Appendix F1

SEM/EDS Data for Test #4, Day-30 Aluminum Coupons

Figures

Tables

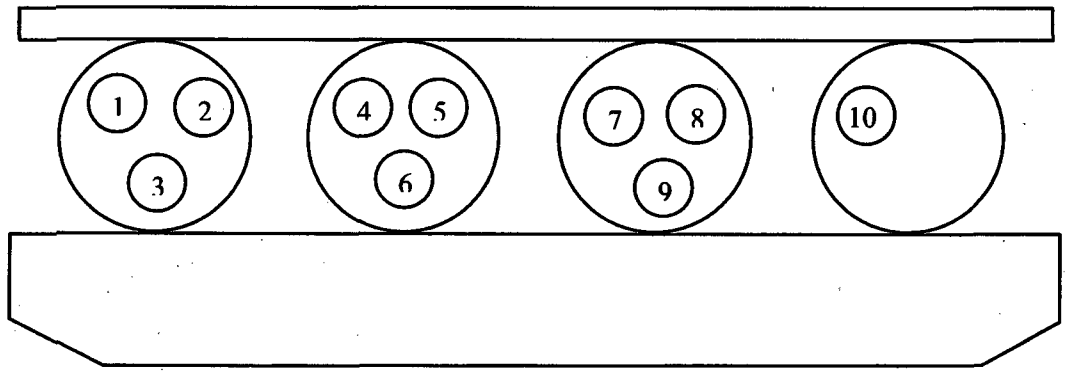
This appendix shows the SEM/EDS results for the metal aluminum coupons under two categories: (1) unsubmerged and (2) submerged. Unsubmerged refers to coupons held in the test tank gas space above the water level of the solution during ICET. Unsubmerged coupons were contacted by the solution only during the 4-hour spraying period at the initial date of the test. In addition, the surface of the unsubmerged coupons may also have been affected by the moisture in the gas space during the test. Submerged refers to the coupons that were under the solution during the test.

The coupon samples were collected on June 23, 2005 (the date Test #4 was shut down) and examined by SEM/EDS on June 29, 2005. The aluminum coupon samples were dried in air before being coated 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.

Transcribed Laboratory Log

Laboratory session from June 29, 2005.
SEM Test #4, Day-30 Aluminum Coupons

1. Unsubmerged Al	3. Sus. Cu	5. Sus. Gal-Steel	7. Sus. Steel
2. Submerged Al	4. Sub. Cu	6. Sub. Gal Steel	8. Sub. Steel
9. Sediment	10. Powder on Sub. Rack		



Unsubmerged Aluminum Coupons

Image:	T4D30AlSusp001	100 ×	SEM image	Figure F1-1
	T4D30AlSusp002	500 ×	SEM image higher magnification	Figure F1-2
	T4D30AlSusp003	1000 ×	SEM annotated image	Figure F1-3
EDS:	T4D30AlSusp01		On particles at Al surface shown in image T4D30AlSusp003	Figure F1-4
	T4D30AlSusp02		On Al coupon surface shown in image T4D30AlSusp003	Figure F1-5

Submerged Al Coupon

Image:	T4D30AlSubm004	100 ×	SEM image of fiberglass	Figure F1-6
	T4D30AlSubm005	500 ×	SEM image higher magnification	Figure F1-7
	T4D30AlSubm006	1000 ×	SEM annotated image	Figure F1-8
EDS:	T4D30AlSubm03		EDS of particles shown in 006	Figure F1-9
	T4D30Alsubm04		EDS of Al surface in 006	Figure F1-10
Image:	T4D30AlSubm007	5000 ×	SEM image higher magnification	Figure F1-11

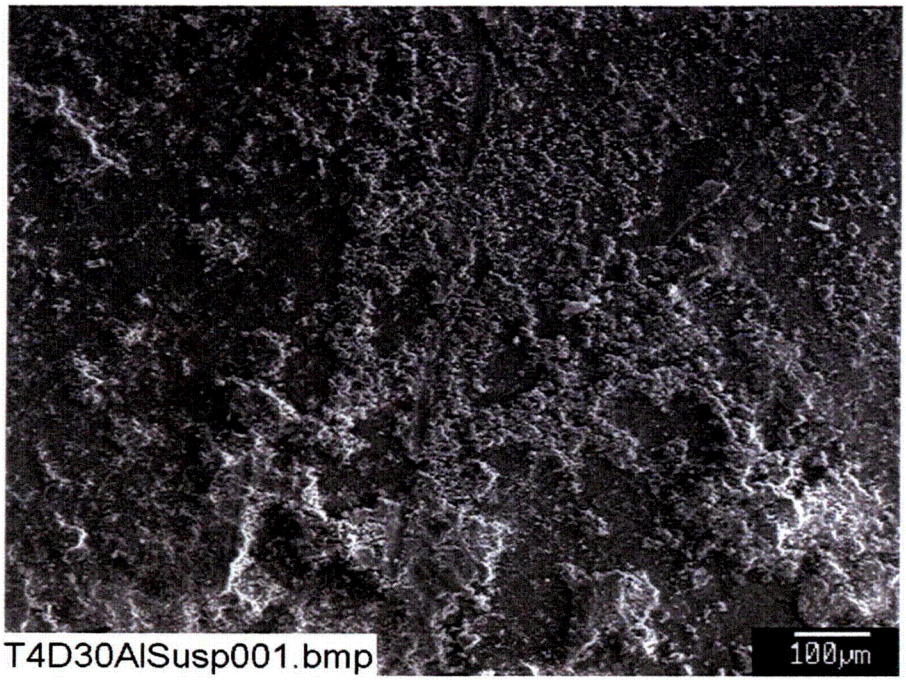


Figure F1-1. **SEM image magnified 100 times for a Test #4, Day-30 unsubmerged aluminum coupon sample. (T4D30AlSusp001.bmp)**

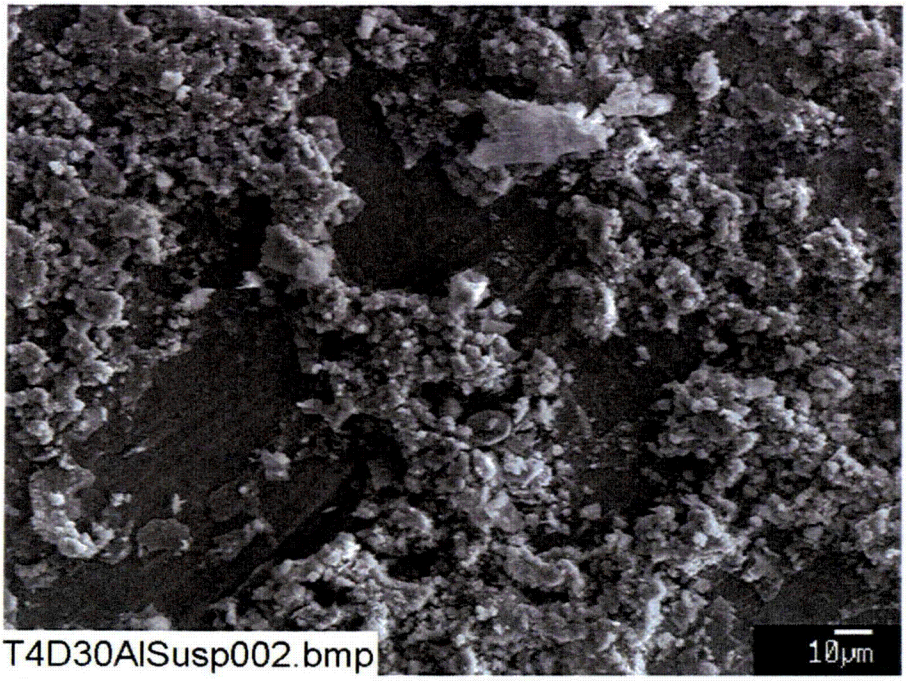


Figure F1-2. **SEM image magnified 500 times for a Test #4, Day-30 unsubmerged aluminum coupon sample. (T4D30AlSusp002.bmp)**

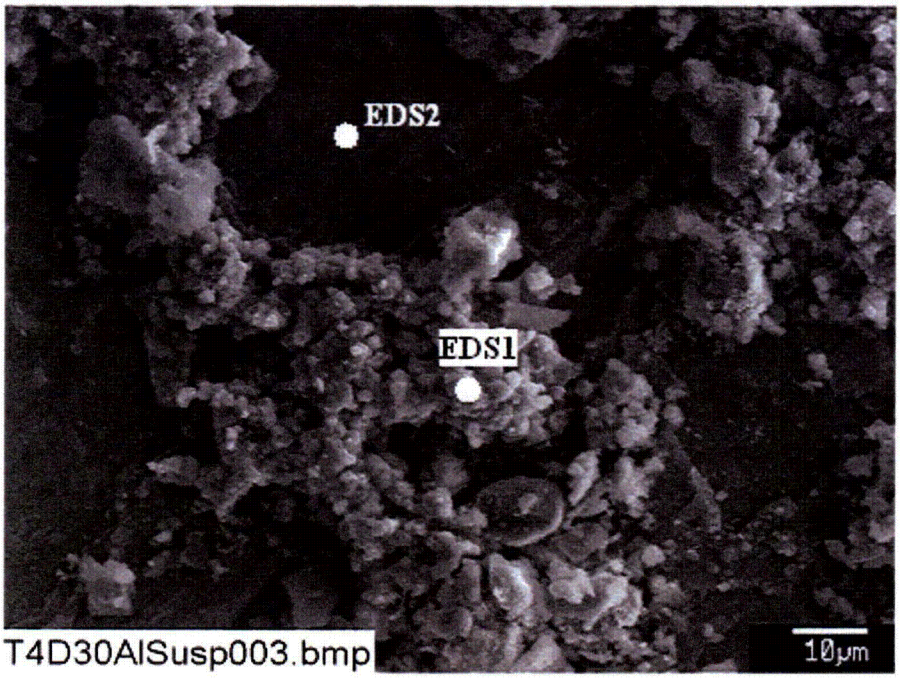


Figure F1-3. **Annotated SEM image magnified 1000 times for a Test #4, Day-30 unsubmerged aluminum coupon sample. (T4D30AlSusp003.bmp)**

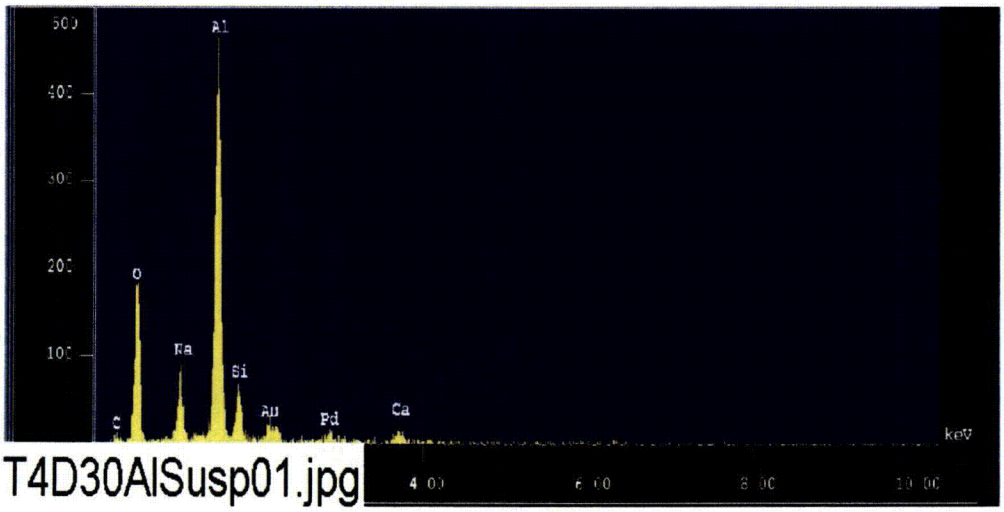


Figure F1-4. **EDS counting spectrum for the deposits (EDS1) on the coupon surface shown in Figure F1-3. (T4D30AlSusp01.jpg)**

The results from the chemical composition analysis for T4D30AlSusp01.jpg are given in Table F1-1.

Table F1-1. Chemical Compositions for T4D30AlSusp01.jpg, Figure F1-4

Jun 29 10:11 2005

Group : NRC
Sample : T4D30 ID# : 1
Comment : Particle on suspended Al
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.069E-09 A
Stage Point : X=86.836 Y=58.400 Z=10.786
Acq. Date : Wed Jun 29 10:06:34 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	49.7713	0.0017	1653 / 8
Na K	Normal	0.81- 1.27	6.5744	0.0007	627 / 6
Al K	Normal	1.26- 1.78	29.7835	0.0010	4103 / 29
Si K	Normal	1.50- 2.07	3.1103	0.0004	400 / 238
Ca K	Normal	3.40- 4.30	1.8580	0.0027	116 / 2
C K	Normal	0.09- 0.46	0.1137	0.0001	6 / 16

Chi_square = 3.1919

Element	Mass%	Atomic%	ZAF	Z	A	F
O	47.921	60.0922	0.9108	0.9888	0.9212	0.9999
Na	8.495	7.4130	1.2222	1.0435	1.1736	0.9980
Al	35.757	26.5870	1.1356	1.0048	1.1313	0.9990
Si	5.079	3.6282	1.5447	0.9930	1.5557	1.0000
Ca	1.975	0.9887	1.0056	1.0019	1.0036	1.0001
C	0.773	1.2909	6.4296	1.0368	6.2020	1.0000
Total	100.000	100.0000				

Normalization factor = 1.0572

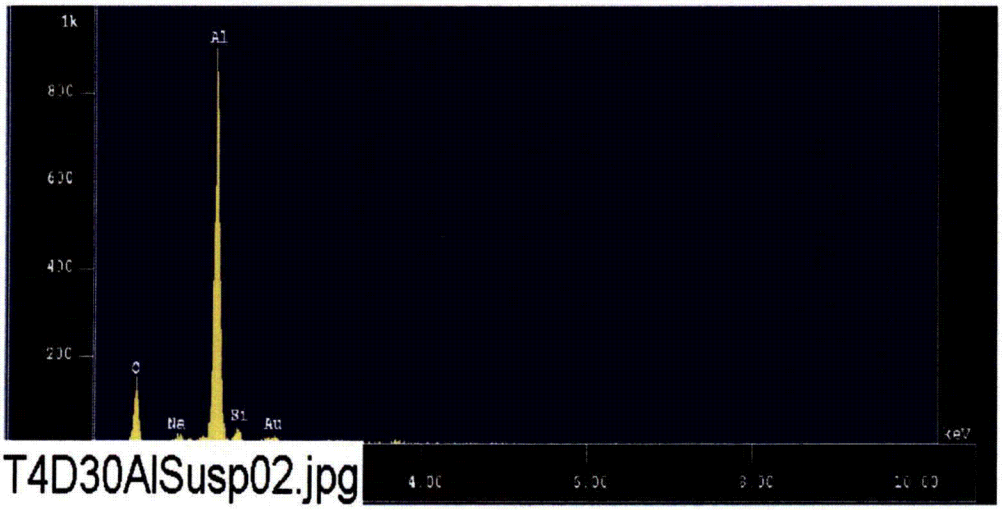


Figure F1-5. **EDS counting spectrum for the flat coupon surface (EDS2) shown in Figure F1-3. (T4D30AlSusp02.jpg)**

The results from the chemical composition analysis for T4D30AlSusp02.jpg are given in Table F1-2.

Table F1-2. Chemical Compositions for T4D30AlSusp02.jpg, Figure F1-5

Jun 29 10:15 2005

Group : NRC
Sample : T4D30 ID# : 2
Comment : Surface of suspended Al
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.068E-09 A
Stage Point : X=86.836 Y=58.400 Z=10.786
Acq. Date : Wed Jun 29 10:13:59 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	36.6938	0.0015	1217 / 4
Na K	Normal	0.81- 1.27	1.3775	0.0004	131 / 8
Al K	Normal	1.26- 1.78	57.7086	0.0014	7943 / 24
Si K	Normal	1.50- 2.07	1.7543	0.0003	225 / 430

Chi_square = 4.6847

Element	Mass%	Atomic%	ZAF	Z	A	F
O	36.111	48.7449	0.9776	0.9856	0.9919	0.9999
Na	1.540	1.4468	1.1106	1.0400	1.0727	0.9956
Al	59.291	47.4571	1.0206	1.0013	1.0198	0.9995
Si	3.058	2.3512	1.7314	0.9893	1.7500	1.0000
Total	100.000	100.0000				

Normalization factor = 1.0067

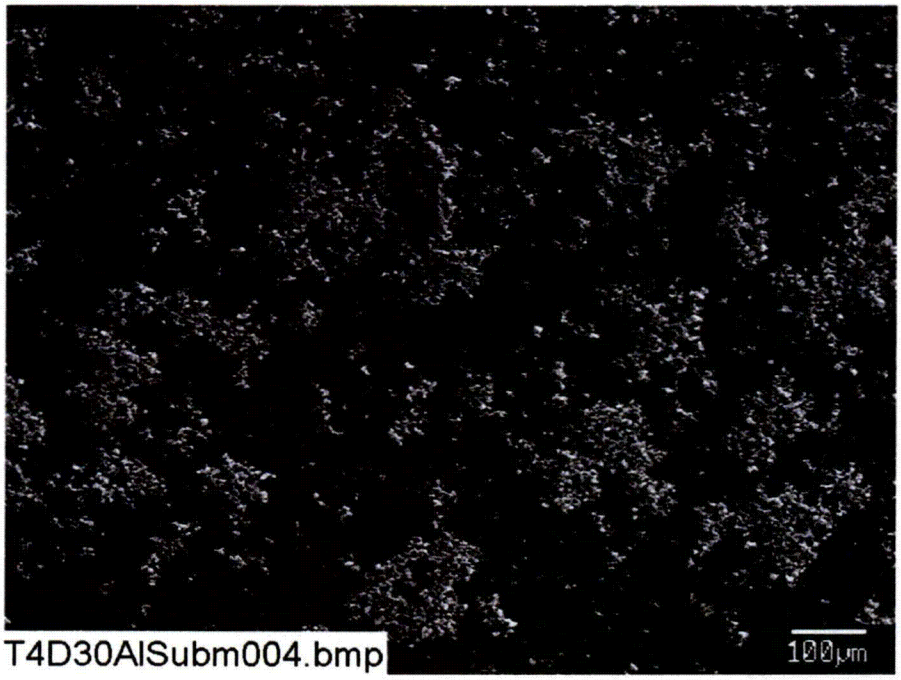


Figure F1-6. **SEM image magnified 100 times for a Test #4, Day-30 submerged aluminum coupon sample. (T4D30AlSubm004.bmp)**

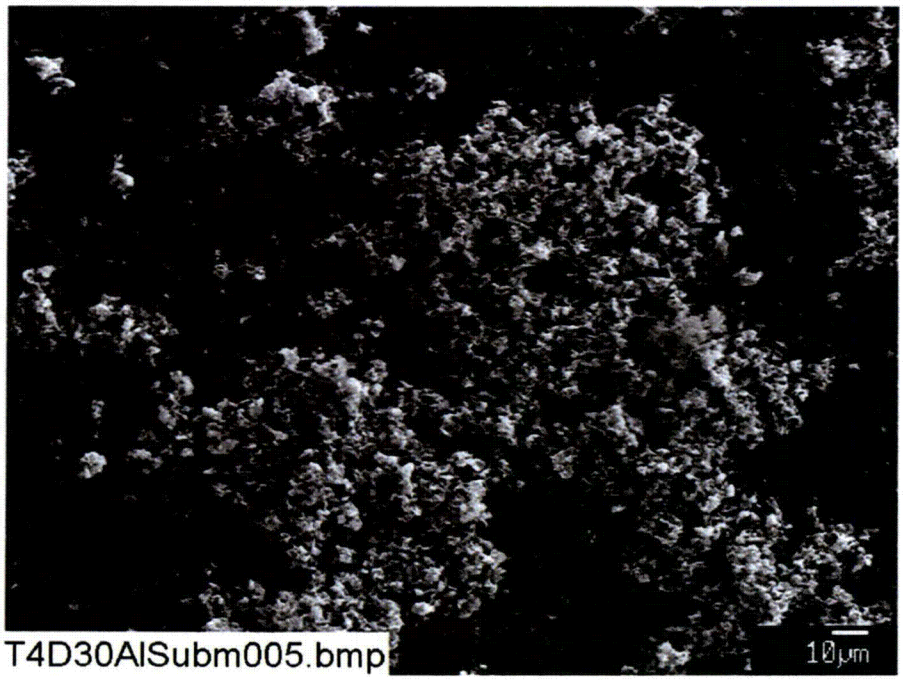


Figure F1-7. **SEM image magnified 500 times for a Test #4, Day-30 submerged aluminum coupon sample. (T4D30AlSubm005.bmp)**

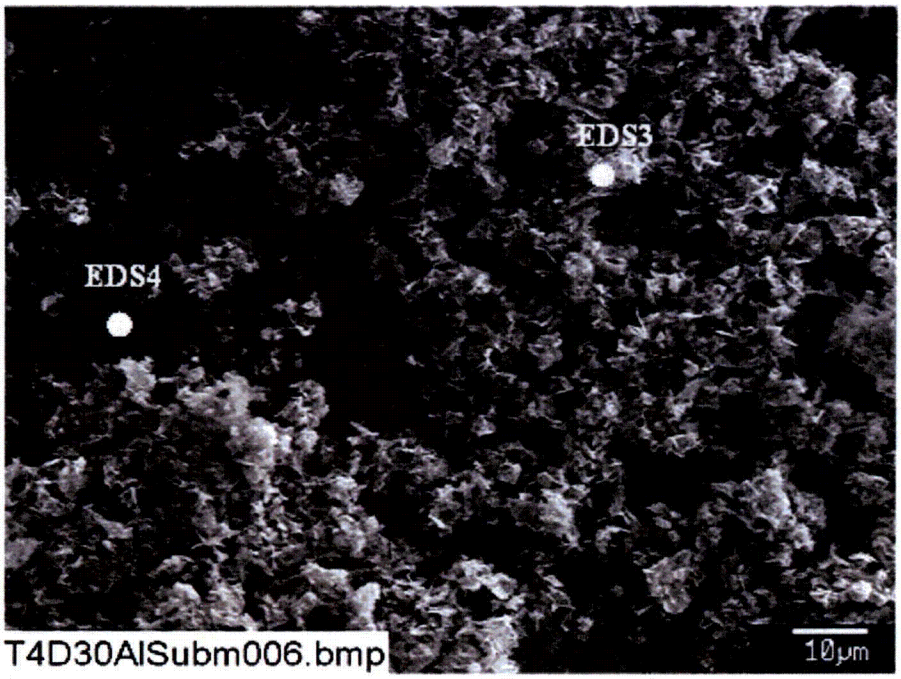


Figure F1-8. **Annotated SEM image magnified 1000 times for a Test #4, Day-30 submerged aluminum coupon sample. (T4D30AlSubm006.bmp)**

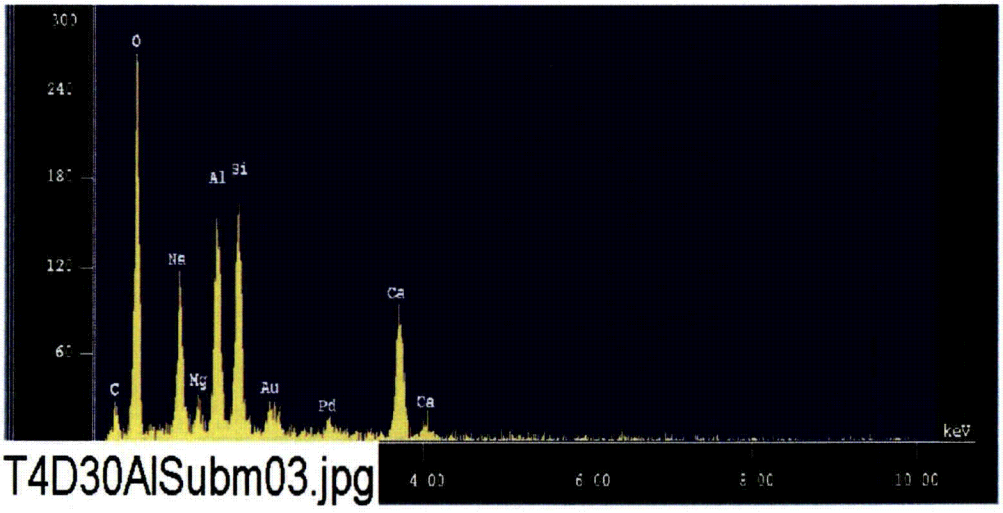


Figure F1-9. **EDS counting spectrum for the deposits (EDS3) on the coupon surface shown in Figure F1-8. (T4D30AlSubm03.jpg)**

The results from the chemical composition analysis for T4D30AlSubm03.jpg are given in Table F1-3.

