# Appendix D

# SEM/EDS Data for White Precipitate, Day-30 Fiberglass, Drain Screen Debris, Pipe Residue, Tank Sediment, Concrete Sample, Latent Debris Baselines, Test #1 Day-30 High-Volume Filtrate

## Figures

## Tables

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Following the conclusion of the 30-day period for Test #1, work was continued for the purpose of identifying the origin and chemical composition of the products that were formed during the test. One question of concern is whether the composition of the white precipitate evolved over the course of the study. To address this question partially, a set of precipitate samples was obtained from archival solutions representing three different time points. The first sample was obtained from unfiltered solution extracted on December 7, 2004, near the middle of the test. The second sample was obtained from an unfiltered solution extracted on November 26, 2004, near the beginning of the test. The third sample was obtained from an unfiltered solution that was archived in 1-l bottles at the end of the test. This appendix contains SEM/EDS data generated on January 18, 2005, for the sampled precipitate.

This appendix also presents additional SEM/EDS data that were generated on January 18, 2005, during a reexamination of Day-30 fiberglass. This analysis was conducted to obtain additional composition spectra and higher-quality images free from streaking caused by charge buildup on the samples.

On January 28, 2005, additional inspections were performed of (1) debris collected from the drain screen, (2) residual deposits scraped from inside the piping after the tank was drained, (3) sediment collected from the bottom of the tank, and (4) concrete chips that were introduced in a SS bag as a unique sample type. In addition, baseline characterizations were obtained for the soil and crushed concrete constituents of the surrogate latent debris.

On December 21, 2004 (termination date for Test 1), a high-volume filter sample was extracted from the tank. SEM/EDS data are presented here for the filtrate that was obtained from that filter paper on February 25, 2005. See Appendix C for optical microscopic characterization of this filtrate.

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# Transcribed Laboratory Log

**Laboratory session from January 18, 2005**

Samples of precipitate from archival bottles at three time points during Test #1.

Sample      #1      ICET-1207-1000-u (from mid-test unfiltered daily water sample)
            #2      ICET-1126-1000-u (from early-test unfiltered daily water sample)
            #3      ICET-12-21-04 #7 (from end-of-test archive bottle #7)

Spatial arrangement of sample material on the SEM sample mounts. Noticeable color variations from cream to brown were evident. Samples arranged to allow examination of entire range.
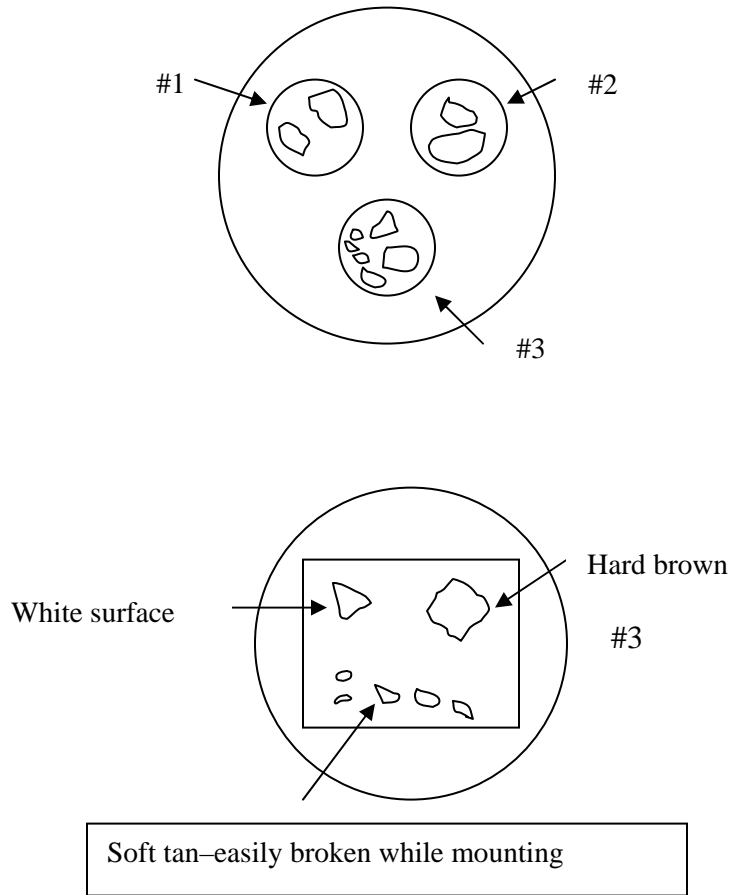




Instrument Conditions:          15-kV, 1-nA beam current, Aperture 2, EDS = T3, 100 s

**File: POST-T1_sample #1**

| Image: | POST-T1001 | 40 × | overview of chip |
|--------|------------|------|------------------|
|        | POST-T1002 | 220 × | on desiccation wrinkle |

| | | | |
|---|---|---|---|
| EDS: 1-1 | | | O > Na > Al >> B, first time that boron has been noted |
| | | | |
| Image: | POST-T1003 | | 2000 × on granular surface approximately 10- × 10-µm area |
| EDS: 1-2 | EDS on area of granular surface | | ~10- × 10-µm area |
| | | | |
| Image: | POST-T1004 | | 1000 × on coarse granular surface in upper part of photo T1001 above |
| EDS | 1-3 | | EDS on area (10 × 10 µm) of coarse granular surface |
| EDS | 1-4 | | particle on surface of image T1004 above |

**File: POST-T1_sample #2**

| | | | |
|---|---|---|---|
| Image: | POST-T1005 | 40 × | overview of one fragment on mount, coarsely granular structure |
| | POST-T1006 | 1000 × | on coarse granular surface |
| EDS | 2-5 | | EDS on area (10 × 10 µm) of granular surface |
| | | | |
| Image: | POST-T1007 | 40 × | overview on other mounted fragment, smooth surface |
| | POST-T1008 | 2000 × | on smooth granular surface |
| EDS | 2-6 | | EDS on one area (10 × 10 µm) of the smooth surface |
| EDS | 2-7 | | on another area of the smooth surface |

**File: POST-T1_sample #3**

| | | | |
|---|---|---|---|
| Image: | POST-T1009 | 65 × | on white surface |
| | POST-T1010 | 1000 × | on white surface, coarse granular, appears to be on underside of smooth brown fragment |
| EDS: | 3-8 | | EDS on area (10 × 10 µm) of coarse white surface in image T1010 |
| | | | |
| Image: | POST-T1011 | 10,000 × | close-up of image T1010 |
| EDS: | 3-9 | | area analysis (10 × 10 µm) on another area of white surface |
| | | | |
| Image: | POST-T1012 | 40 × | overview of "smooth" hard brown fragment, surface wrinkled |
| | POST-T1013 | 500 × | on wrinkled surface |
| | POST-T1014 | 2000 × | close-up on wrinkled surface in image T1013 |
| EDS: | 3-10 | | area analysis (10 × 10 µm) on wrinkled surface in image T1014 |

| Image: | POST-T1015 | 40 × | overview of soft tan particles, concoidal fracturing |
| | POST-T1016 | 2000 × | surface of concoidal fracture |
| EDS: | 3-11 | | area analysis on surface in image T1016 |
| | 3-12 | | area analysis on another concoidal surface of a different particle |

<u>Reexamine samples from Day 30</u>

Sample Definitions:    #3      High-flow sacrificial fiber from perimeter with dark crust
                            #4      High-flow sacrificial fiber from perimeter with white deposits
                                        (visually surveyed but found to be similar to #3, no data saved)
                            #7      Unsoaked fiberglass to serve as "clean" control sample.
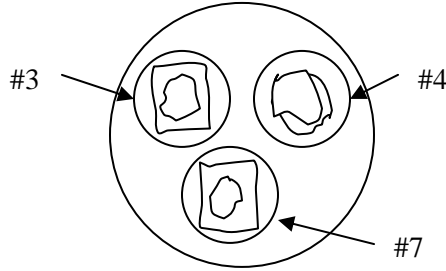


Sample: T1D30 (30 day sample)

**File: T1D30_3, sample #3**

| Image: | T1D30_3017 | 150 × | overview |
|---|---|---|---|
| EDS: | T1D30_3-1 | | poor spectrum (not saved) |
| | T1D30_3-2 | | web-like film between fibers |
| | T1D30_3-3 | | web-like film between fibers |
| | T1D30_3-4 | | web-like film between fibers |

**File: T1D30_7, sample #7**

| Image: | T1D30_7018 | 150 × | overview on clean fiberglass |
|---|---|---|---|
| EDS: | T1D30_7-5 | | EDS on individual thick fiber |
| | T1D30_7-6 | | EDS on individual clump of fiber |

**Post-Test Analysis was done on January 28, 2005**

ICET sample mounted:

Sample     #P1               pump suction debris
           #Sed1             sediment inside pipe (loose particles)
           #Sed2             tank sediment
           #Sed2a            one side of flake
           #Sed2b            other side of flake
           #LD1              latent debris (loose particles of sand and soil)
           #CD1              concrete dust (loose particles of crushed concrete)
           #Conc             concrete fragment
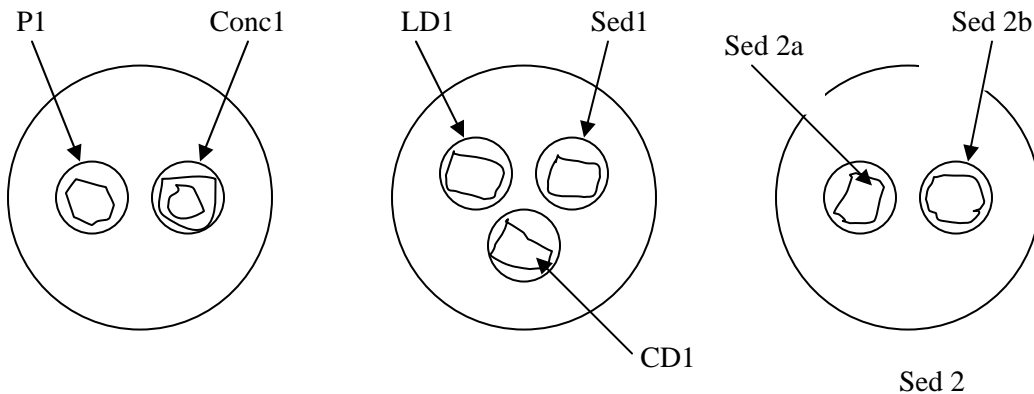


Conditions:     15-kV, 1-nA beam current, Aperture = 2, EDS aperture = 1, Process time = T2

**File: Post-T1, sample #P1**         pump suction debris

Image:     POST-T1017     35 ×        overview of fibers and other debris
           POST-T1018     220 ×       on larger particle (SEI)
           POST-T1019     220 ×       BSE same field as T1018
EDS:       P1-1                       EDS on large flake in T1018
           P1-2                       EDS on small cluster to lower left of
                                      flake in T1018

Image:     POST-T1020     60 ×        SEI at different area
                                      too much charging—beam current set to
                                      0.1 nA
           POST-T1021                 BSE same field as T1020
           POST-T1022     200 ×       SEI on flake (flake charged and jumped
                                      out of view—could not get EDS)
EDS:       P1-3                       EDS on particles in foreground, below
                                      flake

**File: Post-T1, sample conc**                                      concrete fragment

Image:            POST-T1023            43 ×            BSE image overview of portion of concrete fragment. Large aggregate in upper right is quartz.

EDS:              Conc-4                               EDS at 5500 × (20 × 20 μm) average on concrete matrix. Aggregate appears to be mostly quartz

Image:            POST-T1024            110 ×          SEI of another area
                  POST-T1025            110 ×          BSE on same field as T1024
EDS:              Conc-5                               aggregate particle in lower portion of image T1025

                  Conc-6                               surface granules on or in concrete; mostly carbon

                  Conc-7                               concrete matrix behind surface granules


**File: POST-T1, sample CD1**                                       concrete dust

Image:            POST-T1026            40 ×           SEI overview of dust particles mounted on carbon tape; many large particles

                  POST-T1027            40 ×           BSE on same field as T1026; checked particles with EDS; most are quartz
EDS:              CD-8                                 EDS on particles in lower center of image T1027

Image:            POST-T1028            170 ×          SEI on smaller particles at different area
                  POST-T1029            170 ×          BSE on same field as T1028
EDS:              CD-9                                 EDS on particle in lower right of image T1029; most large particles are quartz
Image:            POST-T1030            400 ×          SEI image of particle in center of image T1029


**File: POST-T1, sample LD-1**                                      Latent debris

Image: POST-T1031                       40 ×           SEI overview of dust debris mounted on carbon tape

                  POST-T1032            40 ×           BSE image of same field; large particles are quartz as k-feldspar
EDS:              LD-10                                EDS on large particle at right side of k-feldspar

                  LD-11                                EDS on small bright particle in upper center of image T1032

                  LD-12                                EDS on thin fiber in lower center of image T1032

Image:            POST-T1033            400 ×          SEI of central portion of image T1031
                  POST-T1034            400 ×          BSE image of same field as above
EDS:              LD-13                                EDS on flake at left center of a Na-feldspar

                  LD-14                                EDS on large particle at upper center of image T1034

| Image: | POST-T1035 | 40 × | SEI overview of another area |
|---|---|---|---|
| | POST-T1036 | 40 × | BSE of same field as above |
| EDS: | LD-15 | | EDS on bright particle in lower right of image T1036 |
| | LD-16 | | EDS on round particle slightly right center of image T1036 |
| | LD-17 | | EDS over area (425 × 425 μm) at 250 times; C and Cl in spectrum are from mounting tape |

**File: POST-T1, sample Sed1**  pipe sediment

| Image: | POST-T1037 | 40 × | SEI image overview of particles |
|---|---|---|---|
| | POST-T1038 | 40 × | BSE of same field as above |
| | POST-T1039 | 120 × | SEI close-up |
| | POST-T1040 | 120 × | BSE of same field as above (pretty uniform composition) |
| | POST-T1041 | 370 × | SEI on single particle |
| | POST-T1042 | 3000 × | SEI on small particle |
| EDS: | SED-18 | | EDS over particle in image T1042 at 6000 × (18 × 18 μm) |
| Image: | POST-T1043 | 300 × | SEI on another particle |
| | POST-T1044 | 300 × | BSE of same field as above |
| | POST-T1045 | 5000 × | SEI on center of particle |
| | POST-T1046 | 10,000 × | SEI on same particle |
| | POST-T1047 | 40 × | overview of above |
| EDS: | SED-19 | | EDS on same field as image T1046 at 10,000 × |

**File: POST-T1, sample Sed-2A**  Tank sediment

| Image: | POST-T1048 | 100 × | SEI overview |
|---|---|---|---|
| | POST-T1049 | 100 × | BSE same field as above |
| | POST-T1050 | | SEI on a representative large particle |
| | POST-T1051 | 550 × | SEI on the same large particle |

Note: *Samples Sed2-A and -B, and P1 were overcharged. Samples were recoated.

| Image: POST-T1052 | 43 × | SEI overview |
|---|---|---|
| POST-T1053 | 370 × | SEI 2 large particles |
| POST-T1054 | 370 × | BSE same field as above |
| POST-T1055 | 1000 × | BSE on fuzzy particle |
| POST-T1056 | 5000 × | same particle |
| EDS:  SED-20 | | EDS on fuzzy particle |
| SED-21 | | EDS on another particle—no Fe, Cu, Zn |
| Image:  POST-T1057 | 1000 × | SEI on particle analyzed |
| EDS:  SED-22 | | EDS on large round particle K-feldspar, probably from concrete |

| Image: | POST-T1058 | 150 × | SEI of high-Z particles analyzed above (EDS SED-21 and SED-22) |
|---|---|---|---|

**File: POST-T1, sample Sed-2B**                                 Tank sediment, other side of flake from 2A

| Image: | POST-T1059 | 40 × | SEI overview; number of large particles in view |
|---|---|---|---|
| | POST-T1060 | 40 × | BSE same field as above |
| EDS: | Sed-23 | | EDS on plate; O>Na>Al; similar to filtrate composition from 1/18/05 |
| | Sed-24 | | EDS on particle in above plate |
| Image: | POST-T1061 | 160 × | SEI on agglomerate of small particles and fibers at left side of image T1059 |
| | POST-T1062 | 160 × | BSE same field as above |
| EDS: | Sed-25 | | EDS on particle in agglomerate of image T1062 |

Note: *Returned to sample P1 because of bad charging of the previous sample.

**File: POST-T1, sample P1**                                      pump suction debris after recoating

| Image: | POST-T1063 | 40 × | overview, large particles present in sample |
|---|---|---|---|
| | POST-T1064 | 40 × | BSE on same field as above |
| EDS: | P1-26 | | EDS on large triangular particle |
| | P1-27 | | EDS on flat particle at lower right |
| | P1-28 | | EDS on bright particle in center |

Sample: <u>High-Volume Filter</u> (T1D30) from February 25, 2005

| Image: | T1D30_001 | 40 × | Overview |
|---|---|---|---|
| | T1D30_002 | 300 × | Close-up |
| EDS: | T1D30-EDS1 | | On filtrate |
| | T1D30-EDS2 | | On filtrate |
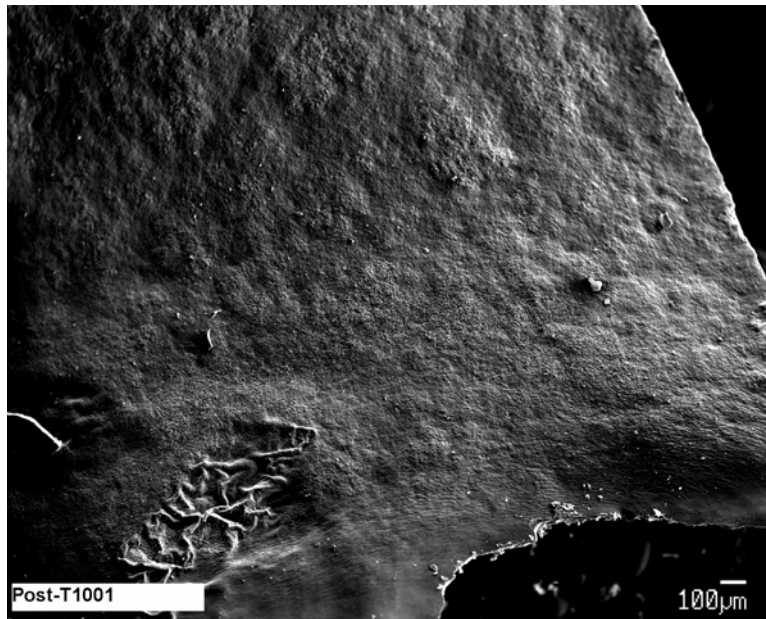
**Figure D-1.** Mid-test unfiltered water sample, post-T1 sample #1 SEM image (POST-T1001) magnified 40 times; overview of the white precipitate chip.
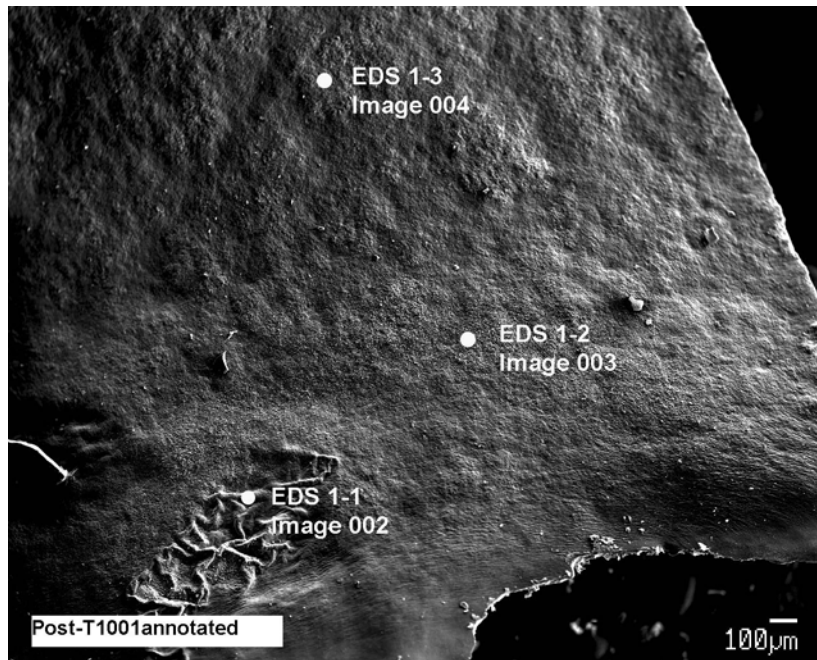


**Figure D-2.** Mid-test unfiltered water sample, post-T1 sample #1 SEM image (POST-T1001 annotated) annotating the EDS locations.
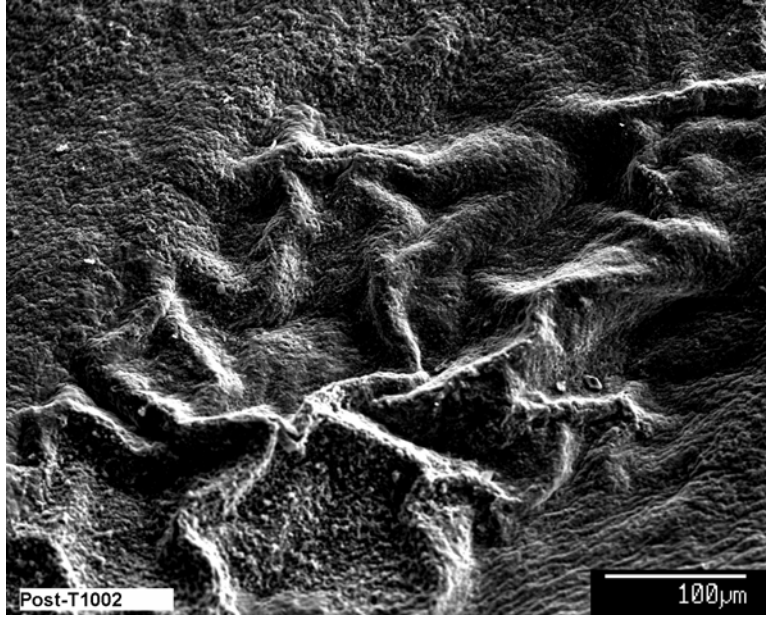
**Figure D-3.**      Mid-test unfiltered water sample, post-T1 sample #1 SEM image (POST-T1002) magnified 220 times on the desiccation wrinkles, as shown in Figure D-2.
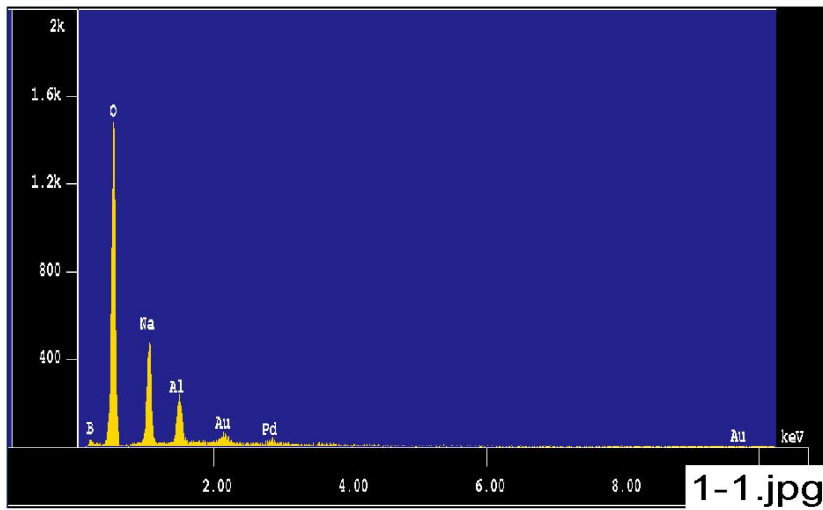


**Figure D-4.**      Mid-test unfiltered water sample, post-T1 sample #1 counting spectrum (EDS 1-1) for the desiccation wrinkles in Figure D-3, showing the chemical compositions of O, Na, Al, and B (first time that B has been noted).

The results from the chemical composition analysis for EDS 1-1 are given in Table D-1.

**Table D-1.      The Chemical Compositions for EDS 1-1**

```
Jan 18 11:17 2005  /tmp/eds_pout.log Page 1


Group      : NRC
Sample     : Post-T1  ID# : 1     From SEM#1 (Day 15)
Comment    : wrinkled surface
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV        Probe Current : 8.587E-10 A
             Stage Point : X=25.049 Y=59.828 Z=10.967
             Acq. Date   : Tue Jan 18 10:46:14 2005


Element    Mode      ROI(KeV)    K-ratio(%)  +/-    Net/Background
  O K      Normal    0.25- 0.77    35.4629   0.0044    11879 /     17
 Na K      Normal    0.81- 1.27     4.5113   0.0016     4342 /     21
 Al K      Normal    1.19- 1.83     1.5205   0.0004     2113 /     19
  B K      Normal    0.00- 0.36     1.2097   0.0002      169 /      7
-------------------------------------------------------------------
                     Chi_square = 44.3126

Element Mass%    Atomic%    ZAF     Z      A      F
     O   61.031  58.8793  0.7366 0.9686 0.7604 1.0000
    Na   13.715   9.2077  1.3011 1.0231 1.2716 1.0001
    Al    4.842   2.7699  1.3629 0.9965 1.3677 1.0000
     B   20.413  29.1432  7.2220 1.1244 6.4230 1.0000
-------------------------------------------------------------------
 Total   100.000 100.0000
Normalization factor =  2.3365
```
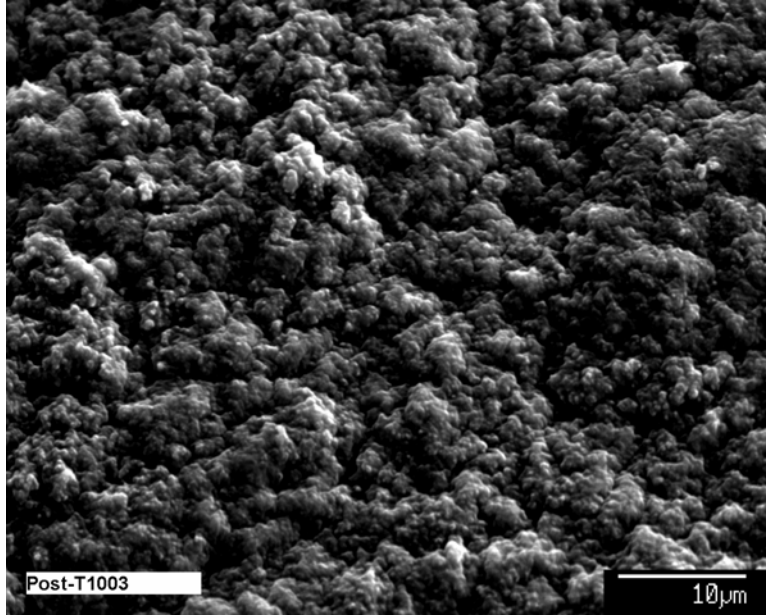
**Figure D-5.**     **Mid-test unfiltered water sample, post-T1 sample #1 image (POST-T1003) magnified 2000 times on granular surface, as shown in Figure D-2.**
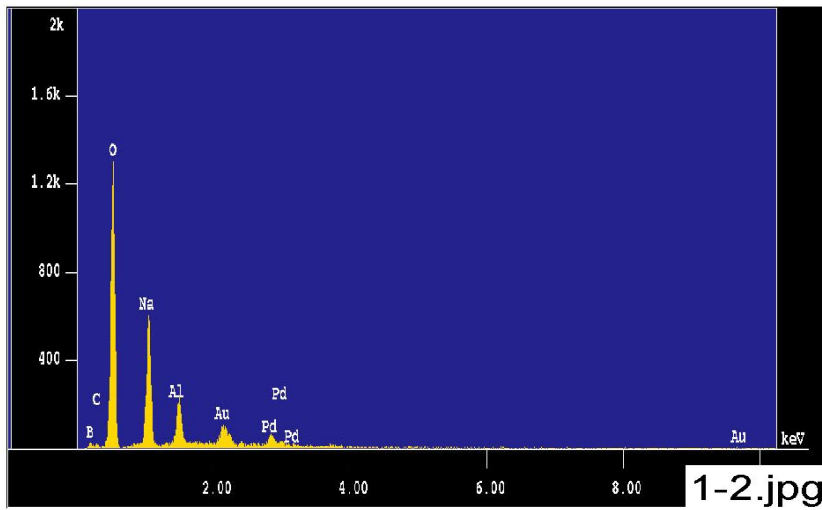


**Figure D-6.**     **Mid-test unfiltered water sample, post-T1 sample #1 counting spectrum (EDS 1-2) on the granular surface, as shown in Figure D-5, within an area about 10 × 10 µm.**
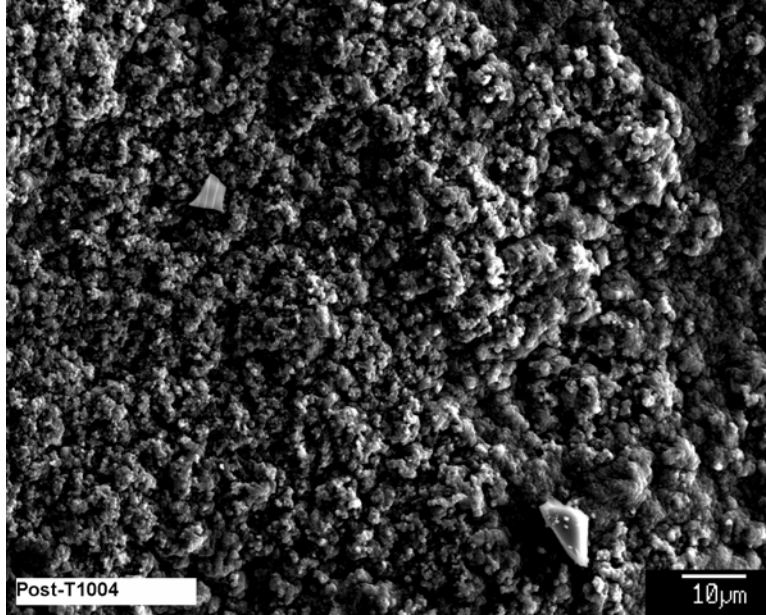
**Figure D-7.** Mid-test unfiltered water sample, post-T1 sample #1 SEM image (POST-T1004) magnified 1000 times on coarse granular surface in upper part of image (POST-T1001), as shown in Figure D-2.
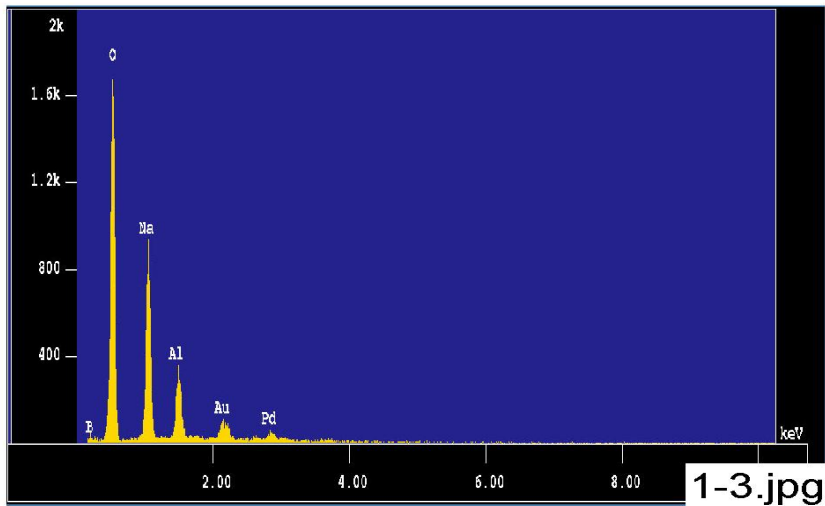


**Figure D-8.** Mid-test unfiltered water sample, post-T1 sample #1 counting spectrum (EDS 1-3) on an area (10 × 10 μm) of the coarse granular surface, as shown in Figure D-7.

The results from the chemical composition analysis for EDS 1-3 are given in Table D-2.

