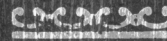


308 --- Q199704250031
Scientific Notebook #040

040



21
300

R

VITREOUS STATE LAB (VSL)

PI: HERSH K. MANAKTALA

SWRI BLDG. #57. TEL: 512-522-5210

The Boorum & PeaseTM Quality Guarantee

The materials and craftsmanship that went into this product are of the finest quality. The pages are thread sewn, meaning they're bound to stay bound. The inks are moisture resistant and will not smear. And the uniform quality of the paper assures consistent rulings, excellent writing surface and erasability. If, at any time during normal use, this product does not perform to your expectations, we will replace it free of charge. Simply write to us:

Boorum & Pease Company
71 Clinton Road, Garden City, NY 11530
Attn: Marketing Services

CNWRA
CONTROLLED
COPY 640

One Good Book Deserves Many Others.

Look for the complete line of Boorum & PeaseTM Columnar, Journal, and Record books. Custom-designed books also available by special order. For more information about our Customized Book Program, contact your office products dealer. See back cover for other books in this series.

Made in U.S.A.

TABLE OF CONTENTS

1

		Date	page no.
1.	"Dry Run" of Partial PCT Measuring WV Glass	1/10/92	5.
2.	Profiling/Calibration of Blue-M Low Temp. Oven	1/23/92	9
3.	Low-Temp. Blue-M Furnace Recalibration	1/31/92	11
4.	Blue-M Low. Temp. Oven Recalibration	1/5/92	13
5.	Leaching Vessel Cleaning	2/10/92	15
6.	Series I (AA) PCT test on Borosilicate Glasses Powdered/Crushed	2/12/92	18
7.	— " — Leaching Test Set-up Data	2/19/92	20-21
8.	Preparation of Leachate Samples for Analyses	2/19/92	22
9.	2 nd Series of PCT on Borosilicate Glass Samples - Cleaning of Leaching Vessels	3/8/92 3/16/92	23 23
10.	Data for PCT Series II (AB) Tests - Test Set-up	3/2/92	25
11.	Series IV (AC) PCT Leaching Tests on Borosilicate Glass	3/9/92	29
12.	Leaching Vessel Cleaning for the new test series	5/26/92	33

**Pages 2 and 3 Are Intentionally
Left Blank**

INITIAL ENTRIES

This lab notebook will be used primarily for documenting leaching studies of vitrified waste forms (glasses). The test specimens will only contain simulated wastes, i.e., no radioactive materials or compounds will be used (unless an exception is clearly identified).

The experiments documented in this lab notebook will be conducted by HERSH K. MANAKTALA or designated laboratory or technical staff who has been qualified and instructed about the proper use of equipment, test materials, and safety precautions.

The objective of the experiments are: (1) to develop vitrified waste leaching capabilities at CNWRA/SWRI, (2) conduct test on standard wasteforms and wasteforms that have been tested in other laboratories for comparison of the results and determine lab to lab variability, and (3) to develop data to support the standardization of the leaching test as an ASTM standard test method.

Calibrated test equipment such as furnaces, beakers, purity of chemicals, test temperatures, frequency of data gathering are described in other documents, viz. TOP-019, TOP-020, and TOP-021.

Appropriate level of details will be documented in this lab notebook for tests conducted under procedures that are not available as CNWRA/SWRI procedures in order to facilitate their reproducibility at a later date.

HKM
10/13/94

"Dry Run" of Partial PCT Using WVDP Glass January 10, 1992.

"Dry Run" of leaching test procedure using West Valley glass - composition typical of WVDP but chemical analyses unknown. Test performed to identify any show stoppers.

- Uncrushed glass black in color. Crushed & sieved glass golden-brown in color. Used sieve size -40 + 200 (unwashed).

- Leaching Vessel used: 22 ml Parr Instrument Type 316L SS with closed top & teflon washer.

Empty Vessel: 61.7654 g. (Vessel alone)

With lid: 115.3046 g. (Vessel + lid + washer)

Complete Assembly: 301.05 g.

Weighed on Mettler PM4600 Delta Range Balance
Serial No. 102461

- Glass powder was weighed on "Redi-Weigh" weighing papers (circles) - Fisher Catalog # 09-878-25A.

- 1 g. (nominal) glass sample was used.

Empty weighing paper: 0.4531 g.

Paper + glass sample: 1.4577 g.

Paper after transferring glass to leaching vessel: 0.4550 g.

Difference in wt. of paper after & before weighing glass: +0.0019 g.

Glass weighed on Mettler AE 240 Analytical Balance Serial No. 101237 in Glo Series Lab.

The difference is attributed to weighing error (balance instability, absorption of moisture in the balance chamber, and possibly due to small amount of glass powder that may have remained stuck to the weighing paper after transfer of the glass powder to the leaching vessel.

- Wt. of leaching vessel with lid & glass:
116.2980 g.

- Addition of 10 ml of Nanopure II water (de-ionized) meeting the requirements of ASTM Grade I DI water was made — using a graduated pipette with a Brinkmann Pipette Helper.

Wt. of leaching vessel + lid + water (10ml)
= 126.2652 g.

- The cap was placed on the vessel & tightened with a nut using the tools appropriate for the leaching assembly.

Wt. of the leaching vessel
assembly complete + glass: 302.04 g.

Wt. of leaching vessel
assembly complete + glass + water (10ml): 312.02 g.

- The glass powder was weighed using calibrated Mettler AE 240 Analytical Balance Serial No. 101237 located in the Geosciences lab in Bldg 57.

- The complete assembly (which is outside the weighing range of the Mettler AE 240 Analytical Balance) was weighed using calibrated Mettler PM 4600 Delta Range Top Loader Pan Balance Serial No. 102461 located in the Geosciences lab in Bldg 57.

- A single layer of teflon tape was used on the leaching vessel threads to facilitate opening.

- The assembled leaching test assembly was placed in a pre-heated forced-air convection oven model — Blue M "Stabil-Therm" Electric Oven with Pro-Set 4 Controller. Serial No. OV-5697 Model No. OV-510A-3 with an operating temperature range of 38°C – 260°C per manufacturer's specifications — The oven draws a line current of 15.5 A.