Table F1-3. Chemical Compositions for T4D30AlSubm03.jpg, Figure F1-9

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Group : NRC
Sample : T4D30 ID# : 3
Comment : Particles on submerged Al
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.067E-09 A
Stage Point : X=74.706 Y=62.388 Z=10.786
Acq. Date : Wed Jun 29 10:30:29 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	0.5999	0.0002	29 / 15
O K	Normal	0.25- 0.77	68.4438	0.0020	2268 / 16
Na K	Normal	0.81- 1.27	9.1569	0.0008	872 / 18
Mg K	Normal	0.97- 1.57	1.2076	0.0002	167 / 24
Al K	Normal	1.26- 1.78	9.9577	0.0007	1369 / 79
Si K	Normal	1.50- 2.07	8.1480	0.0007	1045 / 80
Ca K	Normal	3.40- 4.30	13.3080	0.0052	829 / 4

Chi_square = 3.4940

Element	Mass%	Atomic%	ZAF	Z	A	F
C	2.021	3.2391	4.2355	1.0372	4.0840	0.9999
O	57.541	69.2225	1.0567	0.9892	1.0682	1.0000
Na	10.097	8.4534	1.3860	1.0441	1.3282	0.9994
Mg	1.535	1.2155	1.5981	0.9830	1.6294	0.9978
Al	9.810	6.9977	1.2382	1.0055	1.2340	0.9980
Si	8.534	5.8480	1.3164	0.9937	1.3251	0.9998
Ca	10.462	5.0238	0.9881	1.0029	0.9851	1.0001
Total	100.000	100.0000				

Normalization factor = 0.7956

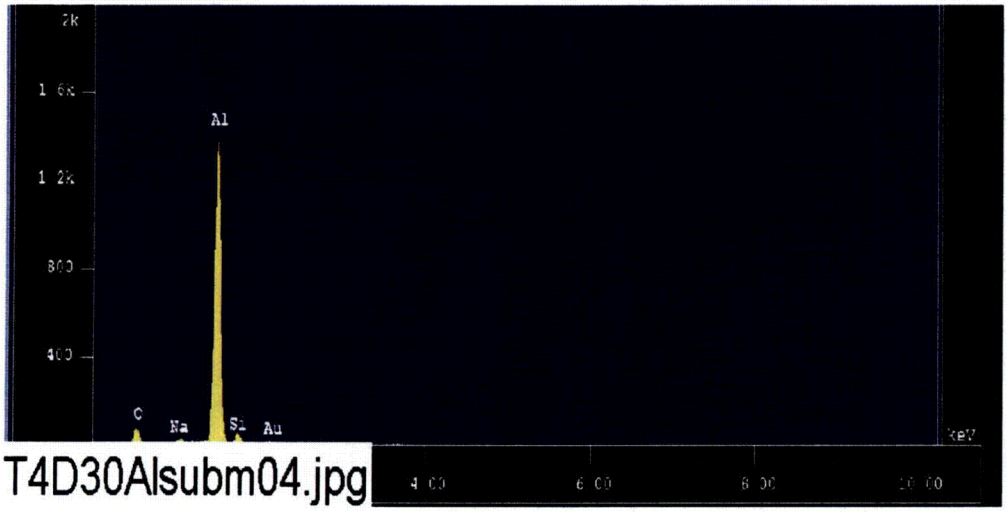

Figure F1-10. EDS counting spectrum for the flat coupon surface (EDS4) shown in Figure F1-8. (T4D30AlSubm04.jpg)

The results from the chemical composition analysis for T4D30Alsubm04.jpg are given in Table F1-4.

Table F1-4. Chemical Compositions for T4D30Alsubm04.jpg, Figure F1-10

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Group : NRC
Sample : T4D30 ID# : 4
Comment : Surface of submerged Al
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.068E-09 A
Stage Point : X=74.653 Y=62.211 Z=10.786
Acq. Date : Wed Jun 29 10:41:51 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	23.8625	0.0013	792 / 7
Na K	Normal	0.81- 1.27	1.5073	0.0005	144 / 13
Al K	Normal	1.26- 1.78	98.1067	0.0019	13504 / 30
Si K	Normal	1.50- 2.07	2.4247	0.0004	311 / 724

Chi_square = 2.7984

Element	Mass%	Atomic%	ZAF	Z	A	F
O	20.718	30.5721	1.1041	0.9815	1.1250	0.9999
Na	1.176	1.2077	0.9922	1.0354	0.9648	0.9933
Al	74.561	65.2404	0.9665	0.9967	0.9702	0.9995
Si	3.545	2.9798	1.8592	0.9847	1.8881	1.0000
Total	100.000	100.0000				

Normalization factor = 0.7863

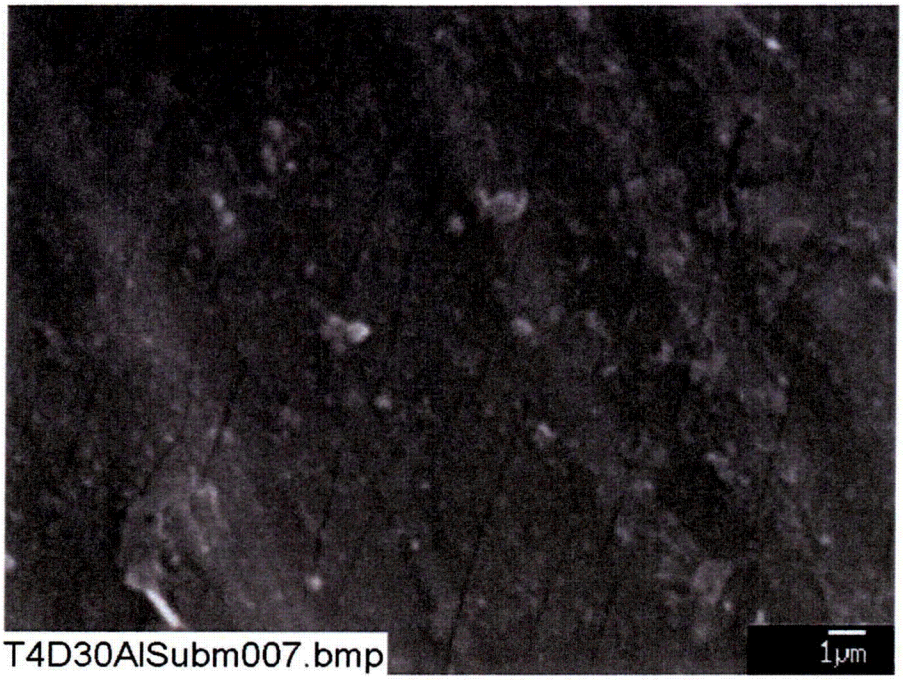


Figure F1-11. SEM image magnified 5000 times for a Test #4, Day-30 submerged aluminum coupon sample. (T4D30AlSubm007.bmp)

Appendix F2

SEM/EDS Data for Test #4, Day-30 Copper Coupons

Figures

Tables

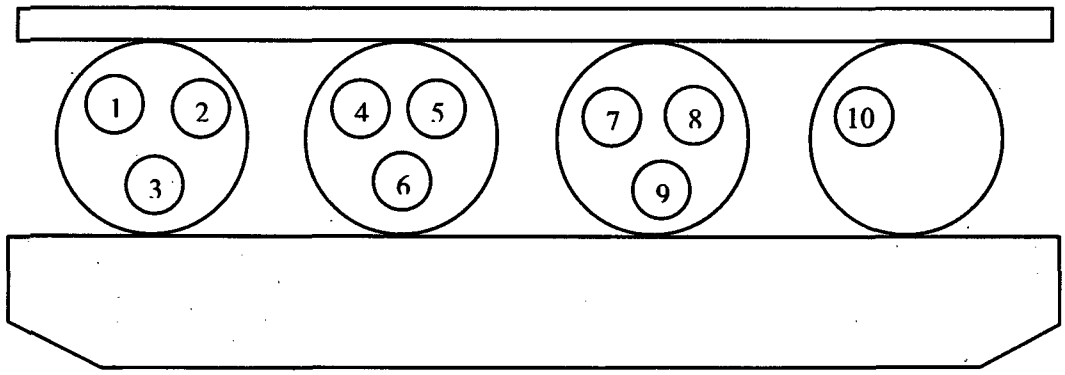
This appendix shows the SEM/EDS results for the metal copper coupons under two categories: (1) unsubmerged and (2) submerged. Unsubmerged refers to coupons held in the test tank gas space above the water level of the solution during ICET. Unsubmerged coupons were contacted by the solution only during the 4-hour spraying period at the initial date of the test. In addition, the surface of the unsubmerged coupons may also be affected by the moisture in the gas space during the test. Submerged refers to the coupons that were under the solution during the test.

The coupon samples were collected on June 23, 2005 (the date Test #4 was shut down), and by SEM/EDS on June 29, 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.

Transcribed Laboratory Log

Laboratory session from June 29, 2005.
SEM Test #4, Day-30 Copper Coupons

1. Unsubmerged Al	3. Sus. Cu	5. Sus. Gal Steel	7. Sus. Steel
2. Submerged Al	4. Sub. Cu	6. Sub. Gal Steel	8. Sub. Steel
9. Sediment	10. Powder on Sub. Rack		



Unsubmerged Copper Coupon

Image:	T4D30CuSusp008	100 ×	SEM image	Figure F2-1
	T4D30CuSusp009	500 ×	SEM image higher magnification	Figure F2-2
	T4D30CuSusp010	1500 ×	SEM annotated image	Figure F2-3
EDS:	T4D30CuSusp05		On particles in T4D30CuSusp010	Figure F2-4

Submerged Copper Coupon

Image:	T4D30CuSubm011	100 ×	SEM image of fiberglass	Figure F2-5
	T4D30CuSubm012	500 ×	SEM image higher magnification	Figure F2-6
	T4D30CuSubm013	1800 ×	SEM annotated image	Figure F2-7
EDS:	T4D30CuSubm06		On bright particles shown in 013	Figure F2-8
	T4D30CuSubm07		Surface shown in 013	Figure F2-9

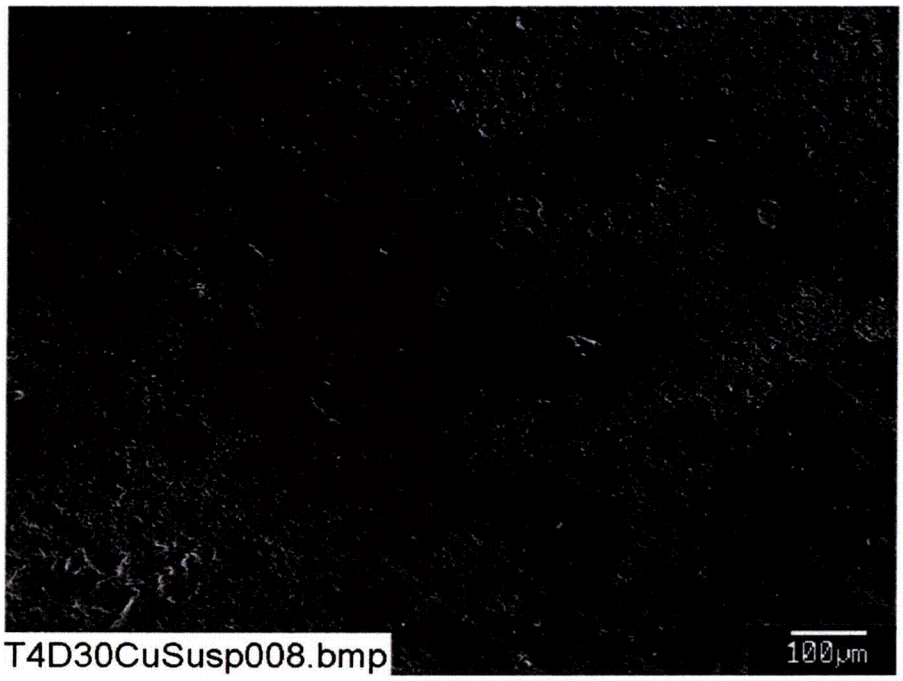


Figure F2-1. **SEM image magnified 100 times for a Test #4, Day-30 unsubmerged copper coupon sample. (T4D30CuSusp008.bmp)**

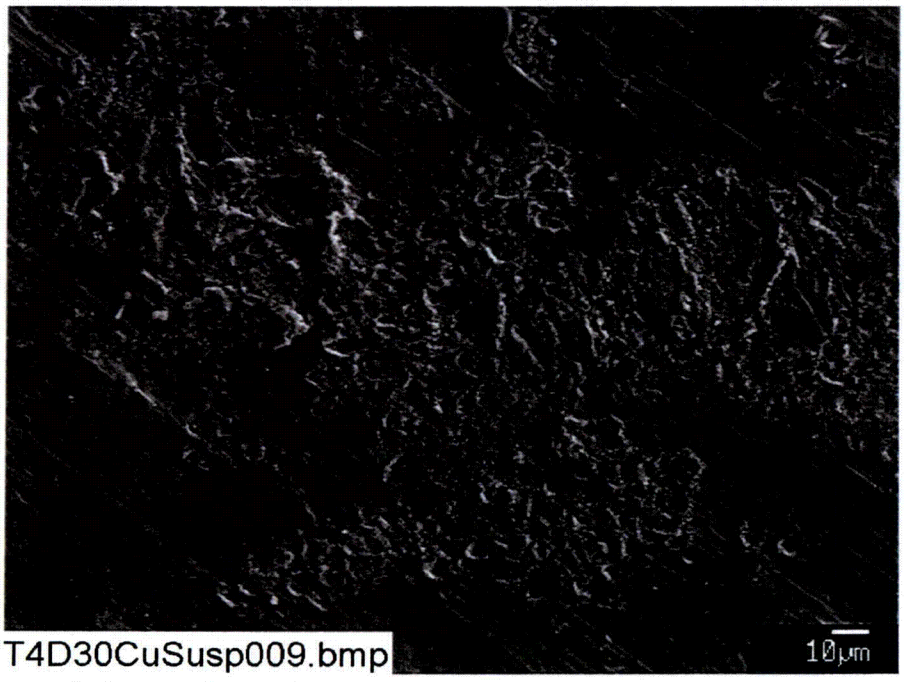


Figure F2-2. **SEM image magnified 500 times for a Test #4, Day-30 unsubmerged copper coupon sample. (T4D30CuSusp009.bmp)**

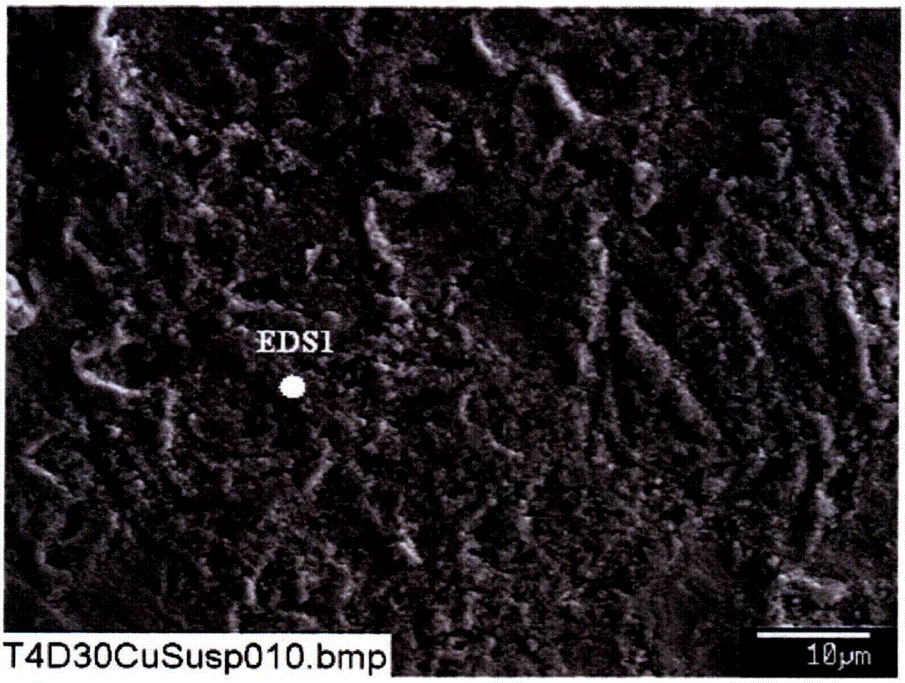


Figure F2-3. **Annotated SEM image magnified 1500 times for a Test #4, Day-30 unsubmerged copper coupon sample. (T4D30CuSusp010.bmp)**

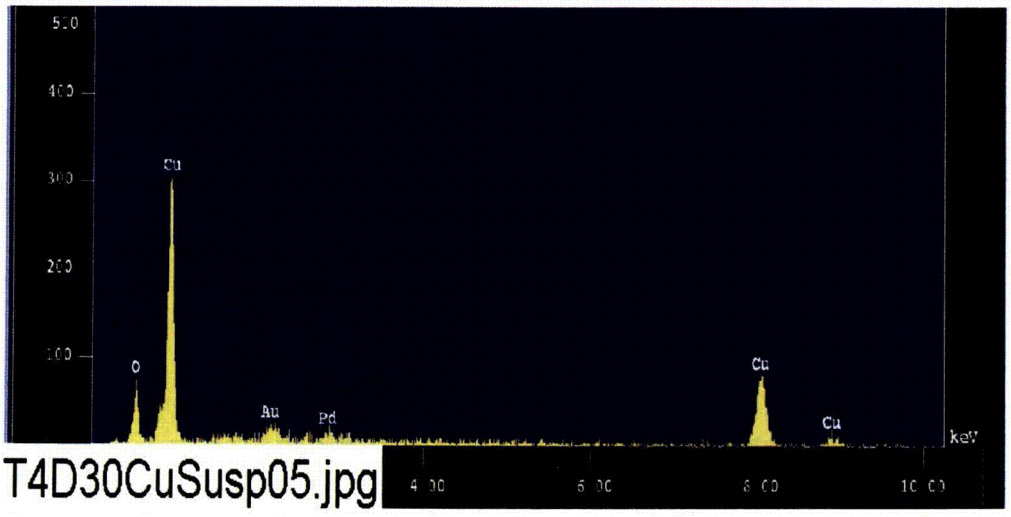


Figure F2-4. **EDS counting spectrum for the deposits (EDS1) on the coupon surface shown in Figure F2-3. (T4D30CuSusp05.jpg)**

The results from the chemical composition analysis for T4D30CuSusp05.jpg are given in Table F2-1.

