Area WAC compressive strength requirement of 500 psi. The CIF Ashcrete sample prepared in the laboratory and an actual Ashcrete Plant sample (June 1999) both met the E-Area WAC for permeability. The permeabilities of both Ashcrete samples were only measured by the falling head method. The correlation factor of 10x relating the falling head and centrifuge methods indicate that the Ashcrete will also meet the $1x10^{-8}$ cm/sec requirement when measured by the centrifuge method.

CIF Blowcrete made with 1 and 40 wt % total solids blowdown solution met the disposal WAC when measured with the falling head method but did not meet the $1x10^{-8}$ cm/sec criteria using the correlation to determine the centrifuge method result.

As the soluble salt loading in a cement-based waste form increases, the permeability increases. This was observed in the simulant samples (Samples 1 and 2) and in actual blowcrete waste samples (Samples 3 and 4). The phenomena occurs because water flowing through the solid waste form dissolves away the salt and thereby opens the porosity. In addition, the salts affect the water/cement ratio required to obtain processable mixtures (the higher the salt content the lower the water/cement ratio required to achieve processability). Also the porosity and permeability increases as the water to cement ratio increases.

Based on the limited samples tested, results obtained by the centrifuge method were one order of magnitude higher (10x more permeable) than those obtained by the falling head method. Differences in the methods include: 1) sample size and sample preparation, 2) pressure applied to the sample and permeant fluid, 3) time to achieve steady state. In addition, the method of saturating the samples was different in the two laboratories making these measurements.

Sources of error for both methods include: 1) discrepancies in the actual surface area versus the assumed surface area. In the falling head method, care must be taken to prevent epoxy from plugging pores in the thin disk-shaped samples. 2) discrepancies in the degree of saturation of the samples tested. Cement-based waste forms are difficult to saturate because a large percentage of the pores are very small. Lack of saturation will result in lower values. Consequently, determination of the degree of saturation must be measured for each sample after the permeability measurement is made. The degree of saturation should be reported along with the permeability measurement.

Either method of measuring permeability is acceptable for qualifying the CIF Ashcrete and Blowcrete for E-Area disposal. The centrifuge method should be used if available because it is a whole body method and will not have end effects.

RECOMMENDATIONS

Additional samples should be tested to confirm the correlation between the centrifuge and falling head results. In addition, permeability results should be reported as a function of the degree of saturation of the sample used in the test.

Currently, waste forms made with 1 and 40 wt. % blowdown solution pass permeability requirements using the falling head method but fail the permeability requirement using the correlation to determine the centrifuge permeability. Formulation modifications should be considered if the current operation of sending the scrubber solution to ETF is no longer possible. Additional testing is required to determine the maximum salt loading in Blowcrete which will result in waste forms that meet the disposal WAC.

QUALITY ASSURANCE

Quality Assurance testing was conducted in accordance with SRS procedures. Results are recorded in Laboratory Notebook WSRC-NB-96-633. Work Authorization Document 112939 authorized this study[5]. WSRC-RP-99-00267 is the Technical Task Plan that details the work to be performed during the study[6]. WSRC-RP-99-00268 covers the Quality Assurance Plan for work activities[7].

Centrifuge analyses performed by UFA Ventures have been accepted to ASTM D18.21 Subcommittee on Ground Water.

REFERENCES

1. "Low –Level Radioactive Waste Acceptance Criteria," WSRC Manual 1S, Procedure 3.17.

2. J.R. Cook, A.D. Yu, "Radiological Performance Assessment of Trench Disposal of Cement-Stabilized Wasteforms" (U), Rev. 0, WSRC-RP-94-218, April 15, 1994.

3. C. A. Langton, "Determination of Percent Saturation of Porous Solid Materials," Manual 12.1, Procedure IWT-OP-131, August 27, 1999.

4. B. W. Walker, "Procedure for the Operation of the Permeameter," Manual 12.1, Procedure IWT-OP-128, June, 1, 1999.

5. J. Simpson, "Work Authorization Document 112939," October, 7, 1998.

6. B. W. Walker, "Technical Task Plan for Developing a Method for Measuring Permeability" (U), WSRC-RP-99-00267, Rev. 0, March 22, 1999.

