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Scientific Notebook #040

VITREOUS STATE LAB (VSL)

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INITIAL ENTRIES

This lab notebook will be used primary 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 ~~to~~ & 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.

~~Det~~ Appropriate ~~det~~ level of details will be documented in this lab note book for tests conducted under procedures that are not ~~a~~ 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 −140 + 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 # 09-898-25A.

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

Empty weighing paper :	0.4531 g.	Glass weighed on Mettler AE 240 Analytical Balance, Serial No. 101237 in Geo Sciences Lab.
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.	

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 (10 ml) = 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 (10 ml): 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 II 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) and was reweighed — The weight was: 312.00 g.

0.0032 % HBM 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 its 0.2 %. This is a conservative estimate as some loss is attributable to evaporation of lubricant/grease on the leaching 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 leaching vessel assembly set-up, exposing the test assembly to the oven environment, and maintaining the required amount of leachant in the vessel throughout the duration of the test to within the specifications.

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

HBM
Jan 17, 1992

Profiling / Calibration of Blue-M Low Temp Oven

Date / Time	HBM 1/23/92 Furnace Setting (°C) (LED Display)	Furnace Reading (LED Display)	External * Mercury Thermometer [-15 to 101°C / 1/10°C grad. Fisher # 15041B SWPI Calib. Nov 1991 ID # 893-3133 QS Lab D]	Ambient Reading # Ext. Mercury Thermometer 20-110°C / 1°C grad. [SWPI # 125-3001 IWPE Lab]	Ambient Reading # Ext. Mercury Thermom. -10 to 260°C / 1°C grad. SWPI # 123-8003 IWPE Lab.
	A	B	C	E	F
1/23/92 4:30 pm	Set at 00°C	22/23 °C	24.5 °C	23.8 °C	22.5°C
5:00	30 °C	↓	↓	↓	↓
5:30	30	34	35.8	23	22
5:30	35				
6:00	35	40/41	42.4	22.5	21.2
6:00	40				
6:30	40	44	45.8	22	21
6:30	45				
7:00	45	46	48.2	21	20
7:00	51				
7:30	51	52	53.6	20.5	20
7:30	55				
8:00	55	55/56	57.7	20.5	19.5
8:00	60				
8:30	60	60/61	62.4	20.2	19.2
1/24/92 7:30 am	60	60/61	62	18	16.5
7:30	65				
8:30	65	65/66	66.8	18	17
8:30	70				
9:00	70	70/71	71.8	18	17
9:00	76				
9:30	76	76/77	77.5	18	17
9:30	80				
10:00	80	80/81	81.7	18.8	17.2
10:00	85				
10:30	84/85	85	85.9	19.2	18
10:30	90				
11:00	90	90/91	91.4	20	18.8

* External thermometer with 20°C marking inside the furnace left-hand side wall port - graduations 0°C to 20°C within the rubber stopper.

Approx. 12" from the furnace - measured by hanging mercury thermometers

↓ Cont. next page -

— Contd. —
⇓

Date/ Time A	Furnace Setting (LED Display) B	Furnace Reading (LED Display) C	External Mercury Thermometer −1 to 101°C (see p. 9) D	Ambient Reading: Ext. Mercury Thermometer 20–110°C (see p. 9) E	Ambient Reading: Ext. Mercury Thermometer −10 to 260°C (see p. 9) F
11:00	95				
11:30	95	95	95.8	20.2	19
11:30	98				
12:00	98	98/99	98.8	20.3	19
	Ext. thermometer upto 90°C marking in the furnace — with 00–90°C range inside the rubber stopper in the L.H.S. wall of the furnace		Changed External Thermometer to −10 to 260°C shown in Column (F.)		Discontinued
12:00	98				
12:30 pm	98	98/99	99.3	20.5	
12:30	106				
1:00	106	106/107	105.3	21	
1:00	110				
1:05	110	110	109	21.0	
1:05	114				
1:10	114	114	114.3	21	
1:10	120				
1:15	120	120/121	120.2	21.1	
1:15	125				
1:20	125	125	124.9	21.2	
1:20	130				
1:25	130	130/131	129.9	21.8	
1:25	135				
1:30	135	135/136	134.2	21.9	
1:30	139				
1:35	139	139	137.8	21.8	
1:35	146				
1:40	146	146	144	21.8	
1:40	150				
1:45	150	150	148	21.8	
2:00	150	150	148		
2:30 pm	150	150	148	21.8	