Table F2-1. Chemical Compositions for T4D30CuSusp05.jpg, Figure F2-4

Jun 29 13:44 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T4D30 ID# : 5
Comment : particles on suspended Cu
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.065E-09 A
Stage Point : X=81.233 Y=71.962 Z=10.786
Acq. Date : Wed Jun 29 13:41:34 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	16.7471	0.0012	554 / 13
Cu K	Normal	7.63- 9.27	105.3640	0.0057	1327 / 2

Chi_square = 1.3920

Element	Mass%	Atomic%	ZAF	Z	A	F
O	11.102	33.1531	0.8154	0.8265	0.9868	0.9998
Cu	88.898	66.8469	1.0378	1.0386	0.9993	1.0000
Total	100.000	100.0000				

Normalization factor = 0.8130

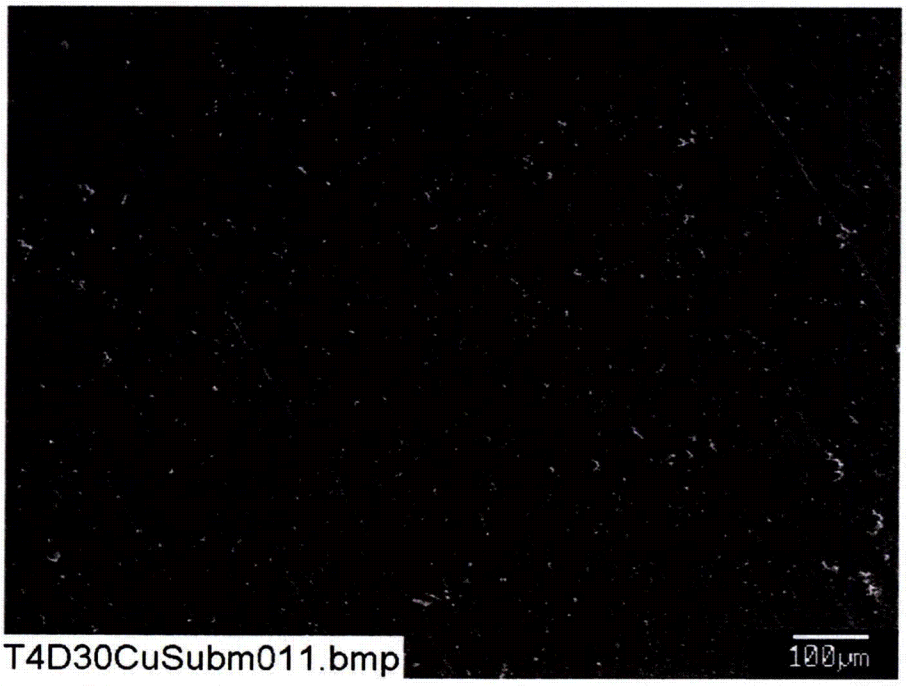


Figure F2-5. **SEM image magnified 100 times for a Test #4, Day-30 submerged copper coupon sample. (T4D30CuSubm011.bmp)**

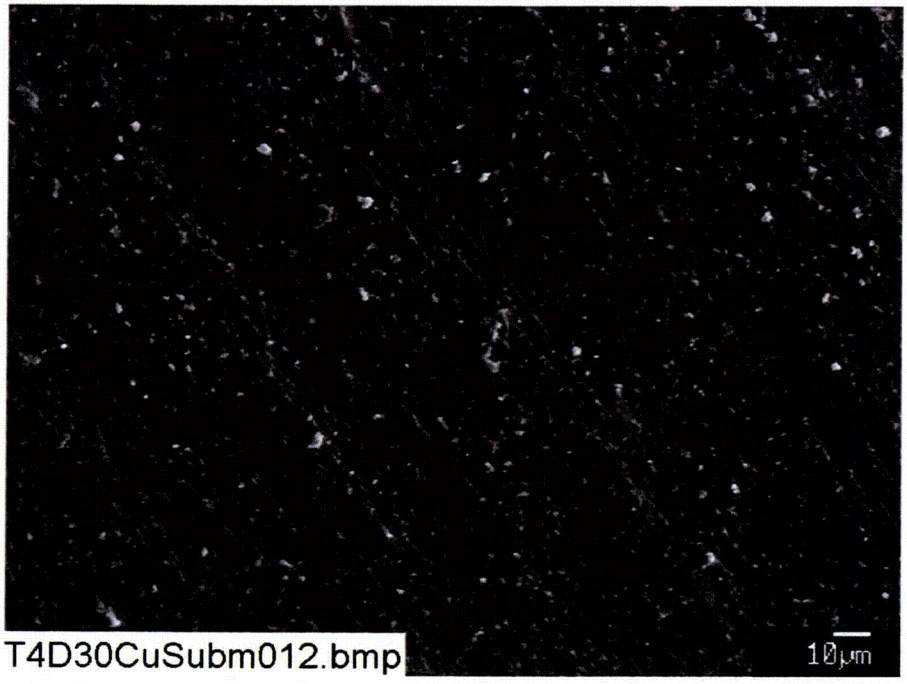


Figure F2-6. **SEM image magnified 500 times for a Test #4, Day-30 submerged copper coupon sample. (T4D30CuSubm012.bmp)**

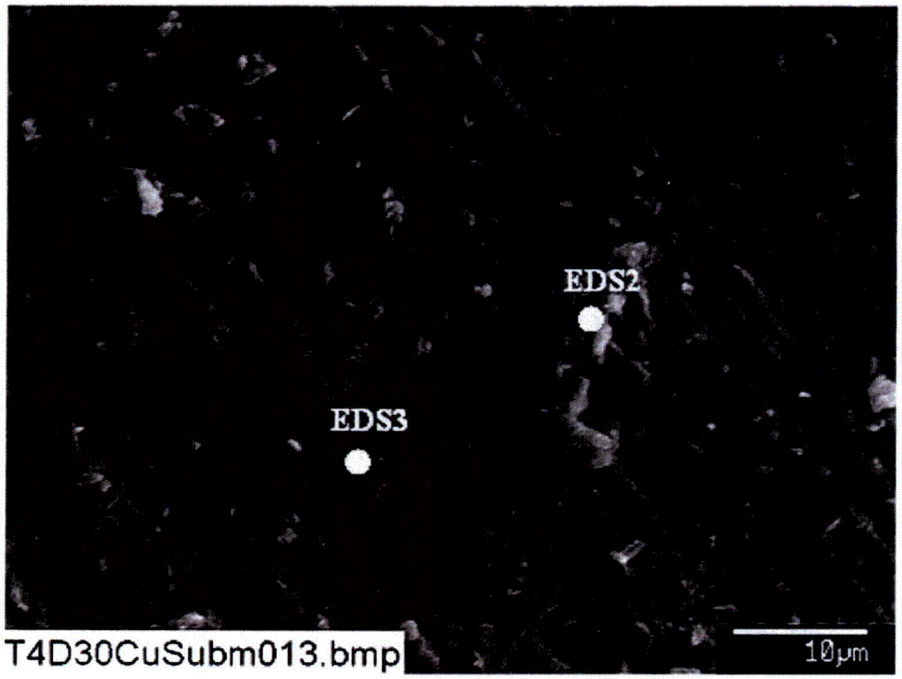


Figure F2-7. **Annotated SEM image magnified 1800 times for a Test #4, Day-30 submerged copper coupon sample. (T4D30CuSubm013.bmp)**

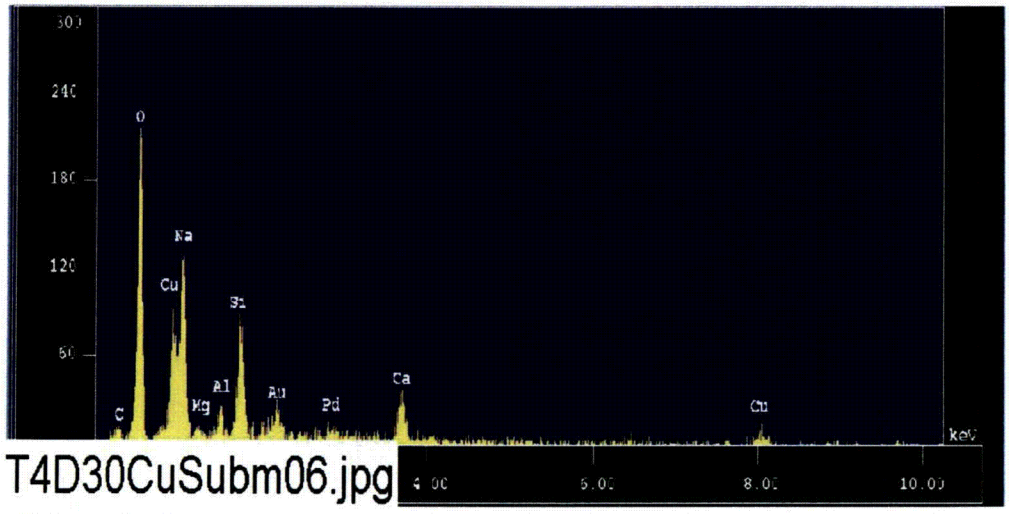


Figure F2-8. **EDS counting spectrum for the deposit (EDS2) on the coupon surface shown in Figure F2-7. (T4D30CuSubm06.jpg)**

The results from the chemical composition analysis for T4D30CuSubm06.jpg are given in Table F2-2.

Table F2-2. Chemical Compositions for T4D30CuSubm06.jpg, Figure F2-8

Jun 29 13:57 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T4D30 ID# : 6
Comment : light particles on submerged Cu
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.065E-09 A
Stage Point : X=57.699 Y=58.376 Z=10.786
Acq. Date : Wed Jun 29 13:54:39 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	55.2961	0.0018	1829 / 10
Na K	Normal	0.81- 1.27	11.0827	0.0010	1053 / 13
Al K	Normal	1.26- 1.78	1.1553	0.0003	159 / 32
Si K	Normal	1.50- 2.07	4.0896	0.0005	523 / 17
Ca K	Normal	3.40- 4.30	4.2931	0.0035	267 / 6
Cu K	Normal	7.63- 9.27	9.7062	0.0023	122 / 2
C K	Normal	0.09- 0.46	0.0000	0.0000	0 / 16
Mg K	Normal	0.97- 1.57	0.0718	0.0001	10 / 36

Chi_square = 2.6388

Element	Mass%	Atomic%	ZAF	Z	A	F
O	52.039	67.6839	0.8346	0.9692	0.8612	0.9999
Na	20.992	19.0006	1.6798	1.0227	1.6423	1.0001
Al	1.925	1.4848	1.4779	0.9846	1.5028	0.9988
Si	6.377	4.7251	1.3829	0.9729	1.4216	0.9999
Ca	4.710	2.4453	0.9729	0.9809	0.9925	0.9994
Cu	13.788	4.5154	1.2597	1.2639	0.9967	1.0000
C	0.000	0.0000	4.0970	1.0163	4.0314	0.9999
Mg	0.169	0.1449	2.0917	0.9627	2.1740	0.9994
Total	100.000	100.0000				

Normalization factor = 1.1276

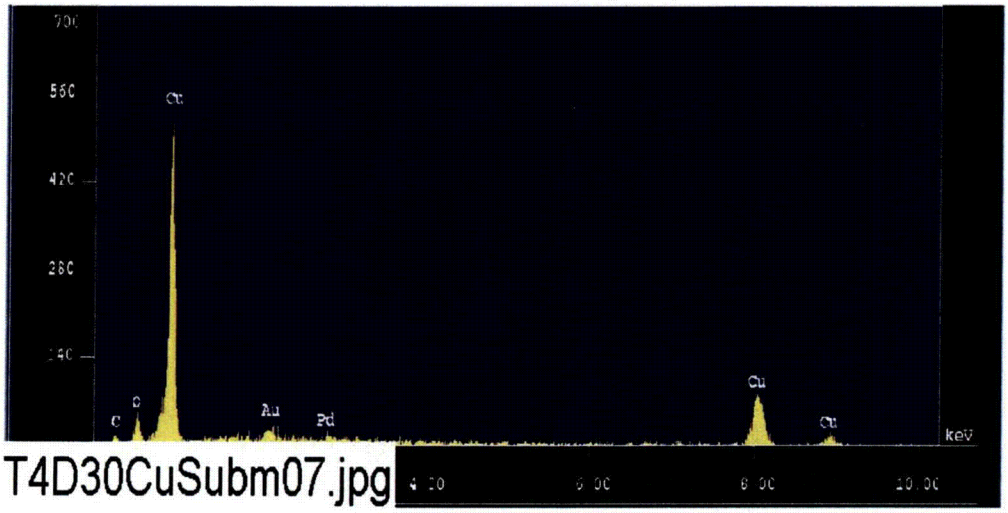


Figure F2-9. **EDS counting spectrum for the flat coupon surface (EDS3) shown in Figure F2-7. (T4D30CuSubm07.jpg)**

The results from the chemical composition analysis for T4D30CuSubm07.jpg are given in Table F2-3.

Table F2-3. Chemical Compositions for T4D30CuSubm07.jpg, Figure F2-9

Jun 29 14:02 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T4D30 ID# : 7
Comment : surface of submerged Cu
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.065E-09 A
Stage Point : X=57.699 Y=58.376 Z=10.786
Acq. Date : Wed Jun 29 13:59:37 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	12.2183	0.0011	404 / 21
Cu K	Normal	7.63- 9.27	113.6477	0.0061	1432 / 0
C K	Normal	0.09- 0.46	2.1513	0.0001	106 / 4

Chi_square = 0.9631

Element	Mass%	Atomic%	ZAF	Z	A	F
O	7.923	20.9480	0.8970	0.8301	1.0807	0.9998
Cu	85.861	57.1606	1.0450	1.0460	0.9991	1.0000
C	6.216	21.8914	3.9968	0.8712	4.5876	1.0000
Total	100.000	100.0000				

Normalization factor = 0.7229

Appendix F3

SEM/EDS Data for Test #4, Day-30 Galvanized Steel Coupons

Figures

Tables

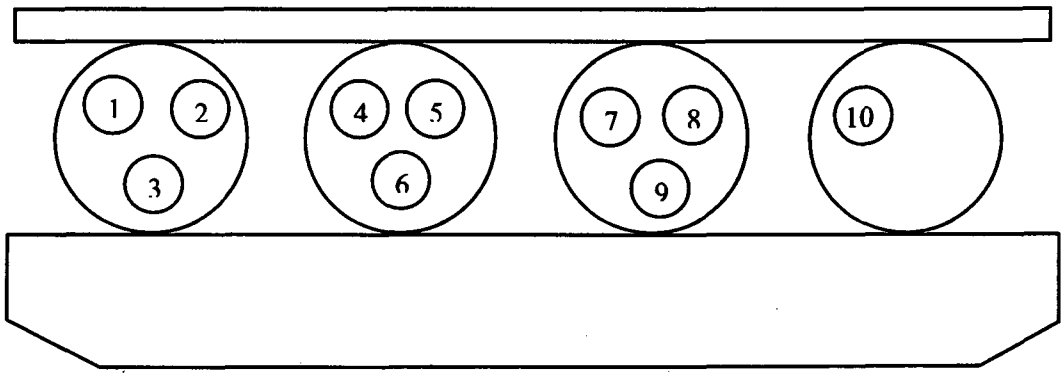
This appendix shows the SEM/EDS results for the metal galvanized steel coupons under two categories: (1) unsubmerged and (2) submerged. Unsubmerged refers to coupons held in the test tank gas space above the water level of the solution during ICET. Unsubmerged coupons were contacted by the solution only during the 4-hour spraying period at the initial date of the test. In addition, the surface of the unsubmerged coupons may also be affected by the moisture in the gas space during the test. Submerged refers to the coupons that were under the solution during the test.

The coupon samples were collected on June 23, 2005 (the date Test #4 was shut down), and examined by SEM/EDS on June 29, 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.

Transcribed Laboratory Log

Laboratory session from June 29, 2005.
SEM Test #4, Day-30 Galvanized Steel Coupons

1. Unsubmerged Al	3. Sus. Cu	5. Sus. Gal Steel	7. Sus. Steel
2. Submerged Al	4. Sub. Cu	6. Sub. Gal Steel	8. Sub. Steel
9. Sediment	10. Powder on Sub. Rack		



Unsubmerged Galvanized Steel Coupon

Image:	T4D30GalSusp014	100 ×	SEM image	Figure F3-1
	T4D30GalSusp015	500 ×	SEM image	Figure F3-2
	T4D30GalSusp016	1800 ×	SEM annotated image	Figure F3-3
EDS:	T4D30GalSusp08		Particles shown in 016	Figure F3-4
	T4D30Galsusp09		Surface shown in 016	Figure F3-5

Submerged Galvanized Steel Coupon

Image:	T4D30GalSubm017	100 ×	SEM image	Figure F3-6
	T4D30GalSubm018	500 ×	SEM image	Figure F3-7
	T4D30GalSubm019	1800 ×	SEM annotated image	Figure F3-8
	T4D30GalSubm10		Particles shown in 019	Figure F3-9
	T4D30GalSubm11		Surface shown in 019	Figure F3-10

Figure F3-1. **SEM image magnified 100 times for a Test #4, Day-30 unsubmerged galvanized steel coupon sample. (T4D30GalSusp014.bmp)**

Figure F3-2. **SEM image magnified 500 times for a Test #4, Day-30 unsubmerged galvanized steel coupon sample. (T4D30GalSusp015.bmp)**

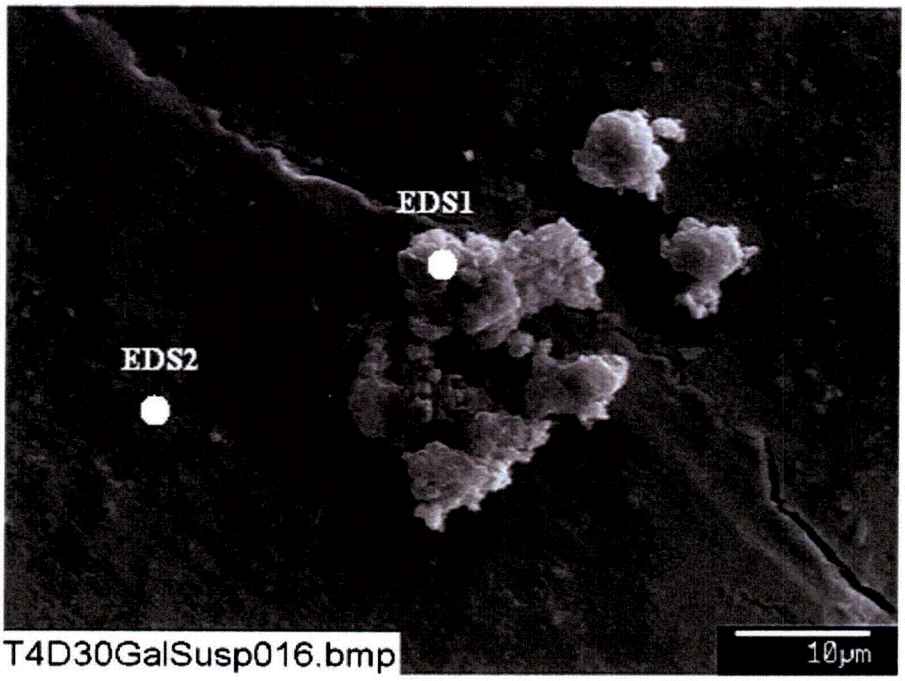


Figure F3-3. **Annotated SEM image magnified 1800 times for a Test #4, Day-30 unsubmerged galvanized steel coupon sample. (T4D30GalSusp016.bmp)**

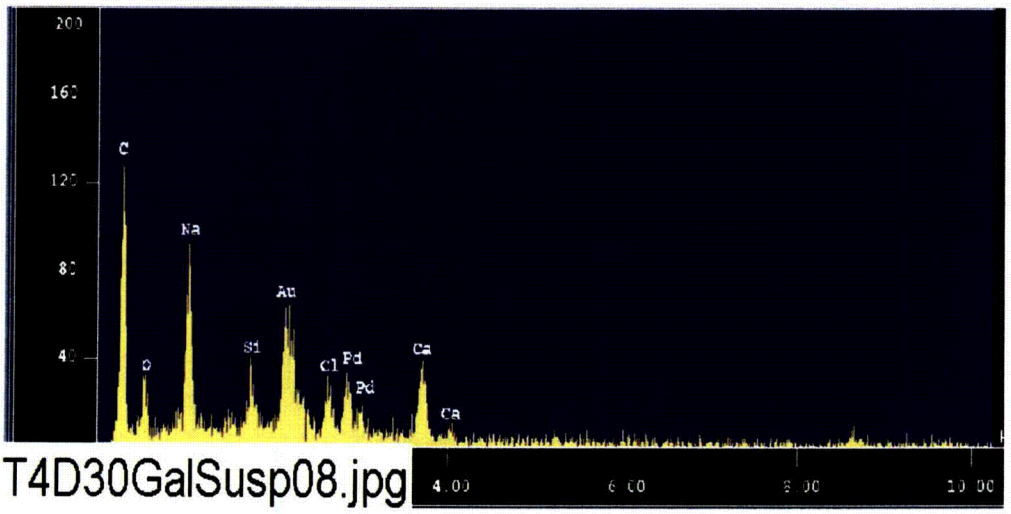


Figure F3-4. **EDS counting spectrum for the deposits (EDS1) on the coupon surface shown in Figure F3-3. (T4D30GalSusp08.jpg)**

The results from the chemical composition analysis for T4D30GalSusp08.jpg are given in Table F3-1.

Table F3-1. Chemical Compositions for T4D30GalSusp08.jpg, Figure F3-4

Jun 29 14:17 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T4D30 ID# : 8
Comment : particle on suspended Gal-steel
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.064E-09 A
Stage Point : X=44.143 Y=59.814 Z=10.786
Acq. Date : Wed Jun 29 14:12:20 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	17.7108	0.0003	868 / 7
O K	Normal	0.25- 0.77	7.9933	0.0009	264 / 43
Na K	Normal	0.81- 1.27	4.1179	0.0007	391 / 7
Si K	Normal	1.50- 2.07	1.2113	0.0004	155 / 14
Cl K	Normal	2.34- 3.06	3.7051	0.0005	324 / 14
Ca K	Normal	3.40- 4.30	5.1818	0.0037	322 / 5

Chi_square = 6.7756

Element	Mass%	Atomic%	ZAF	Z	A	F
C	57.624	69.8633	2.4583	1.0116	2.4303	1.0000
O	22.840	20.7886	2.1589	0.9652	2.2368	1.0000
Na	6.485	4.1080	1.1900	1.0197	1.1668	1.0002
Si	1.750	0.9073	1.0916	0.9716	1.1247	0.9989
Cl	4.786	1.9657	0.9759	1.0255	0.9552	0.9963
Ca	6.515	2.3671	0.9500	0.9837	0.9657	1.0001
Total	100.000	100.0000				

Normalization factor = 1.3235

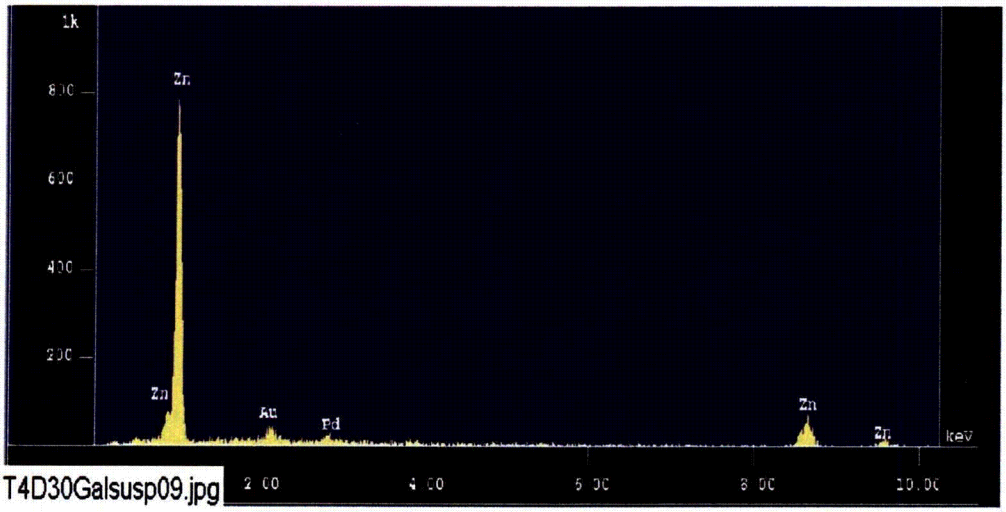


Figure F3-5. **EDS counting spectrum for the flat coupon surface (EDS2) shown in Figure F3-3. (T4D30Galsusp09.jpg)**

Figure F3-6. **SEM image magnified 100 times for a Test #4, Day-30 submerged galvanized steel coupon sample. (T4D30GalSubm017.bmp)**

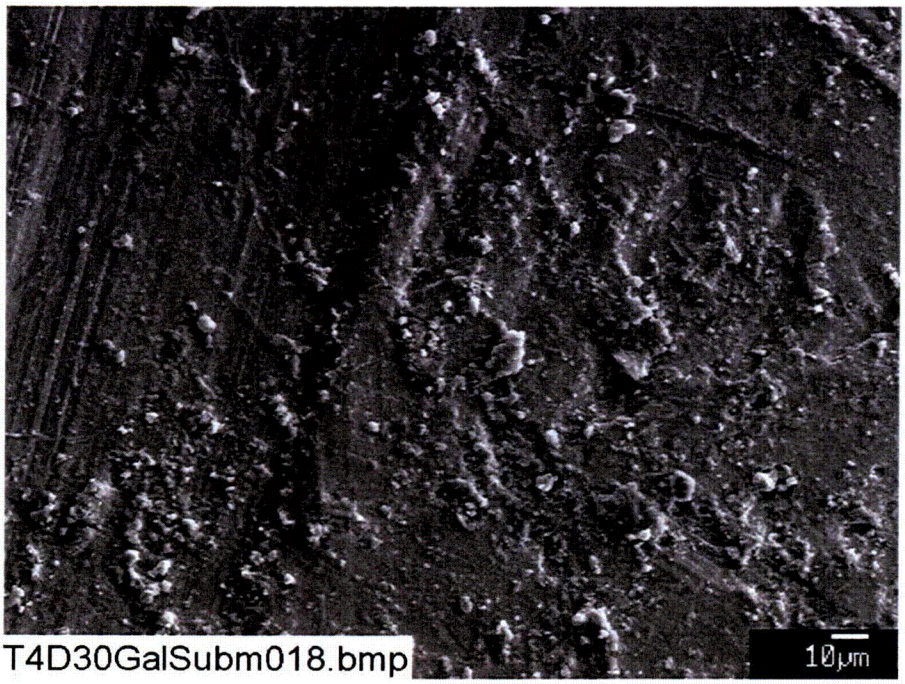


Figure F3-7. **SEM image magnified 500 times for a Test #4, Day-30 submerged galvanized steel coupon sample. (T4D30GalSubm018.bmp)**

