WSRC-RP-92-1029

FILTERING REPRECIPITATED SLURRY (U)

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DE93 006159

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WSRC-RP-92-1029
Keywords: Filters, Cross Flow, Late Washing
Retention: Lifetime

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August 31st, 1992

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Filtering Reprecipitated Slurry (U)

1.0 Abstract

As part of the Late Washing Demonstration, Interim Waste Technology has filtered reprecipitated and non reprecipitated slurry with the Experimental Laboratory Filter (ELF) at TNX. See Attachment 1. Reprecipitated slurry generates higher permeate fluxes than non reprecipitated slurry. Washing reprecipitated slurry may require a defoamer because reprecipitation encourages foaming.

2.0 Introduction

The In-Tank Precipitation (ITP) Process decontaminates radioactive waste in Tank 48 by precipitating Cs^{137} with tetraphenylborate and adsorbing Sr^{90} with sodium titanate. Concentrated tetraphenylborate/sodium titanate slurry is then transferred to Tank 49 for storage. Waste Management will eventually transfer the slurry to the Defense Waste Processing Facility (DWPF) for vitrification, but while waste remains in Tank 49, the $[NO_2^-]$ must remain high to inhibit corrosion. Waste with high $[NO_2^-]$ complicates DWPF processes. To avoid complications, the $[NO_2^-]$ must be reduced to 0.01M.

The Late Washing Process has been proposed to reduce the $[NO_2^-]$ to ≤ 0.01 M. In Late Washing, the KTPB slurry will be washed to reduce the $[NO_2^-]$. Fresh wash water dilutes the nitrite and cross flow filtration removes the wash water after it has reached ambient $[NO_2^-]$.

When slurry remains in Tank 49, cesium tetraphenyl borate undergoes radiolysis, which allows some cesium to dissolve, pass through the filter and contaminate the spent wash water. Reprecipitating the dissolved cesium prevents cesium from contaminating the wash water. This report compares the filterability of reprecipitated and non reprecipitated slurries.

3.0 Experimental

3.1 Equipment

The ELF contains a single sintered stainless steel filter. Late Washing is being designed to use a ITP type cross flow filter. The ITP filter compares with the ELF filter in the following ways:

1. ELF filter internal diameter = 3/8 " — ITP filter internal diameter = 5/8 "
2. ELF filter length = 16 7/8 " — ITP filter Length = 10 '
3. The ELF has 1 element/housing — The ITP filter has 144 elements/housing
4. The ELF permeate/backpulse line is in the center of the housing. The ITP permeate/backpulse line is near the filter exhaust.
5. ELF filter surface area = 0.138 ft^2 — ITP filter surface area = 230 ft^2
6. Both ELF & ITP filters are 0.5 μ Mott HyPulse filters.
7. The ELF uses a Wilden M2 double diaphragm pump with Blacoh surge suppressor. ITP and probably Late Washing will use low shear centrifugal pumps.

Based on surface area the ELF is a 1/1700 model of the ITP filter.

3.2 Cleaning

Performing experiments reproducibly requires starting with a clean filter. Recirculating 1 wt. % oxalic acid and 1 wt. % caustic cleaned the filter. Testing deionized water flux prior to Experiment C & G verified cleanliness. A water flux graph for Experiments C & G appears in Attachment 2.

3.3 Simulant

DWPT prepared non reprecipitated slurry for Experiment C and reprecipitated slurry for Experiment G. The composition appears in Attachment 3. Both slurries had a 2 year equivalent radiation exposure and contained approximately 9.5 wt. % solids. To irradiate slurries DWPT exposed them to $3*10^8$ rad in a ^{60}Co well. This exposure is equivalent to 2 years self irradiation during storage of slurry containing 36 Ci ^{137}Cs/gallon.

3.4 Flux Tests

Transmembrane pressure, flow velocity and reprecipitation affect filter performance. To evaluate these variables two experiments were conducted (Experiments C & G). Each experiment's design was a center point with eight points located around the center in an ellipse (Attachment 4). A one hour test was run at each point, manually backpulsing for approximately one second every fifteen minutes with 90 psi air. The design sequence appears in Attachment 5. Each experiment began at the center, 6 fps & 30 psi. Four points on the ellipse were then tested and the center point was repeated. The next four tests occurred at the remaining points on the ellipse and the final test occurred at the center point. All experiments used the same test sequence.

Flux tests operate in total recycle, so concentrations remain constant. The large feed volume required prohibited operating in once through mode. Pumping and agitation shears the slurry and decreases the permeate flux. Sheared slurry provides a more conservative estimate of filter performance, because slurry which enters the Late Washing Process will have been sheared during ITP.