7. B. W. Walker, "Quality Assurance Plan for Developing a Method for Measuring Permeability" (U), WSRC-RP-99-00268, Rev. 0, March 22, 1999.

Appendix 1.

Characterization of Incinerator Waste

Off Gas Scrubber Solution (Blowdown) Characterization

Soluble solids were 1 % (wt./vol.). Insoluble solids were 0.2 % (wt./vol.). Most of the solids (>99%) were soluble in a hot 30 wt % NaOH solution. The bulk of the insolubles are likely alumina, silica, and probably titanium dioxide. There was no detectable soluble mercury. The density of the blow down was 1.01 g/ml and the pH was 7.5.

Gross alpha was 539 dpm/ml
Gross beta was 240 dpm/ml
Gross gamma was 154 dpm/ml
Tritium was 1302 dpm/ml

Gamma Contributors

Activity	Component
3.85 pCi/ml	K-40
1.31 pCi/ml	Co-60
226 pCi/ml	Cs-137
2.52 pCi/ml	Eu-154
7.69 pCi/ml	Am-241

Scrubber Solution (Blow down) Characterization

Component	(mg/liter)	Component	(mg/liter)
Silver	<0.003	Manganese	0.712
Aluminum	<0.060	Molybdenum	0.776
Arsenic	0.024	Sodium	2459
Boron	7.75	Nickel	0.075
Barium	0.085	Lead	0.037
Beryllium	<0.0004	Selenium	<0.015
Calcium	39.16	Antimony	10.64
Cadmium	0.032	Silicon	69.5
Chromium	0.009	Strontium	0.065
Cesium	<10	Thorium	<0.50
Copper	1.66	Titanium	<0.001
Iron	0.252	Thallium	<0.015
Potassium	92.53	Uranium	<0.15
Magnesium	12.26	Vanadium	0.091
Zinc	2.43	Zirconium	<0.035
Chloride	988	Sulfate	2050
Carbonate	105		

Ash Characterization

The ash was wet quenched and contained 45 +/- 15 wt. % quench water. Ash used in this study was drained of excess water which resulted in a water content of about 25 wt. %. The pH of the water in contact with the ash was 10.6.

Ash Characterization

Component	(mg/liter)	Component	(mg/liter)
Mercury	0.0042	Selenium	0.255
Silver	0.226	Antimony	29.7
Arsenic	6.96	Thallium	0.416
Barium	238	Copper	646
Beryllium	0.266	Iron	6400
Cadmium	2.57	Manganese	153
Chromium	16.9	Sodium	5450
Nickel	87.4	Titanium	3.9
Lead	44.8	Zinc	846
Cobalt	3.43		

Appendix 2.

Permeability Calculations for Waste Forms

Table A2.1 Measurements and Results of Falling Head Permeability Tests. (Refer to equations on page 6 of report.)

