Appendix F2

TEM Data for Test #2, Day-17 Solution Samples

Figures

This appendix presents TEM images and diffraction patterns for Test #2, Day-17 (February 22, 2005) filtered and unfiltered solution samples. The filtered sample was obtained by passing solution through a 0.7-μm fiberglass filter at 60°C. The unfiltered solution samples were extracted from the tank directly. A drop of each solution sample was placed onto a copper grid of 200 mesh. After being dried in air at room temperature, the sample was ready for TEM analysis. The TEM results and diffraction patterns were obtained on February 22, 2005. Diffraction patterns show whether the sample was amorphous or crystalline. When a sample gives clear and significant diffraction patterns, it is crystalline. Otherwise, it is amorphous. The results show that all of the Test #2, Day-17 samples were amorphous.

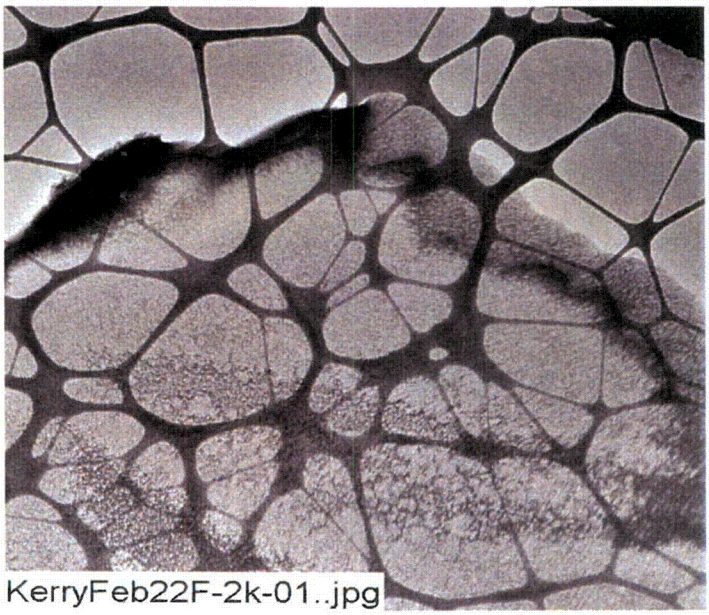


Figure F2-1. Electron micrograph magnified 2000 times for one Test #2, Day-17 filtered sample location. (KerryFeb22F-2k-01)

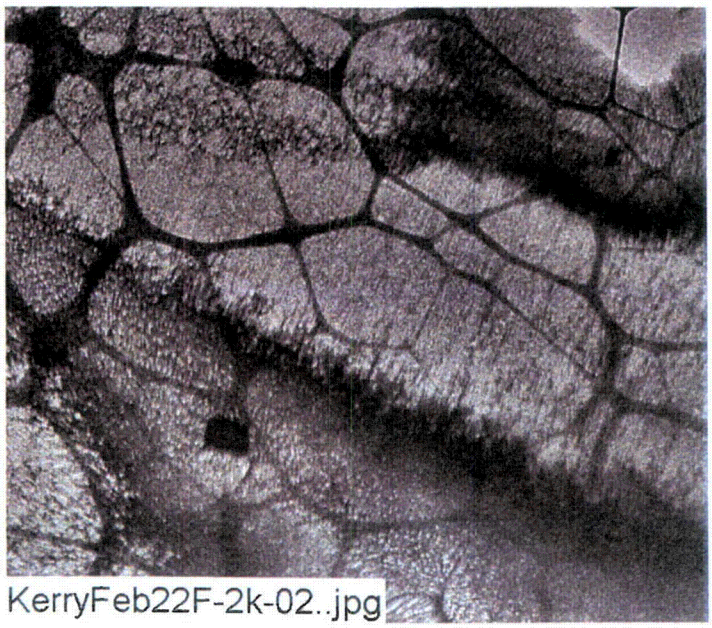


Figure F2-2. Electron micrograph magnified 2000 times for a second Test #2, Day-17 filtered sample location. (KerryFeb22F-2k-02)

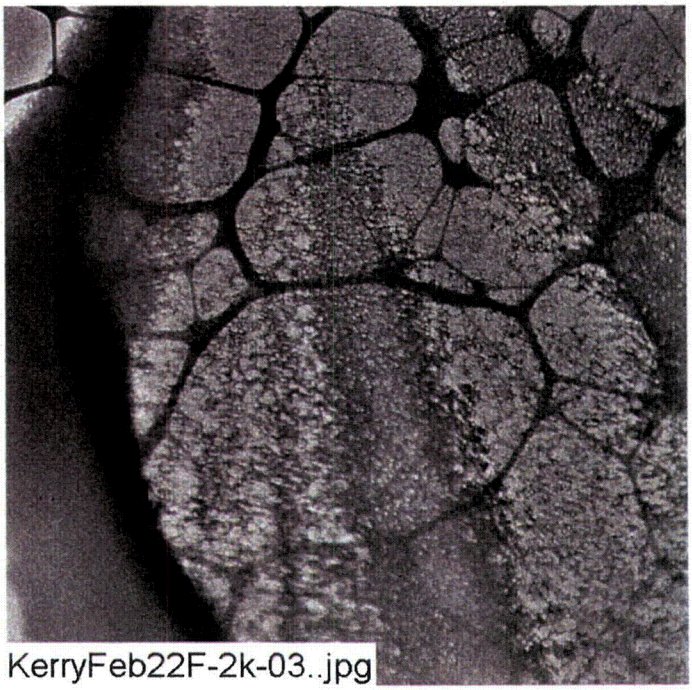


Figure F2-3. Electron micrograph magnified 2000 times for a third Test #2, Day-17 filtered sample location. (KerryFeb22F-2k-03)

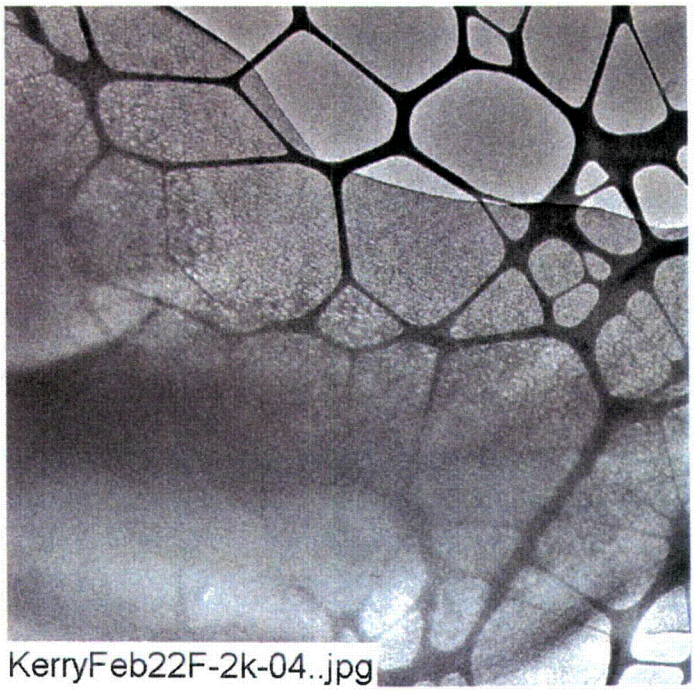


Figure F2-4. Electron micrograph magnified 2000 times for a fourth Test #2, Day-17 filtered sample location. (KerryFeb22F-2k-04)

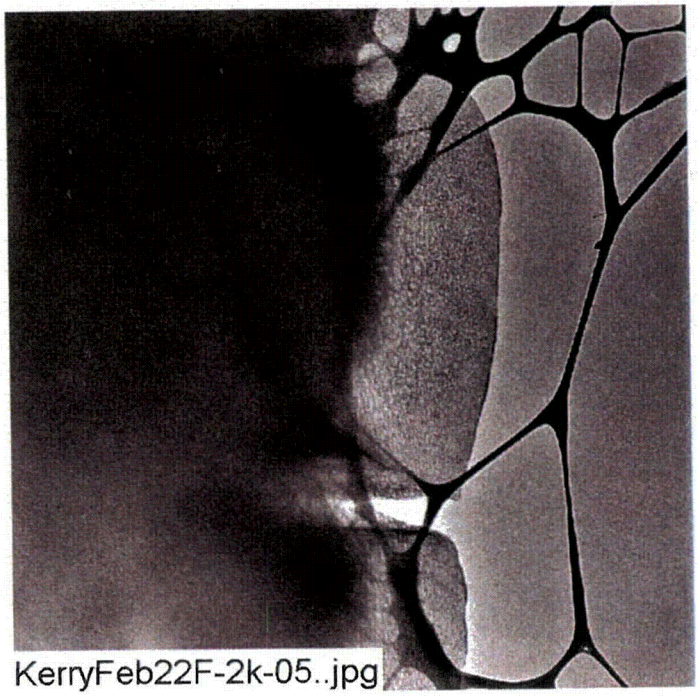


Figure F2-5. Electron micrograph magnified 2000 times for a fifth Test #2, Day-17 filtered sample location. (KerryFeb22F-2k-05)

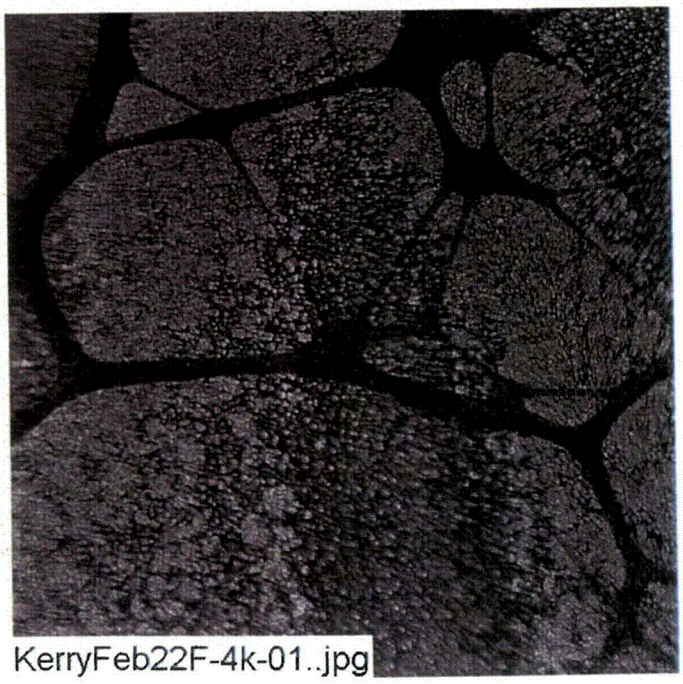


Figure F2-6. Electron micrograph magnified 4000 times for one Test #2, Day-17 filtered sample location. (KerryFeb22F-4k-01)

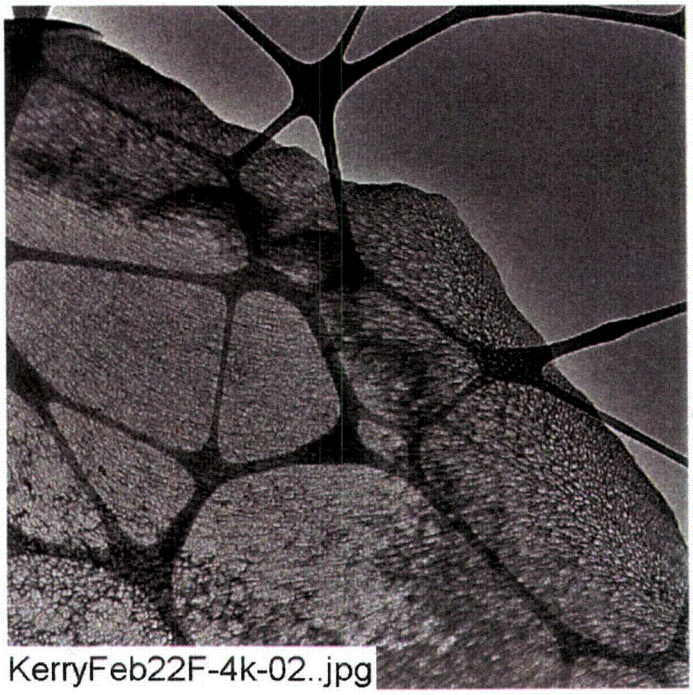


Figure F2-7. Electron micrograph magnified 4000 times for a second Test #2, Day-17 filtered sample location. (KerryFeb22F-4k-02)

Figure F2-8. Electron micrograph magnified 4000 times for a third Test #2, Day-17 filtered sample location. (KerryFeb22F-4k-03)

Figure F2-9. Electron micrograph magnified 4000 times for a fourth Test #2, Day-17 filtered sample location. (KerryFeb22F-4k-04)

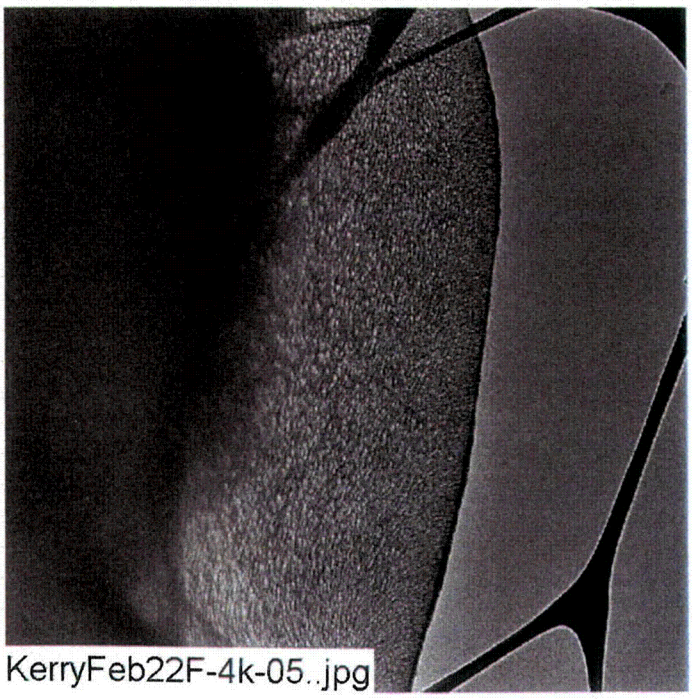


Figure F2-10. Electron micrograph magnified 4000 times for a fifth Test #2, Day-17 filtered sample location. (KerryFeb22F-4k-05)

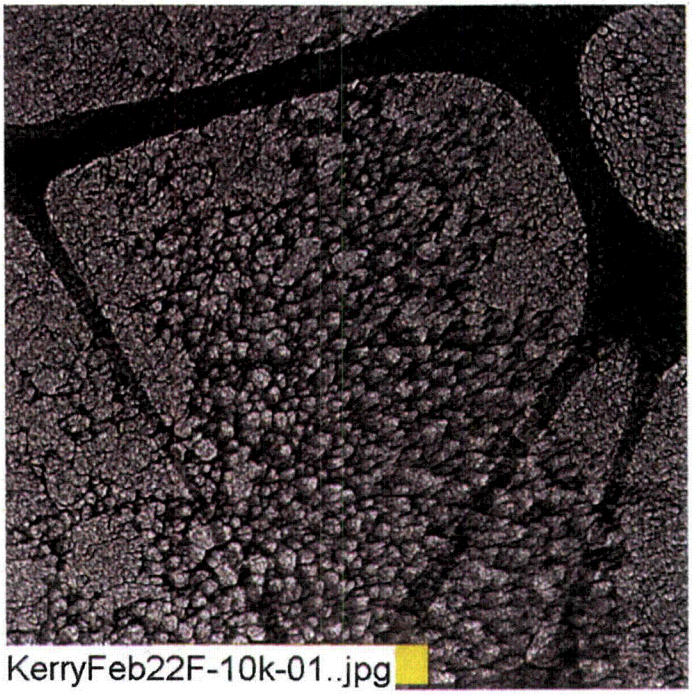


Figure F2-11. Electron micrograph magnified 10,000 times for one Test #2, Day-17 filtered sample location. (KerryFeb22F-10k-01)

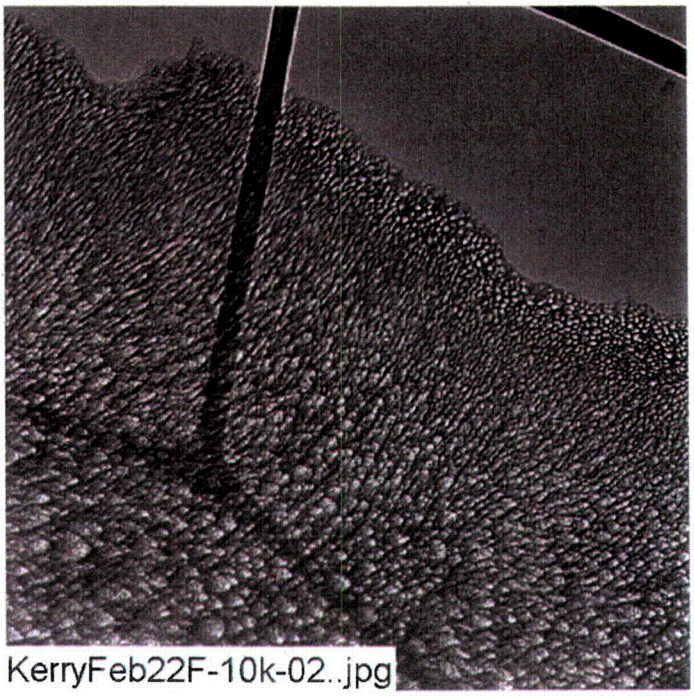


Figure F2-12. Electron micrograph magnified 10,000 times for a second Test #2, Day-17 filtered sample location. (KerryFeb22F-10k-02)

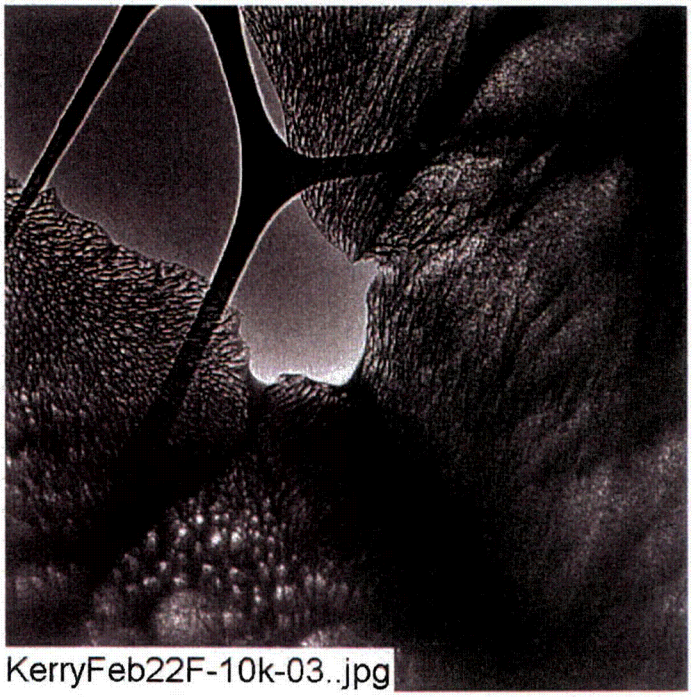


Figure F2-13. Electron micrograph magnified 10,000 times for a third Test #2, Day-17 filtered sample location. (KerryFeb22F-10k-03)

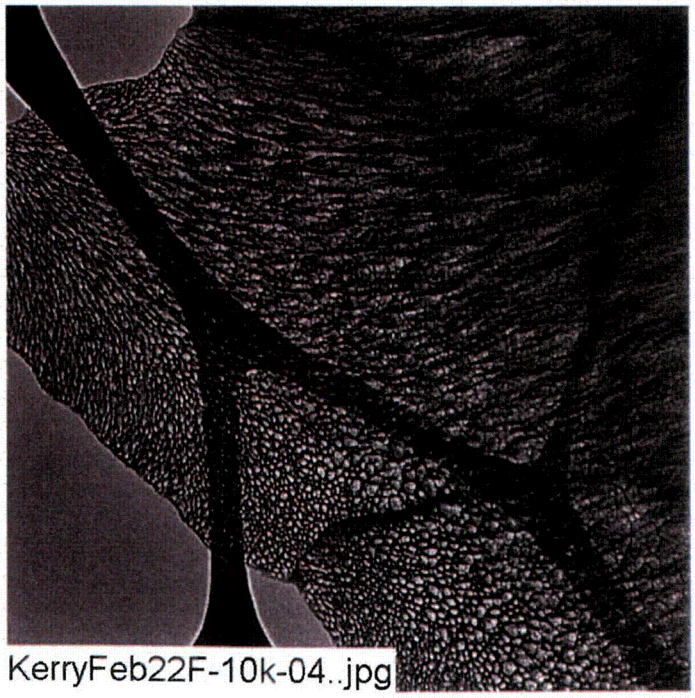


Figure F2-14. Electron micrograph magnified 10,000 times for a fourth Test #2, Day-17 filtered sample location. (KerryFeb22F-10k-04)

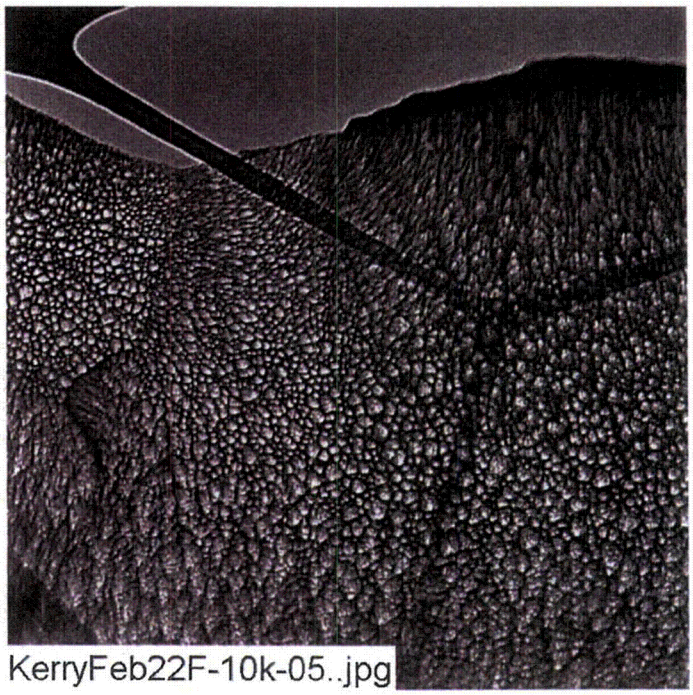


Figure F2-15. Electron micrograph magnified 10,000 times for a fifth Test #2, Day-17 filtered sample location. (KerryFeb22F-10k-05)

