Appendix E1

SEM/EDS Data for Test #5, Day-30 Aluminum Coupons

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This appendix shows the SEM/EDS results for the metal aluminum coupons under two categories: (1) unsubmerged and (2) submerged. Unsubmerged refers to coupons located above the water level of the test tank during ICET tests. Unsubmerged coupons were contacted with the solution only during the initial 4-hour spray phase. In addition, the surface of the unsubmerged coupons may be affected during the test by the moist air in the tank gas space. Submerged refers to the coupons submerged in the solution during the test.

The coupon samples were collected on August 25, 2005 (the date Test #5 was shut down), and examined by SEM/EDS on August 30 and September 6, 2005. The aluminum coupon samples were dried in air before coating with Au/Pd for SEM examination. SEM results present the surface condition of the aluminum coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products.

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Transcribed Laboratory Log

Laboratory session from September 6, 2005. SEM Test #5, Day-30 Aluminum Coupons

Conditions: e=15.0kV, WD=11mm



1--Yellow Deposits on Submerged Rack 2--Sediment (T5D30) 4--Al-Submerged 7--Cu Unsubmerged 10--Steel-Submerged

5--Gal-Steel Unsubmerged 8--Cu-Submerged 11--Drain Collar Interior

3--Al-Unsubmerged 6--Gal-Steel Submerged 9--Steel-Unsubmerged 12--Drain Collar Outside Ext.

Unsubmerged Aluminum Coupons

Image:	T5D30SuspAl007	$100 \times$	SEM image	Figure E1-1
	T5D30SuspA1008	$500 \times$	SEM image	Figure E1-2
EDS:	T5D30SuspAl_flat		EDS on flat surface in 008	Figure E1-3
	T5D30SuspAl_Granul		EDS on particle in 008	Figure E1-4
Image:	T5D30SuspA1009	1000 ×	SEM image higher magnification	Figure E1-5

Submerged Al Coupon

Image:	T5D30SubmAl010	$250 \times$	SEM image	Figure E1-6
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	T5D30SubmAl012	$1200 \times$	SEM annotated	Figure E1-8
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Figure E1-1. SEM image magnified 100 times for a Test #5, Day-30 unsubmerged aluminum coupon sample. (T5D30SuspAl007.bmp)



Figure E1-2. SEM image magnified 500 times for a Test #5, Day-30 unsubmerged aluminum coupon sample. (T5D30SuspAl008.bmp)



Figure E1-3. EDS counting spectrum for the smooth surface (EDS1) of the aluminum coupon shown in Figure E1-2. (T5D30SuspAl~flat.jpg)



Figure E1-4. EDS counting spectrum for the granular particles (EDS2) on the aluminum coupon surface shown in Figure E1-2. (T5D30SuspAl~Granul.jpg)

The results from the chemical composition analysis for T5D30SuspAl_Granul.jpg are given in Table E1-1.

Group Sample Comment Condition	: NRC : T5D30 : Granula : Full Sc Live Ti Acc. Vo Stage P Acq. Da	ID# : 5 r particles ale : 20KeV me : 60.0 lt : 15.0 oint : X=88. te : Tue A	on suspende (10eV/ch,2K 00 sec A KV F 063 Y=64.38 ug 30 13:44	ed Al Aperture Trobe Cur 3 Z=11.8 1:34 2005	# : 1 rent : 1.006E- 48	09 A
Element Al K	Mode Normal Normal	ROI(KeV) 1.26- 1.78 0.31- 0.74	K-ratio(%) 16.5329 20 9428	+/- 0.0023	Net/Backgroun 18452 / 19756 /	d 29 12
			i_square =	45.7092		
Element M Al O	ass% At 46.210 33 53.790 66	omic% ZAF .7485 1.0224 .2515 0.9395	Z 1.0020 1.0 1.0018 0.9	A 2003 1.00 0377 1.00	F 00 00	
Total 1 Normaliza	00.000 100 tion facto	.0000 r = 2.7339				

 Table E1-1.
 Chemical Compositions for T5D30SuspAl~Granul.jpg, Figure E1-4



Figure E1-5. SEM image magnified 1000 times for a Test #5, Day-30 unsubmerged aluminum coupon sample. (T5D30SuspAl009.bmp)



