Appendix D2

ESEM and SEM/EDS Data for Test #2, Day-30 Fiberglass in High- and Low-Flow Zones

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The debris accumulated on fiberglass in the ICET tests is of great interest because it may contribute to additional head loss during recirculation of the coolant following a LOCA. To evaluate the potential for debris accumulation, fiberglass samples submerged in high- and low-flow zones in the tank were examined by ESEM and SEM/EDS.

The fiberglass samples examined in this appendix were extracted on the date Test #2 was terminated (March 7, 2005). Both exterior and interior locations on the fiberglass samples were examined. Microprobe SEM was used to examine the fiberglass samples after they were air dried at room temperature and then coated with gold/palladium. In addition to microprobe SEM, ESEM was used to analyze the wet fiberglass samples without any coating. ESEM was performed under a low-vacuum condition (80 Pa) to minimize any modification of the fiberglass that could occur through the drying process. Microprobe SEM/EDS and ESEM results of Test #2, Day-30 high- and low-flow fiberglass samples were obtained on March 7 and March 9, 2005, respectively. Accompanying EDS results provide a semiquantitative elemental analysis of the debris deposited on the fiberglass.

Microprobe laboratory session from March 7, 2005

T2D30 Samples-NRC



Conditions: 15-kV, 1-nA beam current, Aperture = 2

Sample: Low-Flow Exterior

Image:	T2D30_LoFlo014	43 ×	Overview SE image	Figure D2-1
	T2D30_LoFlo015	500 ×	SE near center	Figure D2-2
	T2D30_LoFlo016	$1500 \times$	SE image on same area	Figure D2-3

Sample: High-Flow Exterior

Image:	T2D30_HiFlo017	40 ×	Overview SE image	Figure D2-4
	T2D30_HiFlo018	230 ×	Center of image 017	Figure D2-5
	T2D30_HiFlo019	$1000 \times$	Lower right of image 018	Figure D2-6
	T2D30_HiFlo020	700 ×	New area	Figure D2-7
EDS:	T2D30EDS13		Film on high-flow fiberglass	Figure D2-8

Sample: High-Flow Interior

Image:	T2D30_HiFlo021	$40 \times$	SE image overview	Figure D2-9
	T2D30_HiFlo022	$180 \times$	SE image near center of 021	Figure D2-10
			image	
	T2D30_HiFlo023	$1000 \times$	Same area	Figure D2-11

EDS:	T2D30EDS14		Film on fiberglass Hi Flow interior	Figure D2-12
Image:	T2D30_HiFlo024	500 ×	SE on different area of sample	Figure D2-13

Sample: Low-Flow Interior

Image:	T2D30_LoFlo025	350 ×	On possible bacteria or Ca Phosphate	Figure D2-14
EDS:	T2D30EDS15		Globular cluster from image 025	Figure D2-15
Image:	T2D30_LoFlo026	$800 \times$	Different area	Figure D2-16

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ESEM Laboratory session from March 8, 2005

T2D30 Low-Vacuum SEM



Conditions: 20-kV, 12-mm Working Distance, 80 Pa pressure

Sample: High-Flow Interior

Image:	T2D30H11	150 ×	Overview	Figure D2-17
	T2D30HI2	1000 ×		Figure D2-18

Sample: High-Flow Exterior

Image:	T2D30HX3	$150 \times$	Overview	Figure D2-19
	T2D30HX4	$1000 \times$		Figure D2-20

Sample: Low-Flow Exterior

Image: T2D30LX5 150 ×

Figure D2-21

T2D30LX6 $1000 \times$ On debris

Sample: Low-Flow Interior

Image: T2D30LI7 150 × T2D30LI8 1000 × Figure D2-23 Figure D2-24



Figure D2-1. SEM image for a Test #2, Day-30 low-flow exterior fiberglass sample. (T2D30_LoFlo014)



Figure D2-2. SEM image of a higher magnification for a Test #2, Day-30 low-flow exterior fiberglass sample. (T2D30_LoFlo015)



Figure D2-3. SEM image of a higher magnification for a Test #2, Day-30 low-flow exterior fiberglass sample. (T2D30_LoFlo016)



Figure D2-4. SEM image for a Test #2, Day-30 high-flow exterior fiberglass sample. (T2D30_HiFlo017)



Figure D2-5. SEM image for a Test #2, Day-30 high-flow exterior fiberglass sample. (T2D30_HiFlo018)



Figure D2-6. SEM image of a higher magnification for a Test #2, Day-30 high-flow exterior fiberglass sample. (T2D30_HiFlo019)



Figure D2-7. SEM image of a higher magnification for a Test #2, Day-30 high-flow exterior fiberglass sample. (T2D30_HiFlo020)



Figure D2-8. EDS counting spectrum for the film deposits or growth on fiberglass, as shown in Figure D2-7. (T2D30EDS13~Film at High Flow Exterior)

The results from the chemical composition analysis for T2D30EDS13 are given in Table D2-1.

Table D2-1. The Chemical Composition for T2D30EDS13 (Figure D2-8)

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Group Sample Comment Condition	: NRC : T2D30 : film : Full Live Acc. Stage Acq.	D ID# : 1 on HiFlow Scale : Time : Volt : Point : Date :	13 20KeV 60.00 20.01 X=48.3 Mon Ma	rglass (10eV/c) 00 sec KV 137 Y=50 ar 7 1 ⁻	n,2Kch) Apert Probe 5.660 Z 7:20:30	ture # e Curre =10.558 2005	: 1 nt : 3.1	247E-10 A
Element C K	Mode Normal	ROI(1 0.09-	KeV) 0.46	K-ratio	b(%) +, 00 0.0	/- Ne	et/Back	ground / 15
OK	Normal	0.25-	0.77	22.758	34 0.0	0029	1420	/ 8
Nak	Normal	1 50-	2.05	1 249		0008	576	/ 4
D K ST V	Normal	1 75-	2.05	1.35	16 0.1	0016	300	/ 34
CI K	Normal	2.34-	3.06	0.470	0.0	0004	143	/ 5
Ca K	Normal	3.39-	4.30	0.423	L2 0.0	0006	103	/ 3
			Ch	i_square	e = 2.	5421		
Element Ma	ass%	Atomic%	ZAF	Z	A	F		
С	0.000	0.0000 6	6.0057	1.0404	5.7730	0.9999		
0 6	58.212	77.9670 (0.7076	0.9928	0.7128	1.0000		
Na :	15.750	12.5283	1.5604	1.0448	1.4934	1.0001		
Si	7.284	4.7427	1.3779	0.9953	1.3873	0.9980		
C1	4./50	1 1225	1 1005	1.1/82	1 0520	0.9993		
Ca	1.806	0.8239	1.0120	1.0156	0.9965	1.0000		
Total 10	00.000 1	00.0000			, 10 (10 (10 (10 (10 (10 (10 (10 (10 (10			
Normalizat	tion fac	tor = 4	.2357					



Figure D2-9. SEM image for a Test #2, Day-30 high-flow interior fiberglass sample. (T2D30_HiFlo021)



Figure D2-10. SEM image for a Test #2, Day-30 high-flow interior fiberglass sample. (T2D30_HiFlo022)



Figure D2-11. SEM image at 1000× magnification for a Test #2, Day-30 high-flow interior fiberglass sample. (T2D30_HiFlo023)



Figure D2-12. EDS counting spectrum for the film deposits or growth on fiberglass, as shown in Figure D2-11. (T2D30EDS14~Film on HiFlow Interior)

The results from the chemical composition analysis for T2D30EDS14 are given in Table D2-2.