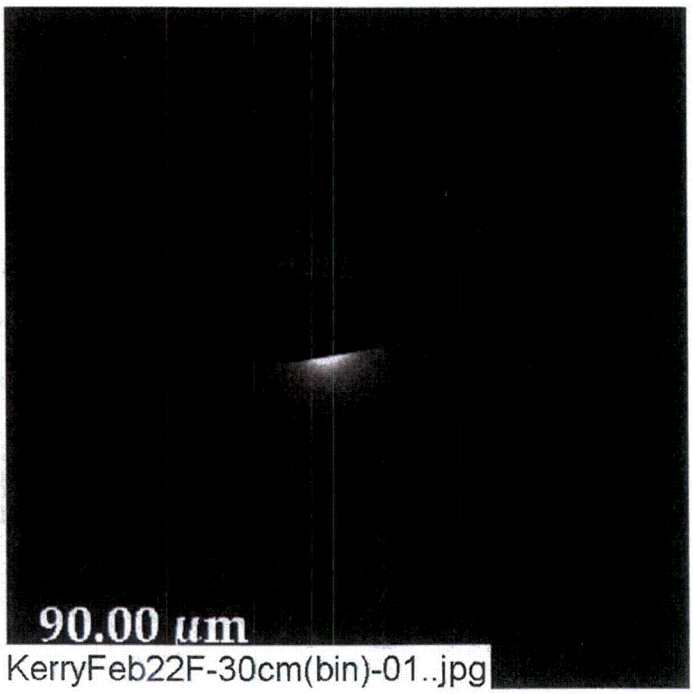


Figure F2-16. TEM image for one Test #2, Day-17 filtered sample solution location. (KerryFeb22F-30cm(bin)-01)

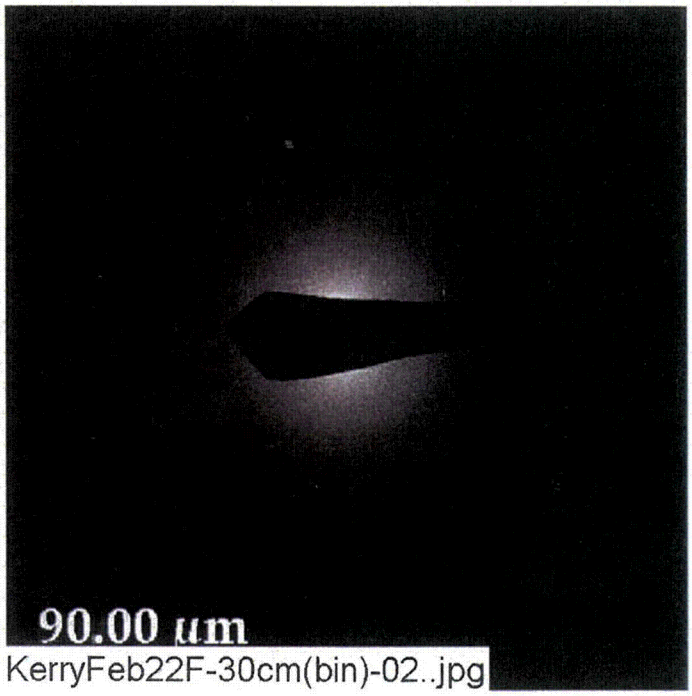


Figure F2-17. TEM image for a second Test #2, Day-17 filtered sample solution location. (KerryFeb22F-30cm(bin)-02)

Figure F2-18. TEM image for a third Test #2, Day-17 filtered sample solution location. (KerryFeb22F-30cm(bin)-03)

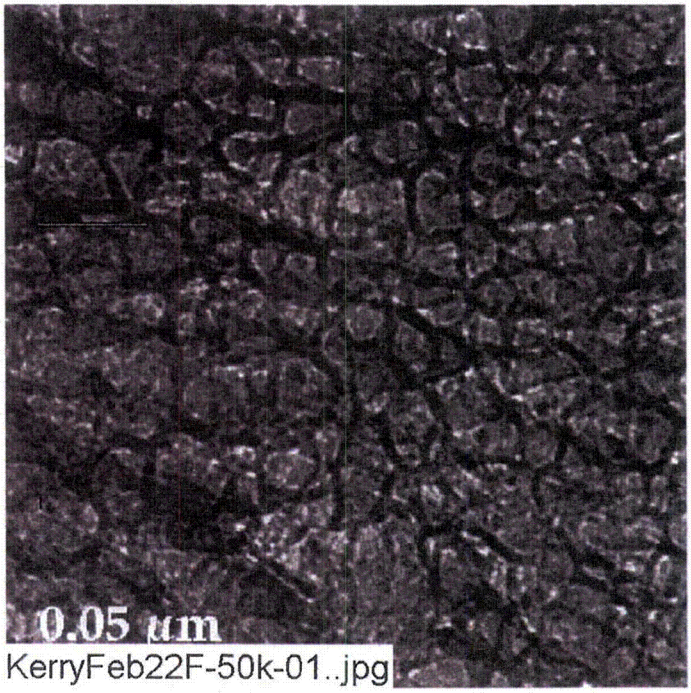


Figure F2-19. Electron micrograph magnified 50,000 times for one Test #2, Day-17 filtered sample location. (KerryFeb22F-50k-01)

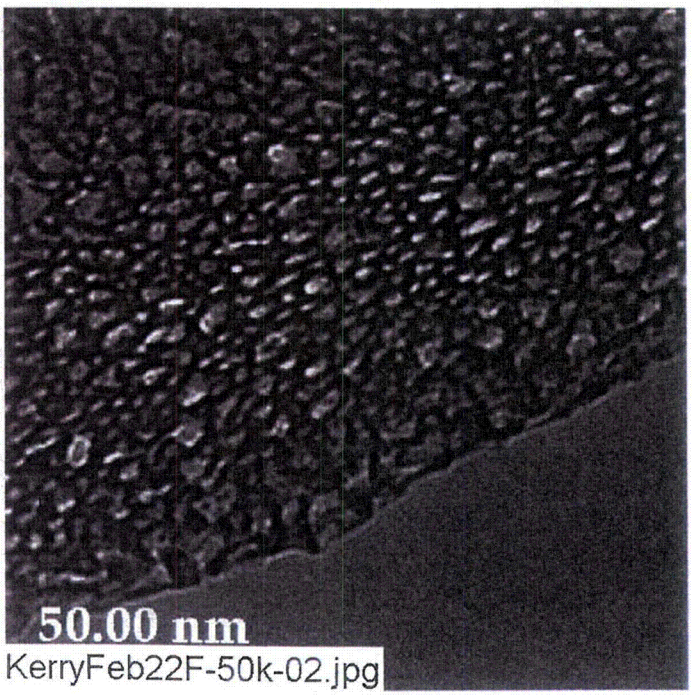


Figure F2-20. Electron micrograph magnified 50,000 times for a second Test #2, Day-17 filtered sample location. (KerryFeb22F-50k-02)

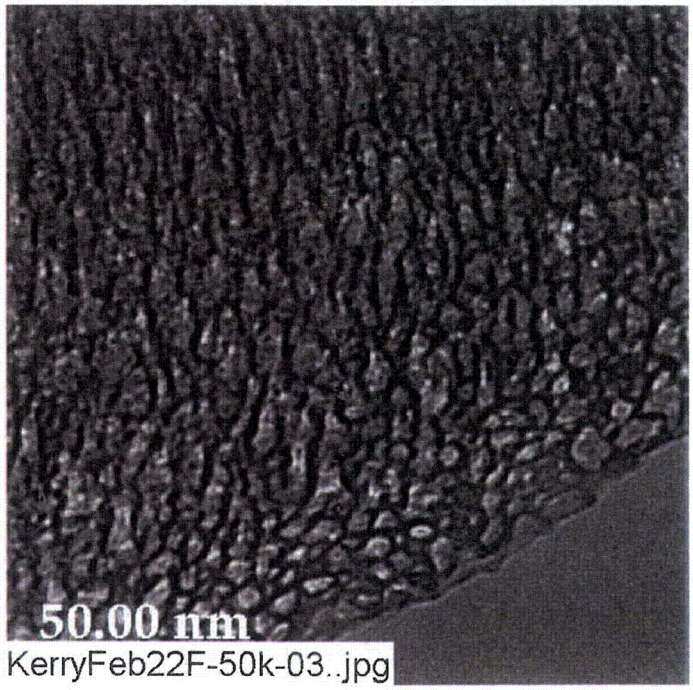


Figure F2-21. Electron micrograph magnified 50,000 times for a third Test #2, Day-17 filtered sample location. (KerryFeb22F-50k-03)

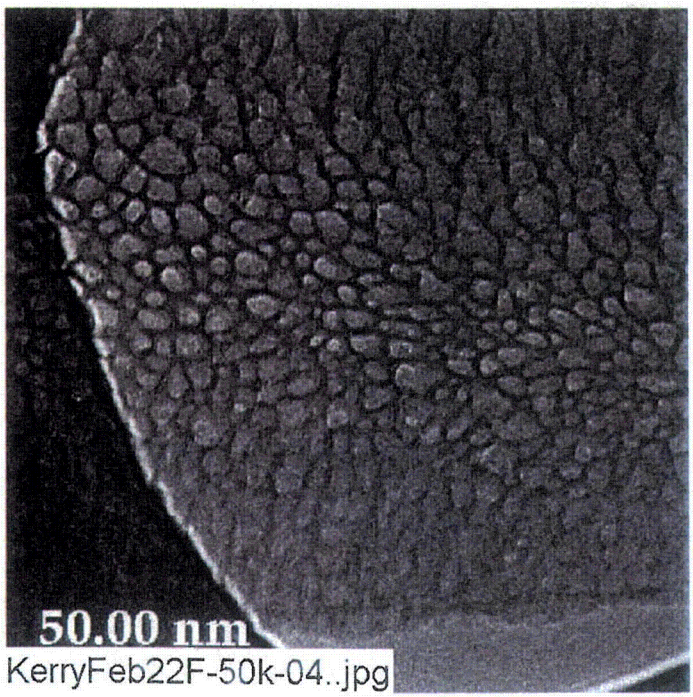


Figure F2-22. Electron micrograph magnified 50,000 times for a fourth Test #2, Day-17 filtered sample location. (KerryFeb22F-50k-04)

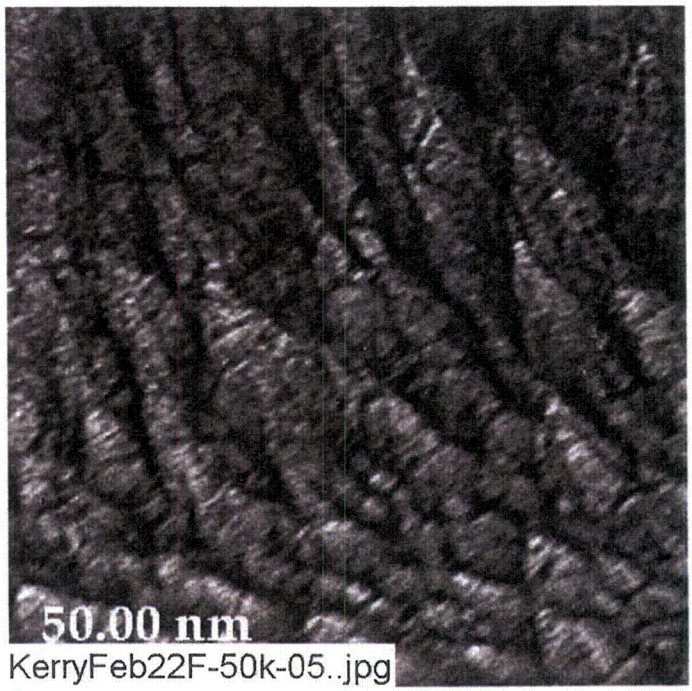


Figure F2-23. Electron micrograph magnified 50,000 times for a fifth Test #2, Day-17 filtered sample location. (KerryFeb22F-50k-05)

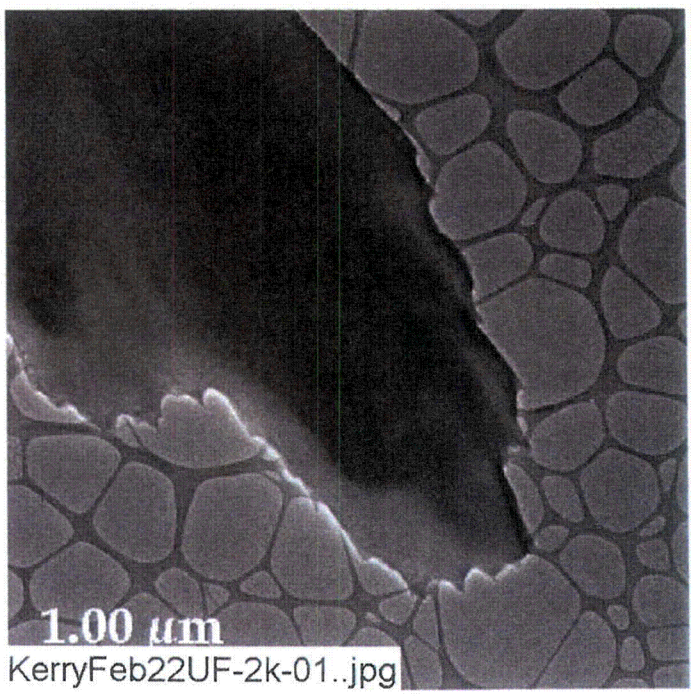


Figure F2-24. Electron micrograph magnified 2000 times for one Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-2k-01)

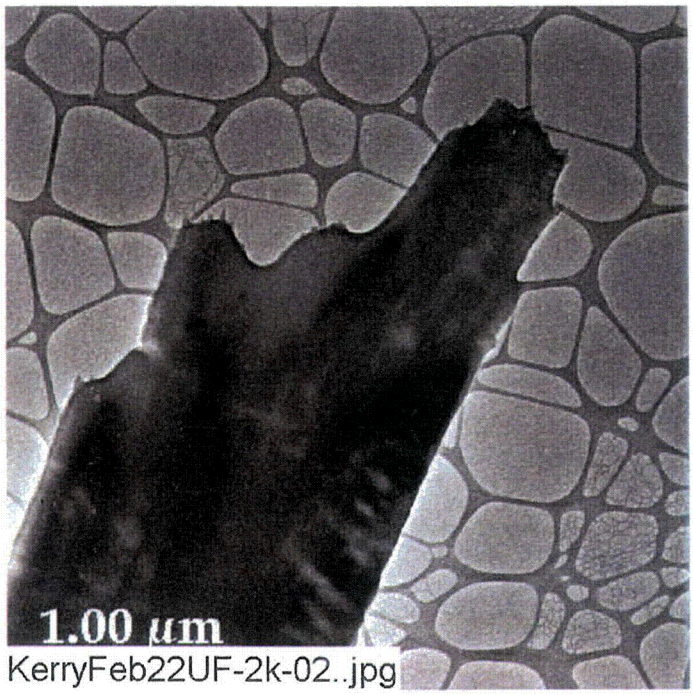


Figure F2-25. Electron micrograph magnified 2000 times for a second Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-2k-02)

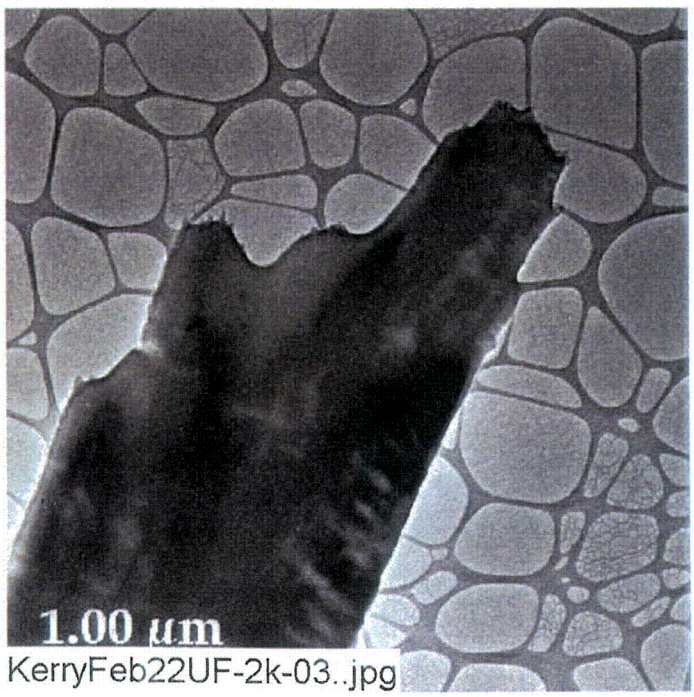


Figure F2-26. Electron micrograph magnified 2000 times for a third Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-2k-03)

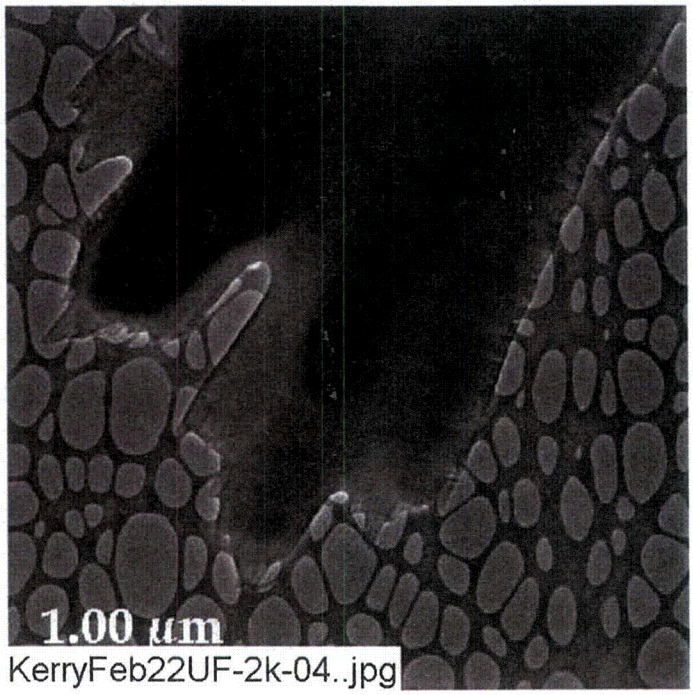


Figure F2-27. Electron micrograph magnified 2000 times for a fourth Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-2k-04)

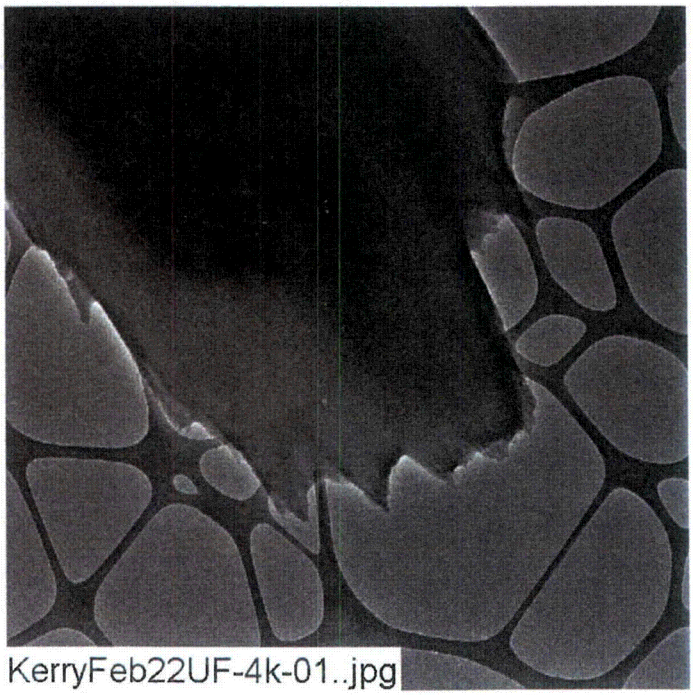


Figure F2-28. Electron micrograph magnified 4000 times for one Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-4k-01)

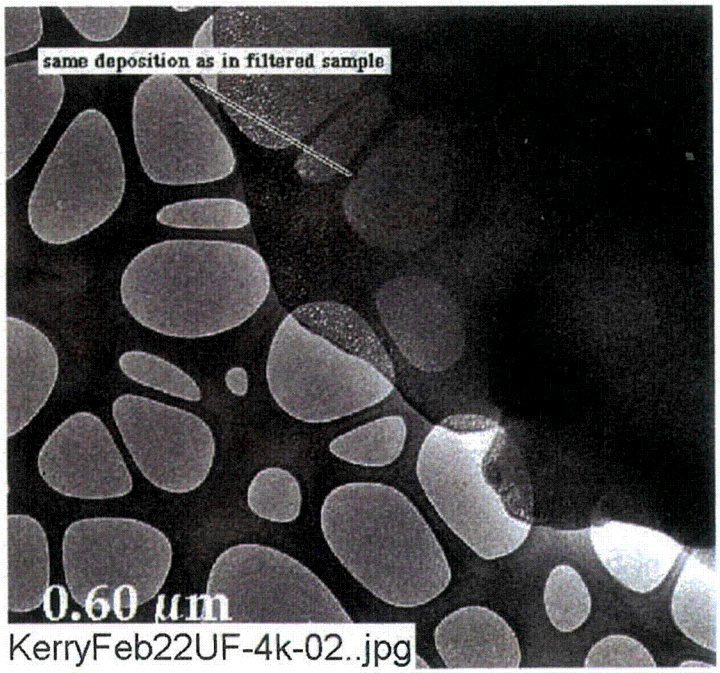


Figure F2-29. Electron micrograph magnified 4000 times for a second Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-4k-02)

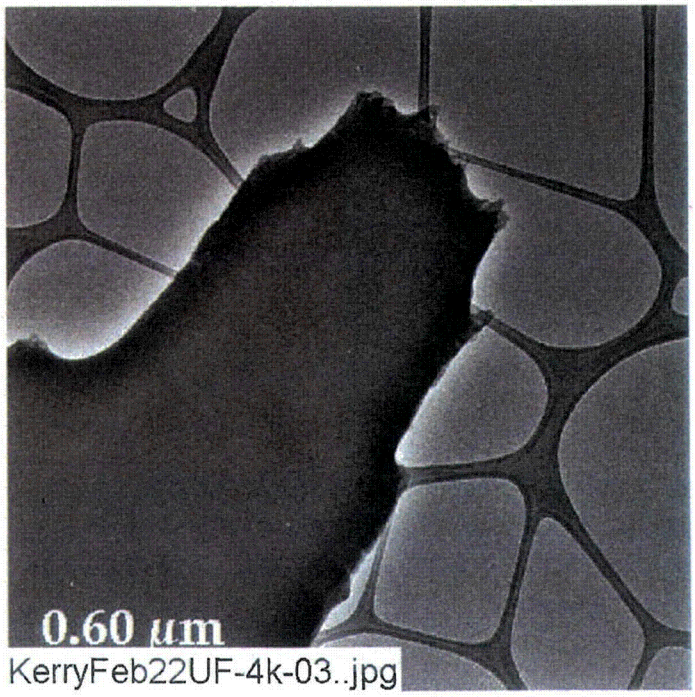


Figure F2-30. Electron micrograph magnified 4000 times for a third Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-4k-03)