**Table D-2.    The Chemical Compositions for EDS 1-3**

```
Jan 18 11:18 2005   /tmp/eds_pout.log Page 1


Group      : NRC
Sample     : Post-T1   ID# : 3    From SEM# 1 (Day 15)
Comment    :  coarse granular surface area analysis
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time    : 100.000 sec    Aperture #    : 1
            Acc. Volt    : 15.0 KV         Probe Current : 8.087E-10 A
            Stage Point : X=24.722 Y=58.294 Z=10.967
            Acq. Date    : Tue Jan 18 11:00:44 2005


Element    Mode    ROI(KeV)    K-ratio(%)  +/-    Net/Background
  O K      Normal  0.25- 0.77   44.4103   0.0048   14010 /      20
 Na K      Normal  0.81- 1.27    8.5492   0.0020    7749 /      32
 Al K      Normal  1.19- 1.83    2.3330   0.0005    3054 /      35
  B K      Normal  0.00- 0.36    0.9836   0.0002     129 /      11
-----------------------------------------------------------------
                    Chi_square = 48.4646

Element Mass%   Atomic%   ZAF      Z      A      F
     O   58.466  59.0811 0.7212 0.9739 0.7406 0.9999
    Na   19.871  13.9741 1.2733 1.0285 1.2380 1.0000
    Al    6.080   3.6433 1.4278 1.0016 1.4255 1.0000
     B   15.582  23.3015 8.6782 1.1306 7.6756 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.8254
```
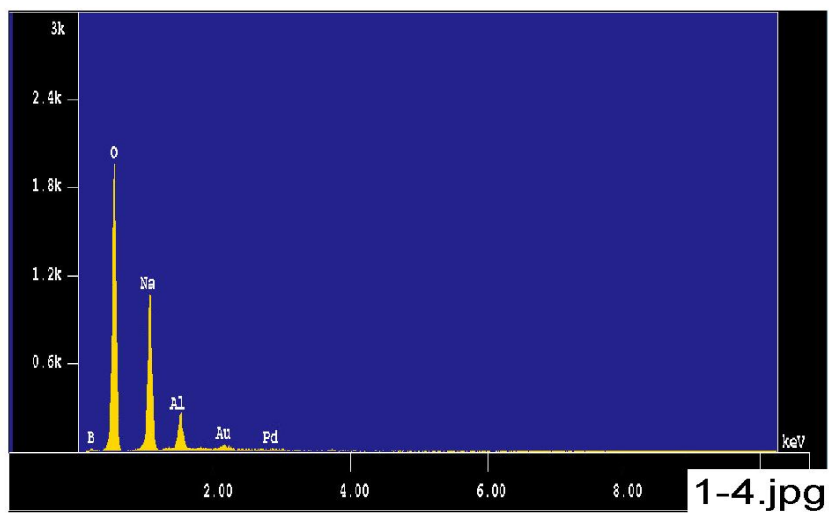
**Figure D-9.**   Mid-test unfiltered water sample, post-T1 sample #1 counting spectrum (EDS 1-4) on the particle on surface of SEM image (POST-T1004), as shown in Figure D-7.
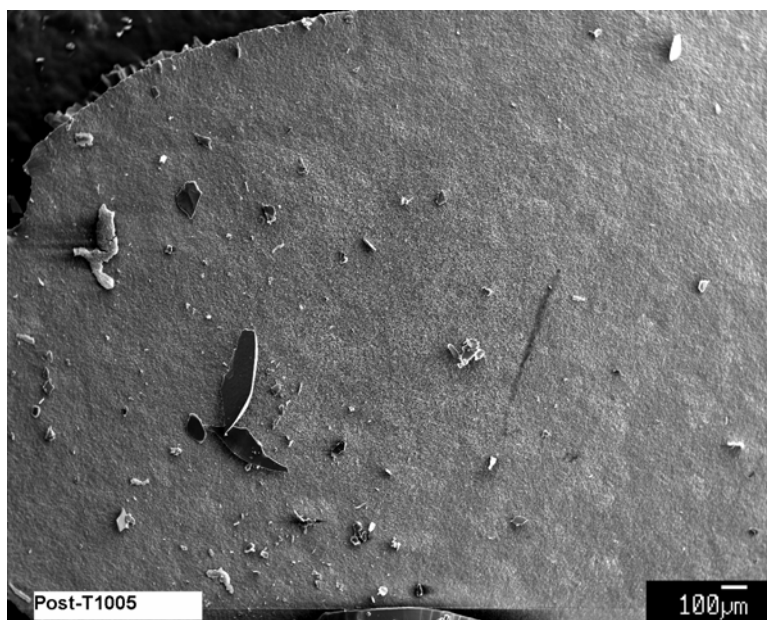


**Figure D-10.**   Mid-test unfiltered water sample, post-T1 sample #2 SEM (POST-T1005) magnified 40 times for the overview of one fragment on mount showing coarsely granular structure.
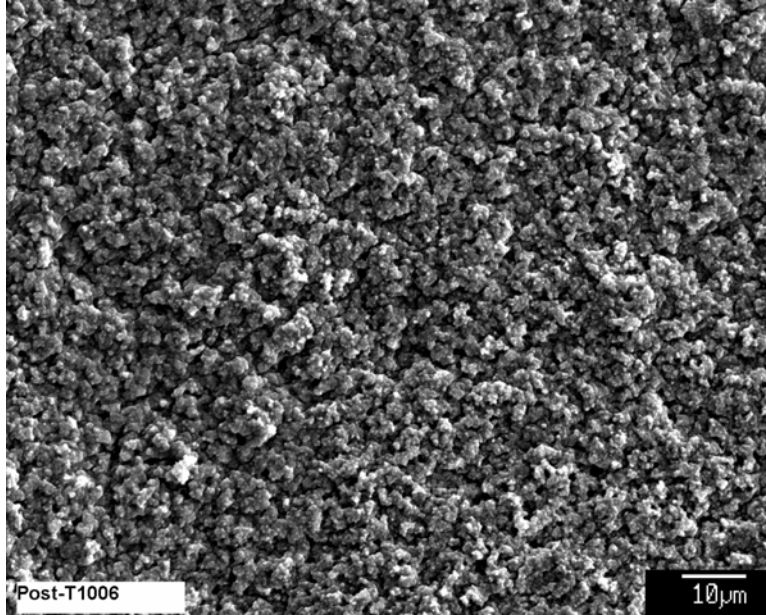
**Figure D-11.** Mid-test unfiltered water sample, post-T1 sample #2 SEM (POST-T1006) magnified 1000 times on the coarsely granular surface, as shown in Figure D-10.
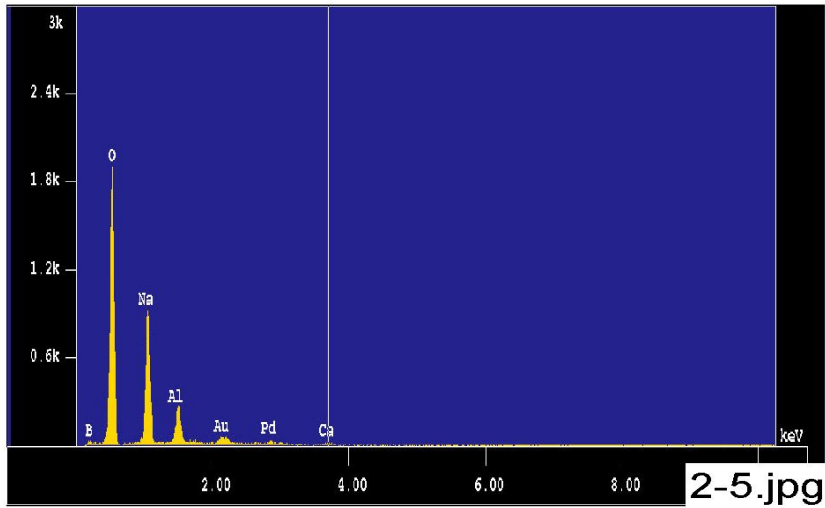


**Figure D-12.** Mid-test unfiltered water sample, post-T1 sample #2 counting spectrum (EDS 2-5) on the granular surface, as shown in Figure D-11.

The results from the chemical composition analysis for EDS 2-5 are given in Table D-3.

**Table D-3.    The Chemical Compositions for EDS 2-5**

```
Jan 18 11:31 2005   /tmp/eds_pout.log Page 1


Group      : NRC
Sample     : Post-T1  ID# : 5        From SEM#2 (beginning)
Comment    :  coarse granular surface area analysis
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 7.220E-10 A
            Stage Point : X=10.586 Y=55.990 Z=10.967
            Acq. Date   : Tue Jan 18 11:28:48 2005


Element    Mode     ROI(KeV)    K-ratio(%)  +/-     Net/Background
  O K      Normal   0.25- 0.77   52.9682   0.0049    14918 /      24
 Na K      Normal   0.81- 1.27    9.8622   0.0021     7981 /      26
 Al K      Normal   1.19- 1.83    2.0175   0.0004     2358 /      32
  B K      Normal   0.00- 0.36    1.0850   0.0002      127 /      12
 Ca K      Normal   3.39- 4.30    0.3343   0.0009      177 /      13
---------------------------------------------------------------------
                      Chi_square = 35.4442

Element Mass%    Atomic%    ZAF      Z      A      F
     O   60.176  61.0351  0.7164  0.9754  0.7344  0.9999
    Na   20.230  14.2795  1.2935  1.0302  1.2555  1.0001
    Al    4.616   2.7762  1.4427  1.0032  1.4381  1.0000
     B   14.456  21.6982  8.4015  1.1325  7.4188  1.0000
    Ca    0.521   0.2110  0.9831  1.0039  0.9794  1.0000
---------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.5859
```
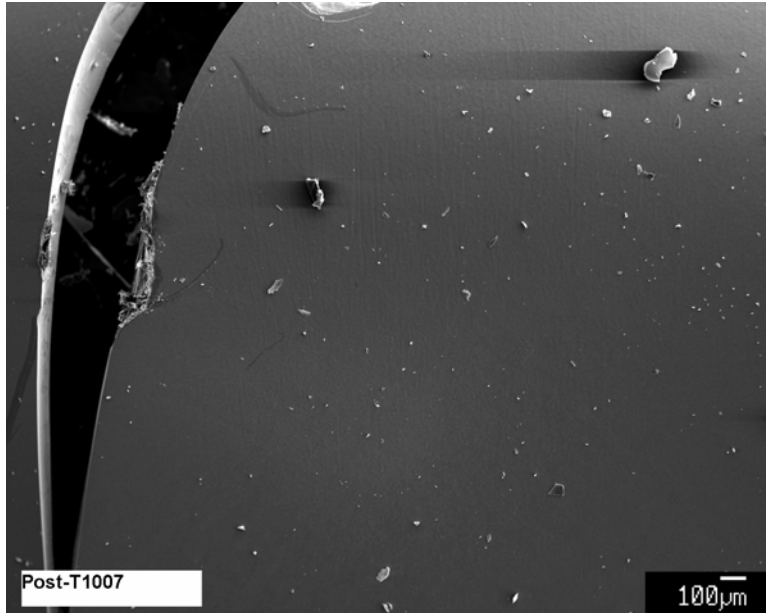
**Figure D-13.** Begin-test unfiltered water sample, post-T1 sample #2 SEM (POST-T1007) magnified 40 times of the overview on the other mounted fragment with smooth surface.
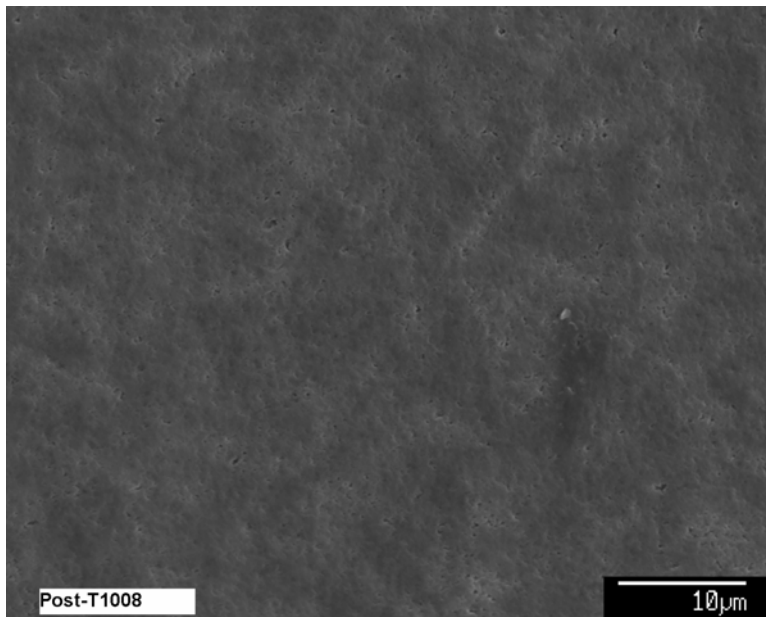


**Figure D-14.** Begin-test unfiltered water sample, post-T1 sample #2 SEM image (POST-T1008) magnified 2000 times on the smooth surface, as shown in Figure D-13.
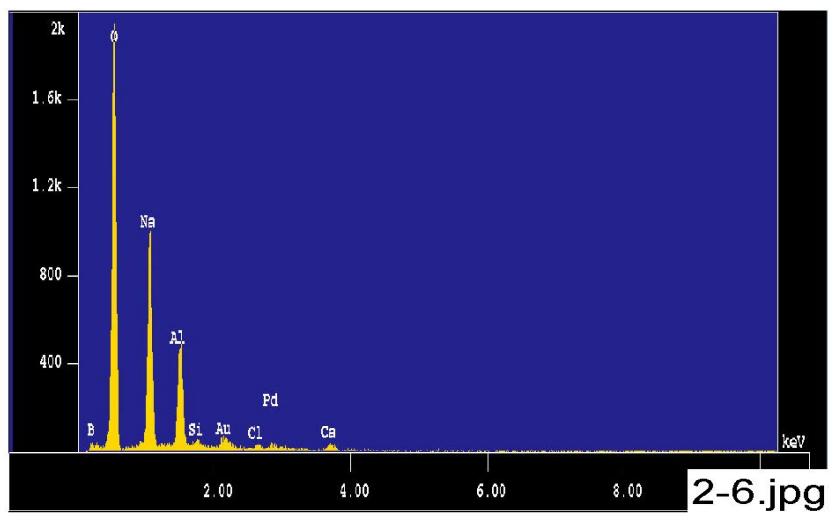
**Figure D-15.** Begin-test unfiltered water sample, post-T1 sample #2 counting spectrum (EDS 2-6) on one area (10 × 10 μm) of the smooth surface, as shown in Figure D-14.

The results from the chemical composition analysis for EDS 2-6 are given in Table D-4.

**Table D-4.      The Chemical Compositions for EDS 2-6**

```
Jan 18 11:46 2005   /tmp/eds_pout.log Page 1


Group      : NRC
Sample     : Post-T1  ID# : 6    SEM#2 (beginning)
Comment    : smooth surface area analysis
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec     Aperture #    : 1
            Acc. Volt   : 15.0 KV         Probe Current : 6.840E-10 A
            Stage Point : X=10.350 Y=59.471 Z=10.967
            Acq. Date   : Tue Jan 18 11:43:23 2005


Element    Mode      ROI(KeV)    K-ratio(%)  +/-     Net/Background
  B K      Normal    0.00- 0.36    1.2246    0.0002      136 /      14
  O K      Normal    0.25- 0.77   59.8482    0.0051    15969 /      28
  Na K     Normal    0.81- 1.27   11.1361    0.0022     8537 /      25
  Al K     Normal    1.19- 1.83    4.2719    0.0006     4730 /      32
  Si K     Normal    1.50- 2.05    0.1862    0.0003      192 /     267
  Cl K     Normal    2.34- 3.06    0.4244    0.0007      300 /      14
  Ca K     Normal    3.39- 4.30    0.7458    0.0010      374 /      10
-------------------------------------------------------------------
                   Chi_square = 26.7443

Element Mass%   Atomic%   ZAF      Z      A      F
   B    14.058  21.4396 8.9766 1.1319 7.9308 1.0000
   O    58.125  59.9001 0.7594 0.9749 0.7790 0.9999
   Na   18.281  13.1106 1.2836 1.0295 1.2470 0.9999
   Al    7.721   4.7183 1.4133 1.0025 1.4099 0.9999
   Si    0.310   0.1818 1.3007 0.9804 1.3267 0.9999
   Cl    0.565   0.2626 1.0402 1.0342 1.0062 0.9996
   Ca    0.941   0.3870 0.9863 1.0030 0.9834 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.2789
```
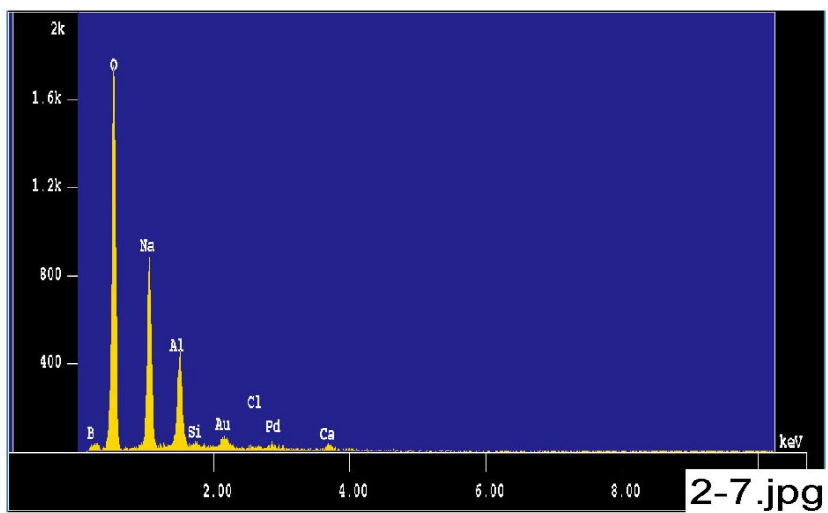
**Figure D-16.** Begin-test unfiltered water sample, post-T1 sample #2 counting spectrum (EDS 2-7) on another area of the smooth surface, as shown in Figure D-14.

The results from the chemical composition analysis for EDS 2-7 are given in Table D-5.


**Table D-5.    The Chemical Compositions for EDS 2-7**


```
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Group      : NRC
Sample     : Post-T1   ID# : 7     SEM#2 (beginning)
Comment    : smooth surface area analysis
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV        Probe Current : 6.705E-10 A
             Stage Point : X=12.518 Y=59.220 Z=10.967
             Acq. Date   : Tue Jan 18 11:49:08 2005


Element    Mode     ROI(KeV)    K-ratio(%)  +/-     Net/Background
  B K      Normal   0.00- 0.36   0.0000     0.0000      0  /     12
  O K      Normal   0.25- 0.77  55.3551     0.0049   14478  /     26
  Na K     Normal   0.81- 1.27  10.1545     0.0020    7631  /     26
  Al K     Normal   1.19- 1.83   3.8370     0.0005    4164  /     32
  Si K     Normal   1.50- 2.05   0.1735     0.0003     176  /    248
  Ca K     Normal   3.39- 4.30   0.7780     0.0010     383  /     15
-------------------------------------------------------------------
                    Chi_square = 31.2526

Element Mass%    Atomic%  ZAF      Z      A      F
  B      0.000    0.0000 11.2455 1.1536 9.7482 1.0000
  O     64.445   73.4935 0.6695 0.9934 0.6740 0.9999
  Na    23.652   18.7708 1.3394 1.0487 1.2774 0.9998
  Al    10.115    6.8395 1.5159 1.0210 1.4849 0.9999
  Si     0.422    0.2742 1.3986 0.9983 1.4010 1.0000
  Ca     1.366    0.6220 1.0099 1.0202 0.9900 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.7390
```
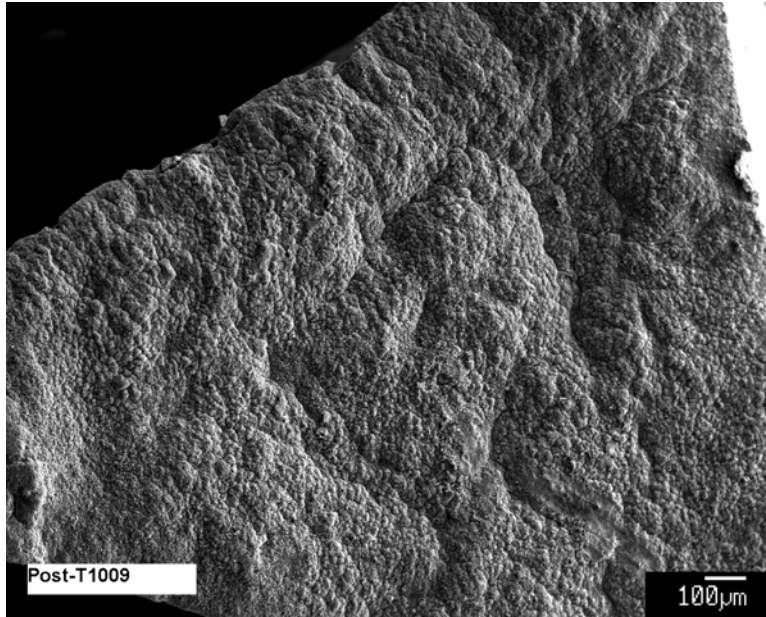
**Figure D-17.** End-test archive, post-T1 sample #3 SEM image (POST-T1009) magnified 65 times on the white surface of the white precipitate.
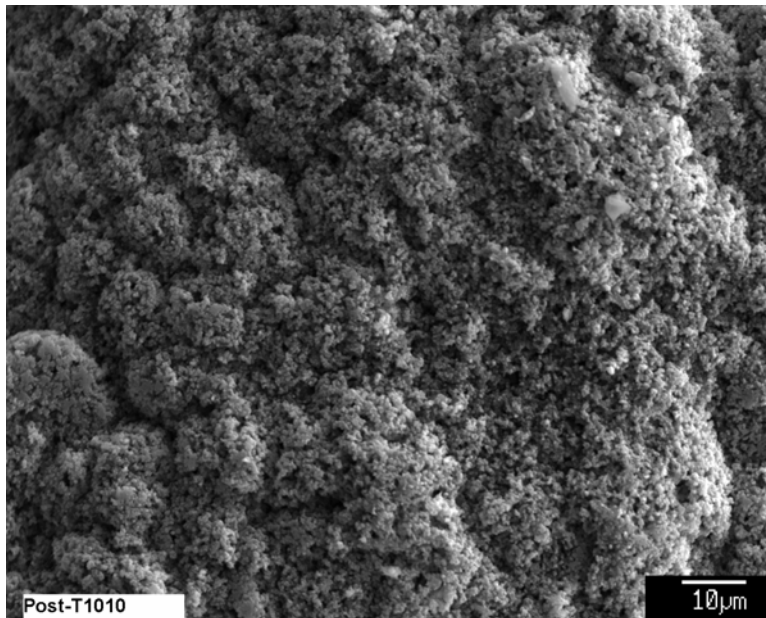


**Figure D-18.** End-test archive, post-T1 sample #3 SEM image (POST-T1010) magnified 1000 times on the white surface in Figure D-17, showing a coarsely granular structure on the underside of the smooth brown fragments.
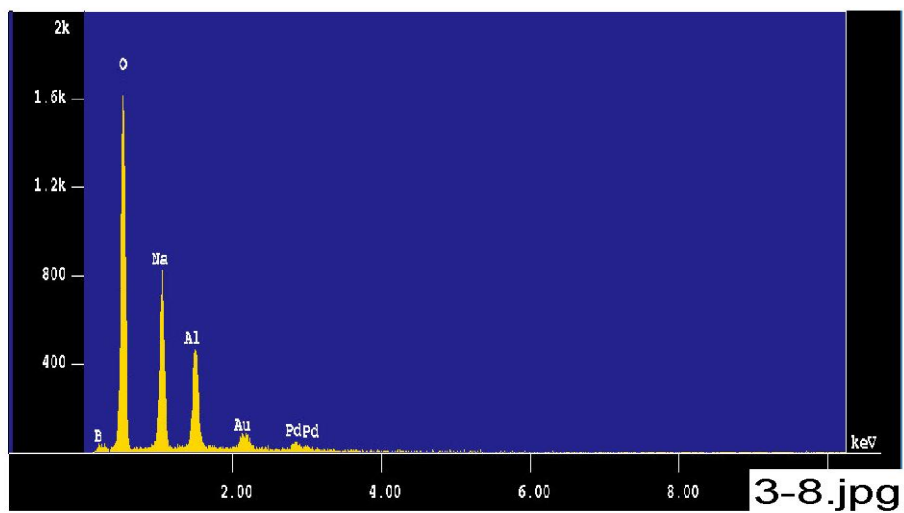
**Figure D-19.** End-test archive, post-T1 sample #3 counting spectrum (EDS 3-8) on an area (10 × 10 m) of the coarse white surface in the image (POST-T1010), as shown in Figure D-18.

The results from the chemical composition analysis for EDS 3-8 are given in Table D-6.

**Table D-6.    The Chemical Compositions for EDS 3-8**

```
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Group      : NRC
Sample     : Post-T1  ID# : 8     SEM#3 (Day 30)
Comment    : white coarse surface area analysis  - bottom of flake
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #     : 1
             Acc. Volt   : 15.0 KV         Probe Current : 6.439E-10 A
             Stage Point : X=16.090 Y=65.380 Z=10.967
             Acq. Date   : Tue Jan 18 12:00:59 2005


Element    Mode      ROI(KeV)    K-ratio(%)  +/-      Net/Background
  O K      Normal   0.25- 0.77    55.1199   0.0048     13845 /      23
  Na K     Normal   0.81- 1.27     9.4895   0.0020      6848 /      28
  Al K     Normal   1.19- 1.83     4.5307   0.0006      4722 /      28
  B K      Normal   0.00- 0.36     0.4438   0.0002        46 /      16
-------------------------------------------------------------------
                       Chi_square = 49.0164

Element Mass%    Atomic%   ZAF      Z       A       F
     O   61.576  66.1306 0.6849 0.9841 0.6960 0.9999
     Na  20.172  15.0761 1.3032 1.0391 1.2545 0.9998
     Al  10.726   6.8301 1.4514 1.0117 1.4346 1.0000
     B    7.527  11.9632 10.3982 1.1426 9.1004 1.0000
-  --------------------------------------------------------------
Total    100.000 100.0000
Normalization factor =  1.6311
```
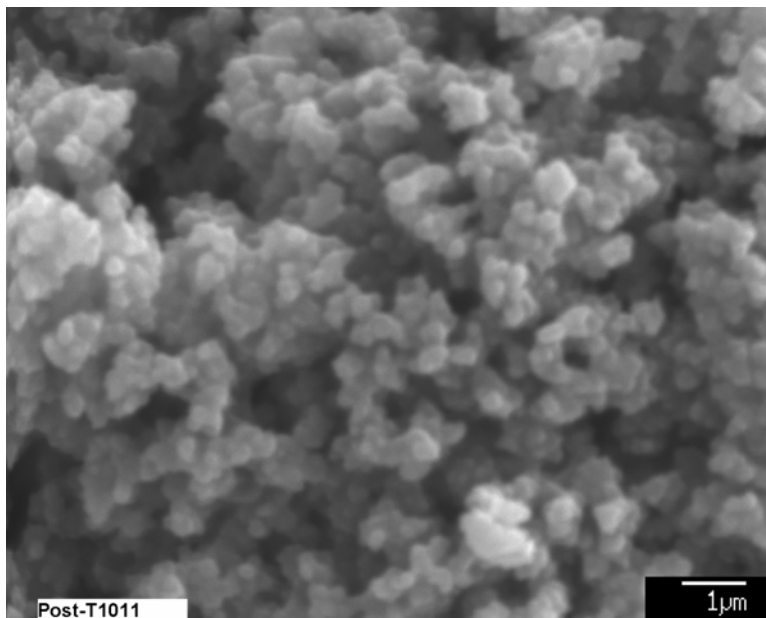
**Figure D-20.** End-test archive, post-T1 sample #3 SEM (POST-T1011) magnified 10,000 times; close-up of the white surface, as shown in Figure D-18.
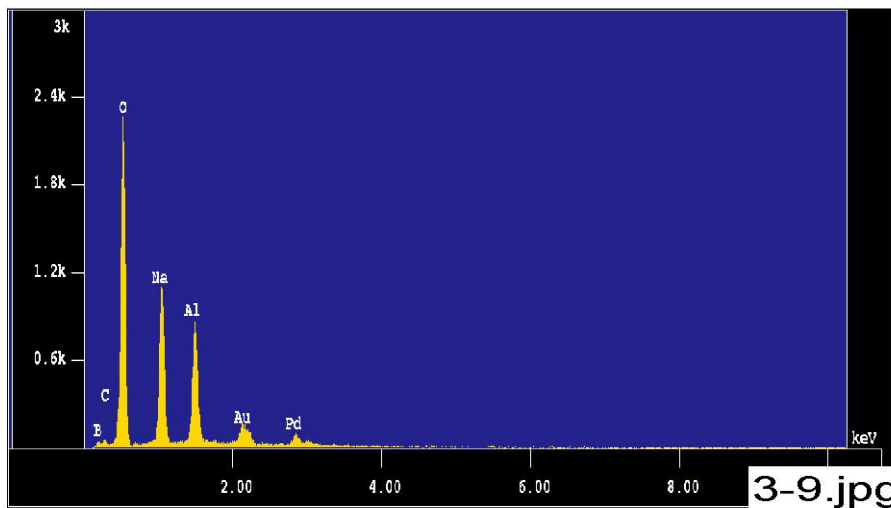


**Figure D-21.** End-test archive, post-T1 sample #3 counting spectrum (EDS 3-9) on another area of the white surface, as shown in Figure D-20.

The results from the chemical composition analysis for EDS 3-9 are given in Table D-7.

**Table D-7.        The Chemical Compositions for EDS 3-9**

```
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Group     : NRC
Sample    : Post-T1  ID# : 9      SEM# 3 (Day 30)
Comment   : white coarse surface area analysis -bottom of flake
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.022E-09 A
            Stage Point : X=16.404 Y=65.334 Z=10.967
            Acq. Date   : Tue Jan 18 12:12:46 2005


Element    Mode    ROI(KeV)    K-ratio(%)  +/-     Net/Background
  O K      Normal  0.25- 0.77   47.7843   0.0056     19050 /      44
  Na K     Normal  0.81- 1.27    8.6181   0.0023      9872 /      42
  Al K     Normal  1.19- 1.83    4.8672   0.0007      8052 /      48
  B K      Normal  0.00- 0.36    0.8142   0.0003       135 /      16
---------------------------------------------------------------------
                         Chi_square = 59.4370

Element Mass%    Atomic%   ZAF      Z      A      F
  O     57.627  59.8374 0.7343 0.9763 0.7522 0.9999
  Na    17.957  12.9762 1.2687 1.0310 1.2309 0.9997
  Al    11.219   6.9075 1.4034 1.0039 1.3980 1.0000
  B     13.197  20.2790 9.8692 1.1335 8.7068 1.0000
- ------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.6424
```
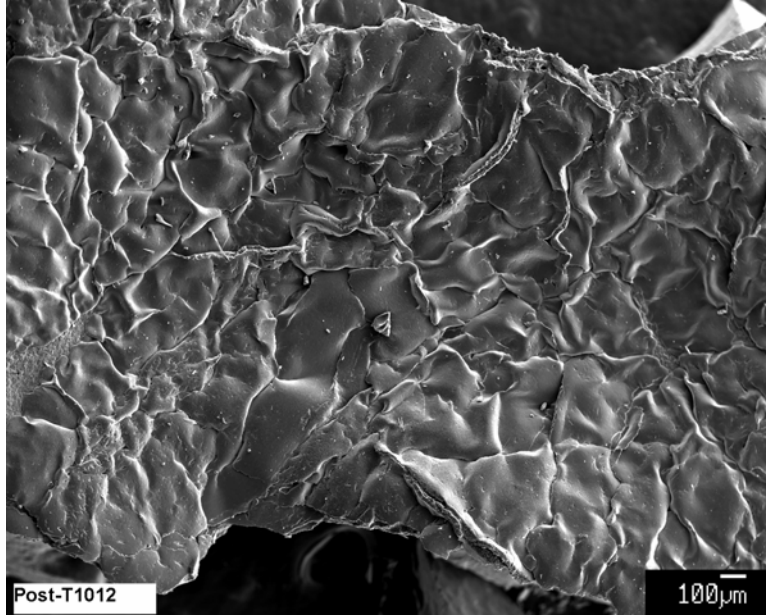
**Figure D-22.** End-test archive, post-T1 sample #3 SEM image (POST-T1012) magnified 40 times; overview of the smooth and hard brown fragment with wrinkled surface.
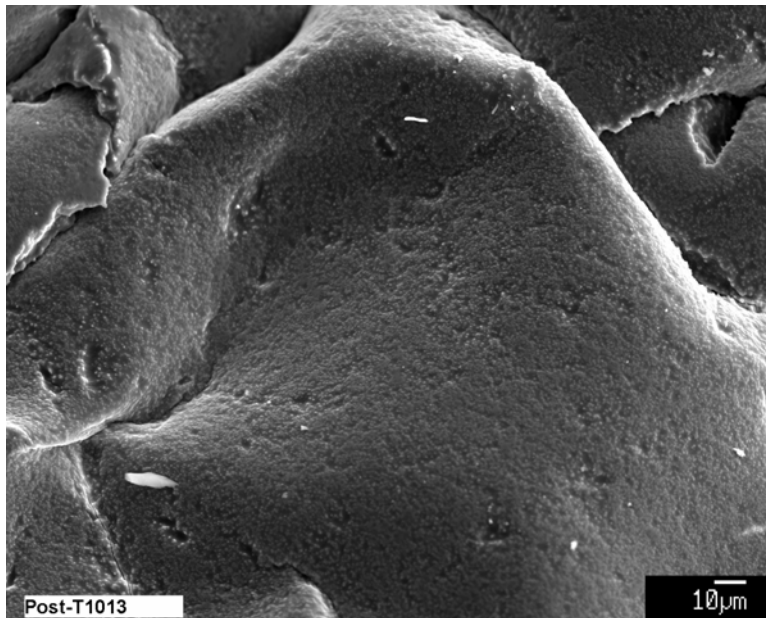


**Figure D-23.** End-test archive, post-T1 sample #3 SEM image (POST-T10013) magnified 500 times on the wrinkled surface, as shown in Figure D-22.
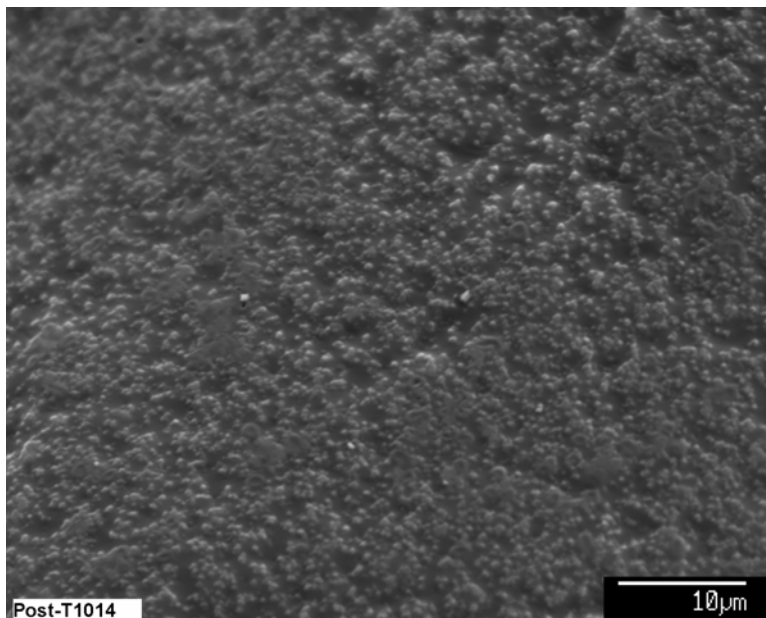
**Figure D-24.** End-test archive, post-T1 sample #3 SEM image (POST-T1014) magnified 2000 times; close-up on the wrinkled surface in SEM image (POST-T1013), as shown in Figure D-23.
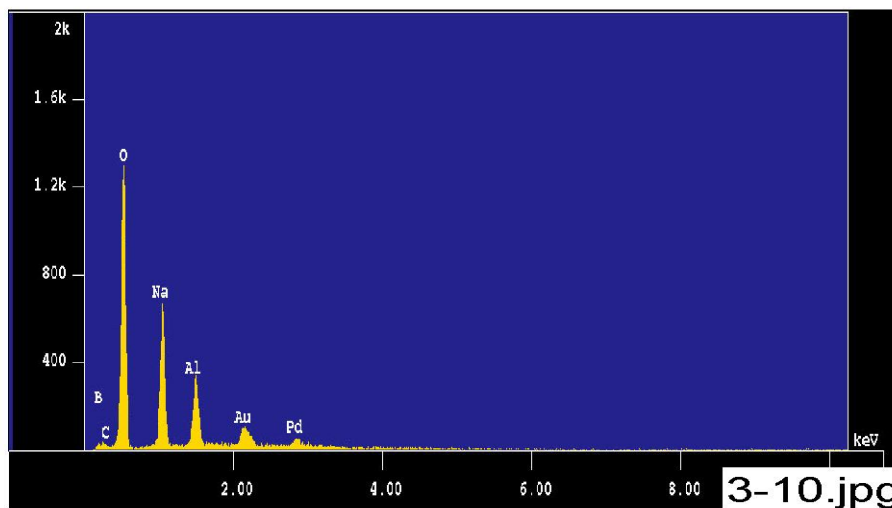


**Figure D-25.** End-test archive, post-T1 sample #3 counting spectrum (EDS 3-10) on an area (10 × 10 µm) of the wrinkled surface in the SEM image (POST-T1014), as shown in Figure D-24.

The results from the chemical composition analysis for EDS 3-10 are given in Table D-8.