Table D2-2. The Chemical Composition for T2D30EDS~14 (Figure D2-12)

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Group Sample Comment Condition	: NRC : T2D30 : film on : Full Sc Live Ti Acc. Vo Stage P Acq. Da	ID# : 14 hi flow fib ale : 20KeV me : 60.0 lt : 20.0 oint : X=43. te : Mon M	erglass int (10eV/ch,2K 00 sec A KV P 227 Y=69.35 Jar 7 17:34	erior ch) perture # robe Curre 7 Z=10.558 :53 2005	: 1 ent : 3.155E-10) A
Element	Mode	ROI (KeV)	K-ratio(%)	+/- 1	Jet/Background	
СК	Normal	0.09- 0.46	0.0000	0.0000	0 /	18
O K	Normal	0.25- 0.77	23.9534	0.0029	1452 /	6
Na K	Normal	0.81- 1.27	0.4162	0.0004	98 /	4
Si K	Normal	1.50- 2.05	0.7810	0.0002	317 /	14
PK	Normal	1.75- 2.38	0.2529	0.0013	55 /	26
Ca K	Normal	3.39- 4.30	0.4296	0.0005	102 /	5
		Ch	i_square =	3.6593	i ne ne per le le competencia de la com	
Element Ma	ass% At	omic% ZAF	Z	A F		
С	0.000 0	.0000 3.8510	1.0451 3.6	851 0.9999)	
0 8	35.150 90	.8939 0.5770	0.9974 0.5	785 1.0000)	
Na	4.620 3	.4320 1.8017	1.0498 1.7	160 1.0002	1	
Si	6.368 3	.8722 1.3235	1.0003 1.3	239 0.9994		
P	1.246 0	.6870 0.7999	1.1842 0.6	756 0.9999	,	
Ca	2.616 1	.1148 0.9886	1.0213 0.9	680 1.0000	ſ	
Total 10 Normalizat	0.000 100 100	.0000 or = 6.1604	nen en nechen en en en en her her her			



Figure D2-13. SEM image of a higher magnification for a Test #2, Day-30 high-flow interior fiberglass sample. (T2D30_HiFlo024)



Figure D2-14. SEM image for a Test #2, Day-30 low-flow interior fiberglass sample. (T2D30_LoFlo025)



Figure D2-15. EDS counting spectrum for the particulate deposits or growth on fiberglass, as shown in Figure D2-14. (T2D30EDS15-Particle on Lowflow Interior)



Figure D2-16. SEM image of a higher magnification for a Test #2, Day-30 low-flow interior fiberglass sample. (T2D30_LoFlo026)



Figure D2-17. ESEM image for a Test #2, Day-30 high-flow interior fiberglass sample. It seems the interior fiberglass is cleaner than exterior fiberglass. (T2D30H11)



Figure D2-18. ESEM image of a higher magnification for a Test #2, Day-30 high-flow interior fiberglass sample. (T2D30HI2)



Figure D2-19. ESEM image for a Test #2, Day-30 high-flow exterior fiberglass sample. (T2D30HX3)



Figure D2-20. ESEM image of a higher magnification for a Test #2, Day-30 high-flow exterior fiberglass sample. (T2D30HX4)



Figure D2-21. ESEM image for a Test #2, Day-30 low-flow exterior fiberglass sample. (T2D30LX5)



Figure D2-22. ESEM image of a higher magnification for a Test #2, Day-30 low-flow exterior fiberglass sample. (T2D30LX6)



Figure D2-23. ESEM image for a Test #2, Day-30 low-flow interior fiberglass sample. (T2D30L17)



Figure D2-24. ESEM image of a higher magnification for a Test #2, Day-30 low-flow interior fiberglass sample. (T2D30L18)

Appendix D3

ESEM and SEM/EDS Data for Test #2, Day-30 Drain Collar Fiberglass

Figures

Figure D3-1.	SEM image for a Test #2, Day-30 fiberglass sample on the drain collar next
	to the drain screen. (T2D30_DrainScreen029)
Figure D3-2.	Backscattered SEM image for a Test #2, Day-30 fiberglass sample on the
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Figure D3-4.	SEM image at 1000× magnification for a Test #2, Day-30 fiberglass sample
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Figure D3-5.	EDS counting spectrum for the center of the image shown in Figure D3-4.
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	The film is rich in silicon, oxygen, aluminum, sodium, and calcium.
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	(T2D30_DrainOutside033)D3-12
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	collar showing deposits or growth on fiberglass.
	<u>(T2D30_DrainOutside034)</u>
Figure D3-9.	Backscattered SEM image for a Test #2, Day-30 exterior fiberglass sample
	on the drain collar. (T2D30_DrainOutside035)
Figure D3-10.	SEM image at a higher magnification for a Test #2, Day-30 exterior
	fiberglass sample on the drain collar showing deposits or growth on
	fiberglass. (T2D30_DrainOutside 036)