- The oven was used in the "factory calibrated" condition, i.e. per factory/manufacturer's certificate the oven was calibrated at 150°C . The operating temperature for the "dry run" test in the exercise used a temperature setting of 90°C . [The error/difference between the set-temp. & the temperature of the leaching vessel was not determined].

- The single leaching vessel was placed in the middle of the oven on wire rack no. 2. The test was initiated at 3:00 pm. The weight at the beginning of the test was 312.02 g.
- The vessel was removed from the oven on January 11, 1992 at 3:00 pm — was allowed to cool to room temperature (~ 1 hour) and was reweighed. The wt. after 1 day was 312.01 g. The test vessel was re-inserted in the oven at 90°C .
- The leaching vessel was removed on January 17, 1992 (after seven days test) at 3:00 pm — it was allowed to cool to RT (~ 1 hr) & was reweighed — The weight was; 312.00 g.

0.0032% ~~ABM~~ 1/17/92

- The calculated weight loss was ~~0.33%~~ after one day, and after seven (7) days the weight loss was 0.0064%. These numbers are well within those considered acceptable by ASTM test requirements for PCT (5% mass loss of water).

- The mass loss of water for 1 day is calculated to be 0.1% and for 7 days it's 0.2%. This is a conservative estimate as some loss is attributable to evaporation of lubricant/grease on the leading vessel assembly threadings. [The vessel used for this test was brand new].

- This completes the first "dry run" to determine any difficulties in operating the oven & the weighing glass, preparing the leading vessel assembly set-up, exposing the test assembly to the oven environment, and maintaining the required amount of headspace in the vessel throughout the duration of the test to within the specifications.

- The DI water used for the test was de-ionized using Ultropure - Reverse Osmosis System - Nanopure II water with a min. of 16.7 megohm-cm resistivity.

ABM

Jan 17, 1992

Jan 23, 1992 . 9

Profiling / Calibration of Blue-M Low Temp Oven


Date / Time	Furnace Setting (°C) (LED Display)	Furnace Reading (LED Display)	External Mercury Thermometer	Ambient Reading #	
				Ext. Mercury Thermometer	Ext. Mercury Thermometer
			-151.01°C / 1/10°C grad. Fisher # 15041B SWPE Calib. 10th 1991 ID # 893-3133 GS Lab D	SWPE # 125-30.01 TWP Cal	SWPE # 123-8003 TWP Cal
	A	B	C	E	F
1/23/92	4:30 pm	Set at 00°C	22/23°C	24.5°C	23.8°C
	5:00 }	30°C	↓	↓	↓
	5:30 }	30	34	35.8	23
	5:30 }	35			
	6:00 }	35	40/41	42.4	22.5
	6:00 }	40			
	6:30 }	40	44	45.8	22
	6:30 }	45			
	7:00 }	45	46	48.2	21
	7:00 }	51			
	7:30 }	51	52	53.6	20.5
	7:30 }	55			
	8:00 }	55	55/56	57.7	20.5
	8:00 }	60			
	8:30 }	60	60/61	62.4	20.2
1/24/92	7:30 am	60	60/61	62	18
	7:30 }	65			
	8:30 }	65	65/66	66.8	18
	8:30 }	70			
	9:00 }	70	70/71	71.8	18
	9:00 }	76			
	9:30 }	76	76/77	77.5	18
	9:30 }	80			
	10:00 }	80	80/81	81.7	18.8
	10:00 }	85			
	10:30 }	84/85	85	85.9	19.2
	10:30 }	90			
	11:00 }	90	90/91	91.4	20

* External Thermometer up to 20°C marking inside the furnace left-hand side wall past the rubber stopper. Cont. next page -

Approx. 12" from the furnace - measured by hanging thermometer

* Column 4 p. 9. Foot note: Hg Thermometer -1 to 101°C with 1/10°C
Calibration - Foster # 15041B - Foster USA 893-313 -
SRI Calibration 10/1/91 Newr. Calib. 10/1/92 ID # 893-3133

Jan 31, 1992.

* Thermometer inserted upto 100°C in the rubber stopper

 The rubber stopper was inserted in the LHS wall of the furnace (in the existing hole).

- Cond. -

A	B	C	D	E
Date/Time	Ext. Hg. Thermometer AMBIENT TEMPERATURE # 123-8002 -10°C to 260°C/ 1°C grad. (EwPE Lab)	Ext. Hg. Thermometer # 123-8003 -10 to 260°C/ 1°C grad. (EwPE Lab)	Furnace Setting (LED display)	Furnace Reading (LED display)
2:30 pm } 3:00 }	22.3	90	90	90/91
3:00 } 3:30 }	22.5	94	95	95
3:30 } 4:00 }	23	98.9	100	100
4:00 } 4:30 }	23	104.5	106	106/107
4:30 } 5:00 }	23	108.1	110	110/111
5:00 } 5:30 }	23	113	115	115/116
5:30 } 6:00 }	22.6	117	120	120
6:00 } 6:30 }	22	122	125	125
6:30 } 7:00 }	22	126.4	130	131
7:00 } 7:30 }	21.8	130	135	135
7:30 } 8:00 }	21.4	133	139	139
8:00 } 8:30 }	21.2	139.8	146	146
8:30 } 9:00 }	21	143.8	150	150
9:00 } 9:30 }	20.8	147.9	155	155
9:30 } 10:00 pm }	20.7	152.0	160	160

Cond.

- Contd. -

A	B	C	D	E
Date/ Time	Ext. Hg. Thermometer AMBIENT Temp. # 123-8002 -10°C to 260°C/ 1°C grad. (EwPE Lab)	Ext. Mercury Thermometer # 123-8003 -10 to 260°C/ 1°C grad. (EwPE Lab)	Furnace Setting (LED display)	Furnace Reading (LED display)
10:00 } 10:30 pm }	20.6	156.6	165	165
10:30 } 11:00 }	20.2	161.5	171	171
11:00 } 11:30 pm }	20.3	165.4	175	175
H Bell 1/31/92				
Blue M Low-Temp. Oven Recalibration Feb 5, 1992 Wed.				
Date/ Time	Ambient Temp. # 123-8002 -10 to 260°C/ 1°C grad. (EwPE Lab)	Furnace Reading (LED display) Ext. Hg. Thermometer # 123-8003 -10 to 260°C/ 1°C grad. (EwPE Lab)	Furnace Setting (LED display)	Furnace Reading (LED display)
9:00 am	18 °C	17.8 °C	0 °C	16 °C
9:55 }			125 °C	
10:08 }	19	115	125	125
10:18 }	19	120.5	125	125
10:45 }	19.2	121.1	125	125
11:00 }	19.2	121.1	125	125
11:00 }			171	
11:30 }	19.5	161.5	171	171
11:30 }			175	
12:00 noon	19.6	165.4	175	175
12:00 }			180	
12:30 pm }	20	170	180	181
12:30 }			185	
1:00 }	20	174	185	185

Cond.