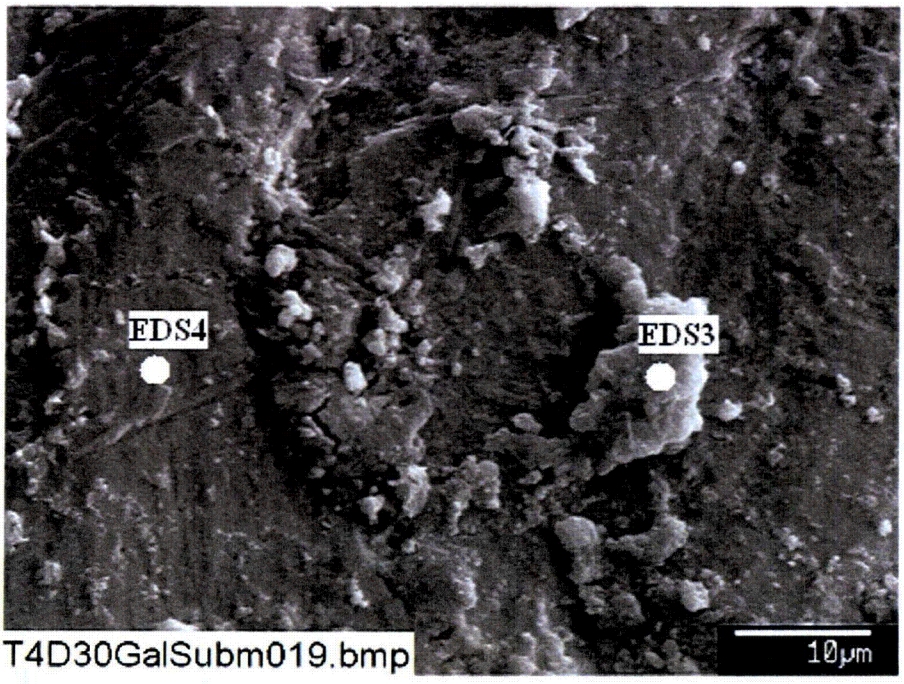


Figure F3-8. **Annotated SEM image magnified 1800 times for a Test #4, Day-30 submerged galvanized steel coupon sample. (T4D30GalSubm019.bmp)**

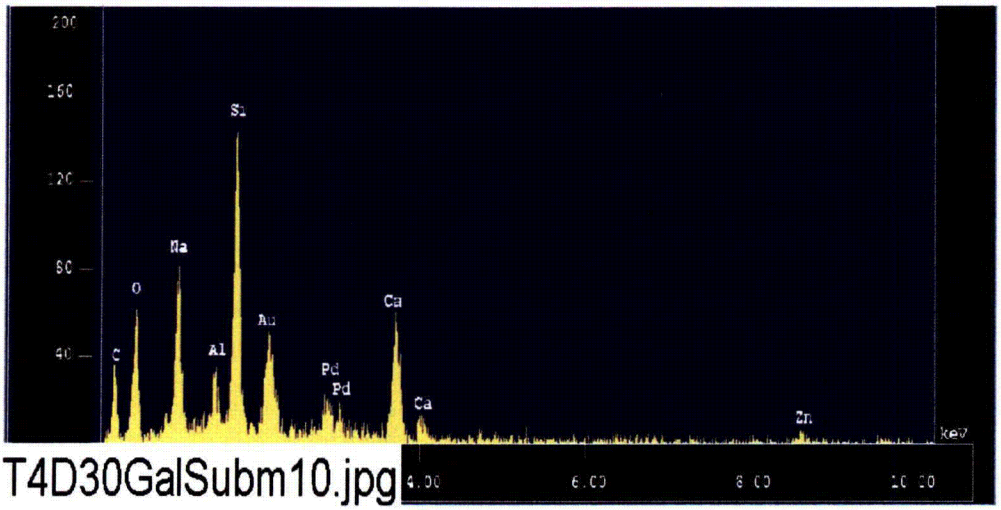


Figure F3-9. **EDS counting spectrum for the deposits (EDS3) on the coupon surface shown in Figure F3-8. (T4D30GalSubm10.jpg)**

The results from the chemical composition analysis for T4D30GalSubm10.jpg are given in Table F3-2.

Table F3-2. Chemical Compositions for T4D30GalSubm10.jpg, Figure F3-9

Jun 29 14:33 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T4D30 ID# : 10
Comment : Particle on submerged Gal-steel
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.067E-09 A
Stage Point : X=50.108 Y=68.926 Z=10.786
Acq. Date : Wed Jun 29 14:30:34 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	4.3159	0.0001	212 / 6
O K	Normal	0.25- 0.77	17.0145	0.0011	564 / 10
Na K	Normal	0.81- 1.27	6.3047	0.0007	600 / 6
Al K	Normal	1.26- 1.78	1.7139	0.0004	236 / 56
Si K	Normal	1.50- 2.07	7.1958	0.0006	923 / 25
Ca K	Normal	3.40- 4.30	8.1648	0.0042	509 / 2

Chi_square = 2.5440

Element	Mass%	Atomic%	ZAF	Z	A	F
C	23.363	34.2953	3.7436	1.0268	3.6461	1.0000
O	38.077	41.9614	1.5476	0.9794	1.5802	1.0000
Na	11.659	8.9415	1.2789	1.0340	1.2371	0.9998
Al	2.933	1.9165	1.1835	0.9960	1.1924	0.9965
Si	12.465	7.8251	1.1980	0.9844	1.2173	0.9997
Ca	11.503	5.0602	0.9743	0.9944	0.9797	1.0001
Total	100.000	100.0000				

Normalization factor = 1.4460

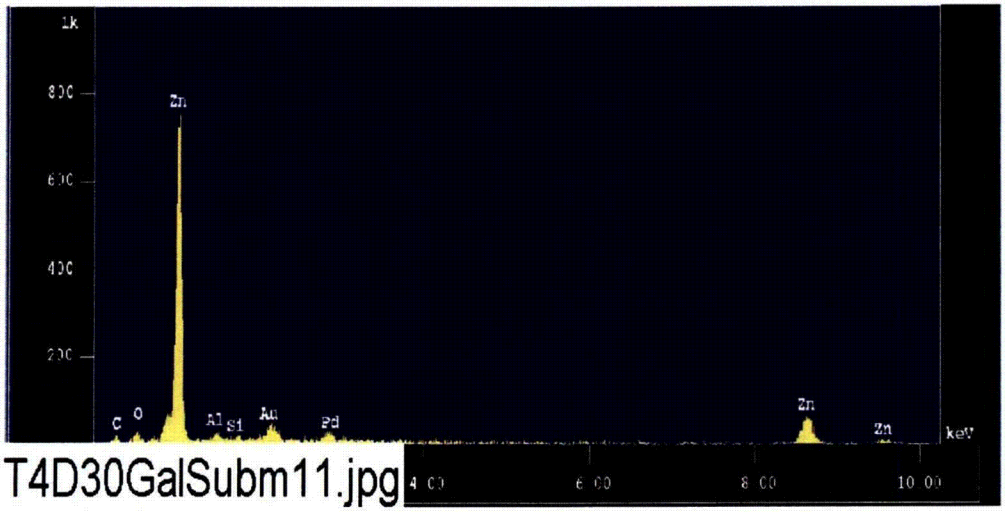


Figure F3-10. EDS counting spectrum for the flat coupon surface (EDS4) shown in Figure F3-8. (T4D30GalSubm11.jpg)

The results from the chemical composition analysis for T4D30GalSubm11.jpg are given in Table F3-3

Table F3-3. Chemical Compositions for T4D30GalSubm11.jpg, Figure F3-10

```
Jun 29 14:37 2005  /tmp/eds_pout.log Page 1


 Group      : NRC
 Sample     : T4D30  ID# : 11
 Comment    : Surface of submerged Gal-steel
 Condition  : Full Scale   : 20KeV(10eV/ch,2Kch)
              Live Time    :  60.000 sec     Aperture #     : 2
              Acc. Volt    : 15.0 KV         Probe Current  : 1.066E-09 A
              Stage Point  : X=50.108 Y=68.926 Z=10.786
              Acq. Date    : Wed Jun 29 14:35:16 2005


 Element      Mode      ROI(KeV)    K-ratio(%)   +/-     Net/Background
   O K       Normal   0.25- 0.77     7.3244    0.0010       243 /       18
  Al K       Normal   1.26- 1.78     0.9436    0.0004       130 /       16
  Si K       Normal   1.50- 2.07     0.4713    0.0003        60 /       22
  Zn K       Normal   8.22-10.03   118.8961    0.0077      1137 /        2
   C K       Normal   0.09- 0.46     2.8301    0.0001       139 /        2
-------------------------------------------------------------------------
                              Chi_square =  0.5995

 Element  Mass%     Atomic%    ZAF       Z       A       F
     O     4.987   12.5657  1.0235  0.8341  1.2272  0.9999
    Al     1.235    1.8457  1.9679  0.8447  2.3300  0.9999
    Si     0.520    0.7459  1.6575  0.8339  1.9877  1.0000
    Zn    83.279   51.3537  1.0528  1.0538  0.9991  1.0000
     C     9.979   33.4891  5.2998  0.8754  6.0542  1.0000
-------------------------------------------------------------------------
 Total   100.000 100.0000
 Normalization factor =  0.6653
```

Appendix F4

SEM/EDS Data for Test #4, Day-30 Steel Coupons

Figures

Tables

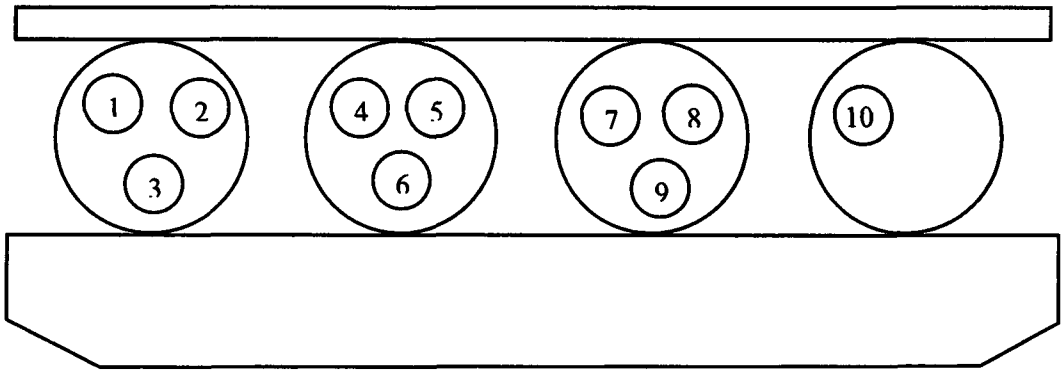
This appendix shows the SEM/EDS results for the metal steel coupons under two categories: (1) unsubmerged and (2) submerged. Unsubmerged refers to coupons held in the test tank gas space above the water level of the solution during ICET tests. Unsubmerged coupons were contacted by the solution only during the 4-hour spraying period at the initial date of the test. In addition, the surface of the unsubmerged coupons may also be affected by the moisture in the gas space during the test. Submerged refers to the coupons that were under the solution during the test.

The coupon samples were collected on June 23, 2005 (the date Test #4 was shut down), and examined by SEM/EDS on June 29, 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.

Transcribed Laboratory Log

Laboratory session from June 29, 2005.
SEM Test #4, Day-30 Steel Coupons

1. Unsubmerged Al	3. Sus. Cu	5. Sus. Gal Steel	7. Sus. Steel
2. Submerged Al	4. Sub. Cu	6. Sub. Gal Steel	8. Sub. Steel
9. Sediment	10. Powder on Sub. Rack		



Unsubmerged Steel Coupons

Image:	T4D30SteelSusp020	100 ×	SEM image	Figure F4-1
	T4D30SteelSusp021	500 ×	SEM image	Figure F4-2
	T4D30SteelSusp022	1800 ×	SEM annotated image	Figure F4-3
EDS:	T4D30SteelSusp12		Particles shown in 022	Figure F4-4
	T4D30SteelSusp13		Surface shown in 022	Figure F4-5

Submerged Steel Coupons

Image:	T4D30SteelSubm023	100 ×	SEM image	Figure F4-6
	T4D30SteelSubm024	500 ×	SEM image	Figure F4-7
	T4D30SteelSubm025	1500 ×	SEM annotated image	Figure F4-8
EDS:	T4D30SteelSubm14		Particles shown in 025	Figure F4-9
	T4D30SteelSubm15		Surface shown in 025	Figure F4-10

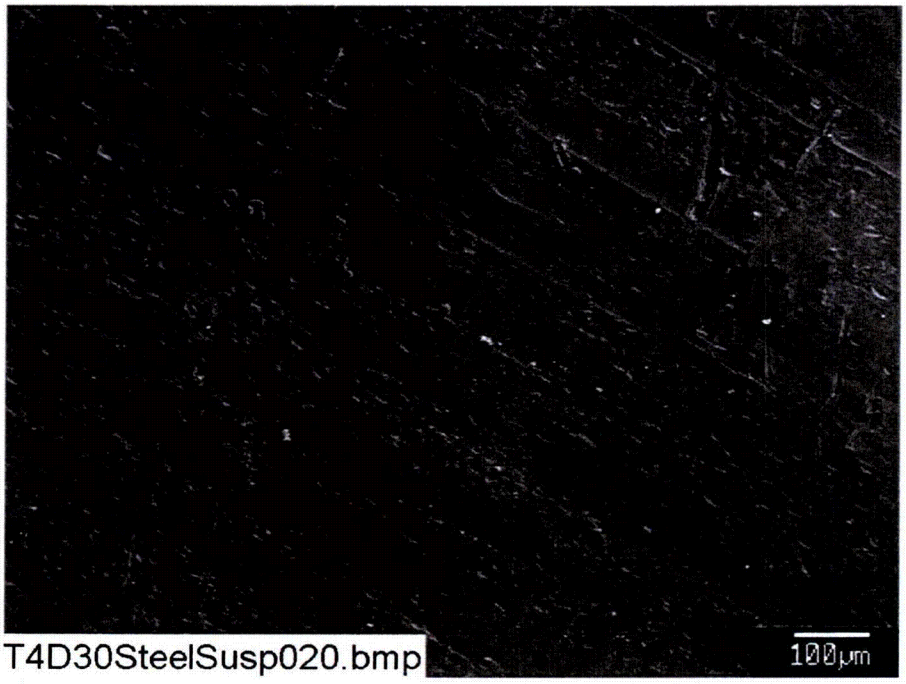


Figure F4-1. **SEM image magnified 100 times for a Test #4, Day-30 unsubmerged uncoated steel coupon sample. (T4D30SteelSusp020.bmp)**

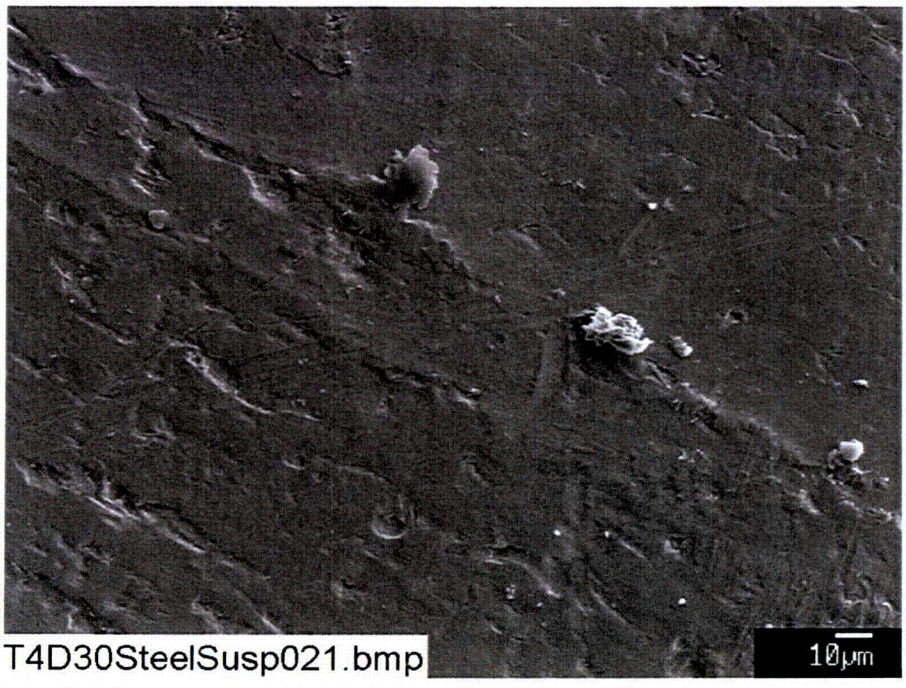


Figure F4-2. **SEM image magnified 500 times for a Test #4, Day-30 unsubmerged uncoated steel coupon sample. (T4D30SteelSusp021.bmp)**

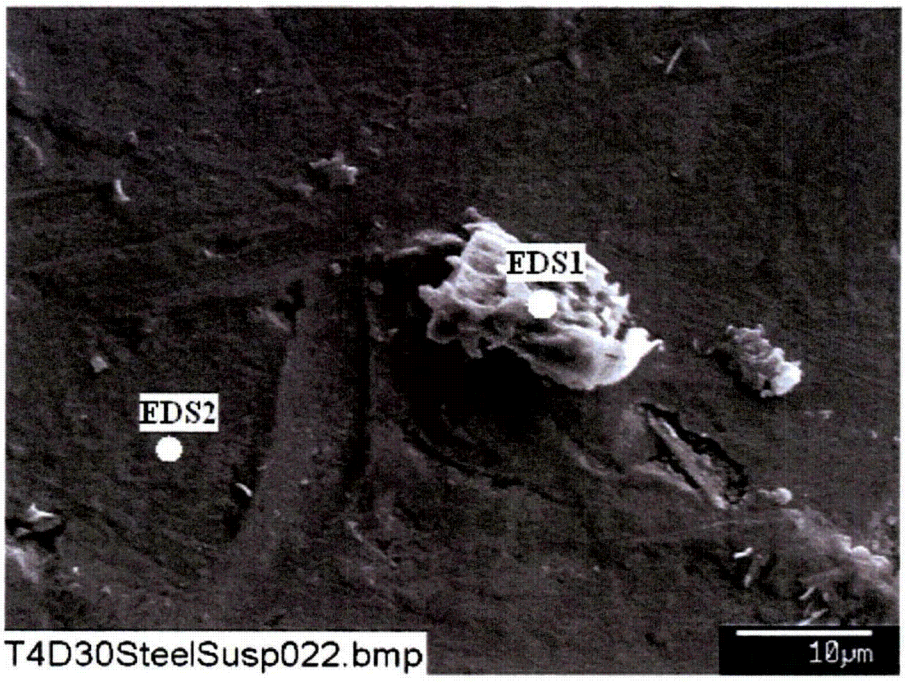


Figure F4-3. **Annotated SEM image magnified 1800 times for a Test #4, Day-30 unsubmerged uncoated steel coupon sample. (T4D30SteelSusp022.bmp)**

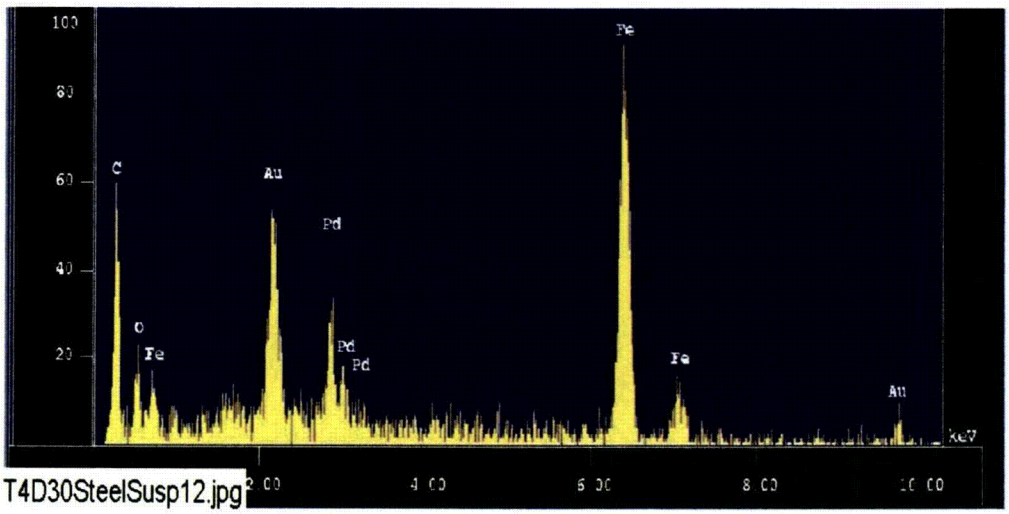


Figure F4-4. **EDS counting spectrum for the white deposits (EDS1) on the coupon surface shown in Figure F4-3. (T4D30SteelSusp12.jpg)**

The results from the chemical composition analysis for T4D30SteelSusp12.jpg are given in Table F4-1.

