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.

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Transcribed Laboratory Log

<u>Laboratory session from June 29, 2005.</u>
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		
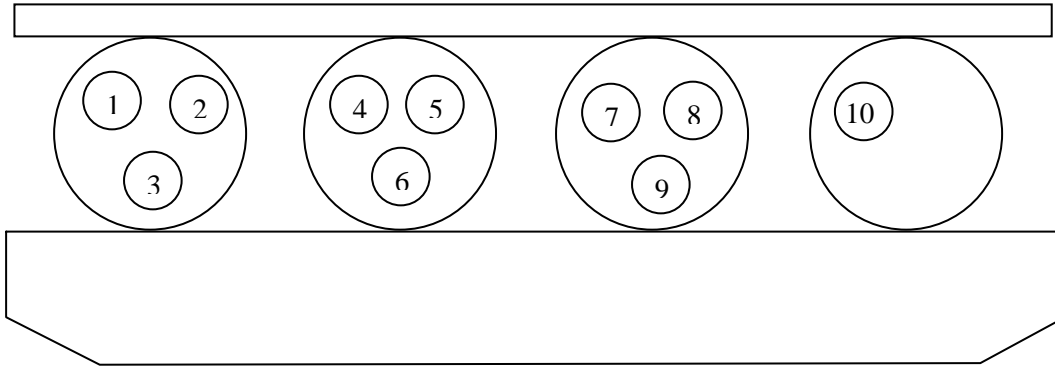
Unsubmerged Steel Coupons