Figure D3-11.	EDS counting spectrum for deposits or growth on fiberglass. The deposits	
	contain a significant amount of carbon. (T2D30EDS18~Drain Outside	
	Particles High C)	D3-14
Figure D3-12.	EDS counting spectrum for a deposit or growth on fiberglass. The deposit	
	contains a significant amount of carbon. (T2D30EDS19)I	D3-14
Figure D3-13.	SEM image for a Test #2, Day-30 interior fiberglass sample on the drain	
	collar. Image shows deposits or growth on fiberglass.	
	(T2D30_DrainInt037)	D3-16
Figure D3-14.	SEM image for a Test #2, Day-30 interior fiberglass sample on the drain	
	collar. Image shows deposits or growth on fiberglass.	
	(T2D30_DrainInt038)	D3-16
Figure D3-15.	SEM image of a higher magnification for a Test #2, Day-30 interior	
	fiberglass sample on the drain collar. The image shows deposits or growth or	ı
	fiberglass. (T2D30_Drain Int039) I	03-17
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	on the drain collar. Deposits appear to have a similar or lower atomic number	r
	as compared with fiberglass. (T2D30_DrainInt040)I	03-17
Figure D3-17.	EDS counting spectrum for the deposits or growth on fiberglass. The	
	deposits are rich in silicon, carbon, oxygen, and calcium. The deposits are	
	very thin, and thus it was difficult to obtain high-quality EDS spectra.	
	(T2D30EDS20)	03-18
Figure D3-18.	ESEM image for a Test #2, Day-30 exterior fiberglass sample on the drain	
	collar. Image shows particulate deposits or growth on fiberglass.	
	(T2D30DO1)	03-20
Figure D3-19.	ESEM image of a higher magnification for a Test #2, Day-30 exterior	
	fiberglass sample on the drain collar. The image shows particulate deposits	
	or growth on fiberglass. (T2D30DO2)	03-20
Figure D3-20.	ESEM image for a Test #2, Day-30 exterior fiberglass sample on the drain	
	collar. Image shows particulate deposits or growth on fiberglass.	
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Figure D3-21.	ESEM image for a Test #2, Day-30 fiberglass sample on the drain collar next	t
	to the drain screen. (T2D30DS4)	03-21
Figure D3-22.	ESEM image for a Test #2, Day-30 fiberglass sample on the drain collar next	t
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Figure D3-23.	ESEM image of a higher magnification for a Test #2, Day-30 fiberglass
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Figure D3-24.	ESEM image for a Test #2, Day-30 interior fiberglass sample on the drain
	collar. Image suggests that interior fiberglass was relatively clean as
	compared with exterior. (T2D30DI7)
Figure D3-25.	ESEM image for a Test #2, Day-30 interior fiberglass sample on the drain
	collar. (T2D30DI8)
Figure D3-26.	ESEM image of a higher magnification for a Test #2, Day-30 interior
	fiberglass sample on the drain collar. The image shows deposits or growth on
	the interior fiberglass, although the quantity was less than for exterior
	samples. (T2D30DI9)

Tables

Table D3-1.	The Chemical Composition for T2D30EDS16 (Figure D3-5)
Table D3-2.	The Chemical Composition for T2D30EDS17 (Figure D3-6)
Table D3-3.	The Chemical Composition for T2D30EDS19 (Figure D3-12) D3-15
Table D3-4.	The Chemical Composition for T2D30EDS20 (Figure D3-17) D3-19

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Chemical deposits accumulated on fiberglass during ICET testing are of great interest because they may cause additional head loss during recirculation of the coolant following a LOCA. The drain collar placed in the test tank represents an area of higher flow velocity, where water continually flows through any accumulated debris. This condition of continual water flow is similar to that found on the face of a recirculation sump screen, so it is important to examine the fiberglass samples recovered from the drain collar via ESEM and SEM/EDS and compare the results with similar examinations of fiberglass from alternative flow regimes.

Fiberglass samples from the drain collar were extracted on the date that Test #2 was shut down (March 7, 2005). Samples located at the outside exterior (away from the drain screen), the inside exterior (next to the drain screen), and the interior of the collar were examined. Microprobe SEM was used to examine the fiberglass samples after they were dried in air at room temperature and coated with carbon. In addition to microprobe SEM, ESEM was used to analyze the wet fiberglass samples. ESEM was performed without any required coating under a low-vacuum condition (80 Pa) to minimize any modification of the sample that might occur through the drying process. Microprobe SEM/EDS and ESEM results of the Test #2, Day-30 drain collar fiberglass samples were obtained on March 9, 2005.

Microprobe laboratory session from March 9, 2005 T2D30 samples from fiberglass drain collar



Conditions: 15-kV, 1-nA beam current, Aperture=2 Note: Bold spots on sketch denote carbon glue used to secure the samples.

Sample: Drain Screenside

Image:	T2D30_DrainScreen029	150 ×	SE	Figure D3-1
	T2D30_DrainScreen030	150 ×	BSE	Figure D3-2
	T2D30_DrainScreen031	90 ×	SE	Figure D3-3
	T2D30_DrainScreen032	1000×	SE	Figure D3-4
EDS:	T2D30EDS16		Center of image 032	Figure D3-5
	T2D30EDS17		Film on fiberglass	Figure D3-6

Sample: Drain Outside

Image:	T2D30_DrainOutside033	90 ×	SE	Figure D3-7		
	T2D30_DrainOutside034	150 ×	SE same area	Figure D3-8		
	T2D30_DrainOutside035	150 ×	BSE same area	Figure D3-9		
	T2D30_DrainOutside036	1000 ×	SE same area	Figure D3-10		
EDS:	T2D30EDS18		Particles on fiberglass, high C content	Figure D3-11		
	T2D30EDS19 Particle on fiberglass, high C Figure D3 content					
Note:	*Very difficult to get EDS	spectrum	of particles. The particles are			

Note: *Very difficult to get EDS spectrum of particles. The particles are very thin and react under the beam. EDS18 and EDS19 are simply replicates at slightly different sample locations.

Image:	T2D30_DrainInt037	90 ×	SE	Figure D3-13
	T2D30_DrainInt038	150 ×	SE new area	Figure D3-14
	T2D30_DrainInt039	$1000 \times$	SE same area	Figure D3-15
	T2D30_DrainInt040	150 ×	BSE same area	Figure D3-16
EDS:	T2D30EDS20		Particles on fiberglass	Figure D3-17

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ESEM laboratory session from March 9, 2005 T2D30 NRC - Fiberglass on Drain Collar ESEM



Conditions: 20-kV, 12-mm Working Distance, 80 Pa pressure

Outside Sample				
Image:	T2D30DO1	150 ×	BSE Overview	Figure D3-18
	T2D30DO2	1000 ×	Same area	Figure D3-19
	T2D30DO3	90 ×	Same area	Figure D3-20

Screen-	Screen-Side Sample					
Image:	T2D30DS4		90 × .	Overview	Figure D3-21	
	T2D30DS5		150 ×	Same area	Figure D3-22	
	T2D30DS6		1000 ×	Same area as above	Figure D3-23	
Interior	Interior Sample					
Image:	T2D30D17	.	90 ×	l st area	Figure D3-24	

T2D30D18150 ×Different areaT2D30D191000 ×Same as above

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Figure D3-25 Figure D3-26

D3-5

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Figure D3-1. SEM image for a Test #2, Day-30 fiberglass sample on the drain collar next to the drain screen. (T2D30_DrainScreen029)



Figure D3-2. Backscattered SEM image for a Test #2, Day-30 fiberglass sample on the drain collar next to the drain screen, illustrating that deposits have a similar atomic number to fiberglass. (T2D30_DrainScreen030)



Figure D3-3. SEM image for a Test #2, Day-30 fiberglass sample on the drain collar next to the drain screen. (T2D30_DrainScreen031)



Figure D3-4. SEM image at 1000× magnification for a Test #2, Day-30 fiberglass sample on the drain collar next to the drain screen. (T2D30_DrainScreen032)



Figure D3-5. EDS counting spectrum for the center of the image shown in Figure D3-4. (T2D30EDS16~Drain Screen Side Center of Image 032)

The results from the chemical composition analysis for T2D30EDS16 are given in Table D3-1.