The flux tests recorded permeate flux at 1 minute intervals before and after backpulsing to determine the effectiveness of the backpulse. The ELF did not contain a backpulse vessel because after backpulsing this vessel would require significant time to refill. Measuring flux immediately after the backpulse was essential to determining the backpulse effectiveness and thus the backpulse vessel was not utilized for these experiments. Operating without a backpulse vessel allowed the the ELF to be backpulsed with air in addition to permeate. This air probably contributed to foaming. Foaming leads to changes in filter performance.

3.5 Calibration

The flow meters were calibrated with graduated cylinder measurements using actual permeate to account for differences in viscosity and specific gravity between water and the simulant. The calibration curves for Experiments C and D appear in Attachments 6 and 7 respectively.

3.6 Results & Conclusions

The Reprecipitation Graph in Attachment 5 demonstrates that reprecipitated slurry generates higher permeate flux than non reprecipitated slurry. For simplicity this graph shows only center points, but at non center points, the reprecipitated slurry also generated higher permeate fluxes. Previous work[1] determined the Mott 0.5 µ filter could filter non reprecipitated slurry and generate fluxes exceeding the Late Washing Facility Design requirements[2]. Reprecipitation should allow the Facility to further exceed design requirements.

Reprecipitation's primary disadvantage is that the reprecipitated slurry foams more readily. Slurry with 2 year equivalent radiation exposure has foamed when exposed to heat and agitation. Since efficient washing requires good agitation and since agitation encourages foaming, a defoamer may be necessary to wash irradiated slurry. 500 ppm SURFYNOL® 104 has proven effective, but lesser concentrations may prove sufficient.

3.7 Error Margins

The ELF uses a diaphragm pump, which causes pressures and flow to fluctuate. The surge suppressor reduces, but does not eliminate fluctuation. Fluctuating pressure and flow make reading pressure and flow gauges difficult. Measurements will have the following error margins:

Measurement	Measuring Device	Typical Value	Error Margins
Concentrate Flow	Flow Meter 1	1.0 - 10.0 gpm	± 0.1 gpm
Permeate Flow (High Flow)	Flow Meter 2	0.1 - 3.0 gpm	± 0.1 gpm
Permeate Flow (Low Flow)	Flow Meter 3	10 - 250 ml/min	± 5 ml/min
Feed Pressure	Pressure Gauge 2	15 - 100 psi	± 4 psi
Concentrate Pressure	Pressure Gauge 3	15 - 100 psi	± 3 psi
Permeate Pressure	Pressure Gauge 5	0 - 75 psi	± 2 psi
Backpulse Duration	Stop watch	1 - 4 seconds	± 0.5 seconds
Backpulse Frequency	Stop watch	5 - 20 minutes	± 0.5 seconds