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

T4D30SteelSusp020.bmp

100µm

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

T4D30SteelSusp021.bmp

10 µm

Figure F4-2. SEM image magnified 500 times for a Test #4, Day-30 unsubmerged uncoated steel coupon sample. (T4D30SteelSusp021.bmp)
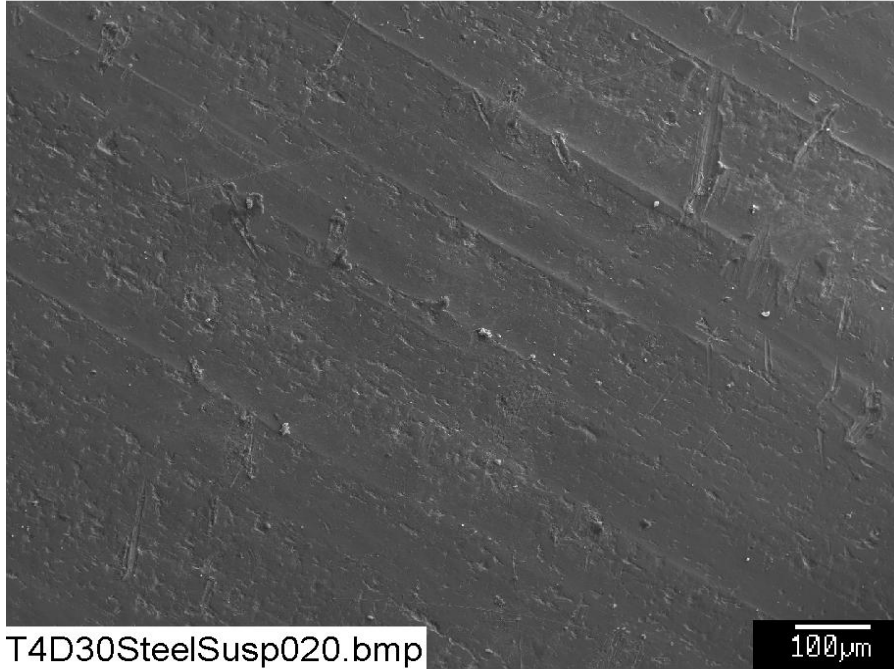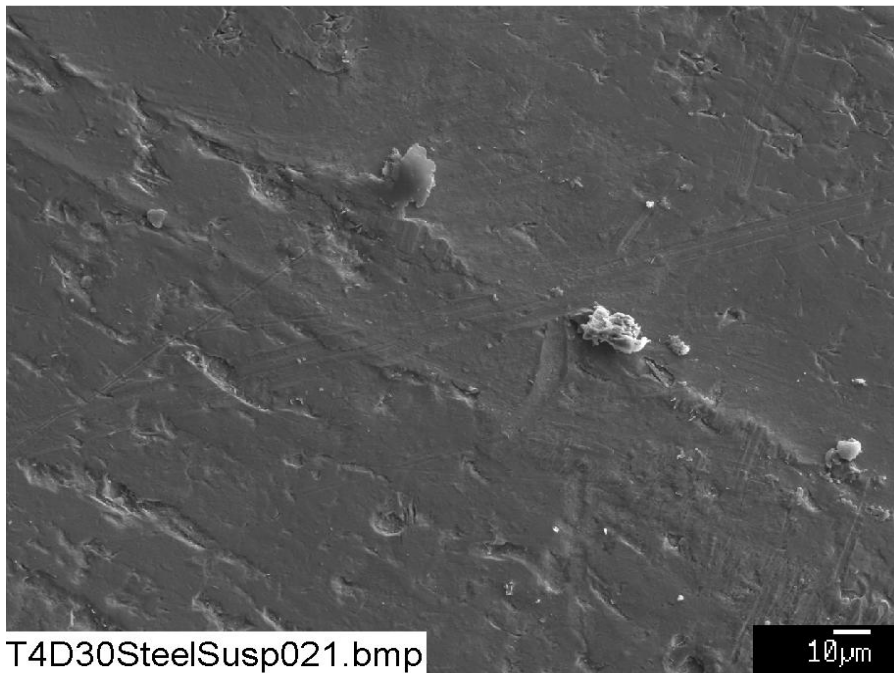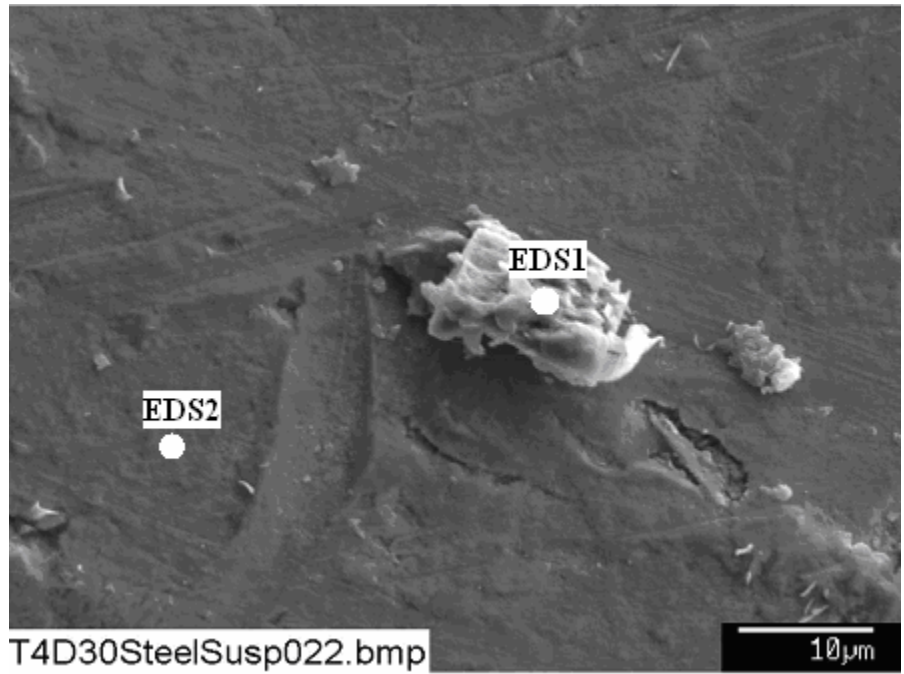
Figure F4-3. Annotated SEM image magnified 1800 times for a Test #4, Day-30 unsubmerged uncoated steel coupon sample. (T4D30SteelSusp022.bmp)
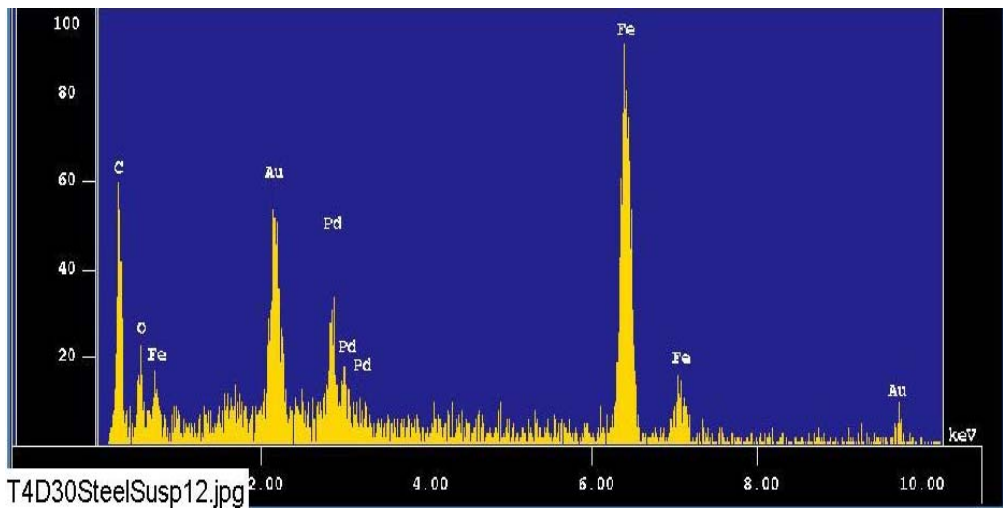

Figure F4-4. EDS counting spectrum for the white deposits (EDS1) on the coupon surface shown in Figure F4-3. (T4D30SteelSusp12.jpg)