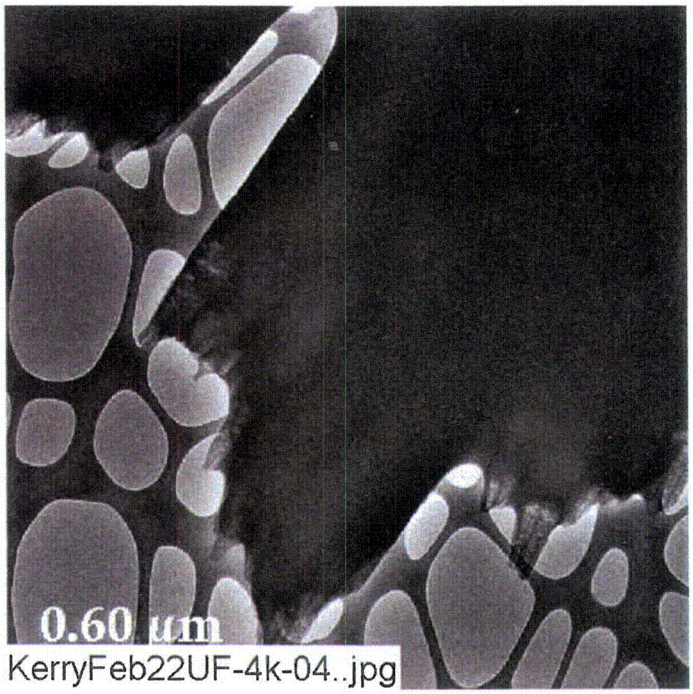


Figure F2-31. Electron micrograph magnified 4000 times for a fourth Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-4k-04)

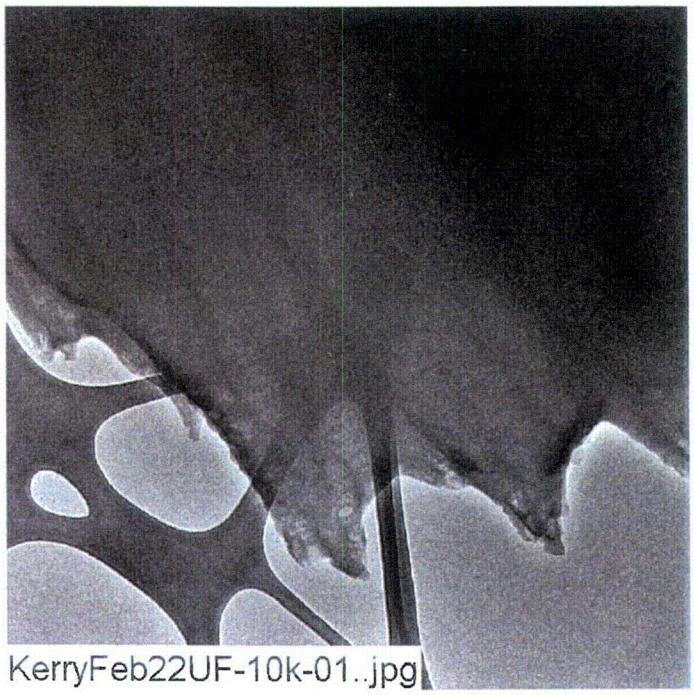


Figure F2-32. Electron micrograph magnified 10,000 times for one Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-10k-01)

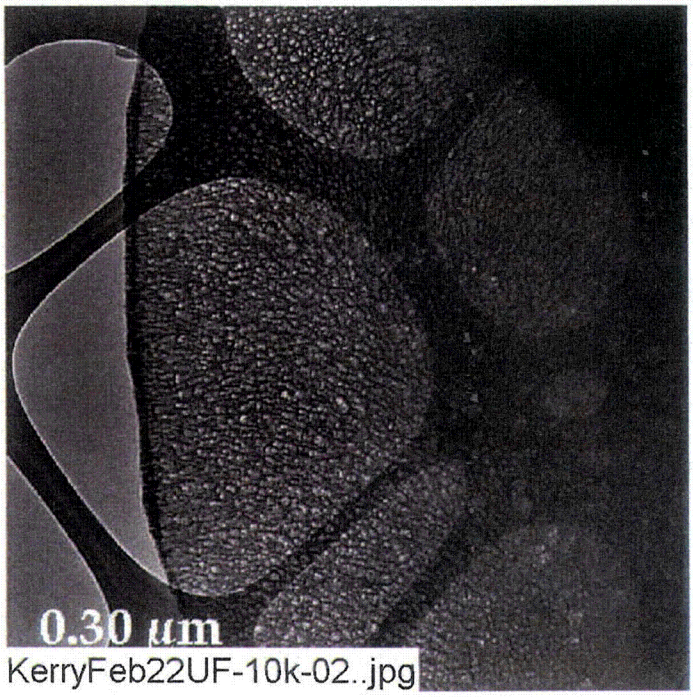


Figure F2-33. Electron micrograph magnified 10,000 times for a second Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-10k-02)

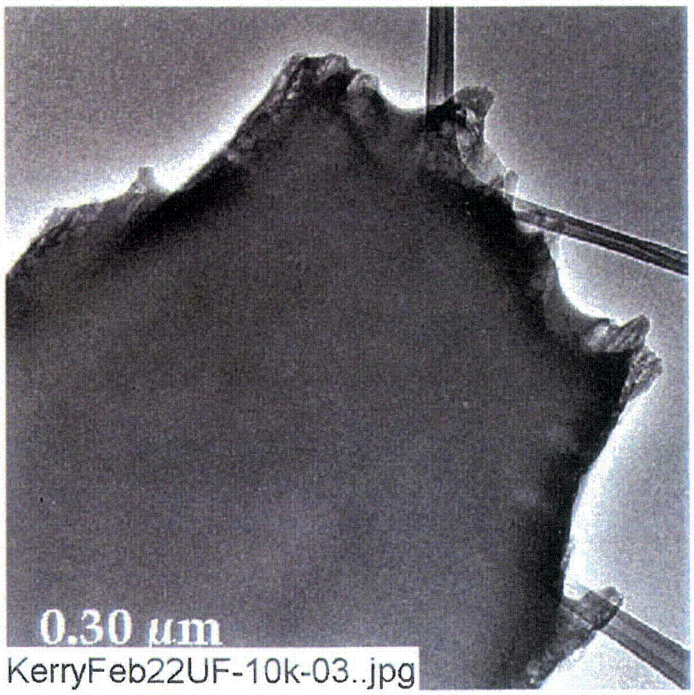


Figure F2-34. Electron micrograph magnified 10,000 times for a third Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-10k-03)

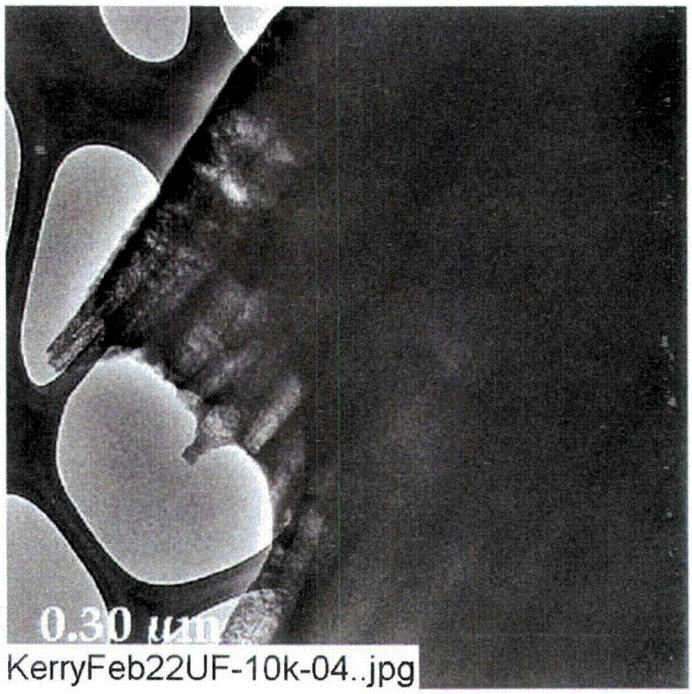


Figure F2-35. Electron micrograph magnified 10,000 times for a fourth Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-10k-04)

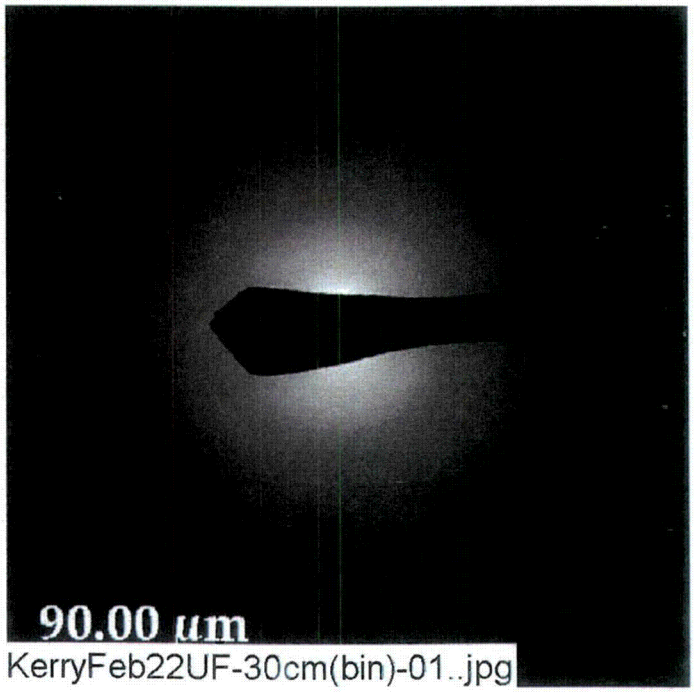


Figure F2-36. TEM image for one Test #2, Day-17 unfiltered sample solution location. (KerryFeb22UF-30cm(bin)-01)

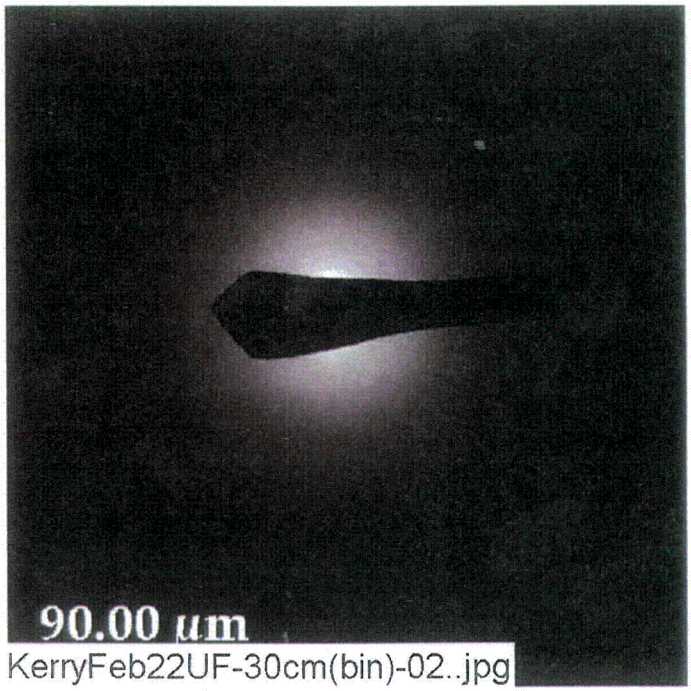


Figure F2-37. TEM image for a second Test #2, Day-17 unfiltered sample solution location. (KerryFeb22UF-30cm(bin)-02)

Figure F2-38. TEM image for a third Test #2, Day-17 unfiltered sample solution location. (KerryFeb22UF-30cm(bin)-03)

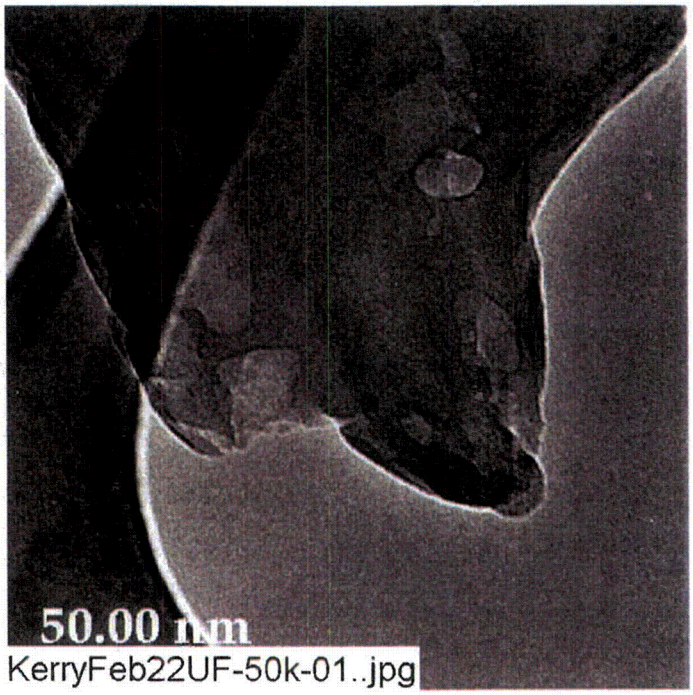


Figure F2-39. Electron micrograph magnified 50,000 times for one Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-50k-01)

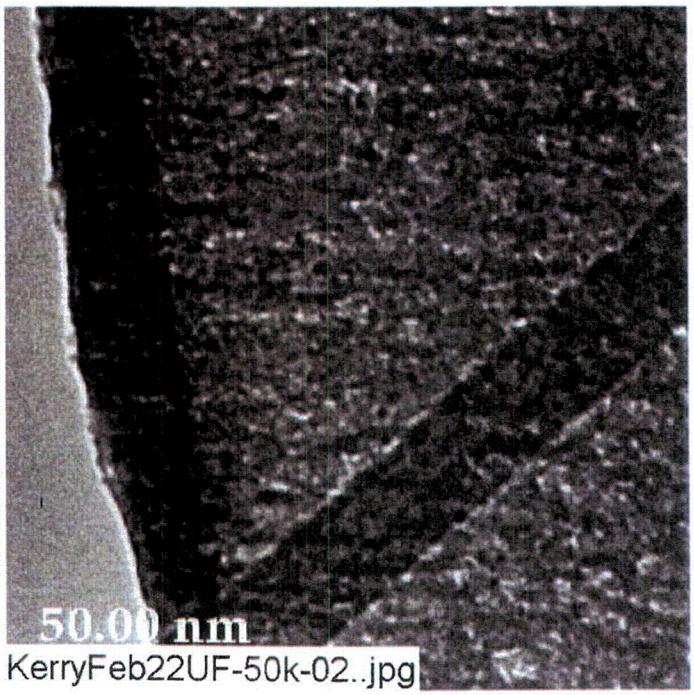


Figure F2-40. Electron micrograph magnified 50,000 times for a second Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-50k-02)

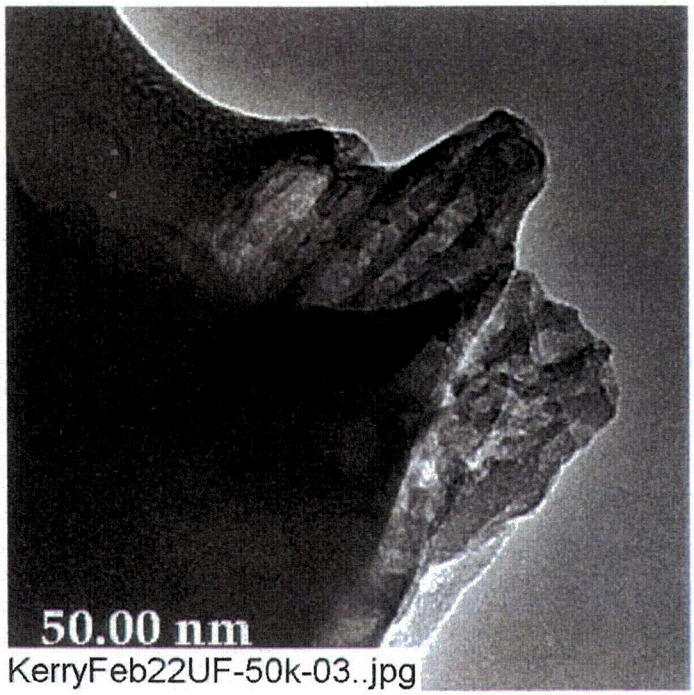


Figure F2-41. Electron micrograph magnified 50,000 times for a third Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-50k-03)

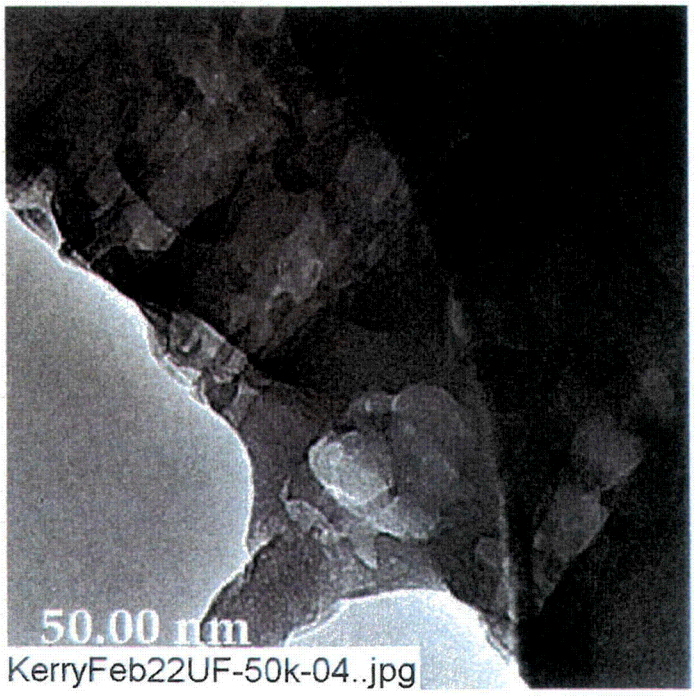


Figure F2-42. Electron micrograph magnified 50,000 times for a fourth Test #2, Day-17 unfiltered sample location. (KerryFeb22UF-50k-04)

Appendix F3

TEM Data for Test #2, Day-30 Filter Samples

Figures

This appendix presents TEM images and diffraction patterns for Test #2, Day-30 (March 7, 2005) filtered and unfiltered solution samples. The filtered samples were obtained by passing solution through a 0.7-μm fiberglass filter at 60°C. The unfiltered solution samples were extracted from the tank directly. A drop of each solution sample was placed onto a copper grid of 200 mesh. After being dried in air at room temperature, the sample was ready for TEM analysis. TEM results and diffraction patterns were obtained on March 7, 2005. Diffraction patterns show whether the sample was amorphous or crystalline. When a sample gives clear and significant diffraction patterns, it is crystalline. Otherwise, it is amorphous. The results show that all of the Test #2, Day-30 samples were amorphous.

Figure F3-1. Electron micrograph magnified 2000 times for one Test #2, Day-30 filtered sample location. (March07F-2k-01)

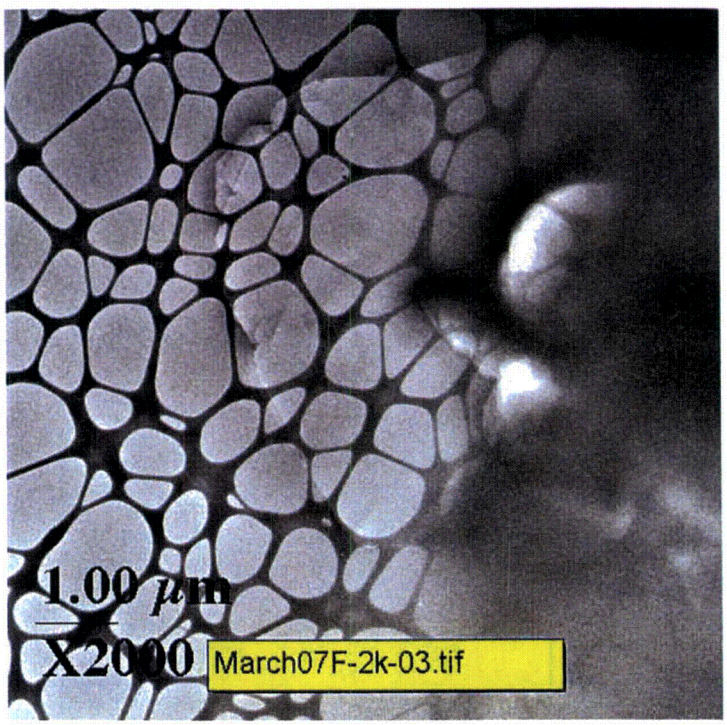


Figure F3-2. Electron micrograph magnified 2000 times for another Test #2, Day-30 filtered sample location. (March07F-2k-03)

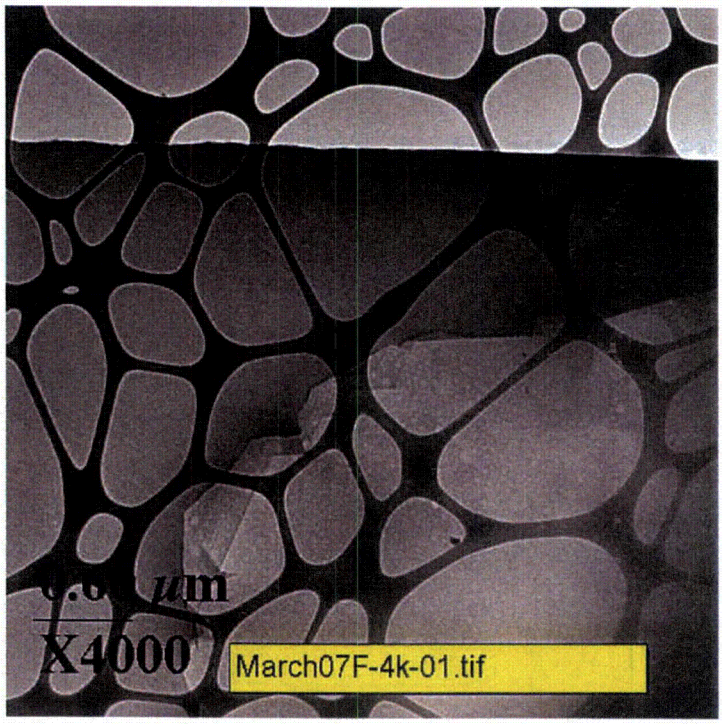


Figure F3-3. Electron micrograph magnified 4000 times for one Test #2, Day-30 filtered sample location. (March07F-4k-01)