4.0 Attachments

4.0.1 Attachment 1 - Experimental Laboratory Filter Sketch

4.0.2 Attachment 2 - Water Flux Tests C & G Graph

4.0.3 Attachment 3 - Simulant Composition

4.0.4 Attachment 4 - Late Washing Flux Test Design

4.0.5 Attachment 5 - Late Washing Flux Test Design Datasheet

4.0.6 Attachment 6 - Irradiated Slurry Calibration Curve

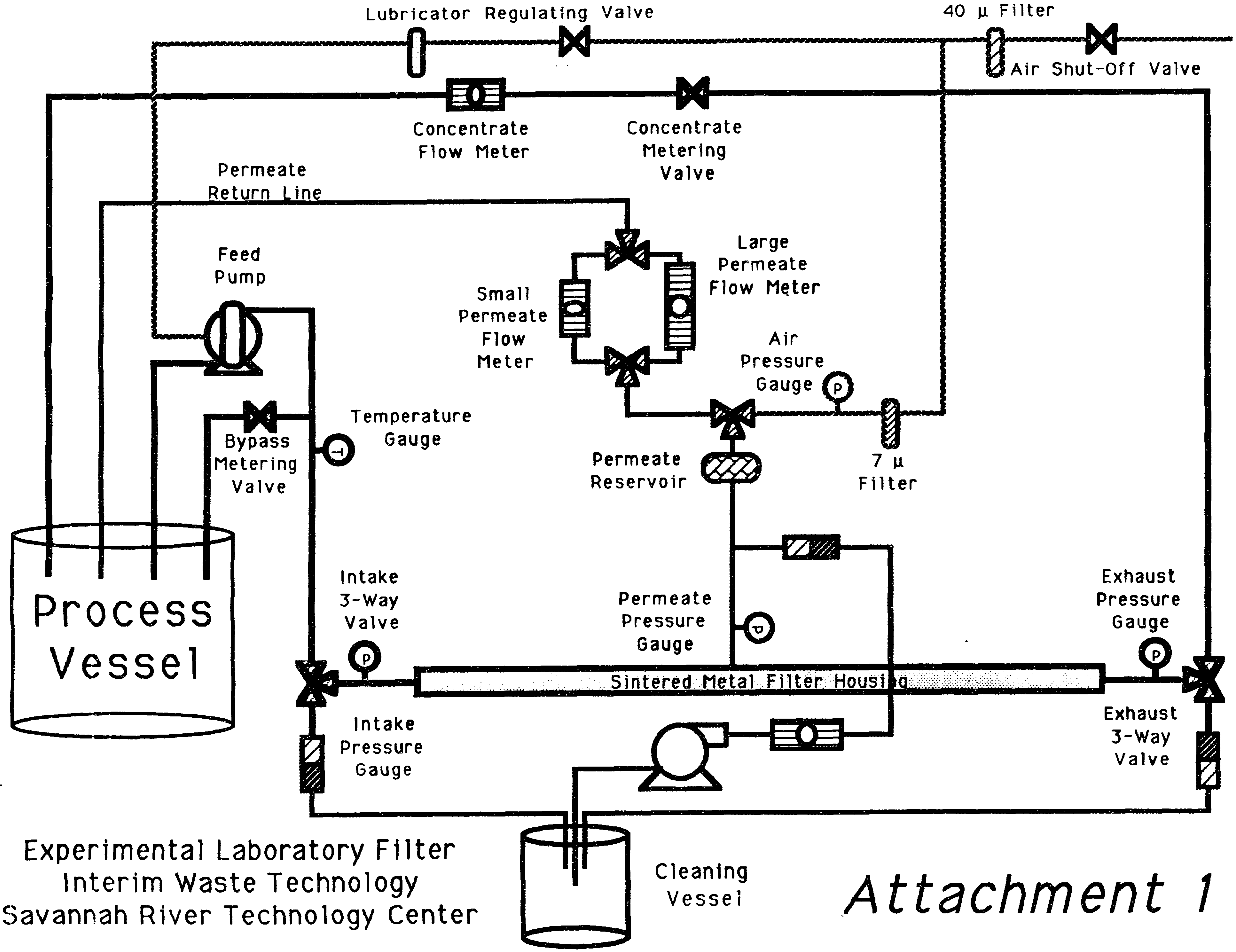
4.0.7 Attachment 7 - Reprecipitated Slurry Calibration Curve