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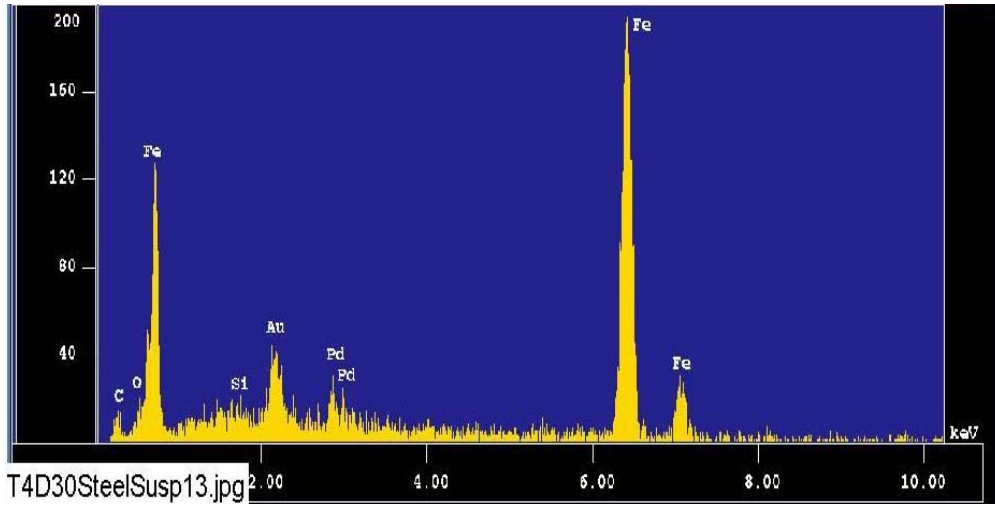
Figure F4-5. EDS counting spectrum for the flat coupon surface (EDS2) shown in Figure F4-3. (T4D30SteelSusp13.jpg)