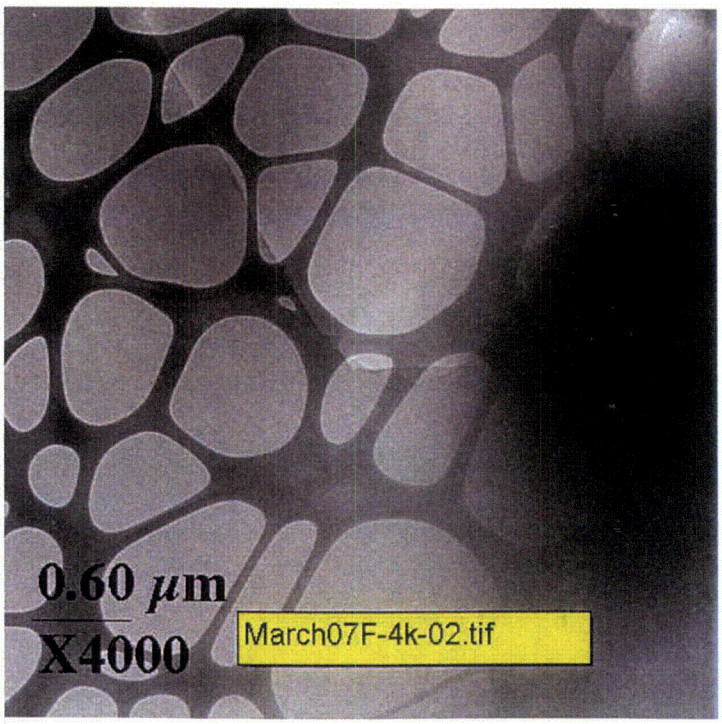


Figure F3-4. Electron micrograph magnified 4000 times for a second Test #2, Day-30 filtered sample location. (March07F-4k-02)

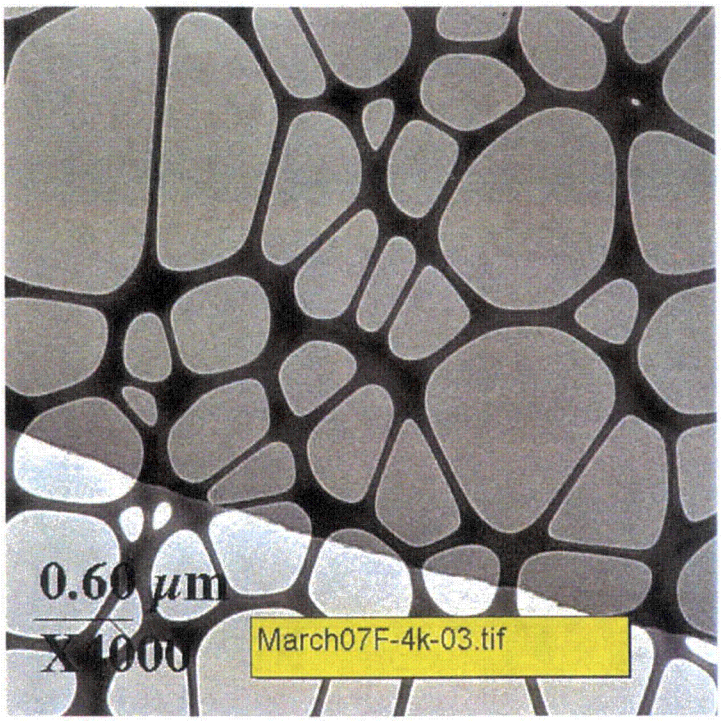


Figure F3-5. Electron micrograph magnified 4000 times for a third Test #2, Day-30 filtered sample location. (March07F-4k-03)

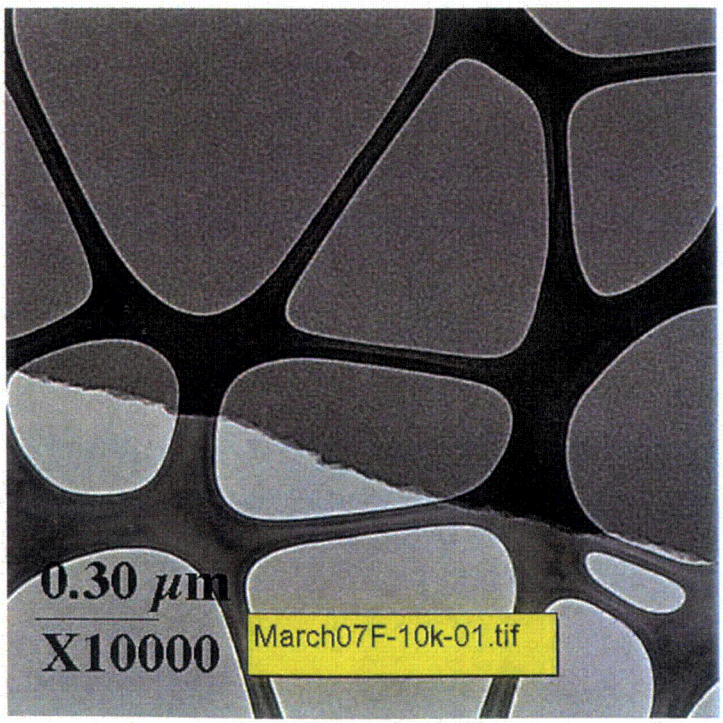


Figure F3-6. Electron micrograph magnified 10,000 times for one Test #2, Day-30 filtered sample location. (March07F-10k-01)

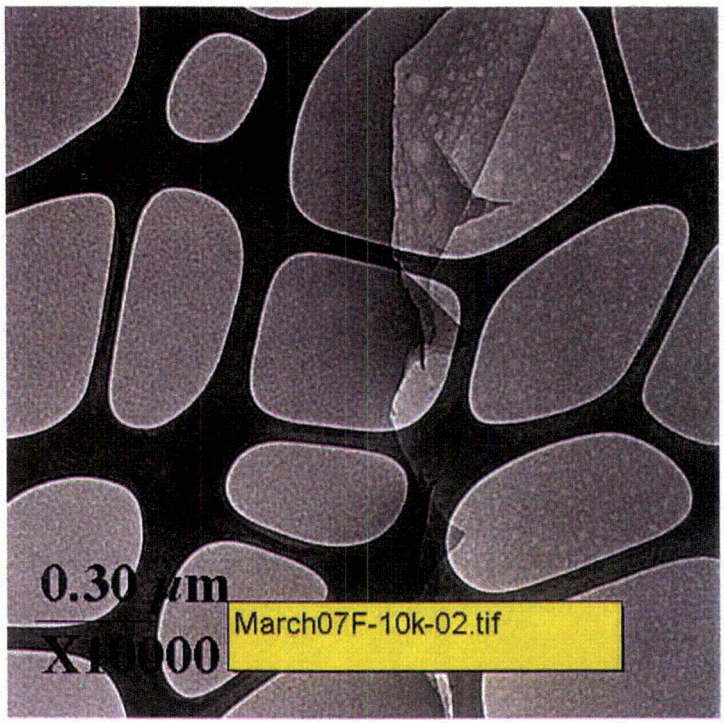


Figure F3-7. Electron micrograph magnified 10,000 times for a second Test #2, Day-30 filtered sample location. (March07F-10k-02)

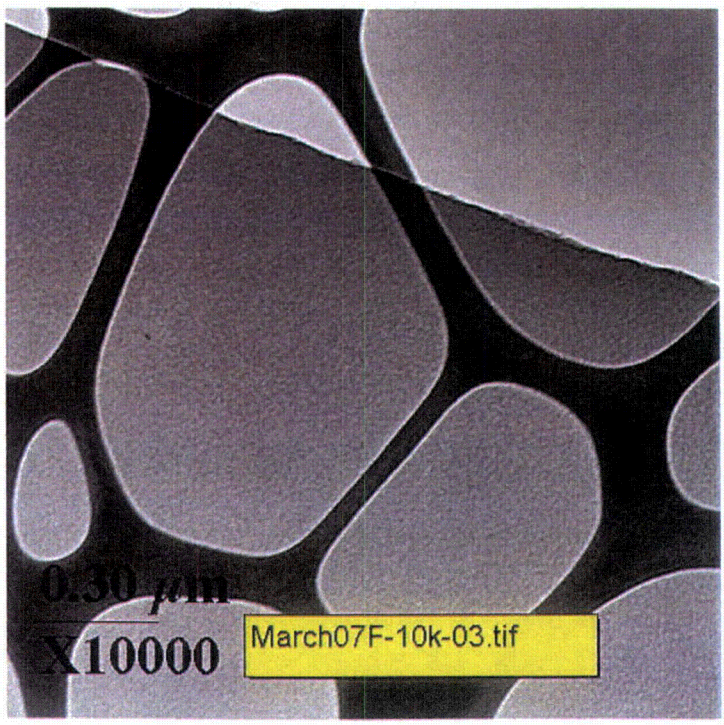


Figure F3-8. Electron micrograph magnified 10,000 times for a third Test #2, Day-30 filtered sample location. (March07F-10k-03)

Figure F3-9. TEM image for a Test #2, Day-30 filtered sample location. (March07F-20cm(bin)-01)

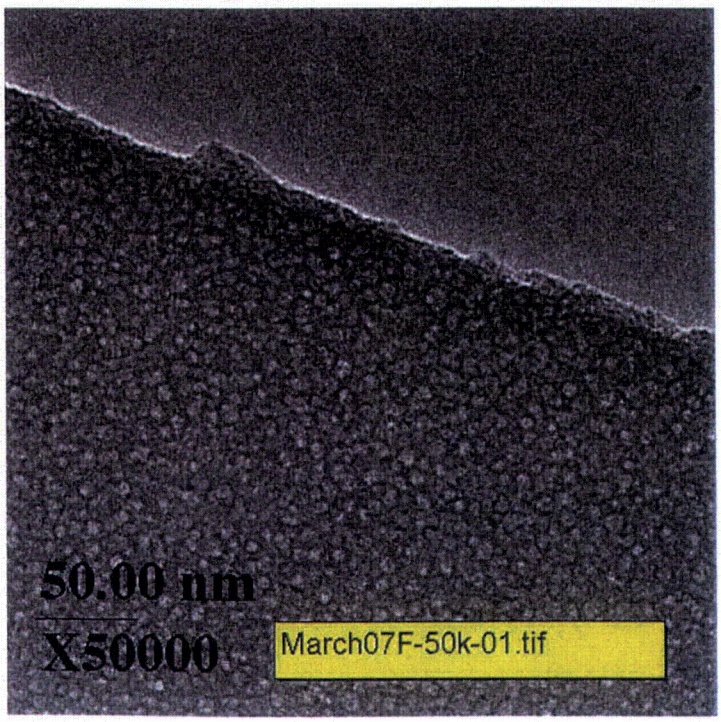


Figure F3-10. Electron micrograph magnified 50,000 times for one Test #2, Day-30 filtered sample location. (March07F-50k-01)

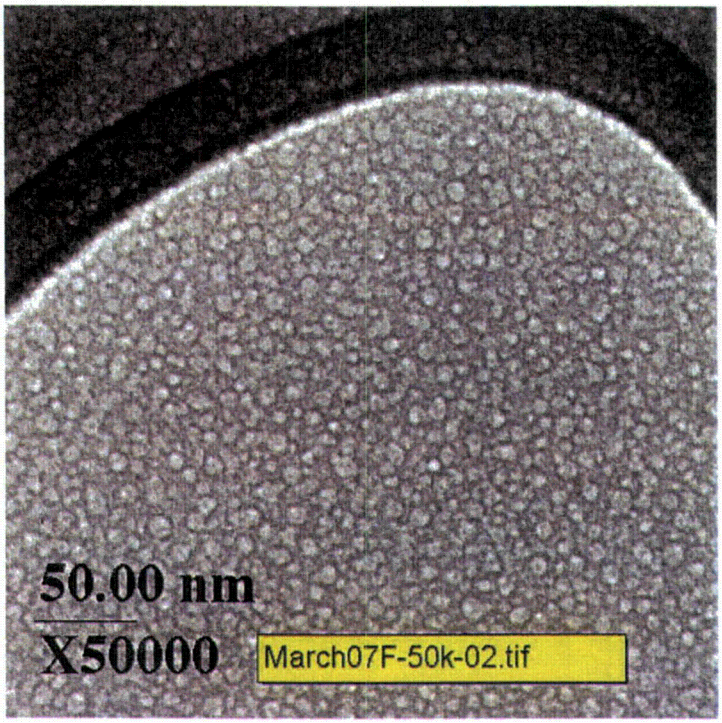


Figure F3-11. Electron micrograph magnified 50,000 times for a second Test #2, Day-30 filtered sample location. (March07F-50k-02)

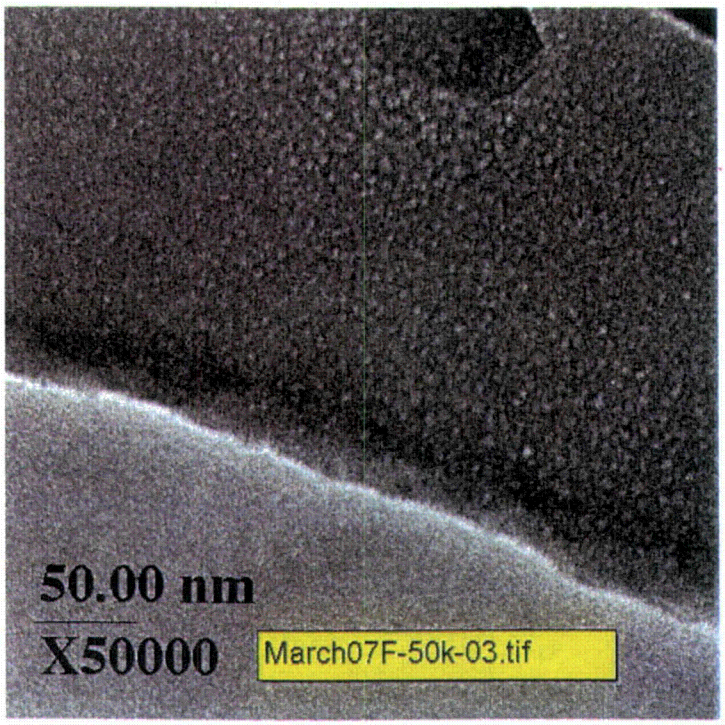


Figure F3-12. Electron micrograph magnified 50,000 times for a third Test #2, Day-30 filtered sample location. (March07F-50k-03)

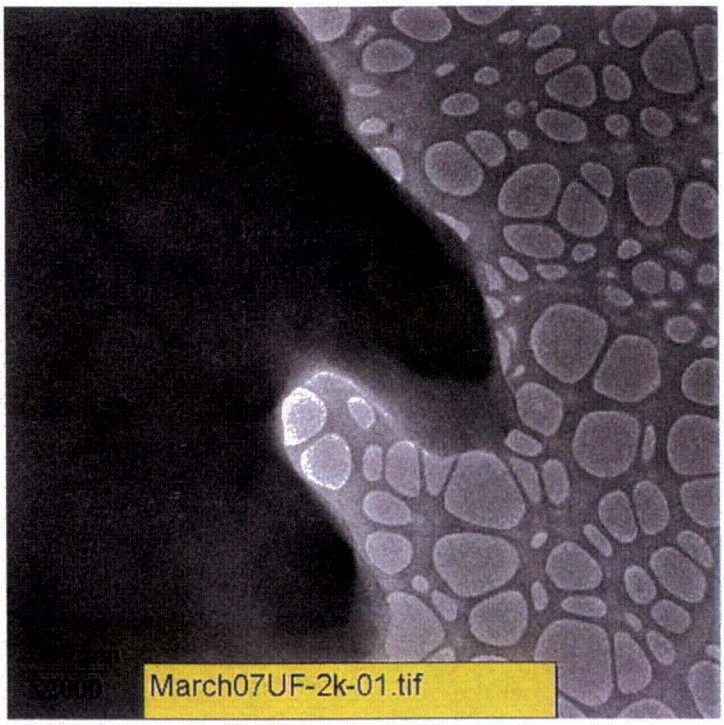


Figure F3-13. Electron micrograph magnified 2000 times for one Test #2, Day-30 unfiltered sample location. (March07UF-2k-01)

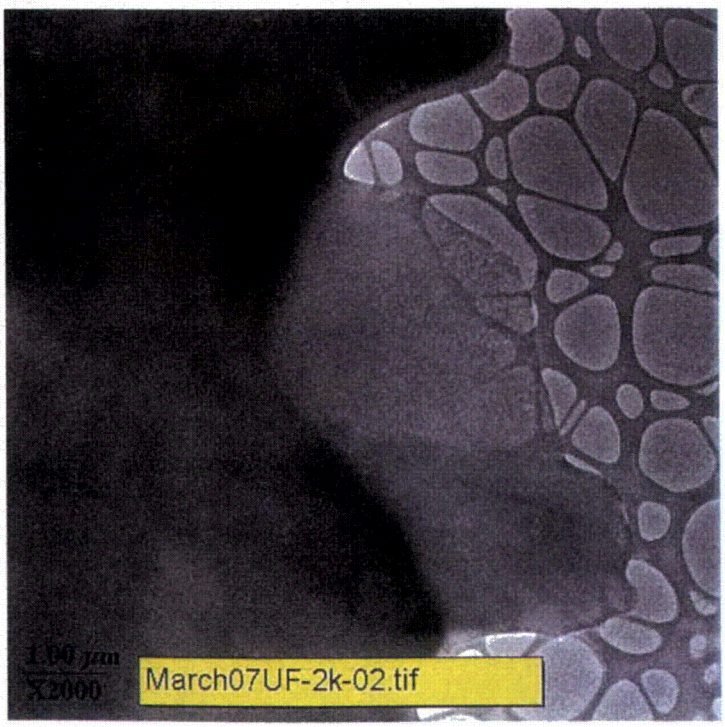


Figure F3-14. Electron micrograph magnified 2000 times for a second Test #2, Day-30 unfiltered sample location. (March07UF-2k-02)

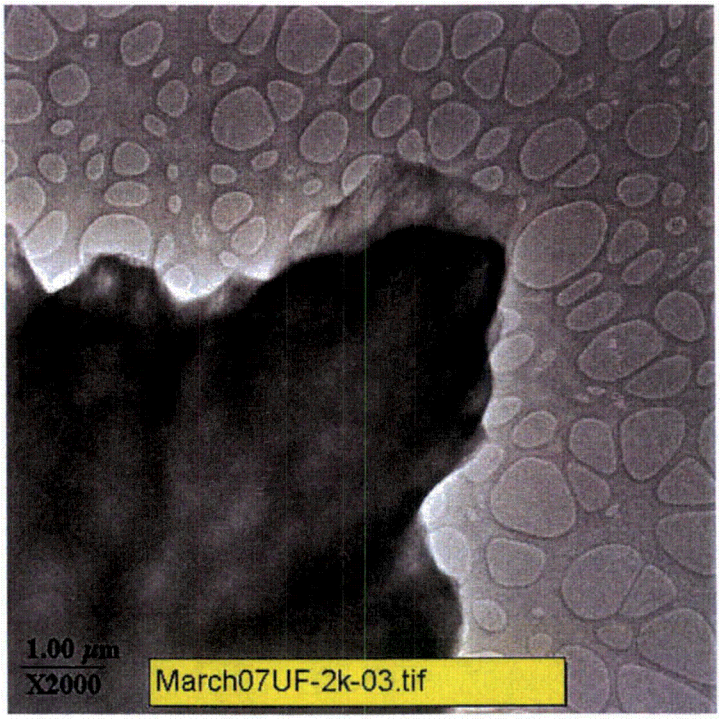


Figure F3-15. Electron micrograph magnified 2000 times for a third Test #2, Day-30 unfiltered sample location. (March07UF-2k-03)

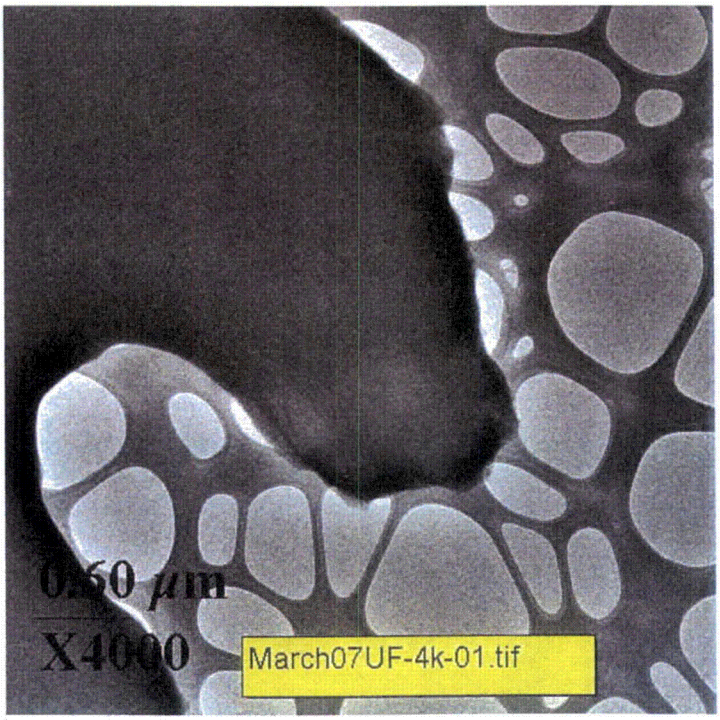


Figure F3-16. Electron micrograph magnified 4000 times for one Test #2, Day-30 unfiltered sample location. (March07UF-4k-01)

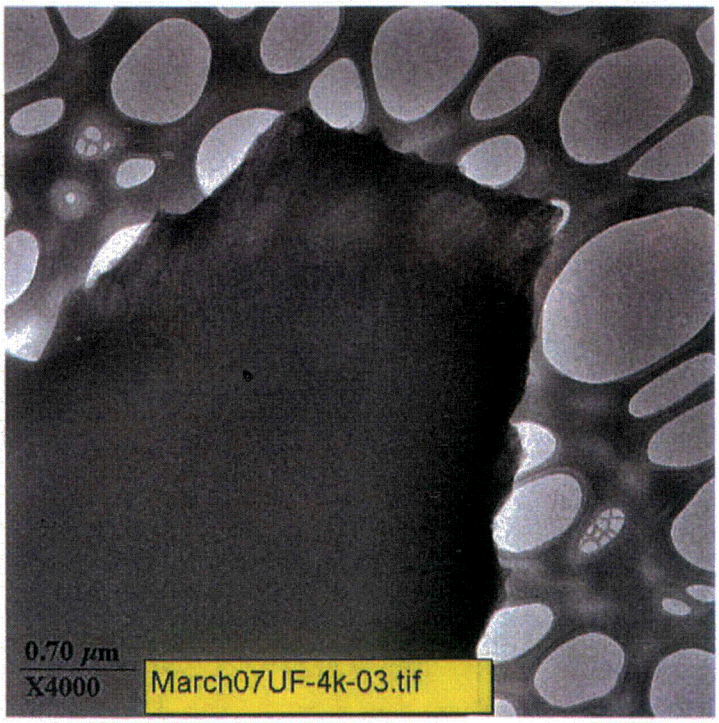


Figure F3-17. Electron micrograph magnified 4000 times for another Test #2, Day-30 unfiltered sample location. (March07UF-4k-03)