4.0.8 Attachment 8 - Reprecipitation Graph

5.0 References

5.0.1 Reference 1 - L. O. Dworjanyn & M. F. Morrissey, "Initial Technical Basis for the Use of a Spare ITP Filter in DWPF Late Washing (U)", WSRC-RP-92-766, June 5th, 1992.

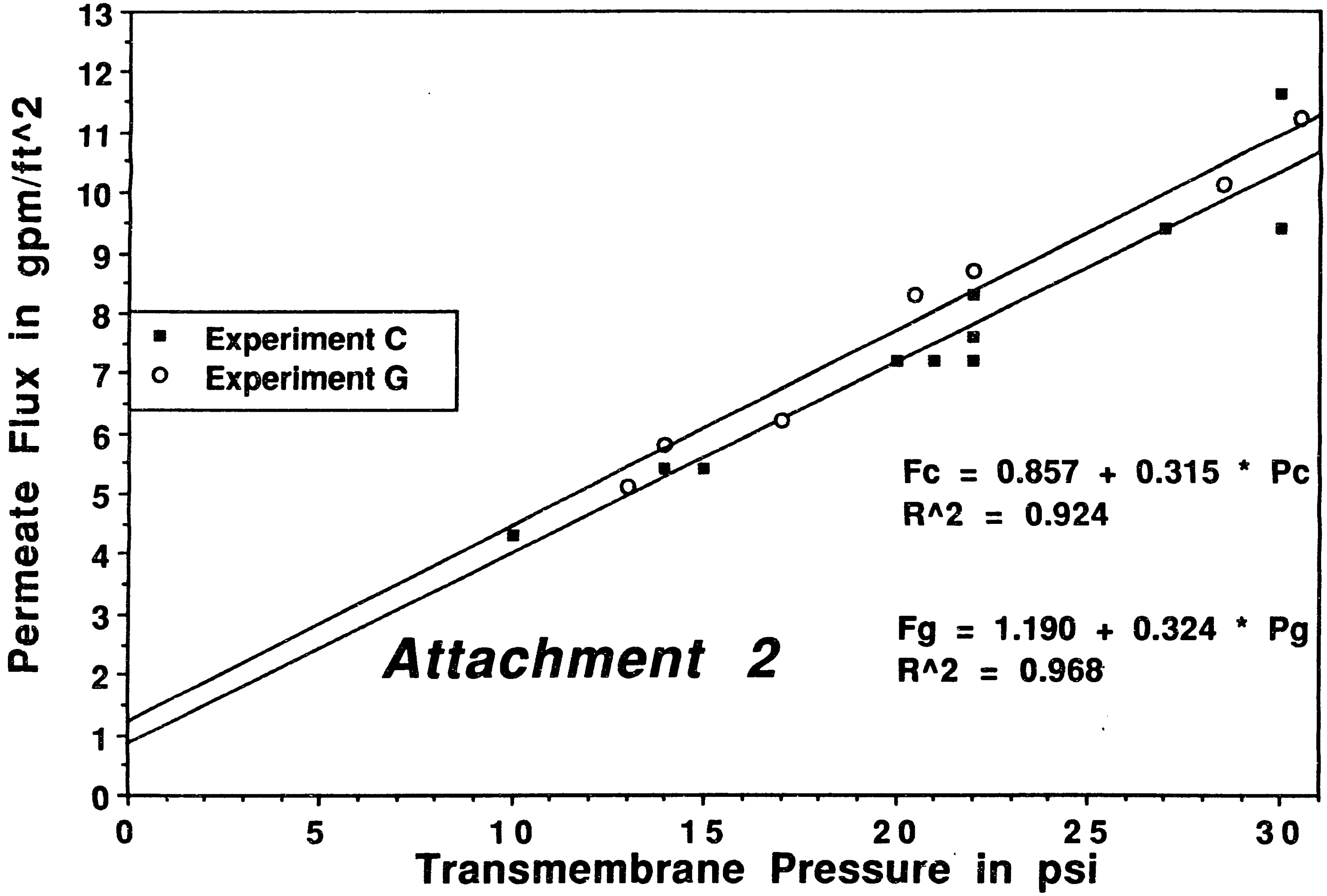
5.0.2 Reference 2 - D. L. Fish & L. F. Landon, "Initial Technical Bases - DWPF Late Washing Facility (U)", WSRC-RP-92-773, June 15th, 1992.