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
```

T4D30SteelSubm023.bmp

100μm

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

T4D30SteelSubm024.bmp

10μm

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

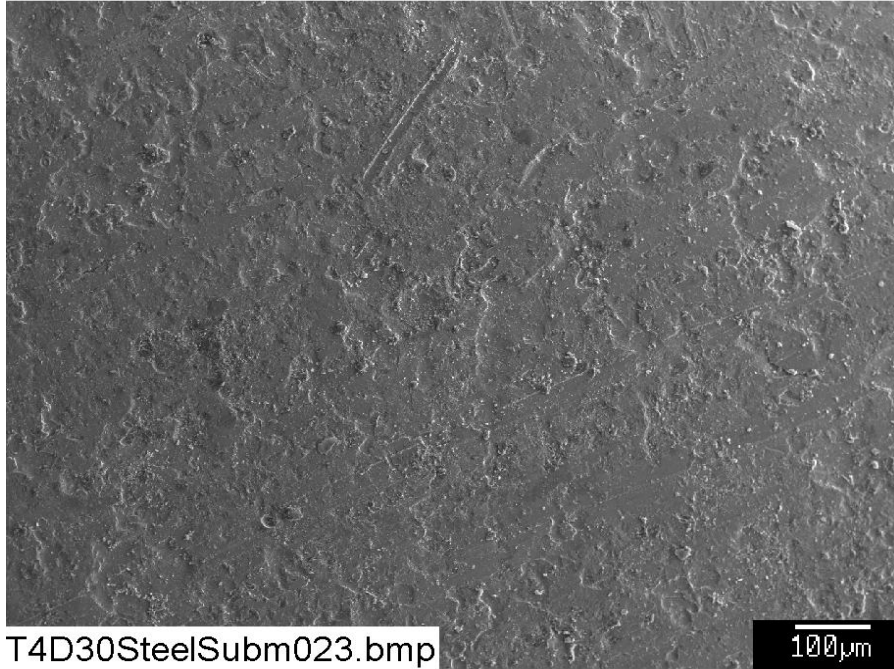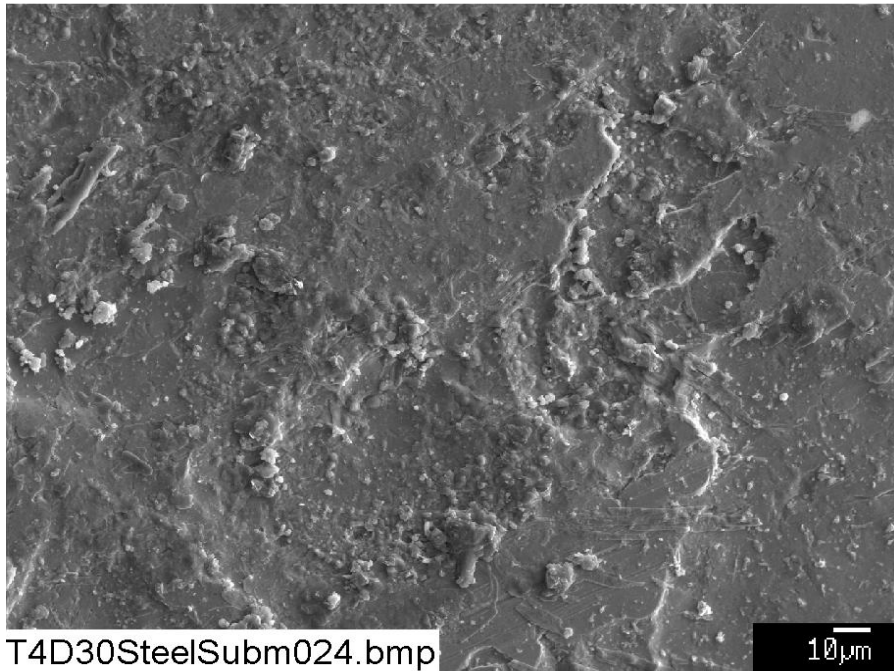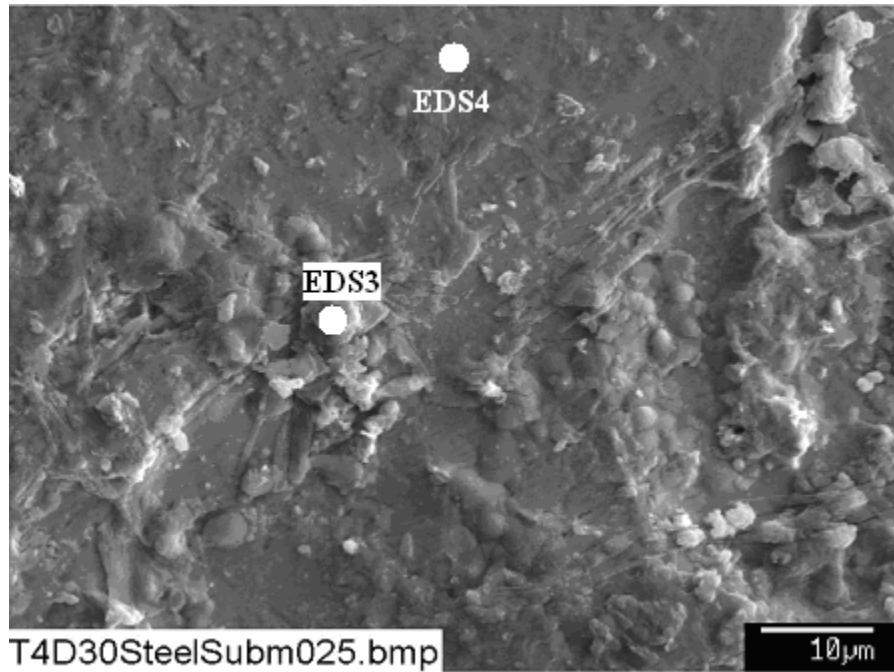**Figure F4-8.** Annotated SEM image magnified 1500 times for a Test #4, Day-30 submerged uncoated steel coupon sample. (T4D30SteelSubm025.bmp)