- Cond. -
↓

A	B	C	D	E
Date/ Time	Ambient Temp - # 123-8002 -10 to 260°C/ 1°C grad. (EWPE Lab)	Furnace Reading Ext. Hg. Thermocouple # 123-8003 -10 to 260°C/ 1°C grad. (EWPE Lab)	Furnace Setting (LED display)	Furnace Reading (LED display)
1:00	20.6	181.9	193	193
1:30			193	
1:30	21	186.6	200	200
2:00			200	
2:00	21	192	207	207
2:30			207	
2:30	21.3	196.7	215	215
3:00			215	
3:00	21.6	205.2	222	222
3:30			222	
3:30	22	210.4	230	230
4:00			230	
4:00	22	216.00	235	235
4:30			235	
4:30	22	220.2	240	240
5:00			240	
5:00	22.2	224.8	245	245
5:30			245	
5:30	22	229.2	250	250
6:00			250	
6:00	22	233	255	255
6:30			255	
7:00	22	237.8	260	260
7:30			260	
8:00	21	237.8	260	260

This completes the calibration/recalibration of the Blue M Low Temp Oven over the full range of operation. Observation: At temp exceeding ~ 180°C the temp. of the furnace door & walls (ext.) is very uneven indicating uneven insulation - leading to perhaps non-uniform temp. inside the furnace. Hptt 2/5/92

Leaching Vessel Cleaning

Leaching vessels were cleaned using the CNWRA TOP-020 procedure titled "Cleaning of Stainless Steel Leaching Vessels."

The leaching vessels were filled 7/8 the volume with DI water & left in the Blue M oven at 90°C with the cap on - but without the teflon washer or the assembly nut. The pH of the DI water (starting) at 10⁰⁰am at RT was 5.85. pH of the bulk water was measured again at 11¹⁰am & was found to be 6.20 pH units. * The pH was measured using Fisher ACCUMET 950 pH/Ion meter without the automatic temperature compensator - Manual input to the pH meter for RT was 25°C.

The pH of the DI water in the 18 cleaned vessels was measured after 24 hours at 90°C and at RT. using Accumet 950 pH meter. The readings are shown on p. 16.

The leaching vessels used in this test were fabricated by Parr Instruments - Type 316 L unsensitized steel with no welds - matching lids. Capacity of the leaching vessels was 45 ml.

* The pH of the DI water at 90°C was measured to be 6.90 pH units -

Leaching Vessel & Lid #	Date/Time	pH at 90°C [manual input to pH meter at 90°C]	Date/Time	pH at RT [manual input to pH meter at 25°C]
1-001	Feb 11, 1992 Tue. 10:38 am	8.18	Feb 11, 1992 11:31 am	7.59
1-002	10:40	8.10	11:31	7.42
1-003	10:39	8.15	11:28	7.58
1-004	10:41	8.15	11:35	7.48
1-005	10:42	8.17	11:29	7.55
1-006	10:43	8.15	11:25	7.63
1-007	10:44	8.14	11:24	7.57
1-008	10:45	8.03	11:30	7.50
1-009	10:46	8.11	11:34	7.48
1-010	10:36	8.16	11:32	7.51
1-011	10:35	8.10	11:28	7.55
1-012	10:34	8.13	11:25	7.59
1-013	10:33	8.19	11:22	7.62
1-014	10:32	8.17	11:32	7.37
1-015	10:24	8.15	11:25	7.56
1-016	10:30	8.19	11:33	7.36
1-017	10:28	7.90	11:26	7.48
1-018	10:37	8.12	11:27	7.48

The leaching vessels were re-washed 10 times each with DI water & then filled to 7/8 full with DI water & placed in a pre-heated oven at 90°C. These vessels were planned to be checked again for pH change the next day.

{ The DI water used (bottle) for the next run had a pH of 5.81 at 25°C (RT)

The pH of the DI water in the leaching vessels on Feb 12, 1992 at 8:30 am is shown on p. 17.

Leaching Vessel & Lid #	pH at 90°C 8:30 am 2/12/92	pH at 25°C 10:00 am 2/12/92	Remarks
1-001	5.71	5.37	
1-002	5.73	5.40	
1-003	5.88	5.56	
1-004	5.50	5.24	
1-005	5.78	5.64	
1-006	5.82	5.76	
1-007	5.83	5.56	
1-008	5.61	5.63	
1-009	5.60	5.46	
1-010	5.40	5.16	
1-011	5.68	5.58	
1-012	5.61	5.44	
1-013	5.35	5.16	
1-014	5.40	5.25	
1-015	5.61	5.37	
1-016	5.82	5.95	
1-017	5.78	5.42	
1-018	6.10	5.73	
1-019	5.30	6.10	
1-020	6.05	5.58	
1-021	5.79	5.57	
1-022	5.93	5.50	

Each vessel was withdrawn from the oven separately & the pH was checked within a few seconds.

This set of cleaned leaching vessels & cleaned teflon washers were used for the first series of PCT tests using 4 types of borosilicate glasses.

HHH
Feb 12, 1992

Feb 12, 1992.