Table F4-1. Chemical Compositions for T4D30SteelSusp12.jpg, Figure F4-4

Jun 29 14:51 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T4D30 ID# : 12
Comment : Particle on suspended steel
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.064E-09 A
Stage Point : X=26.283 Y=57.998 Z=10.786
Acq. Date : Wed Jun 29 14:48:17 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	7.3229	0.0002	359 / 2
O K	Normal	0.25- 0.77	5.2438	0.0007	173 / 21
Fe K	Normal	6.00- 7.44	57.5951	0.0015	1453 / 1

Chi_square = 3.0082

Element	Mass%	Atomic%	ZAP	Z	A	F
C	23.723	54.7789	2.8221	0.9173	3.0766	1.0000
O	5.935	10.2882	0.9859	0.8744	1.1282	0.9995
Fe	70.342	34.9330	1.0639	1.0675	0.9967	1.0000
Total	100.000	100.0000				

Normalization factor = 1.1480

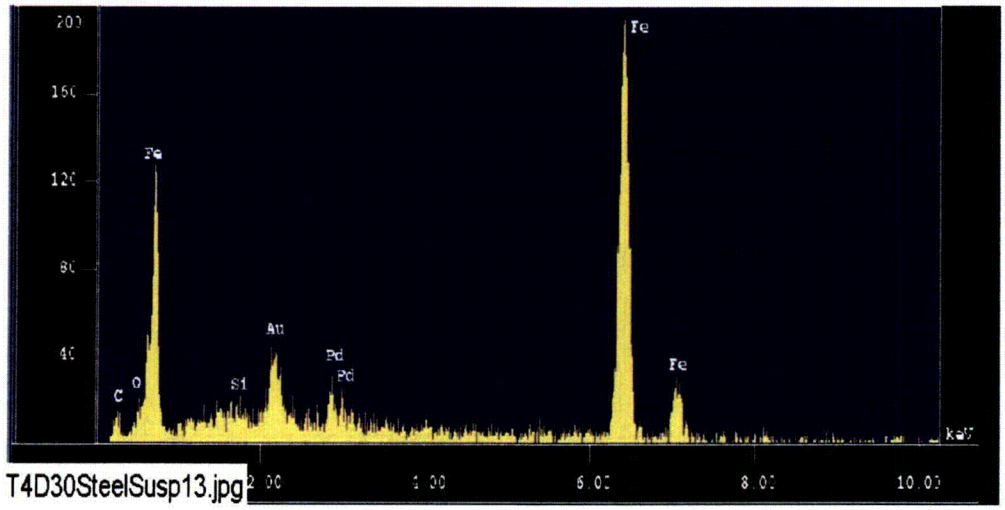


Figure F4-5. **EDS counting spectrum for the flat coupon surface (EDS2) shown in Figure F4-3. (T4D30SteelSusp13.jpg)**

The results from the chemical composition analysis for T4D30SteelSusp13.jpg are given in Table F4-2.

Table F4-2. Chemical Compositions for T4D30SteelSusp13.jpg, Figure F4-5

Jun 29 14:56 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T4D30 ID# : 13
Comment : Surface of suspended steel
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.065E-09 A
Stage Point : X=26.283 Y=57.998 Z=10.786
Acq. Date : Wed Jun 29 14:52:52 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
Fe K	Normal	6.00- 7.44	131.0725	0.0022	3310 / 4
C K	Normal	0.09- 0.46	1.8367	0.0001	90 / 4
Si K	Normal	1.50- 2.07	0.0575	0.0003	7 / 14
O K	Normal	0.25- 0.77	2.0509	0.0010	68 / 17

Chi_square = 0.9378

Element	Mass%	Atomic%	ZAF	Z	A	F
Fe	94.919	80.9892	1.0113	1.0118	0.9995	1.0000
C	3.993	15.8420	3.0361	0.8799	3.4506	1.0000
Si	0.057	0.0961	1.3759	0.8380	1.6420	0.9999
O	1.032	3.0728	0.7025	0.8384	0.8387	0.9991
Total	100.000	100.0000				

Normalization factor = 0.7161

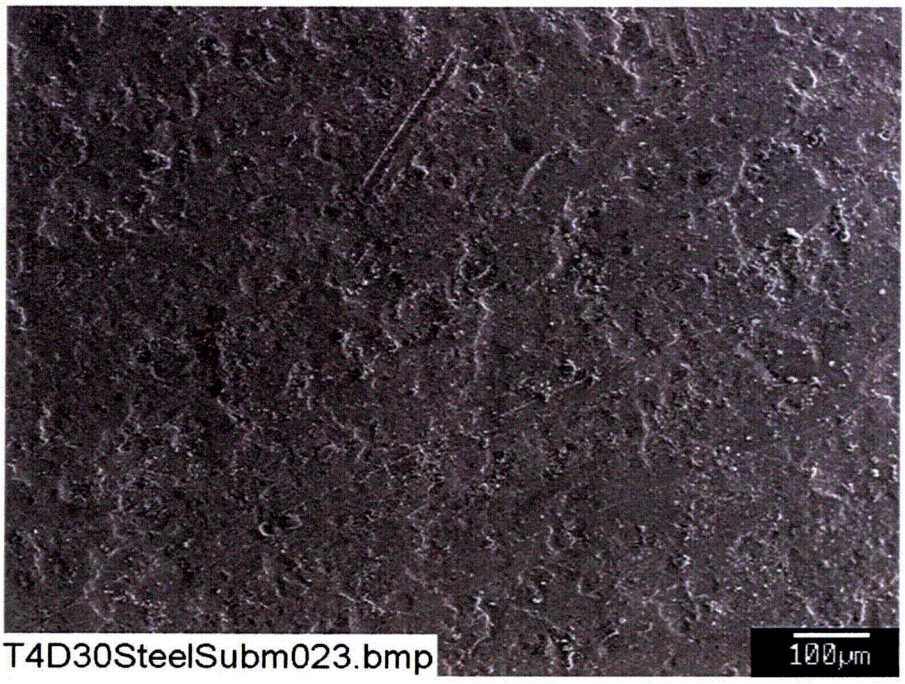


Figure F4-6. **SEM image magnified 100 times for a Test #4, Day-30 submerged uncoated steel coupon sample. (T4D30SteelSubm023.bmp)**

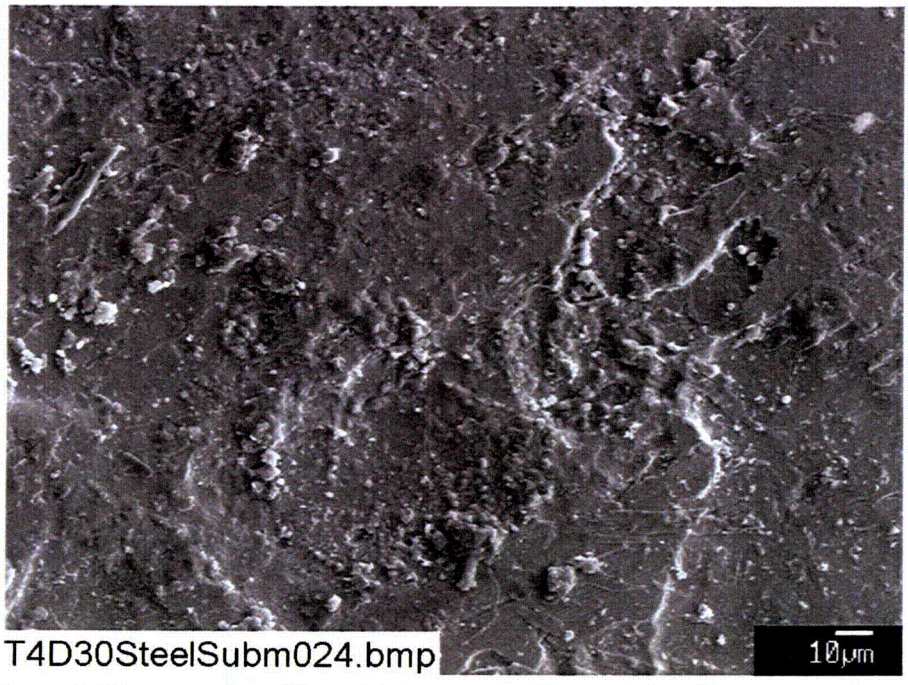


Figure F4-7. **SEM image magnified 500 times for a Test #4, Day-30 submerged uncoated steel coupon sample. (T4D30SteelSubm024.bmp)**

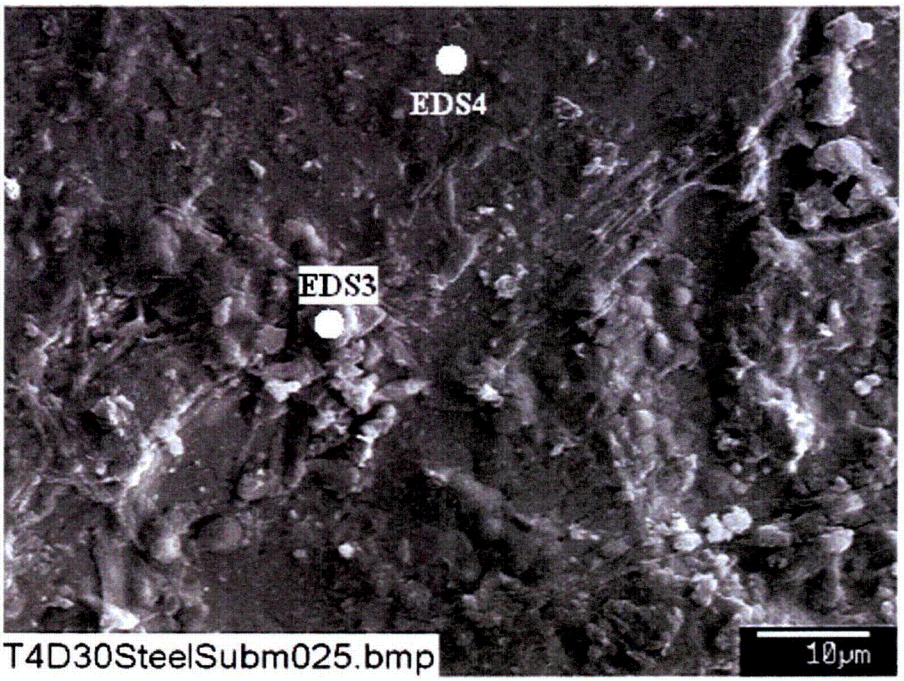


Figure F4-8. **Annotated SEM image magnified 1500 times for a Test #4, Day-30 submerged uncoated steel coupon sample. (T4D30SteelSubm025.bmp)**

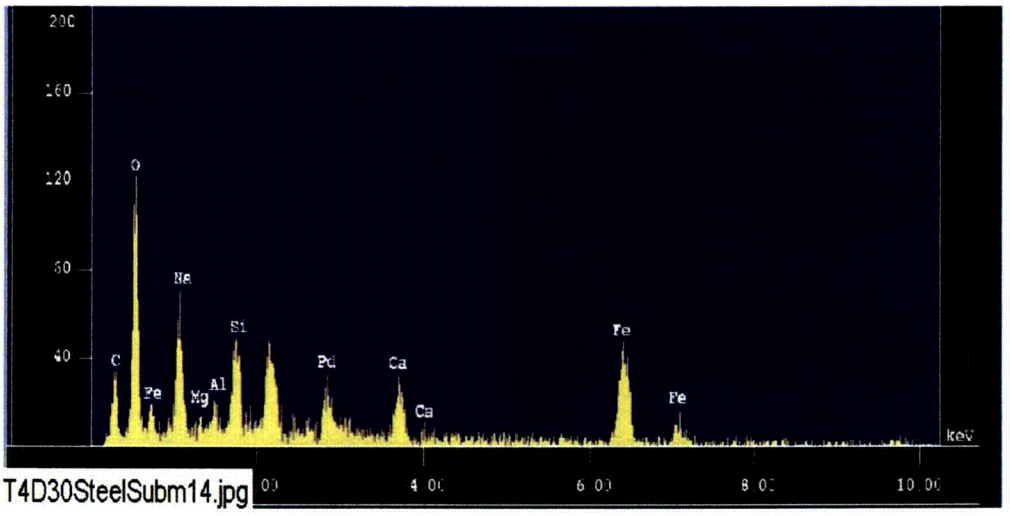


Figure F4-9. **EDS counting spectrum for the deposit (EDS3) shown in Figure F4-8. (T4D30SteelSubm14.jpg)**

The results from the chemical composition analysis for T4D30SteelSubm14.jpg are given in Table F4-3.

Table F4-3. Chemical Compositions for T4D30SteelSubm14.jpg, Figure F4-9

Jun 29 15:08 2005 /tmp/eds_pout.log Page 1

Group : MRC
Sample : T4D30 ID# : 14
Comment : Particles on submerged steel
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.063E-09 A
Stage Point : X=12.785 Y=58.790 Z=10.786
Acq. Date : Wed Jun 29 15:05:38 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	3.5591	0.0002	174 / 8
O K	Normal	0.25- 0.77	30.6742	0.0015	1013 / 13
Na K	Normal	0.81- 1.27	5.0811	0.0006	482 / 13
Al K	Normal	1.26- 1.78	0.7478	0.0003	102 / 22
Si K	Normal	1.50- 2.07	2.0211	0.0004	258 / 13
Ca K	Normal	3.40- 4.30	3.3136	0.0032	206 / 4
Fe K	Normal	6.00- 7.44	29.5118	0.0012	744 / 3
Mg K	Normal	0.97- 1.57	0.0852	0.0001	12 / 10

Chi_square = 2.0712

Element	Mass%	Atomic%	ZAF	Z	A	P
C	12.691	23.9254	3.2608	0.9777	3.3354	1.0000
O	31.600	44.7225	0.9420	0.9322	1.0108	0.9998
Na	11.110	10.9423	1.9994	0.9834	2.0328	1.0002
Al	1.177	0.9881	1.4397	0.9465	1.5219	0.9994
Si	2.951	2.3790	1.3351	0.9351	1.4279	0.9999
Ca	3.349	1.8922	0.9243	0.9416	0.9890	0.9926
Fe	36.931	14.9736	1.1443	1.1473	0.9974	1.0000
Mg	0.190	0.1769	2.0386	0.9256	2.2032	0.9997
Total	100.000	100.0000				

Normalization factor = 1.0936

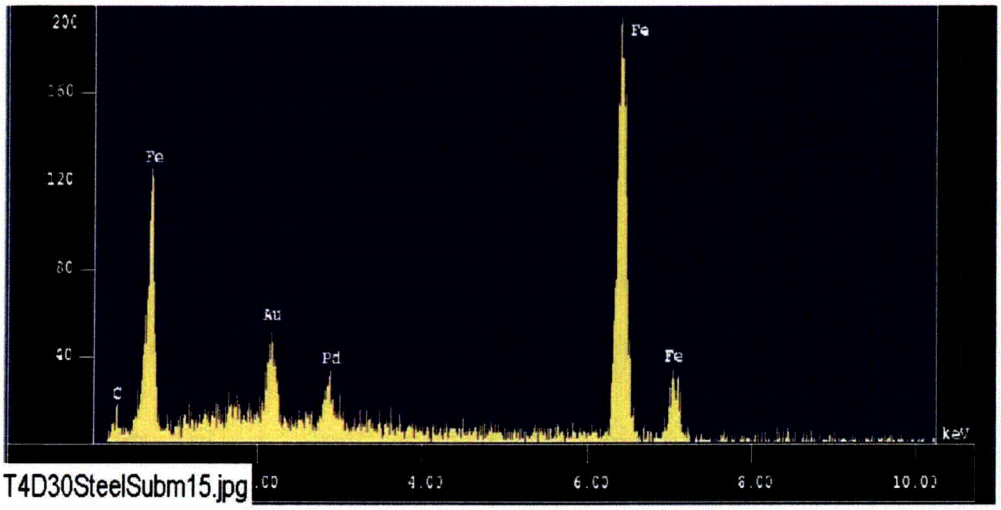


Figure F4-10. EDS counting spectrum for the flat coupon surface (EDS4) shown in Figure F4-8. (T4D30SteelSubm15.jpg)

The results from the chemical composition analysis for T4D30SteelSubm15.jpg are given in Table F4-4.

Table F4-4. Chemical Compositions for T4D30SteelSubm15.jpg, Figure F4-10

```
Jun 29 15:12 2005  /tmp/eds_pout.log Page 1


 Group     : NRC
 Sample    : T4D30  ID# : 15
 Comment   : Surface of submerged steel
 Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   :  60.000 sec    Aperture #    : 2
             Acc. Volt   : 15.0 KV        Probe Current : 1.064E-09 A
             Stage Point : X=12.785 Y=58.790 Z=10.786
             Acq. Date   : Wed Jun 29 15:10:11 2005


 Element     Mode      ROI(KeV)    K-ratio(%)  +/-     Net/Background
  Fe K      Normal   6.00- 7.44   134.9460   0.0022      3404 /       3
   C K      Normal   0.09- 0.46     1.2783   0.0001        63 /       4
-------------------------------------------------------------------------
                              Chi_square =  1.2118

 Element Mass%    Atomic%   ZAF      Z      A      F
    Fe   97.224  88.2786 1.0059 1.0062 0.9997 1.0000
     C    2.776  11.7214 3.0324 0.8759 3.4621 1.0000
-------------------------------------------------------------------------
 Total   100.000 100.0000
 Normalization factor =  0.7162
```

Appendix G

SEM/EDS Data for Test #4, Day-30 Sediment

Figures

Tables

Particulate sediments at the bottom of the tank directly relate to the corrosion products and debris generated during ICET. This appendix lists the probe SEM/EDS and XRD/XRF results for the sediment samples collected from the bottom of the tank on the date Test #4 was shut down (June 23, 2005). The purpose of these analyses is to provide information on the morphology and the composition of the sediments.

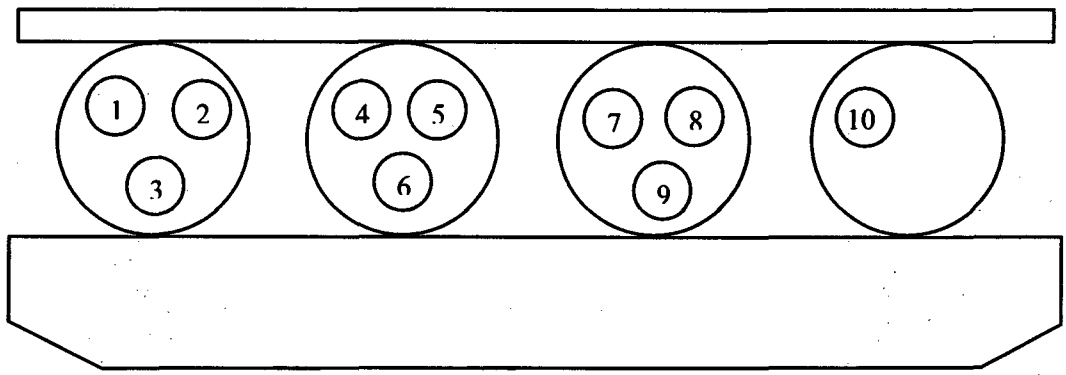
The sediment samples were dried in air before being coated with Au/Pd for probe SEM examination. EDS results provide an elemental composition of the sediment. The SEM/EDS results of the Test #4, Day-30 sediment samples were obtained on June 29, 2005. XRD and XRF analyses were performed on August 25 and July 19, 2005, respectively. Based on XRD results, the sediment sample contained crystalline substances of tobermorite [$Ca_{2.25}(Si_3O_{7.5}(OH)_{1.5})(H_2O)$], $Ca_4[Si_6O_{15}(OH)_2)(H_2O)_5]$, and calcite ($CaCO_3$), similar to the unused raw or unused baked cal-sil samples. XRF results show the chemical composition of the sediment.

Transcribed Laboratory Log

Laboratory session from June 29, 2005.

SEM Test #4, Day-30 Sediment

1. Suspended Al	3. Sus. Cu	5. Sus. Gal Steel	7. Sus. Steel
2. Submerged Al	4. Sub. Cu	6. Sub. Gal Steel	8. Sub. Steel
9. Sediment	10. Powder on Sub. Rack		



Bottom of Tank Sediment Sample

Image:	T4D30SEDMT026	100 ×	SEM image	Figure G-1
	T4D30SEDMT027	500 ×	SEM annotated image	Figure G-2
EDS:	T4D30SEDMT16		EDS on white snow like particle shown in 027	Figure G-3
	T4D30SEDMT17		EDS on dark particle shown in 027	Figure G-4
Image:	T4D30SEDMT028	1000 ×	SEM at higher magnification	Figure G-5

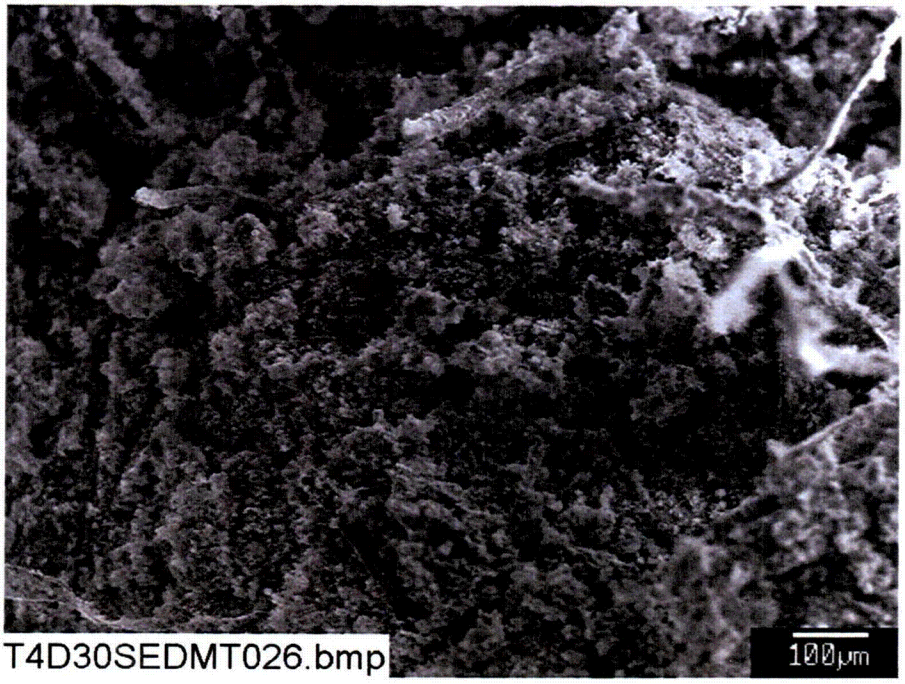


Figure G-1. **SEM image magnified 100 times for a Test #4, Day-30 sediment at the bottom of the tank. (T4D30SEDMT026.bmp)**

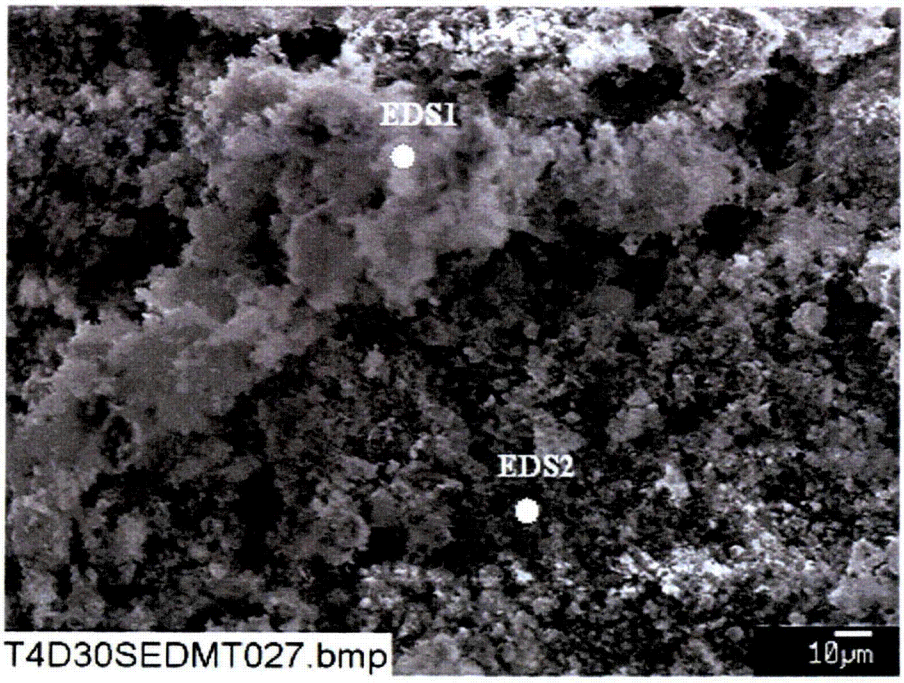


Figure G-2. **Annotated SEM image magnified 500 times for a Test #4, Day-30 sediment at the bottom of the tank. (T4D30SEDMT027.bmp)**

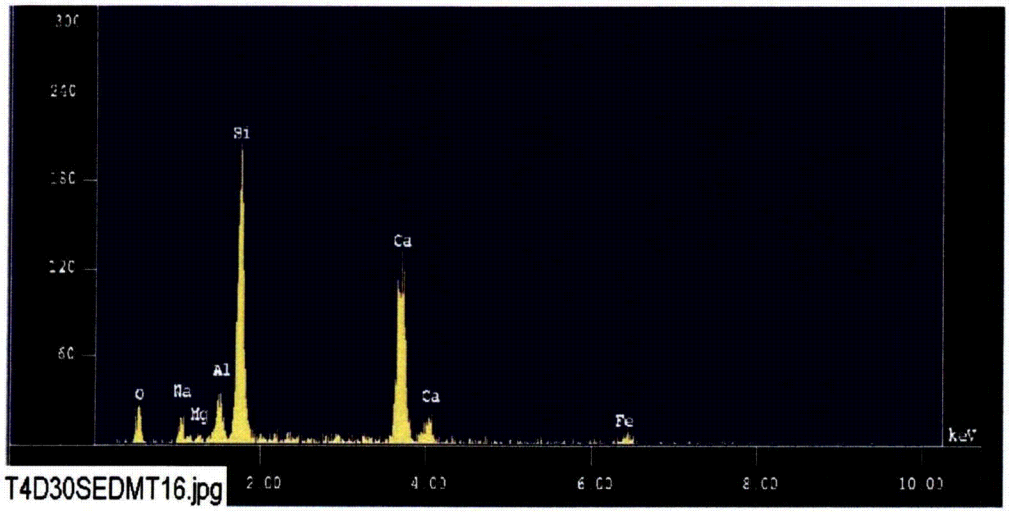


Figure G-3. **EDS counting spectrum for the white snow like deposits (EDS1) shown in Figure G-2. (T4D30SEDMT16.jpg)**

The results from the chemical composition analysis for T4D30SEDMT16.jpg are given in Table G-1.