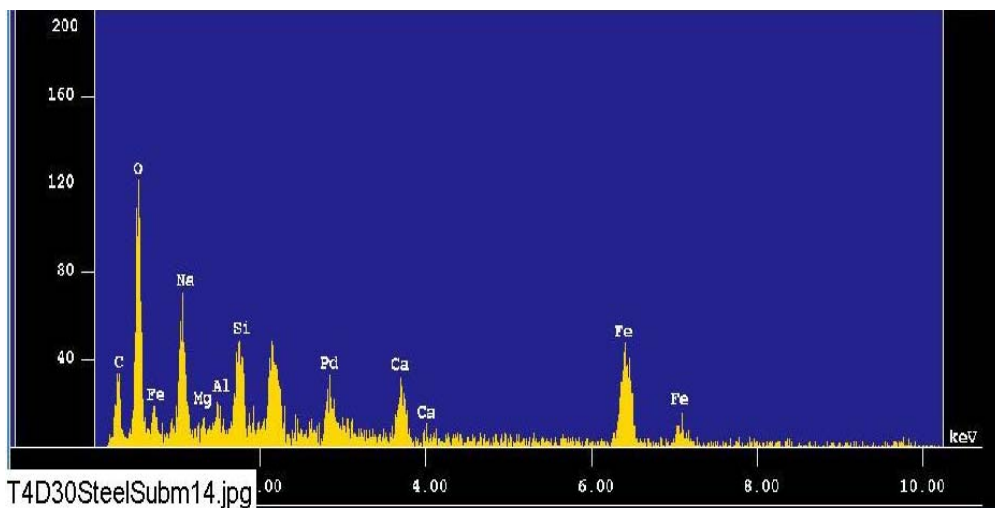

Figure F4-9. EDS counting spectrum for the deposit (EDS3) shown in Figure F4-8. (T4D30SteelSubm14.jpg)

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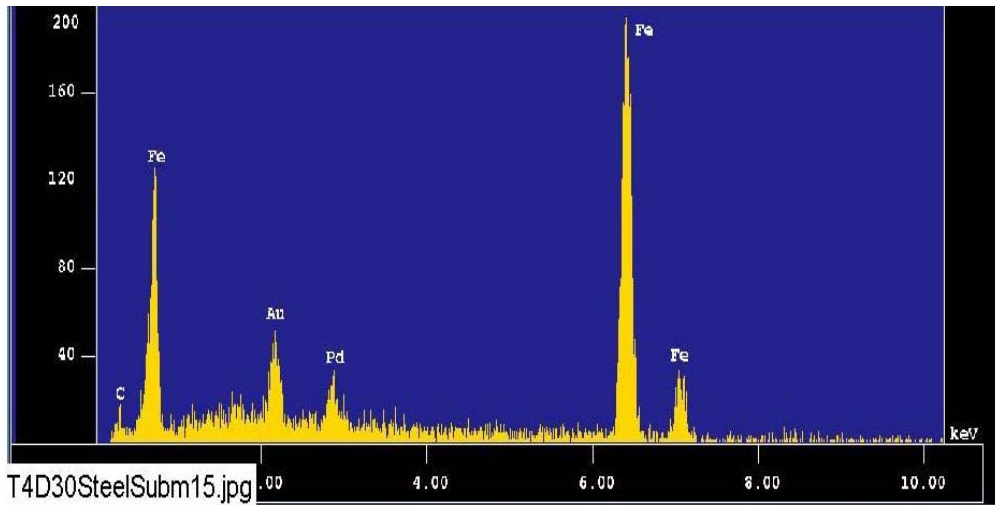
Figure F4-10. EDS counting spectrum for the flat coupon surface (EDS4) shown in Figure F4-8. (T4D30SteelSubm15.jpg)

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
```

