Appendix E3

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

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This appendix shows the SEM/EDS results for the metal galvanized steel 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 galvanized steel coupon samples were dried in air before being coated with Au/Pd for SEM examination. SEM results present the surface condition of the galvanized steel 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 Galvanized Steel 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 Galvanized Steel Coupon

Image:	T5D30SuspGalsteel013	$100 \times$	SEM image	Figure E3-1
	T5D30SuspGalsteel014	$1000 \times$	SEM Annotated image	Figure E3-2
EDS:	T5D30Susp GS needle03		On needle like crystal shown in image 014	Figure E3-3
	T5D30 Susp GS layer04		On cracked skin layer shown in image 014	Figure E3-4

Submerged Galvanized Steel Coupon

Image:	T5D30SubmGalsteel015	$100 \times$	SEM image of fiberglass	Figure E3-5
	T5D30SubmGalsteel016	1000 ×	SEM image higher magnification	Figure E3-6
EDS:	T5D30Subm GS Particle05		On particles shown in image 016	Figure E3-7
	T5D30Subm GS Particle05		On particles shown in image 016	Figure E3-8



Figure E3-1. SEM image magnified 100 times for a Test #5, Day-30 unsubmerged galvanized steel coupon sample. (T5D30SuspGalsteel013.bmp)



Figure E3-2. Annotated SEM image magnified 1000 times for a Test #5, Day-30 unsubmerged galvanized steel coupon sample. (T5D30SuspGalsteel014.bmp)



Figure E3-3. EDS counting spectrum for the needle-like crystal (EDS1) on the unsubmerged, galvanized steel coupon surface shown in Figure E3-2. (T5D30Susp GS needle 03.jpg)

The results from the chemical composition analysis for T5D30Susp GS needle 03.jpg are given in Table E3-1.

Group Sample Comment Condition	: NRC : T5D30 : Needle : Full Sc Live Ti Acc. Vo Stage P Acq. Da	ID# : 9 crystal on s ale : 20KeV me : 60.0 lt : 15.0 oint : X=44. te : Tue A	uspended Ga (10eV/ch,2K 00 sec A KV P 601 Y=63.87 ug 30 14:27	l-steel ch) perture = robe Cur 5 Z=11.8 :56 2005	# : 1 rent : 1.005F 48	:-09 A
Flowont	Mode	DOT (Koll)	K matio (%)		Not /Dackgrou	md
Zn K	Normal	8 10_10 0G	20 0055	0 0087	2501 /	uiu E
	Normal	0.13 - 10.00 0.31 - 0.74	30.3333	0 0125	22808 /	40
СК	Normal	0.11 - 0.47	29.4584	0.0106	297 /	123
Element Ma Zn 4 0 3 C 1 Total 10 Normalizat	ss% At 8.925 17 6.525 53 4.550 28 0.000 100 ion facto	Ch omic% ZAF .6401 1.1674 .8072 1.1161 .5527 0.3653 .0000 r = 1.3522	i_square = Z 1.1710 0.9 0.9160 1.2 0.9195 0.3	7.1611 969 1.00 184 1.00 973 1.00	F 00 00 00	

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Figure E3-4. EDS counting spectrum for the cracked layer (EDS2) on the unsubmerged galvanized steel coupon surface shown in Figure E3-2. (T5D30 Susp GS layer 04.jpg)

The results from the chemical composition analysis for T5D30 Susp GS layer 04.jpg are given in Table E3-2.

Group : NRC Sample : T5D30 ID# : 10 Comment : Cracked layer on suspended Gal-steel Condition : Full Scale : 20KeV(10eV/ch,2Kch) Live Time : 60.000 sec Aperture # : 1 Acc. Volt : 15.0 KV Probe Current : 1.006E-09 A Stage Point : X=44.601 Y=63.875 Z=11.848 Acq. Date : Tue Aug 30 14:32:43 2005						
Flowont	Mode	POT (Koy)	K-ratio(%)	+ / - N	at /Backgroup	h
Zn K	Normal	8.19-10.06	27.9430	0.0081	3177 /	т б
СК	Normal	0.11-0.47	29.1554	0.0102	294 /	100
ОК	Normal	0.31- 0.74	19.0550	0.0110	17975 /	35
		Ch	i_square =	4.1819		
Element Ma	ss% At	omic% ZAF	Z	A F		
Zn 4	9.823 17	.9813 1.1635	1.1671 0.9	970 1.0000		
	6.397 32	.2068 0.3670	0.9167 0.4	003 1.0000		
	3.700 49	.0119 1.1300	0.9133 1.2	667 I.0000		
Total 10 Normalizat	0.000 100 ion facto	.0000 r = 1.5324				