Table G-1. Chemical Compositions for T4D30SEDMT16.jpg, Figure G-3

Jun 29 15:24 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T4D30 ID# : 16
Comment : snow like particle in sediment
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.065E-09 A
Stage Point : X=19.550 Y=69.838 Z=10.786
Acq. Date : Wed Jun 29 15:21:11 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	6.2037	0.0007	205 / 0
Na K	Normal	0.81- 1.27	1.6489	0.0003	157 / 6
Mg K	Normal	0.97- 1.57	0.3435	0.0001	47 / 6
Si K	Normal	1.50- 2.07	10.5531	0.0007	1351 / 15
Ca K	Normal	3.40- 4.30	19.5783	0.0060	1218 / 4
Al K	Normal	1.26- 1.78	1.9653	0.0003	270 / 70
Fe K	Normal	6.00- 7.44	2.2007	0.0005	56 / 2

Chi_square = 1.2925

Element	Mass%	Atomic%	ZAF	Z	A	F
O	24.352	40.1993	2.0616	0.9667	2.1326	1.0000
Na	4.528	5.2016	1.4422	1.0195	1.4157	0.9993
Mg	0.998	1.0843	1.5261	0.9594	1.5950	0.9972
Si	24.295	22.8456	1.2091	0.9690	1.2487	0.9992
Ca	36.326	23.9368	0.9745	0.9748	1.0002	0.9995
Al	4.425	4.3313	1.1825	0.9810	1.2130	0.9938
Fe	5.077	2.4010	1.2116	1.1869	1.0209	1.0000
Total	100.000	100.0000				

Normalization factor = 1.9040

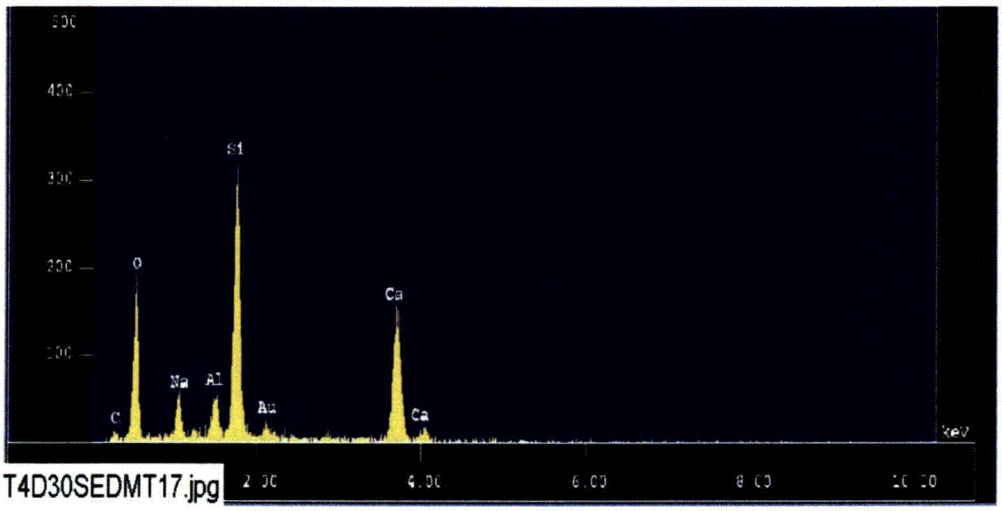


Figure G-4. **EDS counting spectrum for the dark deposits (EDS2) shown in Figure G-2. (T4D30SEDMT17.jpg)**

The results from the chemical composition analysis for T4D30SEDMT17.jpg are given in Table G-2.

Table G-2. Chemical Compositions for T4D30SEDMT17.jpg, Figure G-4

Jun 29 15:28 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T4D30 ID# : 17
Comment : dark particle in sediment
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 1.065E-09 A
Stage Point : X=19.550 Y=69.838 Z=10.786
Acq. Date : Wed Jun 29 15:26:31 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
O K	Normal	0.25- 0.77	45.7598	0.0017	1514 / 5
Na K	Normal	0.81- 1.27	4.6668	0.0006	444 / 12
Al K	Normal	1.26- 1.78	2.9372	0.0004	403 / 132
Si K	Normal	1.50- 2.07	16.4077	0.0009	2100 / 30
Ca K	Normal	3.40- 4.30	21.6864	0.0066	1349 / 4
C K	Normal	0.09- 0.46	0.1986	0.0002	10 / 14

Chi_square = 3.0443

Element	Mass%	Atomic%	ZAF	Z	A	F
O	54.173	69.0385	1.3508	0.9866	1.3691	1.0000
Na	5.887	5.2207	1.4393	1.0412	1.3828	0.9996
Al	3.068	2.3186	1.1920	1.0025	1.1947	0.9952
Si	17.376	12.6144	1.2084	0.9907	1.2203	0.9995
Ca	18.748	9.5372	0.9864	0.9992	0.9871	1.0001
C	0.749	1.2706	4.3013	1.0346	4.1578	0.9999
Total	100.000	100.0000				

Normalization factor = 0.8764

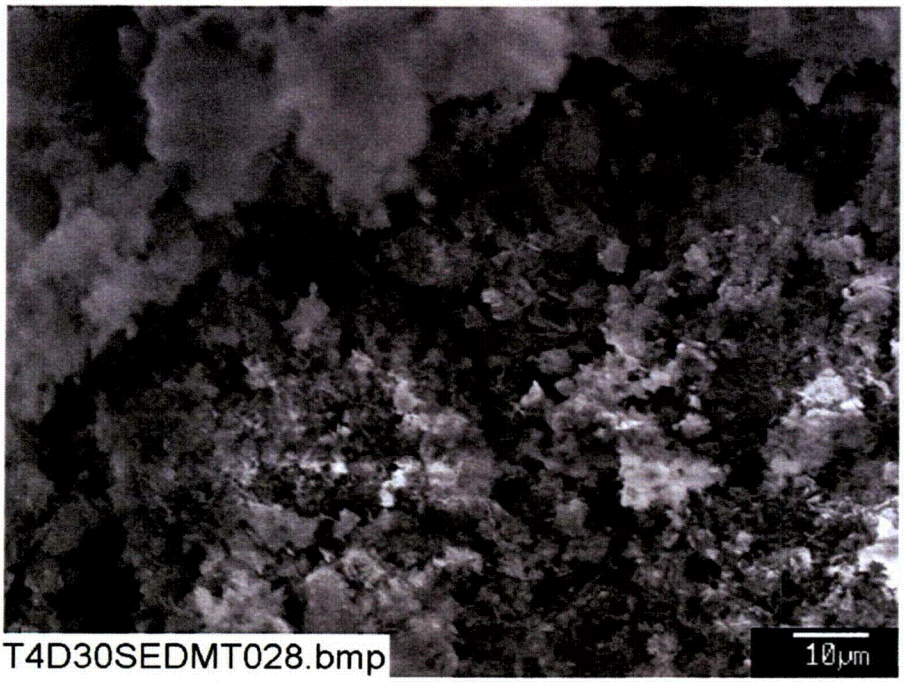


Figure G-5. **SEM image magnified 1000 times for a Test #4, Day-30 sediment at the bottom of the tank. (T4D30SEDMT028.bmp)**

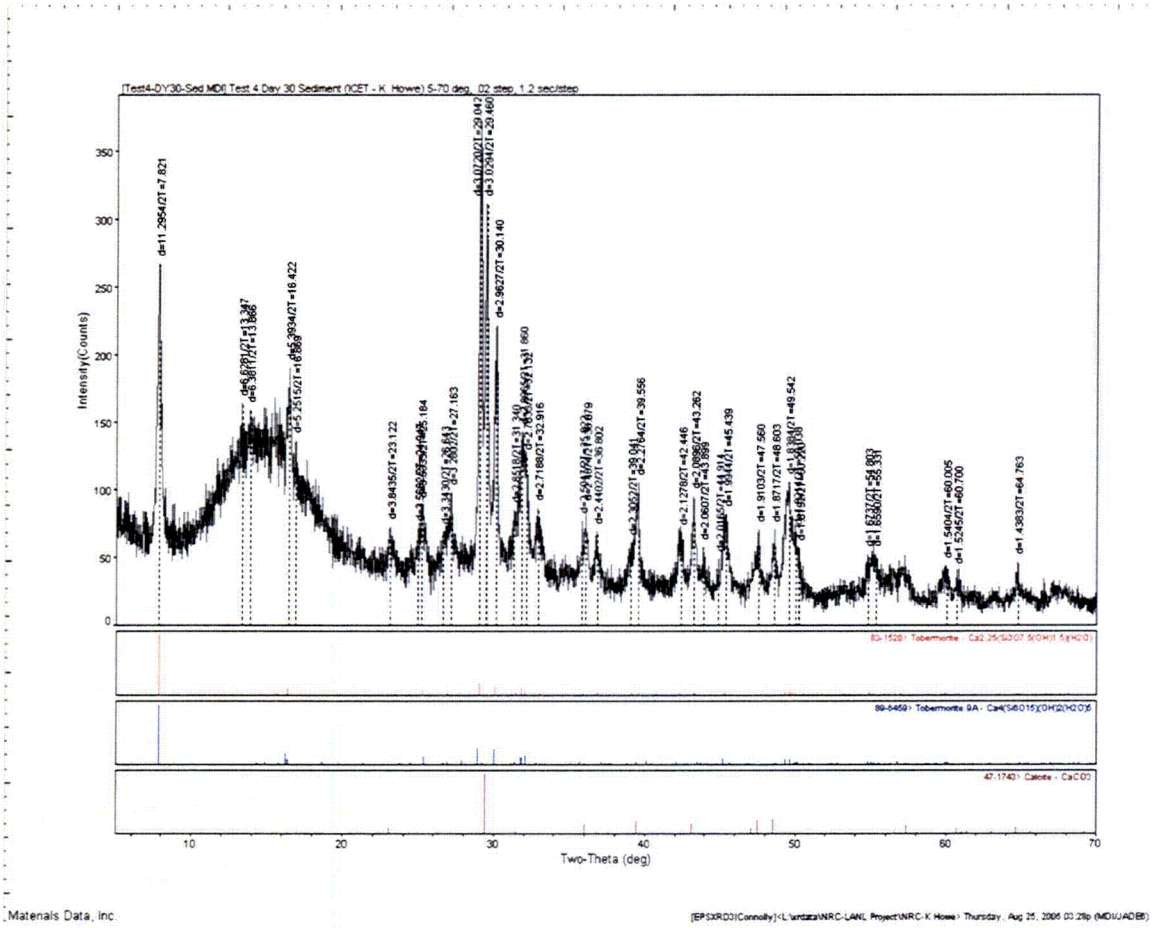


Figure G-6. **XRD result of the possible matching crystalline substances in Test #4, Day-30 sediment.**

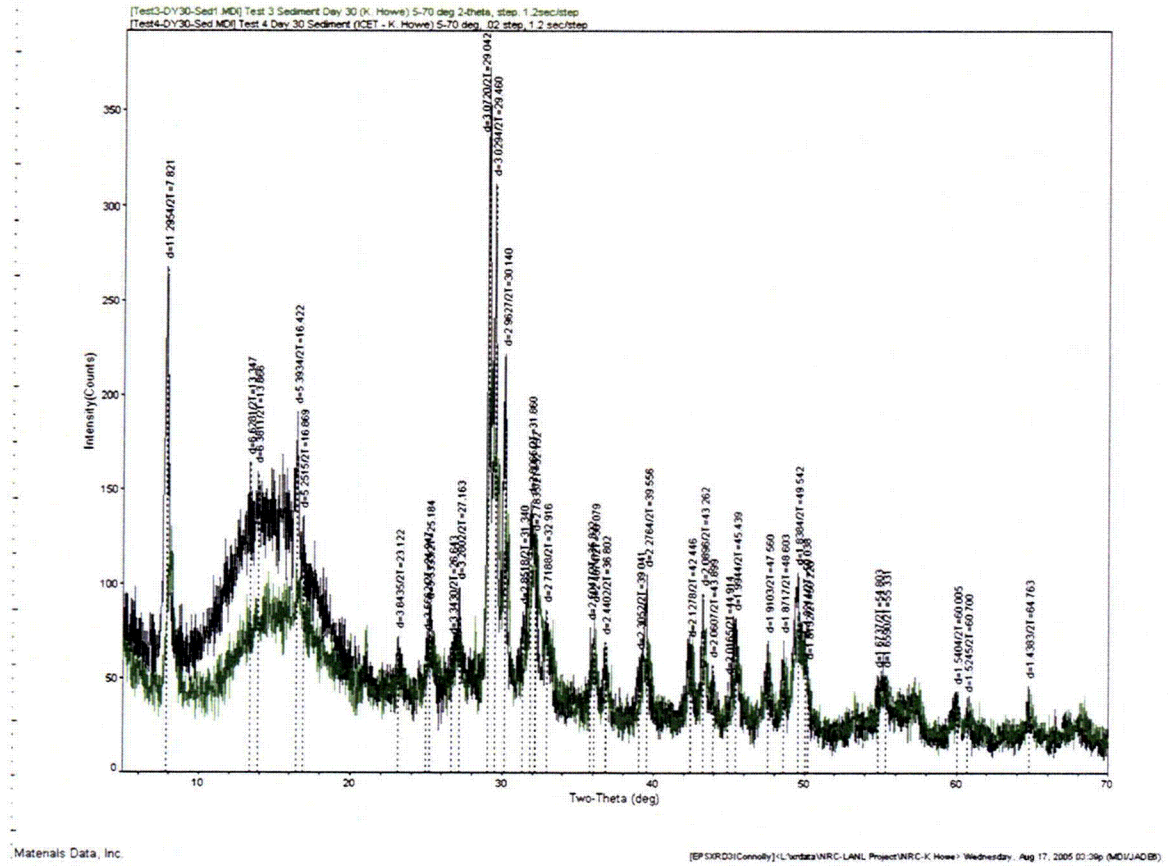


Figure G-7. **XRD results for the comparison between the Test #4, Day-30 sediment (black spectrum) and the Test #3 Day-30 sediment (green spectrum).**

Table G-3. Dry Mass Composition of Test #4, Day-30 Sediment by XRF Analysis (first row is compound; second row is mass composition in percent)

SiO_2	TiO_2	Al_2O_3	Fe_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	$H_20(-)$	$H_2O(+)$ CO_2	P_2O_5	Total	H2O(+) CO2 /DF (10) & Cover. To %
34.20	0.18	4.78	2.18	0.00	0.06	0.66	28.58	5.05	0.24	1.25	23.55	0.15	100.88	1.0241

Appendix H

TEM Data for Test #4 Solution Samples

Figures

This appendix presents TEM images and EDS results for Test #4, Day-4, Day-15, and Day-30 unfiltered solution samples. The unfiltered solution samples were extracted from the tank directly. A tiny drop of the testing solutions was transferred to a copper mesh, followed by drying in air for TEM analysis. The TEM and EDS results were obtained on June 27, 2005.

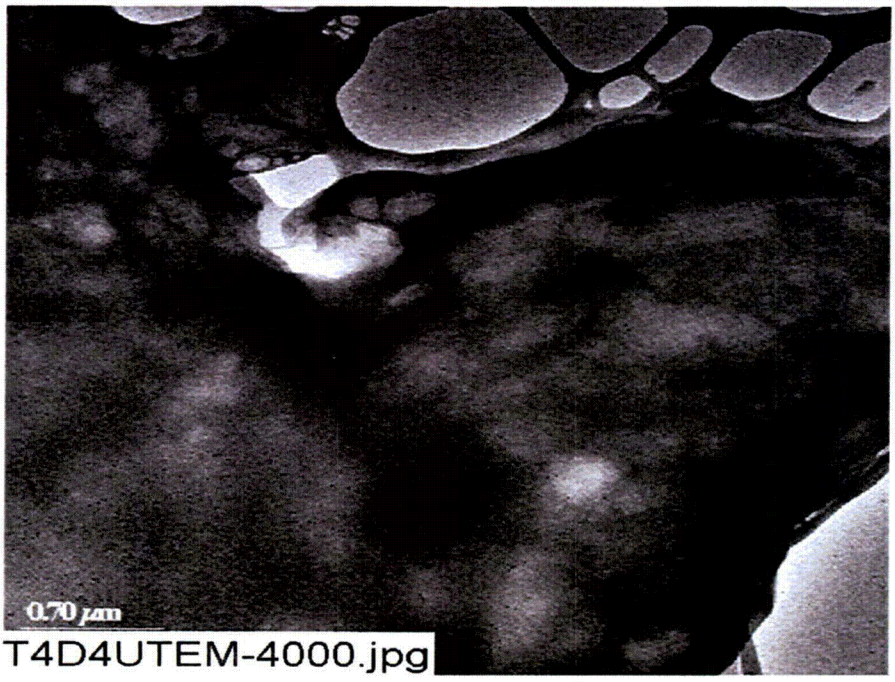


Figure H-1. **TEM magnified 4000 times for one Test #4, Day-4 unfiltered sample location. (T4D4UTEM-4000.jpg)**

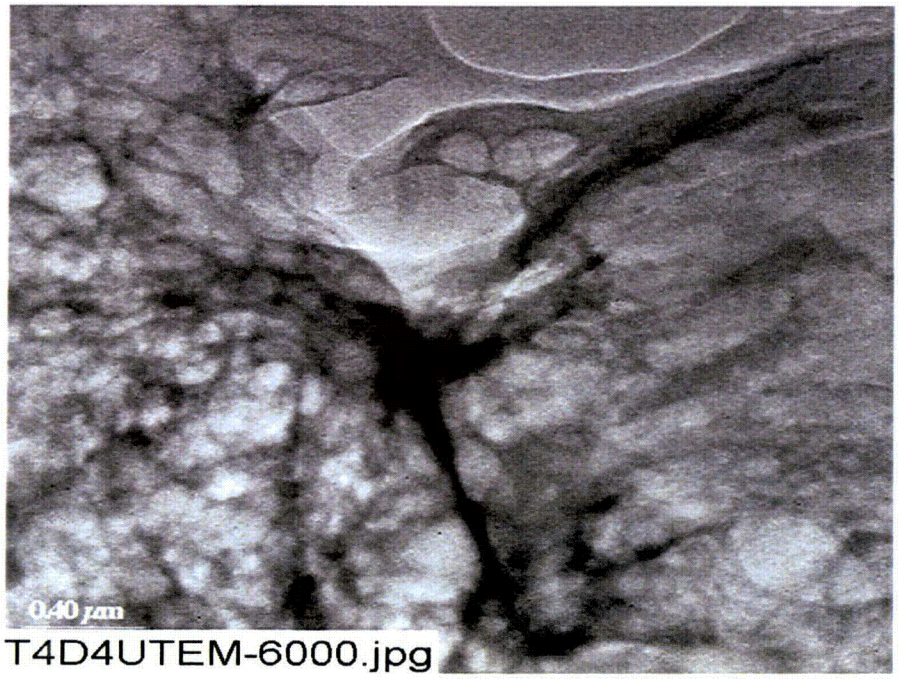


Figure H-2. **TEM magnified 6000 times for one Test #4, Day-4 unfiltered sample location. (T4D4UTEM-6000.jpg)**

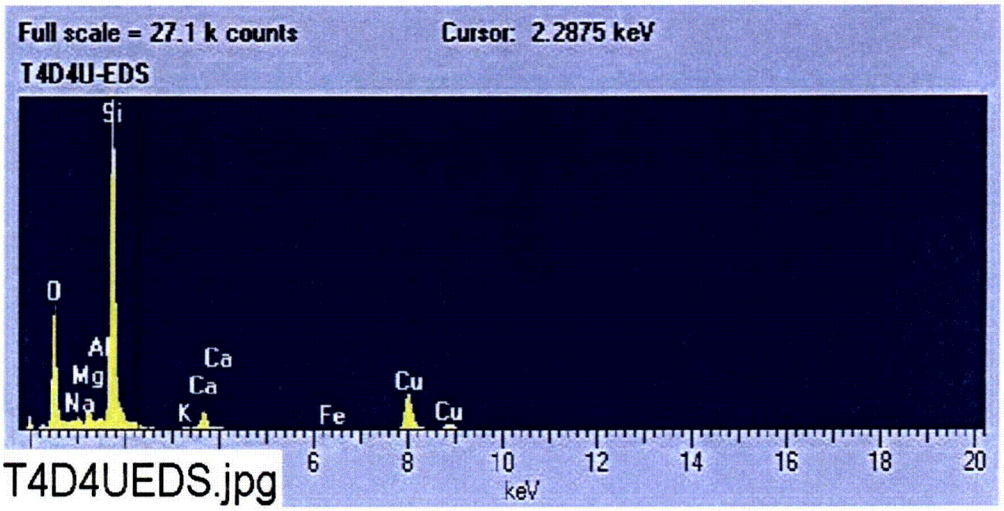


Figure H-3. **TEM energy-dispersive x-ray spectrum for a Test #4 Day-4 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T4D4UEDS.jpg)**