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Appendix G

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

Figures

Tables

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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.
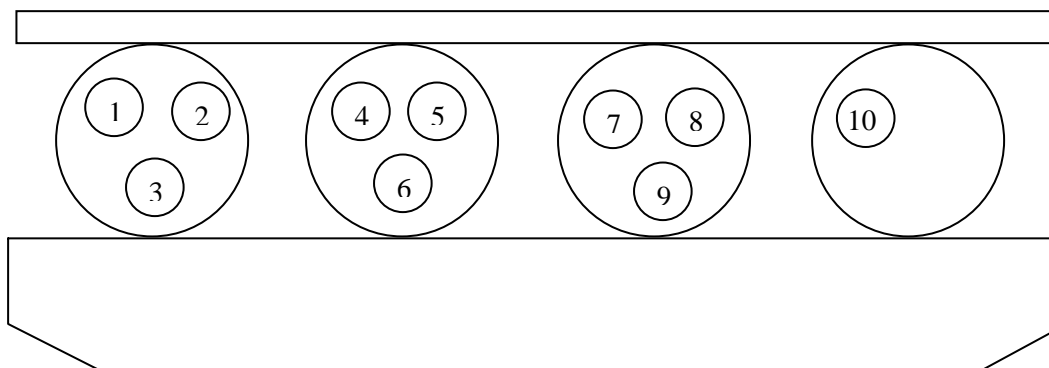
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Transcribed Laboratory Log

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

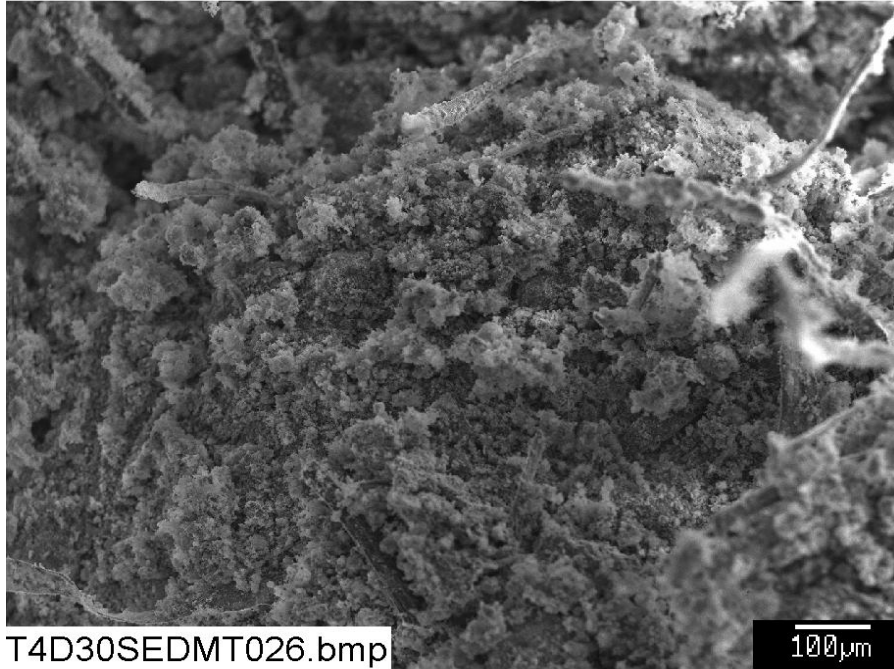**Figure G-1.** **SEM image magnified 100 times for a Test #4, Day-30 sediment at the bottom of the tank. (T4D30SEDMT026.bmp)**
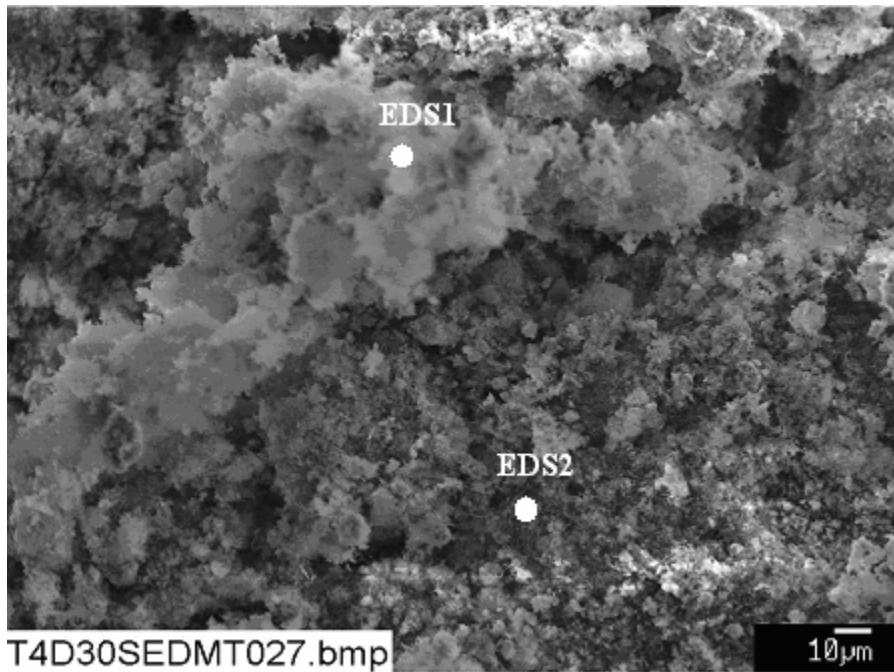