 Table E3-2.
 Chemical Compositions for T5D30 Susp GS layer 04.jpg, Figure E3-4



Figure E3-5. SEM image magnified 100 times for a Test #5, Day-30 submerged galvanized steel coupon sample. (T5D30SubmGalsteel015.bmp)



Figure E3-6. Annotated SEM image magnified 1000 times for a Test #5, Day-30 submerged galvanized steel coupon sample. (T5D30SubmGalsteel016.bmp)



Figure E3-7. EDS counting spectrum for the light deposits (EDS3) on the submerged galvanized steel coupon surface shown in Figure E3-6. (T5D30Subm GS Particle05.jpg)

The results from the chemical composition analysis for T5D30Subm GS Particle05.jpg are given in Table E3-3.

<pre>Group : NRC Sample : T5D30 ID# : 11 Comment : Particles on submerged Gal_steel surface Condition : Full Scale : 20KeV(10eV/ch,2Kch) Live Time : 60.000 sec Aperture # : 1 Acc. Volt : 15.0 KV Probe Current : 1.005E-09 A Stage Point : X=56.678 Y=62.490 Z=11.848 Acq. Date : Tue Aug 30 14:47:04 2005</pre>						
Flowopt	Mode	DOT (Kou)	K-ratio(%)) +/-	Not /Backgro	und
Alk	Mormal	1.26 - 1.78	n 9951	0 0008	1100 /	70
SiK	Normal	1.20 - 1.70 1.50 - 2.07	0 9148	0 0005	1119 /	125
CaK	Normal	3 40- 4 30	0 4234	0 0049	250 /	25
Zn K	Normal	8 19-10 06	17 3542	0.0070	1971 /	7
СК	Normal	0 11 - 0 47	19 5680	0 0121	197 /	121
οĸ	Normal	0.31- 0.74	25.0700	0.0122	23626 /	28
		Ch	i_square =	5.5084		
Element	Mass%	Atomic% ZAF	Z	A	5	
Al	2.628	2.0964 1.5366	0.9406 1.	6342 0.99	96	
Si	2.296	1.7594 1.4602	0.9328 1.	5654 1.00	00	
Ca	0.685	0.3677 0.9409	0.9394 1.0	0025 0.99	91	
Zn	36.016	11.8565 1.2072	1.2114 0.9	9965 1.00	00	
С	12.092	21.6651 0.3595	0.9447 0.3	3805 1.00	00	
0	46.283	62.2547 1.0739	0.9412 1.	1410 1.00	00	
Total Normali:	100.000 1 zation fac	.00.0000 tor = 1.7191				

 Table E3-3.
 Chemical Compositions for T5D30Subm GS Particle05.jpg, Figure E3-7



Figure E3-8. EDS counting spectrum for the grey coupon surface (EDS4) of the submerged galvanized steel shown in Figure E3-6. (T5D30Subm GS Surface06.jpg)

The results from the chemical composition analysis for T5D30Subm GS Surface06.jpg are given in Table E3-4.

Group Sample Comment Conditio	: NRC : T5D30 : Surfac n : Full S Live T Acc. V Stage Acq. D	ID# : 12 e of submerge cale : 20KeV ime : 60.0 olt : 15.0 Point : X=56 ate : Tue 1	ed Gal_ste V(10eV/ch, D00 sec KV .678 ¥=62. Aug 30 14:	el 2Kch) Aperture Probe Cur 490 Z=11.8 52:18 2005	# : 1 rent : 1.006E 48	-09 A
Floment	Mode	DOT (KOV)	K-ratio(s) +/-	Net /Backgrou	md
V] K	Normal	1.26 - 1.78	n 4278	ໍ່ ດີດດາງ	A77 /	58
SiK	Normal	150 - 207	0 1268	0 0004	155 7	91
Zn K	Normal	8.19-10.06	45.4077	0.0105	5163 /	10
OK	Normal	0.31-0.74	2.7433	0.0065	2588 /	58
СК	Normal	0.11- 0.47	21.6706	0.0104	219 /	36
		Cl	ni_square	= 1.5563		
Element	Mass% A	tomic% ZAF	z	А	F	
Al	1.263	1.6261 1.8551	1 0.8522 2	.1770 1.00	00	
Si	0.331	0.4087 1.6373	2 0.8446 1	.9384 1.00	00	
$\mathbf{Z}\mathbf{n}$	77.389 4	1.1116 1.070	5 1.0718 0	.9988 1.00	00	
0	5.427 1	1.7800 1.242	6 0.8547 1	.4540 0.99	99	
С	15.590 4	5.0735 0.4519	9 0.8583 0	.5265 1.00	00	
Total Normaliz	100.000 10 ation fact	0.0000 or = 1.5921				