Series I (AA) PCT test on Borosilicate Glasses Powdered/Crushed

See p. 35
11/15/96

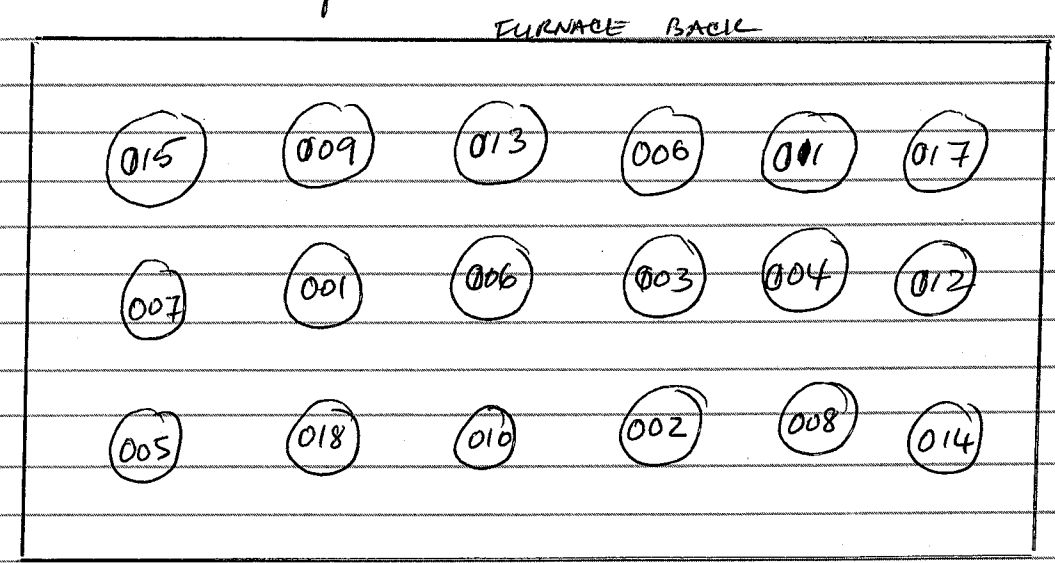
Borosilicate glass samples were crushed using the procedure outlined in CNWRA TOP-019 titled "Preparation of Ground Borosilicate Glass Specimens". Four types of glasses were used in this series, viz. Savannah River Lab supplied 202-G, 202-P, and new ARM glass, and Catholic University supplied SF-10 glass. The CIA glass SF-10 was received in the crushed form mesh -100 + 200 in unwashed condition. At CNWRA this glass was used after washing as outlined in CNWRA procedure TOP-021 "Product Consistency Test (PCT) for Glass Samples".

Both washed & limited amount of unwashed samples plus a few blanks (DI water) only were used. Nominal amount of glass used per leaching vessel in this series was 15g, and the amount of DI water used was 15mL per vessel. The vessel size was 45mL. The specimens and the leaching vessel assemblies were assembled in air atmosphere at ambient temperature.

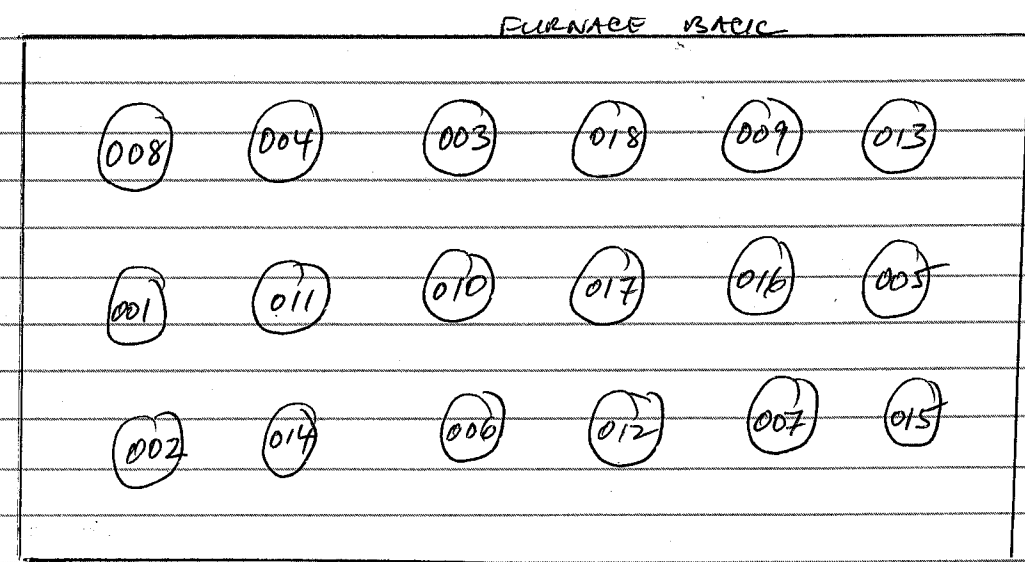
The vessels were hung by teflon coated steel loops. ~~approx~~ The distance from the 1st wire rack in the Blue M oven maintained at 90°C. The distance of the top of the leaching vessels from the top furnace wall (ceiling) was ~ 4.5 inches.

The location of the leaching vessels looking from the top of the furnace on the first day (Feb 12, 1992) at the time of beginning of the test and after 24 hours (after first day weighing (Feb 13, 1992) & then the end of test on Feb 19, 1992 are shown in sketches below. The PCT test was initiated ~ 3:00 pm on Wednesday Feb 12, 1992 & terminated at ~~3:00 pm~~ ^{1:30 pm} on Feb 19, 1992 (Wednesday). The data obtained throughout the 7-day test are shown on p. 20.

Looking
TOP DOWN
2/12/92
3:00 pm
2/13/92
12:30 pm



Looking
TOP DOWN
2/13/92
1:00 pm
2/19/92
1:30 pm



First Series AA. Nominal Glass wt. 1.5g / leaching vcs.