Figure G-2. **Annotated SEM image magnified 500 times for a Test #4, Day-30 sediment at the bottom of the tank. (T4D30SEDMT027.bmp)**
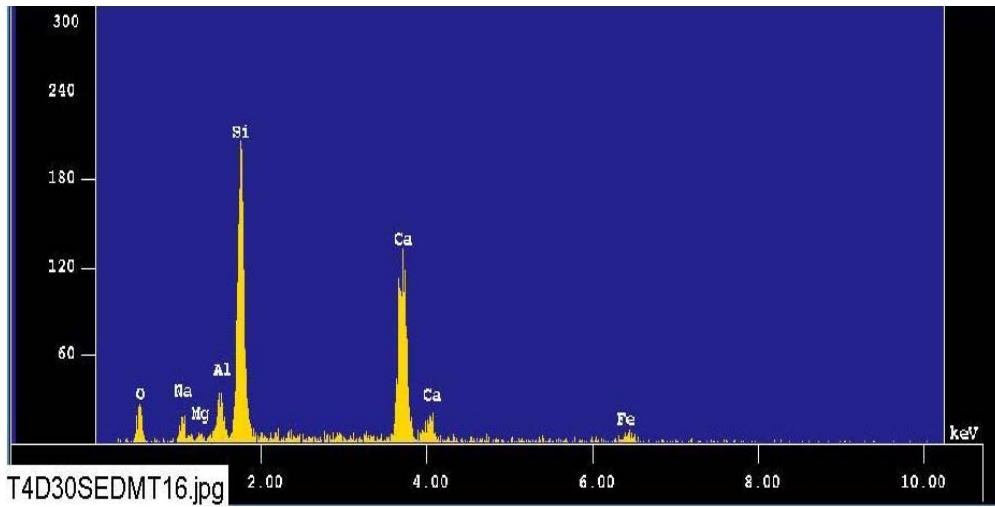
Figure G-3. EDS counting spectrum for the white snow like deposits (EDS1) shown in Figure G-2. (T4D30SEDMT16.jpg)

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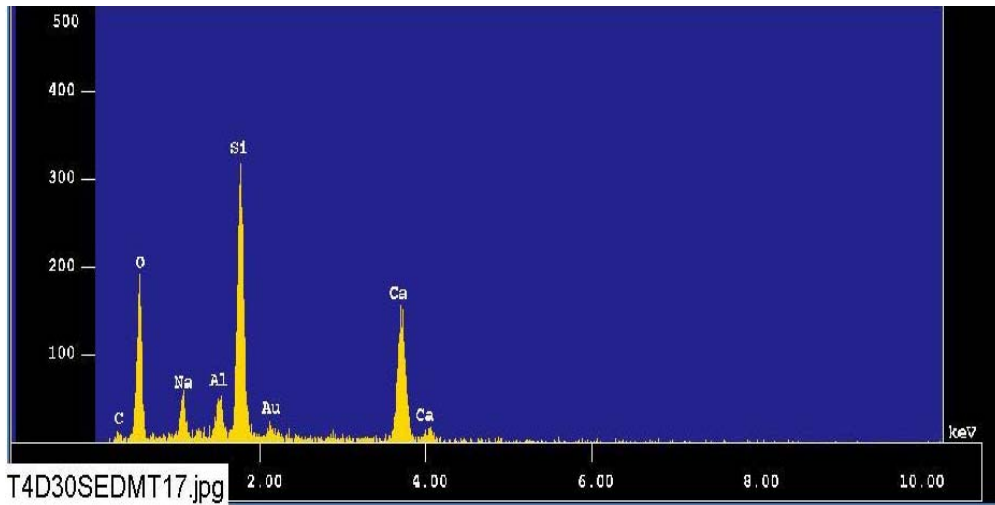
Figure G-4. EDS counting spectrum for the dark deposits (EDS2) shown in Figure G-2. (T4D30SEDMT17.jpg)