 Table E3-4.
 Chemical Compositions for T5D30Subm GS Surface06.jpg, Figure E3-8

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Appendix E4

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

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This appendix shows the SEM/EDS results for the metal steel 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 steel coupon samples were dried in air before being coated with Au/Pd for SEM examination. SEM results present the surface condition of the steel 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 Steel 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 Steel Coupon

Image:	T5D30SuspSteel021	$100 \times$	SEM image	Figure E4-1
	T5D30SuspSteel022	$1000 \times$	SEM image higher magnification	Figure E4-2
EDS:	T5D30SuspSteel023	$1000 \times$	SEM annotated image	Figure E4-3
	T5D30Susp Steel Surface11		On smooth surface shown in image 023	Figure E4-4
	T5D30Susp Steel Particle12		On white particles shown in image 023	Figure E4-5

Submerged Steel Coupon

Image:	T5D30SubmSteel024	$100 \times$	SEM image of fiberglass	Figure E4-6
	T5D30SubmSteel025	$1000 \times$	SEM Annotated image	Figure E4-7
EDS:	T5D30Subm Steel light particle13		EDS of light particles in 025	Figure E4-8
	T5D30Subm Steel dark particle14		EDS of dark particle in 025	Figure E4-9
	T5D30Subm Steel Surface15		EDS of surface in 025	Figure E4-10



Figure E4-1. SEM image magnified 100 times for a Test #5, Day-30 unsubmerged steel coupon sample. (T5D30SuspSteel021.bmp)



Figure E4-2. SEM image magnified 1000 times for a Test #5, Day-30 unsubmerged steel coupon sample. (T5D30SuspSteel022.bmp)



Figure E4-3. Annotated SEM image magnified 1000 times for a Test #5, Day-30 unsubmerged steel coupon sample. (T5D30SuspSteel023.bmp)



Figure E4-4. EDS counting spectrum for the dark coupon surface (EDS1) of the unsubmerged steel shown in Figure E4-3. (T5D30Susp Steel Surface11.jpg)

The results from the chemical composition analysis for T5D30Susp Steel Surface11.jpg are given in Table E4-1.

Group Sample Comment Conditio	: NRC : T5D30 : Surfac on : Full S Live T Acc. V Stage Acq. D	ID# : 17 e of Suspende cale : 20KeV ime : 60.0 olt : 15.0 Point : X=21. ate : Tue A	d Steel (10eV/ch,2F 00 sec 7 KV 7 743 Y=66.86 ug 30 15:38	(ch) Aperture Probe Cur 57 Z=11.8 3:16 2005	# : 1 rent : 1.004E-0 48	9 A
Element Fe K C K	Mode Normal Normal	ROI(KeV) 6.04-7.40 0.11-0.47	K-ratio(%) 57.1720 25.4488	+/- 0.0094 0.0098	Net/Background 15164 / 256 /	18 25
Element Fe c	Mass% A 89.763 6 10 237 3	Ch tomic% ZAF 5.3492 1.0215 4 6509 0 2617	i_square = Z 1.0227 0.9 0.8592 0.3	3.9338 A 1989 1.00	F 00 00	
Total Normali:	100.000 10 zation fact	0.0000 or = 1.5370				

 Table E4-1.
 Chemical Compositions for T5D30Susp Steel Surface11.jpg, Figure E4-4



Figure E4-5. EDS counting spectrum for the light particle (EDS2) on the unsubmerged steel coupon surface shown in Figure E4-3. (T5D30Susp Steel Particle12.jpg)

The results from the chemical composition analysis for T5D30Susp Steel Particle12.jpg are given in Table E4-2.