Table D3-1. The Chemical Composition for T2D30EDS16 (Figure D3-5)

9 17:18 2005 /tmp/eds_pout.log Page 1 Mar

Group : Sample : Comment : Condition :	NRC T2D30 ID# : 16 drain screen side Full Scale : 20KeV(10eV/ch,2Kch) Live Time : 60.000 sec Aperture # : 1 Acc. Volt : 15.0 KV Probe Current : 1.004E-09 A Stage Point : X=75.582 Y=53.863 Z=10.627 Acq. Date : Wed Mar 9 16:16:45 2005
Element CKN OKN NaKN AlKN SiKN PKN CaKN ZnKN	ModeROI(KeV)K-ratio(%)+/-Net/Backgroundormal0.09-0.460.00000.00000 /27ormal0.25-0.7788.97960.00222775 /22ormal0.83-1.282.13160.0068209 /19ormal1.26-1.781.43060.0004211 /53ormal1.50-2.073.51710.0007520 /73ormal1.75-2.3813.86240.00241032 /44ormal3.40-4.309.23720.0047722 /7ormal8.22-10.0310.88800.003598 /3
Element Mass C 0. O 65. Na 2. Al 1. Si 3. P 9. Ca 7. Zn 10.	Chi_square = 2.8663 Atomic% ZAF Z A F 000 0.0000 4.3367 1.0203 4.2504 0.9999 359 81.5376 0.9412 0.9731 0.9672 1.0000 262 1.9637 1.3596 0.9772 1.3894 1.0013 422 1.0518 1.2735 0.9851 1.2952 0.9981 163 2.2478 1.1523 0.9730 1.1881 0.9968 891 6.3735 0.9142 1.1728 0.7798 0.9996 056 3.5137 0.9787 0.9855 0.9933 0.9998 847 3.3119 1.2765 1.2799 0.9973 1.0000
Total 100.	$000 \ 100.0000$

ormalization factor 0.7805



Figure D3-6. EDS counting spectrum for the film between fibers shown in Figure D3-4. The film is rich in silicon, oxygen, aluminum, sodium, and calcium. (T2D30EDS17~Drain Screen Side Film on Fiber Glass)
The results from the chemical composition analysis for T2D30EDS17 are given in Table D3-2.

Table D3-2. The Chemical Composition for T2D30EDS17 (Figure D3-6)

Mar 9 17:20 2005 /tmp/eds_pout.log Page 1

Group Sample Comment Condition	: NRC : T2D30 : film on : Full Sc Live Ti Acc. Vo Stage P Acq. Da	ID# : 17 fiberglass ale : 20KeV me : 60.0 lt : 15.0 oint : X=75. te : Wed M	(10eV/ch,2 00 sec KV 684 Y=53.9 ar 9 16:2	Kch) Aperture Probe Cur 77 Z=10.6 1:30 2005	# : 1 rent : 1.00 27	1E-09 A
Element	Mode	ROI (KeV)	K-ratio(%) +/-	Net/Backgr	round
ОК	Normal	0.25- 0.77	7.9629	0.0018	1866 /	12
Na K	Normal	0.83- 1.28	0.4017	0.0037	296 /	15
Al K	Normal	1.26- 1.78	0.8589	0.0006	954 /	93
Si K	Normal	1.50- 2.07	2.1743	0.0011	2413 /	84
ΡK	Normal	1.75- 2.38	0.6172	0.0018	345 /	129
Ca K	Normal	3.40- 4.30	0.6433	0.0037	378 /	6
Fe K	Normal	6.04- 7.40	0.0946	0.0232	18 /	2
ους δος μαγίνες του στο πολιτικό του τους που		Ch	i_square =	2.9399		
Element Ma	ass% At	omic% ZAF	Z	A	F	
0 5	59.959 73	.0617 0.9746	0.9921 0.	9824 1.00	00	
Na	3.492 2	.9608 1.1249	0.9964 1.	1283 1.00	06	
Al	7.333 5	.2984 1.1051	1.0045 1.	1064 0.99	44	
Si 1	L8.694 12	.9763 1.1128	0.9923 1.	1231 0.99	86	
P	4.794 3	.0176 1.0055	1.1961 0.	8408 0.99	98	
Ca	4.994 2	.4289 1.0048	1.0056 0.	9992 1.00	00	
Fe	0.734 0	.2562 1.0038	1.0013 1.	0018 1.00	08	
Total 10 Normalizat	0.000 100 100	.0000 r = 7.7260	*********			



Figure D3-7. SEM image for a Test #2, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen) showing deposits or growth on fiberglass. (T2D30_DrainOutside033)



Figure D3-8. SEM image for a Test #2, Day-30 exterior fiberglass sample on the drain collar showing deposits or growth on fiberglass. (T2D30_DrainOutside034)



Figure D3-9. Backscattered SEM image for a Test #2, Day-30 exterior fiberglass sample on the drain collar. (T2D30_DrainOutside035)



Figure D3-10. SEM image at a higher magnification for a Test #2, Day-30 exterior fiberglass sample on the drain collar showing deposits or growth on fiberglass. (T2D30_DrainOutside 036)



Figure D3-11. EDS counting spectrum for deposits or growth on fiberglass. The deposits contain a significant amount of carbon. (T2D30EDS18~Drain Outside Particles High C)



Figure D3-12. EDS counting spectrum for a deposit or growth on fiberglass. The deposit contains a significant amount of carbon. (T2D30EDS19)

The results from the chemical composition analysis for T2D30EDS19 are given in Table D3-3.

Table D3-3. The Chemical Composition for T2D30EDS19 (Figure D3-12)

Mar 9 16:58 2005 /tmp/eds_pout.log Page 1

Group : NRC Sample : T2D30 Comment : particl Condition : Full Sc Live Ti Acc. Vo Stage Pe Acq. Da	ID# : 19 es on fiberglass ale : 20KeV(10eV/c me : 120.000 sec lt : 15.0 KV oint : X=81.088 Y=6 te : Wed Mar 9 1	h,2Kch) Aperture # Probe Current 3.027 Z=10.627 6:55:59 2005	: 1 : 9.749E-09 A
Element Mode CK Normal OK Normal AlK Normal SiK Normal ZnK Normal CaK Normal	ROI (KeV)K-rati0.09-0.460.110.25-0.770.031.26-1.780.001.50-2.070.008.22-10.030.103.40-4.300.00	o(%) +/- Net 38 0.0002 59 0.0006 48 0.0002 33 0.0003 99 0.0036 38 0.0018	/Background 771 / 2 164 / 44 104 / 6 71 / 11 145 / 2 44 / 4
Element Mass% Ato C 59.630 79 O 12.718 12 Al 1.176 0 Si 0.710 0 Zn 25.102 6 Ca 0.664 0	Chi_squar Omic% ZAF Z .7022 2.7985 0.9722 .7615 1.8946 0.9274 .6998 1.3104 0.9398 .4060 1.1613 0.9286 .1648 1.2204 1.2263 .2658 0.9225 0.9421	e = 3.5725 A F 2.8786 1.0000 2.0429 1.0000 1.3945 0.9998 1.2506 1.0000 0.9952 1.0000 0.9951 0.9991	
Total 100.000 100 Normalization facto	.0000 r = 187.2003		