Experimental Laboratory Filter
Interim Waste Technology
Savannah River Technology Center

Attachment 1

Water Fluxes for Experiments C & G



Attachment 3

DWPT first blended the salt solution presented below.

Salt Solution					
Compound	Mass Added	Molecular Weight	Moles	Molarity	Concentration
H2O	6,178.35 grams	18.00 g/mol	343.2417 moles	53.9784 M	952,560 ppm
K2CO3	3.45 grams	138.20 g/mol	0.0250 moles	0.0039 M	532 ppm
KNO2	268.95 grams	85.10 g/mol	3.1605 moles	0.4970 M	41,466 ppm
CsNO3	6.72 grams	194.91 g/mol	0.0345 moles	0.0054 M	1,036 ppm
KNO3	25.02 grams	101.10 g/mol	0.2475 moles	0.0389 M	3,858 ppm
KCl	0.24 grams	74.55 g/mol	0.0032 moles	0.0005 M	37 ppm
KF	0.13 grams	58.10 g/mol	0.0022 moles	0.0004 M	20 ppm
Na2SO4	2.87 grams	141.98 g/mol	0.0202 moles	0.0032 M	442 ppm
K2CrO4	0.09 grams	194.19 g/mol	0.0005 moles	0.0001 M	14 ppm
Na2SiO3*9H2O	0.16 grams	284.07 g/mol	0.0006 moles	0.0001 M	25 ppm
K2MnO4	0.01 grams	197.13 g/mol	0.0001 moles	0.0000 M	2 ppm
C2H5B(OH)2	0.06 grams	94.00 g/mol	0.0006 moles	0.0001 M	9 ppm
Total Mass	6,486.05 grams			Total Volume	6.359 liters