Glass Type	Leaching Vessel ID (45ml)	Washed (W) / Unwashed (UW)	Leaching Vessel (Container) (g) *	Glass Specimen Only (g)	Leaching Vessel (Container) + Glass Specimen (g) *	D.I. Water Vol. (mL) at RT
202-G	AA-1-001	W	213.3560	1.5113	214.5595	15
	AA-1-002	W	214.0025	1.5039	215.5111	15
	AA-1-003	W	220.6682	1.5054	222.1738	15
	AA-1-004	UW	216.5918	0.4357	216.9599	4.4
202-P	AA-1-005	W	220.0614	1.5048	221.5663	15
	AA-1-006	W	220.3579	1.5081	221.8644	15
	AA-1-007	W	215.9392	1.5035	217.4500	15
	AA-1-008	UW	215.3948	0.9262	216.3193	9.3
SF-10	AA-1-009	W	220.6948	1.5106	221.7491	15
	AA-1-010	W	215.3062	1.5063	216.8130	15
	AA-1-011	W	215.2687	1.5001	216.7670	15
NEW ARM	AA-1-012	W	219.7972	1.5090	221.3039	15
	AA-1-013	W	216.5547	1.5011	218.0582	15
	AA-1-014	W	218.8355	1.5055	220.3401	15
	AA-1-015	UW	220.9475	0.8178	221.7685	8.2
DI H ₂ O (ASTM E)	AA-1-016	N/A	219.7252	—	—	15
	AA-1-017	N/A	213.5098	—	—	15
	AA-1-018	N/A	216.4350	—	—	15
DI H ₂ O HNO ₃ CUA	AA-1-019	N/A	—	—	—	—
	AA-1-020	N/A	—	—	—	—
	AA-1-021	N/A	—	—	—	—

Feb 12, 1992
3:00 pm

Feb 13, 1992
3:00 pm

Feb 19, 1991
1:30 pm

Leaching Vessel (Container) + Glass Specimen + Water (g) *	Complete Leaching Vessel Assembly (without the hook) (g) #	Calculated Mass of DI Water added (check)	Complete Leaching Vessel Assembly (without the hook) (g) #	Complete Leaching Vessel Assembly (without the hook) (g) #	pH at 25°C (without automatic temp comp) @	Remarks
230.0591	468.37	1.7031	468.36	468.36	10.234	
230.4185	469.66	1.4160	469.65	469.67	8.863	
237.2853	477.03	1.6171	477.02	477.04	10.266	
221.4438	462.86	0.452	462.84	462.86	9.360	
22						
236.6627	476.98	1.6013	476.97	476.98	10.226	
237.1084	476.70	1.7505	476.69	476.70	10.082	
232.5133	472.74	1.5741	472.74	472.74	9.235	
225.6704	464.18	0.9756	464.18	464.19	10.204	
237.2533	476.12	1.5585	476.12	476.13	10.232	
231.7388	472.21	1.4326	472.22	472.22	10.239	
231.7992	472.49	1.5305	472.48	472.49	10.201	
236.3148	477.74	1.5176	477.74	477.75	10.511	
233.1693	473.68	1.6146	473.68	473.69	10.441	
235.3920	475.79	1.5565	475.79	475.80	10.291	
229.9335	469.81	0.7860	469.80	469.81	10.311	
234.7590	476.23	15.0338	476.23	476.24	5.196	
228.5674	468.06	15.0576	468.05	468.07	5.318	
231.3773	471.34	14.9423	471.34	471.33	5.450	
					5.480	DI H ₂ O used for leaching glass before leaching
					0.632	SRL 0.4M HNO ₃
					1.008	CUA 1wt% HNO ₃

* Readings taken on a Sans Mettler Balance INPELab
Mettler PM4600 Delta Range Top Loader Pan Balance QSLab
@ Accumet 450 (Fisher) pH/tem meter without temp automatic
The pH meter was calibrated with pH 7.0 & pH 10.00 Bu
Fisher Solutions

(Calibrated at factory)
Serial No. 10246/
Temp. compensator Serial # 3340
Fisher pH 7.00 ± 0.01 at 25°C, SB-108-500 Lot No. 913828-24]
pH 10.00 ± 0.02 at 25°C, SB 116-500, Lot No. 913908-24]

Feb 19, 1992

Feb 19, 1992

Preparation of Leachate Samples for Analyses

The ^{first} series leaching vessels were removed from the Blue M low-temp oven. The samples were allowed to cool to RT & then weighed. The weights are shown in table on p. 20-21. ^{Essentially} No weight was observed. Therefore, the sample run was considered acceptable for analyses of the leachate.

JHM
10/13/94

Sample (leaching vessels) were opened one at a time & pH measured (unfiltered leachate) using a 3ml aliquot in a hollow stopper made of polyethylene/polypropylene. The data are recorded in the table on p. 20-21.

The leachate was extracted from ^{1 Fe} leaching vessel using a Becton ~~and~~ ^{JHM 10/13/94} Dickinson & Co. 20cc Syringe [Reorder No. 9661] [Becton Dickinson & Co., Rutherford, New Jersey 07070. and a disposable needle Model ~~TS20~~ ^{JHM 10/13/94} [Part No. TS18 "E" Series Stainless Steel Needle with molded nylon "Pink Color" hub] [Mfg. by Electric Switches, Inc. 2478 Fletcher Ave., Los Angeles, Calif. 90039. Tel: 213-660-1310]. The needle had a standard 1 1/2" length with 0.033" ID & 0.049" OD and blunt tip.

After extracting the leachate from the leaching vessel into the syringe, the needle was removed and a 0.45µm Cellulose Acetate Nalgene (25mm disposable syringe filter) [Cataly No. 190-2045 Sterile] was screwed on. The syringe was then emptied into a clean vial. The filtered leachate was then dilute with dilute HNO₃ solution.

prior to submitting to external laboratory for elemental analyses of interest.

JHM
2/19/92

March Feb 06, 1992 Friday

2nd Series of PCT on Borosilicate Glass Samples

Cleaning of Leaching Vessels

Leaching Vessels # 023 then 040 were cleaned using CNWPA TOP-020 Procedure titled "Cleaning of Stainless Steel Leaching Vessels".

The pH of the bulk DI water Nanopure II used for cleaning the leaching vessels was measured as follows:

DI water pH	Friday	3/6/92	3 ⁰⁰ pm	5.995
" "	Sunday	3/8/92	7 ³⁰ pm	5.956
" "	Monday	3/9/92	9 ³⁰ am	5.802

Cleaned leaching vessels were filled 7/8 volume with DI water on Friday 3/6/92 and placed in pre-heated Blue M oven at 90°C. The leaching vessels were removed on Sunday at 7:01 pm & pH measured as shown in Table on p. 24. The vessels were re-cleaned by rinsing them 5 times each with DI water - filling 7/8 vol. with DI water on Sunday at 7:30 pm. - the pH of the bulk DI water was 5.956. The leaching vessels were then placed in preheated Blue M oven at 90°C. The vessels were removed on Monday 3/9/92 at 9:30 am & pH measured as reported in Table on p. 24.