	L	D	A	Man. h 1	Man. h 2	Man. h	P in	P out	H	V2-V1	T2-T1	Q	Perm. K
	(cm)	(cm)	(cm^2)	cm	cm	cm	psig	psig	cm	cm3	hr	(cm^3/sec)	(cm/sec)
Sample 1													
actual	0.80	3.10	7.54	2.00	3.00	2.50	10.00	0.00	705.60	0.00	12.00	0.00E+00	0.00E+00
detect	0.80	3.10	7.54	2.00	3.00	2.50	10.00	0.00	705.60	0.05*	12.00	1.16E-06	1.74E-10
Sample 2													
actual	0.85	3.11	7.59	1.50	2.50	2.00	10.00	0.00	705.10	0.15	2.25	1.85E-05	2.94E-09
detect	0.85	3.11	7.59	1.50	2.50	2.00	10.00	0.00	705.10	0.05*	2.25	6.17E-06	9.80E-10
Sample 3													
actual	0.92	2.90	6.60	1.50	1.40	1.45	10.00	0.00	704.55	0.20	5.50	1.01E-05	2.00E-09
detect	0.92	2.90	6.60	1.50	1.40	1.45	10.00	0.00	704.55	0.05*	5.50	2.53E-06	4.99E-10
Sample 4													
actual	0.99	3.10	7.54	2.00	1.90	1.95	10.00	0.00	705.05	0.90	5.50	4.55E-05	8.46E-09
detect	0.99	3.10	7.54	2.00	1.90	1.95	10.00	0.00	705.05	0.05*	5.50	2.53E-06	4.70E-10
Sample 5													
actual	0.81	3.10	7.54	1.00	0.90	0.95	10.00	0.00	704.05	0.00	5.08	0.00E+00	0.00E+00
detect	0.81	3.10	7.54	1.00	0.90	0.95	10.00	0.00	704.05	0.05*	5.08	2.73E-06	4.17E-10
Sample 6													
actual	1.50	3.20	8.04	1.10	1.00	1.05	10.00	0.00	704.15	0.00	5.00	0.00E+00	0.00E+00
detect	1.50	3.20	8.04	1.10	1.00	1.05	10.00	0.00	704.15	0.05*	5.00	2.78E-06	7.36E-10

* 0.05 cm^3 is the minimum detectable volume which can be measured in the graduated buret. The permeability value reported as detectable is based on achieving steady state flow of 0.05 cm^3 (ml) over the run time interval T2 – T1 reported for each sample. For samples having no actual flow (Q) the permeabilities (K) were reported as less than values in Table III using the minimum detectable flow as the upper-bound estimate.

Appendix 3.

ELE Permeability Procedure

PROCEDURE FOR THE OPERATION OF THE PERMEAMETER (U)

1.0 PURPOSE

The purpose of this procedure is to provide instruction for measuring hydraulic conductivity (water permeability) of waste forms and porous solid materials.

2.0 SCOPE

2.1 This procedure applies to personnel operating the Permeameter.

2.2 The Permeameter has a tank for storing water that is pressurized by a line attached to a laboratory air outlet. The pressure of the air is adjusted by using a pressure regulator. A core sample of the solid waste form is glued into a sample holder which is attached to the apparatus.

2.3 Permeability is determined by measuring the amount of water that flows through the sample over a given time period at a regulated pressure.

3.0 PRECAUTIONS AND LIMITATIONS

3.1 Perform radioactive hood work per the requirements listed on the Radioactive Work Permit.

3.2 When attaching sample holder to apparatus make sure gaskets seal without leaking. Test for gasket leaks with hood window lowered, shield in place, and under low pressure to avoid spraying radioactive liquid out of hood.

3.3 Attach a containment hose around hose from water tank to keep leaks within hood containment.

3.4 Be careful to not tear gloves during sample preparation sawing and sanding to avoid contamination hazards.

4.0 PREREQUISITES ACTIONS

4.1 Obtain samples. Cement waste forms must be cured in sealed containers for a predetermined time. 28 days is the standard time. Other curing times are allowed, however results must be reported and interpreted with respect to curing time.

4.2 Remove solid waste core samples from sample bottles or sample molds. Correctly sized disk forms will reduce the sample preparation. Flatten the top and bottom surface using a hack saw blade and sand paper. Measure the length and diameter of the sample in centimeters and record on data sheet in attachment 2.

4.3 Glue the solid waste core into the sample holder using epoxy resin placed in a syringe. Only the sides of the sample should be glued to the inner surface of the sample holder with no excess glue on the surface of the sample.

4.4 Samples are saturated by immersing in water for 72 hours in a container and applying house vacuum line.

4.5 The weight gain of the sample after saturation must be recorded (on towel dried sample) to estimate the degree of saturation of the sample at the beginning of the test.

4.6 Record a physical description of the sample. Note the size of the aggregate (if present), irregularities in color, texture, cracks, etc.

5.0 PROCEDURE

5.1 Close air valve 2A on permeameter.

5.2 Check permeameter pressure gauge 4A at pressure regulator to verify 0 psig. If system not depressurized contact Researcher before proceeding.