Group Sample Comment Condition	: NRC : T5D30 ID# : : Particles on : Full Scale : Live Time : Acc. Volt : Stage Point : Acq. Date :	18 Suspended Ste 20KeV(10eV/c 60.000 sec 15.0 KV X=21.743 Y=6 Tue Aug 30 1	el h,2Kch) Aperture Probe Cur 6.867 Z=11.8 5:43:38 2005	# : 1 rent : 1.005E-09 48	9 A
Flomont	Modo DOT (Kou) K-rati	A(8) +1-	Not /Paakground	
Ci K	Normal 1 50-	0 17 0 10		108 /	47
FAK	Normal 6.04-	7 40 29 43	50 0.0000	7815 /	13
CK	Normal 0.11-	. 0 47 15 95	59 0.0076	161 /	25
οĸ	Normal 0.31-	0.74 6.15	37 0.0065	5799 /	21
		Chi_squar	e = 2.8510		
Element Ma	ss% Atomic%	ZAF Z	A	F I	
Si	0.348 0.4060	1.3604 0.8714	1.5612 0.99	gg	
Fe 7	6.176 44.6485	1.0562 1.0587	0.9976 1.00	00	
с 1	0.179 27.7411	0.2604 0.8849	0.2942 1.00	00	
0 1	3.297 27.2045	0.8818 0.8813	1.0011 0.99	95	
Total 10 Normalizat	0.000 100.0000 ion factor = 2	. 4503			

Table E4-2.	Chemical Co	mpositions for	T5D30Sus	o Steel Particle12.jpg	Figure E4-5
	Christian Co		102000000		,



Figure E4-6. SEM image magnified 100 times for a Test #5, Day-30 submerged steel coupon sample. (T5D30SubmSteel024.bmp)



Figure E4-7. Annotated SEM image magnified 1000 times for a Test #5, Day-30 submerged steel coupon sample. (T5D30SubmSteel025.bmp)



Figure E4-8. EDS counting spectrum for the light particle (EDS1) on the submerged steel coupon surface shown in Figure E4-7. (T5D30Subm Steel light particle13.JPG)

The results from the chemical composition analysis for T5D30Subm Steel light particle13.JPG are given in Table E4-3.

Table E4-3.	Chemical Compositions for T5D30Subm Steel light particle13.JPG,
	Figure E4-8

Group Sample Comment Conditic	: NRC : T5D3(: Light on : Full Live Acc. Stage Acq.) ID# : ; particl Scale : Time : Volt : Point : Date :	19 e on su 20KeV 60.00 15.0 1 X=76.4 Tue Au	ubmerge (10eV/cl 00 sec KV 455 Y=54 ug 30 10	i Steel 1,2Kch) Apei Prok 1.980 2 5:26:04	ture # be Curr 3=11.00 1 2005	: : 1 cent : 1. 00	0031	5-09 A
Element	Mode	POT (KeV)	K-rati/	v(%) -	7-	Net/Back	aro	md
C K	Normal	0 11 -	0 47	255 63	11 0	0237	2571	7	ала д д
Ő K	Normal	0.35-	0.72	14.192	v3 0.	0039	3803	1	62
Na K	Normal	0.83-	1.28	1.250	01 0.	0063	923	1	34
Al K	Normal	1.26-	1.78	1.15	29 0.	0008	1283	1	66
Si K	Normal	1.50-	2.07	0.633	34 0.	0009	704	7	119
Cl K	Normal	2.32-	3.09	0.513	37 0.	0007	485	7	36
Ca K	Normal	3.40-	4.30	0.810	03 0.	0053	477	1	27
Fe K	Normal	6.04-	7.40	15.780	380.	0051	4184	7	6
			Ch	i_square	ə = 3.	2813			
Element	Mass%	Atomic%	ZAF	Z	A				
С	49.124	65.0147	0.1870	0.9620	0.1944	1.000	00		
0	26.855	26.6826	1.8416	0.9574	1.9235	5 0.999	19		
Na	1.678	1.1603	1.3065	0.9520	1.3704	1.001	15		
Al	1.365	0.8042	1.1523	0.9602	1.2004	0.999	18		
Si	0.695	0.3932	1.0673	0.9487	1.1251	L 0.999	19		
C1	0.522	0.2339	0.9882	1.0049	0.9842	0.999	1		
Ca	0.770	0.3053	0.9247	0.9632	0.9661	L 0.993	17		
Fe	18.991	5.4057	1.1706	1.1807	0.991	5 1.000	00		
Total Normaliz	100.000 1 ation fac	100.0000 tor = 1	.0275						



Figure E4-9. EDS counting spectrum for the dark particle (EDS2) on the submerged steel coupon surface shown in Figure E4-7. (T5D30Subm Steel dark particle14.JPG)

The results from the chemical composition analysis for T5D30Subm Steel dark particle14.JPG are given in Table E4-4.