**Table D-8.      The Chemical Compositions for EDS 3-10**

```
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Group      : NRC
Sample     : Post-T1  ID# : 10     SEM#3 (Day 30)
Comment    : smooth surface area analysis      - top of flake
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV        Probe Current : 9.846E-10 A
             Stage Point : X=14.364 Y=69.744 Z=10.967
             Acq. Date   : Tue Jan 18 12:26:01 2005


Element     Mode     ROI(KeV)    K-ratio(%)  +/-     Net/Background
  O K      Normal   0.25- 0.77    27.4160   0.0042    10530 /      30
  Na K     Normal   0.81- 1.27     5.1217   0.0018     5652 /      22
  Al K     Normal   1.19- 1.83     1.9239   0.0005     3066 /      31
  B K      Normal   0.00- 0.36     0.4361   0.0002       70 /      10
----------------------------------------------------------------------
                          Chi_square = 35.3338

Element Mass%   Atomic%   ZAF      Z      A      F
    O   59.189  61.3093 0.7111 0.9776 0.7274 0.9999
    Na  19.917  14.3574 1.2809 1.0324 1.2409 0.9999
    Al   8.376   5.1446 1.4341 1.0053 1.4265 1.0000
    B   12.518  19.1887 9.4555 1.1351 8.3304 1.0000
 ----------------------------------------------------------------------
 .otal  100.000 100.0000
Normalization factor =  3.0360
```
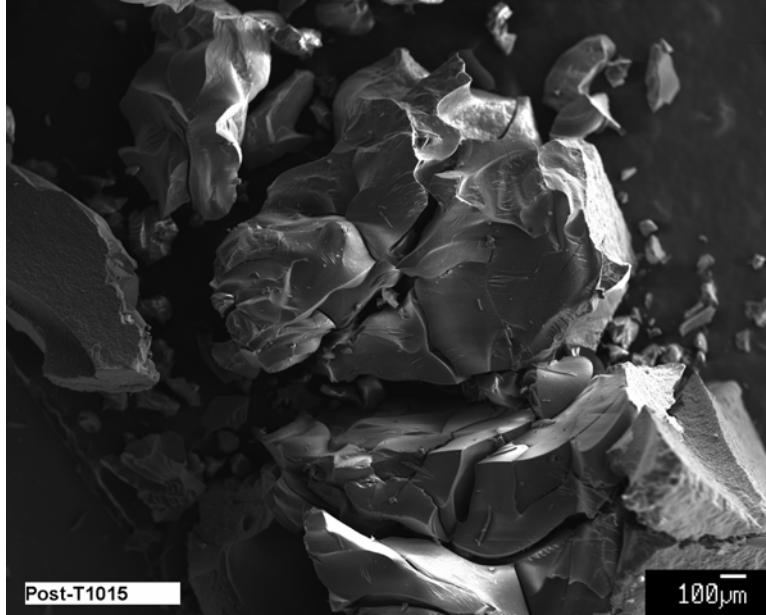
**Figure D-26.** End-test archive, post-T1 sample #3 SEM image (POST-T1015) magnified 40 times; overview of the soft tan particles with concoidal fracturing.
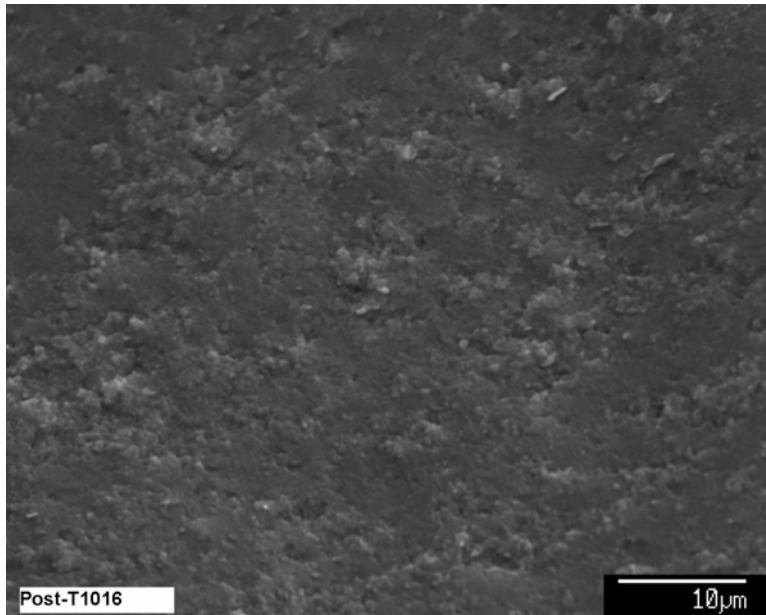


**Figure D-27.** End-test archive, post-T1 sample #3 SEM image (POST-T1016) magnified 2000 times on the surface of the concoidal fracture, as shown in Figure D-26.
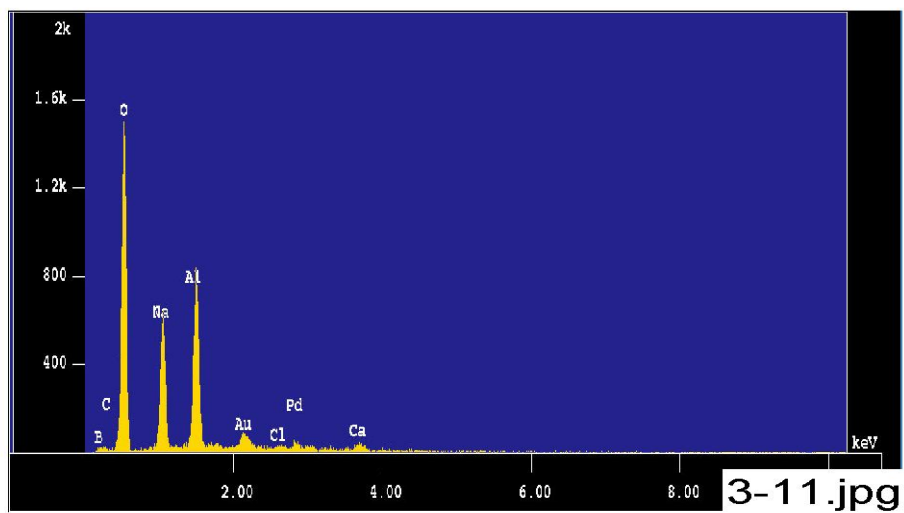
**Figure D-28.** End-test archive, post-T1 sample #3 counting spectrum (EDS 3-11) on an area (10 × 10 μm) on the surface in SEM image POST-T1016, as shown in Figure D-27.

The results from the chemical composition analysis for EDS 3-11 are given in Table D-9.

**Table D-9.    The Chemical Compositions for EDS 3-11**

```
Jan 18 12:38 2005  /tmp/eds_pout.log Page 1


Group      : NRC
Sample     : Post-T1  ID# : 11  SEM#3 (Day 30)
Comment    : concoidal surface area analysis
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec   Aperture #    : 1
            Acc. Volt   : 15.0 KV         Probe Current : 9.560E-10 A
            Stage Point : X=19.341 Y=67.891 Z=10.967
            Acq. Date   : Tue Jan 18 12:36:40 2005


Element    Mode    ROI(KeV)    K-ratio(%)  +/-     Net/Background
  O K     Normal   0.25- 0.77   33.3149   0.0045   12424 /      24
 Na K     Normal   0.81- 1.27    5.0959   0.0018    5460 /      26
 Al K     Normal   1.19- 1.83    4.9523   0.0007    7663 /      28
  B K     Normal   0.00- 0.36    0.0000   0.0000       0 /      14
 Ca K     Normal   3.39- 4.30    0.6178   0.0012     433 /      12
 Cl K     Normal   2.34- 3.06    0.2676   0.0007     264 /      28
-------------------------------------------------------------------
                      Chi_square = 22.8528

Element Mass%   Atomic%   ZAF      Z      A      F
   O    62.147  72.2967 0.7289 0.9919 0.7349 0.9999
   Na   17.338  14.0366 1.3295 1.0471 1.2705 0.9994
   Al   18.165  12.5300 1.4333 1.0193 1.4062 1.0000
    B    0.000   0.0000 12.1965 1.1519 10.5881 1.0000
   Ca    1.604   0.7446 1.0142 1.0182 0.9960 1.0000
   Cl    0.747   0.3921 1.0905 1.0507 1.0384 0.9995
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  2.5592
```
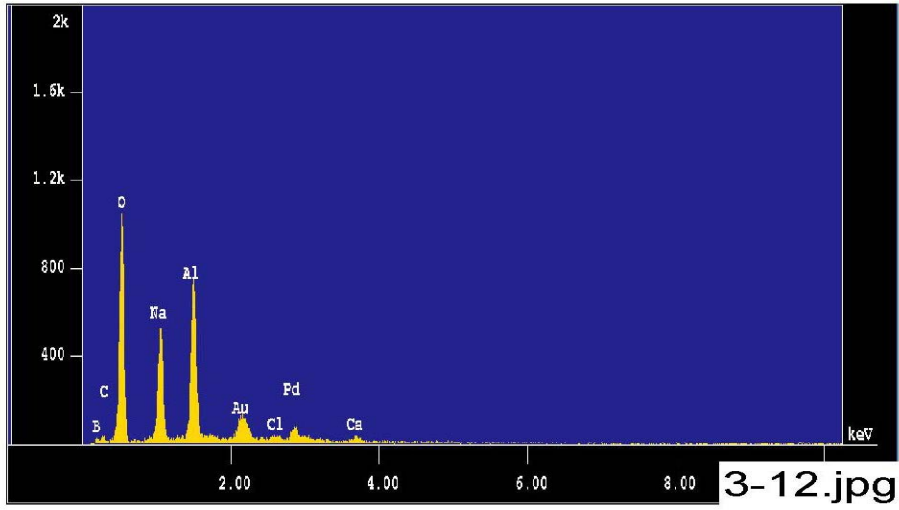
**Figure D-29.    End-test  archive,  post-T1  sample  #3  counting  spectrum  (EDS  3-12)  on  another concoidal  surface  of  a  different  particle  in  SEM  image  POST-T1016,  as  shown  in Figure D-27.**

The results from the chemical composition analysis for EDS 3-12 are given in Table D-10.

**Table D-10.     The Chemical Compositions for EDS 3-12**

```
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Group      : NRC
Sample     : Post-T1  ID# : 12      SEM#3 (Day 30)
Comment    : concoidal surface area analysis
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV         Probe Current : 9.432E-10 A
             Stage Point : X=20.778 Y=66.810 Z=10.967
             Acq. Date   : Tue Jan 18 12:42:24 2005


Element    Mode     ROI(KeV)    K-ratio(%)   +/-     Net/Background
  O K      Normal   0.25- 0.77   24.4363    0.0039      8991 /      30
 Na K      Normal   0.81- 1.27    4.4039    0.0017      4655 /      30
 Al K      Normal   1.19- 1.83    4.5798    0.0007      6992 /      30
  B K      Normal   0.00- 0.36    0.3873    0.0002        59 /      14
 Ca K      Normal   3.39- 4.30    0.4315    0.0011       299 /      15
 Cl K      Normal   2.34- 3.06    0.2999    0.0008       292 /      32
--------------------------------------------------------------------
                       Chi_square = 15.3263

Element Mass%    Atomic%   ZAF      Z      A      F
   O    54.203  58.3833  0.8118  0.9762 0.8316 0.9999
   Na   15.113  11.3286  1.2559  1.0308 1.2192 0.9994
   Al   17.053  10.8912  1.3627  1.0036 1.3578 1.0000
   B    11.587  18.4692 10.9486  1.1335 9.6590 1.0000
   Ca    1.174   0.5046  0.9954  1.0035 0.9920 1.0000
   Cl    0.870   0.4231  1.0623  1.0350 1.0268 0.9996
--------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  2.7325
```
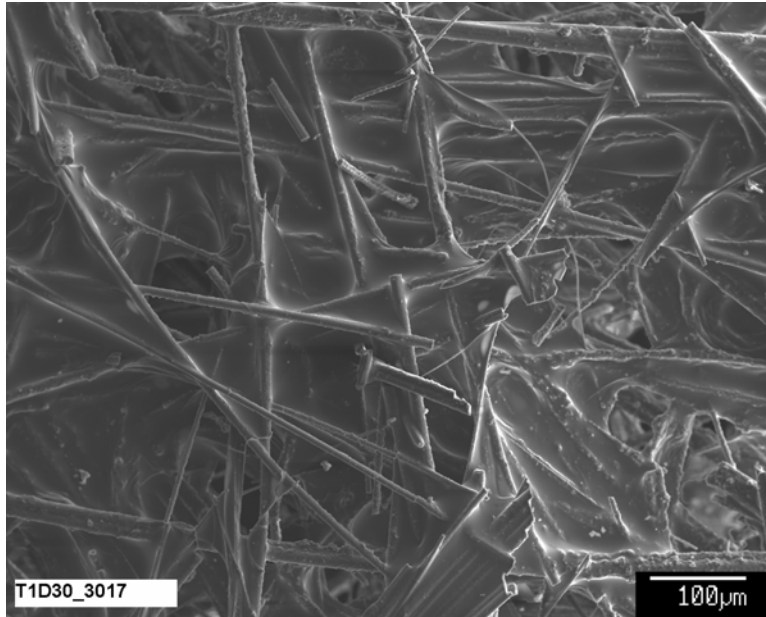
**Figure D-30.** Day-30, T1D30 sample #3 SEM image (T1D30-3017) magnified 150 times; overview of fiberglass from high-flow sacrificial sample.
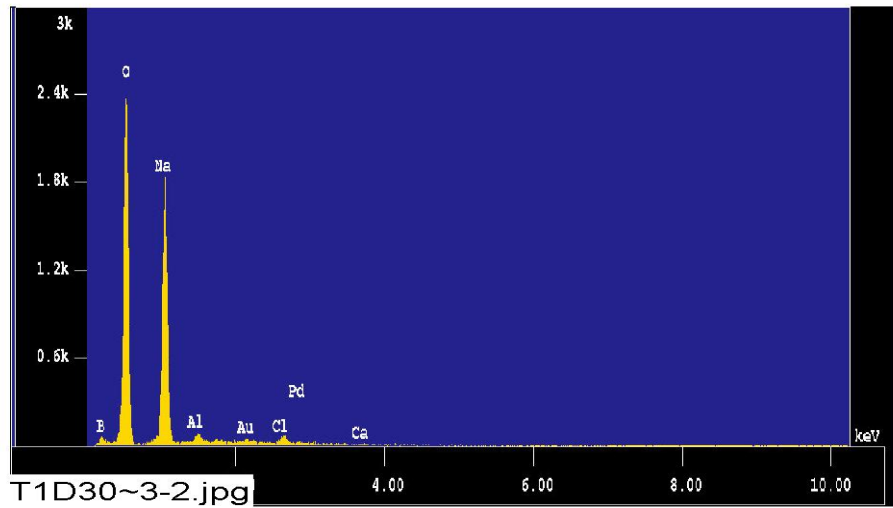


**Figure D-31.** Day-30, T1D30 sample #3 counting spectrum (EDS T1D30-3-2) of the web-like film between the fibers.

The results from the chemical composition analysis for EDS T1D30-3-2 are given in Table D-11.

**Table D-11.    The Chemical Compositions for EDS T1D30-3-2**

```
Jan 18 15:18 2005  /tmp/eds_pout.log Page 1


Group      : NRC
Sample     : T1D30  ID# : 2        From sample #3 (12/21/04)
Comment    : web-like film between fibers
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV        Probe Current : 1.010E-09 A
             Stage Point : X=21.255 Y=59.925 Z= 9.989
             Acq. Date   : Tue Jan 18 15:15:57 2005


Element    Mode     ROI(KeV)    K-ratio(%)  +/-     Net/Background
  O K      Normal   0.25- 0.77   50.7380   0.0058    19990 /      36
 Na K      Normal   0.81- 1.27   13.1256   0.0028    14858 /      35
 Al K      Normal   1.19- 1.83    0.2843   0.0003      465 /      33
  B K      Normal   0.00- 0.36    1.3261   0.0003      217 /      20
 Ca K      Normal   3.39- 4.30    0.1149   0.0009       85 /      19
 Cl K      Normal   2.34- 3.06    0.6698   0.0009      699 /      38
-----------------------------------------------------------------
                     Chi_square = 33.8015

Element Mass%    Atomic%   ZAF      Z      A      F
    O   56.581   57.0707 0.7265 0.9724 0.7472 0.9999
   Na   25.315   17.7694 1.2565 1.0270 1.2231 1.0003
   Al    0.648    0.3878 1.4856 1.0001 1.4855 1.0000
    B   16.227   24.2224 7.9723 1.1289 7.0619 1.0000
   Ca    0.173    0.0697 0.9809 1.0008 0.9801 1.0000
   Cl    1.055    0.4801 1.0259 1.0319 0.9943 0.9999
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.5350
```

D-39
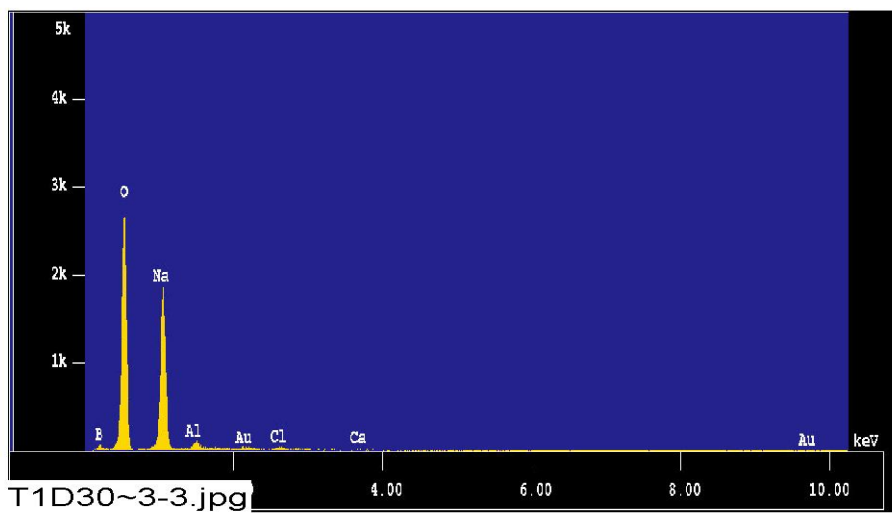
**Figure D-32.** **Day-30, T1D30 sample #3 counting spectrum (EDS T1D30-3-3) of the web-like film between fibers in Figure D-30; replicate analysis at second location.**

The results from the chemical composition analysis for EDS T1D30-3-3 are given in Table D-12.

**Table D-12.    The Chemical Compositions for EDS T1D30-3-3**

```
Jan 18 15:24 2005  /tmp/eds_pout.log Page 1


Group      : NRC
Sample     : T1D30  ID# : 3     From sample #3 (12/21/04)
Comment    : web-like film between fibers
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV        Probe Current : 1.003E-09 A
             Stage Point : X=21.255 Y=59.925 Z= 9.989
             Acq. Date   : Tue Jan 18 15:23:07 2005


Element    Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
  O K      Normal   0.25- 0.77  57.2962   0.0062   22418 /       34
 Na K      Normal   0.81- 1.27  14.2524   0.0029   16022 /       28
 Al K      Normal   1.19- 1.83   0.4161   0.0003     676 /       31
  B K      Normal   0.00- 0.36   1.7870   0.0003     291 /       14
 Ca K      Normal   3.39- 4.30   0.1173   0.0009      86 /       14
 Cl K      Normal   2.34- 3.06   0.2448   0.0008     254 /       28
-------------------------------------------------------------------
                       Chi_square = 36.9226

Element Mass%   Atomic%   ZAF      Z      A      F
    O   56.205  55.6375 0.7308 0.9699 0.7536 0.9999
   Na   23.929  16.4842 1.2508 1.0244 1.2207 1.0003
   Al    0.818   0.4802 1.4645 0.9976 1.4680 1.0000
    B   18.559  27.1876 7.7372 1.1259 6.8718 1.0000
   Ca    0.154   0.0607 0.9760 0.9986 0.9773 1.0000
   Cl    0.335   0.1499 1.0209 1.0295 0.9917 0.9999
-------------------------------------------------------------------
Total  100.000 100.0000
Normalization factor =  1.3423
```

**Figure D-33.    Day-30, T1D30 sample #3 counting spectrum (EDS T1D30-3-4) of the web-like film between fibers in Figure D-30; replicate analysis at a third location.**

The results from the chemical composition analysis for EDS T1D30-3-4 are given in Table D-13.

**Table D-13.     The Chemical Compositions for EDS T1D30-3-4**

```
Jan 18 15:31 2005  /tmp/eds_pout.log Page 1


Group      : NRC
Sample     : T1D30  ID# : 4     From sample #3 (12/21/04)
Comment    : web-like film between fibers
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV         Probe Current : 9.984E-10 A
             Stage Point : X=21.255 Y=59.925 Z= 9.989
             Acq. Date   : Tue Jan 18 15:29:46 2005


Element    Mode     ROI(KeV)    K-ratio(%)   +/-     Net/Background
  O K      Normal   0.25- 0.77   58.2719    0.0062    22695 /      27
 Na K      Normal   0.81- 1.27   13.4543    0.0029    15055 /      44
 Al K      Normal   1.19- 1.83    1.3081    0.0004     2114 /      40
  B K      Normal   0.00- 0.36    1.7140    0.0003      278 /      14
 Ca K      Normal   3.39- 4.30    0.2180    0.0010      160 /      16
 Cl K      Normal   2.34- 3.06    0.2771    0.0008      286 /      27
-----------------------------------------------------------------------
                        Chi_square = 37.2253

Element Mass%   Atomic%   ZAF      Z      A       F
   O    56.633  56.3842 0.7380 0.9705 0.7605 0.9999
   Na   22.285  15.4402 1.2578 1.0250 1.2269 1.0002
   Al    2.494   1.4723 1.4479 0.9983 1.4504 1.0000
   B    17.934  26.4238 7.9459 1.1267 7.0522 1.0000
   Ca    0.281   0.1115 0.9776 0.9992 0.9784 1.0000
   Cl    0.374   0.1679 1.0242 1.0301 0.9944 0.9999
-----------------------------------------------------------------------
Total  100.000 100.0000
Normalization factor =  1.3168
```
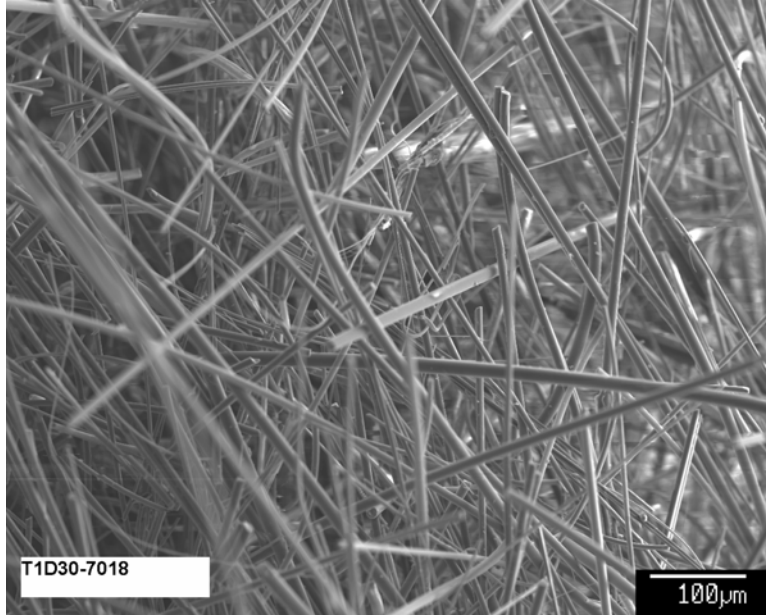
**Figure D-34.   T1D30 sample #7 SEM image (T1D30-7018) magnified 150 times; overview of clean fiberglass.**
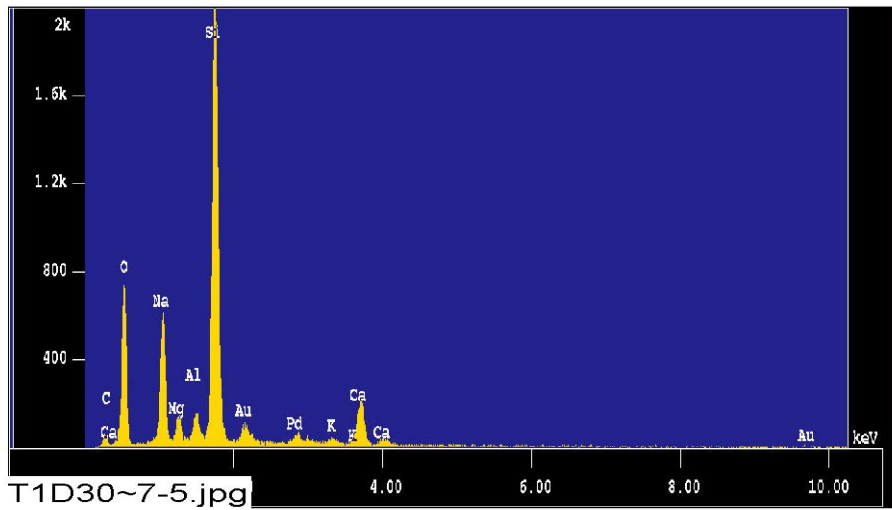


**Figure D-35.   T1D30 sample #7 counting spectrum (EDS T1D30-7-5) on an individual thick fiber.**

The results from the chemical composition analysis for EDS T1D30-7-5 are given in Table D-14.

**Table D-14.     The Chemical Compositions for EDS T1D30-7-5**

```
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Group      : NRC
Sample     : T1D30  ID# : 5
Comment    : clean glass filbers      From sample #7 (12/21/04)
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #     : 1
             Acc. Volt   : 15.0 KV        Probe Current : 1.003E-09 A
             Stage Point : X=15.076 Y=66.231 Z=11.620
             Acq. Date   : Tue Jan 18 15:44:58 2005


Element    Mode      ROI(KeV)    K-ratio(%)   +/-    Net/Background
  O K      Normal   0.25- 0.77    17.6048    0.0036    6888 /      36
 Na K      Normal   0.81- 1.27     4.7404    0.0018    5329 /      83
 Mg K      Normal   0.97- 1.57     0.5839    0.0004     954 /      80
 Al K      Normal   1.19- 1.83     0.6942    0.0004    1127 /     144
 Si K      Normal   1.50- 2.05    14.3850    0.0013   21778 /      94
 Ca K      Normal   3.39- 4.30     3.8044    0.0021    2799 /      24
  C K      Normal   0.09- 0.46     0.0969    0.0004      56 /      76
-----------------------------------------------------------------
                    Chi_square =  9.1028

Element Mass%    Atomic%    ZAF      Z      A      F
  O    41.639   54.6247  1.2000 0.9920 1.2097 1.0000
  Na   11.653   10.6381  1.2471 1.0468 1.1926 0.9990
  Mg    1.750    1.5106  1.5203 0.9854 1.5481 0.9966
  Al    1.834    1.4264  1.3402 1.0187 1.3280 0.9907
  Si   34.048   25.4442  1.2009 0.9959 1.2060 0.9998
  Ca    7.768    4.0679  1.0359 1.0160 1.0197 1.0000
  C     1.309    2.2881  6.8540 1.0403 6.5890 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.9710
```
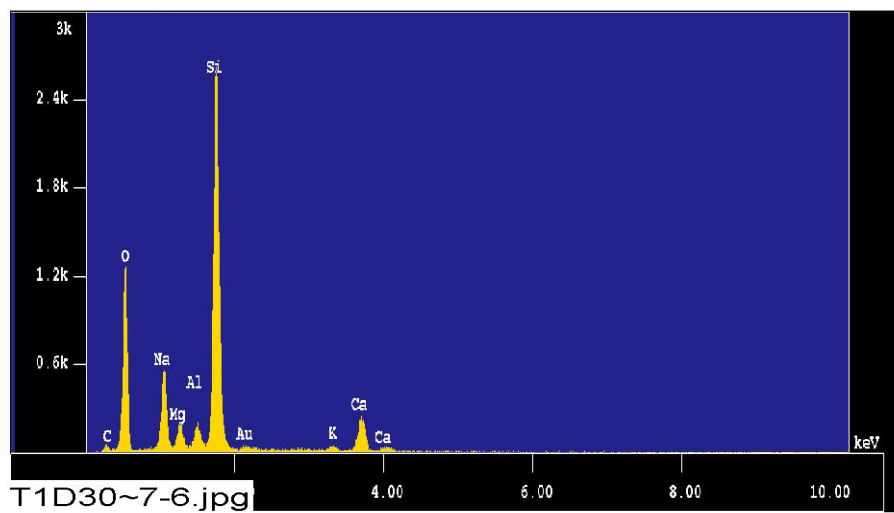
**Figure D-36.  T1D30 sample #7 counting spectrum (EDS T1D30-7-6) on an individual clump fiber.**

The results from the chemical composition analysis for EDS T1D30-7-6 are given in Table D-15.

**Table D-15.    The Chemical Compositions for EDS T1D30-7-6**

```
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Group      : NRC
Sample     : T1D30  ID# : 6
Comment    : clean glass filbers
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #   : 1
            Acc. Volt   : 15.0 KV         Probe Current : 9.958E-10 A
            Stage Point : X=14.777 Y=66.987 Z=11.620
            Acq. Date   : Tue Jan 18 15:54:13 2005


Element    Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
  O K      Normal   0.25- 0.77  27.3788   0.0043    10635 /      48
 Na K      Normal   0.81- 1.27   4.3174   0.0018     4819 /     114
 Mg K      Normal   0.97- 1.57   0.8765   0.0004     1421 /      82
 Al K      Normal   1.19- 1.83   0.8334   0.0004     1343 /     164
 Si K      Normal   1.50- 2.05  17.8096   0.0014    26769 /     112
 Ca K      Normal   3.39- 4.30   4.4556   0.0022     3255 /      23
  K K      Normal   3.01- 3.86   0.6160   0.0018      376 /      24
  C K      Normal   0.09- 0.46   0.0000   0.0000        0 /     135
---------------------------------------------------------------
                       Chi_square = 17.7249

Element Mass%    Atomic%   ZAF     Z      A      F
    O    47.563  61.4845 1.1461 0.9931 1.1541 1.0000
   Na     8.481   7.6293 1.2960 1.0480 1.2378 0.9990
   Mg     1.992   1.6948 1.4995 0.9866 1.5250 0.9967
   Al     1.682   1.2893 1.3316 1.0199 1.3175 0.9910
   Si    32.230  23.7332 1.1939 0.9971 1.1977 0.9998
   Ca     7.004   3.6143 1.0372 1.0173 1.0195 1.0000
    K     1.048   0.5546 1.1230 1.2432 0.9109 0.9917
    C     0.000   0.0000 6.4648 1.0414 6.2081 1.0000
---------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.5157
```

**Figure D-37.** Day-30, post-T1 sample P1 SEM image (Post-T1017) magnified 35 times; overview of the fibers and the other debris.
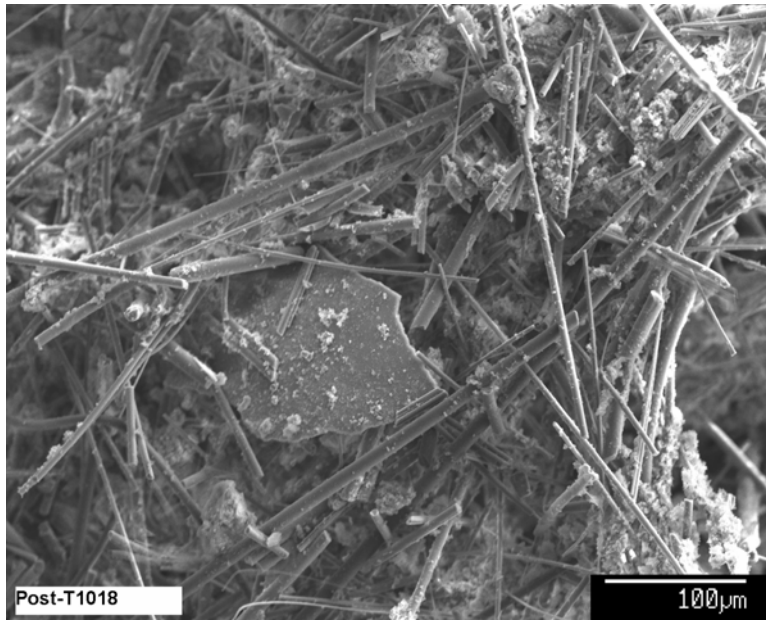


**Figure D-38.** Day-30, post-T1 sample P1 SEM-SE image (Post-T1018) magnified 220 times on large particles at left corner of image T1017, as shown in Figure D-37.
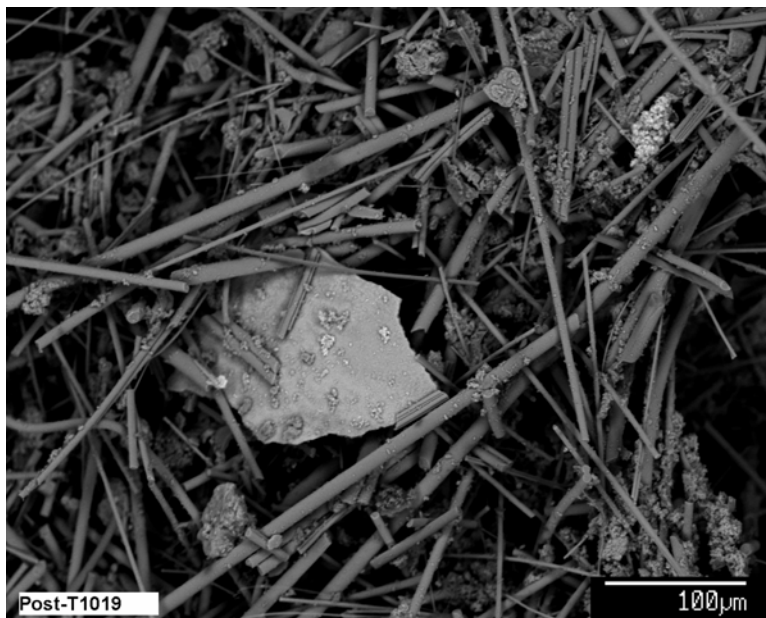
**Figure D-39.   Day-30, post-T1 sample P1 SEM-BSE image (Post-T1019) magnified 220 times on the same field as image T1018, as shown Figure D-38.**
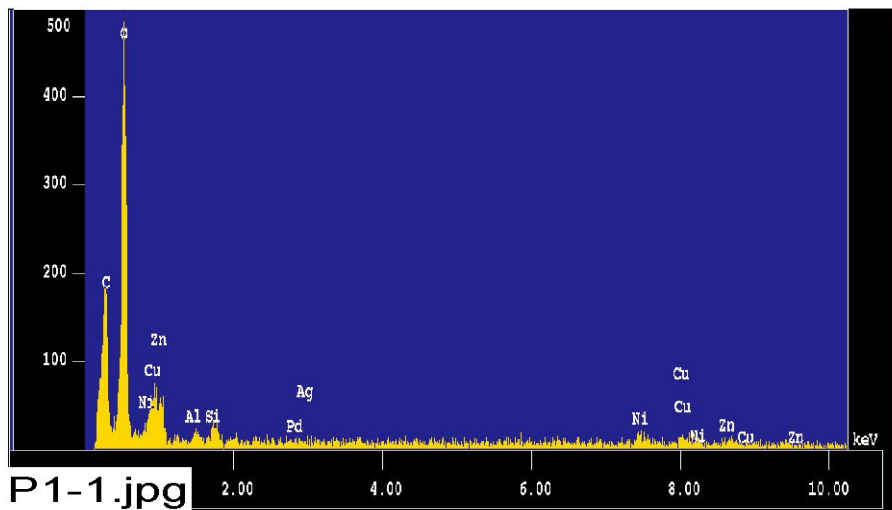