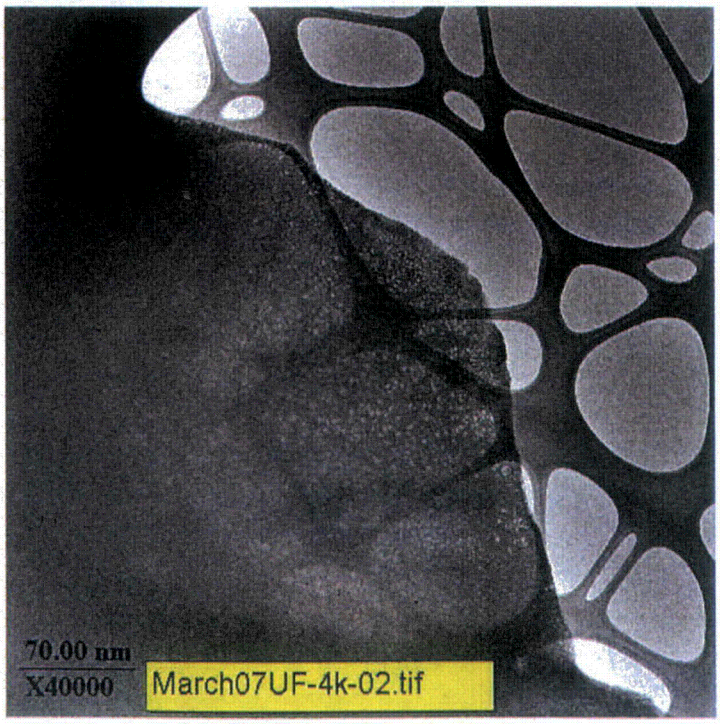


Figure F3-18. Electron micrograph magnified 40,000 times for a Test #2, Day-30 unfiltered sample location. (March07UF-4k-02)

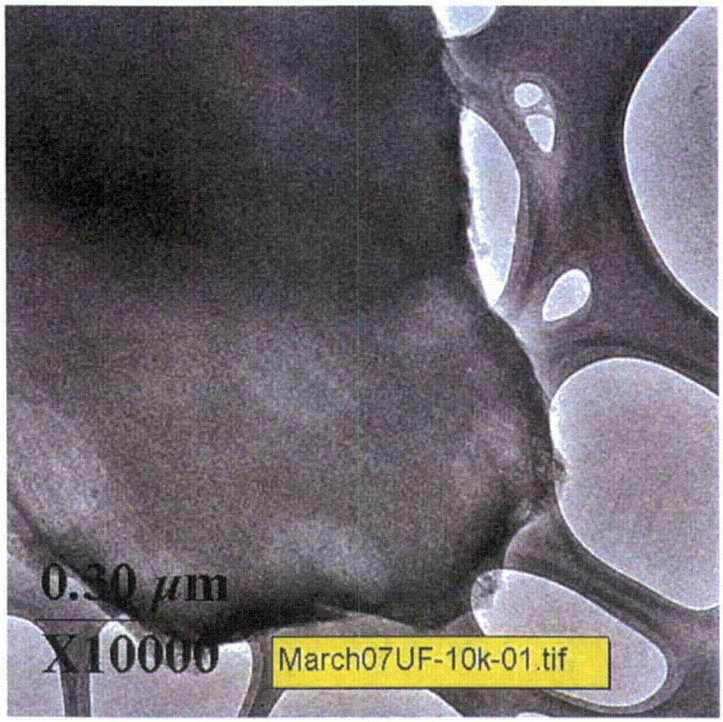


Figure F3-19. Electron micrograph magnified 10,000 times for one Test #2, Day-30 unfiltered sample location. (March07UF-10k-01)

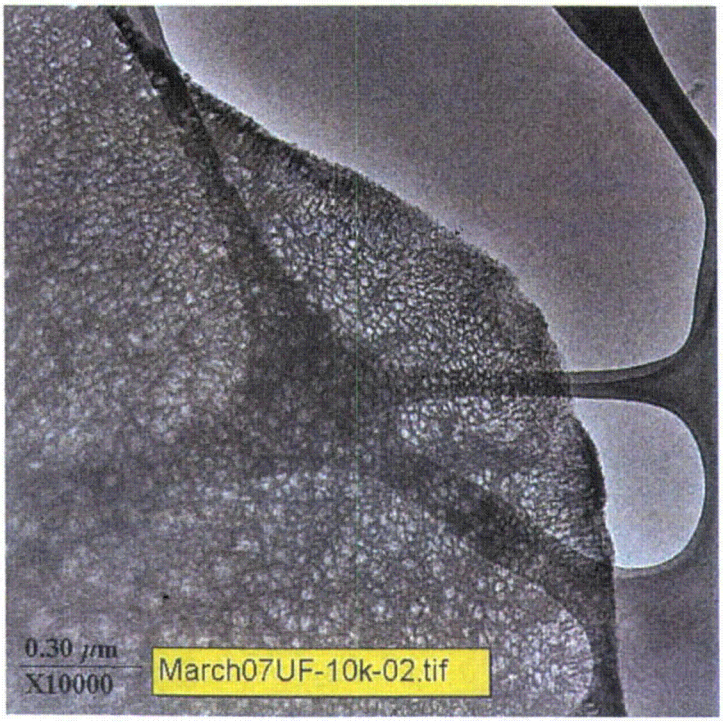


Figure F3-20. Electron micrograph magnified 10,000 times for a second Test #2, Day-30 unfiltered sample location. (March07UF-10k-02)

Figure F3-21. Electron micrograph magnified 10,000 times for a third Test #2, Day-30 unfiltered sample location. (March07UF-10k-03)

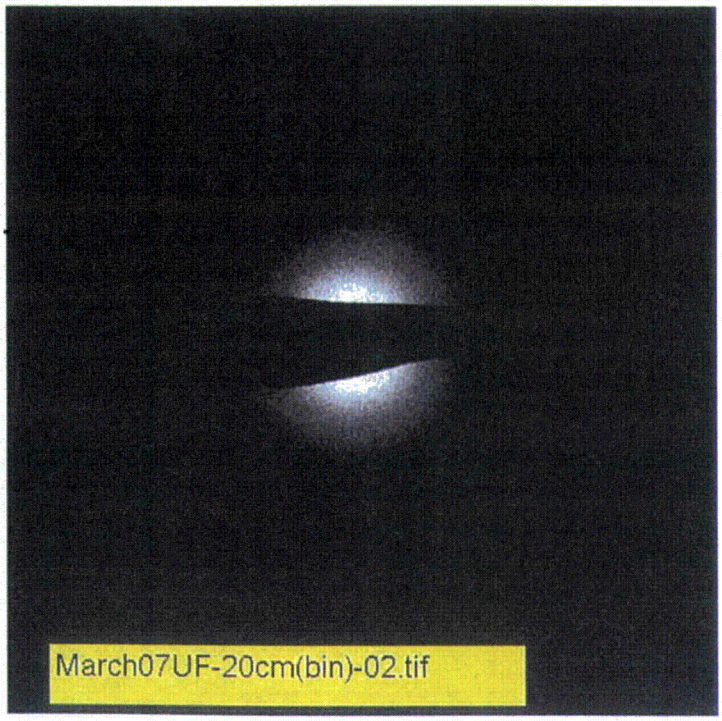


Figure F3-22. TEM image for a Test #2, Day-30 unfiltered sample location. (March07UF-20cm(bin)-02)

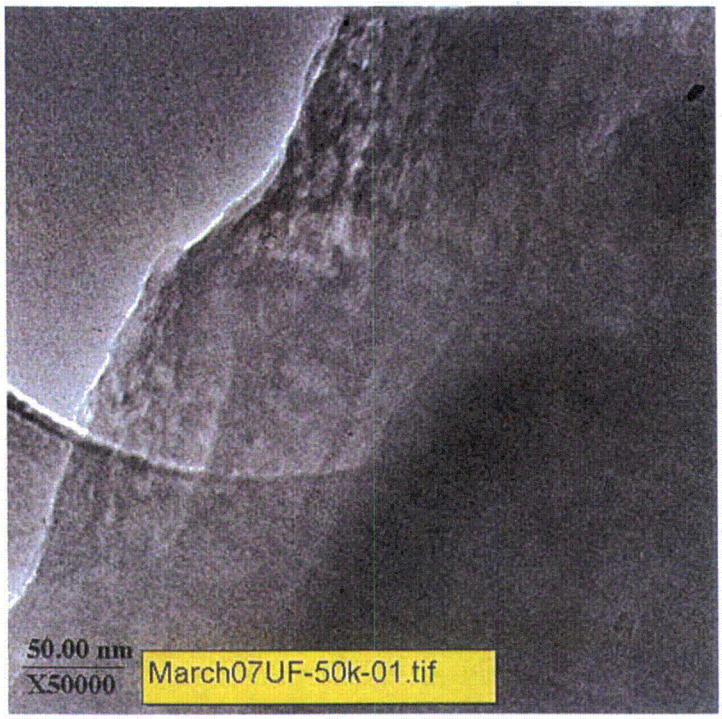


Figure F3-23. Electron micrograph magnified 50,000 times for one Test #2, Day-30 unfiltered sample location. (March07UF-50k-01)

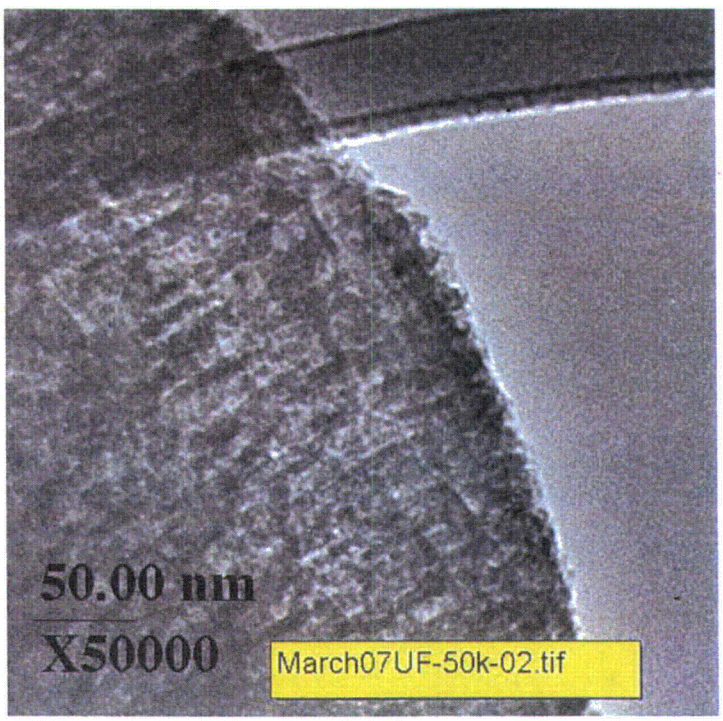


Figure F3-24. Electron micrograph magnified 50,000 times for a second Test #2, Day-30 unfiltered sample location. (March07UF-50k-02)

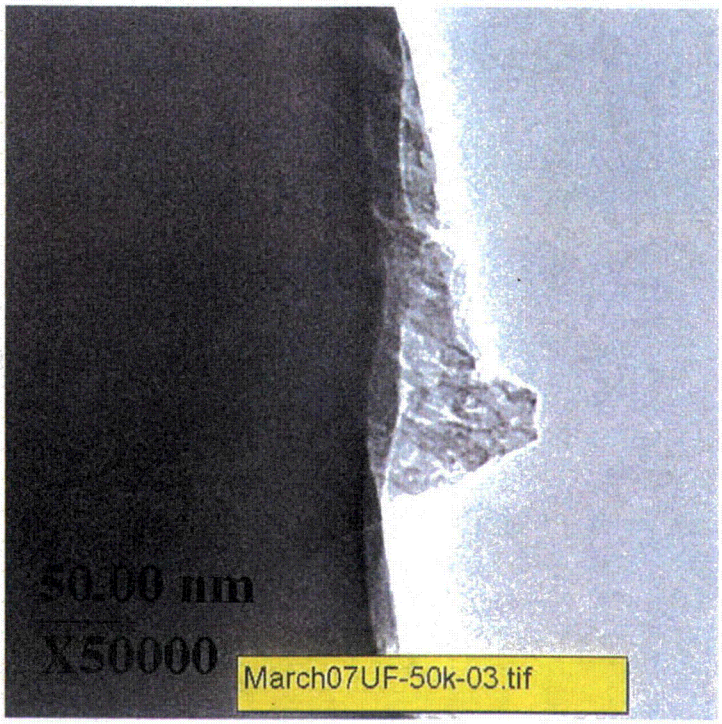


Figure F3-25. Electron micrograph magnified 50,000 times for a third Test #2, Day-30 unfiltered sample location. (March07UF-50k-03)

Appendix G

Test #2 Total Organic Carbon (TOC) Concentration

Tables

This appendix presents the total organic carbon (TOC) levels, which reflect the organic concentration in the Test #2 solution. The organic matter in the solution likely originated from the binding material of the fiberglass used in the tests. These organic materials may potentially become involved in the complexation process with metal, concrete coupons, and fiberglass during ICET tests. The TOC was measured using a TOC analyzer with a UV-persulfate oxidation method.

Potassium hydrogen phthalate with a purity of 99.95% was used to make a standard TOC solution for calibration. The TOC analyzer was calibrated using a five-point calibration curve up to 20 ppm. During measurement of the actual test samples, a TOC standard solution of 10 mg/L was analyzed again to ensure that the instrument was in good condition. The solution samples were extracted on Day 0, Day 15, and Day 30 of Test #2. The samples were prefiltered through a 0.7-μm fiberglass filter at 60°C to remove particulate impurities. The TOC measurements were performed on April 25, 2005. Based on the results, the TOC in the solution generally increased throughout the duration of the test. A substantial TOC increase was found in the Day-15 samples compared with that found in the Day-0 sample. However, only a slight increase in the TOC was observed from the Day-15 to the Day-30 sample.

Table G-1. Total Organic Carbon Results for Test #2 (mg/L)

Test 2	Day 0	Day 15	Day 30
	0.2496	7.2464	7.9276
	0.1332	7.3696	7.8712
	0.102	7.42	7.9096
Mean	0.1616	7.3453	7.9028
Std Dev	0.0778	0.0893	0.0288

Table G-2. Potassium Hydrogen Phthalate Standard TOC Solution

Control (10 ppm standard solution)
10.0511
9.9825
9.995
Mean 10.0095
Std Dev 0.0365

Appendix H

UV Absorbance Spectrum—Day-30 Solution Sample

Figures

Tables

This appendix presents the UV absorbance result of the Test #2, Day-30 solution sample. The purpose of this analysis was to find any distinguishing absorbance peaks that might help to identify organics present in the solution. The solution sample was collected at 60°C through a 0.7-μm fiberglass filter to remove particulate impurities, followed by being scanned by a UV-visible spectrophotometer over wavelengths ranging from 200 to 800 nm. The spectrum of deionized water was used for background subtraction. The test results revealed no distinguishing absorbance peaks that identify organics in the test solution.

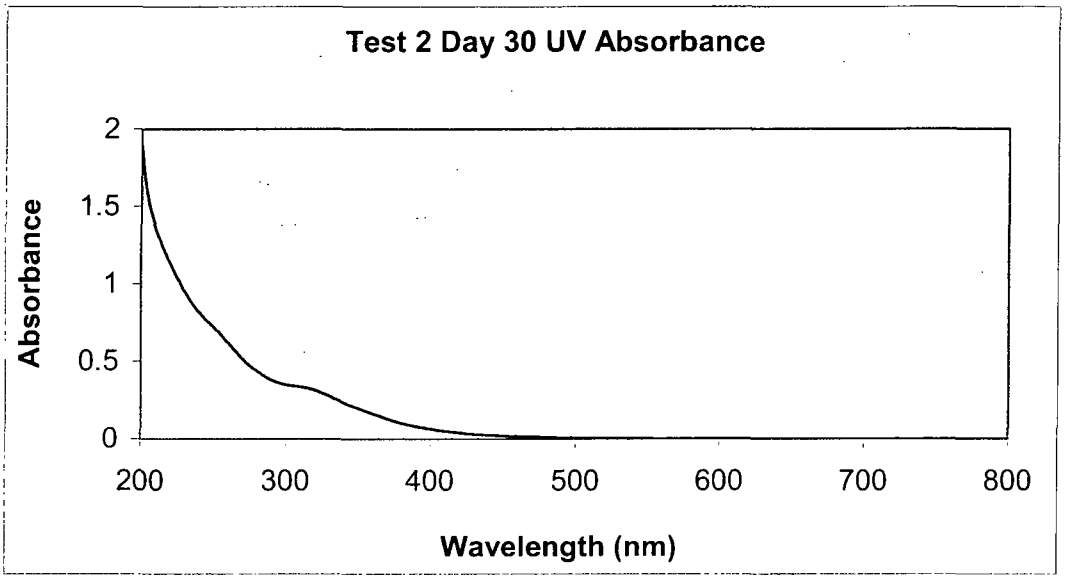


Figure H-1. UV absorbance spectrum for Day-30 solution samples.

Table H-1. Test #2, Day-30 Solution-Sample UV-Absorbance Laboratory Settings

	Test #2, Day 30
Collection Time:	4/21/2005 1:58:32 PM
Operator Name:	
Scan Software Version:	3.00(182)
Parameter List:	
Instrument:	Cary 50
Instrument Version:	3.00
Start (nm):	800.0
Stop (nm):	200.0
X Mode:	Nanometers
Y Mode:	Abs
UV-Vis Scan Rate (nm/min):	600.00
UV-Vis Data Interval (nm):	1.00
UV-Vis Ave. Time (sec):	0.1000
Beam Mode:	Dual Beam
Baseline Correction:	On
Baseline Type:	Baseline correction
Baseline File Name:	
Baseline Std Ref File Name:	
Cycle Mode:	Off
Comments:	
Method Log:	
Method Name:	Default
Date/Time stamp:	4/21/2005 1:46:30 PM
Method Modifications:	
Cell Changer 6 × 6 Changed:	4/21/2005 1:46:34 PM / Old:1 / New:0
UVVIS SAT Changed:	4/21/2005 1:46:59 PM / Old:0.0125 / New:0.1000
NIR SAT Changed:	4/21/2005 1:46:59 PM / Old:0.0125 / New:0.1000
Common SAT Changed:	4/21/2005 1:46:59 PM / Old:0.0125 / New:0.1000
Baseline Correction Changed:	4/21/2005 1:47:42 PM / Old:0 / New:1
Temp Controller Changed:	4/21/2005 1:47:42 PM / Old:0 / New:2
Sipper Type Changed:	4/21/2005 1:47:42 PM / Old:Internal RSA / New:External sipper
End Method Modifications	
<Current Wavelength>	200.1

Appendix I

XRD and XRF Data for Test #2, Day-30 Sediment and Fiberglass in Birdcage

Figures

Tables

This appendix shows XRD and XRF results for Test #2, Day-30 sediment and submerged birdcage fiberglass samples. XRD analysis provided the identity of the existing crystalline materials in the samples, and XRF gave the elemental composition of the samples. The purpose of these analyses is to provide the morphology and the composition of the sediment and the fiberglass samples to understand the chemical reactions that may have occurred during the ICET tests.

The sediment and the birdcage fiberglass samples were collected from the tank on the date that Test #2 was terminated (March 7, 2005). These samples were dried in air at room temperature. XRD and XRF analyses were performed on April 26 and April 28, 2005, respectively. Based on XRD results, the Test #2, Day-30 sediment sample contained some amount of quartz. However, the Test #2, Day-30 birdcage fiberglass sample was mostly amorphous. XRF results show that both the sediment and the fiberglass contained significant amounts of silicon.