Table E4-4.Chemical Compositions for T5D30Subm Steel dark particle14.JPG,
Figure E4-9

Group Sample Comment Conditio	: NRC : T5D3(: Dark on : Full Live Acc. Stage Acq.) ID# : particle Scale : Time : Volt : Point : Date :	20 e on su 20KeV 191.1 15.0 X=76. Tue A	bmerged (10eV/c) 30 sec KV 455 Y=5 ug 30 1	Steel n,2Kch) Aper Prok 4.980 2 7:08:10	ture # e Curre =11.00 2005	: 1 ent : 1.)	. 003E	-09 A
Flement	Mode	POL	KeW)	K-ratio	v(%) -+	/- 1	let / Back	arou	nd
Na K	Normal	0.83-	- 1 28		13 N	0114	9991	igrou /	116
ALK	Normal	1 26-	- 1 78	0 53	16 0.	0013	1884	4	212
SiK	Norma	1.50-	2.07	0.55	49 0.	0008	2159	1	284
CaK	Norma	3.40-	4.30	1.25	63 0.	0109	2354	1	73
Fe K	Norma	6.04-	- 7.40	24.34	40 0.	0113	20548	1	36
ОК	Norma	0.31-	0.74	33.99	78 0.	0267	101858	1	292
СК	Normal	0.11-	0.47	58.53	53 0.	0260	1876	1	551
			Ch	i_squar	∋ = 20.	3524			
Element	Mass%	Atomic%	ZAF	Z	A	F			
Na	1.969	1.7446	1.6775	0.9412	1.7796	5 1.001	5		
Al	0.872	0.6582	1.3195	0.9486	1.3913	0.999	3		
Si	0.890	0.6453	1.2897	0.9408	1.3709	0.999)		
Ca	1.439	0.7313	0.9214	0.9484	0.9798	0.991	6		
Fe	34.836	12.7034	1.1509	1.1573	0.9945	5 1.000	0		
0	43.074	54.8293	1.0190	0.9488	1.0741	0.999)		
C	16.919	28.6879	0.2325	0.9522	0.2442	0.999	9		
Total Normaliz	100.000 zation fac	100.0000 stor = 1	1.2433						



Figure E4-10. EDS counting spectrum for the submerged steel coupon surface (EDS3) shown in Figure E4-7. (T5D30Subm Steel Surface15.jpg)

The results from the chemical composition analysis for T5D30Subm Steel Surface15.jpg are given in Table E4-5.

Table E4-5.	Chemical Compositions for T	5D30Subm Steel	Surface15.jpg, Figure
	E4-10		

Group Sample Comment Conditio	: NRC : T5D3 : Surf. n : Full Live Acc. Stag Acq.	0 ID# : 21 ace of submer Scale : 20K Time : 83 Volt : 15. e Point : X=7 Date : Tue	ged Steel eV(10eV/ch, .100 sec 0 KV 6.455 Y=54 Aug 30 17	,2Kch) Aperture Probe Cur .980 Z=11.0 :15:50 2005	# : 1 rrent : 1.003 000 5	E-09 A
Element	Mode	ROI(KeV)	K-ratio	(%) +/-	Net/Backgro	und
Na K	Norma	1 0.83-1.2	8 0.6403	0.0069	655 /	72
Fe K	Norma	1 6.04-7.4	0 58.7678	8 0.0113	21567 /	30
Co K	Norma	1 6.53-8.0	0.1879	0.0017	64 /	66
O K	Norma	1 0.31- 0.7	4 5.4760	0 0.0120	7133 /	270
СК	Norma	1 0.11-0.4	7 28.026	0.0128	390 /	86
Al K	Norma	1 1.26- 1.7	8 0.5087	7 0.0009	784 /	108
			Chi_square	= 8.3983		
Element	Mass%	Atomic% ZA	F Z	A	F	
Na	1.768	2.7579 2.09	87 0.8635 2	2.4268 1.00)16	
Fe	80.660	51.7984 1.04	32 1.0450 (0.9983 1.00	000	
Co	0.264	0.1607 1.06	80 1.0710 ($0.9972 \ 1.00$	000	
0	6.395	14.3354 0.88	77 0.8717	1.0189 0.99	94	
C	9.925	29.6345 0.26	92 0.8754 (0.3075 1.00	000	
Al	0.988	1.3132 1.47	61 0.8693	1.6981 1.00	000	
Total Normaliz	100.000 ation fa	100.0000 ctor = 1.315	i6			