Figure D3-13. SEM image for a Test #2, Day-30 interior fiberglass sample on the drain collar. Image shows deposits or growth on fiberglass. (T2D30_DrainInt037)



Figure D3-14. SEM image for a Test #2, Day-30 interior fiberglass sample on the drain collar. Image shows deposits or growth on fiberglass. (T2D30_DrainInt038)



Figure D3-15. SEM image of a higher magnification for a Test #2, Day-30 interior fiberglass sample on the drain collar. The image shows deposits or growth on fiberglass. (T2D30_Drain Int039)



Figure D3-16. Backscattered SEM image for a Test #2, Day-30 interior fiberglass sample on the drain collar. Deposits appear to have a similar or lower atomic number as compared with fiberglass. (T2D30 DrainInt040)



Figure D3-17. EDS counting spectrum for the deposits or growth on fiberglass. The deposits are rich in silicon, carbon, oxygen, and calcium. The deposits are very thin, and thus it was difficult to obtain high-quality EDS spectra. (T2D30EDS20)

The results from the chemical composition analysis for T2D30EDS20 are given in Table D3-4.

Table D3-4. The Chemical Composition for T2D30EDS20 (Figure D3-17)

Mar 9 17:16 2005 /tmp/eds_pout.log Page 1

Group : NRC Sample : T2D30 ID# : 20 Comment : particles on fiberglass (Condition : Full Scale : 20KeV(10eV/ch,2Kch) Live Time : 120.000 sec Aperture # : 1 Acc. Volt : 15.0 KV Probe Current : 1.375E-09 Stage Point : X=68.013 Y=68.140 Z=10.627 Acq. Date : Wed Mar 9 17:14:10 2005	A
ElementModeROI(KeV)K-ratio(%)+/-Net/BackgroundC KNormal0.09-0.460.04200.000140 /O KNormal0.25-0.770.14250.000592 /Si KNormal1.50-2.070.02920.000289 /Ca KNormal3.40-4.300.02250.001336 /	2 3 2 1
Chi_square = 0.7730 Element Mass% Atomic% ZAF Z A F C 30.132 39.0987 2.5156 1.0305 2.4412 0.9999 O 55.239 53.8107 1.3590 0.9832 1.3822 1.0000 Si 8.443 4.6849 1.0136 0.9850 1.0293 0.9998 Ca 6.187 2.4058 0.9650 1.0007 0.9642 1.0001	
Total 100.000 100.0000 Normalization factor = 285.2892	



Figure D3-18. ESEM image for a Test #2, Day-30 exterior fiberglass sample on the drain collar. Image shows particulate deposits or growth on fiberglass. (T2D30DO1)



Figure D3-19. ESEM image of a higher magnification for a Test #2, Day-30 exterior fiberglass sample on the drain collar. The image shows particulate deposits or growth on fiberglass. (T2D30DO2)



Figure D3-20. ESEM image for a Test #2, Day-30 exterior fiberglass sample on the drain collar. Image shows particulate deposits or growth on fiberglass. (T2D30DO3)



Figure D3-21. ESEM image for a Test #2, Day-30 fiberglass sample on the drain collar next to the drain screen. (T2D30DS4)



Figure D3-22. ESEM image for a Test #2, Day-30 fiberglass sample on the drain collar next to the drain screen. (T2D30DS5)



Figure D3-23. ESEM image of a higher magnification for a Test #2, Day-30 fiberglass sample on the drain collar next to the drain screen. (T2D30DS6)



Figure D3-24. ESEM image for a Test #2, Day-30 interior fiberglass sample on the drain collar. Image suggests that interior fiberglass was relatively clean as compared with exterior. (T2D30D17)



Figure D3-25. ESEM image for a Test #2, Day-30 interior fiberglass sample on the drain collar. (T2D30D18)



Figure D3-26. ESEM image of a higher magnification for a Test #2, Day-30 interior fiberglass sample on the drain collar. The image shows deposits or growth on the interior fiberglass, although the quantity was less than for exterior samples. (T2D30D19)

Appendix D4

ESEM and SEM/EDS Data for Test #2, Day-30 Birdcage Fiberglass

Figures

Figure D4-1.	SEM image for a Test #2, Day-30 exterior fiberglass sample within the					
	birdcage. The image shows particulate deposits or growth on fiberglass.					
	(TD30_BirdcageExt041)	D4-5				
Figure D4-2.	EDS counting spectrum for the bright particle on the lower-left side of Figure					
	D4-1. (T2D30EDS21)	D4-5				
Figure D4-3.	Backscatter SEM image for a Test #2, Day-30 exterior fiberglass sample					
	within the birdcage. (T2D30_BirdcageExt042)	D4-7				
Figure D4-4.	SEM image for a Test #2, Day-30 exterior fiberglass sample within the					
	birdcage. The field is the same as that shown in Figure D4-3.					
	(T2D30_BirdcageExt043)I	D4-7				
Figure D4-5.	SEM image of a higher magnification for a Test #2, Day-30 exterior					
	fiberglass sample within the birdcage. (T2D30_BirdcageExt044)I	D 4-8				
Figure D4-6.	EDS counting spectrum for the crystal-shaped deposit at the lower-left side of					
	Figure D4-5. (T2D30EDS22)	D4-8				
Figure D4-7.	EDS counting spectrum for the deposits attached to fiberglass, as shown in					
	Figure D4-5. (T2D30EDS23)	04-9				
Figure D4-8.	SEM image for a Test #2, Day-30 interior fiberglass sample within the					
	birdcage. The image shows deposits or growth on the fiberglass.					
	(T2D30_BirdcageInt045)De	4-11				
Figure D4-9.	SEM image for a Test #2, Day-30 interior fiberglass sample within the					
	birdcage. The image shows deposits or growth on the fiberglass.					
	(T2D30_BirdcageInt046)D4	4-11				
Figure D4-10.	Backscattered SEM image for a Test #2, Day-30 interior fiberglass sample					
	within the birdcage. The atomic number of the deposits is similar to that of the					
	fiberglass. (T2D30_BirdcageInt047)D	4-12				

Figure D4-11. SEM image of a higher magnification for a Test #2, Day-30 interior fiberglass					
sample within the birdcage. The image shows deposits or growth on the					
fiberglass. (T2D30_BirdcageInt048) D4-12					
Figure D4-12. EDS counting spectrum for deposits on the fiberglass, as shown in Figure					
D4-11. (T2D30EDS24)					
Figure D4-13. ESEM image for a Test #2, Day-30 exterior fiberglass sample within the					
birdcage. (T2D30BX9) D4-15					
Figure D4-14. ESEM image for a Test #2, Day-30 exterior fiberglass sample within the					
birdcage. (T2d30bx10)					
Figure D4-15. ESEM image of a higher magnification for a Test #2, Day-30 exterior					
fiberglass sample within the birdcage. (T2d30bx11)D4-16					
Figure D4-16. ESEM image of a higher magnification from another area for a Test #2, Day-					
30 exterior fiberglass sample within the birdcage. (T2d30bx12) D4-16					
Figure D4-17. ESEM image for a Test #2, Day-30 interior fiberglass sample within the					
birdcage. The image shows deposits or growth on the fiberglass.					
(T2d30bi13)D4-17					
Figure D4-18. ESEM image of a higher magnification for a Test #2, Day-30 interior					
fiberglass sample within the birdcage. The image shows deposits or growth on					
the fiberglass. (T2d30bi14)D4-17					