Cleaning / Cleanliness Measurement of
Leaching vessels for PCT Series II Test.

Leaching Vessel & Lid ID 45 ml 3 size.	pH measured at RT 7 ⁰⁰ pm 3/8/92 Sunday	#	pH measured at RT 9 ³⁰ am 3/9/92 Monday	Remarks
023	4.997		5.719	
024	5.036		5.728	
025	5.079		5.645	
026	5.125		5.773	
027	4.952		5.719	
028	5.015		5.668	
029	4.897		5.659	
030	5.024		5.694	
031	5.152		5.741	
032	5.069		5.562	
033	5.333		5.702	
034	4.930		5.751	
035	5.188		5.788	
036	5.280		5.684	
037	5.284		5.733	
038	5.425		5.777	
039	4.839		5.668	
040	5.237		5.647	

pH measured at RT -
Fisher pH/Ion meter Model # 950 Serial No. 3340
with automatic temperature compensator.

pH of DI water prior to exposure in leaching vessel
in autoclave (on Friday 3/6/92 at 3:00 pm) was 5.789.

* pH of DI water prior to exposure in leaching
vessel in autoclave (on Sunday 3/8/92 at
7:30 pm) was 5.956.

Hills
3/9/92.

Data for PCT Series II Test

3/2/92

• The Series II PCT was run using 5 types of
borosilicate glass samples, viz. 202-P, 202-G,
SF-10, ARM, and NBS glasses.

• The leaching vessels used numbered 001
thru 022 (vessel # 009 and 017, 018 and 019 were
not used). The cleaning was performed
using CNWRA TOP-020 procedure titled
"Cleaning of Stainless Steel Leaching Vessels".
45 ml size vessels with teflon washers
were used for the test.

• The glass specimens were prepared according
to the CNWRA TOP-019 procedure titled
"Preparation of Ground Borosilicate Glass
Specimens". All except SF-10 and ARM
glasses were crushed in CNWRA lab.
SF-10 glass was supplied by CEA in
the crushed (unwashed) form sieve size
-100+200. It was not re-sieved. While
ARM glass was supplied by SRL in
the crushed form (unwashed). Both
SF-10 & ARM as well as the CNWRA crushed
glasses (202-P & 202-G and NBS) were
washed prior to leaching.

• In this Series II run, three nominal
masses of glass sample size were used,
viz. 1.5g, 2.5g, and 4.0g, and the amt.
of DI water used was 15 ml, 25 ml, and
40 ml respectively -

• The pH of ^{bulk} DI water used for leaching
vessel cleaning was 6.180 (March 2, 1992,
9⁰⁰ am).

• The data collected for this series of tests
is reported on p. 26-27.

9:00 am 3/2/92 (C)					
Glass Type (A)	Leaching Vessel pH & Leach after vessel cleaning (B) 3/4/92	pH of @ DI H ₂ O in leaching vessel after cleaning & exp. to 90°C for 1 hr (C)	# Leaching Vessel Empty (g) (D)	* Glas Specimen (g) (E)	DI Water (leachate) mL (F)
DI H ₂ O	AB-1-001	5.907	213.34	-	15 mL
	AB-1-002	6.296	213.99	-	25 mL
	AB-1-003	6.482	220.65	-	40 mL
202-P	AB-1-004	6.025	216.57	1.5032	15
	AB-1-005	6.822	220.05	2.5039	25
	AB-1-006	6.847	220.34	4.0064	40
SF-10	AB-1-007	5.973	215.93	1.5006	15
	AB-1-008	5.980	215.38	2.5023	25
	AB-1-010	5.965	215.29	4.0001	40
ARM	AB-1-011	5.950	215.26	1.5005	15
	AB-1-012	6.016	219.79	2.5009	25
	AB-1-013	6.185	216.55	4.0025	40
202-G	AB-1-014	5.773	218.82	1.5000	15
	AB-1-015	5.938	220.92	2.5012	25
	AB-1-016	6.343	219.71	4.0075	40
NBS- SRM-623	AB-1-020	5.633	218.98	1.5038	15
	AB-1-021	5.928	210.64	2.5038	25
	AB-1-022	5.933	214.33	4.0048	40

@ pH was measured by Fisher ACCUMET Model 450 pH/
buffer solutions.

Weight measurements taken on Mettler PM 4600 Delta

* Glas specimen weighings taken on Mettler AB 240 An
Glas Specimen particle size was -100 + 200 -
condition.

NOTE: (1) The Location of the sample leaching vessels
(2) pH of DI water used for acidifying leachate
3/4/92 Monday 12:00 noon = 6.412

3/2/92 12:00 noon 3/3/92 12:00 noon 3/4/92 12:00 noon 3/4/92 1:00 pm					
Leaching # DI Water Vessel + Glas Specimen (g) (G)	Leaching # Vessel + Glas + DI Water (g) (H)	Leaching # Vessel + Glas + DI H ₂ O + Nut + Hanger (g) (I)	Leaching # Vessel + Glas + DI H ₂ O + Nut + Hanger (g) (J)	Leaching # Vessel + Glas + DI H ₂ O + Nut + Hanger (g) (K)	@ pH of the leachate (L)
-	228.31	468.98	468.98	468.98	5.123
-	238.94	480.50	480.49	480.49	5.411
-	260.65	502.66	502.65	502.64	5.552
218.08	233.10	476.75	476.76	476.75	10.521
222.56	247.57	490.40	490.40	490.40	10.556
224.35	264.35	506.12	506.13	506.11	10.560
217.43	232.38	474.93	474.92	474.93	10.196
217.88	242.89	483.61	483.61	483.61	10.233
219.29	259.23	502.05	502.05	502.04	10.227
216.75	231.75	474.65	474.65	474.65	10.431
222.28	247.27	491.28	491.27	491.28	10.452
220.54	260.54	503.18	503.18	503.18	10.470
220.32	235.28	477.98	477.96	477.97	10.305
223.43	248.39	490.37	490.36	490.37	10.358
223.72	263.72	507.58	507.59	507.59	10.427
220.49	235.47	475.39	475.40	475.40	8.604
213.17	238.15	481.77	481.78	481.77	8.725
218.32	258.27	501.21	501.20	501.19	8.618

100 Meter, Serial No. 3340. Standardized with pH 7.00 & 10.00
Hill 2/11/92

Range Top Loader Pan Balance, Serial No. 102461 (GS Lab)

analytical Balance Serial No. 101237 (GS Lab)

All crushed glass samples were tested in washed
on 3/2/92 and 3/3/92 then 3/4/92 are shown on p-28

Location of Series II (AB)
Specimens (Leaching vessels)

Back of the Furnace

3/2/92
noon.