* Column Δ p. 9. Foot Note: Hg Thermometer −1 to 101°C with 1/10°C Calibration — Fisher # 150418 — Fisher USA 893-3313 — SCORE Calibration 10 Oct 1991 Next Calib. 10 Oct 1992 ID # 893-3133. 1/24/92

Low-Temp. Blue M Furnace Re-Calibration

Jan 31, 1992.

Date/ Time (A)	External Hg. Thermometer Ambient Temp. # 123-8002 −10 to 260°C/1°C range (IWPE Lab) (B)	Ext. Hg. Therm.* FURNACE Thermometer # 123-8003 −10 to 260°C/1°C range (IWPE Lab) (C)	Furnace Thermometer Setting LED Display (D)	Furnace Thermometer Reading LED Display (E)
Jan 31, 1992 8:00 am	17.5°C	17.5°C	0°C	0°C
8:00	18°C		20°C	
8:30	18°C	23°C	20°C	22°C
8:30			25	
9:00	18	29.5	25	29
9:00			30	
9:30	18	34.6	30	33/34
9:30			35 ?	
10:00	19	42	~~35~~	41
10:00			40	
10:30	19.2	44.2	40	43
10:30			45	
11:00	20	47.2	45	46
11:00			52	
11:30	20.1	54	52	53
11:30			60	
12:00 pm	20.5	61.5	60	60/61
12:00			65	
12:30 pm	20.8	66	65	65/66
12:30			71	
1:00	21	72.3	71.	72
1:00			76	
1:30	21.4	76.8	76	76/77
1:30			80	
2:00	22	81	80	81
2:00			85	
2:30	22	84.8	84/85	85
		⇓ — cont'd —		

* Thermometer inserted upto 10°C in the rubber stopper (10°C, 0°C). The rubber stopper was inserted in the LHS wall of the furnace (in the existing hole).

- Cont'd. -

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			90	
3:00	22.3	90	90	90/91
3:00			95	
3:30	22.5	94	95	95
3:30			100	
4:00	23	98.9	100	100
4:00			106	
4:30	23	104.5	106	106/107
4:30			110	
5:00	23	108.1	110	110/111
5:00			115	
5:30	23	113	115	115/116
5:30			120	
6:00	22.6	117	120	120
6:00			125	
6:30	22	122	125/126	126
6:30			130	
7:00	22	126.4	130	131
7:00			135	
7:30	21.8	130	135	135
7:30			139	
8:00	21.4	133	139	139
8:00			146	
8:30	21.2	139.8	146	146
8:30			150	
9:00	21	143.8	150	150
9:00			155	
9:30	20.8	147.9	155	155
9:30			160	
10:00 pm	20.7	152.0	160	160

Cont'd.

- Cont'd. -

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			165	
10:30 pm	20.6	156.6	165	165
10:30			171	
11:00	20.2	161.5	171	171
11:00			175	
11:30 pm	20.3	165.4	175	175

HAllt 1/31/92

Blue M Low-Temp. Oven Recalibration Feb 5, 1992 Wed.

Date/Time	A	Ambient Temp. # 123-8002 -10 to 260°C/ 1°C grad. (EWPE Lab) B	Furnace Reading (~~LED display~~) Ext Hg Thermometer # 123-8003 -10 to 260°C C	Furnace Setting (LED display) °C D	Furnace Reading (LED display) °C E
Wednesday Feb 5, 1992	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

Cont'd.