**Figure D-40.   Day-30, post-T1 sample P1 counting spectrum (EDS P1-1) on the large flake in image T1018, as shown in Figure D-38.**
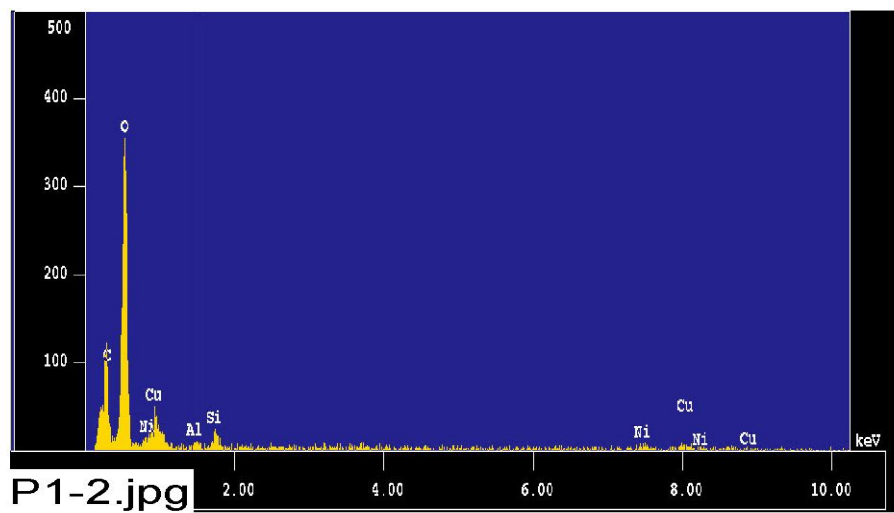
**Figure D-41.** Day-30, post-T1 sample P1 counting spectrum (EDS P1-2) on the small cluster to lower left of large flake shown in image T1018, as shown in Figure D-38.
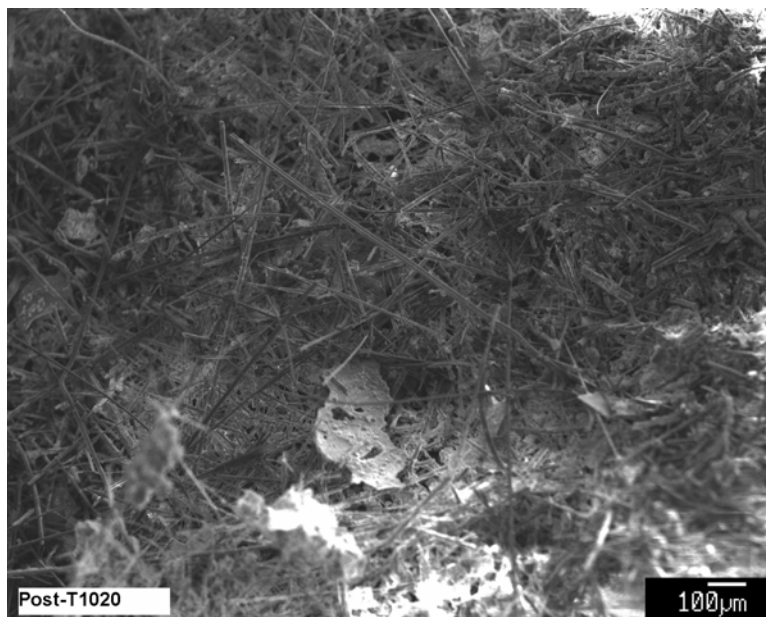


**Figure D-42.** Day-30, post-T1 sample P1 SEM-SE image (post-T1020) magnified 60 times on a different area of sample P1.
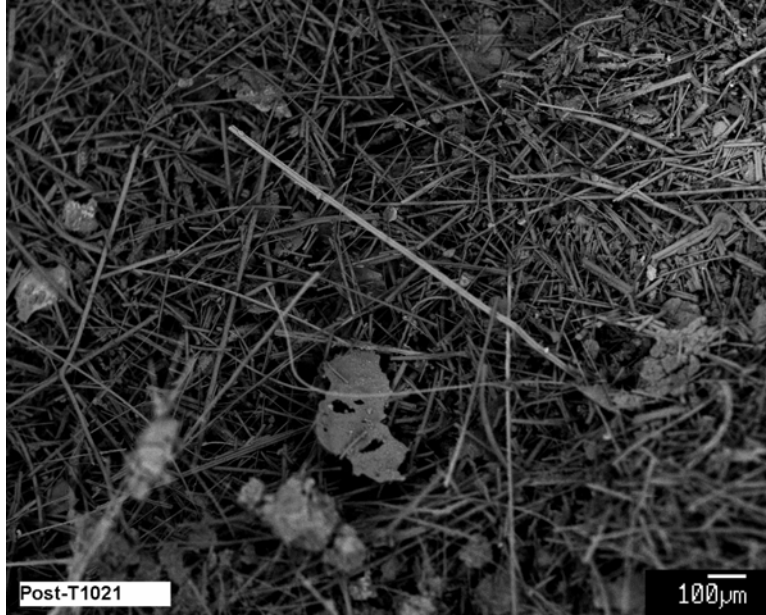
**Figure D-43.** Day-30, post-T1 sample P1 SEM-BSE image (Post-T1021) magnified 60 times on the same field as image T1020, as shown in Figure D-42.
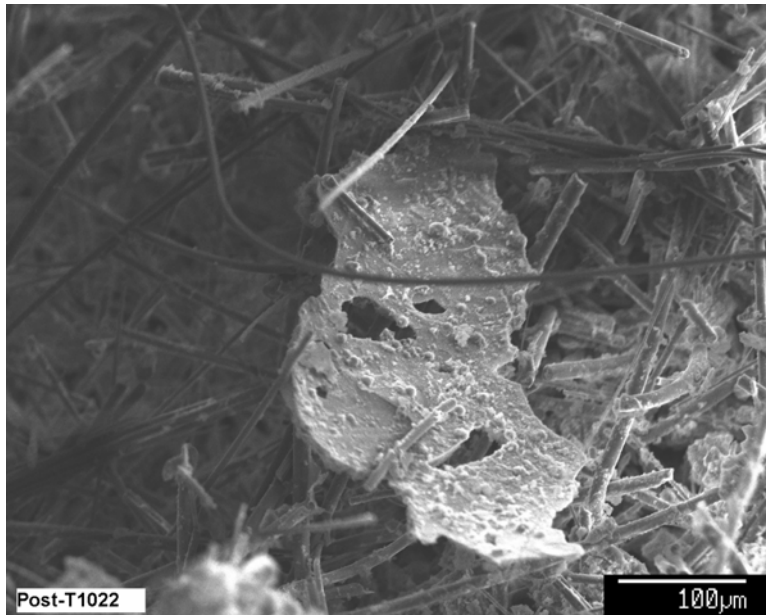


**Figure D-44.** Day-30, post-T1 sample P1 SEM-SE image (Post-T1022) magnified 200 times on the flake, as shown in Figure D-43.
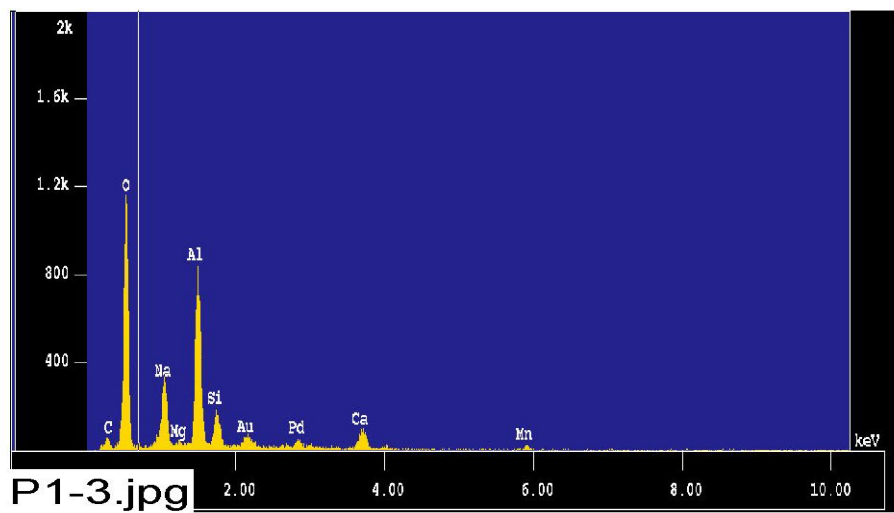
**Figure D-45.** Day-30, post-T1 sample P1 counting spectrum (EDS P1-3) on the particles in foreground below the flake shown in image T1021.

The results from the chemical composition analysis for EDS P1-3 are given in Table D-16.

**Table D-16.     The Chemical Compositions for EDS P1-3**

```
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Group      : NRC
Sample     : Post-T1_1-28-05 ID# : 3
Comment    : P1 particles
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #      : 1
            Acc. Volt   : 15.0 KV        Probe Current : 6.753E-10 A
            Stage Point : X=81.768 Y=61.832 Z=10.582
            Acq. Date   : Fri Jan 28 10:03:44 2005


Element     Mode      ROI(KeV)    K-ratio(%)  +/-    Net/Background
  O K       Normal   0.25- 0.77    39.9049   0.0044   10512 /      39
 Na K       Normal   0.83- 1.28     2.7379   0.0085    2268 /      30
 Mg K       Normal   1.03- 1.52     0.0604   0.0010      63 /     448
 Al K       Normal   1.26- 1.78     5.7166   0.0015    7138 /      79
 Si K       Normal   1.50- 2.07     1.1338   0.0010    1415 /     376
 Ca K       Normal   3.40- 4.30     1.7209   0.0063    1136 /      14
 Mn K       Normal   5.53- 6.82     1.1673   0.0008     282 /       7
  C K       Normal   0.09- 0.46     0.0000   0.0000       0 /     112
-----------------------------------------------------------------------
                         Chi_square =  8.7099

Element Mass%    Atomic%   ZAF      Z      A      F
   O    67.623  78.8082 0.7645 0.9886 0.7733 1.0000
   Na    7.221   5.8567 1.1899 0.9931 1.1973 1.0007
   Mg    0.157   0.1201 1.1709 0.9957 1.1772 0.9989
   Al   14.925  10.3139 1.1779 1.0013 1.1772 0.9993
   Si    3.112   2.0657 1.2380 0.9891 1.2518 0.9999
   Ca    3.755   1.7467 0.9843 1.0029 0.9824 0.9990
   Mn    3.208   1.0887 1.2397 1.2426 0.9977 1.0000
   C     0.000   0.0000 3.9691 1.0365 3.8297 0.9999
-----------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  2.2166
```

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**Figure D-46.** Day-30, post-T1-sample-Conc SEM-BSE image (Post-T1023) magnified 43 times; overview of portion of concrete fragment.
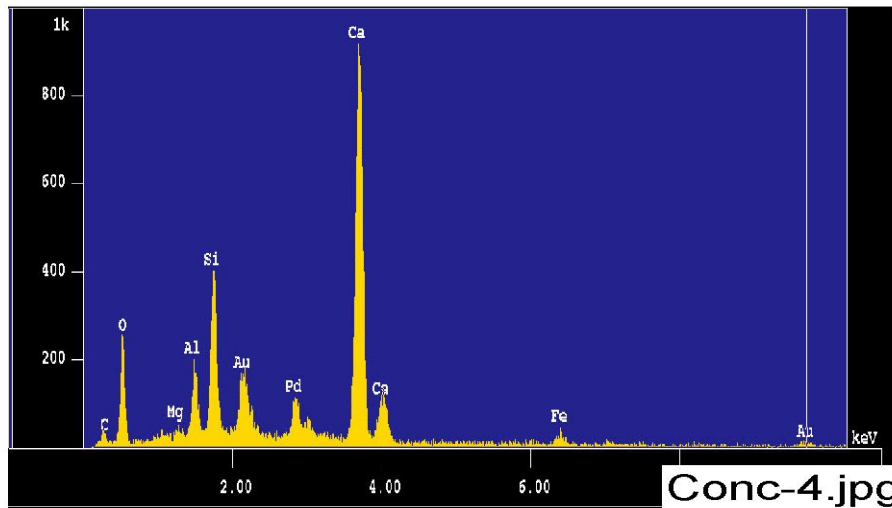


**Figure D-47.** Day-30, post-T1-sample-Conc counting spectrum (EDS Conc-4) on an average area (20 × 20 µm) of the concrete matrix magnified 5500 times.

The results from the chemical composition analysis for EDS Conc-4 are given in Table D-17.

**Table D-17.    The Chemical Compositions for EDS Conc-4**
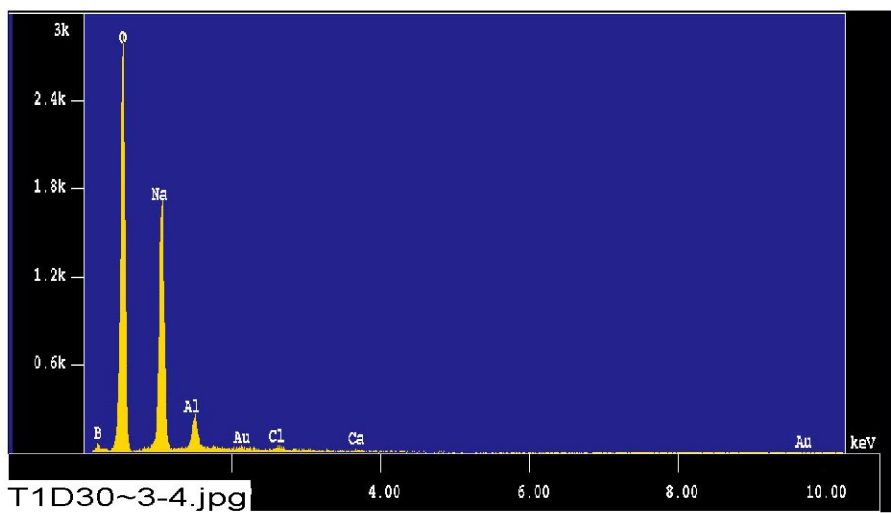
```
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Group     : NRC
Sample    : PostT1_1-28-05  ID# : 4
Comment   : Concrete
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.038E-09 A
            Stage Point : X=69.901 Y=61.652 Z=10.565
            Acq. Date   : Fri Jan 28 10:30:24 2005


Element    Mode     ROI(KeV)    K-ratio(%)  +/-    Net/Background
  O K      Normal   0.25- 0.77    5.6143   0.0021    2273 /     30
  Mg K     Normal   1.03- 1.52    0.1044   0.0010     168 /     98
  Al K     Normal   1.26- 1.78    0.7042   0.0008    1352 /    133
  Si K     Normal   1.50- 2.07    1.8080   0.0014    3468 /    130
  Ca K     Normal   3.40- 4.30   11.5423   0.0159   11712 /     18
  Fe K     Normal   6.04- 7.40    0.8690   0.0473     284 /     16
----------------------------------------------------------------------
                         Chi_square =  2.4699

Element Mass%   Atomic%   ZAF      Z      A      F
   O    43.793  64.5383  2.0502  0.9681  2.1178  1.0000
   Mg    0.453   0.4392  1.1408  0.9741  1.1708  1.0002
   Al    3.090   2.7000  1.1532  0.9792  1.1804  0.9977
   Si    7.508   6.3025  1.0914  0.9670  1.1299  0.9989
   Ca   41.877  24.6354  0.9536  0.9778  0.9756  0.9996
   Fe    3.279   1.3846  0.9919  0.9699  1.0219  1.0008
----------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  3.8047
```

**Figure D-48.** **Day-30, post-T1-sample-Conc SEM-SE image (Post-T1024) magnified 110 times on another area of the sample from the concrete fragment.**
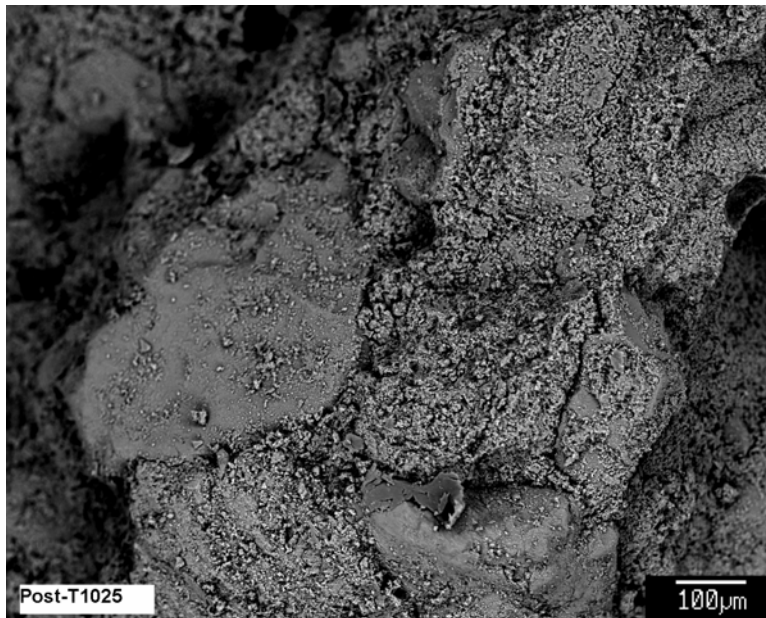


**Figure D-49.** **Day-30, post-T1-sample-Conc SEM-BSE image (Post-T1025) magnified 110 times on the same field as in image Post-T1024 (see Figure D-48).**
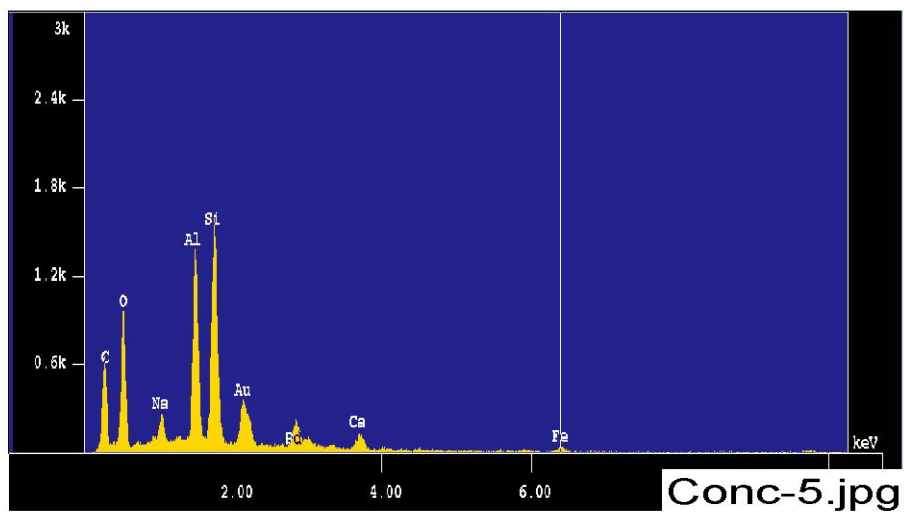
**Figure D-50.** Day-30, post-T1-sample-Conc counting spectrum (EDS Conc-5) on the aggregate particle in lower portion of image Post-T1025, as shown in Figure D-49.

The results from the chemical composition analysis for EDS Conc-5 are given in Table D-18.

**Table D-18.    The Chemical Compositions for EDS Conc-5**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 5
Comment    : Concrete aggregate
Condition : Full Scale   : 20KeV(10eV/ch,2Kch)
            Live Time    : 100.000 sec    Aperture #    : 1
            Acc. Volt    : 15.0 KV        Probe Current : 1.085E-09 A
            Stage Point : X=71.968 Y=63.927 Z=10.565
            Acq. Date    : Fri Jan 28 10:43:57 2005


Element      Mode     ROI(KeV)    K-ratio(%)  +/-     Net/Background
  C K       Normal   0.09- 0.46    6.4490    0.0009    4049 /      87
  O K       Normal   0.25- 0.77   20.8346    0.0042    8818 /     310
 Na K       Normal   0.83- 1.28    1.0347    0.0086    1377 /      96
 Al K       Normal   1.26- 1.78    5.6526    0.0020   11340 /     525
 Si K       Normal   1.50- 2.07    6.8576    0.0027   13750 /     686
 Ca K       Normal   3.40- 4.30    1.2480    0.0076    1324 /      35
 Fe K       Normal   6.04- 7.40    0.9509    0.0533     324 /      10
----------------------------------------------------------------------
                        Chi_square = 17.2326

Element Mass%   Atomic%   ZAF      Z      A      F
    C   33.796  44.7339 3.6197 1.0266 3.5261 1.0000
    O   42.274  42.0080 1.4015 0.9794 1.4310 1.0000
   Na    1.561   1.0798 1.0424 0.9843 1.0583 1.0007
   Al    8.522   5.0215 1.0414 0.9928 1.0523 0.9968
   Si   10.725   6.0712 1.0803 0.9810 1.1013 0.9999
   Ca    1.758   0.6972 0.9727 0.9964 0.9765 0.9997
   Fe    1.365   0.3885 0.9912 0.9949 0.9955 1.0008
----------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.4478
```

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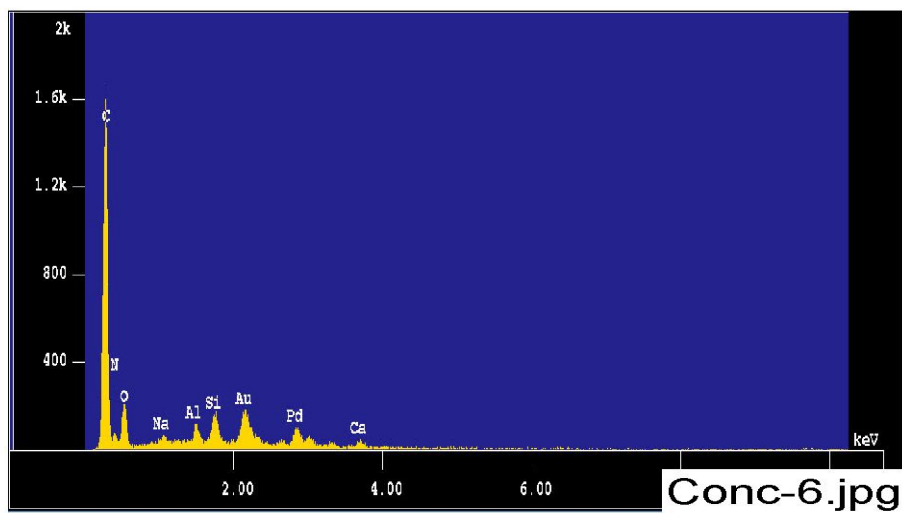
**Figure D-51.** Day-30, post-T1-sample-Conc counting spectrum (EDS Conc-6) on the surface granules on or in the concrete, as shown in image T1025.
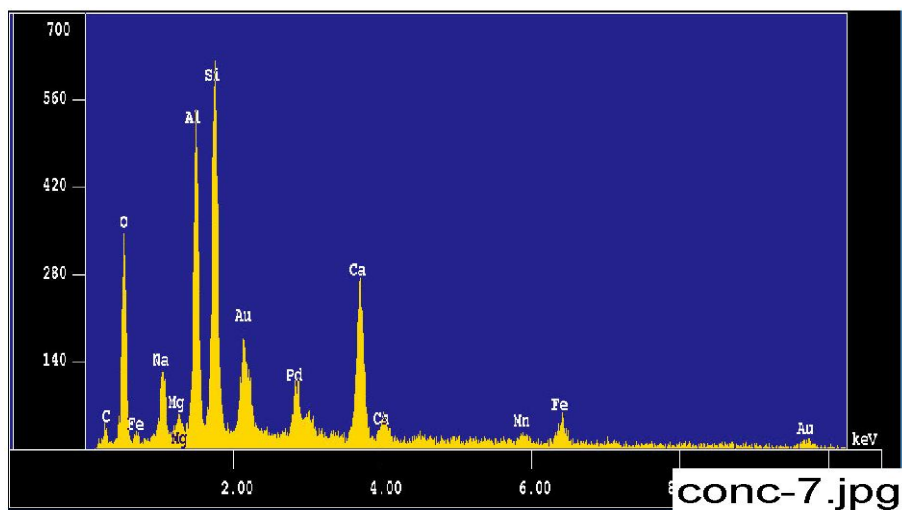


**Figure D-52.** Day-30, post-T1-sample-Conc counting spectrum (EDS Conc-7) on the concrete matrix behind surface granules, shown in image T1025.

The results from the chemical composition analysis for EDS Conc-7 are given in Table D-19.

**Table D-19.    The Chemical Compositions for EDS Conc-7**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 7
Comment    : Concrete matrix
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV        Probe Current : 1.104E-09 A
             Stage Point : X=71.902 Y=63.995 Z=10.565
             Acq. Date   : Fri Jan 28 10:51:55 2005


Element    Mode     ROI(KeV)   K-ratio(%)  +/-     Net/Background
  C K      Normal   0.09- 0.46   0.0000    0.0000      0 /      22
  O K      Normal   0.25- 0.77   7.5549    0.0026   3254 /      22
 Na K      Normal   0.83- 1.28   0.6424    0.0057    870 /      32
 Al K      Normal   1.26- 1.78   2.1633    0.0012   4416 /     220
 Si K      Normal   1.50- 2.07   2.6236    0.0017   5353 /     282
 Ca K      Normal   3.40- 4.30   2.9940    0.0094   3231 /      26
 Fe K      Normal   6.04- 7.40   1.2796    0.0545    444 /      10
 Mg K      Normal   1.03- 1.52   0.1059    0.0010    181 /     221
 Mn K      Normal   5.53- 6.82   0.6354    0.0009    251 /      14
---------------------------------------------------------------
                       Chi_square =  2.2310

Element Mass%   Atomic%   ZAF      Z      A      F
    C     0.000   0.0000 4.9670 1.0196 4.8717 0.9999
    O    44.480  61.9981 1.2202 0.9723 1.2551 0.9999
   Na     3.815   3.7002 1.2307 0.9761 1.2601 1.0006
   Al    12.105  10.0042 1.1597 0.9837 1.1830 0.9966
   Si    15.039  11.9407 1.1880 0.9715 1.2233 0.9997
   Ca    14.137   7.8657 0.9786 0.9830 0.9973 0.9983
   Fe     6.077   2.4265 0.9843 0.9761 1.0076 1.0008
   Mg     0.586   0.5374 1.1465 0.9785 1.1735 0.9985
   Mn     3.762   1.5272 1.2272 1.2146 1.0104 1.0000
---------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  4.8249
```
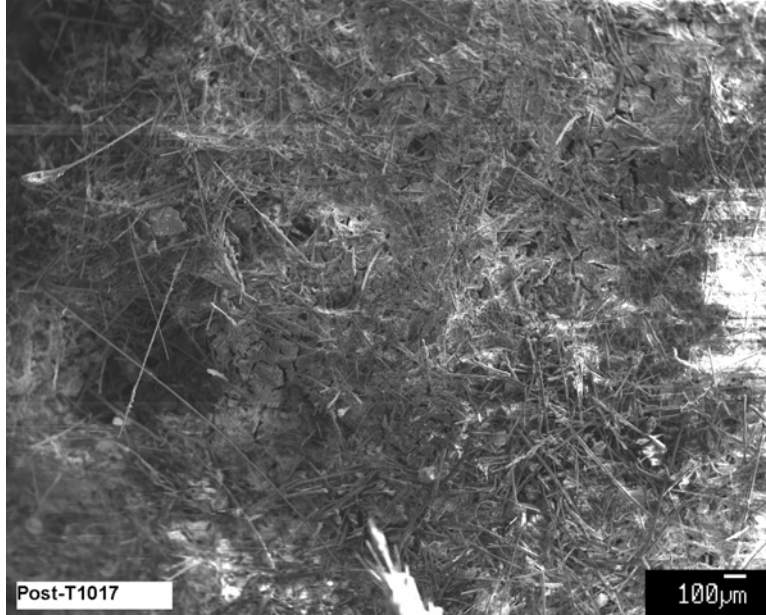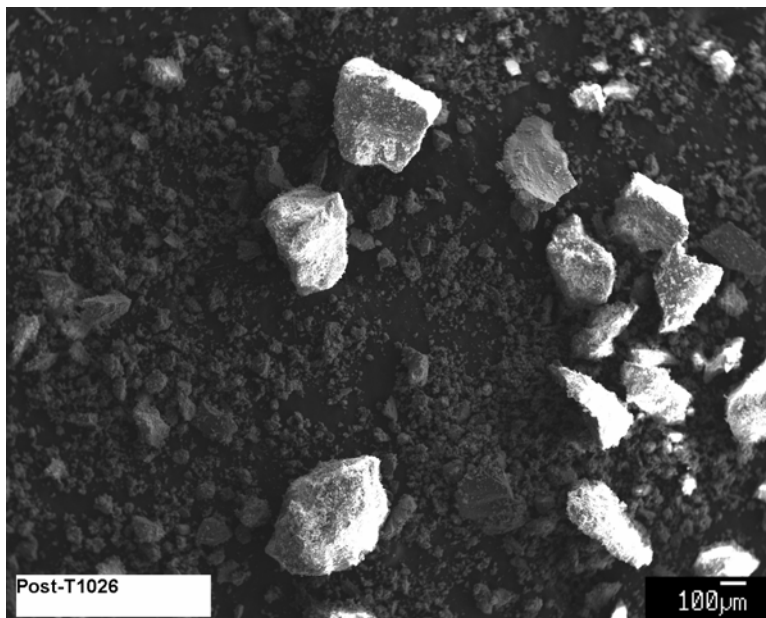
**Figure D-53.** Day-30, post-T1-sample-CD1 SEM-SE image (Post-T1026) magnified 40 times; overview of the dust particles mounted on carbon tape.
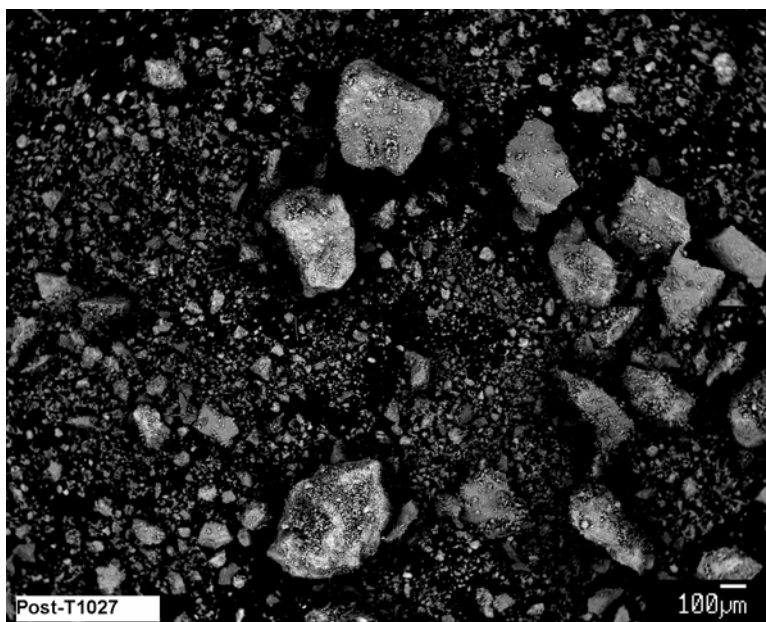


**Figure D-54.** Day-30, post-T1-sample-CD1 SEM-BSE image (Post-T1027) magnified 40 times on the same field as in image Post-T1026 (see Figure D-53).
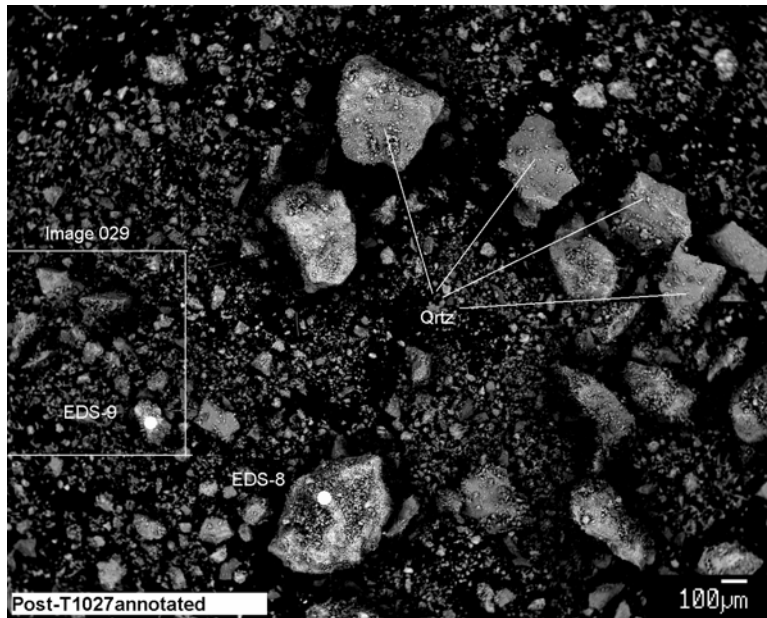
**Figure D-55.** Day-30, post-T1-sample-CD1 SEM-SE image (Post-T1027 annotated) magnified 40 times, annotating the EDS locations.
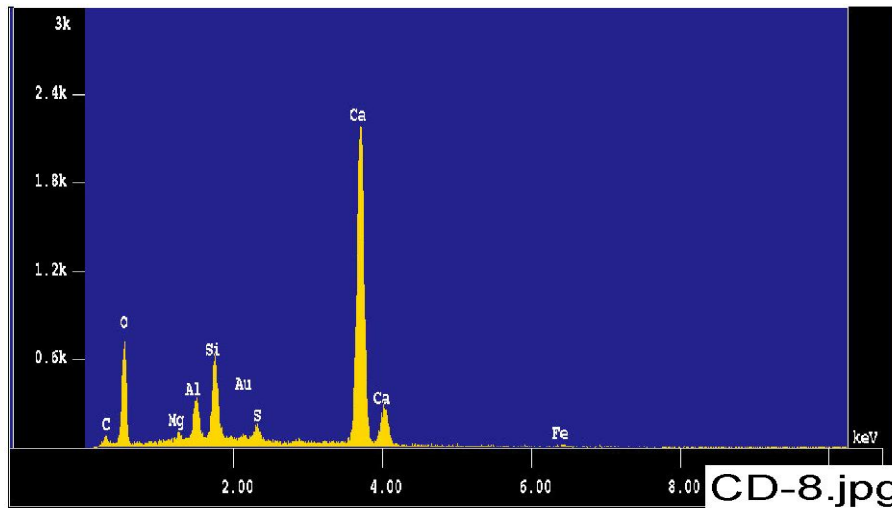


**Figure D-56.** Day-30, post-T1-sample-CD1 counting spectrum (EDS CD-8) on particle in the lower center of image Post-T1027 (see Figure D-55).

The results from the chemical composition analysis for EDS CD-8 are given in Table D-20.