5.3 Close valve S1 on sample holder head assembly.

5.4 Close valve 2C on exit of bottom of sample holder head assembly.

5.5 Close valve T1 on permeameter tank.

5.6 Close valve 2B on tank manometer tube.

5.7 Position the sample holder to the permeameter to contact sealing gaskets on sample assembly and tighten retaining bolts.

5.8 Open valve S1 on sample assembly.

5.9 Check for leaks by placing hood window as low as possible, taping a plastic bag as a shield in front of the permeameter, applying 2 to 5 psig pressure to system by opening Valve 2A, and adjusting the pressure regulator.

5.10 Close valve 2A if leaks are found and perform steps 5.11, 5.12, 5.13, 5.14, 5.15, and 5.16. If no leaks are found close valve 2A and skip steps 5.11 through 5.16.

5.11 Close valve S1.

5.12 Position the sample holder to the permeameter to contact sealing gaskets on sample assembly and tighten retaining bolts.

5.13 Open valve S1.

5.14 Check for leaks by placing hood window as low as possible, taping a plastic bag as a shield in front of the permeameter, applying 2 to 5 psig pressure to system by opening Valve 2A and adjusting the pressure regulator.

5.15 Repeat steps 5.10 through 5.14 until leaks are stopped.

5.16 Turn off valve 2A after leaks have been stopped.

5.17 Add water to buret tube attached to sample holder assembly until close to midpoint level is achieved.

5.18 Add small amount of oil to buret tube on sample holder assembly to stop water evaporation.

5.19 Open air valve 2A and adjust pressure regulator to achieve pressure setting required by researcher.

5.20 Open valve S1 on sample holder assembly.

5.21 Wait 30 minutes to let air in system disappear.

5.22 Close valve S1.

5.23 Close valve 2A.

5.24 Adjust pressure regulator to depressurize system.

5.25 Open valve 2B on tank manometer tube.

5.26 Determine tank level by looking at water level in tank manometer tube.

5.27 If level is not around mid point of tank level remove valve T1 on permeameter tank, insert small funnel, and add water until level is around midpoint.

5.28 Measure distance from top of tank manometer tube level to top of sample holder manometer tube level and record as h1 on data sheet.

5.29 Reattach and close valve T1 to permeameter tank.

5.30 Close valve 2B.

5.31 Open valve 2A and adjust pressure regulator to achieve pressure setting required by researcher.

5.32 Open valve S1 on sample holder, record buret level as V1 on data sheet, and exact time of day S1 is opened as T1 on data sheet.

5.33 Record pressures on gauges 4A as Pin and 4B as Pout on data sheet.

5.34 After period of time required by researcher close valve S1, record exact time valve S1 is closed on data sheet as T2, and buret level in sample holder as V2 on data sheet.

5.35 Close valve 2A and adjust pressure regulator to allow PG4A to read 0 psig.

5.36 Open valve 2B on tank manometer tube.

5.37 Measure distance from top of tank manometer tube level to top of sample holder manometer tube level and record as h2 on data sheet.

5.38 Close valve 2B on tank manometer tube.

5.39 Perform the Permeability calculations using data from the data sheet.

$R = 0.5xD$ (cm)

$A = 3.14 \times R \times R$ (cm^2)

$h = (h1 + h2)/2$ (cm)

$V = V2 - V1$ (cm^3)
$T = (T2 - T1) \times 3600$ (sec)

$Q = V/T$ (cm^3/sec)

$H = (Pin - Pout)70.31 + h$ (cm)

K = Permeability = QL/AH (cm/s)

5.40 Determine the degree of saturation of the sample after completion of the permeability test.[1]

5.41 Samples which produce no flow condition after 48 hours of testing should be run again. One or more of the following actions should be taken.

1) The thickness of the sample should be reduced.
2) The sample surfaces (flat top and bottom) should be sanded and cleaned of dust clogging pores.
3) Pressure (H) in the permeability cell should be increased.
4) Time of the experiment should be increased beyond 48 hours.