— Cont'd. —

A	B	C	D	E
Date/ Time	Ambient Temp. # 123-8002 -10 to 260°C/ 1°C grad. (EWPE Lab)	Furnace Reading Ext. Hg. Thermometer # 123-8003 -10 to 260°C/ 1°C grad (EWPE Lab)	Furnace Setting (LED display)	Furnace Reading (LED display)
1:00			193	
1:30	20.6	181.9	193	193
1:30			200	
2:00	21	186.6	200	200
2:00			207	
2:30	21	192	207	207
2:30			215	
3:00	21.3	196.7	215	215
3:00			222	
3:30	21.6	205.2	222	222
3:30			230	
4:00	22	210.4	230	230
4:00			235	
4:30	22	216.00	235	235
4:30			240	
5:00	22	220.2	240	240
500			245	
5:30	22.2	224.8	245	245
5:30			250	
6:00	22.	229.2	250	250
6:00			255	
6:30	22	233	255	255
7:00			260	
7:30	22	237.8	260	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. HHill 2/5/92

Londeen
~~Fuscaldo~~
Feb 10, 1992

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 in 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 10:00 am at RT was 5.85. pH of the bulk water was measured again at 11:10 am & 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 316L unsensitized steel with no welds — matching lids. Capacity of the leaching vessels was 45 ml.

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* 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 of 90°C]	Date/Time	pH at RT [Manual input to pH meter of 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-washes 10 times each with D.I. 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 (bulk) for the next run ~~is~~ 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 pm~~ ~~2:00 pm~~ 8:30 am ~~are~~ shown on p. 17.

HHM 2/11/92

Leaching Vessel & Lid #	pH at 90°C 8:30 am 2/12/92 HHM 2/12/92	pH at 25°C 10:00 pm 2/12/92 HHM 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 HHM 2/12/92	
1-022	5.93	5.50	

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

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

HHM Feb 11, 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 SE.10 glass. The CNA glass SE-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 15 g. and the amount of DI water used was 15 mL per vessel. The vessel size was 45 mL. The specimens ~~wer~~ & the leaching vessel assemblies were assembled in air atmosphere at ambient temperature.

The vessels were hung by teflon coated steel loops. ~~approx~~ ~~The dist~~ 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 ≈ 45.5 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) & from 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~~ 1:30 pm on Feb 19, 1992 (Wednesday). The data obtained throughout the 7-day test are shown on p. 20.

HRell 2/19/92

LOOKING TOP DOWN

2/12/92 3:00 pm.