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
```
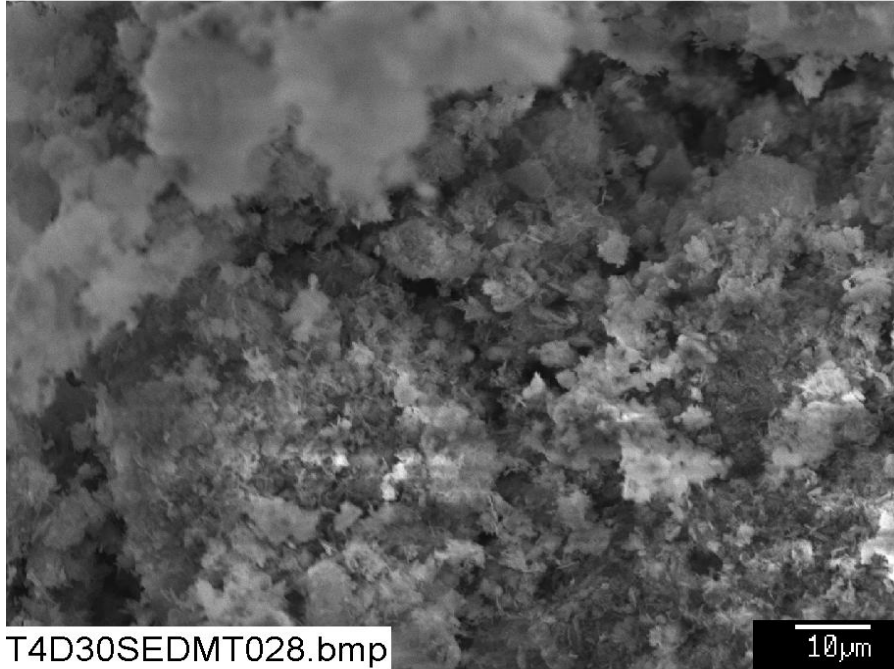
T4D30SEDMT028.bmp
10μm

Figure G-5. SEM image magnified 1000 times for a Test #4, Day-30 sediment at the bottom of the tank. (T4D30SEDMT028.bmp)
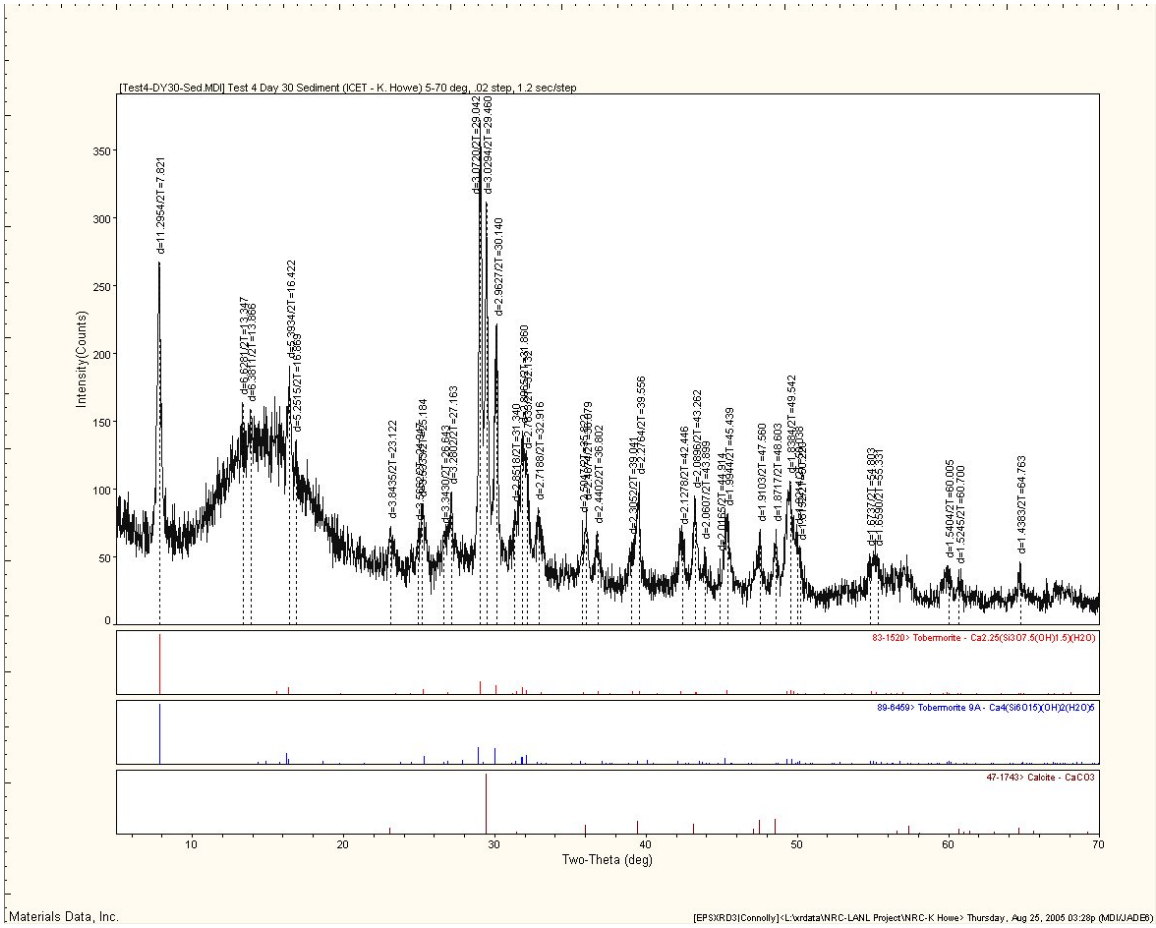
Figure G-6. XRD result of the possible matching crystalline substances in Test #4, Day-30 sediment.
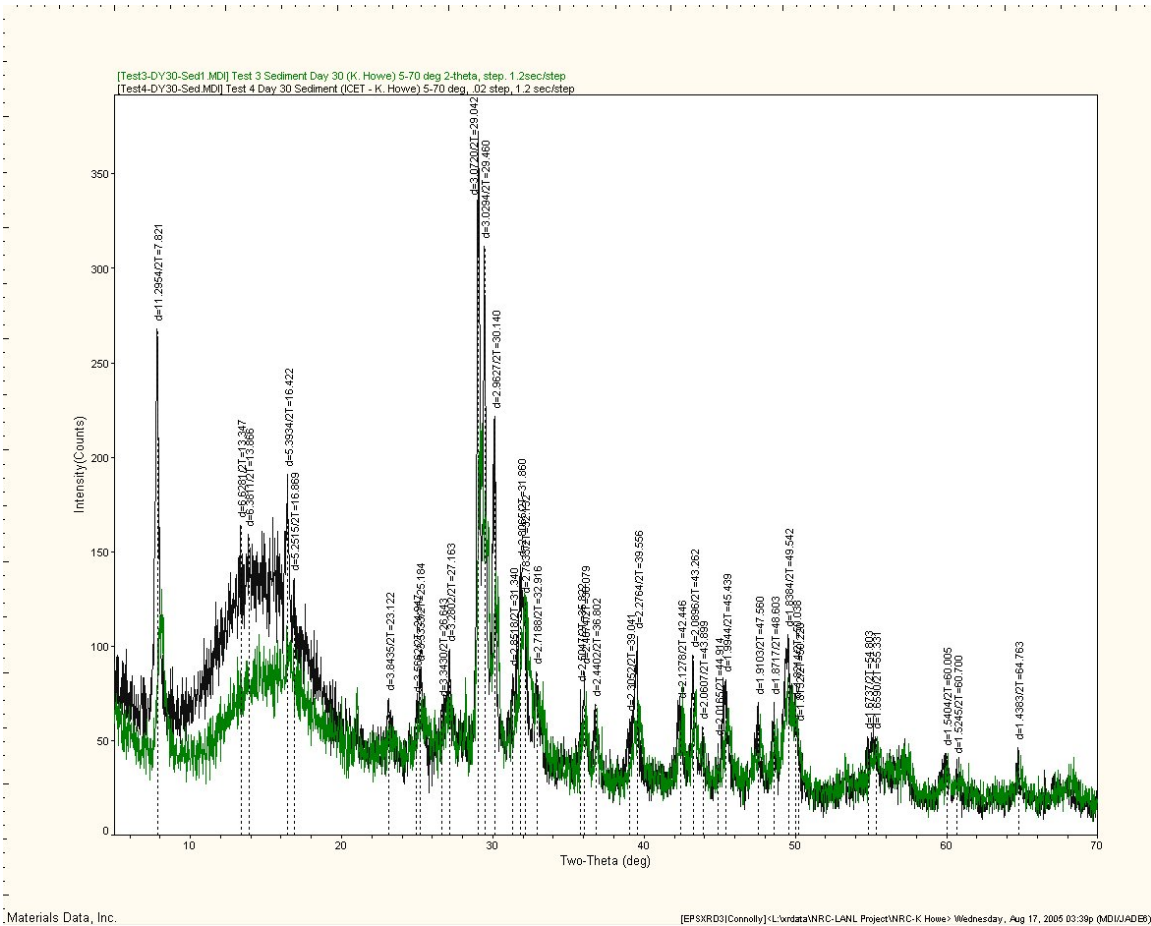
Figure G-7. XRD results for the comparison between the Test #4, Day-30 sediment (black spectrum) and the Test #3 Day-30 sediment (green spectrum).

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	$H_2O(-)$	$H_2O(+)$ CO_2	P_2O_5	Total	$H_2O(+)$ CO_2 /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

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