**Table D-20.    The Chemical Compositions for EDS CD-8**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 8
Comment    : Concrete dust particle
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV        Probe Current : 1.146E-09 A
             Stage Point : X=43.880 Y=75.323 Z=10.565
             Acq. Date   : Fri Jan 28 11:15:28 2005


Element      Mode     ROI(KeV)    K-ratio(%)  +/-    Net/Background
  O K       Normal   0.25- 0.77   15.2816    0.0036    6832 /      50
  Mg K      Normal   1.03- 1.52    0.2151    0.0014     382 /     148
  Al K      Normal   1.26- 1.78    1.1786    0.0011    2497 /     259
  Si K      Normal   1.50- 2.07    2.4899    0.0018    5273 /     210
  S K       Normal   2.04- 2.71    0.9062    0.0014    1099 /      42
  Ca K      Normal   3.40- 4.30   26.1728    0.0242   29320 /      33
  Fe K      Normal   6.04- 7.40    0.2300    0.0445      83 /      12
-----------------------------------------------------------------
                        Chi_square =  5.1089

Element Mass%    Atomic%   ZAF      Z      A      F
     O   50.188  70.2325 2.0081 0.9733 2.0631 1.0000
    Mg    0.399   0.3673 1.1339 0.9796 1.1568 1.0006
    Al    2.215   1.8380 1.1492 0.9848 1.1689 0.9983
    Si    4.403   3.5098 1.0812 0.9726 1.1133 0.9986
     S    1.477   1.0310 0.9963 1.1195 0.8961 0.9932
    Ca   40.943  22.8710 0.9565 0.9840 0.9720 1.0000
    Fe    0.375   0.1505 0.9980 0.9769 1.0208 1.0008
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.6355
```
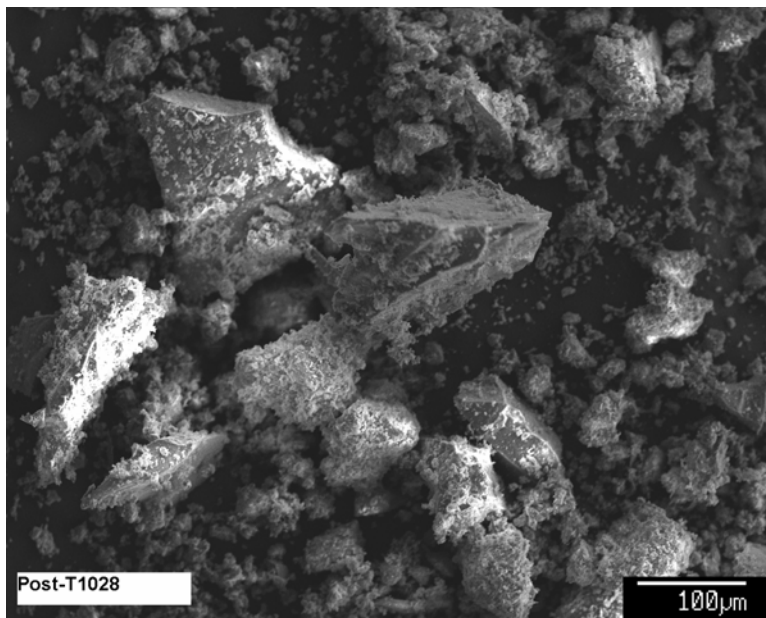
**Figure D-57.** Day-30, post-T1-sample-CD1 SEM-SE image (Post-T1028) magnified 170 times on the smaller particles at different area in the overview image, as shown in Figure D-55.



**Figure D-58.** Day-30, post-T1-sample-CD1 SEM-BSE image (Post-T1029) magnified 170 times on the same field as in image Post-T1028 (see Figure D-57).
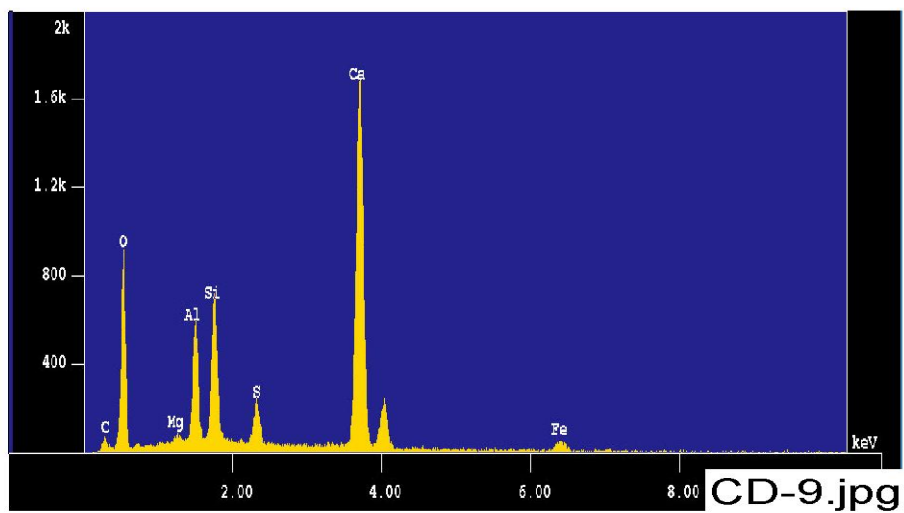
**Figure D-59.** Day-30, post-T1-sample-CD1 counting spectrum (EDS CD-9) on particle in the lower right of image Post-T1029 (see Figure D-58).

The results from the chemical composition analysis for EDS CD-9 are given in Table D-21.

**Table D-21.** **The Chemical Compositions for EDS CD-9**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 9
Comment    : Concrete dust particle
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #   : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.156E-09 A
            Stage Point : X=45.016 Y=75.364 Z=10.565
            Acq. Date   : Fri Jan 28 11:27:56 2005


Element    Mode      ROI(KeV)   K-ratio(%)  +/-    Net/Background
  O K      Normal    0.25- 0.77   17.5957   0.0038    7935 /    44
  Mg K     Normal    1.03- 1.52    0.1183   0.0014     212 /   252
  Al K     Normal    1.26- 1.78    2.2695   0.0014    4851 /   268
  Si K     Normal    1.50- 2.07    2.8894   0.0019    6173 /   292
  S K      Normal    2.04- 2.71    1.9982   0.0015    2444 /    44
  Ca K     Normal    3.40- 4.30   19.4335   0.0215   21960 /    32
  Fe K     Normal    6.04- 7.40    1.5351   0.0612     558 /    11
----------------------------------------------------------------
                       Chi_square =  5.5950

Element Mass%    Atomic%   ZAF      Z      A      F
      O   51.920  71.1181 1.7330 0.9747 1.7780 1.0000
     Mg    0.228   0.2056 1.1328 0.9810 1.1546 1.0001
     Al    4.431   3.5988 1.1467 0.9862 1.1649 0.9981
     Si    5.421   4.2296 1.1018 0.9740 1.1329 0.9985
      S    3.450   2.3580 1.0140 1.1213 0.9087 0.9952
     Ca   31.948  17.4687 0.9655 0.9857 0.9798 0.9997
     Fe    2.602   1.0212 0.9957 0.9790 1.0163 1.0008
----------------------------------------------------------------
Total  100.000 100.0000
Normalization factor =  1.7027
```

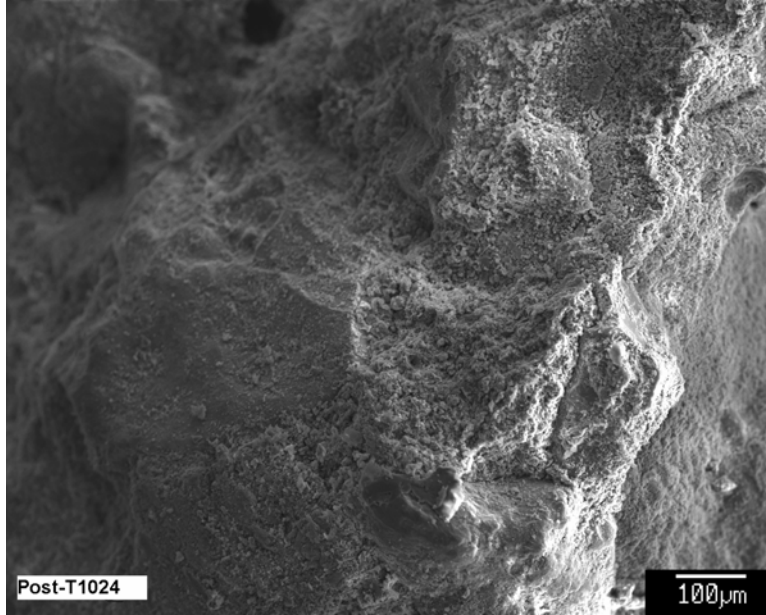**Figure D-60.** Day-30, post-T1-sample-CD1 SEM-SE image (Post-T1030) magnified 400 times on the particle in the center of image Post-T1029 (see Figure D-58).



**Figure D-61.** Day-30, post-T1-sample-LD1 SEM-SE image (Post-T1031) magnified 40 times; overview of the dust sample mounted on carbon tape.
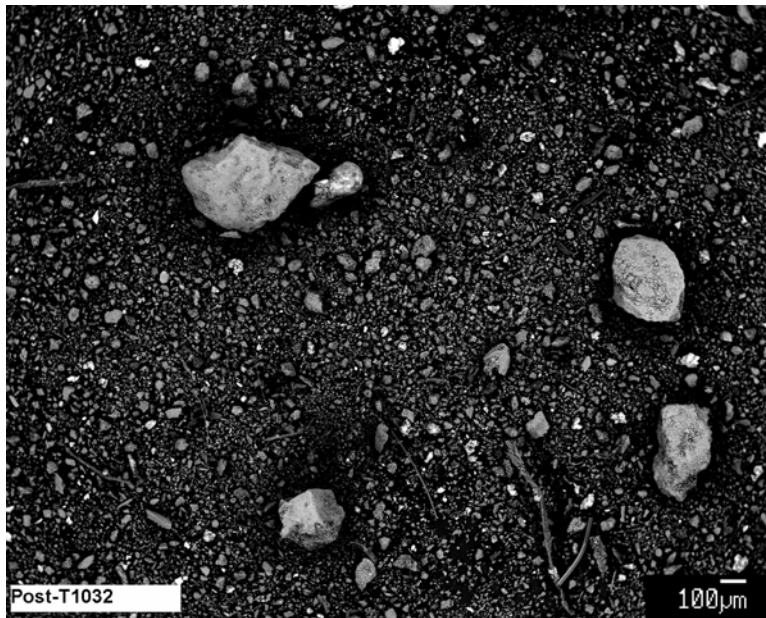
**Figure D-62.** Day-30, post-T1-sample-LD1 SEM-BSE image (Post-T1032) magnified 40 times on the same field as in image Post-T1031 (see Figure D-61); large particles are quartz as in K-feldspar.



**Figure D-63.** Day-30, post-T1-sample-LD1 SEM-BSE image (Post-T1032 annotated) annotating the EDS locations.

**Figure D-64.  Day-30, post-T1-sample-CD1 counting spectrum (EDS LD-10) on large particle at right of k-feldspar (see Figure D-63).**

The results from the chemical composition analysis for EDS LD-10 are given in Table D-22.

**Table D-22.   The Chemical Compositions for EDS LD-10**

```
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Group     : NRC
Sample    : PostT1_1-28-05  ID# : 10
Comment   : K-feldspar in latent dust sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #     : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.165E-09 A
            Stage Point : X=46.733 Y=67.407 Z=10.565
            Acq. Date   : Fri Jan 28 11:45:29 2005


Element    Mode      ROI(KeV)    K-ratio(%)  +/-     Net/Background
  O K      Normal   0.25- 0.77    27.0588   0.0047    12297 /      68
 Na K      Normal   0.83- 1.28     0.8837   0.0077     1263 /      82
 Mg K      Normal   1.03- 1.52     0.2752   0.0016      497 /     346
 Al K      Normal   1.26- 1.78     3.0567   0.0016     6585 /     720
 Si K      Normal   1.50- 2.07    10.0455   0.0032    21627 /     420
  K K      Normal   3.01- 3.86     4.5094   0.0034     3221 /      41
 Fe K      Normal   6.04- 7.40     1.9483   0.0596      714 /      13
 Ca K      Normal   3.40- 4.30     0.5385   0.0061      613 /      46
---------------------------------------------------------------------
                        Chi_square =  8.3513

Element Mass%    Atomic%   ZAF      Z      A      F
   O    55.460  70.6207 1.0754 0.9850 1.0918 1.0000
   Na    1.971   1.7468 1.1705 0.9892 1.1826 1.0006
   Mg    0.569   0.4770 1.0852 0.9917 1.0960 0.9984
   Al    6.501   4.9088 1.1159 0.9971 1.1256 0.9943
   Si   21.253  15.4161 1.1101 0.9849 1.1275 0.9996
   K     9.494   4.9464 1.1047 1.2332 0.8971 0.9985
   Fe    3.702   1.3506 0.9971 0.9923 1.0040 1.0008
   Ca    1.050   0.5335 1.0226 0.9975 1.0256 0.9996
---------------------------------------------------------------------
Total    100.000 100.0000
Normalization factor =  1.9059
```

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**Figure D-65.  Day-30, post-T1-sample-LD1 counting spectrum (EDS LD-11) on the small bright particle in the upper center of image Post-T1032, as shown in Figure D-63.**

The results from the chemical composition analysis for EDS LD-11 are given in Table D-23.

**Table D-23.    The Chemical Compositions for EDS LD-11**

```
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Group       : NRC
Sample      : PostT1 1-28-05  ID# : 11
Comment     : ZnCl      . latent dust sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.167E-09 A
            Stage Point : X=46.743 Y=66.378 Z=10.565
            Acq. Date   : Fri Jan 28 11:50:31 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
  O K       Normal   0.25- 0.77  19.9450   0.0045     9080 /    114
  Cl K      Normal   2.34- 3.06   7.9463   0.0025     9584 /     78
  Zn K      Normal   8.22-10.03  45.6297   0.0180     5999 /     13
--------------------------------------------------------------------
                         Chi_square =  6.8473

Element Mass%    Atomic%   ZAF      Z      A      F
    O    24.790  54.6092 0.9731 0.8709 1.1175 0.9999
    Cl   10.643  10.5798 1.0486 0.9156 1.1455 0.9999
    Zn   64.567  34.8110 1.1079 1.1086 0.9993 1.0000
--------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.2772
```
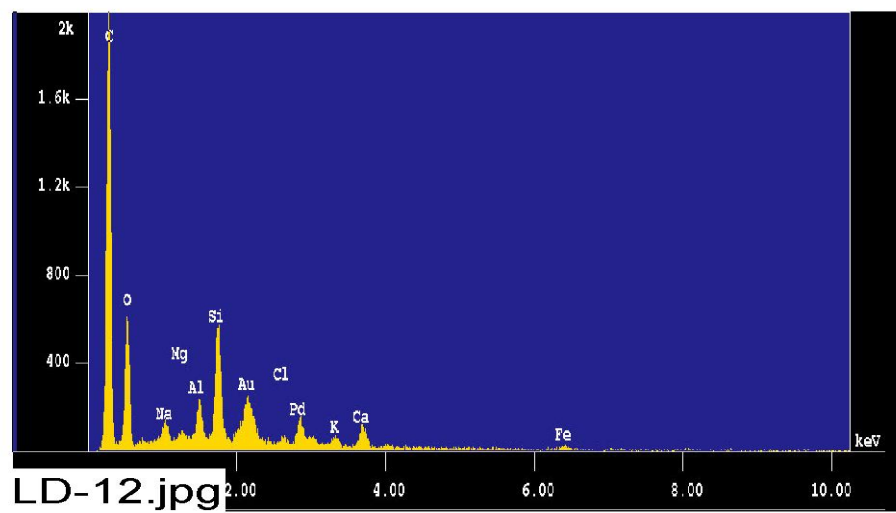
**Figure D-66.**   **Day-30, post-T1-sample-LD1 counting spectrum (EDS LD-12) on the thin fiber in the lower center of image Post-T1032, as shown in Figure D-63.**

The results from the chemical composition analysis for EDS LD-12 are given in Table D-24.

**Table D-24.    The Chemical Compositions for EDS LD-12**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 12
Comment    : Fiber in latent dust sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.169E-09 A
            Stage Point : X=46.614 Y=68.050 Z=10.565
            Acq. Date   : Fri Jan 28 11:56:29 2005


Element     Mode      ROI(KeV)   K-ratio(%)  +/-     Net/Background
  C K       Normal   0.09- 0.46   22.4454    0.0014    15184 /      57
  O K       Normal   0.25- 0.77   13.1602    0.0034     6001 /     839
 Na K       Normal   0.83- 1.28    0.4650    0.0066      667 /      65
 Mg K       Normal   1.03- 1.52    0.1354    0.0014      245 /     158
 Al K       Normal   1.26- 1.78    0.7357    0.0010     1590 /     237
 Si K       Normal   1.50- 2.07    2.3036    0.0017     4976 /     188
 Cl K       Normal   2.34- 3.06    0.5360    0.0009      648 /      62
  K K       Normal   3.01- 3.86    0.8042    0.0021      576 /      39
 Ca K       Normal   3.40- 4.30    1.1517    0.0068     1316 /      26
--------------------------------------------------------------------
                        Chi_square = 64.9276

Element Mass%    Atomic%   ZAF      Z      A      F
    C   57.981  66.9895 1.8889 1.0178 1.8559 1.0000
    O   33.753  29.2766 1.8754 0.9713 1.9309 1.0000
   Na    0.635   0.3834 0.9988 0.9766 1.0213 1.0014
   Mg    0.176   0.1003 0.9496 0.9797 0.9685 1.0008
   Al    1.016   0.5225 1.0098 0.9855 1.0259 0.9987
   Si    3.083   1.5232 0.9786 0.9740 1.0050 0.9997
   Cl    0.721   0.2824 0.9842 1.0327 0.9550 0.9979
    K    1.138   0.4037 1.0344 1.2240 0.8488 0.9956
   Ca    1.497   0.5183 0.9505 0.9910 0.9591 1.0001
--------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.3676
```

Results from an alternative chemical composition analysis for EDS LD-12 using a different set of elements from those selected in the previous table are given in Table D-25. The high mass fraction of carbon is from the tape used for mounting the latent dust.

**Table D-25.      The Chemical Compositions for Alternative Analysis of EDS LD-12**

```
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  Group      : NRC
  Sample     : PostT1_1-28-05  ID# : 12
  Comment    : Fiber in latent dust sample
  Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec     Aperture #    : 1
             Acc. Volt   : 15.0 KV          Probe Current : 1.169E-09 A
             Stage Point : X=46.614 Y=68.050 Z=10.565
             Acq. Date   : Fri Jan 28 11:56:29 2005


  Element     Mode     ROI(KeV)    K-ratio(%)  +/-     Net/Background
    C K       Normal   0.09- 0.46   22.4454    0.0014    15184 /      57
    O K       Normal   0.25- 0.77   13.1602    0.0034     6001 /     839
   Na K       Normal   0.83- 1.28    0.4650    0.0066      667 /      65
   Mg K       Normal   1.03- 1.52    0.1354    0.0014      245 /     158
   Al K       Normal   1.26- 1.78    0.7357    0.0010     1590 /     237
   Si K       Normal   1.50- 2.07    2.3036    0.0017     4976 /     188
   Cl K       Normal   2.34- 3.06    0.5360    0.0009      648 /      62
    K K       Normal   3.01- 3.86    0.8042    0.0021      576 /      39
   Ca K       Normal   3.40- 4.30    1.1517    0.0068     1316 /      26
  -----------------------------------------------------------------
                          Chi_square = 64.9276

  Element Mass%    Atomic%   ZAF      Z      A      F
        C   57.981  66.9895 1.8889 1.0178 1.8559 1.0000
        O   33.753  29.2766 1.8754 0.9713 1.9309 1.0000
       Na    0.635   0.3834 0.9988 0.9766 1.0213 1.0014
       Mg    0.176   0.1003 0.9496 0.9797 0.9685 1.0008
       Al    1.016   0.5225 1.0098 0.9855 1.0259 0.9987
       Si    3.083   1.5232 0.9786 0.9740 1.0050 0.9997
       Cl    0.721   0.2824 0.9842 1.0327 0.9550 0.9979
        K    1.138   0.4037 1.0344 1.2240 0.8488 0.9956
       Ca    1.497   0.5183 0.9505 0.9910 0.9591 1.0001
  -----------------------------------------------------------------
  Total   100.000 100.0000
  Normalization factor =  1.3676
```

**Figure D-67.** Day-30, post-T1-sample-LD1 SEM-SE image (Post-T1033) magnified 400 times on the central portion of image Post-T1031, as shown in Figure D-61.
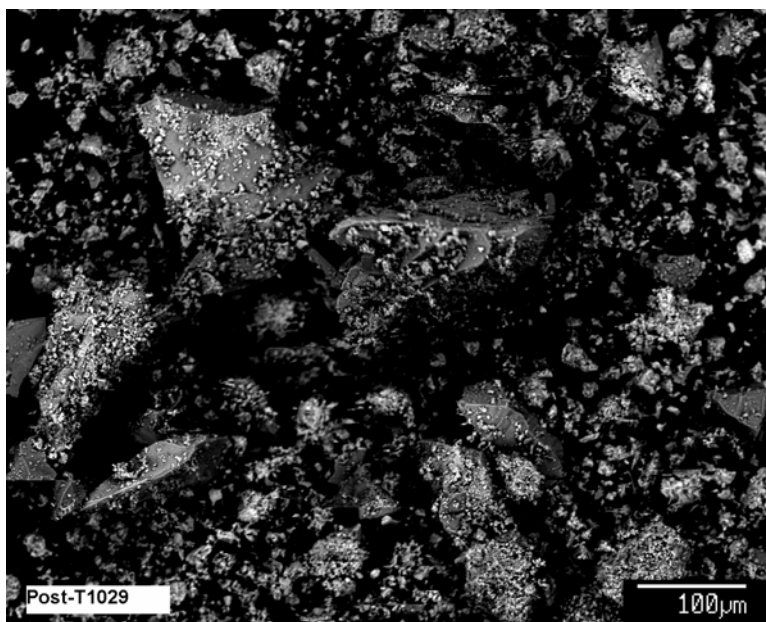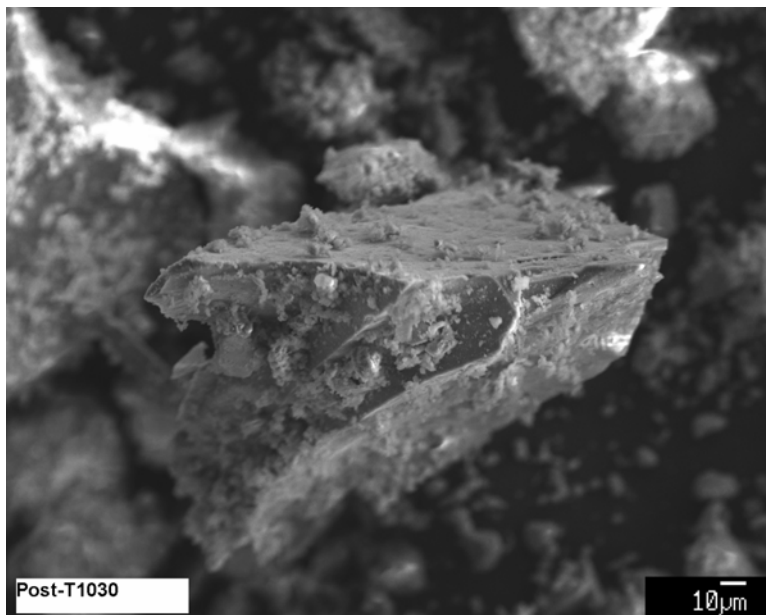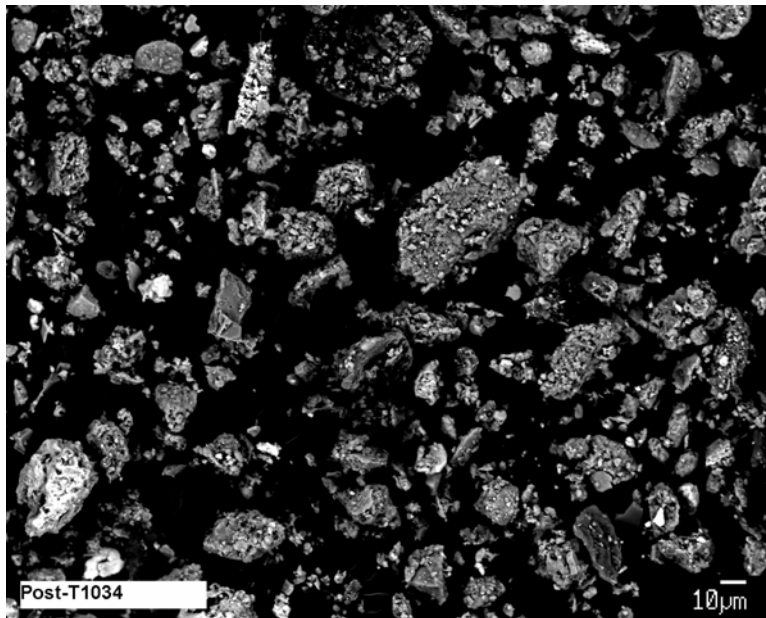


**Figure D-68.** Day-30, post-T1-sample-LD1 SEM-BSE image (Post-T1034) magnified 400 times on the same field as in image Post-T1033 (see Figure D-67).
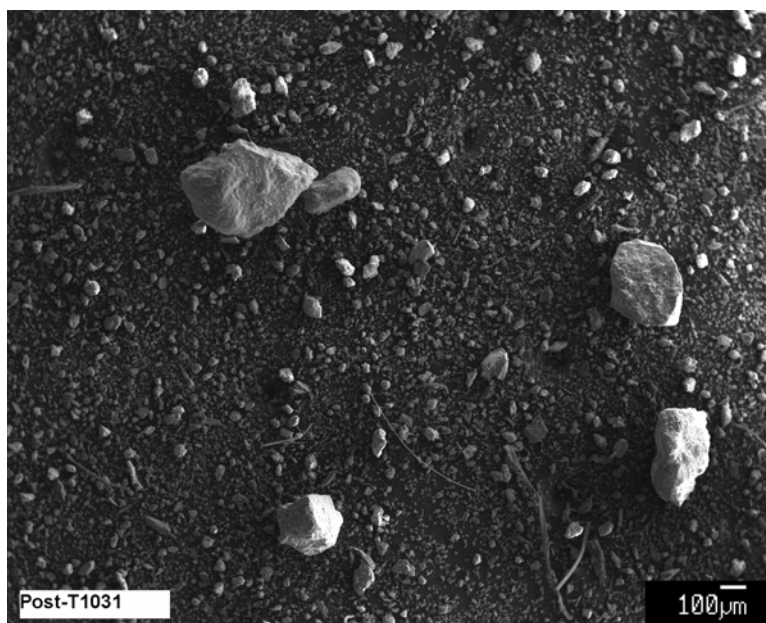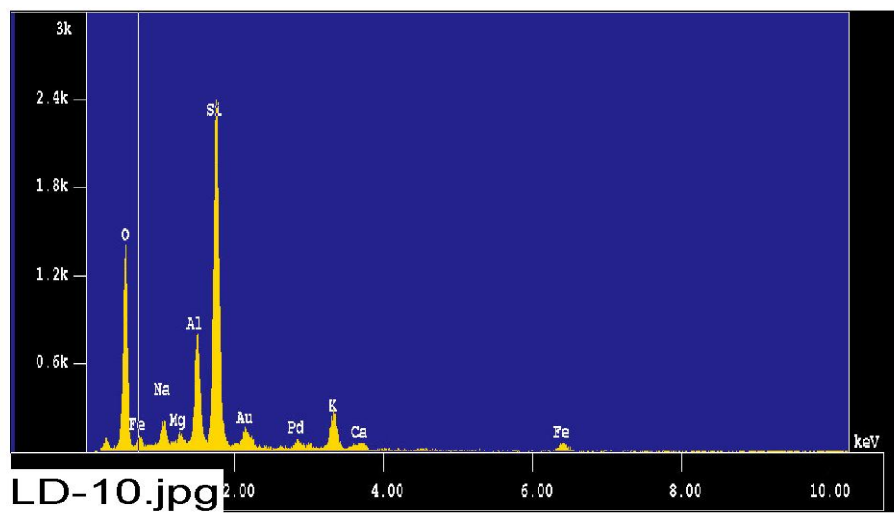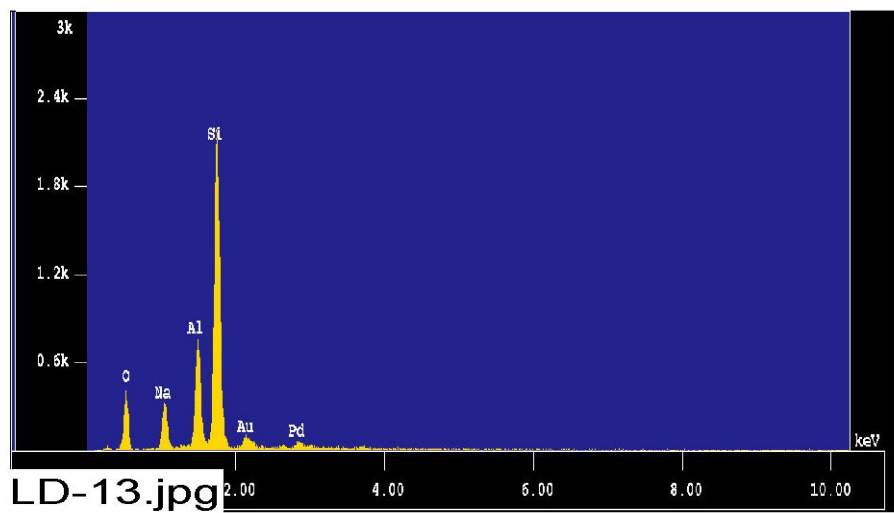
**Figure D-69.** Day-30, post-T1-sample-LD1 counting spectrum (EDS LD-13) on the flake at the left center of Na-feldspar in image Post-T1034 (see Figure D-68).

The results from the chemical composition analysis for EDS LD-13 are given in Table D-26.

**Table D-26.    The Chemical Compositions for EDS LD-13**

```
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Group      : NRC
Sample     : PostTl_1-28-05  ID# : 13
Comment    : Na-feldspar in latent dust sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #     : 1
             Acc. Volt   : 15.0 KV         Probe Current : 1.169E-09 A
             Stage Point : X=46.619 Y=67.410 Z=10.565
             Acq. Date   : Fri Jan 28 12:07:32 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-     Net/Background
  O K       Normal   0.25- 0.77   7.8674   0.0026     3588 /      18
 Na K       Normal   0.83- 1.28   1.7705   0.0081     2539 /      24
 Al K       Normal   1.26- 1.78   2.9692   0.0015     6418 /     686
 Si K       Normal   1.50- 2.07   9.1928   0.0030    19859 /     367
 Ca K       Normal   3.40- 4.30   0.1851   0.0046      212 /      16
-----------------------------------------------------------------
                       Chi_square =  5.7053

Element Mass%    Atomic%   ZAF      Z       A       F
    O   36.363  49.3783 1.1117 0.9948 1.1176 1.0000
    Na   6.851   6.4742 0.9308 0.9988 0.9324 0.9994
    Al  12.829  10.3298 1.0393 1.0067 1.0446 0.9883
    Si  43.160  33.3861 1.1293 0.9943 1.1359 1.0000
    Ca   0.796   0.4317 1.0347 1.0065 1.0279 1.0001
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  4.1574
```
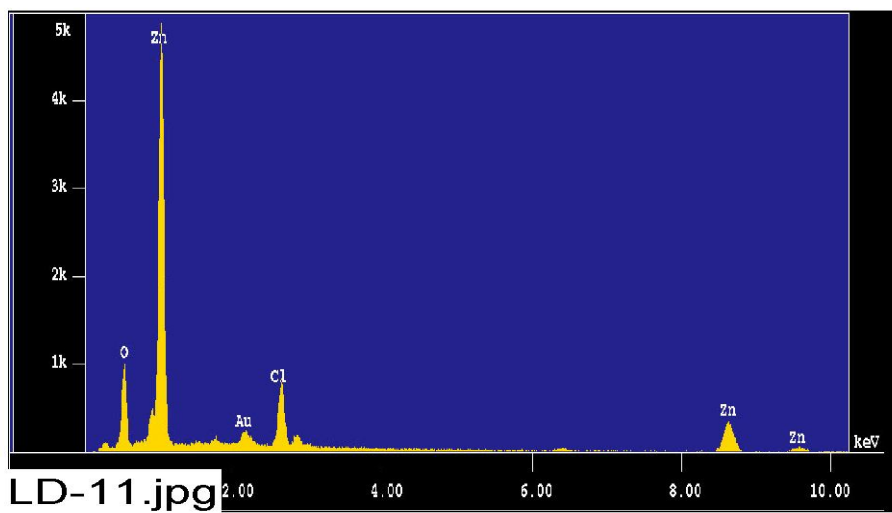
**Figure D-70.** **Day-30, post-T1-sample-LD1 counting spectrum (EDS LD-14) on the large particle at the upper center of image Post-T1034 (see Figure D-68).**

The results from the chemical composition analysis for EDS LD-14 are given in Table D-27.

**Table D-27.    The Chemical Compositions for EDS LD-14**

```
Jan 28 12:12 2005  /tmp/eds_pout.log Page 1



Group      : NRC
Sample     : PostT1_1-28-05  ID# : 14
Comment    : Particle in latent dust sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec   Aperture #    : 1
            Acc. Volt   : 15.0 KV       Probe Current : 1.170E-09 A
            Stage Point : X=46.619 Y=67.410 Z=10.565
            Acq. Date   : Fri Jan 28 12:10:44 2005


Element     Mode    ROI(KeV)   K-ratio(%)  +/-    Net/Background
  O K      Normal  0.25- 0.77   16.6818   0.0037    7614 /      37
 Mg K      Normal  1.03- 1.52    0.4320   0.0016     783 /     377
 Al K      Normal  1.26- 1.78    4.1712   0.0018    9024 /     864
 Si K      Normal  1.50- 2.07   11.7041   0.0035   25306 /     544
  K K      Normal  3.01- 3.86    4.1207   0.0033    2956 /      37
 Ca K      Normal  3.40- 4.30    0.2675   0.0057     306 /      42
 Fe K      Normal  6.04- 7.40    1.0424   0.0520     383 /      16
-----------------------------------------------------------------
                     Chi_square =  6.5270

Element Mass%    Atomic%   ZAF     Z      A      F
   O    45.973  61.6192 1.2142 0.9856 1.2319 1.0000
   Mg    0.985   0.8687 1.0044 0.9922 1.0156 0.9968
   Al   10.049   7.9867 1.0615 0.9975 1.0729 0.9919
   Si   29.495  22.5196 1.1103 0.9852 1.1274 0.9996
   K    10.506   5.7615 1.1233 1.2330 0.9117 0.9992
   Ca    0.630   0.3372 1.0377 0.9973 1.0407 0.9998
   Fe    2.363   0.9072 0.9986 0.9914 1.0065 1.0008
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  2.2697
```
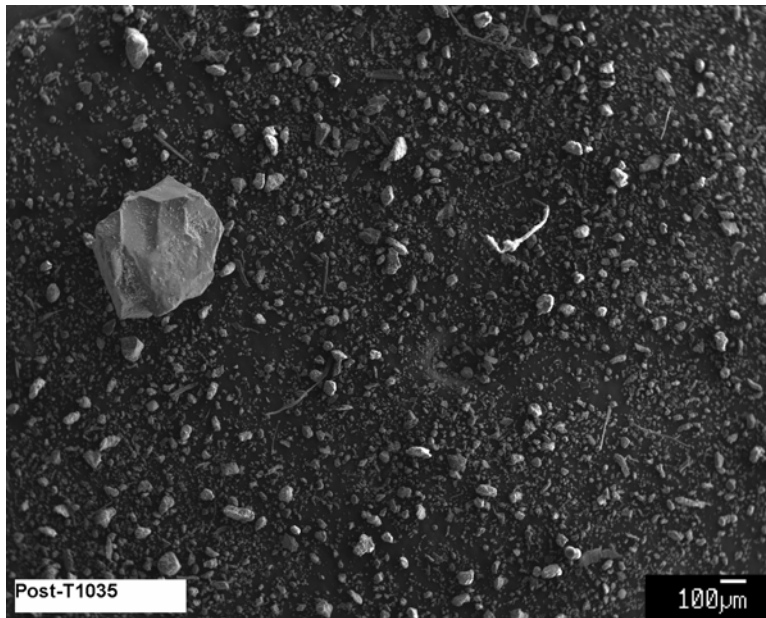
**Figure D-71.** Day-30, post-T1-sample-LD1 SEM-SE image (Post-T1035) magnified 40 times; overview of another area of the dust sample mounted on carbon tape.
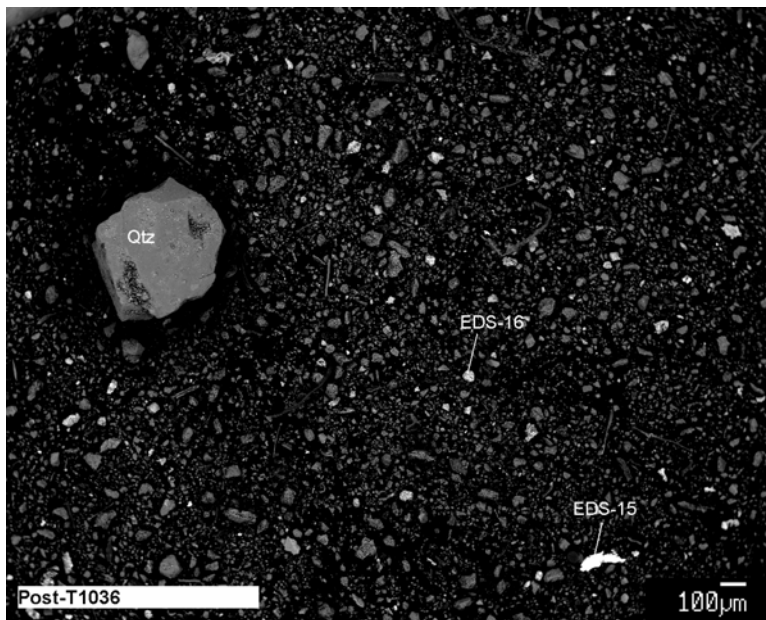


**Figure D-72.** Day-30, post-T1-sample-LD1 SEM-BSE image (Post-T1036) magnified 40 times on the same field as in image Post-T1035 (see Figure D-71).
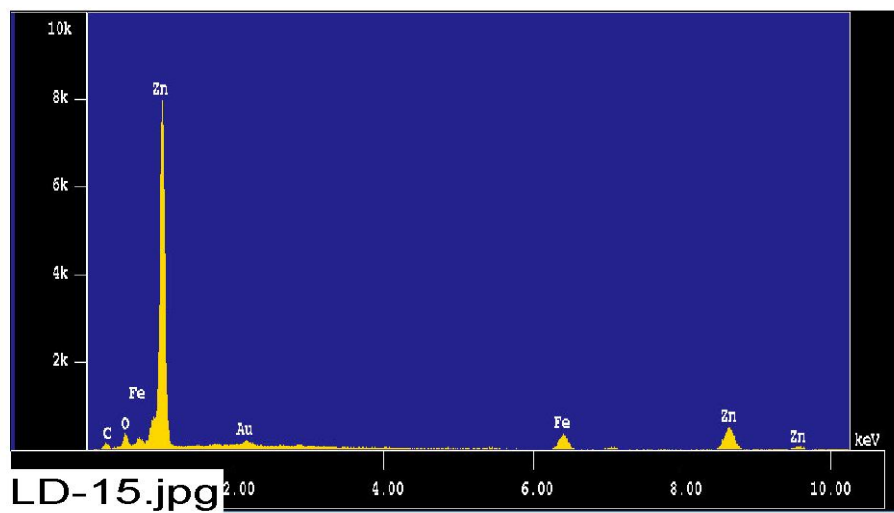
**Figure D-73.** **Day-30, post-T1-sample-LD1 counting spectrum (EDS LD-15) on the bright particle in the lower right of image Post-T1036 (see Figure D-72).**

The results from the chemical composition analysis for EDS LD-15 are given in Table D-28.

**Table D-28.     The Chemical Compositions for EDS LD-15**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 15
Comment    : Brite particle in latent dust sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #     : 1
             Acc. Volt   : 15.0 KV         Probe Current : 1.169E-09 A
             Stage Point : X=50.691 Y=62.647 Z=10.565
             Acq. Date   : Fri Jan 28 12:26:15 2005


Element     Mode      ROI(KeV)   K-ratio(%)  +/-    Net/Background
   O K      Normal   0.25- 0.77    7.5209    0.0034    3430 /     186
  Si K      Normal   1.50- 2.07    0.1738    0.0012     375 /     160
  Cl K      Normal   2.34- 3.06    0.4364    0.0014     527 /     119
  Fe K      Normal   6.04- 7.40   10.6827    0.1340    3926 /      34
  Zn K      Normal   8.22-10.03   70.4669    0.0221    9280 /      26
------------------------------------------------------------------
                        Chi_square =  5.9022

Element Mass%    Atomic%   ZAF      Z      A      F
    O    7.386  24.1083 0.8675 0.8226 1.0547 0.9998
    Si   0.293   0.5444 1.4880 0.8183 1.8185 1.0000
    Cl   0.513   0.7559 1.0388 0.8625 1.2045 0.9998
    Fe   9.186   8.5894 0.7596 0.8043 1.0084 0.9365
    Zn  82.622  66.0020 1.0357 1.0327 1.0029 1.0000
------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.1321
```

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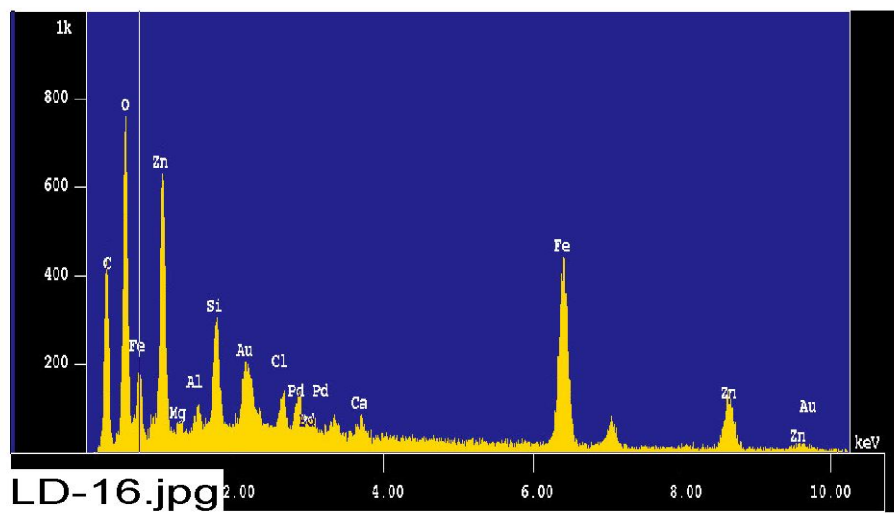
**Figure D-74.**  **Day-30, post-T1-sample-LD1 counting spectrum (EDS LD-16) on the round particle at slightly right of the center of image Post-T1036 (see Figure D-72).**

The results from the chemical composition analysis for EDS LD-16 are given in Table D-29.