Table I-1. XRF Results for Test-2 Day-30 Sediment and Birdcage Fiberglass

Project name:- ICP - XRF Conversion (LANL)

Instrument:- XRF

Analyst:- Mehdi & Blake

Analysis Date:- 4/28/05 Sample ID: Test #2, Day-30 sediment and Test #2, Day-30 fiberglass in birdcage

Sample ID	Ratio Si/SiO_2	XRF (%) SiO_2	ICP (%) Si	ICP (mg/kg) Si
Test #2 Sediment	0.467	37.38	17.4	174440
T2D30 Fiberglass in Birdcage	0.467	50.42	23.5	235293
Sample ID	Ratio Al/Al_2O_3	XRF (%) Al_2O_3	ICP (%) Al	ICP (mg/kg) Al
Test #2 Sediment	0.529	16.06	8.50	85024
T2D30 Fiberglass in Birdcage	0.529	4.6	2.44	24353
Sample ID	Ratio P/P_2O_5	XRF (%) P_2O_5	ICP (%) P	ICP (mg/kg) P
Test #2 Sediment	0.437	10.01	4.37	43697
T2D30 Fiberglass in Birdcage	0.437	2.78	1.21	12136
Sample ID	Ratio Na/Na_2O	XRF (%) Na_2O	ICP (%) Na	ICP (mg/L) Na
Test #2 Sediment	0.742	3.96	2.94	29382
T2D30 Fiberglass in Birdcage	0.742	12.29	9.12	91188
Sample ID	Ratio Ca/CaO	XRF (%) CaO	ICP (%) Ca	ICP (mg/kg) Ca
Test #2 Sediment	0.714	5.08	3.63	36286
T2D30 Fiberglass in Birdcage	0.714	6.83	4.88	48786
Sample ID	Ratio Mn/MnO	XRF (%) MnO	ICP (%) Mn	ICP (mg/kg) Mn
Test #2 Sediment	0.775	0.110	0.085	852
T2D30 Fiberglass in Birdcage	0.775	0.030	0.023	232

Sample ID	Ratio Mg/MgO	XRF (%) MgO	ICP (%) Mg	ICP (mg/kg) Mg
Test #2 Sediment	0.603	1.42	0.857	8566
T2D30 Fiberglass in Birdcage	0.603	2.62	1.580	15805

Sample ID	Ratio K/K_2O	XRF (%) K_2O	ICP (%) K	ICP (mg/kg) K
Test #2 Sediment	0.830	0.720	0.598	5978
T2D30 Fiberglass in Birdcage	0.830	0.570	0.473	4732

Sample ID	Ratio Fe/Fe_2O_3	XRF (%) Fe_2O_3	ICP (%) Fe	ICP (mg/kg) Fe
Test #2 Sediment	0.700	0.780	0.546	5457
T2D30 Fiberglass in Birdcage	0.700	0.550	0.385	3848

Sample ID	Ratio Ti/TiO_2	XRF (%) TiO_2	ICP (%) Ti	ICP (mg/kg) Ti
Test #2 Sediment	0.600	0.100	0.060	600
T2D30 Fiberglass in Birdcage	0.600	0.030	0.018	180

Sample ID	$H_2O(-)$	$H_2O(+)CO_2$	Total (%)
Test #2 Sediment	5.73	21.1	102
T2D30 Fiberglass in Birdcage	3.26	11.2	95

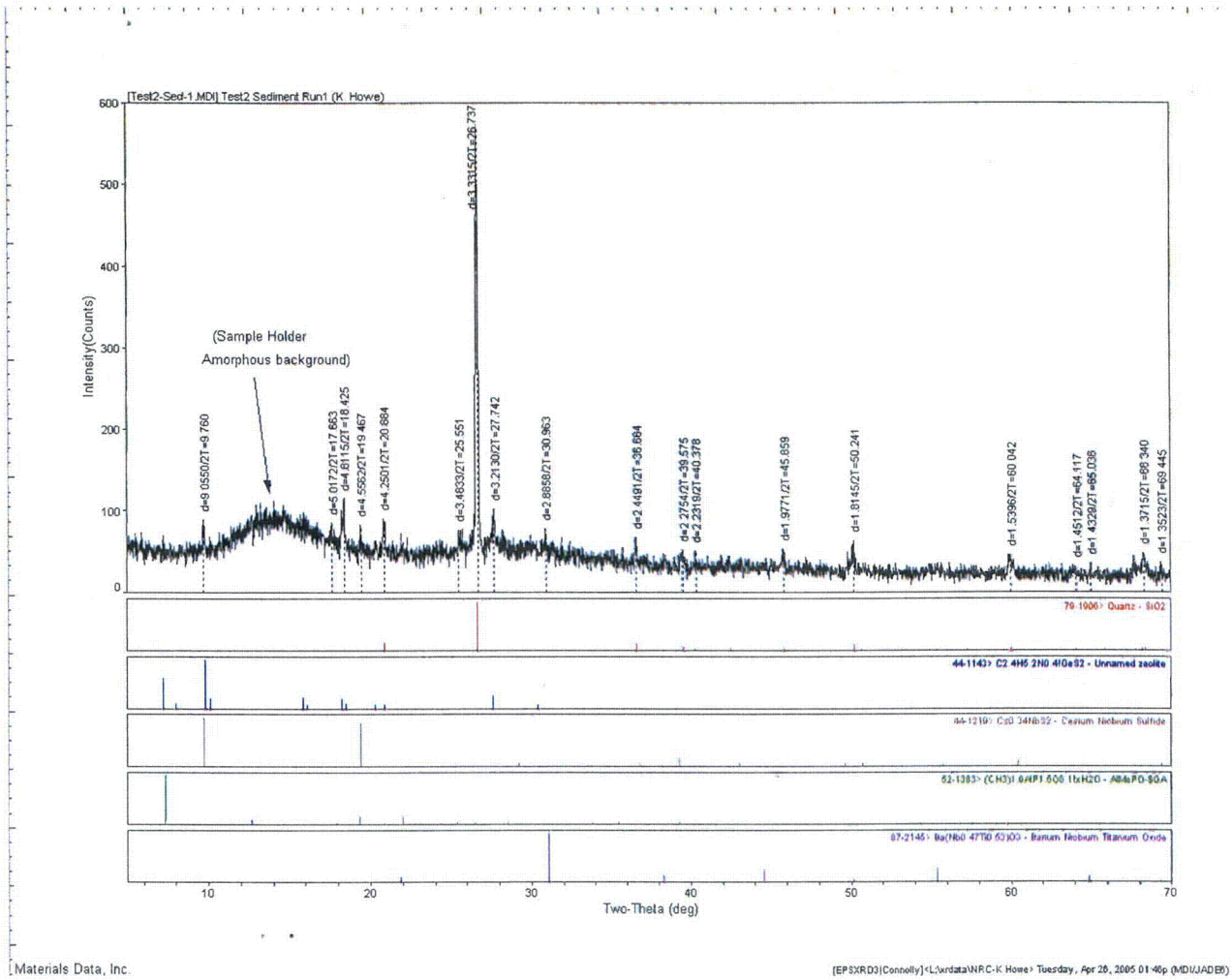


Figure I-1. XRD results for Test #2, Day-30 sediment.

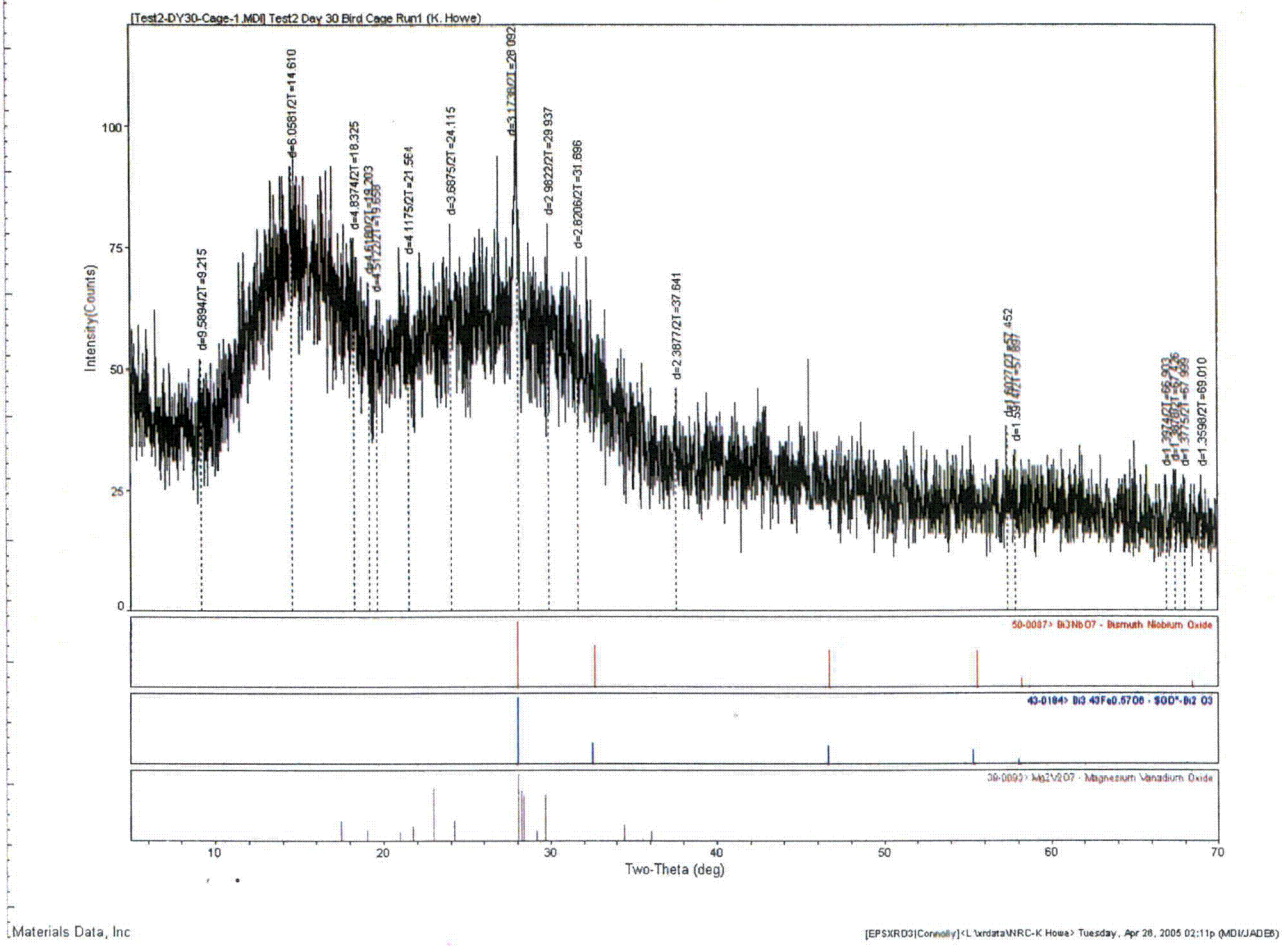


Figure I-2. XRD results for the Test #2, Day-30, Cage-1 match.

Appendix J

ESEM and SEM/EDS Data for Test #2, Day-4 Filtrate and Fiberglass Samples

Figures

Tables

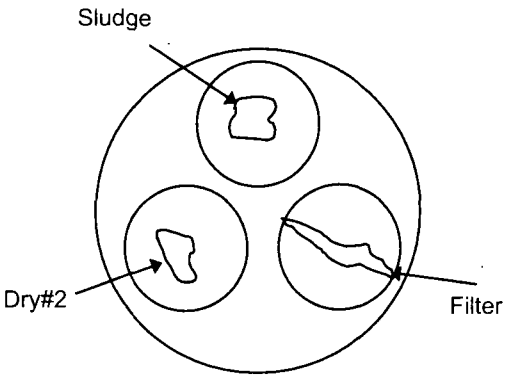
This appendix presents ESEM and SEM/EDS images for filtrate collected from a filtered solution sample and for sacrificial fiberglass samples removed from the test tank on Day 4 (February 9, 2005) of Test #2. The filtered solution sample was passed through a 0.7-μm fiberglass filter at 60°C. SEM/EDS results for the resulting filtrate are presented here. These SEM results were obtained on February 25, 2005.

The fiberglass samples were taken from material encased in a small (~4 in. × 4 in.) stainless-steel mesh envelope that had been submerged in the test tank until being removed on Day 4. Fiberglass samples were examined both hydrated (its condition when removed from the tank) and dry (air dried at room temperature).

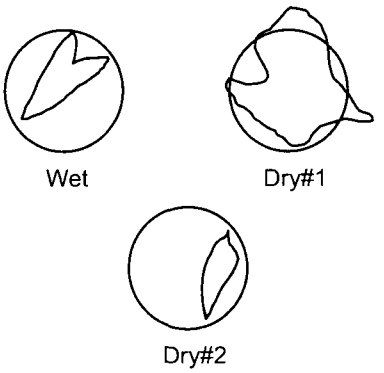
Transcribed Laboratory Log

Laboratory session from February 9, 2005

NRC Test #2, Day 4



SEM MicroProbe Sample Arrangement



ESEM Sample Arrangement

Instrument Conditions: ESEM work in low-vacuum mode with BSE imaging, 20-kV, Working Distance = 8 mm, Aperture = 2, low vacuum = 100 Pa (starting pressure)

Hydrated Sample

Image:	T2D4001	110 ×	Overview	Figure J-1
	T2D4002	600 ×	Close-up near center of image 001	Figure J-2
	T2D4003	500 ×	Close-up of film near center of image 001	Figure J-3
	T2D4004	110 ×	New area	Figure J-4
	T2D4005	500 ×	Right center of image 004	Figure J-5

Dry #1 Sample

Image:	T2D4006	120 ×	Overview	Figure J-6
	T2D4007	500 ×	On deposits between fibers	Figure J-7
	T2D4008	130 ×	Overview new area	Figure J-8
	T2D4009	500 ×	More deposits	Figure J-9

Dry #2 Sample

Image:	T2D4010	40 ×	Overview	Figure J-10
	T2D4011	250 ×	On deposits w/ crystals	Figure J-11
EDS:	D2-1		On smooth deposits	Figure J-12
	D2-2		On crystal mass on fibers	Figure J-13
	D2-3		On smooth cracked deposits	Figure J-14
Image:	T2D4012	200 ×	Crystals on fibers	Figure J-15
EDS:	D2-4		On mass of crystals on fiber, center of image 012	Figure J-16

Filter Sample

Image:	T2D4015	40 ×	Overview of filtrate surface	Figure J-17
	T2D4016	2000 ×	Close-up of filtrate	Figure J-18
EDS:	F-7		On filtrate	Figure J-19
	F-8		On filtrate	Figure J-20

Note: EDS F-7 and F-8 are replicates of homogeneous filtrate material.

Figure J-1. Test #2, Day-4 ESEM image of a hydrated sample, overview. (T2D4001)

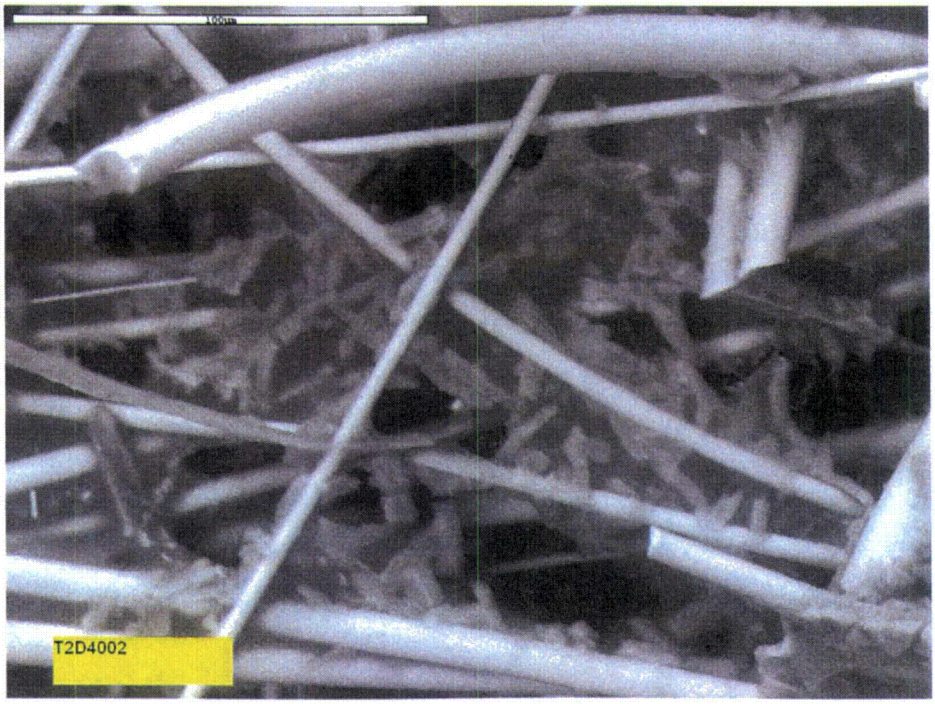


Figure J-2. Test #2, Day-4 ESEM image of a hydrated sample, close-up near the center of the image in Figure J-1.

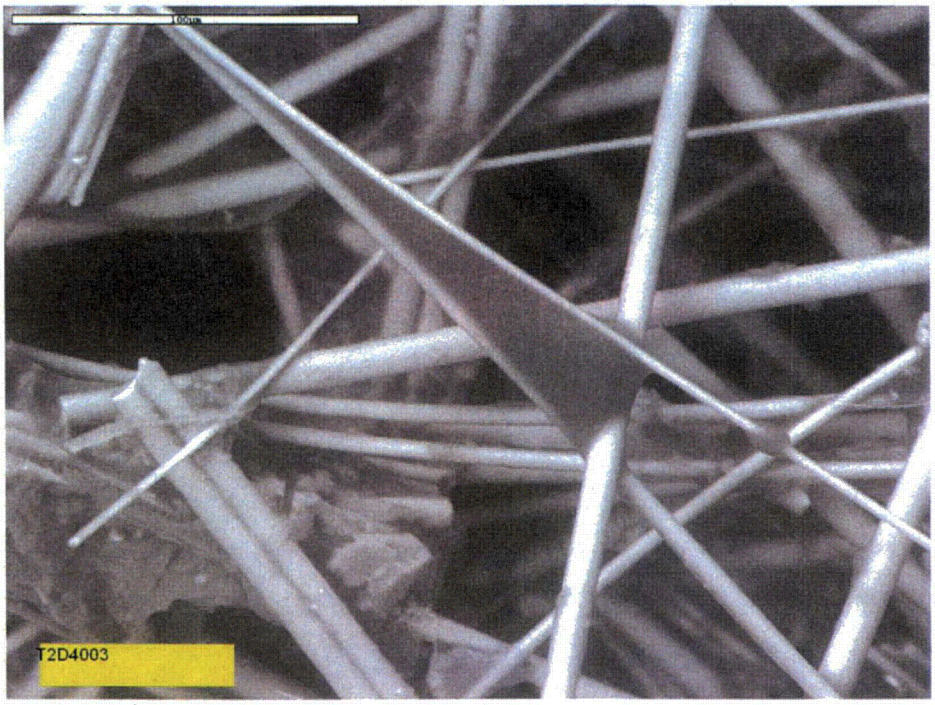


Figure J-3. **Test #2, Day-4 ESEM image, hydrated sample close-up on the film near the center of the image in Figure J-1. (T2D4003)**

Figure J-4. **Test #2, Day-4 ESEM image of a new area within the hydrated sample. (T2D4004)**

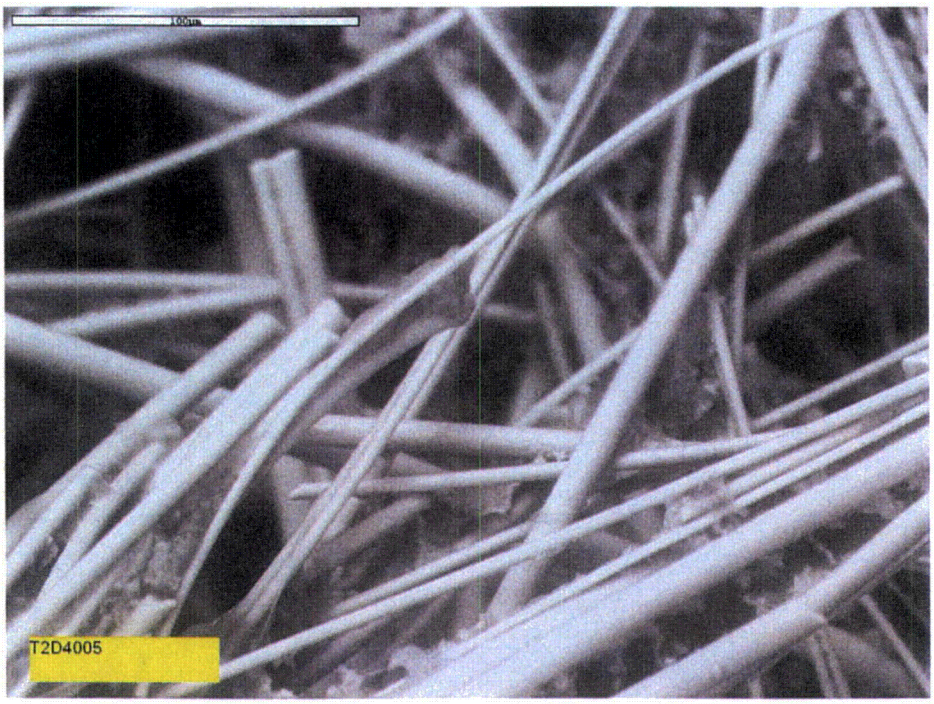


Figure J-5. Test #2, Day-4 ESEM image of the hydrated sample at the right center of Figure J-4. (T2D4005)

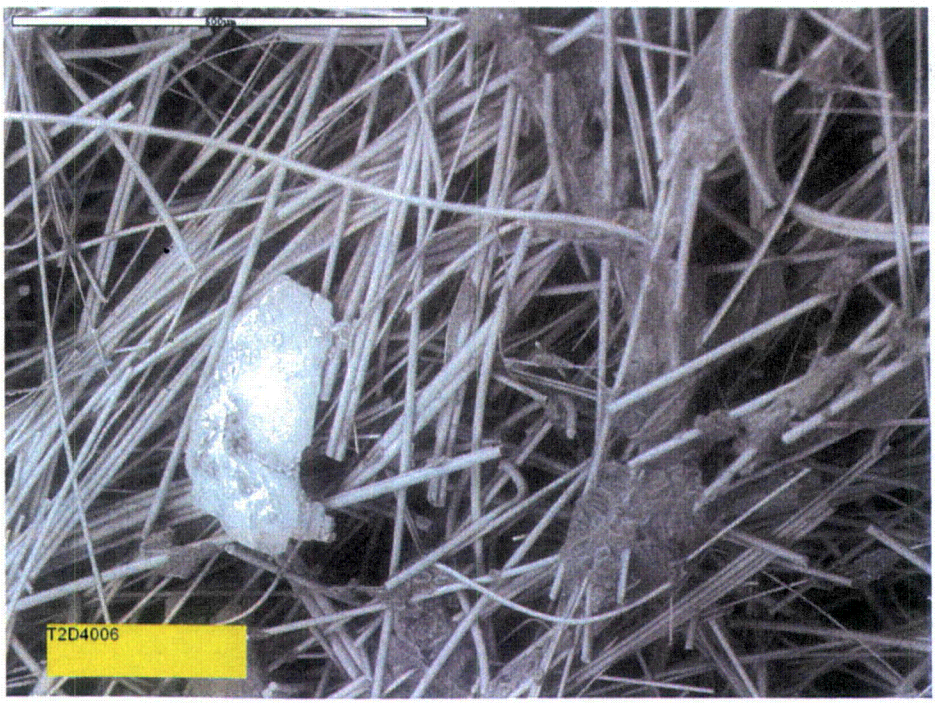


Figure J-6. Test #2, Day-4 SEM image of dry sample #1, overview. (T2D4006)

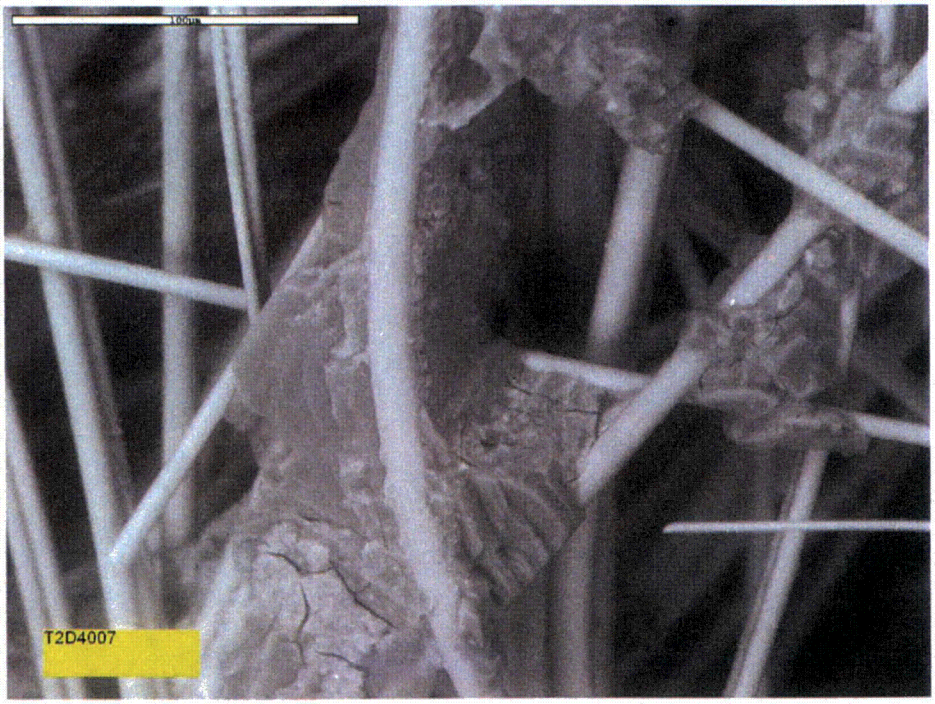


Figure J-7. Test #2, Day-4 SEM dry sample #1, image of deposits between fibers. (T2D4007)