5.42 Confirm steady state flow condition as follows: Calculate K for two hour intervals 0-2, 2-4, 4-6, 6-8, etc. if measurable flow is obtained after 2 hours. If not calculate K for four hour intervals. If flow is not obtained after 4 hours use 8 hour intervals. Continue the permeability test for a minimum of 4 time intervals, regardless of the length of the interval required to achieve detectable flow. Compare the 4 K values and determine whether steady state is achieved or approached. If the last two values are within 5 % of each other steady state can be assumed. If not, continue testing. Record and report flow volumes and K values for all four time intervals.

6.0 RECORDS

6.1 Data will be recorded on the data sheet and in a Lab Notebook.

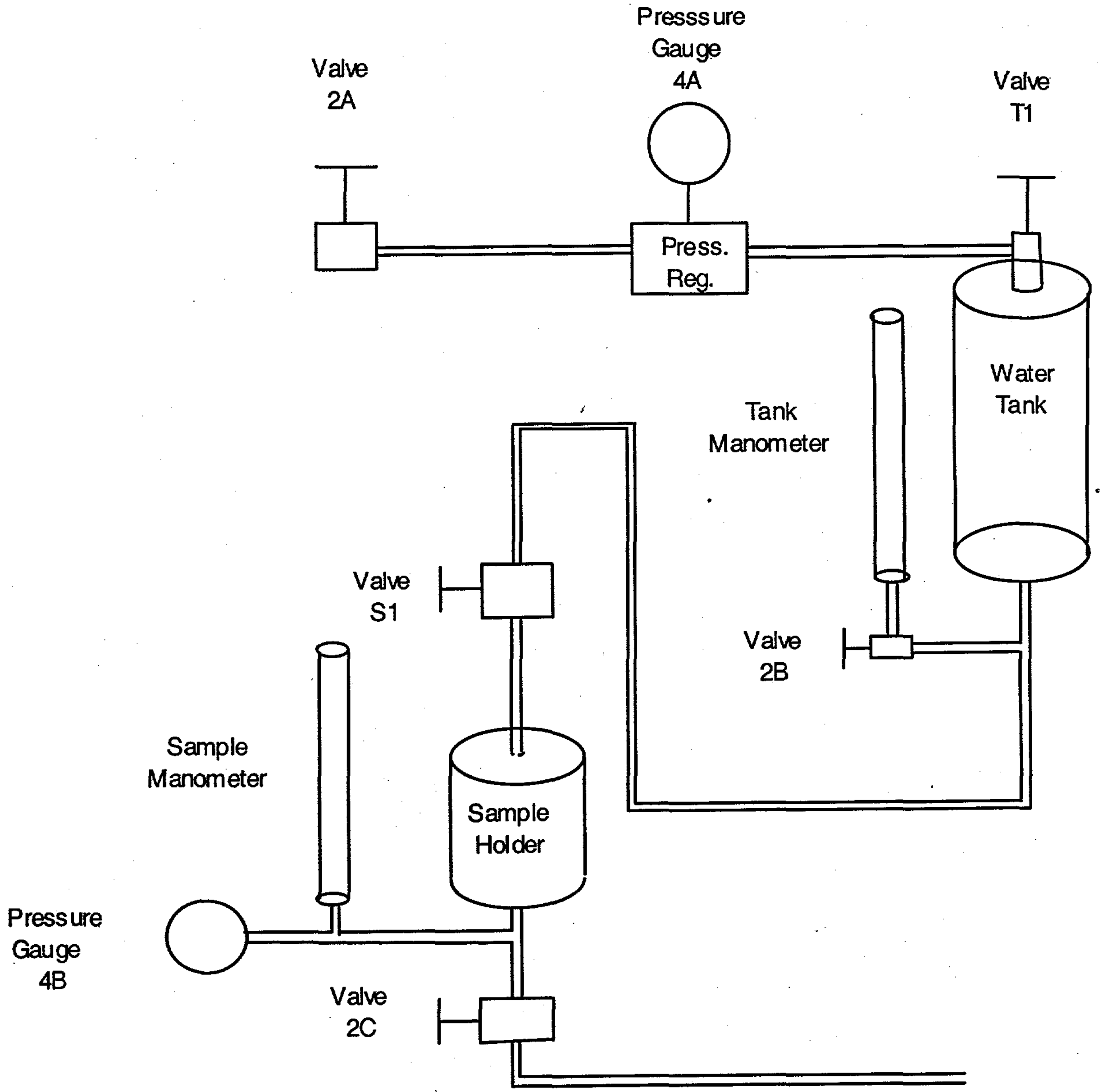
7.0 REFERENCES

1. C. A. Langton, "Determination of Percent Saturation of Porous Solid Materials," Manual 12.1, Procedure IWT-OP-131, August 27, 1999.

8.0 ATTACHMENTS

Attachment 1. Permeameter Sketch

Attachment 2. Data Sheet



Attachment 1. Permeameter Sketch

Attachment 2. ELE Hydraulic Conductivity (Water Permeability) Data Sheet

Project/Task ________________

Sample ID ________________

Test Date ________________

Operator ________________

Physical Description of Sample ____

Weight of sample before saturation ______________________ grams

Weight of sample (towel dried) after saturation ______________________ grams

Percent Saturation of sample after completion of permeability test (Determined using Procedure IWT-OP-131). ___________________

<u>Time Interval 1 Measurements</u>

Step 4.1, L = ______ cm

Step 4.1, D = ______ cm

Step 5.28, h1 = ______ mls

Step 5.32, V1 = ______ mls

Step 5.32, T1 = ______ hrs