Figure E1-6. SEM image magnified 250 times for a Test #5, Day-30 submerged aluminum coupon sample. (T5D30SubmAl010.bmp)



Figure E1-7. SEM image magnified 500 times for a Test #5, Day-30 submerged aluminum coupon sample. (T5D30SubmAl011.bmp)



Figure E1-8. Annotated SEM image magnified 1200 times for a Test #5, Day-30 submerged aluminum coupon sample. (T5D30SubmAl012.bmp)



Figure E1-9. EDS counting spectrum for the dark porous surface (EDS1) of the aluminum coupon shown in Figure E1-8. (T5D30SubmAl~dark01.jpg)

The results from the chemical composition analysis for T5D30SubmAl~dark01.jpg are given in Table E1-2.

Group Sample Comment Conditior	: NRC : T5D30 : Dark p I : Full S Live T Acc. V Stage Acq. D	ID# : 7 borous layer c Scale : 20Kev 'ime : 60.0 Nolt : 15.0 Point : X=50. Date : Tue P	on submerge 7(10eV/ch,21 000 sec 5 KV 1 175 Y=52.1 Aug 30 14:0	d Al Kch) Aperture # Probe Curr 47 Z=11.84 3:41 2005	: 1 ent : 1.006 8	E-09 A
Element	Mode	ROI (KeV)	K-ratio(%) +/-	Net/Backgro	und
Na K	Normal	0.83- 1.28	0.9587	0.0065	710 /	37
Al K	Normal	1.26- 1.78	5.5632	0.0015	6209 /	133
Si K	Normal	1.50- 2.07	2.4170	0.0006	2961 /	364
Ca K	Normal	3.40-4.30	3.2624	0.0073	1925 /	13
Fe K	Normal	6.04-7.40	1.5120	0.0023	402 /	9
СК	Normal	0.11-0.47	50.6476	0.0123	511 /	98
ОК	Normal	0.31 - 0.74	21.2659	0.0113	20061 /	35
Cu K	Normal	7.61-9.28	0.9478	0.0014	138 /	3
		Cł	i_square =	4.7269		
Element h	lass% A	tomic% ZAF	Z	A F		
Na	1.838	1.3545 1.1743	0.9804 1.	1967 1.000	9	
Al	10.097	6.3412 1.1119	0.9887 1.	1261 0.998	7	
Si	4.972	2.9996 1.2602	0.9810 1.	2848 0.999	9	
Ca	5.147	2.1761 0.9665	0.9913 0.9	9757 0.999	2	
Fe	2.971			9962 0.994	.b	
c	23.844 3	3.64V/ V.2884	1 0.9910 0.7		0	
0	49.149 5	02.05/5 1.4159		4334 1.000	0	
cu	1.902	0.5207 1.2014	1.2030 0.3	5572 1.000	•	and a state of the
Total 1 Normaliza	100.000 10 ation fact	00.0000 cor = 1.6323				

 Table E1-2.
 Chemical Compositions for T5D30SubmAl~dark01.jpg, Figure E1-9



Figure E1-10. EDS counting spectrum for the light powder (EDS2) on the aluminum coupon surface shown in Figure E1-8. (T5D30SubmAl~light02.jpg)

The results from the chemical composition analysis for T5D30SubmAl~light02.jpg are given in Table E1-3.