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Tables

Table D4-1.	The Chemical Composition for T2D30EDS21	D4-6
Table D4-2.	The Chemical Composition for T2D30EDS23	04-10
Table D4-3.	The Chemical Composition for T2D30EDS2	04-14

This appendix lists the ESEM/SEM/EDS results for the fiberglass samples within a birdcage submerged in the test solution. The purpose of this analysis was to determine the degree and the extent of particulate debris attached to the fiberglass. The fiberglass samples were extracted from the birdcage on March 7, 2005, the date Test #2 was shut down. Both exterior and interior fiberglass samples were examined. Microprobe SEM was used to examine the fiberglass samples after they were air dried and coated with gold/palladium. In addition to microprobe SEM, ESEM was used to analyze the wet fiberglass samples without any coating and under a low-vacuum condition (i.e., 80 Pa) to minimize the potential for modification of the fiberglass samples that might occur through the drying process. Microprobe SEM/EDS and ESEM results of the Test #2, Day-30 birdcage fiberglass samples were obtained on March 9, 2005.

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Transcribed Laboratory Log

Microprobe laboratory session from March 9, 2005

T2D30 Samples—NRC



Conditions: 15-kV, 1-nA beam current, Aperture = 2 Note: Bold spots in sketch denote carbon glue used to secure samples.

Sample Birdcage Exterior

Image:	T2D30_BirdcageExt041	90 ×	BSE	Figure D4-1
EDS:	T2D30EDS21		Bright particle left side of image 041	Figure D4-2
Image:	T2D30_BirdcageExt042	150 ×	BSE	Figure D4-3
	T2D30_BirdcageExt043	150 ×	SE	Figure D4-4
	T2D30_BirdcageExt044	1000 ×	SE	Figure D4-5
EDS:	T2D30EDS22	2	EDS on lath-like crystal	Figure D4-6
	T2D30EDS23		Particles on fiberglass	Figure D4-7

Sample Birdcage Interior

Image:	T2D30_BirdcageInt045	90 × 🕚	SE image	Figure D4-8
	T2D30_BirdcageInt046	150 ×	SE image same area	Figure D4-9
	T2D30_BirdcageInt047	150 ×	BSE same area	Figure D4-10
	T2D30_BirdcageInt048	$1000 \times$	SE same area	Figure D4-11
EDS:	T2D30EDS24	•	Globules on fiberglass	Figure D4-12

ESEM laboratory session from March 9, 2005

Birdcage Sample Exterior

Image:	T2D30BX9	95 ×	On debris/precipitate	Figure D4-13
	T2D30BX10	150 ×	On glass fibers	Figure D4-14
	T2D30BX11	1000 ×	On fibers	Figure D4-15
	T2D30BX12	700 ×	On particle	Figure D4-16

Birdcage Sample Interior

Image:	T2D30BI13	150 ×	On fibers	Figure D4-17
	T2D30BI14	1000 × .		Figure D4-18



Figure D4-1. SEM image for a Test #2, Day-30 exterior fiberglass sample within the birdcage. The image shows particulate deposits or growth on fiberglass. (TD30_BirdcageExt041)



Figure D4-2. EDS counting spectrum for the bright particle on the lower-left side of Figure D4-1. (T2D30EDS21)

The results from the chemical composition analysis for T2D30EDS21 are given in Table D4-1.

Table D4-1. The Chemical Composition for T2D30EDS21

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Group Sample Comment Conditio	: NRC : T2D3 : brig on : Full Live Acc. Stag Acq.	ID# : 21 t particle Scale : 20KeV(10eV/d Time : 60.000 sec Volt : 15.0 KV Point : X=36.780 Y=5 Date : Wed Mar 9 1	ch,2Kch) Aperture # Probe Current 58.457 Z=10.627 7:26:42 2005	: 1 : 5.403E-10 A
Element O K Na K P K Ca K Zn K	Mode Norma Norma Norma Norma	ROI(KeV) K-rational Control (KeV) Control (KeV) K-rational Control (KeV	LO(%) +/- Net/H 202 0.0022 25 306 0.0151 22 34 0.0034 35 304 0.0050 5 336 0.0071	Background 572 / 15 298 / 23 577 / 28 586 / 11 719 / 2
Element	Mass%	Chi_squar Atomic% ZAF Z	re = 3.0137 A F	
0	35.828	61.5751 1.0317 0.9179	1.1240 1.0000	
Na	2.043	2.4438 1.5945 0.9209	9 1.7289 1.0014	
P	19.372	17.1970 0.9581 1.1032	2 0.8685 0.9999	
Ca	3.013	2.0671 0.9537 0.9240) 1.0329 0.9993	
Zn	39.743	16.7170 1.1826 1.1840	0.9988 1.0000	
Total Normaliz	100.000 ation fa	00.0000 tor = 1.7073		



Figure D4-3. Backscatter SEM image for a Test #2, Day-30 exterior fiberglass sample within the birdcage. (T2D30_BirdcageExt042)



Figure D4-4. SEM image for a Test #2, Day-30 exterior fiberglass sample within the birdcage. The field is the same as that shown in Figure D4-3. (T2D30_BirdcageExt043)



Figure D4-5. SEM image of a higher magnification for a Test #2, Day-30 exterior fiberglass sample within the birdcage. (T2D30_BirdcageExt044)



Figure D4-6. EDS counting spectrum for the crystal-shaped deposit at the lower-left side of Figure D4-5. (T2D30EDS22)



Figure D4-7. EDS counting spectrum for the deposits attached to fiberglass, as shown in Figure D4-5. (T2D30EDS23)

The results from the chemical composition analysis for T2D30EDS23 are given in Table D4-2.