Step 5.33, Pin = ______ psig

Step 5.33, Pout = ______ psig

Step 5.34, V2 = ______ mls

Step 5.34, T2 = ______ hrs

Step 5.37, h2 = ______ mls

Perform calculations per Step 5.39,

R = _____ cm
A = _____ cm^2
h = _____ cm
V = _____ cm^3
T = _____ sec
Q = _____ cm^3/s
H = _____ cm
K = Permeability = _____ cm/s

Time Interval 2 Measurements

Step 4.1, L = ______ cm

Step 4.1, D = ______ cm

Step 5.28, h1 = ______ mls

Step 5.32, V1 = ______ mls

Step 5.32, T1 = ______ hrs

Step 5.33, Pin = ______ psig

Step 5.33, Pout = ______ psig

Step 5.34, V2 = ______ mls

Step 5.34, T2 = ______ hrs

Step 5.37, h2 = ______ mls

Perform calculations per Step 5.39,
R = _____ cm
A = _____ cm^2
h = _____ cm
V = _____ cm^3
T = _____ sec
Q = _____ cm^3/s
H = _____ cm
K = Permeability = _____ cm/s

Time Interval 3 Measurements

Step 4.1, L = ______ cm

Step 4.1, D = ______ cm

Step 5.28, h1 = ______ mls

Step 5.32, V1 = ______ mls

Step 5.32, T1 = ______ hrs

Step 5.33, Pin = ______ psig

Step 5.33, Pout = ______ psig

Step 5.34, V2 = ______ mls

Step 5.34, T2 = ______ hrs

Step 5.37, h2 = ______ mls

Perform calculations per Step 5.39,

R = _____ cm
A = _____ cm^2
h = _____ cm
V = _____ cm^3
T = _____ sec
Q = _____ cm^3/s
H = _____ cm
K = Permeability = _____ cm/s

Time Interval 4 Measurements

Step 4.1, L = ______ cm

Step 4.1, D = ______ cm

Step 5.28, h1 = ______ mls

Step 5.32, V1 = ______ mls

Step 5.32, T1 = ______ hrs

Step 5.33, Pin = ______ psig

Step 5.33, Pout = ______ psig

Step 5.34, V2 = ______ mls

Step 5.34, T2 = ______ hrs

Step 5.37, h2 = ______ mls

Perform calculations per Step 5.39,

R = _____ cm
A = _____ cm^2
h = _____ cm
V = _____ cm^3
T = _____ sec
Q = _____ cm^3/s
H = _____ cm
K = Permeability = _____ cm/s