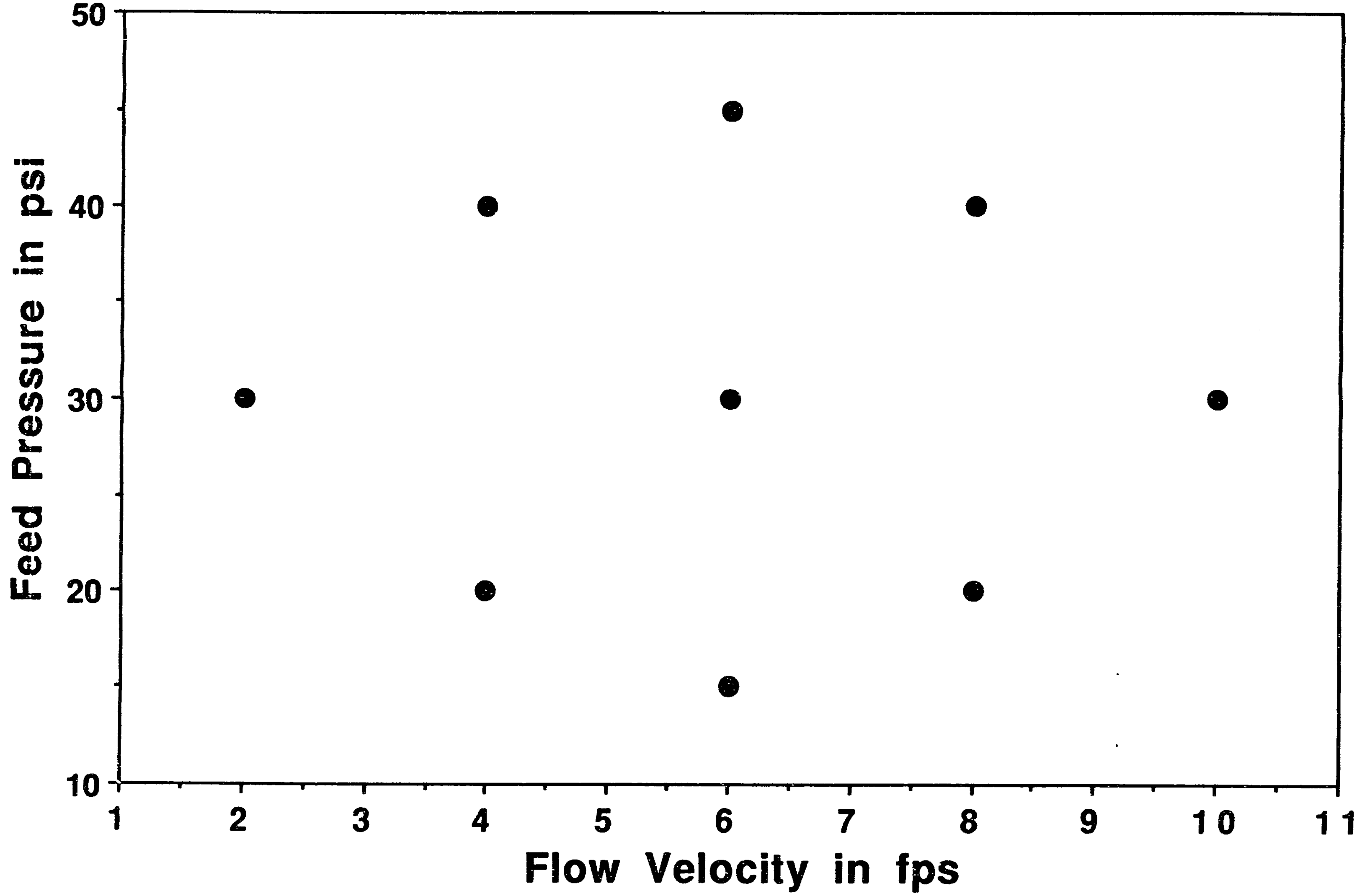
DWPT then added salt solution to NaTPB solution, sodium titante slurry & diphenyl mercury.

Slurry					
Compound	Mass Added	Molecular Weight	Moles	Molarity	Concentration
Salt Solution	3,243.03 grams	N/A	N/A	N/A	N/A
NaTPB Solution	3,534.90 grams	341.79 g/mol	1.7375 moles	0.2532 M	504,986 ppm
NaTi2O5H Slurry	152.50 grams	453.73 g/mol	0.0309 moles	0.0045 M	21,786 ppm
(C6H5)2Hg	22.07 grams	478.18 g/mol	0.0462 moles	0.0067 M	3,153 ppm
H2O	47.50 grams	18.00 g/mol	2.6389 moles	0.3845 M	6,786 ppm
Total Mass	7,000.00 grams			Total Volume	6.863 liters

Final Slurry Composition					
Compound	Mass Added	Molecular Weight	Moles	Molarity	Concentration
H2O	6,216.22 grams	18.00 g/mol	345.3454 moles	50.3218 M	888,031 ppm
Na	41.13 grams	22.99 g/mol	1.7891 moles	0.2607 M	5,876 ppm
K	66.75 grams	39.10 g/mol	1.7072 moles	0.2488 M	9,536 ppm
CO3	0.75 grams	60.00 g/mol	0.0125 moles	0.0018 M	107 ppm
NO2	47.41 grams	30.00 g/mol	1.5802 moles	0.2303 M	6,772 ppm
NO3	6.48 grams	46.00 g/mol	0.1410 moles	0.0205 M	926 ppm
Cl	0.06 grams	35.45 g/mol	0.0016 moles	0.0002 M	8 ppm
F	0.02 grams	20.00 g/mol	0.0011 moles	0.0002 M	3 ppm
SO4	0.97 grams	96.06 g/mol	0.0101 moles	0.0015 M	139 ppm
CrO4	0.03 grams	116.00 g/mol	0.0002 moles	0.0000 M	4 ppm
SiO3	0.02 grams	76.09 g/mol	0.0003 moles	0.0000 M	3 ppm
MnO4	0.00 grams	118.94 g/mol	0.0000 moles	0.0000 M	0 ppm
C2H5B(OH)2	0.03 grams	94.00 g/mol	0.0003 moles	0.0000 M	4 ppm
TPB	593.86 grams	341.79 g/mol	1.7375 moles	0.2532 M	84,838 ppm
Ti2O5H	5.46 grams	176.80 g/mol	0.0028 moles	0.0004 M	779 ppm
(C6H5)2Hg	22.07 grams	478.18 g/mol	0.0462 moles	0.0067 M	3,153 ppm
Total Mass	7,000.00 grams			Total Volume	6.863 liters