2/13/92 12:30 pm

FURNACE BACK

015	009	013	006	011	017
007	001	006	003	004	012
005	018	010	002	008	014

LOOKING TOP DOWN

2/13/92 1:00 pm

→ 2/19/92 1:30 pm

FURNACE BACK

008	004	003	018	009	013
001	011	010	017	016	005
002	014	006	012	007	015

First Series AA. Nominal Glass Wt. 1.5g / leaching vessel

Glass Type	Leaching Vessel ID (45 ml)	Washed (W)/ Unwashed (UW)	Leaching* Vessel (Container) (mass) (g)	Glass Specimen only (g)	Leaching* Vessel (Container) + Glass Specimen (g)	D.I. Water Vol. (mL) at RT	Feb 12, 1992 3:00 pm: Leaching* Vessel (Container) + Glass Specimen + Water (g)	Feb 12, 1992 3:00 pm: Complete# Leaching Vessel Assembly (without the hook) (g)	Calculated Mass of DI water added. (check)	Feb 13, 1992 3:00 pm: Complete# Leaching Vessel Assembly (without the hook) (g)	Feb 19, 1991 1:30 pm: Complete# Leaching Vessel Assembly (without the hook) (g)	pH@ at 25°C (without automatic temp comp)	Remarks.
202-G	AA-1-001	W	213.3560	1.5113	214.5595	15	230.0591	468.37	1.7031	468.36	468.36	10.234	
	AA-1-002	W	214.0025	1.5039	215.5111	15	230.4185	469.66	1.4160	469.65	469.67	8.863	
	AA-1-003	W	220.6682	1.5054	222.1738	15	237.2853	477.03	1.6171	477.02	477.04	10.266	
	AA-1-004	UW	216.5918	0.4357	216.9599	4.4	221.4438	462.86	0.452	462.84	462.86	9.360	
202-P	AA-1-005	W	220.0614	1.5048	221.5663	15	236.6627	476.98	1.6013	476.97	476.98	10.226	
	AA-1-006	W	220.3579	1.5081	221.8644	15	237.1084	476.70	1.7505	476.69	476.70	10.082	
	AA-1-007	W	215.9392	1.5035	217.4500	15	232.5133	472.74	1.5741	472.74	472.74	9.235	
	AA-1-008	UW	215.3948	0.9262	216.3193	9.3	225.6704	464.18	0.9756	464.18	464.19	10.204	
SF-10	AA-1-009	W	220.6948	1.5106	221.7491	15	237.2533	476.12	1.5585	476.12	476.13	10.232	
	AA-1-010	W	215.3062	1.5063	216.8130	15	231.7388	472.21	1.4326	472.22	472.22	10.239	
	AA-1-011	W	215.2687	1.5001	216.7670	15	231.7992	472.49	1.5305	472.48	472.49	10.201	
New ARM	AA-1-012	W	219.7972	1.5090	221.3039	15	236.3148	477.74	1.5176	477.74	477.75	10.511	
	AA-1-013	W	216.5547	1.5011	218.0582	15	233.1693	473.68	1.6146	473.68	473.69	10.441	
	AA-1-014	W	218.8355	1.5055	220.3401	15	235.3920	475.79	1.5565	475.79	475.80	10.291	
	AA-1-015	UW	220.9475	0.8178	221.7685	8.2	229.9335	469.81	0.7860	469.80	~~475~~ 469.81	10.311	
DI H_2O (ASTM I)	AA-1-016	N/A	219.7252	—		15	234.7590	476.23	15.0338	476.23	476.24	5.196	
	AA-1-017	N/A	213.5098	—		15	228.5674	468.06	15.0576	468.05	468.07	5.318	
	AA-1-018	N/A	216.4350	—		15	231.3773	471.34	~~15~~ 14.9423	471.34	471.33	5.450	
DI H_2O	AA-1-019	N/A										5.480	DI H_2O used for leaching glass before leaching.
HNO_3 SRL 0.4M acid	AA-1-020	N/A										0.632	SRL 0.4M HNO_3
HNO_3 CUA 1 vol.% acid	AA-1-021	N/A										1.008	CUA 1 vol% HNO_3

* Readings taken on a Sartorius Mettler Balance IWPE Lab (Calibrated at factory)
\# Mettler PM 4600 Delta Range Top Loader Pan Balance QS Lab Serial No. 10246)
@ Accumet 950 (Fisher) pH/Ion meter (without temp. compensation) Serial # 3340
The pH meter was calibrated with pH 7.0 & pH 10.00 Buffer Solutions. [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. 913808-24]

HKM 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.

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

The leachate was extracted from the leaching vessel using a Becton ~~Dict~~ Dickinson & Co. 20cc Syringe [Reorder No. 9661] [Becton Dickinson & Co, Rutherford, New Jersey 07070. and a disposable needle Model ~~FS20~~ [Part No. TS18 "E" Series Stainless Steel Needle with molded nylon "Pink Color" hub] [Mfg. by Electric Switches, Inc. 2478 Fletcher Drive, 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 0.45 μm Cellulose Acetate Nalgene (25 mm disposable syringe filter) [~~model~~ Catalog No. 190-2045 Sterile] was screwed on. The syringe was then emptied into a clean vial. The filtered leachate was then diluted with dilute HNO_3 solution

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

2/19/92

March ~~Feb~~ 06, 1992 Friday

2nd Series of PCT on Borosilicate Glass Samples

Cleaning of Leaching Vessels

Leaching Vessels # 023 thru 040 were cleaned using CNWRA 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 7:00 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 pre-heated 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 size.	pH measured at RT # 7:00 pm 3/8/97 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 5.799.