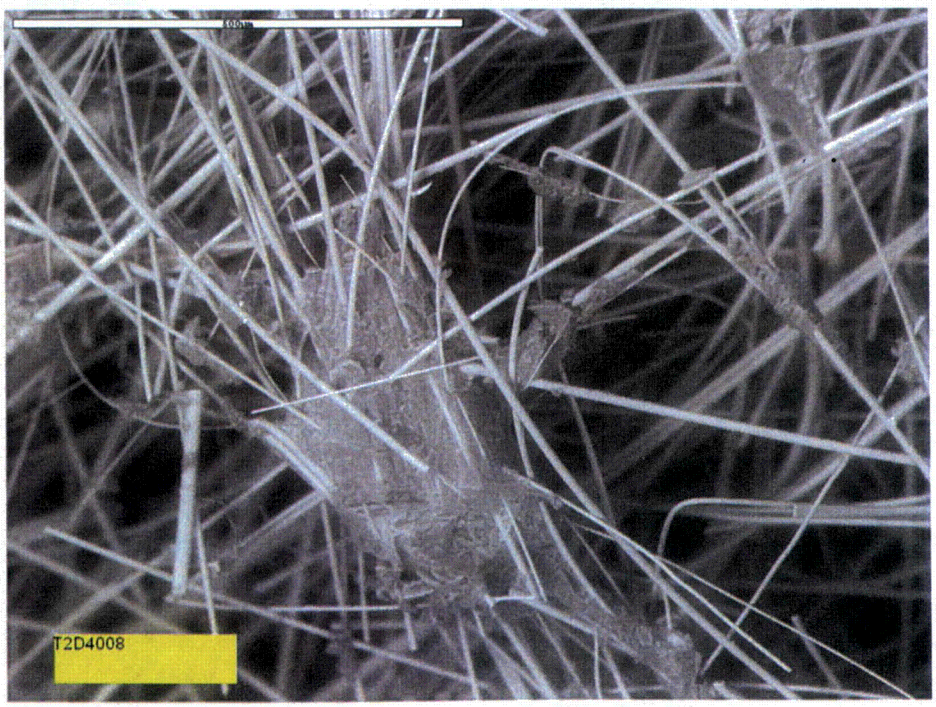


Figure J-8. Test #2, Day-4 SEM image, overview of a new area of dry sample #1. (T2D4008)

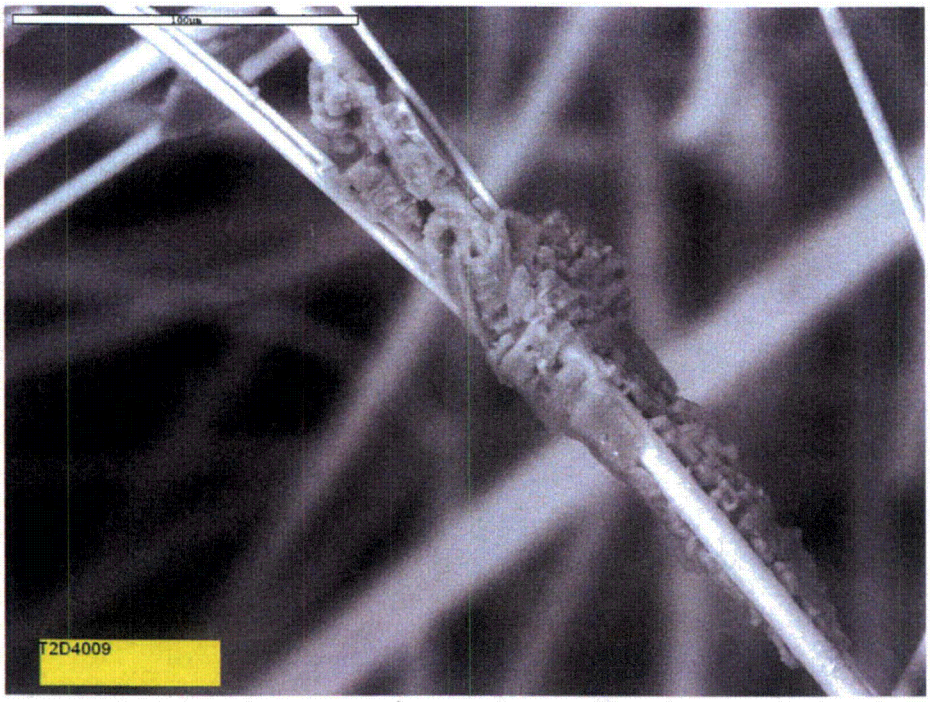


Figure J-9. **Test #2, Day-4 SEM image of deposits on fibers of dry sample #1 at 500 × magnification. (T2D4009)**

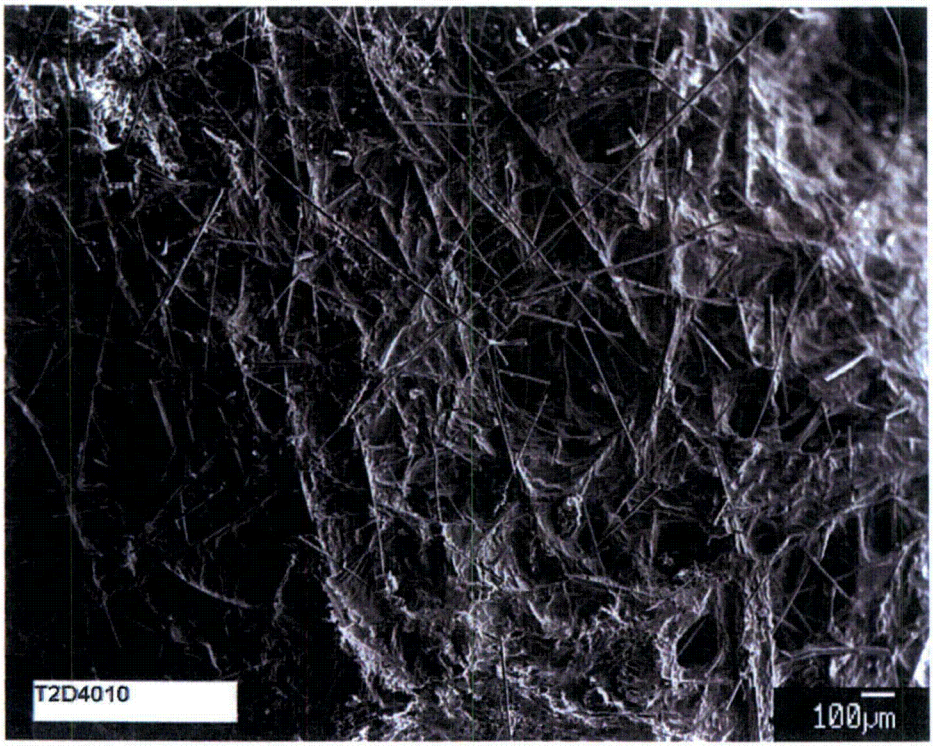


Figure J-10. **Test #2, Day-4 SEM image, overview of dry sample #2. (T2D4010)**

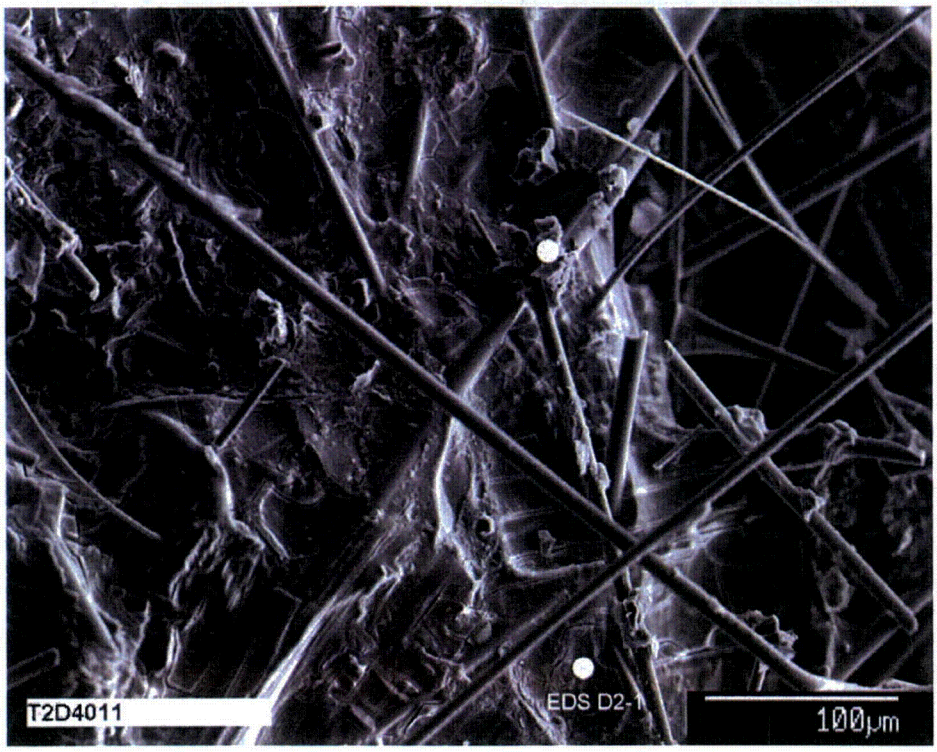


Figure J-11. Test #2, Day-4 SEM image of dry sample #2 deposits with crystals. (T2D4011)

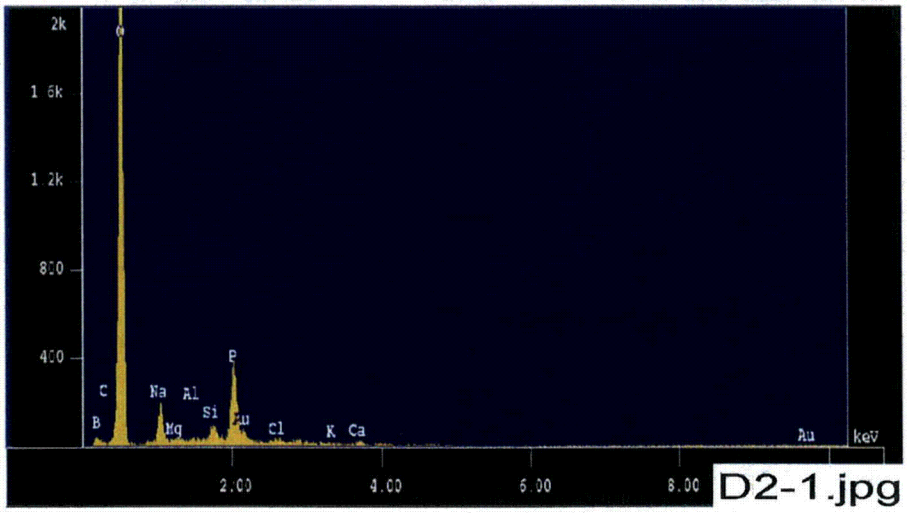


Figure J-12. Test #2, Day-4, dry sample #2 counting spectrum on the smooth deposits of image T2D4011 (Figure J-11). (D2-1)

The results from the chemical composition analysis for EDS D2-1 are given in Table J-1.

Table J-1. Chemical Composition for EDS D2-1

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Group : NRC
Sample : T2D4 ID# : 1
Comment : Gunk filling between fibers
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 100.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.030E-09 A
Stage Point : X=81.229 Y=64.853 Z=10.566
Acq. Date : Wed Feb 9 15:56:03 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
B K	Normal	0.00- 0.36	1.0120	0.0002	169 / 10
O K	Normal	0.25- 0.77	43.9423	0.0053	17656 / 26
Na K	Normal	0.83- 1.28	1.1193	0.0064	1414 / 26
Si K	Normal	1.50- 2.07	0.2683	0.0009	511 / 113
P K	Normal	1.75- 2.38	3.9584	0.0035	3798 / 64
Ca K	Normal	3.40- 4.30	0.2361	0.0040	238 / 14

Chi_square = 39.2421

Element	Mass%	Atomic%	ZAF	Z	A	F
B	15.604	22.5349	6.8867	1.1324	6.0813	1.0000
O	72.999	71.2365	0.7420	0.9755	0.7606	1.0000
Na	2.913	1.9780	1.1622	0.9805	1.1836	1.0014
Si	0.619	0.3442	1.0306	0.9774	1.0580	0.9966
P	7.357	3.7082	0.8301	1.1786	0.7043	1.0000
Ca	0.509	0.1982	0.9627	0.9931	0.9693	1.0001
Total	100.000	100.0000				

Normalization factor = 2.2389

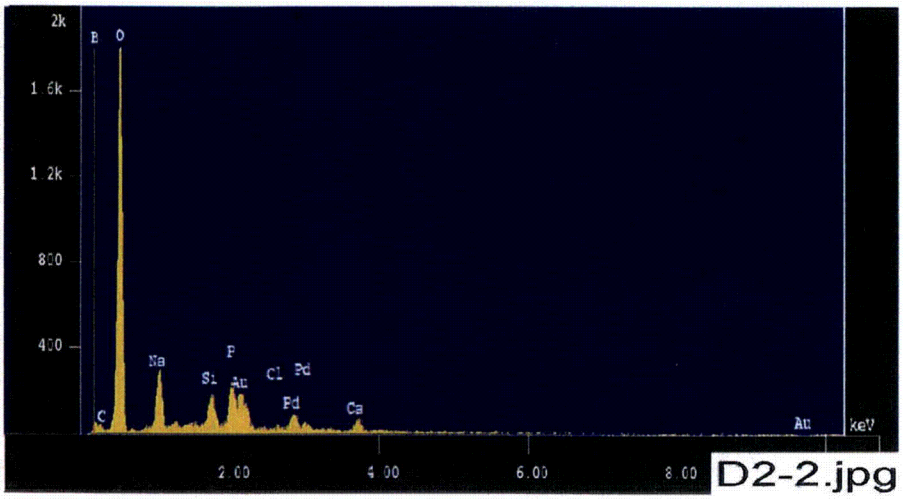

Figure J-13. **Test #2, Day-4, dry sample #2 counting spectrum on the crystal mass on fibers of image T2D4011 (Figure J-11). (D2-2)**

The results from the chemical composition analysis for EDS D2-2 are given in Table J-2.

Table J-2. Chemical Composition for EDS D2-2

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Group : NRC
Sample : T2D4 ID# : 2
Comment : Crystals on fibers
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 100.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.030E-09 A
Stage Point : X=81.182 Y=64.792 Z=10.566
Acq. Date : Wed Feb 9 16:03:32 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
B K	Normal	0.00- 0.36	0.9884	0.0003	165 / 9
O K	Normal	0.25- 0.77	37.3026	0.0050	14988 / 32
Na K	Normal	0.83- 1.28	1.7930	0.0083	2266 / 40
Si K	Normal	1.50- 2.07	0.6948	0.0011	1323 / 112
P K	Normal	1.75- 2.38	1.8701	0.0033	1794 / 102
Ca K	Normal	3.40- 4.30	0.7027	0.0056	708 / 18
Cl K	Normal	2.34- 3.06	0.3090	0.0008	329 / 31

Chi_square = 26.4409

Element	Mass%	Atomic%	ZAF	Z	A	F
B	16.075	23.2987	6.6100	1.1313	5.8430	1.0000
O	70.748	69.2911	0.7708	0.9745	0.7910	1.0000
Na	5.077	3.4601	1.1507	0.9795	1.1731	1.0015
Si	1.782	0.9942	1.0424	0.9763	1.0696	0.9982
P	3.881	1.9632	0.8433	1.1773	0.7166	0.9996
Ca	1.661	0.6492	0.9604	0.9919	0.9681	1.0001
Cl	0.777	0.3435	1.0221	1.0345	0.9888	0.9992
Total	100.000	100.0000				

Normalization factor = 2.4605

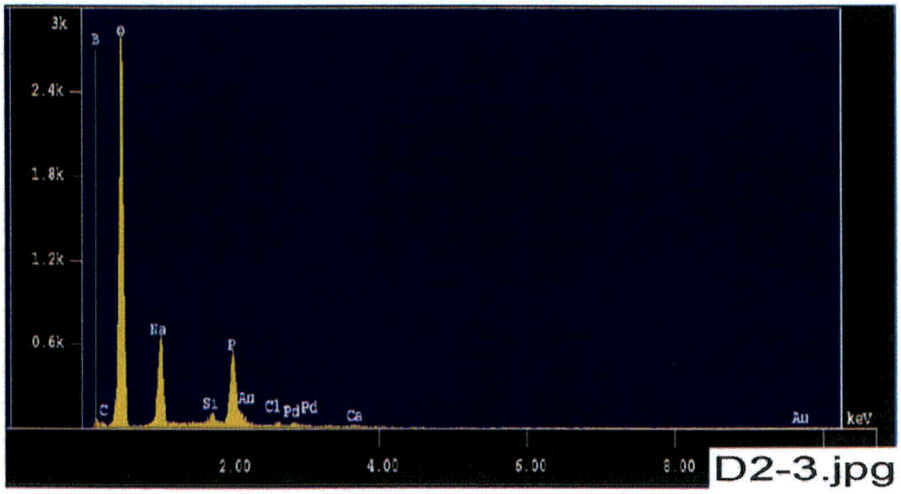

Figure J-14. **Test #2, Day-4, dry sample #2 counting spectrum on the smooth cracked deposit on image T2D4011 (Figure J-11). (D2-3)**

The results from the chemical composition analysis for EDS D2-3 are given in Table J-3.

Table J-3. Chemical Composition for EDS D2-3

```
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 Group      : NRC
 Sample     : T2D4  ID# : 3
 Comment    : Cracked gunk
 Condition  : Full Scale   : 20KeV(10eV/ch,2Kch)
              Live Time    : 100.000 sec    Aperture #     : 1
              Acc. Volt    : 15.0 KV        Probe Current  : 1.027E-09 A
              Stage Point  : X=81.136 Y=64.586 Z=10.566
              Acq. Date    : Wed Feb  9 16:17:50 2005


 Element     Mode      ROI(KeV)    K-ratio(%)   +/-    Net/Background
   B K       Normal   0.00- 0.36     1.6161    0.0003      269 /      16
   O K       Normal   0.25- 0.77    57.3942    0.0061    22993 /      42
  Na K       Normal   0.83- 1.28     4.0848    0.0114     5146 /      33
  Si K       Normal   1.50- 2.07     0.2639    0.0009      501 /     154
   P K       Normal   1.75- 2.38     5.9164    0.0043     5660 /      76
  Ca K       Normal   3.40- 4.30     0.0703    0.0042       71 /      22
  Cl K       Normal   2.34- 3.06     0.3471    0.0008      368 /      34
-------------------------------------------------------------------------
                                Chi_square = 37.8736

 Element Mass%    Atomic%   ZAF      Z      A      F
      B   17.669  25.5719 7.2292 1.1279 6.4095 1.0000
      O   66.963  65.4859 0.7714 0.9716 0.7940 1.0000
     Na    6.865   4.6718 1.1112 0.9766 1.1362 1.0014
     Si    0.415   0.2309 1.0385 0.9734 1.0704 0.9966
      P    7.444   3.7603 0.8319 1.1738 0.7089 0.9998
     Ca    0.102   0.0399 0.9614 0.9890 0.9720 1.0001
     Cl    0.542   0.2391 1.0323 1.0314 1.0009 1.0000
-------------------------------------------------------------------------
 Total   100.000 100.0000
 Normalization factor =  1.5124
```

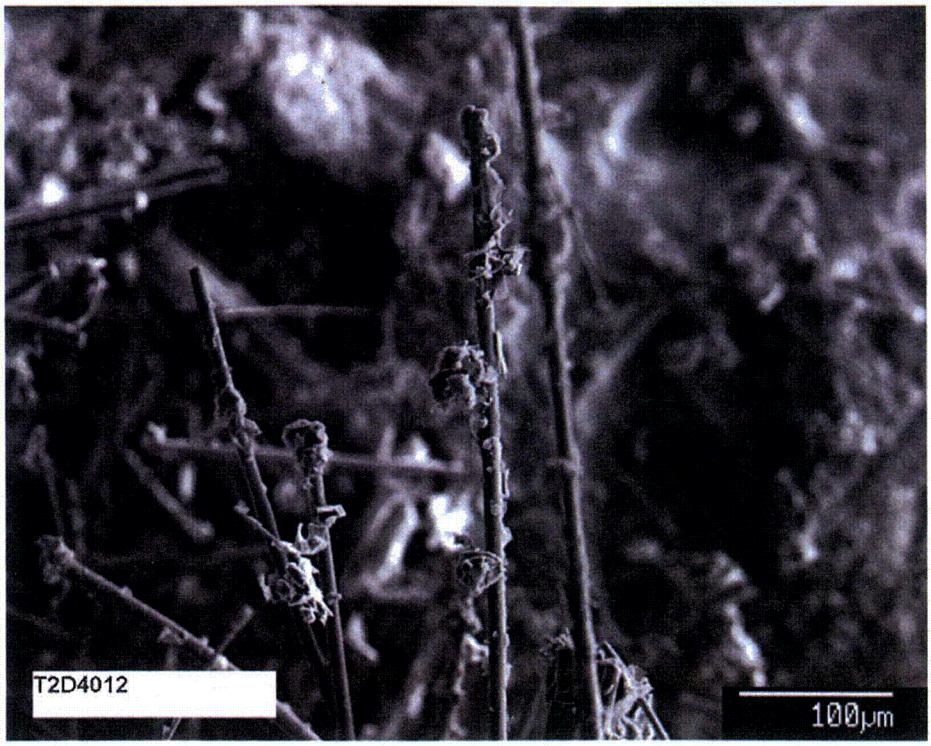

Figure J-15. Test #2, Day-4 SEM image of crystals on fibers of dry sample #2 at 200 × magnification. (T2D4012)

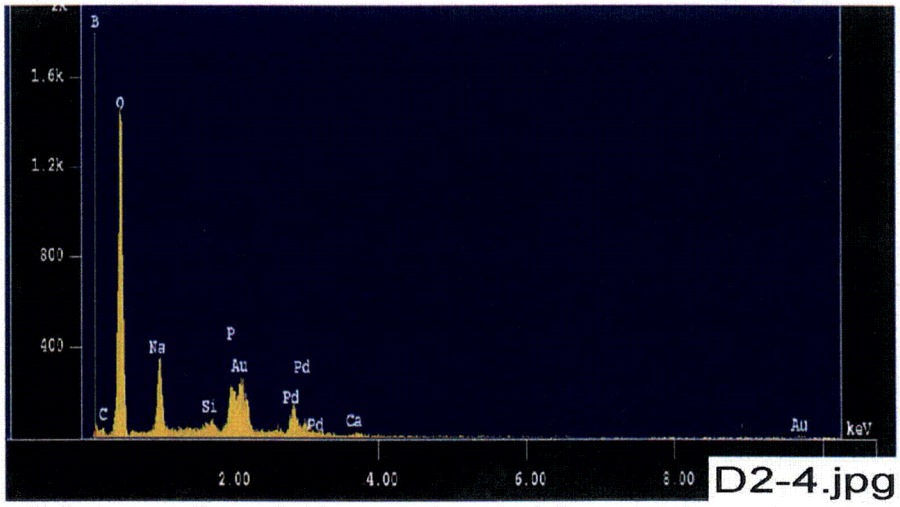

Figure J-16. Test #2, Day-4, dry sample #2 counting spectrum on the mass of crystals on fibers at the center of image T2D4012 (Figure J-12) but taken from an agglomeration at the tip of a fiber. (D2-4)

The results from the chemical composition analysis for EDS D2-4 are given in Table J-4.