Table D4-2. The Chemical Composition for T2D30EDS23

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Group : 1 Sample : 7 Comment : 1 Condition : 1 I Z	NRC T2D30 ID# : 23 Darticles on fiberg Full Scale : 20KeV Live Time : 120.0 Acc. Volt : 15.0 Stage Point : X=35. Acq. Date : Wed M	glass 7(10eV/ch,2Kc 000 sec Ar KV Pr 661 Y=58.464 Mar 9 17:47:	ch) perture # cobe Curren 4 Z=10.627 :47 2005	: 1 t : 1.020E-10 A	4
Element M OK No NaK No MgK No AlK No SiK No PK No CaK No	Mode ROI (KeV) ormal 0.25-0.77 ormal 0.83-1.28 ormal 1.03-1.52 ormal 1.26-1.78 ormal 1.50-2.07 ormal 1.75-2.38 ormal 3.40-4.30	K-ratio(%) 23.8450 1.5443 0.4403 1.8853 3.3376 3.3673 2.8112	+/- Ne 0.0014 0.0028 0.0005 0.0004 0.0006 0.0014 0.0031	t/Background 1139 / 6 232 / 10 84 / 38 427 / 28 755 / 38 384 / 43 336 / 2	
Element Mass O 62.7 Na 4.6 Mg 1.2 Al 5.6 Si 9.8 P 8.3 Ca 7.3	Ch Atomic% ZAF 41 75.5081 0.9977 58 3.9012 1.1437 84 1.0170 1.1059 81 4.0539 1.1426 37 6.7440 1.1176 99 5.2214 0.9458 99 3.5544 0.9980	i_square = Z 0.9907 1.00 0.9951 1.14 0.9978 1.10 1.0033 1.14 0.9910 1.13 1.1947 0.79 1.0045 0.99	2.0568 A F 070 1.0000 485 1.0007 091 0.9993 431 0.9963 808 0.9973 920 0.9996 934 1.0001	r rom o de la constanción de la constan L	
Total 100.0	00 100.0000		n nin na	ರ್ ನಾಯಾ ನಾ ವಿ. ಕೇಂದ್ರ ವ್ಯಾಪ್ ಕೇಂದ್ರ ಕೊಂಡಿಗಳು ಆಗ್ರೆ ಕೇಂದ್ರ	

Normalization factor = 2.6373



Figure D4-8. SEM image for a Test #2, Day-30 interior fiberglass sample within the birdcage. The image shows deposits or growth on the fiberglass. (T2D30 BirdcageInt045)



Figure D4-9. SEM image for a Test #2, Day-30 interior fiberglass sample within the birdcage. The image shows deposits or growth on the fiberglass. (T2D30 BirdcageInt046)



Figure D4-10. Backscattered SEM image for a Test #2, Day-30 interior fiberglass sample within the birdcage. The atomic number of the deposits is similar to that of the fiberglass. (T2D30_BirdcageInt047)



Figure D4-11. SEM image of a higher magnification for a Test #2, Day-30 interior fiberglass sample within the birdcage. The image shows deposits or growth on the fiberglass. (T2D30_BirdcageInt048)



Figure D4-12. EDS counting spectrum for deposits on the fiberglass, as shown in Figure D4-11. (T2D30EDS24)

The results from the chemical composition analysis for T2D30EDS24 are given in Table D4-3.

Table D4-3. The Chemical Composition for T2D30EDS2

Mar 9 18:08 2005 /tmp/eds_pout.log Page 1

	Group Sample Comment Condition	: NRC : T2D30 : globulo : Full So Live T Acc. Vo Stage D Acq. Da	ID# : 24 es on fibergla cale : 20KeV ime : 120.00 olt : 15.0 H Point : X=49.1 ate : Wed Ma	ass birdcage (10eV/ch,2Kd 00 sec Ag KV P: 105 Y=62.519 ar 9 18:06	e interior ch) perture # robe Curren 9 Z=10.627 :33 2005	: 1 t : 1.019E-1	.0 A
-	Element O K Na K Mg K Al K Si K P K Ca K	Mode Normal Normal Normal Normal Normal Normal	ROI (KeV) 0.25- 0.77 0.83- 1.28 1.03- 1.52 1.26- 1.78 1.50- 2.07 1.75- 2.38 3.40- 4.30	K-ratio(%) 10.6755 0.6105 0.0691 0.1947 0.8895 0.6913 0.4040	+/- Ne 0.0009 0.0018 0.0003 0.0002 0.0004 0.0009 0.0018	t/Background 509 / 92 / 13 / 44 / 201 / 79 / 48 /	4 3 10 8 12 14 2
	Element Ma O 7 Na Mg Al Si P Ca	At 3.306 82 6.327 4 0.700 0 1.996 5 8.560 5 5.609 2 3.502 5	Comic% ZAF 2.8087 0.7875 4.9737 1.1884 0.5202 1.1607 1.3369 1.1755 5.5082 1.1035 3.2731 0.9305 1.5793 0.9941	Z 0.9951 0.79 0.9997 1.18 1.0024 1.19 1.0080 1.16 0.9959 1.11 1.2006 0.77 1.0101 0.98	A F 914 1.0000 375 1.0011 578 1.0001 597 0.9969 103 0.9981 752 0.9998 341 1.0001		*5
	Total 10 Normalizat	0.000 100 ion facto	0.0000 or = 8.7200	na venerana eta eta eta eta eta eta eta eta eta et	102.071 (PD-) (TT-) (TT-	y. nurrali angen ginangen kina kina kina kina kina kina kina kin	

D4-14



Figure D4-13. ESEM image for a Test #2, Day-30 exterior fiberglass sample within the birdcage. (T2D30BX9)



Figure D4-14. ESEM image for a Test #2, Day-30 exterior fiberglass sample within the birdcage. (T2d30bx10)



Figure D4-15. ESEM image of a higher magnification for a Test #2, Day-30 exterior fiberglass sample within the birdcage. (T2d30bx11)



Figure D4-16. ESEM image of a higher magnification from another area for a Test #2, Day-30 exterior fiberglass sample within the birdcage. (T2d30bx12)



Figure D4-17. ESEM image for a Test #2, Day-30 interior fiberglass sample within the birdcage. The image shows deposits or growth on the fiberglass. (T2d30bi13)



Figure D4-18. ESEM image of a higher magnification for a Test #2, Day-30 interior fiberglass sample within the birdcage. The image shows deposits or growth on the fiberglass. (T2d30bi14)

Appendix E

SEM/EDS Data for T2D30 Sediment

Figures

Figure E-1.	SEM image for a Test #2, Day-30 sediment sample at 150 × magnification.	
	(T2D30SEDMT003)	E - 4
Figure E-2.	SEM image for a Test #2, Day-30 sediment sample at 100 × magnification.	
	(T2D30SEDMT006)	E-4
Figure E-3.	EDS counting spectrum for the circularly layered material close to the right	
	edge of Figure E-2. (T2D30SED1)	E-5
Figure E-4.	SEM image for a Test #2, Day-30 sediment sample at 300 × magnification.	
	(T2D30SEDMT004)	E - 7
Figure E-5.	EDS counting spectrum for the porous structured material shown in Figure	
	E-3. (T2D30SED2)	E-7

Tables

 Table E-1.
 The Chemical Composition for T2D30SED1 (Figure E-2)

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Particulate sediments at the bottom of the tank directly relate to the corrosion products and debris generated during ICET tests. This appendix lists the SEM/EDS results for the sediment samples collected from the bottom of the tank on the date Test #2 was shut down (March 7, 2005). The purpose of these analyses is to provide information on the morphology and the composition of the sediment to evaluate the potentially occurring chemical reactions during ICET tests.