007 015 016 011 013 002

012 008 004 003 010 001

021 005 014 022 020 006

Back of the Furnace

3/3/92
2:00 pm
→ 10 min!
3/9/92
12:00 noon

015 007 011 014 022 020

003 004 010 016 001 021

012 008 013 002 006 005

Holly

3/9/92

Series III (AC) PCT Leaching Tests on
Borosilicate Glass

3/9/92

The series III (AC) PCT leaching tests on glass samples was ^{almost} identical to series II test, i.e.

- 5 types of borosilicate glass samples - 100+200 mesh size were used.
- all samples were leached in washed condition
- exception: the ARM glass was also crushed at CNUSRA (for series II SRL crushed ARM glass was used)
- sample sizes were 1.5g, 2.5g and 4.0g respectively.
- 45 mL leaching vessels with teflon washers were used.
- leaching time was 7 days at 90°C in a forced air convection furnace (Blue M).
- Upon completion of the experiment the leachate pH was measured in unfiltered condition, and then the leachate was filtered using 25-mm dia 0.45 µm cellulose acetate NALGENE filter prior to acidifying the leachate for elemental analysis using ICP.
- The leached glass samples were recorded to the extent possible for microscopic analysis at a later date — or for further leaching.
- Data obtained from Series III leaching tests are shown in Table on p. 30-31.

pH = 6.032
3/9/92 1:00 pm

3/9/92
12:00 noon

3/10/92
noon

3/16/92
1:00 pm

3/16/92

Glass Type	Leaching Vessel + lid ID	Wt. of Leaching Vessel Empty (g)	Wt. of Glass Sample (g)	Wt. of Leaching Vessel + Glass Sample (g)	DE H ₂ O volume (mL)	Wt. of Leaching Vessel + Glass Sample + Water (g)	Wt. of Leaching Assembly (Glass + H ₂ O + Leaching Vessel + Nut) (g)	Wt. of Leaching Assembly incl. Hook (g)	Wt. of Leaching Assembly incl. Hook (g)	pH of the leachate unfiltered	Temp at which pH measured °C	Remarks
(A)	(B)	(C)	(D)	(E)	(F)	(G)	(H)	(I)	(J)	pH	°C	
ARM	AC-1-023	214.56	4.0005	218.56	40 mL	258.42	501.73	501.73	501.72	10.352	21.0 °C	
	AC-1-024	214.51	2.5012	217.00	25 mL	242.00	487.69	487.69	487.69	10.362	21.4	
	AC-1-025	213.09	1.5013	214.59	15 mL	229.56	470.14	470.14	470.13	10.255	22.0	
SF-10	AC-1-026	213.05	4.0045	217.05	40	257.01	500.38	500.38	500.37	10.145	21.7	
	AC-1-027	213.13	2.5011	215.63	25	240.55	485.25	485.25	485.24	10.131	21.1	
	AC-1-028	214.92	1.5001	216.42	15	231.39	474.01	474.01	474.01	10.066	23	
202-G	AC-1-029	213.26	4.0004	217.23	40	257.16	500.12	500.12	500.12	10.301	22.2	
	AC-1-030	213.78	2.5010	216.28	25	241.24	489.06	489.06	489.06	10.266	22.2	
	AC-1-031	212.75	1.5003	214.26	15	229.23	472.05	472.05	472.04	10.239	21.8	
NBS-SRM-623	AC-1-032	215.75	4.0041	219.77	40	259.69	505.88	505.88	505.88	8.796	21.6	
	AC-1-033	211.74	2.5010	214.24	25	239.21	481.82	481.82	481.81	8.758	22.1	
	AC-1-034	214.26	1.5000	215.76	15	230.74	474.47	474.46	474.46	8.638	21.5	
DI H ₂ O (ASTM I)	AC-1-035	212.92	-	-	40	252.86	496.04	496.04	496.03	6.015	21.4	Something observed at bottom of the vessel @ parallel H ₂ O 3/16/92
	AC-1-036	213.15	-	-	25	238.13	481.89	481.89	481.88	5.554	21.2	
	AC-1-037	213.39	-	-	15	228.33	469.00	469.00	468.99	5.252	21.9	
202-P	AC-1-038	214.15	4.0026	218.16	40	258.17	500.98	500.98	500.98	10.504	21.3	
	AC-1-039	214.86	2.5000	217.36	25	242.33	480.95	480.93	480.93	10.447	21.4	
	AC-1-040	212.49	1.5000	213.99	15	228.99	471.71	471.71	471.70	10.372	21.2	