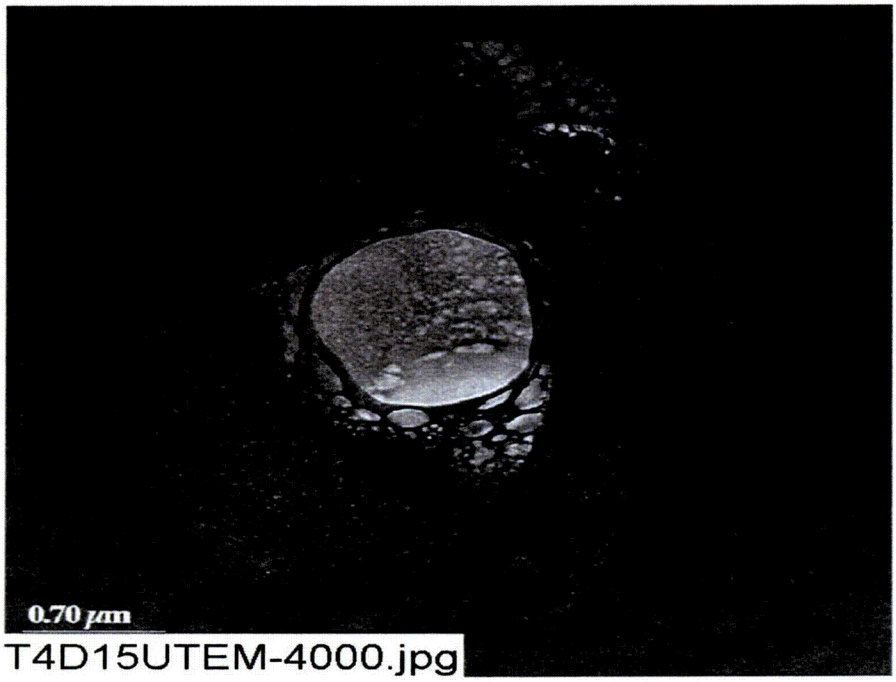


Figure H-4. **TEM magnified 4000 times for one Test #4, Day-15 unfiltered sample location. (T4D15UTEM-4000.jpg)**

Figure H-5. **TEM magnified 6000 times for one Test #4, Day-15 unfiltered sample location. (T4D15UTEM-6000.jpg)**

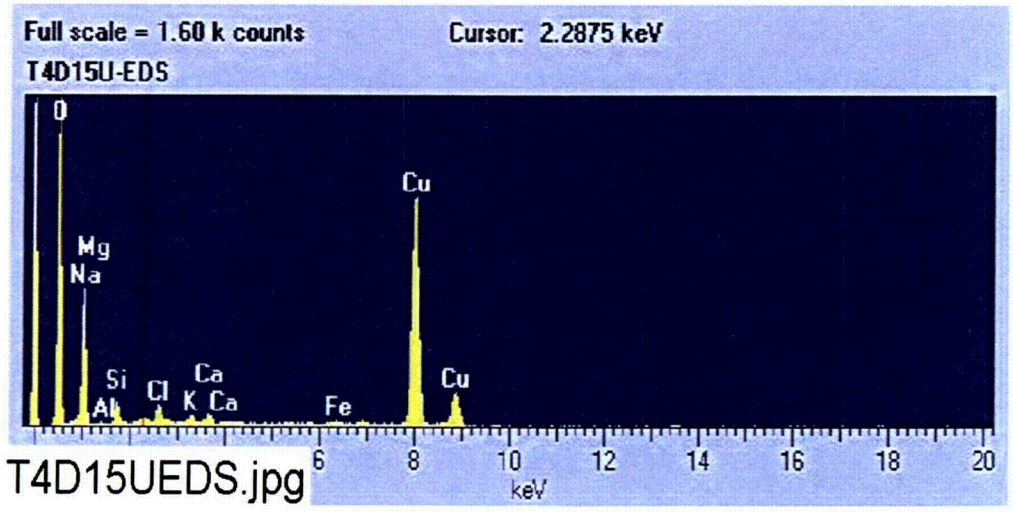


Figure H-6. **TEM energy-dispersive x-ray spectrum for a Test #4 Day-15 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T4D15UEDS.jpg)**

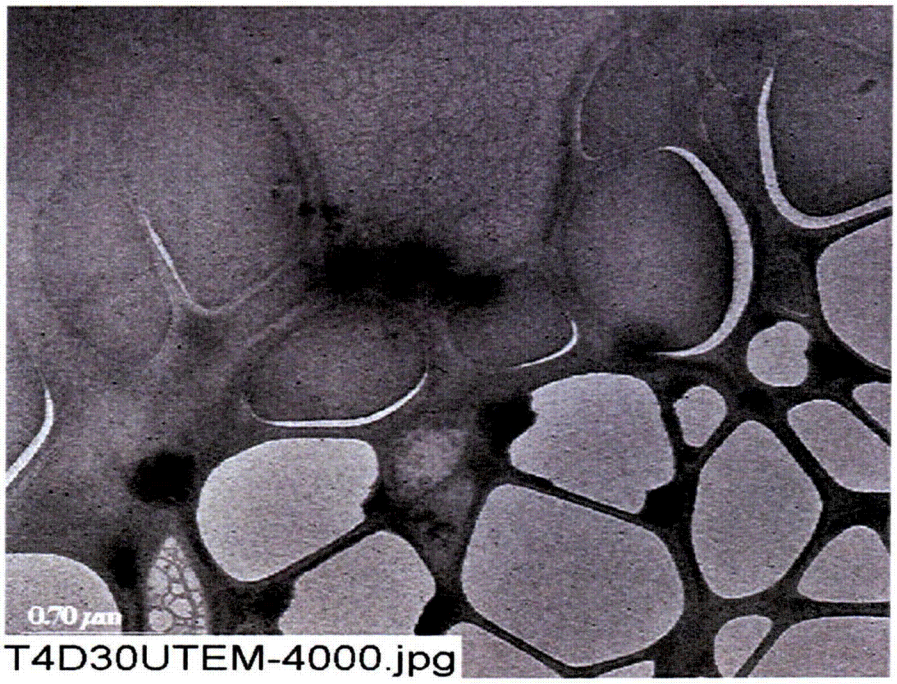


Figure H-7. **TEM magnified 4000 times for one Test #4, Day-30 unfiltered sample location. (T4D30UTEM-4000.jpg)**

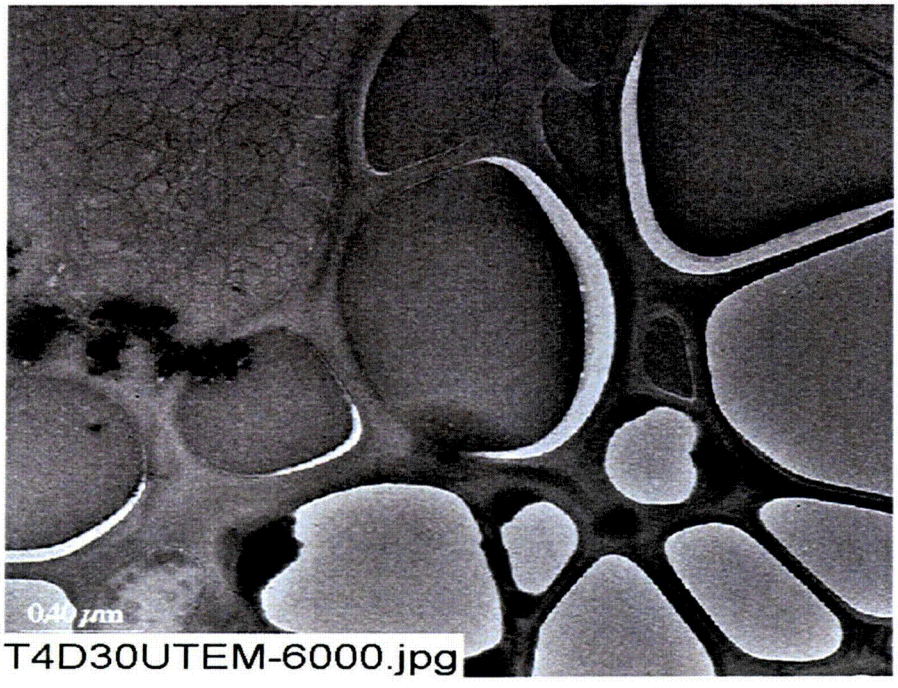


Figure H-8. **TEM magnified 6000 times for one Test #4, Day-30 unfiltered sample location. (T4D30UTEM-6000.jpg)**

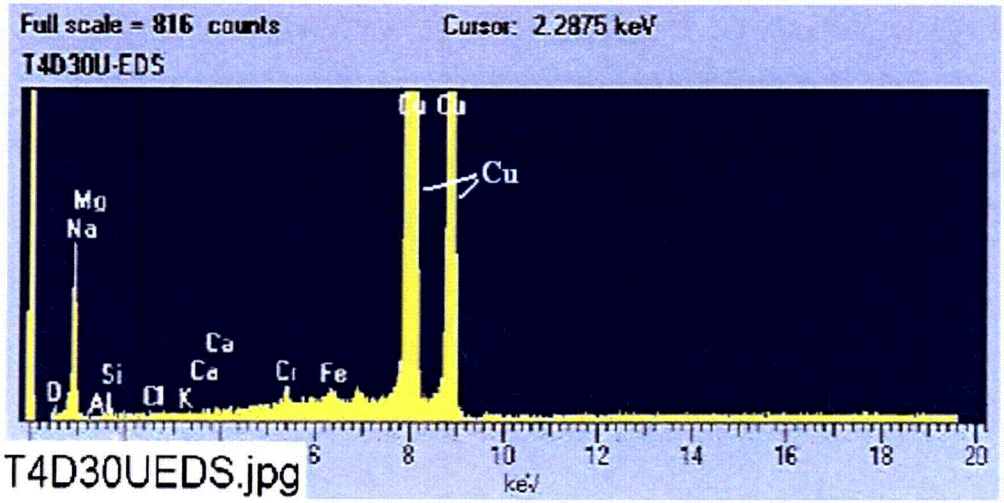


Figure H-9. **TEM energy-dispersive x-ray spectrum for a Test #4 Day-30 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T4D30UEDS.jpg)**

Appendix I

UV Absorbance Spectrum—Day-30 Solution Samples

Figures

Tables

This appendix presents the UV absorbance result of the Test #4, Day-30 solution sample. The purpose of this analysis was to find any distinguishing absorbance peaks to identify the organics present in the solution. The solution sample was collected through a 0.7-μm fiberglass filter at 60°C to remove particulate impurities, followed by being scanned over the wavelength ranging from 200 to 800 nm by a UV-visible spectrophotometer. The spectrum of DI water was used as background subtraction. From the result, no distinguishing absorbance peaks were found because of the well-mixed nature of the test solution.

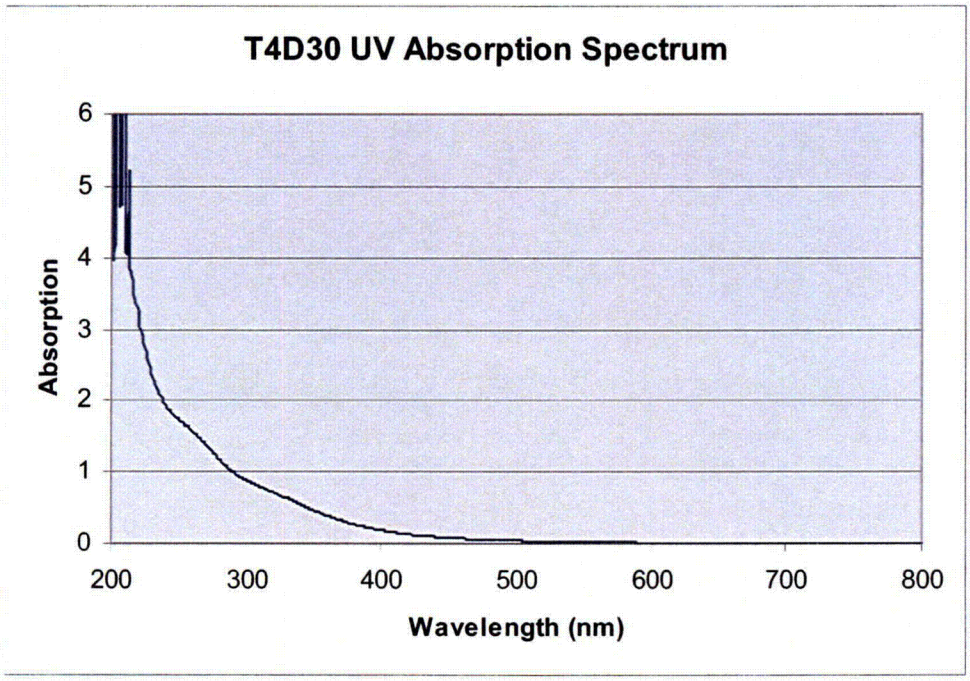


Figure I-1. **UV absorbance spectrum for Test #4, Day-30 solution samples.**

Table I-1. Test #4, Day-30 Solution Sample Laboratory Settings

Test #4, Day 30	
Collection Time:	7/21/2005 5:15:30 PM
Operator Name:	
Scan Software Version:	3.00(182)
Parameter List:	
Instrument:	Cary 50
Instrument Version:	3.00
Start (nm):	800.0
Stop (nm):	200.0
X Mode:	Nanometers
Y Mode:	Abs
UV-Vis Scan Rate (nm/min):	600.00
UV-Vis Data Interval (nm):	1.00
UV-Vis Ave. Time (sec):	0.1000
Beam Mode:	Dual Beam
Baseline Correction:	On
Baseline Type:	Baseline Correction
Baseline File Name:	
Baseline Std Ref File Name:	
Cycle Mode:	Off
Comments:	
Method Log:	
Method Name:	Default
Date/Time stamp:	7/21/2005 5:09:23 PM
Method Modifications:	
Cell Changer 6x6 Changed:	7/21/2005 5:09:27 PM/Old:1/New:0
UVVIS SAT Changed:	7/21/2005 5:10:05 PM/Old:0.0125/New:0.1000
NIR SAT Changed:	7/21/2005 5:10:05 PM/Old:0.0125/New:0.1000
Common SAT Changed:	7/21/2005 5:10:05 PM/Old:0.0125/New:0.1000
Baseline Correction Changed:	7/21/2005 5:10:10 PM/Old:0 / New:1
Temp Controller Changed:	7/21/2005 5:10:10 PM/Old:0 / New:2
Sipper Type Changed:	7/21/2005 5:10:10 PM/Old:Internal RSA/New:External Sipper
End Method Modifications	
<Current Wavelength>	200

Appendix J

ICET Test #4: Pre-Test, Test, and Post-Test Project Instructions

The ICET series is conducted under the guidance of project instructions (PIs), which identify the steps to follow for certain activities. These PIs are revised or rewritten as needed for each test. For Test #4, new PIs were written to address pre-test operations and test operations. The post-test operations PI was not changed from Test #3. These three PIs are included in this appendix to more completely describe the test apparatus and chemical solution preparations, the test startup and daily sampling, and the steps followed after test shutdown.

1.0 INTRODUCTION

1.1 PURPOSE

The purpose of this instruction is to ensure that all data acquisition, test samples, testing supplies, chemicals, and related materials are ready and accounted for prior to testing. In addition, this instruction provides instructions on preparing the chemical test apparatus for testing.

1.2 SCOPE

The pre-test operations preparation will ensure that successful initiation of the testing activity is achieved.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.c, March 30, 2005
- Chemical Additive Analysis Revisions – ICET-CALC-007, November 11, 2004; Test #4 Addendum, May 10, 2005
- Laboratory Safety Guidelines
- ASTM A 380 – 99, Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems
- Material Safety Data Sheets (MSDS) for all chemicals involved

2.0 PREREQUISITES

The data acquisition setup and inspection; instrument calibration; and the coupon receipt, preparation, inspection, and storage tasks must be completed in full prior to the completion of this activity. Fiberglass and calcium silicate (cal-sil) samples must be weighed and their planned locations in the tank identified. That data must be recorded.

2.1 Training Requirements

The following personnel training is required for this task:

1) LabVIEW and computer data acquisition training
2) Chemical handling training, specifically for ethyl alcohol, ammonium hydroxide, and lithium hydroxide.
3) Safe lift execution training

2.2 Equipment Requirements

The following equipment is required to perform this activity: computer with installed LabVIEW software, data acquisition system, and fully assembled and calibrated ICET test apparatus.

Safety equipment must be available: goggles, gloves, lab coats, eye wash station.

3.0 DOCUMENTATION REQUIRED

MSDSs must be available for all chemicals used.

A lab notebook must be maintained throughout the pre-test operations instruction. Contained within the lab notebook will be the date, times, description of activities, and quantities of chemicals added, number of cleanings, and physical observations of the tank cleaning and preparation procedures.

4.0 HAZARDS

The hazards associated with this activity include potential injuries associated with chemical handling.

5.0 INSTRUCTIONS

1. Ensure that all testing materials and supplies are ready and on-site. See checklist at the end of this document. Verify that eye wash station is operational. Note: The following solutions are not used in this instruction, but are to be prepared in advance of entering ICET-PI-016, "Test Operations, Test #4 (cal-sil, fiberglass, and NaOH at pH 10)." After preparation, clearly label the containers with the solutions and place in an area restricted for ICET Project test use.
2. Prepare 21. 2 g of concrete dust and 63.7 g of latent debris.
3. Prepare LiOH solution: dissolve 0.663 g of lithium hydroxide (LiOH) into about 100 mL water in a 250-mL sample container.
4. Prepare NaOH-LiOH-HCl solution.
   a. Add about 10 gallons of RO water to a 10-L polypropylene container.
   b. Add 212 mL of 12.24 N HCl to the water in the container.
   c. Dissolve 0.663 g of LiOH into the water in the container.
   d. Dissolve 8.47 kg of NaOH into the water in the container. Add the NaOH slowly and mix until dissolved so the solution does not over-heat.
   e. Properly label and store the container.
5. Prepare NaOH solution for spray nozzle feed.
   a. Add about 0.5 gallons of RO water to a 1-gallon polypropylene container.

b. Dissolve 614 g of NaOH into the water in the container. Add the NaOH slowly and mix until dissolved so the solution does not over-heat.
c. Dilute with additional RO water until the volume is 1 gallon.

6. Prepare laboratory control sample (LCS). See ICET-PI-005, "Chemical Sampling and Analysis," for details on the laboratory control sample.
7. Start the data acquisition system. Verify that the data acquisition system is monitoring flow rate, pump speed, temperature, and pH.
8. Clean the tank and piping.
   a. Cleaning should commence as soon after a test is completed as possible, to prevent material from hardening in the tank or piping and to maximize the time available for cleaning.
   b. Cleaning chemicals may consist of weak acids (e.g., acetic acid, citric acid, or dilute mineral acids), weak bases (e.g., ammonium hydroxide), week organic solvents (e.g., ethanol), or detergents/surfactants (e.g., trisodium phosphate, sodium dodecyl sulfate), as necessary. Cleaning solutions can be heated if necessary. Note that the discharge limit to the sanitary sewer is a maximum temperature of 140 °F and pH between 5.0 and 11.5. Cleaning solutions that are not within this range should be neutralized before discharge.
   c. During cleaning, the pump should be run and water directed through both recirculation lines (through the spray nozzles and lower headers)
   d. The sample line should be removed from the piping, physically cleaned, and carefully inspected. If the sample line cannot be adequately cleaned, it should be replaced.
   e. After each cleaning step, the tank and piping should be thoroughly rinsed with tap water or demineralized water.
   f. After each cleaning step, a segment of pipe should be removed, and the interior of the pipe visually inspected.
   g. Cleanliness criteria: When the tank visually appears to be satisfactorily cleaned, the tank and piping should be thoroughly rinsed with demineralized water. The interior surfaces of the tank and piping shall be free of any deposits that can be removed by vigorous scrubbing. Demineralized water drained from the tank should have turbidity less than 0.3 NTU and conductivity less than 50 uS/cm.
9. Tank is now ready for testing. Proceed immediately to Instruction No. ICET-PI-016, "Test Operations, Test #4 (cal-sil, fiberglass, and NaOH at pH 10).

6.0 ATTACHMENTS

No forms are attached to this document.

7.0 Materials Checklist

_____ lithium hydroxide, 0.663 g

_____ NaOH pellets, 9.085 kg

_____ 212 mL of 12.24 N HCl

_____ tap water supply

_____ demineralized water production system

_____ chemical handling safety equipment (lab coat, goggles, rubber gloves)

_____ analytical balance

_____ top loading balance

_____ chemical spatula

_____ chemical scoop

_____ weigh boats

_____ two 5-gallon plastic containers

_____ 250 mL graduated cylinder

_____ 250-mL HDPE or PP bottle

_____ 2.5 gallons ethanol

_____ 2.5 gallons ammonium hydroxide

_____ turbidimeter and associated equipment

_____ conductivity meter and associated equipment

1.0 INTRODUCTION

1.1 PURPOSE

The intent of the instruction is to outline the steps that are to be followed during testing.

1.2 SCOPE

This activity forms the core of the entire Chemical Effects Testing project. All activities involved in this project affect and are affected by this activity.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.c, March 30, 2005
- ASTM Standard G 4-01
- ASTM Standard D 3370-95a
- ASTM Standard G 31-72
- Material Safety Data Sheets (MSDS) for all chemicals involved
- LabVIEW operation manual
- Laboratory Safety Guidelines
- Chemical Additive Analysis Revisions – ICET-CALC-007, November 11, 2004; Test #4 Addendum, May 10, 2005. Note: This addendum used a target pH range of 9.7-10.3.
- John Gisclon email to Bhagwat Jain, Cal-sil Information Used in Test 3, March 31, 2005

2.0 PREREQUISITES

All sample coupons must be placed in their corresponding racks. Also, the pre-operation test preparation activity must be completed in full.

2.1 Training Requirements

The following personnel training is required for this task:
1) LabVIEW and computer data acquisition training.
2) Chemical handling training for all chemicals involved.

2.2 Equipment Requirements

The following equipment is required to perform this activity: computer with installed LabVIEW software, data acquisition system, and fully assembled and calibrated ICET test apparatus.

Safety equipment must be available: goggles, gloves, lab coats, hard hats, steel-toed shoes, eye wash station, hydrogen detector and hydrogen removal system.

3.0 DOCUMENTATION REQUIRED

A lab notebook must be maintained throughout the testing procedure. In addition, a binder will be maintained that includes pertinent test instructions and the completed daily log sheets (see Attachment A). The daily log sheet contains the date, times, physical description, and quantity of fiberglass and water samples obtained each day. In addition, the daily log sheet contains information from the data acquisition system (DAS), the water samples taken, and other test information.