Probe SEM was used to examine the sediment samples after drying in air at room temperature, followed by being coated with carbon. EDS results provide a semiquantitative elemental analysis of the sediment after calibration. Probe SEM/EDS results of the Test #2, Day-30 sediment samples were obtained on April 12, 2005.

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Transcribed Laboratory Log

Laboratory session from April 12, 2005.

T2D30 Sediment Samples



Sediment Samples

Image:	T2D30SEDMT003	150 ×	SEM image	Figure E-1
	T2D30SEDMT006	100 ×	SEM image sediment sample	Figure E-2
EDS:	T2D30SED1		Spot of the layer show in 006	Figure E-3
Image:	T2D30SEDMT004	300 ×	Porous structure	Figure E-4
EDS:	T2D30SED2		Of porous material in Image 004	Figure E-5



Figure E-1. SEM image for a Test #2, Day-30 sediment sample at 150 × magnification. (T2D30SEDMT003)



Figure E-2. SEM image for a Test #2, Day-30 sediment sample at 100 × magnification. (T2D30SEDMT006)



Figure E-3. EDS counting spectrum for the circularly layered material close to the right edge of Figure E-2. (T2D30SED1)

The results from the chemical composition analysis for T2D30SED1 are given in Table E-1.

Table E-1. The Chemical Composition for T2D30SED1 (Figure E-2)

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Figure E-4. SEM image for a Test #2, Day-30 sediment sample at 300 × magnification. (T2D30SEDMT004)







Appendix F1

TEM Data for Test #2, Day-4 Solution Samples

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	(KerryFeb09F-30-cm (bin)-02)
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	(KerryFeb09-30cm(bin)-01)

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(KerryFeb09-30cm(bin)-02)
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Figure F1-27. Electron micrograph magnified 10,000 times for a second Test #2, Day-4
filtered sample location. (KerryFeb09F-10k-02)
Figure F1-28. Electron micrograph magnified 10,000 times for a third Test #2, Day-4
filtered sample location. (KerryFeb09F-10k-03)

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This appendix presents TEM images and diffraction patterns for Test #2, Day-4 (February 9, 2005) filtered and unfiltered solution samples. The filtered samples were obtained by passing solution through a 0.7- μ m fiberglass filter at 60°C. The unfiltered solution samples were extracted from the tank directly. A drop of each solution sample was placed onto a copper grid of 200 mesh. After being dried in air at room temperature, the sample was ready for TEM analysis. The TEM results and diffraction patterns were obtained on February 9, 2005. Diffraction patterns show whether the sample was amorphous or crystalline. When a sample gives clear and significant diffraction patterns, it is crystalline. Otherwise, it is amorphous. The results show that all of the Test #2, Day-4 samples were amorphous.

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Figure F1-1. TEM image for a Test #2, Day-4 filtered sample solution. (KerryFeb09F-3-cm(bin)-01)



Figure F1-2. TEM image for a second Test #2, Day-4 filtered sample solution. (KerryFeb09F-30-cm (bin)-02)



Figure F1-3. Electron micrograph magnified 50,000 times for one Test #2, Day-4 filtered sample location. (KerryFeb09F-50k-01)



Figure F1-4. Electron micrograph magnified 50,000 times for a second Test #2, Day-4 filtered sample location. (KerryFeb09F-50k-02)



Figure F1-5. Electron micrograph magnified 50,000 times for a third Test #2, Day-4 filtered sample location. (KerryFeb09F-50k-03)



Figure F1-6. Electron micrograph magnified 2000 times for one Test #2, Day-4 unfiltered sample location. (KerryFeb09-2k-01)



Figure F1-7. Electron micrograph magnified 2000 times for a second Test #2, Day-4 unfiltered sample location. (KerryFeb09-2k-02)



Figure F1-8. Electron micrograph magnified 4000 times for one Test #2, Day-4 unfiltered sample location. (KerryFeb09-4k-01)



Figure F1-9. Electron micrograph magnified 4000 times for a second Test #2, Day-4 unfiltered sample location. (KerryFeb09-4k-02)



Figure F1-10. Electron micrograph magnified 4,000 times for a third Test #2, Day-4 unfiltered sample location. (KerryFeb09-4k-03)



Figure F1-11. Electron micrograph magnified 10,000 times for one Test #2, Day-4 unfiltered sample location. (KerryFeb09-10k-01)



Figure F1-12. Electron micrograph magnified 10,000 times for a second Test #2, Day-4 unfiltered sample location. (KerryFeb09-10k-02)



Figure F1-13. Electron micrograph magnified 10,000 times for a third Test #2, Day-4 unfiltered sample location. (KerryFeb09-10k-03)



Figure F1-14. TEM image for one Test #2, Day-4 unfiltered sample location. (KerryFeb09-30cm(bin)-01)



Figure F1-15. TEM image for a second Test #2, Day-4 unfiltered sample location. (KerryFeb09-30cm(bin)-02)



Figure F1-16. TEM image for a third Test #2, Day-4 unfiltered sample location. (KerryFeb09-30cm (bin)-03)



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Figure F1-17. Electron micrograph magnified 50,000 times for one Test #2, Day-4 unfiltered sample location. (KerryFeb09-50k-01)



Figure F1-18. Electron micrograph magnified 50,000 times for a second Test #2, Day-4 unfiltered sample location. (KerryFeb09-50k-02)



Figure F1-19. Electron micrograph magnified 50,000 times for a third Test #2, Day-4 unfiltered sample location. (KerryFeb09-50k-03)



Figure F1-20. Electron micrograph magnified 2000 times for one Test #2, Day-4 filtered sample location. (KerryFeb09F-2k-01)



Figure F1-21. Electron micrograph magnified 2000 times for a second Test #2, Day-4 filtered sample location. (KerryFeb09F-2k-02)



Figure F1-22. Electron micrograph magnified 2000 times for a third Test #2, Day-4 filtered sample location. (KerryFeb09F-2k-03)

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Figure F1-23. Electron micrograph magnified 4000 times for one Test #2, Day-4 filtered sample location. (KerryFeb09F-4k-01)



Figure F1-24. Electron micrograph magnified 4000 times for a second Test #2, Day-4 filtered sample location. (KerryFeb09F-4k-02)



Figure F1-25. Electron micrograph magnified 4000 times for a third Test #2, Day-4 filtered sample location. (KerryFeb09F-4k-03)



Figure F1-26. Electron micrograph magnified 10,000 times for one Test #2, Day-4 filtered sample location. (KerryFeb09F-10k-01)



Figure F1-27. Electron micrograph magnified 10,000 times for a second Test #2, Day-4 filtered sample location. (KerryFeb09F-10k-02)



Figure F1-28. Electron micrograph magnified 10,000 times for a third Test #2, Day-4 filtered sample location. (KerryFeb09F-10k-03)