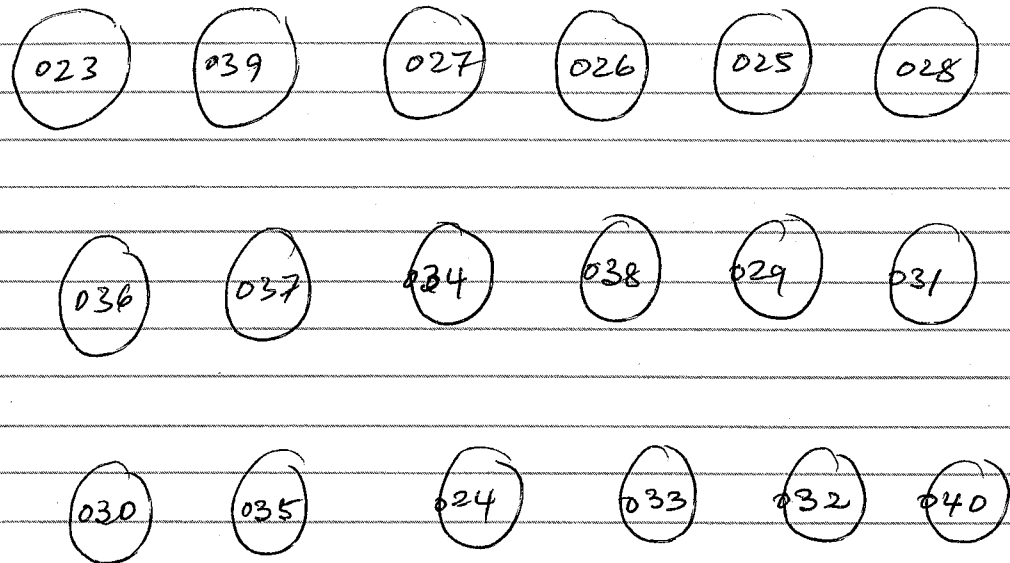
Monday 3/16/92 DE H₂O used for preparing det
S&L 25 mL + rest DE H₂O to 1 liter (Cup & Spout)
* pH Reading with ATC (Automatic Temp Compensation).
NOTE: Leachate extraction method was the same as for series I & II samples

HNO₃ and solution for acidifying leachate (OPMA HNO₃)
20 mL + rest
pH = 5.882
RT ~ 20.7 °C
H₂O 3/16/92

Location of
Series (L. CAE)
samples in
the furnace

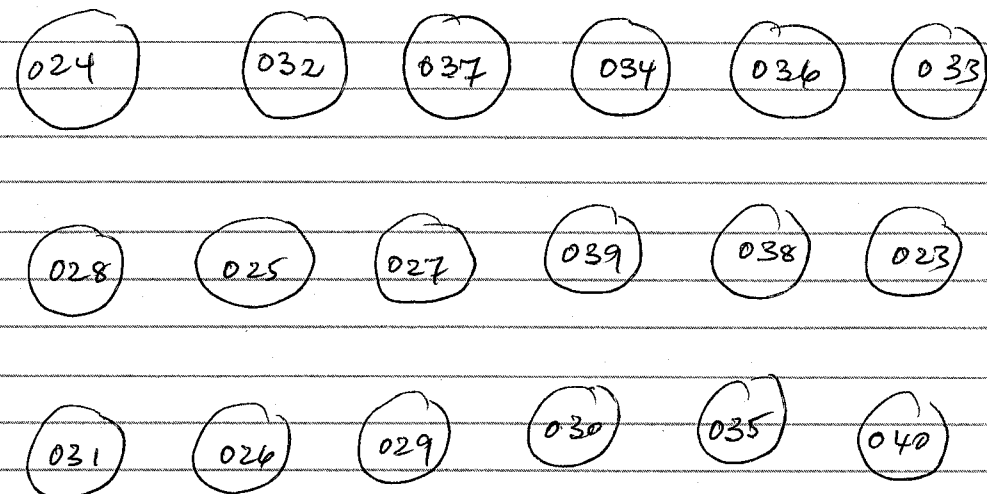
Back of the furnace

First day
3/9/92
Started
1:00pm.
Monday



Back of the furnace

2nd time
7th day
3/10/92 -
3/14/92
1:00pm



HHLV
3/16/92

1992 3/26/92
Tuesday May 26, 1992 10/13/92

Leaching Vessel Cleaning for the new Test Series
36 leaching vessels (304L SS) 45ml Capacity
were cleaned using TOP- procedure.

The leaching vessels were filled with DI water
of ~~6.282~~ pH and covered with matching
lids & left to stay at RT ^{90°C HHLV 5/26/92} over night in the oven.
The pH readings at 3³⁰pm 5/26/92 and
on the following day are shown below.
RT = 21.0 °C at 3³⁰pm

Leaching vessel No.	5/26/92 pH 3 ³⁰ pm	Leaching vessel No.	5/26/92 pH 3 ³⁰ pm
1-001		-029	
-002		-030	
-003		-031	
-004		-032	
-005		-033	
-006		-034	
-007	6.864	-035	
-008		-036	
-010		-037	
-011		-038	
-012	7.026	-039	
-013		-040	
-014			
-015			
-016			
-020			
-021			
-022	6.561		
-023			
-024			
-025			
-026			
-027			
-028			

Vessels to be re-cleaned
some observed some
of some
5/26/92

Pages 1 through 33 of this Scientific Notebook were reviewed for compliance with QAP-001 in response to Corrective Action Request 94-02. Corrections and clarifications were made as appropriate. In some cases, the date of a change will reflect the date of this review rather than the date of the original Scientific Notebook entry.

Randy Folck

SWRI-QA

12/09/94

This project is closed per NRC
funding.
J. H. Marshall
11/1/96

Three borosilicate glass sample types were received from Savannah River Laboratory (SRL) for round-robin tests at CNWRA. This exercise is a part of CNWRA leaching test procedure calibration, and which will prepare the lab for conducting long-term tests more appropriate for performance characterization of vitrified wasteforms. The samples were brought back to CNWRA by H. Marshall after the Aug. 7-8, 1991, meeting with SRL & DOE personnel at SRL, Aiken, SC. The samples are labeled: (1) SRL-202-G, SRL-202-P, and SRL-ARM. Approx. 100g. of each of the first 3 types of glass were obtained. The samples consisted of 10 or more broken pieces of various sizes. A draft document M-910814.01 dated August 14, 1991, has been issued to records mgr. by H. Marshall to record suggested tests to be performed to the samples obtained. The referenced document has been discussed with and a copy provided to the NRC (Rick Weller, Ron Ballard, Chuck Interrante, Mel Silverberg, Tom Ahn, Phil Reed, et al.).

J. H. Marshall September 4, 1991

Added to this
notebook by
N. J. Griffith
11/16/96

From notebook
029 -
needs to be in
040

I have reviewed this scientific notebook and find it in compliance with QAP-001. There is sufficient information regarding procedures used for conducting tests, acquiring and analyzing data so that another qualified individual could repeat the activity.

N. Sridhar 2/24/97

Narasi Sridhar
Manager, Engineered Barrier System and Waste Solidification System