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

HHM 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 CGA 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 1.5 g, 2.5 g, and 4.0 g., 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, 9:00 am).

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

Glass Type (A)	Leaching Vessel & Lid ID (B)	pH of @ DI H_2O in leaching vessel after cleaning & exp. to 90°C (C)	Leaching Vessel Empty # (g) (D)	Glass Specimen * (g) (E)	DI water (leachate) mL (F)	Leaching # Vessel + Glass Specimen (g) (G)	Leaching # Vessel + Glass + DI water (g) (H)	Leaching # Vessel + Glass + DI H_2O + Nut + Hanger (g) (I)	Leaching # Vessel + Glass + DI H_2O + Nut + Hanger (g) (J)	Leaching # Vessel + Glass + DI H_2O + Nut + Hanger (g) (K)	pH of @ the leachate (L)	Remarks (M)
		9:00 am 3/2/92				3/2/92 12:00 noon		3/3/92 12:00 noon		3/9/92 12:00 noon	3/9/92 1:00 pm	
DI H_2O	AB-1-001	5.907	213.34	-	15 mL	-	228.31	468.98	468.98	468.98	5.123	
	AB-1-002	6.296	213.99	-	25 mL	-	238.94	480.50	480.49	480.49	5.411	
	AB-1-003	6.482	220.65	-	40 mL	-	260.65	502.66	502.65	502.64	5.552	
202-P	AB-1-004	6.025	216.57	1.5032	15	218.08	233.10	476.75	476.76	476.75	10.521	
	AB-1-005	6.822	220.05	2.5039	25	222.56	247.57	490.40	490.40	490.40	10.556	
	AB-1-006	6.847	220.34	4.0064	40	224.35	264.35	506.12	506.13	506.11	10.560	
SF-10	AB-1-007	5.973	215.93	1.5006	15	217.43	232.38	474.93	474.92	474.93	10.196	
	AB-1-008	5.980	215.38	2.5023	25	217.88	242.89	483.61	483.61	483.61	10.233	
	AB-1-010	5.965	215.29	4.0001	40	219.29	259.23	502.05	502.05	502.04	10.227	
ARM	AB-1-011	5.950	215.26	1.5005	15	216.75	231.75	474.65	474.65	474.65	10.431	
	AB-1-012	6.016	219.79	2.5009	25	222.28	247.27	491.28	491.27	491.28	10.452	
	AB-1-013	6.185	216.55	4.0025	40	220.54	260.54	503.18	503.18	503.18	10.470	
202-G	AB-1-014	5.773	218.82	1.5000	15	220.32	235.28	477.98	477.96	477.97	10.305	
	AB-1-015	5.938	220.92	2.5012	25	223.43	248.39	490.37	490.36	490.37	10.358	
	AB-1-016	6.343	219.71	4.0075	40	223.72	263.72	507.58	507.59	507.59	10.427	
NBS-SRM-623	AB-1-020	5.633	218.98	1.5038	15	220.49	235.47	475.39	475.40	475.40	8.604	
	AB-1-021	5.928	210.64	2.5038	25	213.17	238.15	481.77	481.78	481.77	8.725	
	AB-1-022	5.933	214.33	4.0048	40	218.32	258.27	501.21	501.20	501.19	8.618	

@ pH was measured by Fisher ACCUMET Model 950 pH/ion Meter, Serial No. 3340. ~~Calibrated~~ Standardized with pH 7.00 & 10.00 buffer solutions. HHH 3/19/92

Weight measurements taken on Mettler PM 4600 Delta Range Top Loader Pan Balance, Serial No. 102461 (GS Lab)

* Glass specimen weighing taken on Mettler AE 240 Analytical Balance Serial No. 101237 (GS Lab)

Glass Specimen particle size was -100 + 200 — All crushed glass samples were tested in washed condition.