**Table D-29.    The Chemical Compositions for EDS LD-16**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 16
Comment    : Brite particle in latent dust sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.168E-09 A
            Stage Point : X=50.484 Y=62.853 Z=10.565
            Acq. Date   : Fri Jan 28 12:31:06 2005


Element    Mode      ROI(KeV)   K-ratio(%)  +/-    Net/Background
  O K      Normal    0.25- 0.77   17.1582  0.0039     7818 /     231
 Mg K      Normal    1.03- 1.52    0.1158  0.0012      210 /     354
 Al K      Normal    1.26- 1.78    0.2078  0.0007      449 /     142
 Si K      Normal    1.50- 2.07    1.0789  0.0014     2329 /     111
 Cl K      Normal    2.34- 3.06    1.0453  0.0013     1262 /      72
 Ca K      Normal    3.40- 4.30    0.4189  0.0066      478 /      40
 Fe K      Normal    6.04- 7.40   12.0769  0.1311     4434 /      17
 Zn K      Normal    8.22-10.03   16.3616  0.0116     2153 /      10
-----------------------------------------------------------------
                      Chi_square =   8.8330

Element Mass%    Atomic%   ZAF      Z      A      F
   O    30.426   60.3020 0.8300 0.8879 0.9350 0.9998
   Mg    0.466    0.6074 1.8827 0.8923 2.1073 1.0013
   Al    0.734    0.8626 1.6531 0.8966 1.8445 0.9995
   Si    3.145    3.5505 1.3645 0.8851 1.5419 0.9998
   Cl    2.349    2.1005 1.0516 0.9344 1.1261 0.9995
   Ca    0.815    0.6445 0.9103 0.8918 1.0245 0.9964
   Fe   22.085   12.5395 0.8560 0.8786 1.0040 0.9704
   Zn   39.980   19.3929 1.1438 1.1362 1.0066 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =   2.1364
```
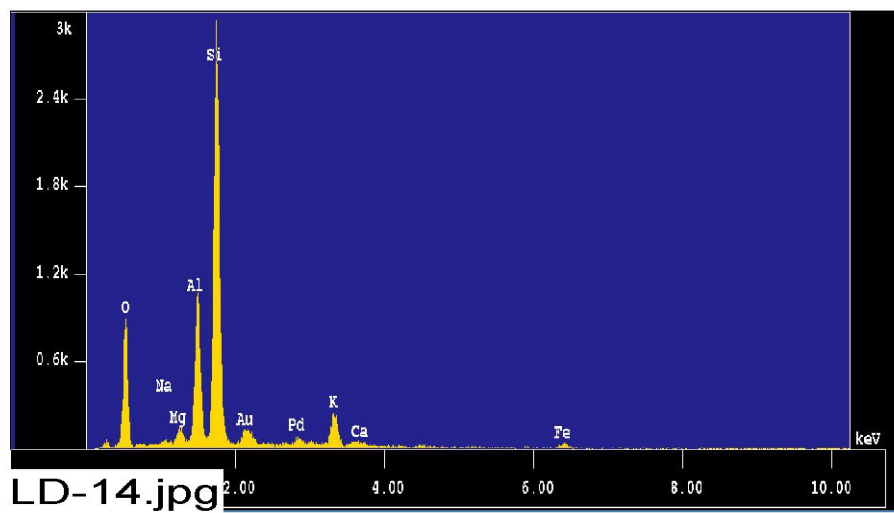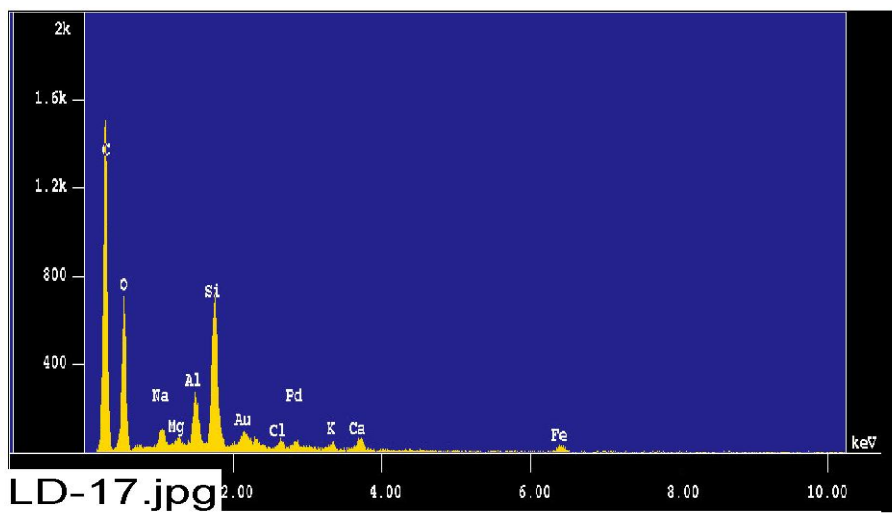
**Figure D-75.** **Day-30, Post-T1-sample-LD1 counting spectrum (EDS LD-17) over an area of 425 × 425 µm, magnified 250 times. (C and Cl peaks are from the mounting tape).**

The results from the chemical composition analysis from EDS LD-17 are given in Table D-30.

**Table D-30.    The Chemical Compositions for EDS LD-17**

```
Jan 28 12:38 2005  /tmp/eds_pout.log Page 1



Group     : NRC
Sample    : PostT1_1-28-05  ID# : 17
Comment   : Area of latent dust sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #   : 1
            Acc. Volt   : 15.0 KV        Probe Current : 1.168E-09 A
            Stage Point : X=50.719 Y=62.405 Z=10.565
            Acq. Date   : Fri Jan 28 12:37:20 2005


Element    Mode      ROI(KeV)   K-ratio(%)  +/-     Net/Background
  O K      Normal    0.25- 0.77   14.6419   0.0034    6671 /     648
 Mg K      Normal    1.03- 1.52    0.1506   0.0012     273 /     146
 Al K      Normal    1.26- 1.78    0.9264   0.0010    2001 /     241
 Si K      Normal    1.50- 2.07    2.9558   0.0018    6380 /     135
 Ca K      Normal    3.40- 4.30    0.6744   0.0056     770 /      21
 Fe K      Normal    6.04- 7.40    1.0013   0.0459     368 /       4
  K K      Normal    3.01- 3.86    0.6405   0.0017     459 /      23
-------------------------------------------------------------------
                        Chi_square = 42.6433

Element Mass%    Atomic%   ZAF      Z      A      F
    O   65.858  79.3709 0.9080 0.9874 0.9196 1.0000
   Mg    0.831   0.6587 1.1135 0.9944 1.1208 0.9991
   Al    5.237   3.7421 1.1410 0.9998 1.1462 0.9956
   Si   16.317  11.2023 1.1144 0.9876 1.1286 0.9998
   Ca    3.338   1.6060 0.9992 1.0009 0.9991 0.9992
   Fe    4.950   1.7090 0.9979 0.9965 1.0007 1.0008
    K    3.470   1.7110 1.0936 1.2372 0.8885 0.9949
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  4.9538
```
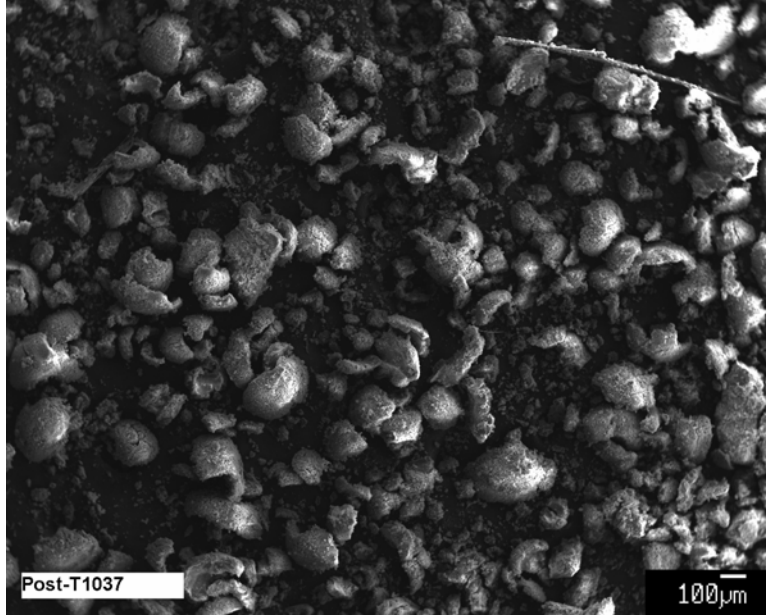
**Figure D-76.**   Day-30, post-T1-sample-Sed1 SEM-SE image (Post-T1037) magnified 40 times; overview of particles.



**Figure D-77.**   Day-30, post-T1-sample-Sed1 SEM-BSE image (Post-T1038) magnified 40 times on the same particles, as shown in Figure D-76.

**Figure D-78.** Day-30, post-T1-sample-Sed1 SEM-SE image (Post-T1039) magnified 120 times; close-up of particles.



**Figure D-79.** Day-30, Post-T1-sample-Sed1 SEM-BSE image (Post-T1040) on the same field, as shown in Figure D-78.
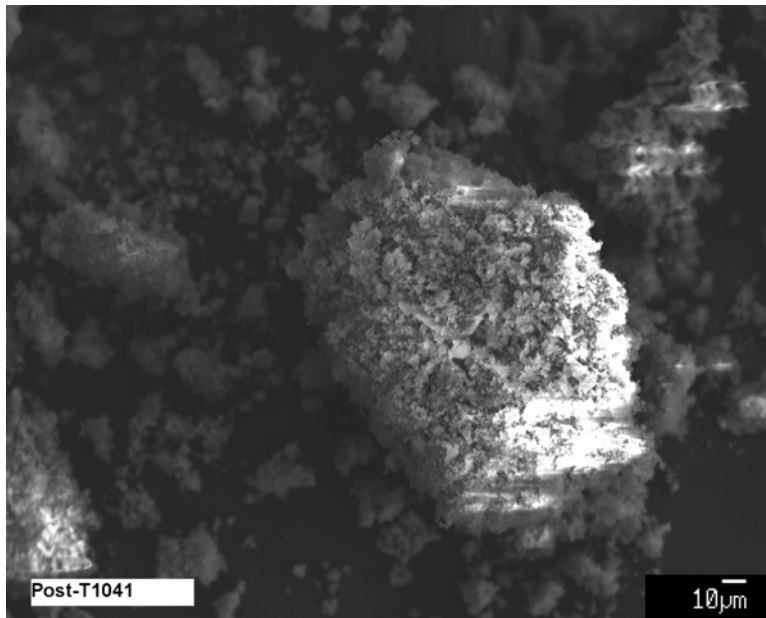
**Figure D-80.** Day-30, post-T1-sample-Sed1 SEM-SE image (Post-T1041) magnified 370 times on a single particle in image Post-T1-1040, as shown in Figure D-79.
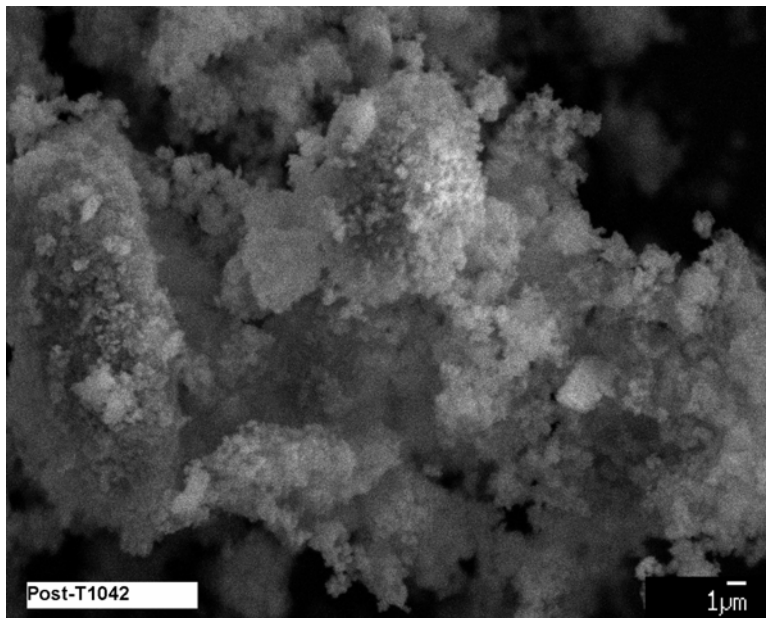


**Figure D-81.** Day-30, post-T1-sample-Sed1 SEM-SE image (Post-T1042) magnified 3000 times on the small particles in image Post-T1040, as shown in Figure D-79.
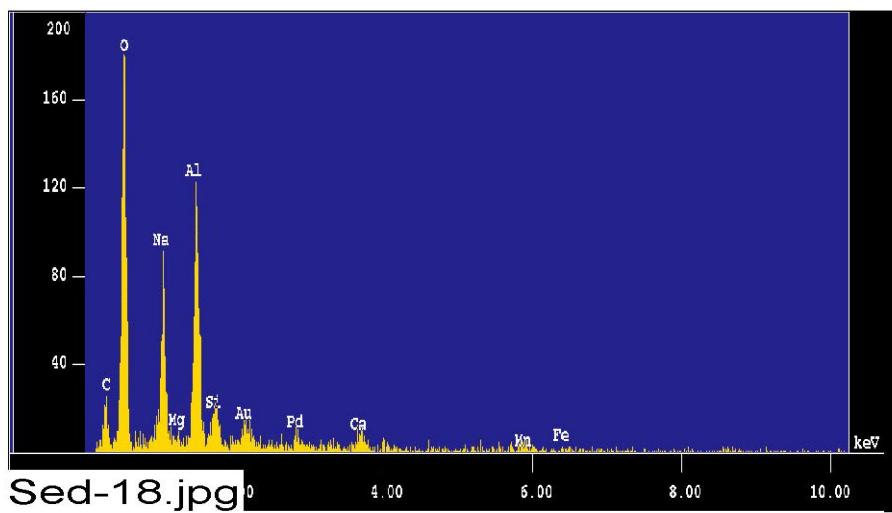
**Figure D-82.** Day-30, post-T1-sample-Sed1 counting spectrum (EDS SED-18) over the particles in an area (18 × 18 μm) in image Post-T1042 (see Figure D-81), magnified 6000 times.

The results from the chemical composition analysis for EDS SED-18 are given in Table D-31.

**Table D-31.     The Chemical Compositions for EDS SED-18**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 18
Comment    : Pipe sediment sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
              Live Time   : 100.000 sec    Aperture #     : 1
              Acc. Volt   : 15.0 KV         Probe Current : 2.991E-10 A
              Stage Point : X=37.268 Y=67.741 Z=10.565
              Acq. Date   : Fri Jan 28 13:51:02 2005


Element      Mode     ROI(KeV)    K-ratio(%)  +/-     Net/Background
   C K      Normal   0.09- 0.46    0.0000    0.0000        0 /      20
   O K      Normal   0.25- 0.77   14.2631    0.0018     1664 /      16
  Na K      Normal   0.83- 1.28    1.5969    0.0039      586 /       2
  Al K      Normal   1.26- 1.78    1.6726    0.0005      925 /      10
  Si K      Normal   1.50- 2.07    0.2688    0.0004      149 /      52
  Ca K      Normal   3.40- 4.30    0.3276    0.0021       96 /       3
  Mn K      Normal   5.53- 6.82    0.3310    0.0003       35 /       0
  Fe K      Normal   6.04- 7.40    0.0159    0.0120        1 /       1
-----------------------------------------------------------------
                      Chi_square =  1.8808

Element Mass%    Atomic%   ZAF      Z      A      F
   C      0.000    0.0000 3.9321 1.0376 3.7901 0.9999
   O     67.010   77.4509 0.7082 0.9896 0.7157 0.9999
  Na     12.316    9.9063 1.1626 0.9942 1.1684 1.0008
  Al     13.480    9.2382 1.2149 1.0024 1.2126 0.9995
  Si      2.226    1.4655 1.2481 0.9902 1.2604 0.9999
  Ca      2.141    0.9880 0.9852 1.0042 0.9820 0.9991
  Mn      2.722    0.9162 1.2395 1.2444 0.9961 1.0000
  Fe      0.106    0.0349 0.9982 1.0007 0.9967 1.0008
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  6.6339
```
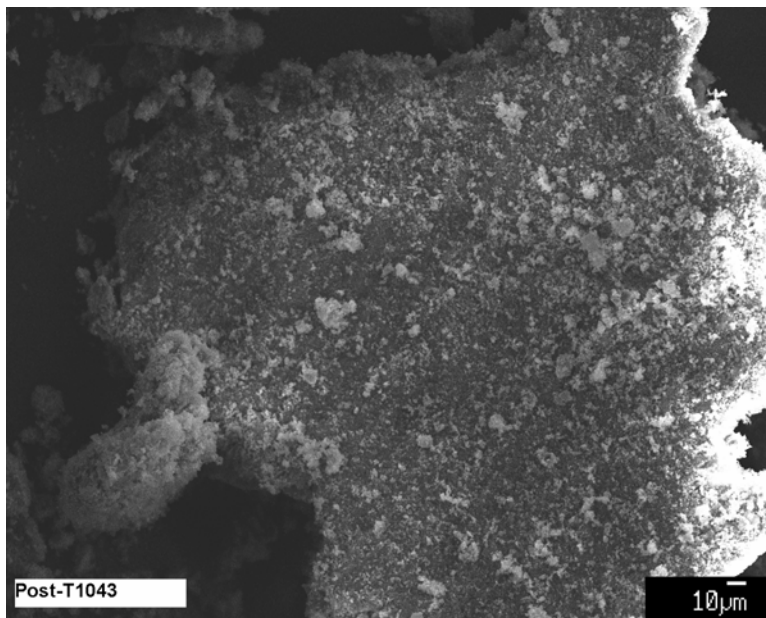
**Figure D-83.** Day-30, post-T1-sample-Sed1 SEM-SE image (Post-T1043) magnified 300 times on another particle in image Post-T1040, as shown in Figure D-79.
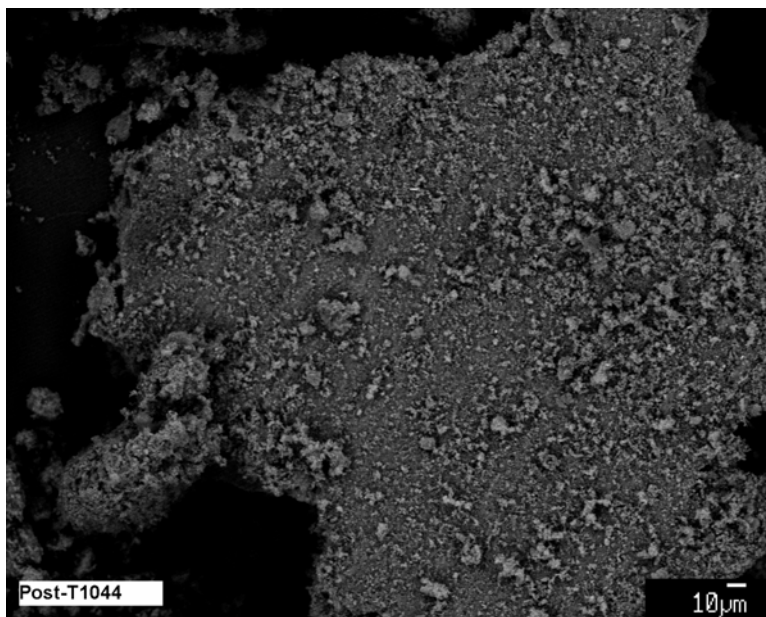


**Figure D-84.** Day-30, post-T1-sample-Sed1 SEM-BSE image (Post-T1044) magnified 300 times on the same particle in image Post-T1043, as shown in Figure D-83.
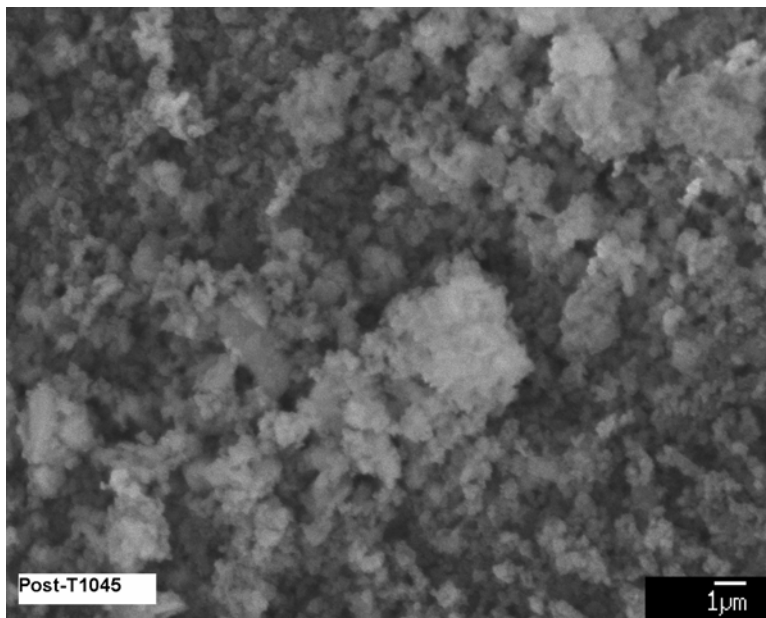
**Figure D-85.** Day-30, post-T1-sample-Sed1 SEM-SE image (Post-T1045) magnified 5000 times on the center particle in image Post-T1043, as shown in Figure D-83.



**Figure D-86.** Day-30, post-T1-sample-Sed1 SEM-SE image (Post-T1046) magnified 10,000 times on the same particle in image Post-T1043, as shown in Figure D-83.

**Figure D-87.** Day-30, post-T1-sample-Sed1 SEM-SE image (Post-T1047) magnified 40 times; overview of the particles in images Post-T1043, -T1044, -T1045, and -T1046, as shown in Figure D-83 to Figure D-86.
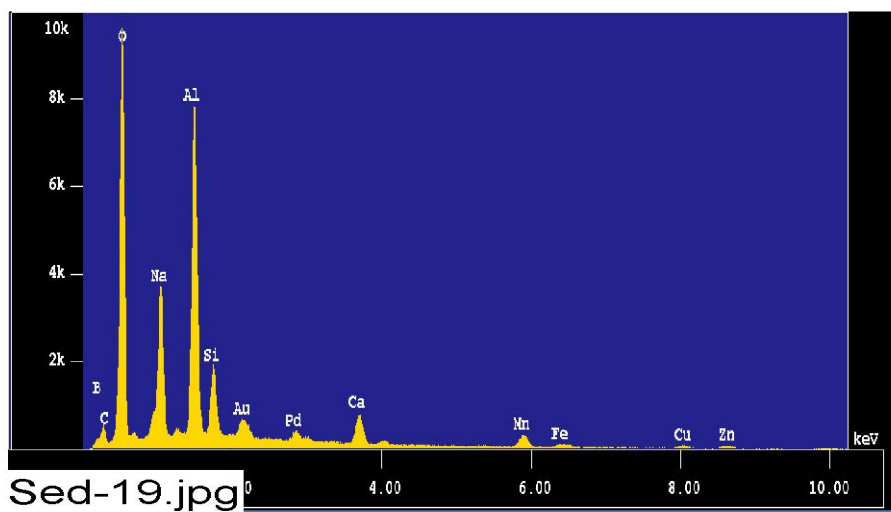


**Figure D-88.** Day-30, post-T1-sample-Sed1 counting spectrum (EDS SED-19) for the same field as in image Post-T1046 (see Figure D-86), magnified 10,000 times.

The results from the chemical composition analysis for EDS SED-19 are given in Table D-32.

**Table D-32.     The Chemical Compositions for EDS SED-19**

```
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Group     : NRC
Sample    : PostT1_1-28-05  ID# : 19
Comment   : Pipe sediment sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 5.884E-09 A
            Stage Point : X=36.914 Y=68.416 Z=10.565
            Acq. Date   : Fri Jan 28 14:08:16 2005


Element     Mode      ROI(KeV)     K-ratio(%)   +/-      Net/Background
  O K       Normal    0.25- 0.77    40.2864    0.0134     92468 /     381
 Na K       Normal    0.83- 1.28     3.1848    0.0412     22988 /     376
 Mg K       Normal    1.03- 1.52     0.1428    0.0033      1302 /    4531
 Al K       Normal    1.26- 1.78     6.1469    0.0046     66877 /     856
 Si K       Normal    1.50- 2.07     1.3594    0.0034     14782 /    3944
 Ca K       Normal    3.40- 4.30     1.5660    0.0181      9007 /     154
 Mn K       Normal    5.53- 6.82     2.2207    0.0029      4681 /      76
 Fe K       Normal    6.04- 7.40     0.3664    0.1051       678 /      58
 Cu K       Normal    7.63- 9.27     1.2864    0.0090      1124 /      46
 Zn K       Normal    8.22-10.03     1.5860    0.0125      1051 /      31
------------------------------------------------------------------------
                         Chi_square = 31.9346

.lement Mass%    Atomic%   ZAF      Z      A      F
    O    58.714  74.0315 0.7799 0.9730 0.8016 0.9999
    Na    7.760   6.8088 1.3038 0.9771 1.3332 1.0008
    Mg    0.346   0.2873 1.2969 0.9796 1.3248 0.9993
    Al   14.542  10.8723 1.2659 0.9849 1.2861 0.9994
    Si    3.264   2.3446 1.2849 0.9729 1.3209 0.9999
    Ca    2.853   1.4357 0.9747 0.9853 0.9910 0.9982
    Mn    5.018   1.8425 1.2091 1.2190 0.9990 0.9929
    Fe    0.662   0.2392 0.9674 0.9799 0.9989 0.9883
    Cu    3.053   0.9691 1.2698 1.2712 0.9989 1.0000
    Zn    3.788   1.1689 1.2781 1.2801 0.9984 1.0000
------------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.8688
```

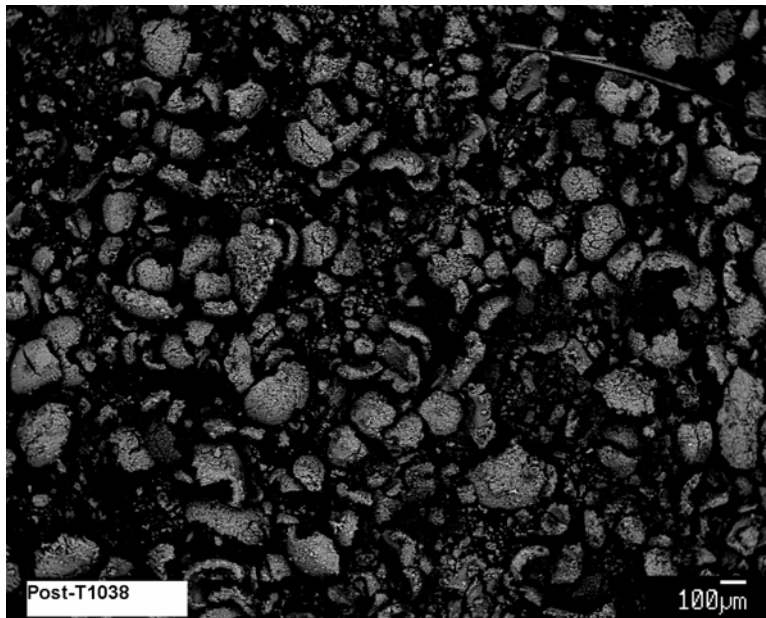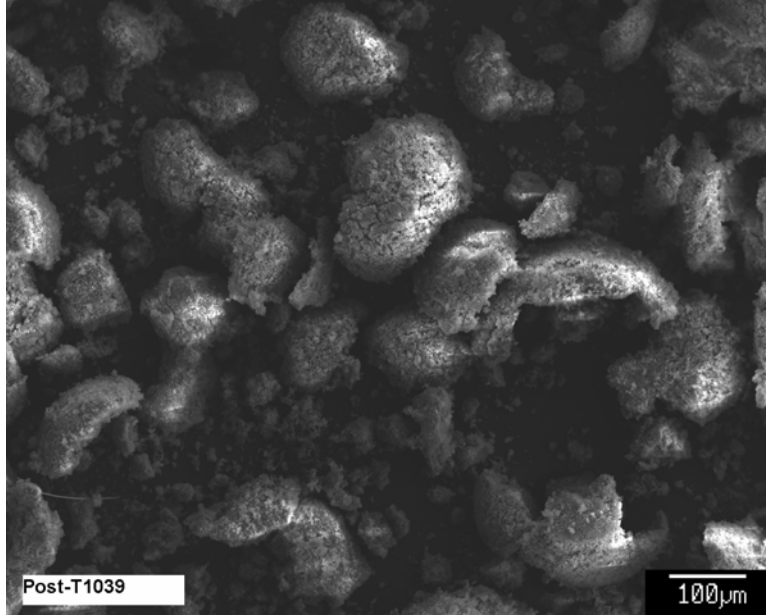**Figure D-89.** Day-30, post-T1-sample-Sed2A SEM-SE image (Post-T1048) magnified 100 times; overview of tank-sediment sample Sed2A.
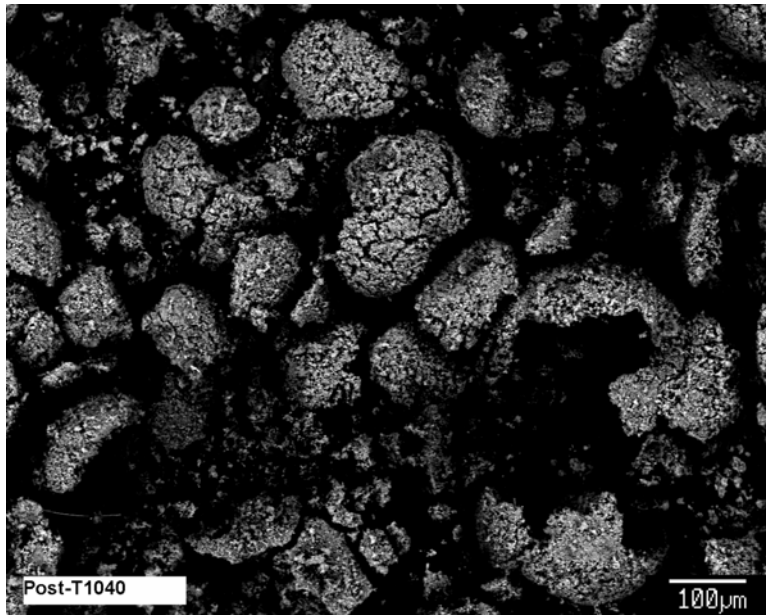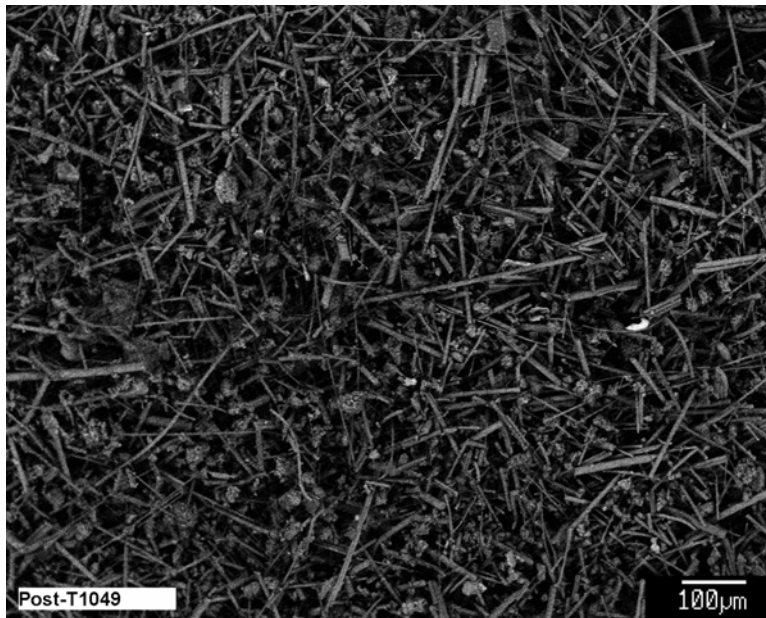