Group Sample Comment Conditio	: NRC : T5D30 : Brigh on : Full Live Acc. Stage Acq.) ID# : 4 st layer o Scale : Time : Volt : Point : Date :	3 20KeV 60.0 15.0 X=46.3 Tue A	merged ; (10eV/cl 00 sec KV 894 ¥=50 ug 30 14	Al h, 2Kc Pr 0.390 4:12:	h) ertu obe (Z=1: 44 2(re # Curre 1.848 005	: 1 nt : 1	. 006E-1	09 A
Element	Mode	POT (1	(AN)	K-ratio	a (%)	+1-	M	et /Bacl	aroun	a
Na K	Normal	0 83-	1 28	0 92	22	0 00	67	683	/	43
ALK	Normal	1.26-	1.78	6.22	83	0.00	15	6951	1	132
Si K	Normal	1.50-	2.07	2.31	39	0.00	06	2834	1	415
Ca K	Normal	3.40-	4.30	3.65	87	0.00	78	2159	1	22
ОК	Normal	0.31-	0.74	27.07	11	0.012	28	25537	7	34
СК	Normal	0.11-	0.47	74.52	73	0.014	49	752	1	144
Fe K	Normal	6.04-	7.40	1.12	69	0.002	22	299	1	4
Cu K	Normal	7.61-	9.28	2.36	61	0.00	16	345	1	3
			Ch	i_squar	e =	5.71	09			
Element	Mass%	Atomic%	ZAF	Z		A	F			
Na	1.431	1.0376	1.2008	0.9788	1.22	56 1.	.0010			
Al	9.046	5.5894	1.1241	0.9871	1.13	99 0.	.9990			
Si	3.757	2.2298	1.2564	0.9794	1.28	30 0.	. 9999			
Ca	4.554	1.8942	0.9633	0.9899	0.97	37 0	.9994			
0	49.656	51.7418	1.4196	0.9861	1.43	95 1	. 0000			
С	25.910	35.9628	0.2691	0.9893	0.27	20 1	.0000			
Fe	1.738	0.5188	1.1936	1.2136	0.99	54 0	.9881			
Cu	3.909	1.0256	1.2785	1.2835	0.99	62 1	. 0000			
Total Normaliz	100.000 1 ation fac	.00.0000 tor = 1	. 2921							

 Table E1-3.
 Chemical Compositions for T5D30SubmAl~light02.jpg, Figure E1-9

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Appendix E2

SEM/EDS Data for Test #5, Day-30 Copper Coupons

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	copper coupon sample. (T5D30SuspCu017.bmp)E2-5
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Table E2-4.	Chemical Compositions for T5D30Subm Cu particle10.jpg, Figure	
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This appendix shows the SEM/EDS results for the metal copper coupons under two categories: (1) unsubmerged and (2) submerged. Unsubmerged coupons were contacted with the solution only during the initial 4-hour spray phase. In addition, the surface of the unsubmerged coupons may be affected during the test by the moist air in the tank gas space. Submerged refers to the coupons submerged in the solution during the test.

The coupon samples were collected on August 25, 2005 (the date Test #5 was shut down), and examined by SEM/EDS on August 30 and September 6, 2005. The copper coupon samples were dried in air before being coated with Au/Pd for SEM examination. SEM results present the surface condition of the copper coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products.

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Transcribed Laboratory Log

Laboratory session from September 6, 2005. SEM Test #5, Day-30 Copper Coupons

Conditions: e=15.0kV, WD=11mm



1--Yellow Deposits on Submerged Rack 2--Sediment (T5D30) 4--Al-Submerged 7--Cu Unsubmerged 10--Steel-Submerged

5--Gal-Steel Unsubmerged 8--Cu-Submerged 11--Drain Collar Interior

3--Al-Unsubmerged 6--Gal-Steel Submerged 9--Steel-Unsubmerged 12--Drain Collar Outside Ext.

Unsubmerged Copper Coupons

Image:	T5D30SuspCu017	$100 \times$	SEM image	Figure E2-1
	T5D30SuspCu018	$1000 \times$	SEM Annotated image higher magnification	Figure E2-2
EDS:	T5D30SuspCu particle07		On light particles shown in 018	Figure E2-3
	T5D30SuspCu Surface08		On coupon surface shown in 018	Figure E2-4

Submerged Copper Coupon

Image:	T5D30SubmCu019	$100 \times$	SEM image of fiberglass	Figure E2-5
	T5D30SubmCu020	1000 ×	SEM image higher magnification	Figure E2-6
EDS:	T5D30Subm Cu surface09		EDS of smooth particles in 020	Figure E2-7
	T5D30Subm Cu particle10		EDS of white particles in 020	Figure E2-8



Figure E2-1. SEM image magnified 100 times for a Test #5, Day-30 unsubmerged copper coupon sample. (T5D30SuspCu017.bmp)



Figure E2-2. Annotated SEM image magnified 1000 times for a Test #5, Day-30 unsubmerged copper coupon sample. (T5D30SuspCu018.bmp)



Figure E2-3. EDS counting spectrum for the light particle (EDS1) on the copper coupon surface shown in Figure E2-2. (T5D30Susp Cu particle07.jpg)

The results from the chemical composition analysis for T5D30Susp Cu particle07.jpg are given in Table E2-1.