Note: (1) The Location of the sample leaching vessels on 3/2/92 and 3/3/92 ~~then~~ 3/9/92 are shown on p. 28

(2) pH of DI water used for acidifying leachate 3/9/92 Monday 12 noon = 6.412

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

3/2/92 noon.

Back of the Furnace

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 → turn! 3/9/92 12:00 noon

015	007	011	014	022	020
003	004	010	016	001	021
012	008	013	002	006	005

HRM 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 CNWRA (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 analyses using ICP.
  - The leached glass samples were recovered to the extent possible for microscopic analyses 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

Glass Type (A)	Leaching Vessel + Lid ID (B)	Wt. of Leaching Vessel Empty (g) (C)	Wt. of Glass Sample (g) (D)	Wt. of Leaching Vessel + Glass Sample (g) (E)	DI H_2O Volume (mL) (F)	Wt. of Leaching Vessel + Glass Sample + Water (g) (G)	Wt. of Leaching Assembly (Glass + H_2O + Leaching vessel + Nut) mid-Hook (g) (H)	Wt. of Leaching Assembly mid Hook (g) (I)	Wt. of Leaching Assembly mid Hook (g) (J)	pH* of the leachate unfiltered pH	Temp at which pH measured °C	Remarks.
						3/9/92 12:00 noon	3/10/92 noon	3/16/92 1:00 pm	3/16/92			
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_2O (ASTM I)	AC-1-035	212.92	-	-	40	252.86	496.04	496.04	496.03	6.015	21.4	Something observed at the bottom of the vessel (barnacle) HPell 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 DI H_2O used for preparing dist. HNO_3 acid solution for acidifying leachate (COPY MA HNO_3)

HPell 3/16/92 ~~S&L 2 Sand + rest DI H_2O to water~~ ~~CUX & SORT~~ ~~Zone + rest~~

pH = 5.882

RT ~ 20.7°C.

HPell 3/16/92

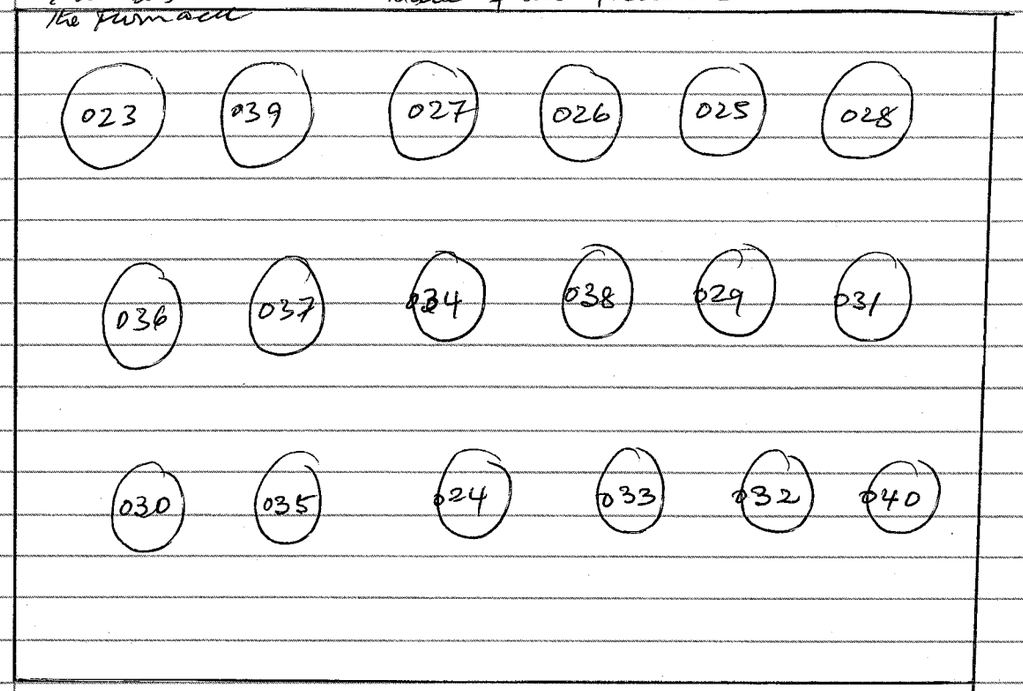
* pH Reading with ATC (automatic temp compensation).