The electronic data that are acquired are backed up daily and stored in a separate location each testing day. Refer to ICET-PI-001, Data Acquisition Setup and Inspection.

4.0 HAZARDS

The hazards associated with this activity include tipping of the chemical tank assembly, ingestion and/or respiration of any chemicals involved, and scalding and/or burning hazards involved in daily tank venting, and possible hydrogen gas generation from corrosion reactions. Appropriate measures to control hydrogen gas must be in place before operations commence.

Lifting hazards associated with the tank lid and coupon racks are also associated with this activity.

5.0 INSTRUCTIONS

1. Because of the time required for heating the tank contents and dissolving chemicals, this sequence should be started at least 48 hours before the scheduled time t = 0. Pre-test operations preparation should be complete before proceeding with this sequence.
2. Ensure that all testing materials and supplies are ready and on-site (see checklist at end of this instruction).
3. Add 239 gallons of RO water to the tank by pumping water from the RO skid through the totalizing flow meter. Record flow to the nearest 0.5 gallon.
4. Verify valves are positioned as follows:

Valve	Description	Position
V-1	tank drain	closed
V-2	pump isolation	open
V-3	instrument loop supply	open
V-4	instrument loop discharge	open
V-5	instrument loop bypass	closed
V-6	in-line filter isolation	open
V-7	recirculation line supply	open
V-8	tank spray nozzle supply	closed
V-9	sample line	closed
V-10	recirculation line injection	closed

5. Start pump and adjust to flow rate of approximately 25 gpm.
6. Start computer, start LabVIEW, verify that flow rate, pump speed, temperature, and pH are being recorded properly.
7. Turn on heater and allow water in tank to heat to 60 °C ± 2 °C. (This may take up to 20 hours.)
8. Add the pre-mixed NaOH-LiOH-HCl solution.
9. Add 15.14 kg of boric acid (H_3BO_3), weighing in approx. 2 kg increments, recording the weight of each increment to the nearest 10 g.
10. Allow the water to circulate until the solution is visibly clear, indicating that the boric acid is completely dissolved.
11. Allow water in tank to heat to 65 °C ± 2 °C.
12. Take grab water sample for analysis for the parameters identified in steps a – h below. Also record physical appearance of the sample (clarity, presence of gelatinous material, etc). All Day 1 and subsequent samples will be analyzed by Assaigai Analytical Laboratory. In addition, periodic test samples and laboratory control samples (LCSs) will also be analyzed by the UNM laboratory.
    a. pH
    b. temperature
    c. turbidity
    d. viscosity
    e. total suspended solids (TSS)
    f. dissolved oxygen (DO)
    g. chloride
    h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved
13. Add 21.2 g of concrete dust and 63.7 g of latent debris samples (prepared earlier), wait 10 minutes, take 100 mL water sample for particulate size distribution, density, and TSS.
14. Stop pump.
15. Add the pre-determined amount of cal-sil dust. This will be approximately 43.5 lb.
16. Place coupon racks, fiberglass holders, and cal-sil holders into tank. This is done in accordance with previously determined quantities, size distributions, and locations.

(Details of the cal-sil preparation and size distributions are given in the referenced email.)

17. Verify locations of coupon racks, fiberglass holders, and cal-sil holders.
18. Verify the tank temperature is 62 °C. (Because the tank lid may have to be removed to unplug nozzles, the test will be started with the water temperature at its upper limit.)
19. Start pump and adjust pump speed to 25 gpm.
20. Open valve V-8 (tank spray nozzle supply) to direct water to nozzles and adjust valves V-7 (recirculation line supply) and V-8 (tank spray nozzle supply) until nozzle flowmeter is reading 3.5 gpm. Verify total flow is still 25 gpm and adjust variable frequency drive (VFD) if necessary.
21. Record date and time at which nozzle flow started. This is time t = 0 for the test.
22. Note that the spray phase will begin with the crane attached to the tank lid. The objective is to be able to carefully monitor possible nozzle blockage and take immediate action to prevent it. At the first sign that a nozzle may be starting to plug, the spray flow rate should be increased rapidly to 5-10 gpm for approximately 5 s. (As long as the nozzle spray pattern is not affected however, the spray flow should remain at 3.5 gpm.) If a nozzle should block in spite of the increased flow rate, the tank lid should be removed and a stainless steel wire used to clear the nozzle exit. The tank lid must be replaced as soon as possible to limit the expected temperature decrease.
23. Flow through the nozzles should be monitored every 5 minutes by looking through the tank view windows. After 15 minutes, if there have been no spray nozzle blockages, the monitoring frequency can be increased to every 15 minutes. (Note that the spray flow pattern from each nozzle can be observed through the view windows and restrictions to the spray pattern are readily observable.)
24. At t = 0, start chemical metering pump and inject pre-measured NaOH solution into the spray line. The objective here is to add the 1-gallon NaOH solution in 30 minutes.
25. At 30 minutes, shut off chemical metering pump and isolate this line.
26. Take a measurement of hydrogen concentration. At 2-hour increments, repeat the hydrogen concentration measurement. If the concentration reaches 10% of the flammability limit, purge the tank atmosphere. This needs to be repeated until the hydrogen concentration has been determined to be below 10% of the flammability limit, and then the frequency of hydrogen concentration measurements is to be re-evaluated.
27. At t = 4 hours, stop the spray flow by closing valve V-8.
28. At any time following the spray phase, the crane may be removed from the tank lid.
29. After closing valve V-8 (at t = 4 hours), take water grab sample for analysis for the parameters listed below. Record the time of sample collection.
    a. pH
    b. temperature
    c. turbidity
    d. viscosity

e. chloride
f. total suspended solids (TSS)
g. dissolved oxygen (DO)
h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved.

30. At t = 24 hours, and daily thereafter, take water grab sample for analysis for the parameters listed below. (The LANL PI will propose a different sampling frequency to the project sponsors if test data support it.) Record the time of sample collection.
    a. pH
    b. turbidity
    c. viscosity
    d. temperature
    e. total suspended solids (TSS)
    f. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved. An exception is that B, Li, K, Pb, and chloride analyses will be performed only at t = days 15 and 30. Also, dissolved oxygen will be measured at day 30.
31. During each daily water sample collection, look inside tank (through windows) and record observations. If the tank water level indicates that the water volume is 245 gallons or less, add RO water to bring the volume up to 250 gallons and record the amount added.
32. At t = 24 hours, weekly thereafter, and at the end of the test, collect 100 mL water sample for particulate size distribution and density analysis, to be performed at AALI. The particulate size ranges to be used will be as close as possible to those called out in the test plan: (in microns), 1-10, 11-25, 26-50, 51-75, 76-100, and > 100 microns.
33. At t = 24 hours, weekly thereafter, and at the end of the test, collect water samples for strain rate viscosity measurements (see PI-010 for sample details.)
34. After 2 to 5 days of testing, it is anticipated that the solution will be stable and no suspended particles will be visible. If that is the case, insert the three types of fiberglass samples described in Attachment B. Note that one of the samples (long, narrow stainless steel holder) is to be placed in front of the water distribution headers, and the others are to be placed behind the headers. (The date and time of the addition of these samples will be recorded in the lab notebook.)
35. At 3 days $\leq$ t $\leq$ 5 days, 14 days $\leq$ t $\leq$ 16 days and at the end of the test, collect a sacrificial fiberglass sample to be inspected and examined with SEM.
36. At 24 hours, at 14 days $\leq$ t $\leq$ 16 days and at the end of the test, run 1L of water through a nucleopore filter. The filter will be taken for SEM analysis as specified in ICET-PI-007. (Note that depending on the solution, some filter material will not work well for this operation. If possible, use a nucleopore filter for SEM analysis, and then collect a second sample on nitrocellulose filter for later digestion and ICP analysis.)
37. Shut down pump
38. Indicate end of test on the data acquisition system and shut down the data acquisition software.
39. Proceed directly to PI-008 Post-Test Operations.

6.0 ATTACHMENTS

Attachment A. Daily Log Sheet

Attachment B. Test #4 Fiberglass Sample Addition after Test Start

7.0 MATERIAL CHECKLIST

_____ boric acid, 15.14 kg

_____ pre-mixed NaOH-LiOH-HCl solution

_____ pre-mixed NaOH solution

_____ concrete dust, 21.2 g

_____ latent debris, 63.7 g

_____ Nucleopore filter

_____ chemical handling safety equipment (lab coat, goggles, rubber gloves)

_____ top-loading balance

_____ weigh pan for 2 kg aliquots of boric acid

_____ stainless steel filter paper holder

_____ 500 mL graduated cylinder (for TSS)

_____ totalizing flow meter

_____ sample containers (see Chemical Sampling Instruction)

_____ analytical equipment (see Chemical Sampling Instruction)

_____ pre-assembled coupon racks

_____ pre-assembled fiberglass baskets, total of 2.2 lb of fiberglass

_____ pre-assembled cal-sil baskets, total of 26.7 lb of cal-sil

_____ pre-measured cal-sil dust, 43.5 lb

_____ coupon handling safety equipment (hard hat, leather gloves, boots)

_____ computer disks for backup of Labview data

_____ Masterflex peristaltic pump and tubing

_____ demineralized water production system

Attachment A. Daily Log Sheet

Daily Log Sheet
Integrated Chemical Effects Test (Test # 4)

Date: _____ Time of sample collection: _____
Sample taking and data reduction by _____ and _____

Sample bottle identification:
Assaigai (total): _____
Assaigai (filtered): _____
UNM (total): _____
UNM (filtered): _____

Control system readings:
Temperature: _____ Flow: _____ pH: _____

Analyses:
Volume filtered for TSS: _____ pH: _____
Temperature: _____ Dissolved oxygen: _____
Turbidity (at 60 °C): _____ (at 23 °C; and 10 min.)_____
Viscosity, unfiltered (60 °C): _____ (at 23 °C) _____
Viscosity, filtered (60 °C): _____ (at 23 °C) _____
Water Level: _____ Water Added: _____
Hydrogen: _____ Other: _____
Fiberglass or other samples taken: _____
TSS filter #: _____ TSS (mg/L): _____

Comments:

Observations written in lab notebook by _____
☐ Continued on back

Attachment B. Test #4 Fiberglass Sample Addition after Test Start

Recent experience gained in ICET Test #3 with adding large quantities of Calcium Silicate (Cal-Sil) debris to the tank suggests that contamination of fiber samples with suspended particulate may complicate the post-test identification and analysis of chemical products that may be contained within the samples. Past experience also suggests that the circulating tank solution will clarify after 1 to 2 days, providing an opportunity to introduce fiber samples for immersion in the chemical environment without substantially shortening the exposure time and while avoiding the complications of Cal-Sil contamination. (Several other fiber samples are immersed at the initiation of the test so that they are directly exposed to large quantities of the Cal-Sil particulate).

This attachment to ICET-PI-16, Rev 0 addresses the addition of three types of containers for fiberglass samples that are to be inserted in the test solution between Days 2 and 5 after substantial water clarity has been achieved and as needed to match operations schedules. These containers include: (A) a long (5-6 in.), thin (1/4 to ½ in.), narrow (1-2 in.) stainless steel mesh envelope that will be placed in front of a discharge hole on one of the water distribution headers (a "high-flow" area of the tank); (B) a nylon mesh envelope of approximately 4 to 6 inches square containing 5 to 10 g of fiberglass, and (C) 3 to 5, two-inch diameter pucks of fiberglass that are prepared in rings of CPVC and encased in typical envelopes of stainless steel mesh. Each container will hold less than 10 g of fiberglass and their fiberglass contents are considered negligible with respect to the total test amount of fiberglass. The nylon mesh envelope and pucks will be placed in "low-flow" areas of the tank behind the water distribution headers. Technical descriptions and justifications for each item follow.

Test Item (A): thin sample in high flow

This sample may provide evidence of whether chemical deposits are enhanced or inhibited by the direct impingement of water flow. The large aspect ratio of this envelope (length/width) is designed to avoid large perturbations in the inlet flow patterns that would occur by placing a large flat object near the distribution headers. Post-test examination of this sample will be made using typical ESEM and SEM/EDS survey techniques.

Test Item (B): nylon mesh envelope

ICET Test #1 results indicated a possible preference for chemical deposits to form near the interface between fiberglass and the stainless steel mesh that was used to form the sample envelopes. The introduction of a nonmetallic casing material may permit a comparison of this effect under exposure to a similar chemical environment. Nylon mesh was selected as a suitably inert material for constructing an envelope for this fiber. Based on qualitative assessments and recommendations of material performance made by chemical supply vendors (see for instance, chemical resistance information given at

www.eldonjames,com/html), nylon is expected to exhibit "Excellent" resistance to NaOH solutions of up to 50% concentration and "Good" resistance to industrial concentrations of boric acid. This container material has not been submitted for independent bench-scale leaching tests in the ICET solution. The small quantity of foreign material is not expected to perturb interpretation of results from ICET Test #4 regardless of its performance characteristics. Post test examination of this sample will be made using typical ESEM and SEM/EDS survey techniques.

Test Item (C): Fiber pucks

The MOU established between NRC and EPRI for conduct of the ICET series specifically excludes modification of the apparatus for the purpose of obtaining in-line flow head loss data. However, the presence of chemical deposits observed on and within fiberglass samples obtained from ICET Test #1 to Test #2 raises questions regarding the potential of these products to impede water flow and about their behavior under flowing conditions, for example, whether they will be adherent or wash out of the fiber matrix. Unanswered questions also remain regarding the possible formation of deposits in the presence of flow, but without a direct mechanism of studying formation under flow, it may be useful to examine whether the deposits can form in fiberglass that represents a prototypical debris bed. Two of the attributes that may distinguish fiberglass on a debris bed from fiberglass in a debris flock are (a) degree of mechanical separation between fibers, and (b) degree of compaction in the bed.

Between 3 and 5 fiberglass pucks will be prepared as shown in Figure 1 for jacketing in a stainless steel envelope and immersion in the Test #4 test solution after the water clarity has improved. Approximately 5-10 grams of dry fiberglass are required to fill the ½-inch thick, 2-in. diameter sample ring. The sample ring is cut from a 2-inch diameter CPVC pipe to provide a standard dimension for any flow testing that may be desired after the conclusion of Test #4 and to avoid the introduction of unapproved foreign materials in the test tank. Before introducing the fiberglass to the mold, it will first be agitated in a kitchen blender for at least 2 minutes in two batches, each batch containing approximately half the debris and approximately 2 quarts of water. The purpose of agitation is to separate fibers from the raw flocks of manufactured insulation. The batches will be sequential poured into the mold placed across a mesh screen. Gentle manual tamping may be required to ensure uniformity of the bed.

Figure 1. Example fiber pucks prepared for immersion in ICET test solution.

The introduction of fiberglass pucks will provide the following technical opportunities for post-test examination:

1) Any observed chemical deposits will be relatively free from contamination of Cal-Sil.
2) ESEM and SEM/EDS examines can be made for the presence of deposits inside of a relatively compact debris bed.
3) If exams 1 and 2 are positive, the pucks will provide a concentrated quantity of the deposit for possible extraction, isolation and identification.
4) The pre-measured dry mass of the fiber may permit a determination of dry mass for any deposited chemical products. This may provide a first step towards quantifying rates and quantities of formation.
5) The convenient form of the debris in the mold will facilitate any head loss testing that is deemed interesting or necessary as a post-test analysis activity. Samples of this type could be placed either within a continuously circulating closed loop or within a static head drain column for measurement of flow loss. The primary objective of any such examinations would be the direct comparison of fresh, identically prepared samples with cultured samples that have been exposed to the test environment. Expected variability between the samples suggests that several replicates should be prepared for comparison. Any work of this type will be conducted under a separate approved procedure.

<u>Note:</u> The purpose of this attachment is to describe the samples to be added to Test #4 after test initiation. Any post-test evaluations of these samples other than ESEM and SEM/EDS will be done under separate procedure/project instructions. In

addition, any post-test head loss testing will require appropriate documentation and sponsor approvals.

1.0 INTRODUCTION

1.1 PURPOSE

The intent of this instruction is to ensure that the experimental samples are removed from the test apparatus, the test apparatus is rinsed and inspected, and the test apparatus is made ready for subsequent pre-test operations.

1.2 SCOPE

This activity marks the end of one chemical effects test run. Experimental sample removals and inspections, test apparatus rinsing, and preparations for cleaning and subsequent tests are addressed here.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.c, March 30, 2005
- ASTM Standard G 4-01
- ASTM Standard G 31-72
- ICET-PI-002, Coupon Receipt, Preparation, Inspection, and Storage, November 19, 2004
- ICET-PI-014, Rev. 0, Test Operations, Test #3 (cal-sil and fiberglass, with TSP, April 5, 2005
- ICET-PI-005, Rev. 1, Chemical Sampling and Analysis, February 3, 2005
- Laboratory safety guidelines
- ICET Project Safety Plan

2.0 PREREQUISITES

All test operation PI criteria must be completed prior to conducting this task.

2.1 Training Requirements

- Laboratory Safety Guidelines
- ICET Project Safety Plan

2.2 Equipment Requirements

A city tap water supply outlet is required for this activity and chemical handling and lifting safety equipment. A reverse osmosis unit is required for the final flush.

3.0 DOCUMENTATION REQUIRED

Documentation related to test parameters, chemical water analyses, coupon and fiberglass examinations, and daily test operations are outlined elsewhere. In this instruction, the steps required to remove samples from the test apparatus and to make it ready for the next test are outlined. In addition, observations as to the test apparatus' condition are obtained and recorded here.

4.0 HAZARDS

The hazards associated with this activity include ingestion/respiration and/or dermal and eye contact with residual chemicals. Lifting hazards associated with the tank lid and coupon racks are also associated with this activity.

5.0 INSTRUCTIONS

1) On the last day of testing, collect water samples and perform analyses as outlined in ICET-PI-014 and ICET-PI-005.
2) Remove 10L of water from the test apparatus and store at test temperature, for future analyses
3) Shut off the recirculation pump.
4) Remove the small fiberglass samples for SEM examination.
5) Leave one heater on and continue to monitor tank water temperature.
6) Isolate and drain the test apparatus piping.
7) Remove the tank lid.
8) Before removing coupon racks or insulation samples, examine and take photographs and notes of the inside of the tank, the coupons and racks, and the insulation samples.
9) Remove the six non-submerged coupon racks to a staging area for drying and post-test examinations (refer to ICET-PI-002).
10) Take additional photographs of the inside of the tank.
11) Drain the tank slowly, down to the level that uncovers the submerged rack, but keeping the water level above the heater.
12) Remove the submerged coupon rack to the staging area.
13) Repeat step # 10.
14) Turn off the heater.
15) Completely drain the tank, taking precautions so that the sediment on the bottom of the tank is not disturbed any more than necessary.
16) Store water that was drained from the test apparatus until it is cleared for disposal or shipment. (This step was just moved from later in the PI – the old step #26.)
17) When the tank is drained, repeat step # 10. Note especially the locations and orientations of the remaining samples.
18) Remove the remaining insulation samples to the staging area to dry.

19) Ensure that all samples removed from the tank are clearly marked as to their location and orientation within the tank.
20) After all samples have been removed, repeat step # 10.
21) Inspect the interior of the tank, noting any observations.
22) Note the presence of any sediment. Carefully remove as much sediment as possible, noting any unique aspects of it, such as location. Place the sediment in plastic containers with lids, marking the location of the sediment in the tank.
23) Remove the tank drain screen and remove the insulation sample for future analysis.
24) Remove the flow meter from the loop and take pictures of the flow meter interior.
25) Remove any deposits within the flow meter and place the deposits in plastic containers with lids. This is to keep the samples hydrated.
26) Remove a section of pipe, take pictures of the pipe interior, and remove and store any deposits there.
27) Replace the flow meter and piping section.
28) Rinse the tank with tap water and drain the water.
29) Fill the system with 250 gallons of tap water and circulate water through the spray nozzles and recirculation headers for at least 60 minutes. Repeat with de-mineralized water.
30) If any signs of deterioration are observed on the inside of the test apparatus tank, remove selected insulation on the tank. Inspect the stainless steel tank for any abnormalities.

6.0 ATTACHMENTS

No forms are attached to this document.

NRC FORM 335 (9-2004) NRCMD 3.7

U.S. NUCLEAR REGULATORY COMMISSION

BIBLIOGRAPHIC DATA SHEET

(See instructions on the reverse)

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J. Dallman, B. Letellier, J. Garcia, J. Madrid, W. Roesch; Los Alamos National Laboratory

D. Chen, K. Howe; University of New Mexico

L. Archuleta, F. Sciacca; Omicron Safety & Risk Technologies, Inc.

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Los Alamos National Laboratory
PO Box 1663
Los Alamos, NM 87545

University of New Mexico
Department of Civil Engineering
Albuquerque, NM 87110

Omicron Safety & Risk Technologies, Inc.
2500 Louisiana Blvd., Suite 410
Albuquerque, NM 87199-3065

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Division of Fuel, Engineering & Radiological Research
Office of Nuclear Regulatory Research
U.S. Nucelar Regulatory Commission
Washington, DC 20555-0001

10. SUPPLEMENTARY NOTES

B. P. Jain, NRC Project Manager; prepared in cooperation with Electric Power Research Institute

11. ABSTRACT *(200 words or less)*

A 30-day test was conducted in the Integrated Chemical Effects Test (ICET) project test apparatus. The test simulated the chemical environment present inside a pressurized water reactor containment water pool after a loss-of-coolant accident. The initial chemical environment contained 15.41 kg of boric acid, 8.47 kg of sodium hydroxide, 0.663 g of lithium hydroxide, and 212 mL of hydrochloric acid. An additional amount of sodium hydroxide (614 g) was added with the spray beginning at time zero and lasting until 30 minutes into the test. The test was conducted for 30 days at a constant temperature of 60°C (140°F). The materials tested within this environment included representative amounts of submerged and unsubmerged aluminum, copper, concrete, zinc, carbon steel, and insulation samples (80% calcium silicate and 20% fiberglass). Representative amounts of concrete dust and latent debris were also added to the test solution. The test solution pH varied from 9.5 to 9.8 over the first two days, rose to 9.9 on Day 8, and then stayed between 9.7 and 9.9 for the remainder of the test. The test solution turbidity decreased to less than 3 nephelometric turbidity units (NTU) after 24 hours. The turbidity averaged 0.5 NTU over the last three weeks of the test. Observations of the test solution indicated that no chemical byproducts were visible in the water and no precipitation occurred as samples cooled from test temperature to room temperature. The appearance of the submerged metallic coupons was largely unchanged throughout the test. Post-test examinations showed very little weight changes and only minimal visible deposits on some coupons. The unsubmerged coupons exhibited some streaking, but little or no weight changes. The bottom of the tank was filled with reddish-brown sediment, but no gel-like deposits were present. The test solution remained clearly Newtonian for the entire test. Aluminum was detectable in the solution for only the first 24 hours.

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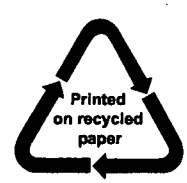

Federal Recycling Program

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