Group Sample Comment Conditio	: NRC : T5D30 : Parti n : Full Live Acc. Stage Acq.	ID# : 13 cles on suspe Scale : 20Ke Time : 60. Volt : 15.0 Point : X=16 Date : Tue	nded Cu V(10eV/ch,2 000 sec KV .143 Y=53.1 Aug 30 15:0	Kch) Aperture Probe Cur 49 Z=11.8 4:14 2005	# : 1 rent : 1.0051 48	5-09 A
Element	Mode	ROT (KeW)	K-ratio(%	1 +/-	Net/Backgrou	md
Na K	Normal	0.83 - 1.28	2.3307	0.0092	1724 Z	64
Al K	Normal	1.26-1.78	5.5249	0.0015	6160 /	395
Si K	Normal	1.50- 2.07	9.9056	0.0011	12122 /	391
Ca K	Normal	3.40- 4.30	1.8982	0.0067	1119 /	16
Fe K	Normal	6.04-7.40	0.8441	0.0021	224 /	9
Cu K	Normal	7.61-9.28	5.2252	0.0023	760 /	7
ОК	Normal	0.31- 0.74	28.9157	0.0133	27250 /	37
СК	Normal	0.11- 0.47	22.6341	0.0128	228 /	155
		c	hi_square =	4.3581		
Element	Mass%	Atomic% ZAF	Z	A	P	
Na	3.761	3.1269 1.244	5 0.9775 1.	2722 1.00	08	
Al	8.435	5.9747 1.177	3 0.9854 1.	1993 0.99	62	
Si	16.555	11.2653 1.288	8 0.9775 1.	3185 0.99	99	
Ca	2.419	1.1534 0.982	7 0.9864 0.	9968 0.99	94	
Fe	1.290	0.4413 1.178	1 1.2064 0.	9981 0.97	83	
Cu	8.607	2.5887 1.270	1 1.2739 0.	9971 1.00	00	
0	46.196	55.1835 1.232	0 0.9852 1.	2504 1.00	00	
С	12.737	20.2662 0.433	9 0.9886 0.	4390 1.00	00	
Total Normaliz	100.000 1 ation fac	00.0000 tor = 1.2968				

 Table E2-1.
 Chemical Compositions for T5D30Susp Cu particle07.jpg, Figure E2-3



Figure E2-4. EDS counting spectrum for the copper coupon surface (EDS2) shown in Figure E2-2. (T5D30Susp Cu Surface08.jpg)

The results from the chemical composition analysis for T5D30Susp Cu Surface08.jpg are given in Table E2-2.

Group Sample Comment Conditio	: NRC : T5D30 I : Surface on : Full Sca Live Tim Acc. Vol Stage Po Acq. Dat	D# : 14 of suspende le : 20KeV e : 60.0 t : 15.0 int : X=16. e : Tue A	d Cu (10eV/ch,2K 00 sec A KV P 143 Y=53.14 ug 30 15:09	ch) perture # robe Curre 9 Z=11.848 :59 2005	: 1 ent : 1.005E-0	9 A
Element	Mode	ROI(KeV)	K-ratio(%)	+/- 1	let/Background	
Cu K	Normal	7.61-9.28	37.9341	0.0051	5518 /	9
0 K	Normal	0.31- 0.74	9.8150	0.0087	9250 /	52
СК	Normal	0.11- 0.47	26.8105	0.0098	270 /	60
		Ch	i_square =	2.8337		
Element	Mass% Ato	mic% ZAF	Z	A F		
Cu	67.862 31.	6586 1.1006	1.1031 0.9	978 1.0000)	
0	17.836 33.	0455 1.1180	0.8760 1.2	764 0.9999		
С	14.302 35.	2958 0.3282	0.8796 0.3	731 1.0000)	
Total Normali:	100.000 100. zation factor	0000 = 1.6254				

Table E2-2. Chemical Compositions for T5D30Susp Cu Surface08.jpg, Figure E2-4



Figure E2-5. SEM image magnified 100 times for a Test #5, Day-30 submerged copper coupon sample. (T5D30SubmCu019.bmp)



Figure E2-6. SEM image magnified 1000 times for a Test #5, Day-30 submerged copper coupon sample. (T5D30SubmCu020.bmp)



Figure E2-7. EDS counting spectrum for the grey copper coupon surface (EDS3) shown in Figure E2-6. (T5D30Subm Cu surface09.jpg)

The results from the chemical composition analysis for T5D30Subm Cu surface09.jpg are given in Table E2-3.