NOTE: leachate extraction method was the same as for series I & II samples

Location of Series III (AC) samples in the Furnace

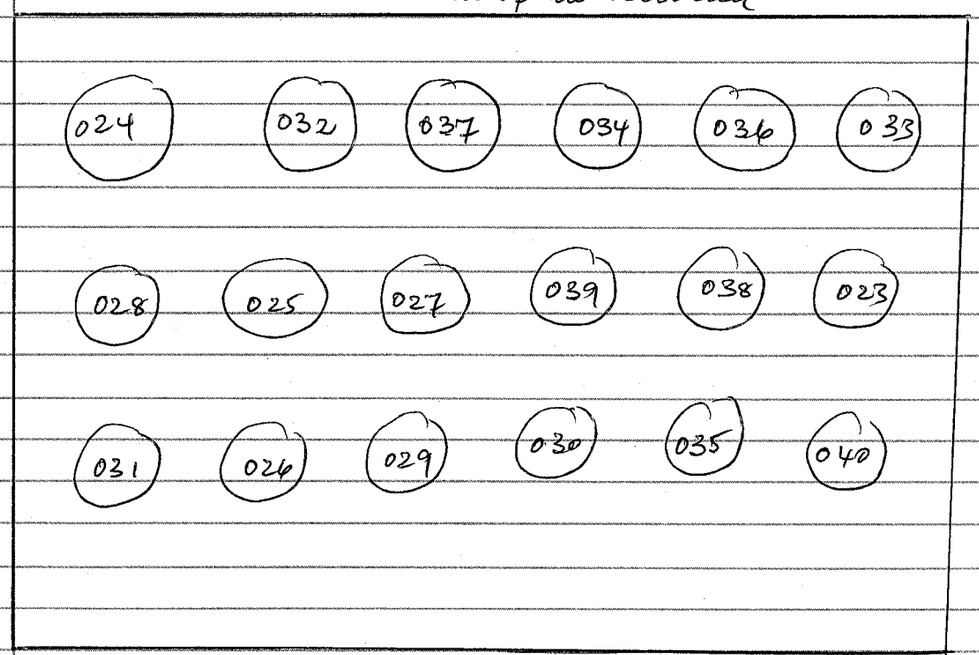
Back of the Furnace

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



Back of the Furnace

2nd thru' 7th day 3/10/92 – 3/16/92 1:00 pm.



HHW 3/16/92

Tuesday May 26, ~~1990~~ 1992 HHW 10/13/94

Leaching Vessel Cleaning for the next Test Series

36 leaching vessels (304L Ss) 45 ml capacity were cleaned using TOP- procedure.

The leaching vessels were filled with DI water of ~~6.102~~ 6.282 HHW 5/26/92 pH and covered with matching lids & left to stay at ~~RT~~ 90°C HHW 5/26/92 over night in the oven. The pH readings at 3:30 pm 5/26/90 and on the following day are shown below.

RT = 21.0 °C at 3:30 pm

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			-036		
-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 visible contamination observed at the bottom of some leaching vessels. HHW 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 Folk
SwRI-QA
12/09/94

This project is closed per NRC funding.
Hersh H. Manaktala
11/1/96

Three borosilicate glass samples 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. Manaktala after the Aug. 7-8, 1991, meeting with SRL & DOE personnel at SRL, Aiken, S.C. The samples are labled: (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 ~~to~~ 20 or more broken pieces of various sizes. A draft document M-910814.01 dated August 14, 1991, has been issued ~~to record~~ ~~sug~~ by H. Manaktala 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, Tae Ahn, Phil Reed, et al).
Hersh H. Manaktala September 4, 1991

Added to this notebook by N. Sridhar 11/15/96.

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