# Appendix E

### SEM/EDS Data for T2D30 Sediment

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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.

#### **Transcribed Laboratory Log**

Laboratory session from April 12, 2005.

T2D30 Sediment Samples



#### **Sediment Samples**

Image:	T2D30SEDMT003	$150 \times$	SEM image	Figure E-1
	T2D30SEDMT006	$100 \times$	SEM image sediment sample	Figure E-2
EDS:	T2D30SED1		Spot of the layer show in 006	Figure E-3
Image:	T2D30SEDMT004	$300 \times$	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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Group Sample Comment Condition	: NRC : Sedime : T2D30 : Full S Live T Acc. V Stage Acq. D	ent ID# layered cale : ime : Colt : Point : ate :	: 1 sedim 20KeV 60.0 15.0 X=86. Tue A	ent (10eV/c 00 sec KV 820 Y=5 pr 12 1	h,2Kch Ape Pro 7.236 3:28:0	) rture # be Curr Z=10.92 7 2005	: 1 ent : 4 7	.371E	-09 A
Element C K O K Na K Mg K Al K Si K P K Ca K Cu K Zn K	Mode Normal Normal Normal Normal Normal Normal Normal Normal Normal	ROI(P 0.09- 0.25- 0.81- 0.97- 1.19- 1.50- 1.75- 3.39- 7.63- 8.22-1	CeV) 0.46 0.77 1.27 1.57 1.83 2.05 2.38 4.30 9.27 0.03	K-rati 4.52 30.96 0.22 0.98 8.35 2.83 9.98 7.57 0.26 0.65	o(%) 08 0 57 0 89 0 30 0 15 0 77 0 89 0 85 0 19 0 58 0	+/- .0013 .0080 .0033 .0008 .0016 .0012 .0090 .0045 .0048 .0069	Net/Bac 6861 31679 673 4198 35453 11233 24403 14581 102 194	kgrou: / / / / / /	nd 798 628 316 412 290 2308 700 62 10
Element Ma C 2 O 4 Na Mg Al 1 Si P Ca Cu Zn	ASS% At 20.663 30 5.036 49 0.316 ( 1.403 5 0.778 5 9.217 5 7.778 3 0.344 ( 0.868 0 0.000 100	tomic% 0.4778 4 9.8700 1 0.2434 1 1.0223 1 7.0766 1 2.2689 1 5.2716 0 3.4382 1 0.0959 1 0.2353 1	Ch: ZAF .4603 .4193 .3462 .3928 .2594 .2370 .9004 .0016 .2820 .2917	i_square Z 1.0251 0.9778 1.0323 0.9720 1.0050 0.9827 1.1798 1.0043 1.2856 1.2954	A 4.3512 1.4515 1.3050 1.4370 1.2560 1.2628 0.7635 0.9974 0.9972 0.9971	F 1.0000 1.0000 0.9993 0.9973 0.9975 0.9968 1.0000 1.0000 1.0000	) ) 7 } ) )		

Normalization factor = 1.0247



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



Figure E-5. EDS counting spectrum for the porous structured material shown in Figure E-3. (T2D30SED2)

## Appendix F1

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

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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- $\mu$ 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.



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)



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)



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)