Group Sample Comment Conditio	: NRC : T5D3 : Surf. on : Full Live Acc. Stag Acq.	0 ID# : 15 ace of submerge Scale : 20KeV Time : 60.0 Volt : 15.0 e Point : X=11 Date : Tue A	ed Cu 7(10eV/ch 000 sec KV 640 Y=63 Aug 30 15	,2Kch) Aperture Probe Cu .166 Z=11.5 :20:19 200	# : 1 crent : 1.005 348 5	Е-09 А
Flowowt	Mode	DOT (KOH)	V-ratio	(2) 11	Not /Dackers	und
LI CHIERIC	Norma	1 196 - 179	n-Latio	$\binom{3}{2}$ 0 0010	net/backyro	
GiK	Norma	1 1.20 - 1.70 1 1.50 - 2.07	0 7/3	7 0 0005	910 7	189
CaK	Norma	1 3 40 - 4 30	1 224	1 0 0066	722 /	24
Cu K	Norma	1 7.61 - 9.28	24.415	2 0.0042	3551 /	
ОК	Norma	1 0.31-0.74	15.740	5 0.0101	14834 /	41
СK	Norma	1 0.11- 0.47	37.159	3 0.0132	375 /	85
		cl	ni_square	= 3.4035		
Element	Mass%	Atomic% ZAF	z	A	F	
Al	4.915	4.1748 1.503	0.9203	1.6344 0.9	997	
Si	1.647	1.3440 1.4543	8 0.9126	1.5937 1.0	000	
Ca	1.717	0.9819 0.921	L 0.9183	1.0049 0.9	982	
Cu	43.542	15.7063 1.171:	L 1.1747 (0.9969 1.0	000	
0	29.739	42.6032 1.240	5 0.9212	1.3468 1.0	000	
C	18.441	35.1897 0.3259	0.9247	0.3524 1.0	000	
Total Normaliz	100.000 zation fac	100.0000 ctor = 1.5228				

 Table E2-3.
 Chemical Compositions for T5D30Subm Cu surface09.jpg, Figure E2-7



Figure E2-8. EDS counting spectrum for the light particles (EDS4) on the coupon surface shown in Figure E2-6. (T5D30Subm Cu particle10.jpg)

The results from the chemical composition analysis for T5D30Subm Cu particle10.jpg are given in Table E2-4.

Comment Conditio	: Part: on : Full Live Acc. Stage Acq.	icles on Scale : Time : Volt : Point : Date :	submer 20KeV 60.0 15.0 1 X=11. Tue A	ged Cu (10eV/cl 00 sec KV 640 Y=6: ug 30 1!	1,2Kch) Aper Prob 3.166 Z 5:24:43	ture # e Curre =11.848 2005	: 1 nt : 1.0	05E-09 A
Element	Mode	ROI	KeV)	K-ratio	b (%) +	/- N	et/Backg	round
Al K	Norma	1.26-	1.78	2.65	38 0.	0011	2964 /	85
Si K	Normal	l 1.50-	2.07	1.019	95 0.	0005	1248 /	247
Ca K	Normal	L 3.40-	4.30	2.27	51 0.	0071	1341 /	27
Cu K	Normal	L 7.61-	9.28	11.13	16 0.	0031	1619 /	7
СК	Normal	l 0.11-	0.47	69.282	230.	0160	698 /	88
0 К	Normal	L 0.31-	0.74	15.11	560.	0099	14246 /	38
			Ch	i_squar	e = 4.	8904		
Element	Mass%	Atomic%	ZAF	Z	A	F		
Al	5.525	3.7131	1.2874	0.9559	1.3474	0.9995		
Si	2.175	1.4045	1.3219	0.9482	1.3942	0.9999		
Ca	3.453	1.5625	0.9405	0.9570	0.9839	0.9988		
Cu	22.087	6.3036	1.2293	1.2346	0.9957	1.0000		
С	30.146	45.5152	0.2696	0.9588	0.2812	1.0000		
	36.614	41.5011	1.5007	0.9555	1.5705	1.0000		

 Table E2-4.
 Chemical Compositions for T5D30Subm Cu particle10.jpg, Figure E2-8