Flux Test Design

Attachment 4



Attachment 5

Late Washing Flux Test Design Datasheet

Test	F conc	V flow	P feed	T	P conc	F perm	P perm	P trans	P flux
1	2.06 gpm	6 fps	30 psi						
2	2.75 gpm	8 fps	40 psi						
3	0.69 gpm	2 fps	30 psi						
4	2.06 gpm	6 fps	15 psi						
5	2.75 gpm	8 fps	20 psi						
6	2.06 gpm	6 fps	30 psi						
7	1.38 gpm	4 fps	40 psi						
8	2.06 gpm	6 fps	45 psi						
9	3.44 gpm	10 fps	30 psi						
10	1.38 gpm	4 fps	20 psi						
11	2.06 gpm	6 fps	30 psi						

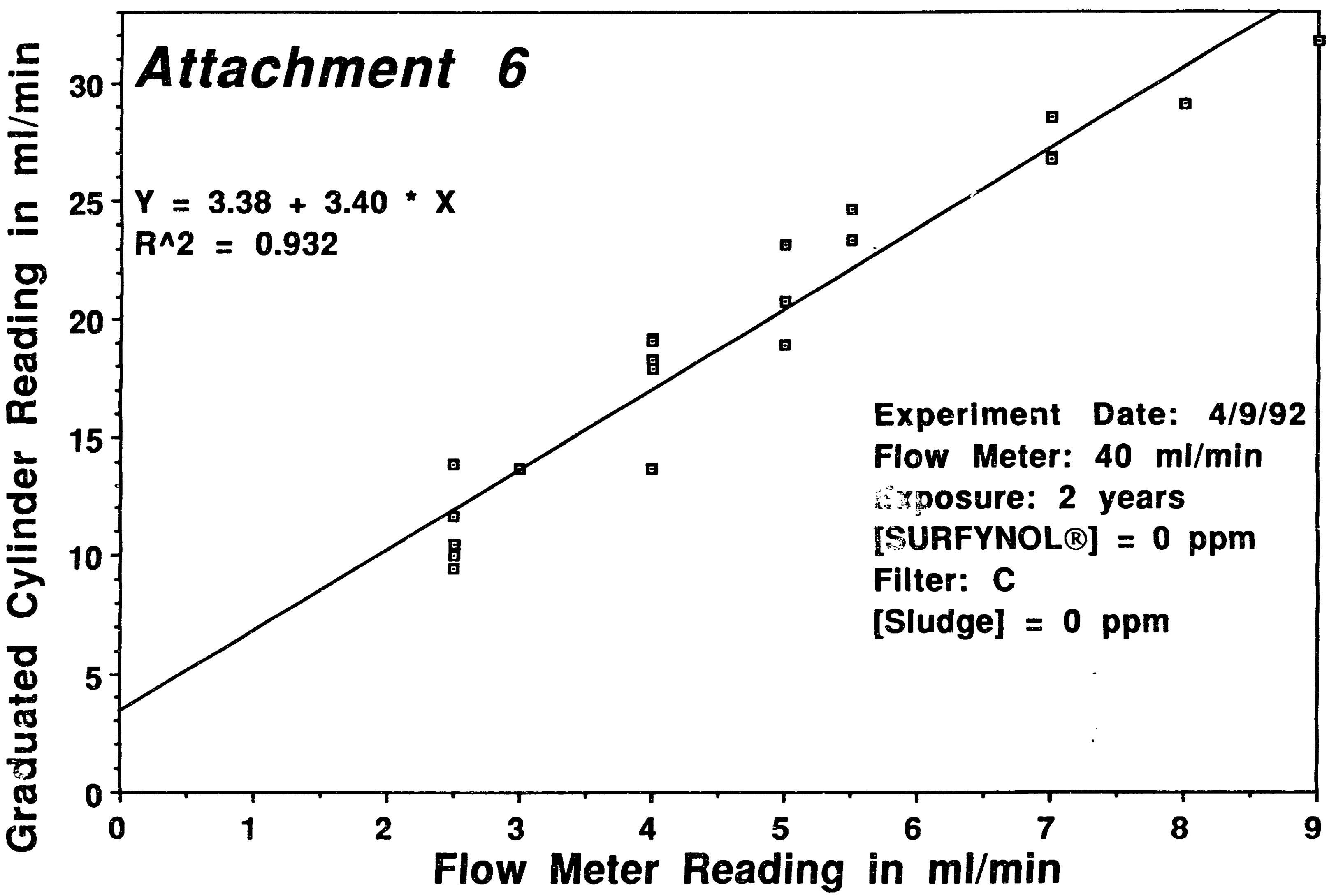
2 Hour Static Interval

Test	F conc	V flow	P feed	T	P conc	F perm	P perm	P trans	P flux
12	2.06 gpm	6 fps	30 psi						
13	2.75 gpm	8 fps	40 psi						
14	0.69 gpm	2 fps	30 psi						
15	2.06 gpm	6 fps	15 psi						
16	2.75 gpm	8 fps	20 psi						
17	2.06 gpm	6 fps	30 psi						
18	1.38 gpm	4 fps	40 psi						
19	2.06 gpm	6 fps	45 psi						
20	3.44 gpm	10 fps	30 psi						
21	1.38 gpm	4 fps	20 psi						
22	2.06 gpm	6 fps	30 psi						