**Figure D-90.** Day-30, post-T1-sample-Sed2A SEM-BSE image (Post-T1049) on the same field as image Post-T1048, as shown in Figure D-89.
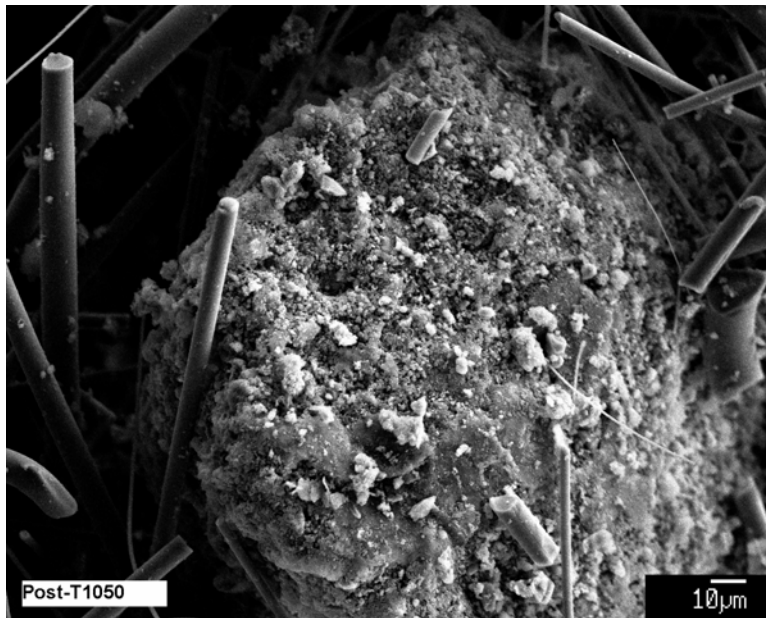
**Figure D-91.    Day-30, post-T1-sample-Sed2A SEM-SE image (Post-T1050) higher magnification of a representative large particle in image Post-T1049, as shown in Figure D-90.**
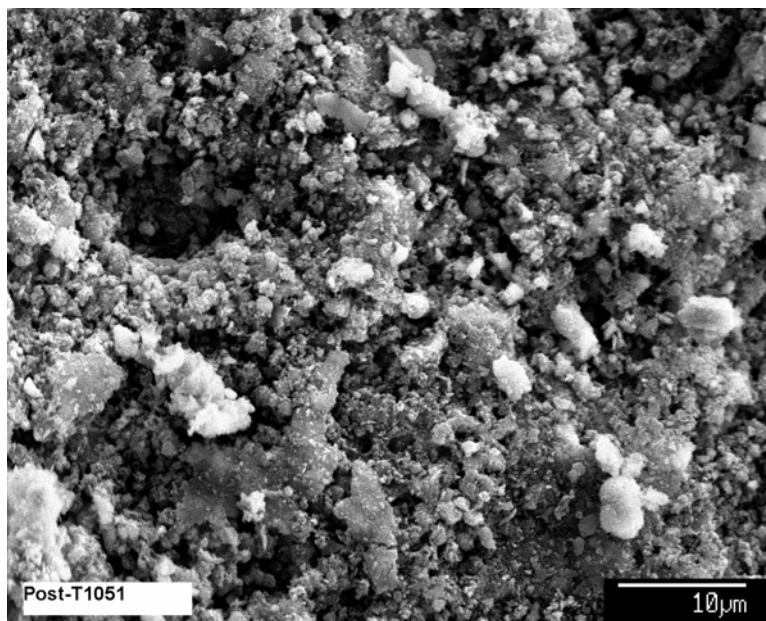


**Figure D-92.    Day-30, post-T1-sample-Sed2A SEM-SE image (Post-T1051) magnified 550 times on the same particle as in image Post-T1050 (see Figure D-91).**
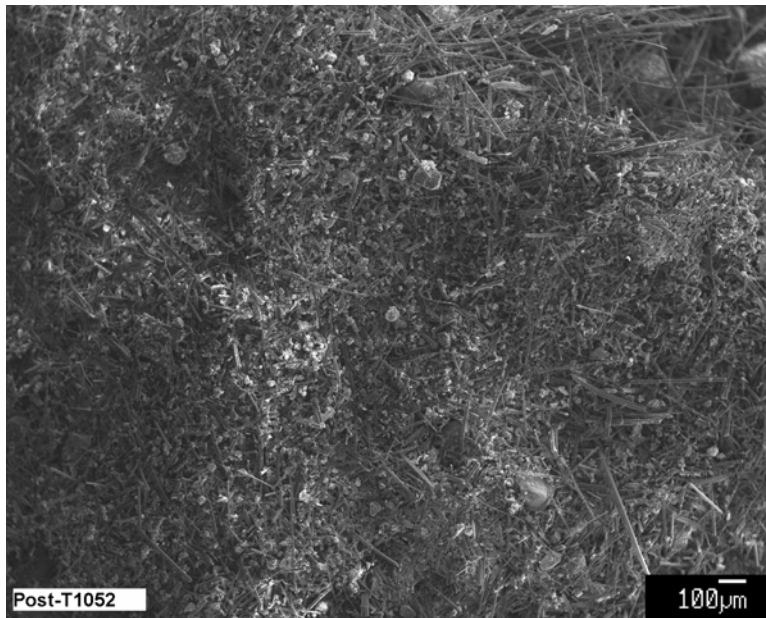
**Figure D-93.** Day-30, post-T1-sample-Sed2A SEM-SE image (Post-T1052) magnified 43 times; overview of tank-sediment sample Sed2A.
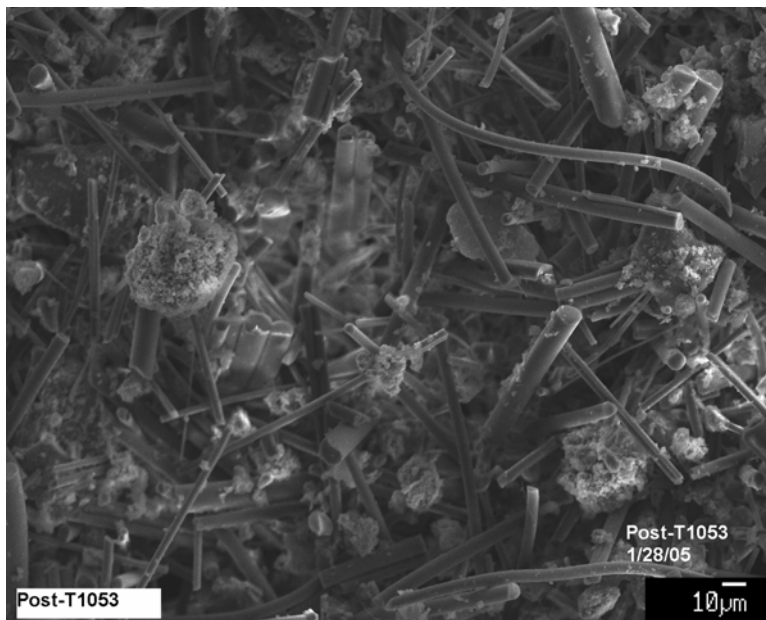


**Figure D-94.** Day-30, post-T1-sample-Sed2A SEM-SE image (Post-T1053) magnified 370 times on two large particles in image Post-T1052 (see Figure D-93).

**Figure D-95.** Day-30, post-T1-sample-Sed2A SEM-BSE image (Post-T1054) magnified 370 times on the same two particles as in image Post-T1053 (see Figure D-94).
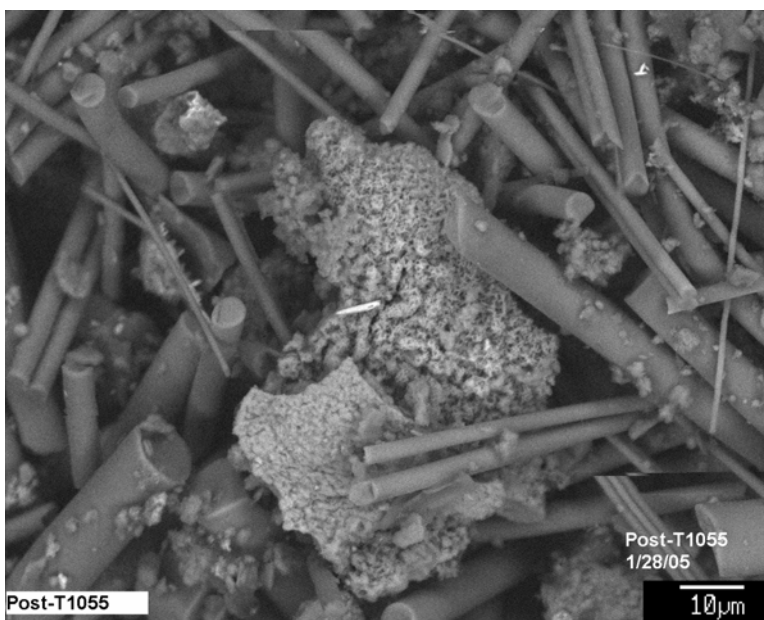


**Figure D-96.** Day-30, post-T1-sample-Sed2A SEM-SE image (Post-T1055) magnified 1000 times on fuzzy particle in image Post-T1054 (see Figure D-95).
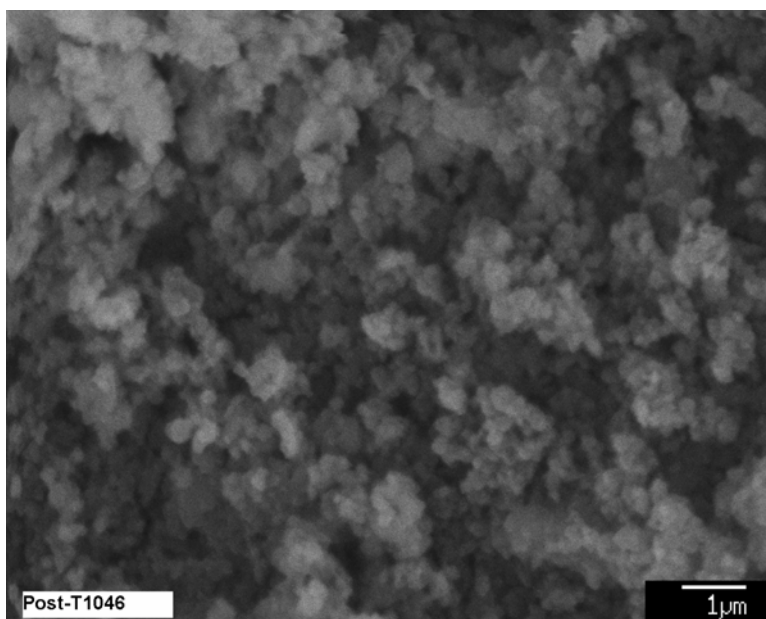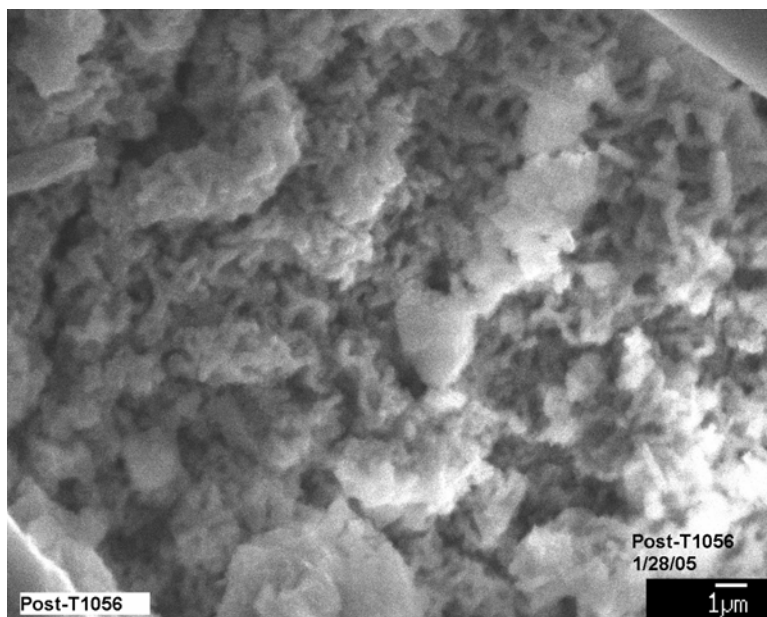
**Figure D-97.** Day-30, post-T1-sample-Sed2A SEM-SE image (Post-T1056) magnified 5000 times on the same particle as in image Post-T1055 (see Figure D-96).
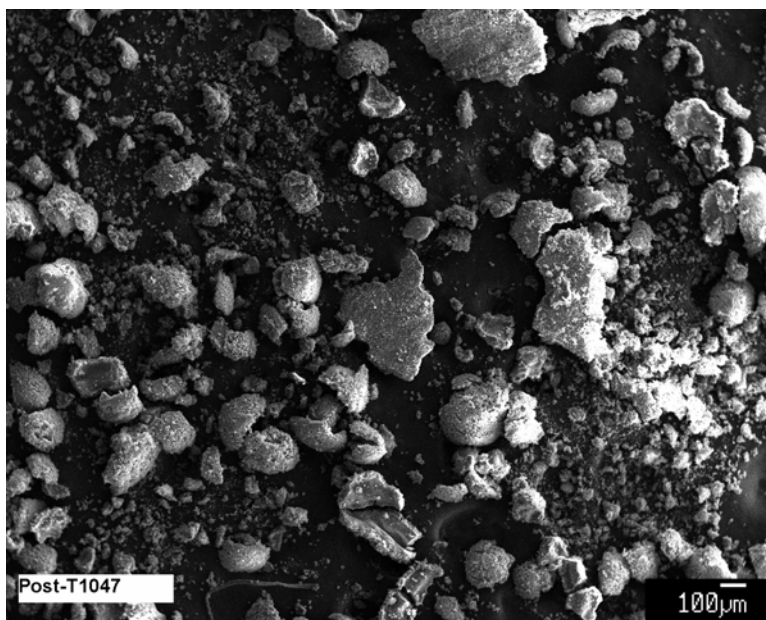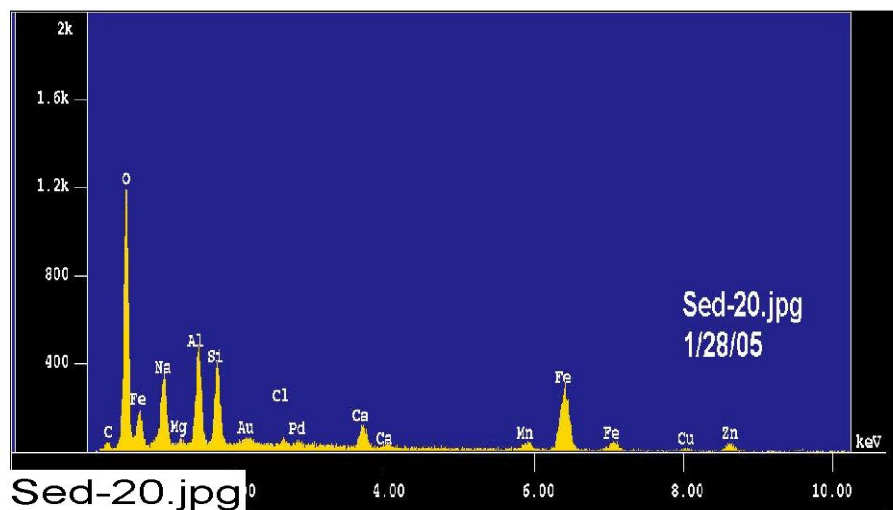


**Figure D-98.** Day-30, post-T1-sample-Sed2A counting spectrum (EDS SED-20) on the fuzzy particle, as shown in Figure D-97.

The results from the chemical composition analysis for EDS SED-20 are given in Table D-33.

**Table D-33.    The Chemical Compositions for EDS SED-20**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 20
Comment    : Tank sediment sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec     Aperture #    : 1
            Acc. Volt   : 15.0 KV         Probe Current : 1.049E-09 A
            Stage Point : X=22.235 Y=61.110 Z=12.288
            Acq. Date   : Fri Jan 28 15:12:25 2005


Element    Mode     ROI(KeV)    K-ratio(%)  +/-     Net/Background
  O K      Normal   0.25- 0.77   27.8526    0.0049   11397 /      54
 Na K      Normal   0.83- 1.28    0.4496    0.0129     579 /      38
 Mg K      Normal   1.03- 1.52    0.1386    0.0011     225 /     354
 Al K      Normal   1.26- 1.78    2.0430    0.0012    3963 /     142
 Si K      Normal   1.50- 2.07    1.7540    0.0014    3400 /     276
 Cl K      Normal   2.34- 3.06    0.4256    0.0009     461 /      30
 Ca K      Normal   3.40- 4.30    1.2765    0.0072    1309 /      20
 Fe K      Normal   6.04- 7.40    9.3292    0.1066    3077 /      14
 Zn K      Normal   8.22-10.03    5.3859    0.0070     637 /       4
 Mn K      Normal   5.53- 6.82    1.2839    0.0012     482 /      13
 Cu K      Normal   7.63- 9.27    1.2101    0.0038     189 /       7
---------------------------------------------------------------------
                    Chi_square =   4.8801

Element Mass%    Atomic%   ZAF      Z      A      F
     O   46.310  70.7546 0.8015 0.9337 0.8586 0.9999
    Na    1.569   1.6684 1.6827 0.9371 1.7934 1.0013
    Mg    0.443   0.4449 1.5394 0.9392 1.6380 1.0006
    Al    6.045   5.4760 1.4264 0.9441 1.5121 0.9992
    Si    4.679   4.0727 1.2861 0.9322 1.3799 0.9999
    Cl    0.939   0.6471 1.0632 0.9853 1.0802 0.9989
    Ca    2.492   1.5198 0.9412 0.9418 1.0035 0.9959
    Fe   17.787   7.7854 0.9191 0.9325 1.0011 0.9846
    Zn   13.617   5.0918 1.2188 1.2124 1.0053 1.0000
    Mn    3.069   1.3655 1.1523 1.1611 1.0018 0.9906
    Cu    3.051   1.1737 1.2154 1.2056 1.0081 1.0000
---------------------------------------------------------------------
Total    100.000 100.0000
Normalization factor =  2.0743
```
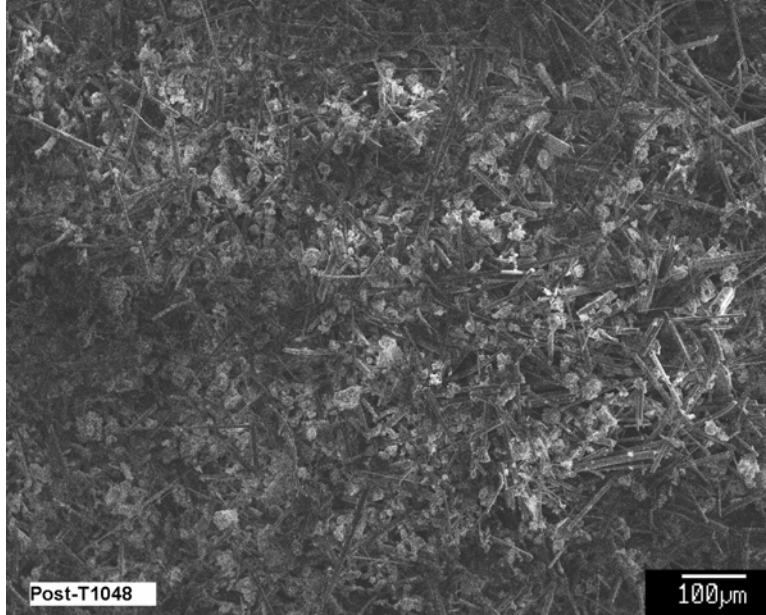
**Figure D-99.   Day-30, post-T1-sample-Sed2A counting spectrum (EDS SED-21) on another particle of tank-sediment sample Sed2A.**

The results from the chemical composition analysis for EDS SED-21 are given in Table D-34.

**Table D-34.     The Chemical Compositions for EDS SED-21**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 21
Comment    : Tank sediment sample
Condition : Full Scale   : 20KeV(10eV/ch,2Kch)
             Live Time    : 100.000 sec    Aperture #      : 1
             Acc. Volt    : 15.0 KV         Probe Current : 4.347E-10 A
             Stage Point : X=21.632 Y=61.902 Z=12.288
             Acq. Date    : Fri Jan 28 15:17:22 2005


Element      Mode     ROI(KeV)    K-ratio(%)  +/-    Net/Background
  O K      Normal   0.25- 0.77    26.3138   0.0029     4462 /        40
  Na K     Normal   0.83- 1.28     2.3020   0.0068     1228 /        32
  Al K     Normal   1.26- 1.78     4.1307   0.0011     3320 /       234
  Si K     Normal   1.50- 2.07     7.5148   0.0017     6037 /       198
  Ca K     Normal   3.40- 4.30     0.7705   0.0040      327 /        10
   F K     Normal   0.46- 0.87     2.8319   0.0019      274 /        60
-----------------------------------------------------------------------
                              Chi_square =   5.2691

Element Mass%    Atomic%    ZAF      Z       A       F
     O   47.350  57.4238 0.8368 0.9899 0.8455 0.9997
    Na    5.724   4.8309 1.1563 0.9943 1.1623 1.0005
    Al   10.135   7.2883 1.1410 1.0024 1.1435 0.9954
    Si   18.840  13.0151 1.1658 0.9902 1.1774 1.0000
    Ca    1.659   0.8029 1.0010 1.0037 0.9972 1.0001
     F   16.292  16.6389 2.6752 1.0987 2.4353 0.9998
-----------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  2.1505
```

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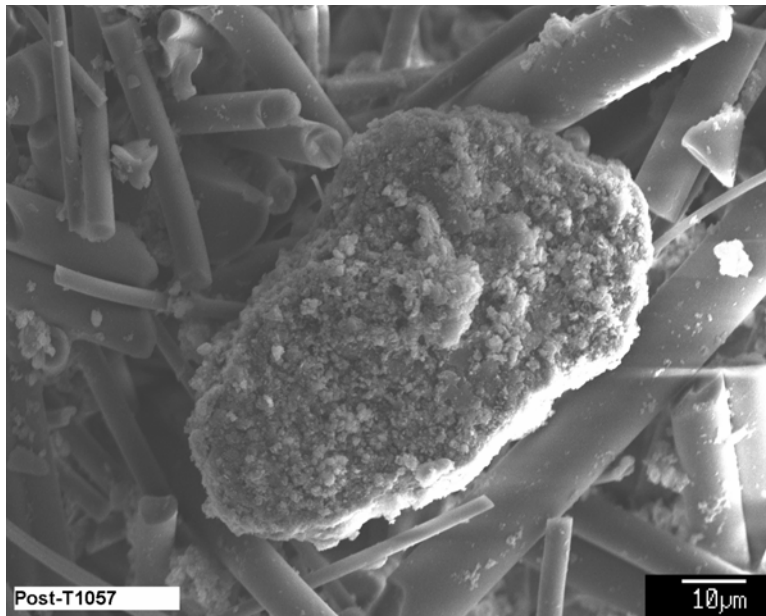
Post-T1057                                    10μm

**Figure D-100. Day-30, post-T1-sample-Sed2A SEM-SE image (Post-T1057) magnified 1000 times on the particle analyzed in the counting spectrum (EDS SED-21), as shown in Figure D-99.**



sed-22.jpg
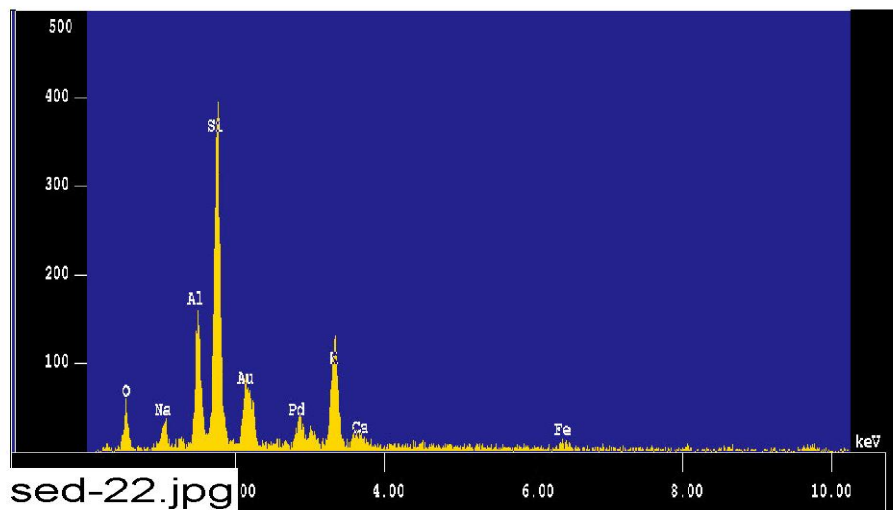
**Figure D-101. Day-30, post-T1-sample-Sed2A counting spectrum (EDS SED-22) on a large round particle of K-feldspar, probably from the concrete sample.**

The results from the chemical composition analysis for EDS SED-22 are given in Table D-35.

**Table D-35.     The Chemical Compositions for EDS SED-22**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 22
Comment    : Tank sediment sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #    : 1
             Acc. Volt   : 15.0 KV        Probe Current : 4.322E-10 A
             Stage Point : X=21.652 Y=61.673 Z=12.288
             Acq. Date   : Fri Jan 28 15:23:18 2005


Element     Mode    ROI(KeV)    K-ratio(%)  +/-     Net/Background
  O K       Normal  0.25- 0.77    2.6175   0.0010      441 /        7
 Na K       Normal  0.83- 1.28    0.3307   0.0029      175 /        8
 Al K       Normal  1.26- 1.78    1.5862   0.0007     1268 /      129
 Si K       Normal  1.50- 2.07    4.1906   0.0013     3347 /       93
 Ca K       Normal  3.40- 4.30    0.2405   0.0034      102 /       23
  K K       Normal  3.01- 3.86    5.7503   0.0022     1524 /       17
 Fe K       Normal  6.04- 7.40    0.6338   0.0299       86 /        6
----------------------------------------------------------------------
                    Chi_square =  1.1162

Element Mass%    Atomic%    ZAF     Z      A      F
    O   26.266  42.2610 1.8669 0.9646 1.9353 1.0000
    Na   1.927   2.1575 1.0839 0.9681 1.1192 1.0003
    Al   9.032   8.6170 1.0593 0.9753 1.0936 0.9932
    Si  24.485  22.4414 1.0870 0.9631 1.1300 0.9988
    Ca   1.391   0.8934 1.0759 0.9732 1.1057 0.9998
    K   33.547  22.0848 1.0854 1.2037 0.9028 0.9987
    Fe   3.351   1.5448 0.9837 0.9647 1.0189 1.0008
----------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  5.3752
```
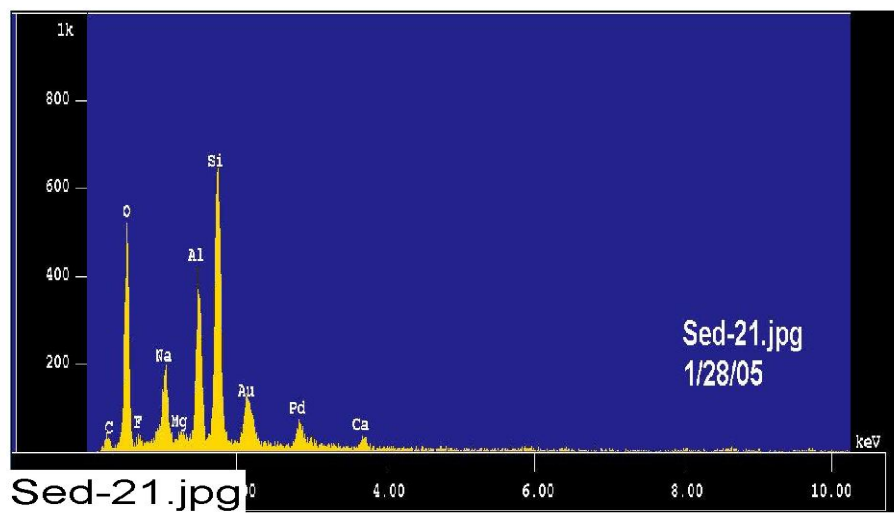
**Figure D-102. Day-30, post-T1-sample-Sed2A SEM-SE image (Post-T1058) of the high-Z particles analyzed in EDS counting spectra EDS-21 and -22, magnified 150 times, as shown in Figure D-99 and Figure D-101.**
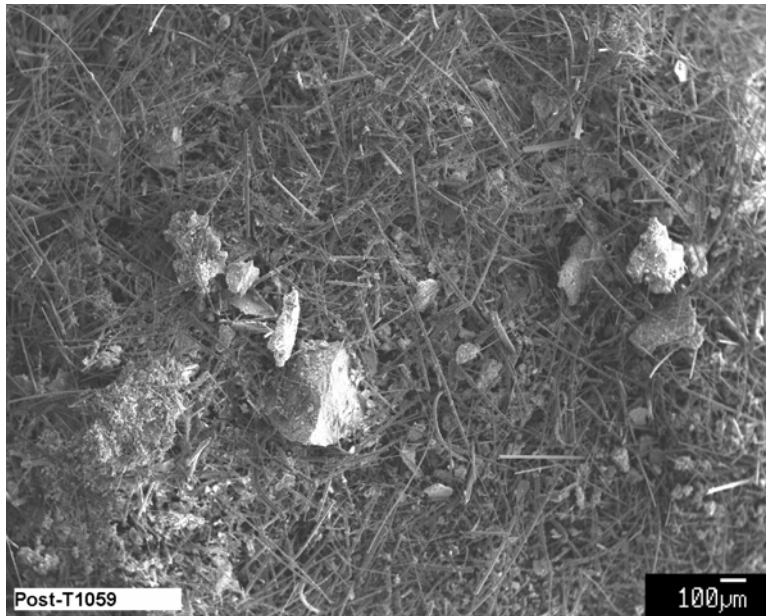


**Figure D-103. Day-30, post-T1-sample-Sed2B SEM-SE image (Post-T1059) magnified 40 times; overview of large particles in tank-sediment sample Sed2B.**
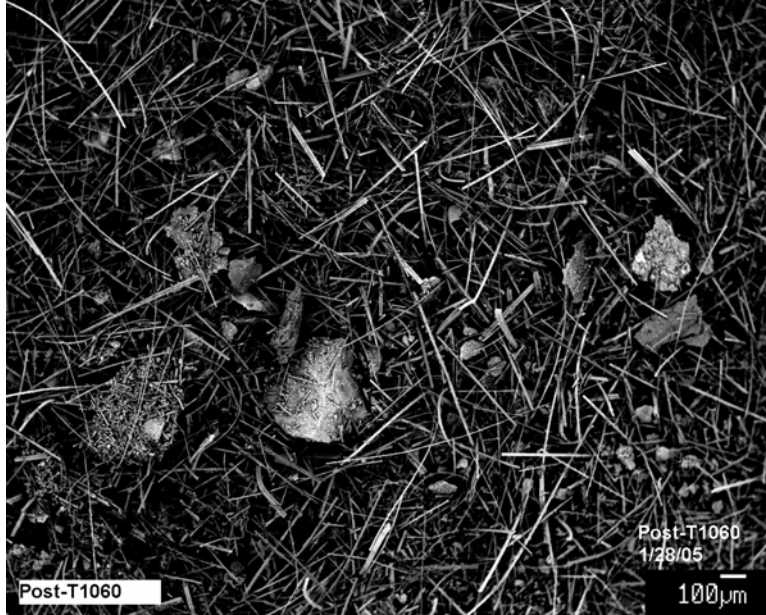
**Figure D-104.** Day-30, post-T1-sample-Sed2B SEM-BSE image (Post-T1060) magnified 40 times on the same particles shown above.



**Figure D-105.** Day-30, post-T1-sample-Sed2B SEM-BSE image (Post-T1060annotated) magnified 40 times on the same particles as in image Post-T1060 (see Figure D-104).

**Figure D-106.** Day-30, post-T1-sample-Sed2B counting spectrum (EDS SED-23) on the plate, as shown in Figure D-105. This sample shows O>Na>Al, which is similar to the filtrate composition from January 18, 2005.

The results from the chemical composition analysis for EDS SED-23 are given in Table D-36.

**Table D-36.**     **The Chemical Compositions for EDS SED-23**

```
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Group     : NRC
Sample    : PostT1_1-28-05  ID# : 23
Comment   : Tank sediment sample
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 4.184E-10 A
            Stage Point : X= 8.337 Y=63.713 Z=12.288
            Acq. Date   : Fri Jan 28 15:41:44 2005


Element     Mode     ROI(KeV)    K-ratio(%)  +/-    Net/Background
  O K      Normal   0.25- 0.77    41.2748   0.0035    6737 /       16
 Na K      Normal   0.83- 1.28     5.3562   0.0081    2749 /       16
 Al K      Normal   1.26- 1.78     2.5107   0.0008    1942 /       34
 Si K      Normal   1.50- 2.07     0.6362   0.0007     492 /      115
 Ca K      Normal   3.40- 4.30     0.9001   0.0041     368 /        7
 Mn K      Normal   5.53- 6.82     0.7900   0.0006     118 /        2
 Fe K      Normal   6.04- 7.40     0.8879   0.0277     117 /        4
-----------------------------------------------------------------------
                      Chi_square =  6.1401

Element Mass%    Atomic%   ZAF      Z      A      F
     O   67.967  78.3590 0.6757 0.9879 0.6840 0.9999
    Na   15.696  12.5931 1.2024 0.9924 1.2102 1.0012
    Al    7.758   5.3038 1.2680 1.0006 1.2677 0.9996
    Si    1.894   1.2441 1.2218 0.9885 1.2360 0.9999
    Ca    2.149   0.9891 0.9798 1.0024 0.9788 0.9987
    Mn    2.380   0.7992 1.2364 1.2421 0.9954 1.0000
    Fe    2.155   0.7117 0.9958 0.9989 0.9961 1.0008
-----------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  2.4371
```
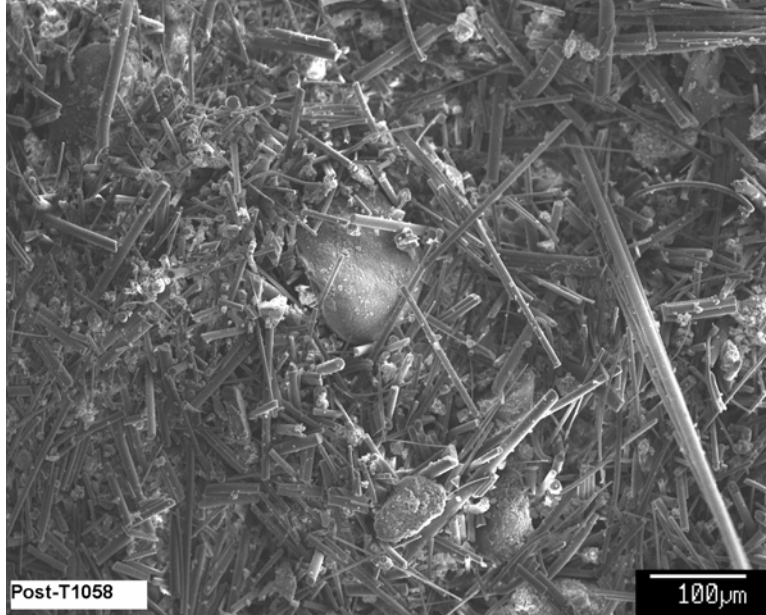
**Figure D-107.** Day-30, post-T1-sample-Sed2B counting spectrum (EDS SED-24) for the particles on the plate in image Post-T1060 (see Figure D-105).

The results from the chemical composition analysis for EDS SED-24 are given in Table D-37.

**Table D-37.     The Chemical Compositions for EDS SED-24**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 24
Comment    : Tank sediment sample
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #   : 1
             Acc. Volt   : 15.0 KV        Probe Current : 4.195E-10 A
             Stage Point : X= 8.337 Y=63.713 Z=12.288
             Acq. Date   : Fri Jan 28 15:46:01 2005


Element    Mode     ROI(KeV)    K-ratio(%)  +/-    Net/Background
   C K     Normal   0.09- 0.46   0.0000     0.0000        0 /      58
   O K     Normal   0.25- 0.77  39.6299     0.0037     6485 /      38
   F K     Normal   0.46- 0.87   2.0252     0.0029      189 /      72
  Na K     Normal   0.83- 1.28   4.3170     0.0083     2222 /      39
  Mg K     Normal   1.03- 1.52   0.6122     0.0011      398 /     292
  Al K     Normal   1.26- 1.78   4.0630     0.0011     3152 /     226
  Si K     Normal   1.50- 2.07   7.5171     0.0017     5827 /     185
  Ca K     Normal   3.40- 4.30   2.4193     0.0055      992 /      14
  Mn K     Normal   5.53- 6.82   1.1727     0.0007      176 /       6
  Fe K     Normal   6.04- 7.40   0.7872     0.0314      104 /       4
--------------------------------------------------------------------
                       Chi_square =  4.2987

Element Mass%   Atomic%   ZAF      Z      A      F
    C    0.000   0.0000 4.7675 1.0337 4.6124 0.9999
    O   52.424  64.0771 0.8428 0.9859 0.8551 0.9998
    F    9.581   9.8616 3.0142 1.0942 2.7551 0.9998
   Na    8.097   6.8877 1.1951 0.9902 1.2060 1.0007
   Mg    1.139   0.9165 1.1859 0.9928 1.1954 0.9992
   Al    7.634   5.5326 1.1971 0.9983 1.2031 0.9968
   Si   13.858   9.6491 1.1746 0.9861 1.1913 0.9999
   Ca    3.759   1.8341 0.9900 0.9993 0.9915 0.9992
   Mn    2.277   0.8106 1.2373 1.2374 0.9999 1.0000
   Fe    1.230   0.4306 0.9953 0.9950 0.9996 1.0008
--------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.5695
```

**Figure D-108.** Day-30, post-T1-sample-Sed2B SEM-SE image (Post-T1061) magnified 160 times on the particles and fibers at left side of image Post-T1059 (see Figure D-103).
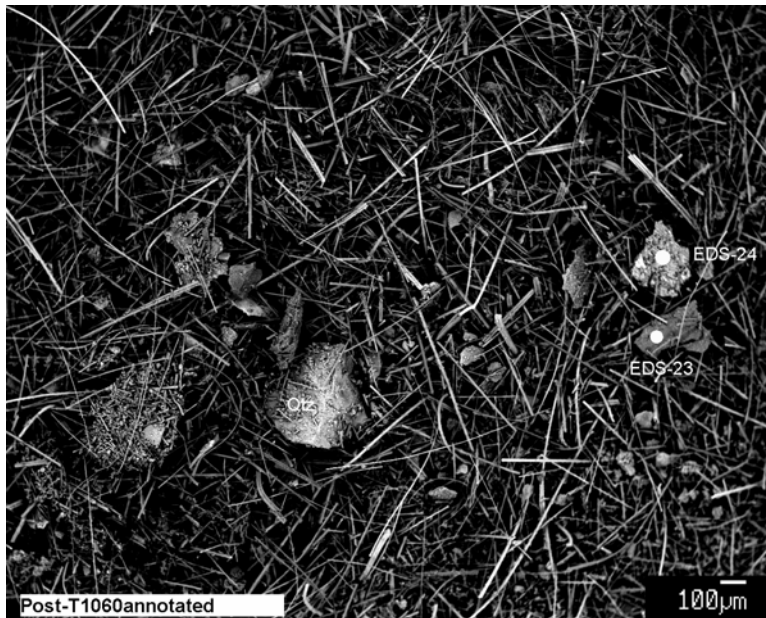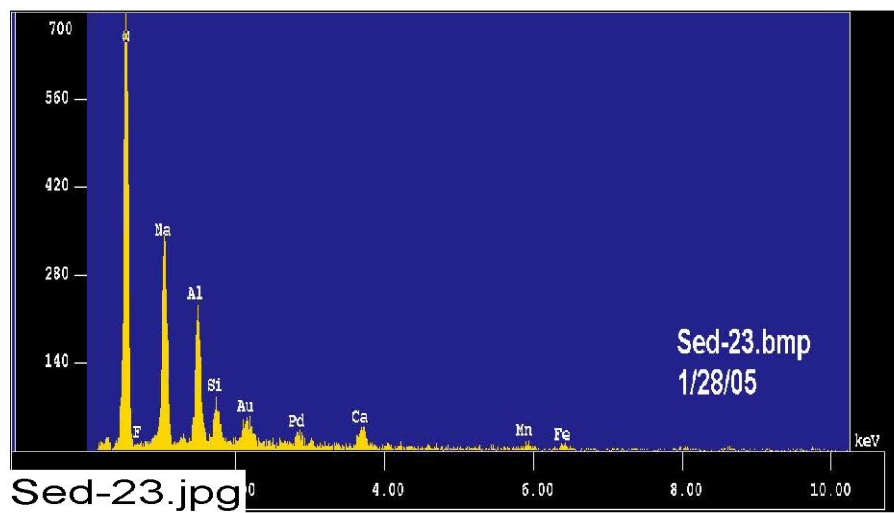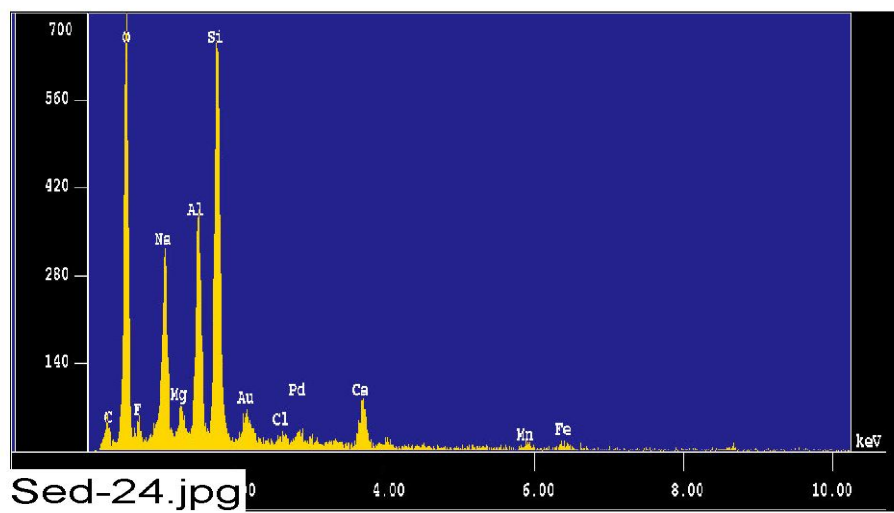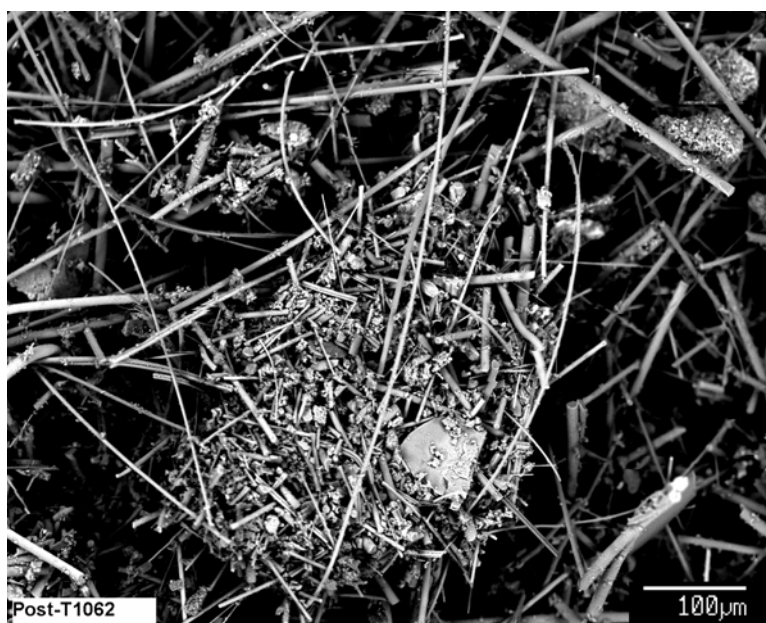


**Figure D-109.** Day-30, post-T1-sample-Sed2B SEM-BSE image (Post-T1062) magnified 160 times on the same field as in image Post-T1061 (see Figure D-108).
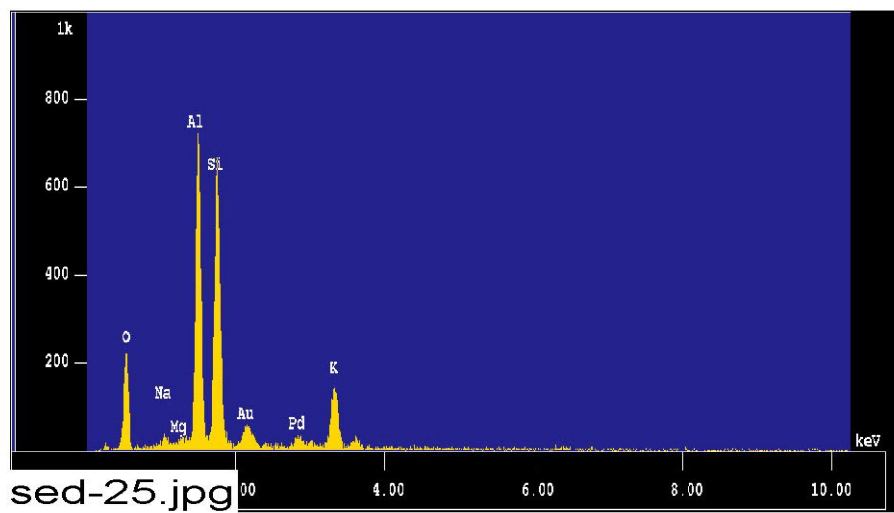
**Figure D-110.** Day-30, post-T1-sample-Sed2B counting spectrum (EDS SED-25) on the particle in agglomerate image Post-T1062 (see Figure D-109).

The results from the chemical composition analysis for EDS SED-25 are given in Table D-38.

**Table D-38.  The Chemical Compositions for EDS SED-25**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 25
Comment    : Tank sediment sample
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time  : 100.000 sec    Aperture #    : 1
             Acc. Volt  : 15.0 KV        Probe Current : 4.259E-10 A
             Stage Point : X= 9.289 Y=64.031 Z=12.288
             Acq. Date  : Fri Jan 28 15:57:48 2005


Element    Mode     ROI(KeV)    K-ratio(%)  +/-    Net/Background
  O K      Normal   0.25- 0.77   12.2389    0.0019    2033 /      14
 Al K      Normal   1.26- 1.78    7.7756    0.0014    6123 /     206
 Si K      Normal   1.50- 2.07    7.3272    0.0017    5767 /     346
  K K      Normal   3.01- 3.86    7.2242    0.0024    1886 /      15
 Na K      Normal   0.83- 1.28    0.3054    0.0033     160 /      25
 Mg K      Normal   1.03- 1.52    0.1299    0.0008      86 /     228
------------------------------------------------------------------
                        Chi_square =  3.9525

Element Mass%    Atomic%   ZAF     Z      A      F
     O   40.610  56.4293 1.4063 0.9807 1.4340 1.0000
    Al   19.030  15.6793 1.0373 0.9923 1.0508 0.9948
    Si   20.371  16.1251 1.1784 0.9800 1.2031 0.9994
     K   18.942  10.7696 1.1113 1.2263 0.9062 1.0000
    Na    0.747   0.7224 1.0369 0.9846 1.0534 0.9998
    Mg    0.300   0.2743 0.9792 0.9871 0.9962 0.9959
------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  2.3594
```
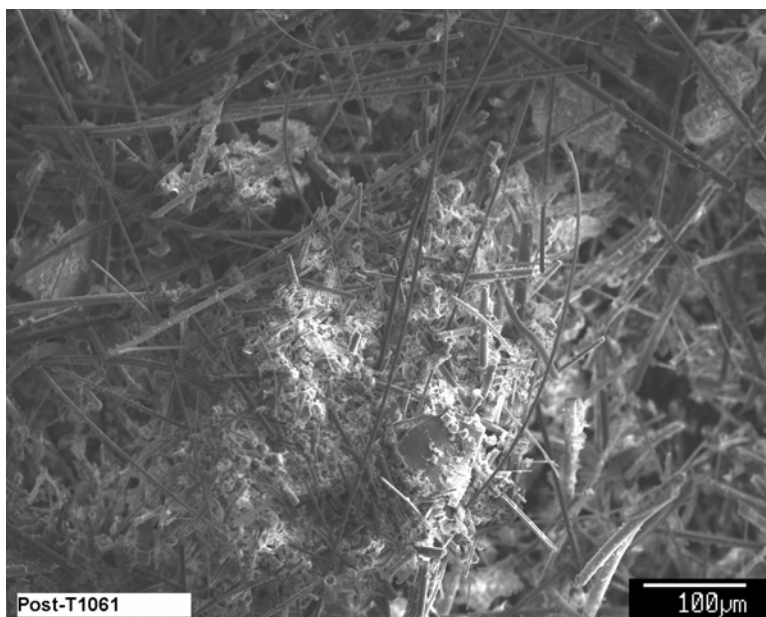
**Figure D-111.** Day-30, post-T1-sample-P1 SEM-SE image (Post-T1063) magnified 40 times; overview of the large particles present in the pump-suction-debris sample.



**Figure D-112.** Day-30, post-T1-sample-P1 SEM-BSE image (Post-T1064) magnified 40 times on the same large particles as in image Post-T1063 (see Figure D-111).

**Figure D-113. Day-30, post-T1-sample-P1 SEM-SE image (Post-T1064 annotated) annotating the EDS locations.**
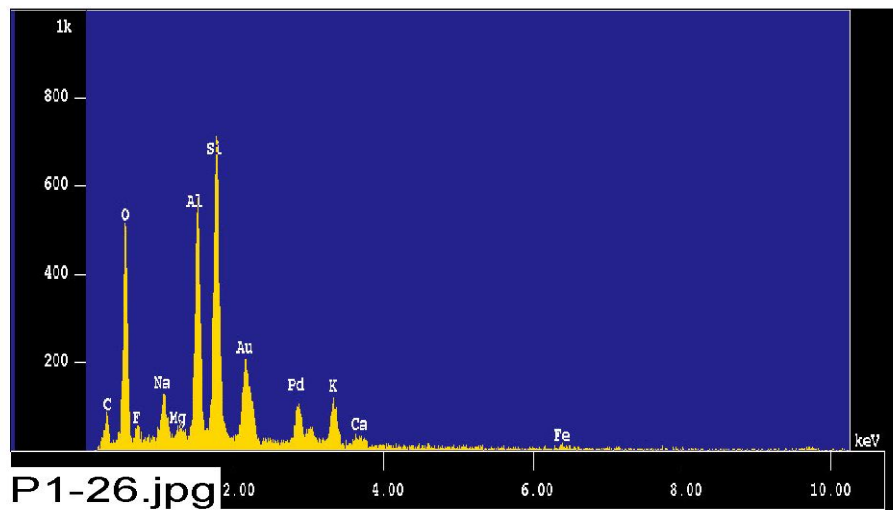


**Figure D-114. Day-30, post-T1-sample-P1 counting spectrum (EDS P1-26) on the triangular large particle in image Post-T1064 (see Figure D-113).**

The results from the chemical composition analysis for EDS P1-26 are given in Table D-39.

**Table D-39.     The Chemical Compositions for EDS P1-26**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 26
Comment    : Pump Suction Debris
Condition : Full Scale  : 20Kev(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #     : 1
            Acc. Volt   : 15.0 KV        Probe Current : 4.196E-10 A
            Stage Point : X=70.335 Y=64.112 Z=12.288
            Acq. Date   : Fri Jan 28 16:14:09 2005


Element     Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
  C K      Normal   0.09- 0.46   1.4540   0.0003     353 /      59
  O K      Normal   0.25- 0.77  28.7309   0.0030    4703 /      46
  F K      Normal   0.46- 0.87   5.1768   0.0022     484 /      72
 Na K      Normal   0.83- 1.28   1.5083   0.0060     776 /      42
 Al K      Normal   1.26- 1.78   5.9468   0.0013    4614 /     226
 Si K      Normal   1.50- 2.07   7.8631   0.0018    6097 /     299
  K K      Normal   3.01- 3.86   5.2345   0.0023    1347 /      32
 Fe K      Normal   6.04- 7.40   0.8985   0.0331     119 /       6
-----------------------------------------------------------------
                     Chi_square =  4.0233

Element Mass%    Atomic%    ZAF      Z       A       F
   C     8.303  13.0283  4.2517  1.0269  4.1405  1.0000
   O    40.921  48.2017  1.0604  0.9794  1.0829  0.9997
   F    18.488  18.3398  2.6588  1.0871  2.4460  0.9999
  Na     2.433   1.9946  1.2011  0.9839  1.2198  1.0008
  Al     8.989   6.2784  1.1254  0.9920  1.1380  0.9969
  Si    12.026   8.0693  1.1386  0.9799  1.1623  0.9997
   K     7.644   3.6842  1.0872  1.2279  0.8855  0.9999
  Fe     1.196   0.4036  0.9910  0.9898  1.0005  1.0008
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.3432
```

**Figure D-115. Day-30, post-T1-sample-P1 counting spectrum (EDS P1-27) on the flat particle at lower right corner of image Post-T1064 (see Figure D-113).**

The results from the chemical composition analysis for EDS P1-27 are given in Table D-40.

**Table D-40.    The Chemical Compositions for EDS P1-27**

```
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Group      : NRC
Sample     : PostT1_1-28-05  ID# : 27
Comment    : pump suction screen debris
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 4.189E-10 A
            Stage Point : X=70.335 Y=64.112 Z=12.288
            Acq. Date   : Fri Jan 28 16:20:03 2005


Element    Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
  C K      Normal  0.09- 0.46    0.2744   0.0004      67 /       46
  O K      Normal  0.25- 0.77   32.5575   0.0032    5320 /       48
  F K      Normal  0.46- 0.87    3.9728   0.0020     371 /       58
 Na K      Normal  0.83- 1.28    3.8472   0.0077    1977 /       28
 Al K      Normal  1.26- 1.78    7.4580   0.0014    5777 /       38
 Si K      Normal  1.50- 2.07    0.2973   0.0007     230 /      367
 Ca K      Normal  3.40- 4.30    1.8105   0.0052     741 /       12
-----------------------------------------------------------------
                           Chi_square =  6.2495

Element Mass%    Atomic%   ZAF      Z      A      F
      C    2.094  3.2545 4.0445 1.0324 3.9179 1.0000
      O   48.264 56.3066 0.7857 0.9847 0.7982 0.9997
      F   20.048 19.6968 2.6747 1.0930 2.4476 0.9998
     Na    8.725  7.0835 1.2020 0.9892 1.2143 1.0007
     Al   16.804 11.6242 1.1942 0.9974 1.1975 0.9998
     Si    0.711  0.4725 1.2673 0.9853 1.2863 0.9999
     Ca    3.354  1.5620 0.9819 0.9992 0.9826 1.0001
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.8867
```
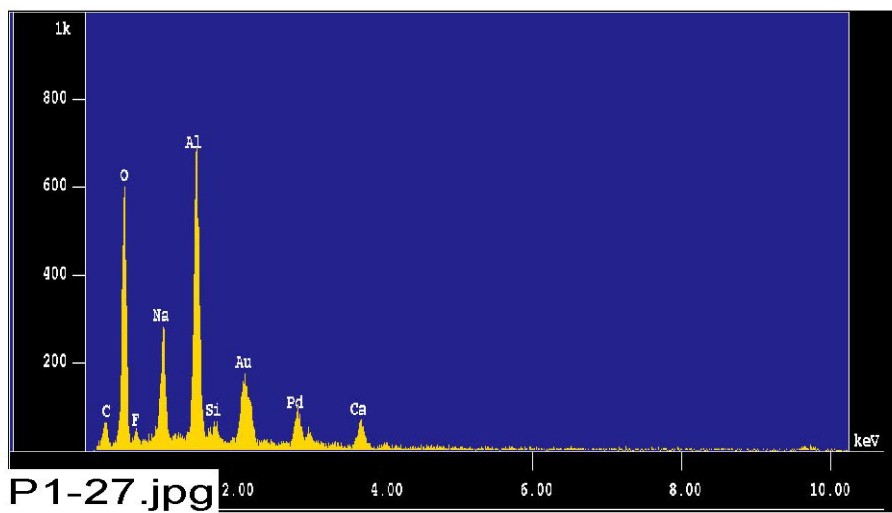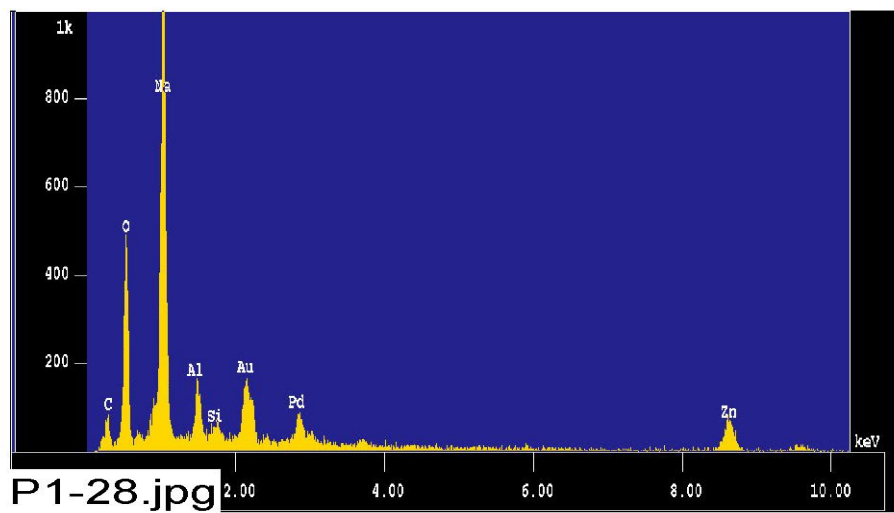
**Figure D-116. Day-30, post-T1-sample-P1 counting spectrum (EDS P1-28) on bright particle in the center of image Post-T1064 (see Figure D-113).**

The results from the chemical composition analysis for EDS P1-28 are given in Table D-41.

**Table D-41.** **The Chemical Compositions for EDS P1-28**

```
Jan 28 16:25 2005   /tmp/eds_pout.log Page 1


Group      : NRC
Sample     : PostT1_1-28-05  ID# : 28
Comment    : pump suction screen debris
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   : 100.000 sec    Aperture #    : 1
            Acc. Volt   : 15.0 KV        Probe Current : 4.194E-10 A
            Stage Point : X=70.335 Y=64.112 Z=12.288
            Acq. Date   : Fri Jan 28 16:24:11 2005


Element     Mode     ROI(KeV)    K-ratio(%)  +/-     Net/Background
  O K      Normal   0.25- 0.77    27.2716   0.0030    4462 /      58
  Na K     Normal   0.83- 1.28     0.0000   0.0000       0 /      43
  Al K     Normal   1.26- 1.78     1.4018   0.0008    1087 /      45
  Zn K     Normal   8.22-10.03    26.4466   0.0088    1250 /       6
  Si K     Normal   1.50- 2.07     0.2317   0.0007     180 /      96
   C K     Normal   0.09- 0.46     1.2827   0.0003     311 /      52
--------------------------------------------------------------------
                       Chi_square =   4.4939

Element Mass%    Atomic%   ZAF      Z      A      F
    O    38.917  59.7633 0.9217 0.9084 1.0148 0.9999
    Na    0.000   0.0000 1.7609 0.9116 1.9289 1.0015
    Al    3.598   3.2767 1.6582 0.9183 1.8058 0.9999
    Zn   47.937  18.0168 1.1708 1.1742 0.9971 1.0000
    Si    0.502   0.4394 1.4004 0.9067 1.5446 1.0000
    C     9.046  18.5038 4.5552 0.9529 4.7807 1.0000
--------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.5482
```
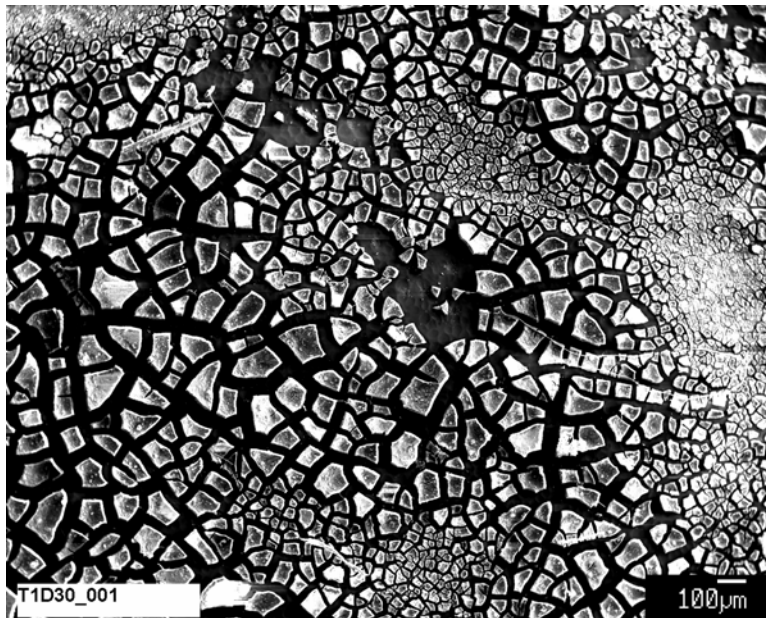
**Figure D-117. Day-30, high-volume sample Microprobe image (T1D30_001) magnified 40 times; overview of the filtrate.**
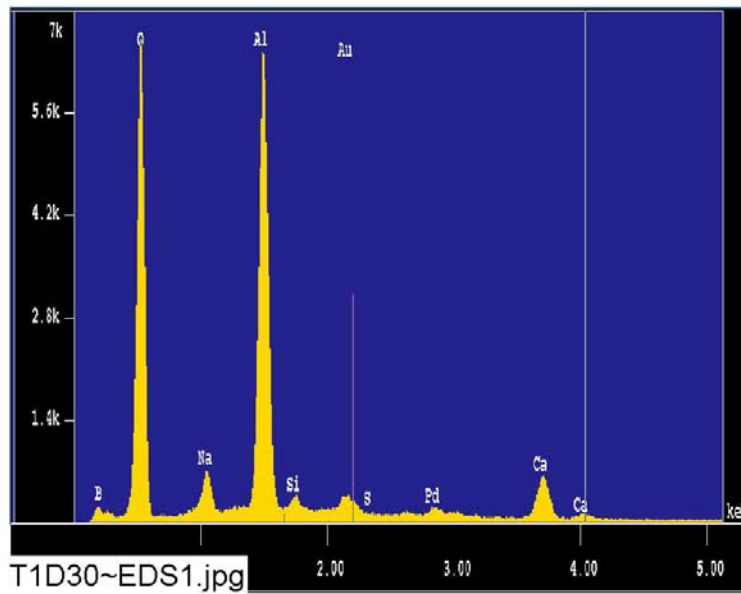


**Figure D-118. Day-30, high-volume sample counting spectrum (T1D30~EDS1) for the filtrate.**

The results from the chemical composition analysis for EDS (T1D30~EDS1) are given in Table D-42.

**Table D-42.     The Chemical Compositions for EDS (T1D30-EDS1)**

```
Feb 25 10:29 2005  /tmp/eds_pout.log Page 1


Group      : NRC
Sample     : T1D30_EDS1  ID# : 1
Comment    : filtrate
Condition  : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   :  60.000 sec    Aperture #    : 2
             Acc. Volt   : 15.0 KV         Probe Current : 2.869E-08 A
             Stage Point : X=51.618 Y=62.980 Z=10.097
             Acq. Date   : Fri Feb 25 10:17:16 2005


Element     Mode     ROI(KeV)    K-ratio(%)  +/-     Net/Background
  O K      Normal   0.25- 0.77    59.9054   0.0095    53380 /     140
 Al K      Normal   1.26- 1.78    13.0861   0.0041    55272 /     259
 Si K      Normal   1.50- 2.07     0.3594   0.0017     1517 /    3145
 Ca K      Normal   3.40- 4.30     3.3380   0.0154     7454 /      63
 Na K      Normal   0.83- 1.28     1.5755   0.0137     4415 /     156
  B K      Normal   0.00- 0.36     2.8524   0.0005     1058 /      52
-------------------------------------------------------------------
                       Chi_square = 105.5553

Element Mass%   Atomic%   ZAF     Z      A      F
     O   57.362  55.9594 0.9173 0.9628 0.9527 1.0000
    Al   14.240   8.2372 1.0424 0.9762 1.0680 0.9998
    Si    0.425   0.2363 1.1334 0.9646 1.1751 0.9999
    Ca    3.311   1.2893 0.9501 0.9801 0.9693 1.0001
    Na    1.736   1.1785 1.0555 0.9678 1.0900 1.0006
     B   22.927  33.0993 7.6998 1.1177 6.8889 1.0000
-------------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  1.0439
```
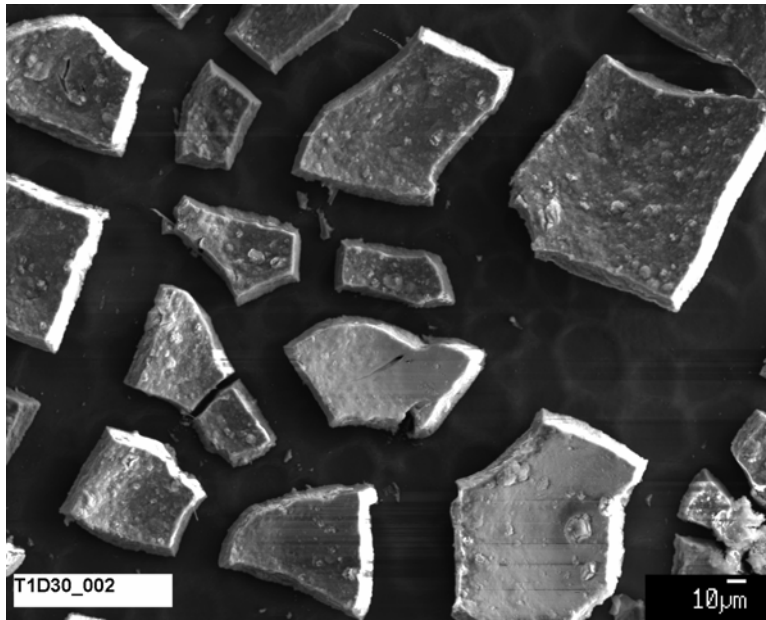
**Figure D-119.** Day-30, high-volume sample microprobe image (T1D30_002) magnified 300 times, close-up of the filter.
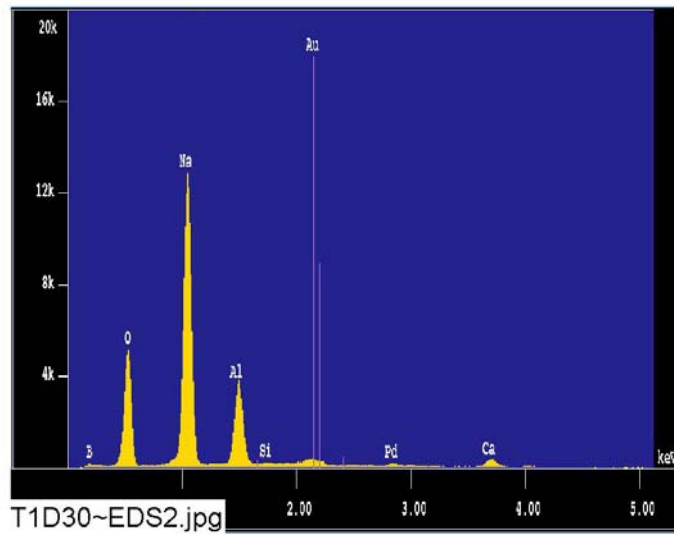


**Figure D-120.** Day-30, high-volume sample counting spectrum (T1D30~EDS2) on the filter, as shown in Figure D-119.

The results from the chemical composition analysis for EDS (T1D30-EDS2) are given in Table D-43.


**Table D-43.    The Chemical Compositions for EDS (T1D30-EDS2)**


```
Feb 25 10:36 2005  /tmp/eds_pout.log Page 1



Group     : NRC
Sample    : T1D30_EDS2  ID# : 2
Comment   : filtrate
Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
            Live Time   :  60.000 sec    Aperture #    : 2
            Acc. Volt   : 15.0 KV        Probe Current : 2.841E-08 A
            Stage Point : X=51.946 Y=63.301 Z=10.071
            Acq. Date   : Fri Feb 25 10:32:31 2005


Element      Mode     ROI(KeV)   K-ratio(%)  +/-    Net/Background
  O K      Normal   0.25- 0.77    49.2514   0.0089    43458 /    178
 Al K      Normal   1.26- 1.78     7.4252   0.0032    31056 /    234
 Si K      Normal   1.50- 2.07     0.1899   0.0016      794 /   1939
 Ca K      Normal   3.40- 4.30     1.9646   0.0133     4344 /     84
 Na K      Normal   0.83- 1.28    37.3248   0.0465   103570 /    196
  B K      Normal   0.00- 0.36     2.1713   0.0005      797 /     62
-----------------------------------------------------------------
                         Chi_square = 63.9091

Element Mass%   Atomic%   ZAF      Z      A      F
     O   39.105  40.6471 0.8807 0.9629 0.9147 0.9999
    Al    8.360   5.1523 1.2488 0.9759 1.2797 0.9999
    Si    0.207   0.1224 1.2075 0.9643 1.2523 1.0000
    Ca    1.696   0.7035 0.9573 0.9790 0.9778 1.0001
    Na   30.078  21.7569 0.8938 0.9676 0.9227 1.0011
     B   20.555  31.6177 10.4997 1.1180 9.3919 1.0000
-----------------------------------------------------------------
Total   100.000 100.0000
Normalization factor =  0.9016
```