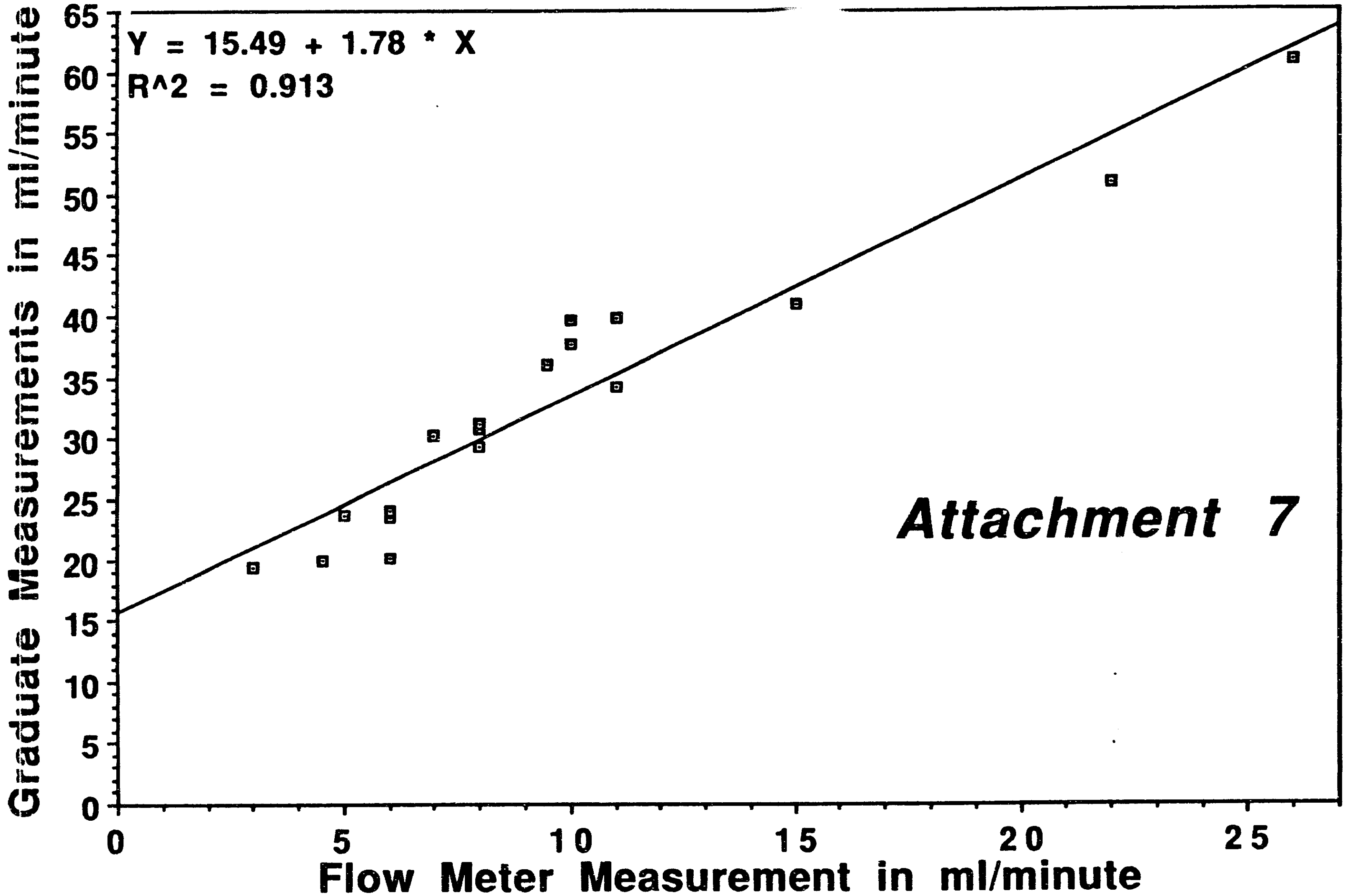
24 Hour Static Interval

Test	F conc	V flow	P feed	T	P conc	F perm	P perm	P trans	P flux
23	2.06 gpm	6 fps	30 psi						
24	2.75 gpm	8 fps	40 psi						
25	0.69 gpm	2 fps	30 psi						
26	2.06 gpm	6 fps	15 psi						
27	2.75 gpm	8 fps	20 psi						
28	2.06 gpm	6 fps	30 psi						
29	1.38 gpm	4 fps	40 psi						
30	2.06 gpm	6 fps	45 psi						
31	3.44 gpm	10 fps	30 psi						
32	1.38 gpm	4 fps	20 psi						
33	2.06 gpm	6 fps	30 psi						

Irradiated Slurry Calibration Curve



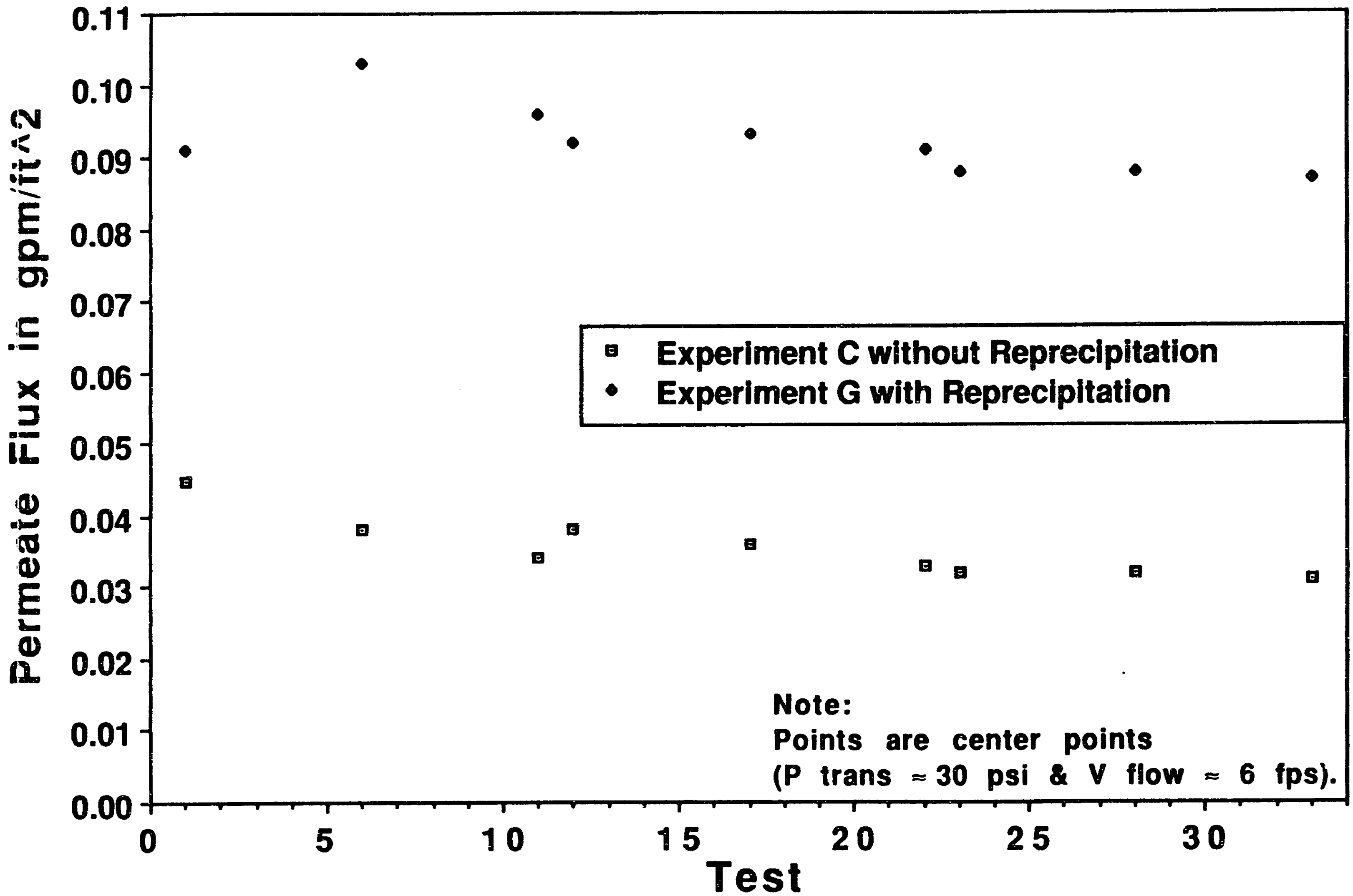
Reprecipitated Slurry Calibration Curve



Attachment 7

Reprecipitation Graph

Attachment 8



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