Table J-4. Chemical Composition for EDS D2-4

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Group : NRC
Sample : T2D4 ID# : 4
Comment : Xtals on fibers
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 100.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.025E-09 A
Stage Point : X=81.535 Y=63.667 Z=10.566
Acq. Date : Wed Feb 9 16:25:19 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
B K	Normal	0.00- 0.36	1.2483	0.0003	208 / 16
O K	Normal	0.25- 0.77	31.5962	0.0047	12633 / 34
Na K	Normal	0.83- 1.28	1.9840	0.0085	2495 / 36
Si K	Normal	1.50- 2.07	0.1494	0.0008	283 / 108
P K	Normal	1.75- 2.38	1.5144	0.0034	1446 / 68
Ca K	Normal	3.40- 4.30	0.1440	0.0043	144 / 23

Chi_square = 23.4498

Element	Mass%	Atomic%	ZAF	Z	A	F
B	21.555	30.0161	6.0487	1.1237	5.3829	1.0000
O	67.735	63.7363	0.7509	0.9681	0.7757	1.0000
Na	6.299	4.1245	1.1121	0.9732	1.1410	1.0015
Si	0.441	0.2365	1.0343	0.9702	1.0677	0.9984
P	3.580	1.7400	0.8280	1.1700	0.7077	1.0000
Ca	0.390	0.1467	0.9501	0.9862	0.9633	1.0001
Total	100.000	100.0000				

Normalization factor = 2.8548

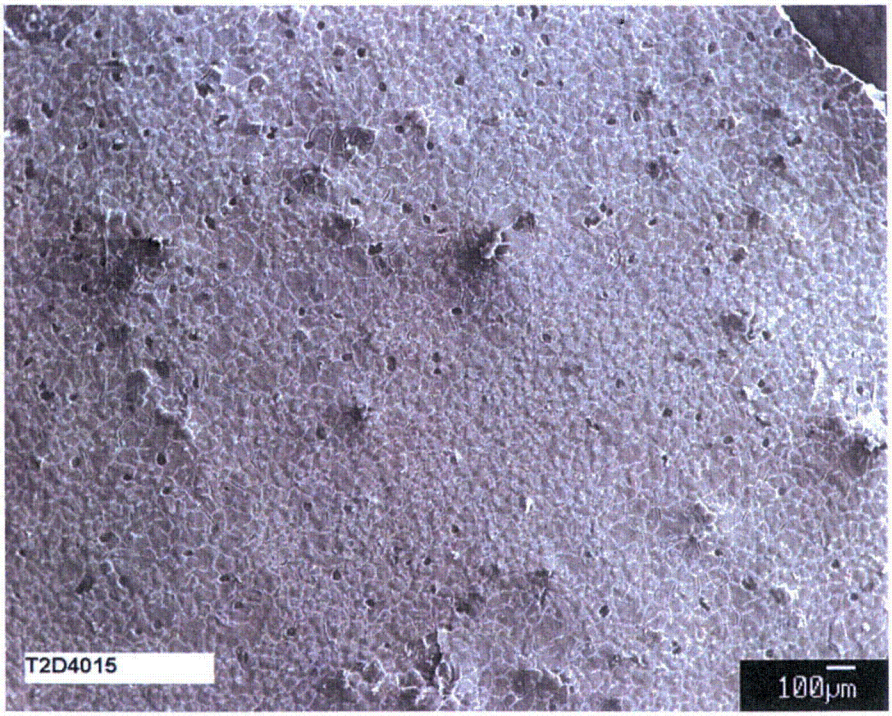

Figure J-17. Test #2, Day-4 SEM image, overview of the filtrate surface for the filter sample. (T2D4015)

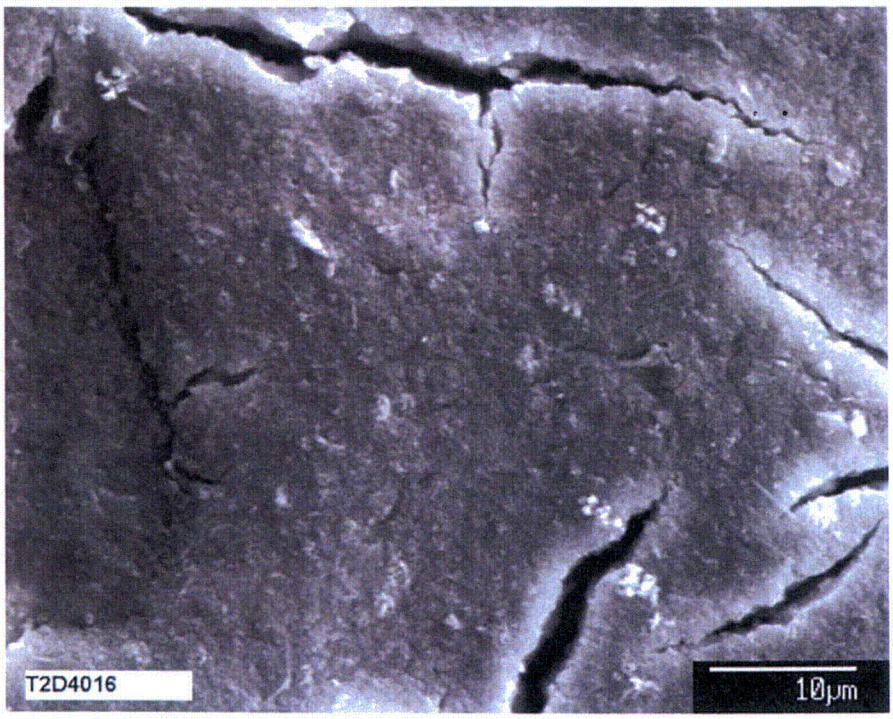

Figure J-18. Test #2, Day-4 SEM image of a close-up of filtrate for the filtrate sample, magnification is 2000 ×. (T2D4016)

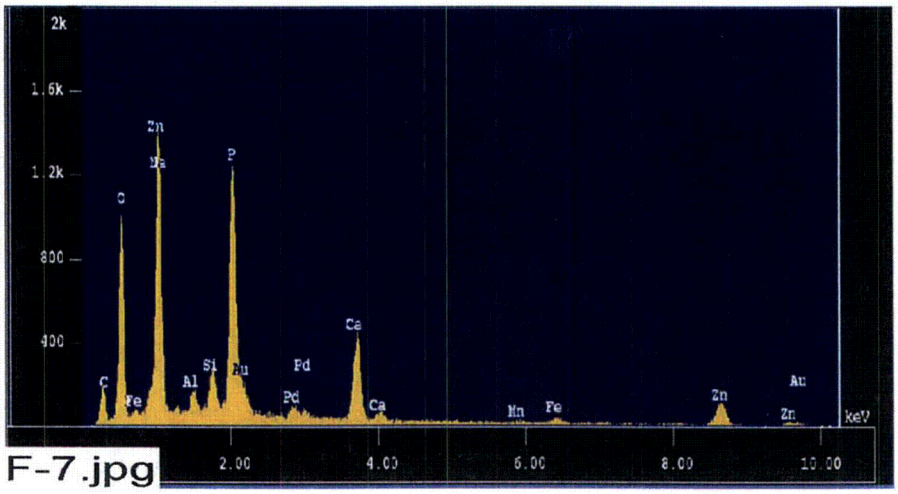

Figure J-19. Test #2, Day-4 filter sample counting spectrum for filtrate. (F-7)

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

Table J-5. Chemical Composition for EDS F-7

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Group : NRC
Sample : T2D4 ID# : 7
Comment : Filtrate
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 100.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.151E-09 A
Stage Point : X=70.927 Y=64.105 Z=10.447
Acq. Date : Wed Feb 9 16:58:16 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	0.9733	0.0006	648 / 104
O K	Normal	0.25- 0.77	22.5680	0.0047	10133 / 118
Al K	Normal	1.26- 1.78	0.4614	0.0009	982 / 155
Si K	Normal	1.50- 2.07	0.7686	0.0014	1635 / 380
P K	Normal	1.75- 2.38	12.6137	0.0066	13524 / 196
Ca K	Normal	3.40- 4.30	4.7780	0.0122	5376 / 36
Fe K	Normal	6.04- 7.40	0.7955	0.0560	288 / 22
Zn K	Normal	8.22-10.03	13.8176	0.0110	1792 / 8
Mn K	Normal	5.53- 6.82	0.2228	0.0010	92 / 20
Na K	Normal	0.83- 1.28	0.4176	0.0235	590 / 93

Chi_square = 4.9155

Element	Mass%	Atomic%	ZAF	Z	A	F
C	7.802	14.9047	5.5330	0.9853	5.6158	1.0000
O	39.654	56.8718	1.2129	0.9394	1.2911	1.0000
Al	0.921	0.7829	1.3773	0.9502	1.4523	0.9980
Si	1.339	1.0937	1.2023	0.9383	1.2871	0.9955
P	16.843	12.4771	0.9217	1.1307	0.8154	0.9997
Ca	6.660	3.8131	0.9622	0.9486	1.0151	0.9993
Fe	1.061	0.4357	0.9202	0.9401	1.0059	0.9730
Zn	24.469	8.5891	1.2224	1.2235	0.9991	1.0000
Mn	0.375	0.1566	1.1615	1.1704	1.0083	0.9843
Na	0.877	0.8753	1.4496	0.9430	1.5352	1.0013
Total	100.000	100.0000				

Normalization factor = 1.4487

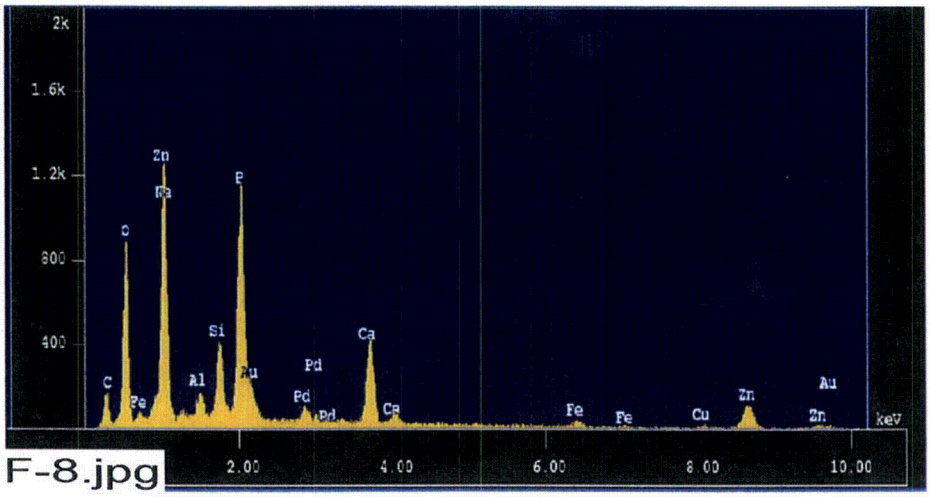

Figure J-20. Replicate Test #2, Day-4 filter sample counting spectrum for filtrate. (F-8)

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

Table J-6. Chemical Composition for EDS F-8

```
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 Group     : NRC
 Sample    : T2D4  ID# : 8
 Comment   : Filtrate
 Condition : Full Scale  : 20KeV(10eV/ch,2Kch)
             Live Time   : 100.000 sec    Aperture #     : 1
             Acc. Volt   : 15.0 KV        Probe Current  : 1.140E-09 A
             Stage Point : X=69.995 Y=63.989 Z=10.447
             Acq. Date   : Wed Feb  9 17:10:35 2005

 Element     Mode      ROI(KeV)    K-ratio(%)   +/-    Net/Background
   C K       Normal   0.09- 0.46     1.1312    0.0005      746 /      97
   O K       Normal   0.25- 0.77    18.7823    0.0040     8352 /     116
  Al K       Normal   1.26- 1.78     0.4612    0.0009      972 /     185
  Si K       Normal   1.50- 2.07     1.4997    0.0016     3159 /     359
   P K       Normal   1.75- 2.38    11.3019    0.0063    12002 /     232
  Ca K       Normal   3.40- 4.30     4.7008    0.0119     5238 /      34
  Fe K       Normal   6.04- 7.40     0.9343    0.0551      335 /      10
  Zn K       Normal   8.22-10.03    14.3829    0.0110     1847 /       7
  Na K       Normal   0.83- 1.28     0.3713    0.0223      519 /      71
  Cu K       Normal   7.63- 9.27     0.7781    0.0041      132 /      10
-------------------------------------------------------------------------
                                Chi_square =  4.8053

 Element Mass%     Atomic%    ZAF      Z      A      F
      C    9.451   18.4623 5.6231 0.9795 5.7408 1.0000
      O   35.077   51.4416 1.2569 0.9339 1.3459 1.0000
     Al    0.958    0.8327 1.3973 0.9445 1.4824 0.9980
     Si    2.706    2.2607 1.2144 0.9326 1.3073 0.9960
      P   15.710   11.9007 0.9356 1.1238 0.8327 0.9997
     Ca    6.694    3.9187 0.9584 0.9426 1.0175 0.9993
     Fe    1.265    0.5314 0.9111 0.9337 1.0064 0.9696
     Zn   25.930    9.3071 1.2134 1.2144 0.9992 1.0000
     Na    0.813    0.8294 1.4731 0.9374 1.5694 1.0013
     Cu    1.396    0.5154 1.2074 1.2074 1.0000 1.0000
-------------------------------------------------------------------------
 Total   100.000 100.0000
 Normalization factor =  1.4858
```

Appendix K

ICET Test #2: Pre-Test, Test, and Post-Test Project Instructions

Contents

Tables

The ICET test series is conducted under the guidance of project instructions (PIs) that identify the steps to follow for certain activities. These PIs are revised or rewritten as needed for each test. For Test #2, new PIs were written to address pre-test operations and test operations. The PI that addresses post-test operations was revised for Test #2. These three PIs are included in this appendix to describe more completely the test apparatus and chemical solution preparations, the test startup and daily sampling, and the steps followed after test shutdown.

K1 ICET TEST #2 PRE-TEST OPERATIONS

K1.1 INTRODUCTION

K1.1.1 PURPOSE

The first intent of this instruction is to ensure that all data acquisition, testing samples, testing supplies, and related material are ready and accounted for before testing. The second intent of this instruction is to prepare the chemical tank for testing.

K1.1.2 SCOPE

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

K1.1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.a, October 6, 2004
- TSP Chemical Additive Analysis, Test #2—ICET-CALC-011
- Laboratory Safety Guidelines
- ASTM A 380–99, Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems
- Material Safety Data Sheets (MSDS) for all chemicals involved

K1.2 PREREQUISITES

The data acquisition setup and inspection; calibration; and the coupon receipt, preparation, inspection, and storage tasks must be completed in full prior to performance of this activity. Fiberglass samples must be weighed and their planned locations in the tank identified. That data must be entered into the fiberglass spreadsheet.

K1.2.1 TRAINING REQUIREMENTS

The following personnel training is required for this task:

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

K1.2.2 EQUIPMENT REQUIREMENTS

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

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

K1.3 DOCUMENTATION REQUIRED

A LabVIEW operational manual is required for this task. In addition, MSDSs must be available for all chemicals used.

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

K1.4 HAZARDS

The hazards associated with this activity include tipping of the chemical tank assembly and potential injuries associated with chemical handling.

K1.5 INSTRUCTIONS

1. Ensure that all testing materials and supplies are ready and on-site. See checklist at the end of this document. Verify that eye wash station is operational. Note: The following solutions are not used in this instruction, but are to be prepared in advance of entering ICET-PI-011, "Test Operations, Test #2 (TSP at pH = 7)." After preparation, clearly label the containers with the solutions and place in an area restricted for ICET Project test use.
2. Prepare TSP Solution Batch 1.
   a. Heat about 1.5 gallons of demineralized water, add 300 g of boric acid (H_3BO_3), and stir until the boric acid is dissolved. Pour the solution into a 5-gallon plastic container. Dissolve the boric acid in multiple batches if necessary.
   b. Add additional demineralized water to the 5-gallon plastic container until it contains about 4 gallons.
   c. Dissolve 1893 g of TSP (Na_3PO_4-$12H_2O$) into the water in the container.
   d. Dilute with additional demineralized water until the volume is 5 gallons.
3. Prepare TSP Solution Batch 2.
   a. Heat about 1.5 gallons of demineralized water, add 300 g of boric acid (H_3BO_3), and stir until the boric acid is dissolved. Pour the solution into a 5-gallon plastic container. Dissolve the boric acid in multiple batches if necessary.
   b. Add additional demineralized water to the 5-gallon plastic container until it contains about 4 gallons.
   c. Dissolve 1893 g of TSP (Na_3PO_4-$12H_2O$) into the water in the container.
   d. Add 214 mL of 12.115 N hydrochloric acid (HCl) to the water in the container.
   e. Dilute with additional demineralized water until the volume is 5 gallons.
4. Prepare laboratory control sample (LCS). See the Chemical Sampling and Analysis Plan for details on the laboratory control sample.
5. Start the data acquisition system. During step 6 verify that the data acquisition system is monitoring flow rate, pump speed, temperature, and pH.

6. Flush tank.
    a. Add 100 gallons of tap water to the tank.
    b. Start pump at 25 gpm and circulate water for 5 minutes, directing water through both recirculation lines.
    c. Check for leaks. If any leaks are present, repair at this time.
    d. Drain water (see step 7). Water should be visually clear, if not, repeat flush tank sequence until water is visually clear.
7. Clean tank. Note: The tank internal will be inspected at the best method for cleaning from ASTM A 380–99 will be chosen. This may require modification to the following steps a–f.
    a. Fill tank with 22.5 gallons of tap water and 2.5 gallons of ammonium hydroxide.
    b. Start pump at 25 gpm and circulate water for 5 minutes, directing water/ ammonium hydroxide mixture through both recirculation lines.
    c. Drain tank.
    d. Fill tank with 22.5 gallons of tap water and 2.5 gallons of ethanol.
    e. Start pump as 25 gpm and circulate water for 5 minutes, directing water/ethanol mixture through both recirculation lines.
    f. Drain tank.
8. Rinse tank.
    a. Measure and record turbidity and conductivity of tap water (see Chemical Sampling and Analysis instruction sheet).
    b. Add 50 gallons of tap water to the tank.
    c. Start pump at 25 gpm and circulate water for 5 minutes, directing water through both recirculation lines.
    d. Drain rinse water. Measure and record turbidity and conductivity of rinse water. Turbidity and conductivity should be within 10 percent of initial values. If not, repeat rinse tank sequence until turbidity and conductivity are within limits.
9. Final rinse.
    a. Fill tank with 50 gallons of RO water.
    b. Start pump at 25 gpm and circulate water for 30 minutes, directing water through both recirculation lines.
    c. Drain tank.
    d. Repeat steps 9.a, 9.b, and 9.c.
    e. Drain the entire system. Repeat steps 9.a, 9.b, and 9.c. until water conductivity is less than 50 μS/cm.
    f. Drain tank.
10. Tank is now ready for testing. Proceed immediately to Instruction No. ICET-PI-004.

K1.6 ATTACHMENTS

No forms are attached to this document.

K1.7 MATERIALS CHECKLIST

_____ lithium hydroxide, 1.197 g

_____ TSP, 3.785 kg

_____ tap water supply

_____ demineralized water production system

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

_____ analytical balance

_____ top loading balance

_____ chemical spatula

_____ six 1-gallon HDPE or PP bottles

_____ 500-mL volumetric flask

_____ 500-mL HDPE or PP bottle

_____ 2.5 gallons ethanol

_____ 2.5 gallons ammonium hydroxide

_____ turbidimeter and associated equipment

_____ conductivity meter and associated equipment

K2 ICET TEST #2 TEST OPERATIONS

K2.1 INTRODUCTION

K2.1.1 PURPOSE

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

K2.1.2 SCOPE

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

K2.1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.a, October 6, 2004
- ASTM Standard G 4-01
- ASTM Standard D 3370-95a
- ASTM Standard G 31-72
- Material Safety Data Sheets (MSDS) for all chemicals involved
- LabVIEW operation manual
- Laboratory Safety Guidelines
- TSP Chemical Additive Analysis; Test #2—ICET-CALC-011

K2.2 PREREQUISITES

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

K2.2.1 TRAINING REQUIREMENTS

The following personnel training is required for this task:

1. LabVIEW and computer data acquisition training.
2. Chemical handling training for all chemicals involved.

K2.2.2 EQUIPMENT REQUIREMENTS

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

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

K2.3 DOCUMENTATION REQUIRED

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

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

K2.4 HAZARDS

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

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

K2.5 INSTRUCTIONS

1. Pre-Operation Preparation should be complete before proceeding with this sequence.
2. Ensure that all testing materials and supplies are ready and on-site (see checklist at end of this instruction).
3. Add 240 gallons of RO water to the tank by pumping water from the RO skid through the totalizing flow meter. Record flow to the nearest 0.5 gallon.
4. Verify valves are positioned as follows:

Table K2-1. Valve Positions for ICET Test #2

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

5. Start pump and adjust to flow rate of approximately 25 gpm.
6. Start computer, start LabVIEW, verify that flow rate, pump speed, temperature, and pH are being recorded properly.
7. Turn on heater and allow water in tank to heat to 60 °C ± 2 °C.

8. Add the pre-mixed LiOH solution.

9. Add 14.54 kg of boric acid (H_3BO_3), weighing in approx. 2 kg increments, recording the weight of each increment to the nearest 10 g.

10. Allow the water to circulate until the solution is visibly clear, indicating that the boric acid is completely dissolved.

11. Allow water in tank to heat to 65 °C ± 2 °C.

12. Take grab water sample for analysis for the parameters identified in steps a–h below. Also record physical appearance of the sample (clarity, presence of gelatinous material, etc.). All Day 1 and subsequent samples will be analyzed by Assaigai Analytical Laboratory. In addition, periodic test samples and laboratory control samples (LCSs) will also be analyzed by the UNM laboratory.

    a. pH

    b. temperature

    c. turbidity

    d. viscosity

    e. total suspended solids (TSS)

    f. dissolved oxygen (DO)

    g. chloride

    h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved

13. Add concrete dust and latent debris samples (prepared earlier), wait 10 minutes, take 100 mL water sample for particulate size distribution, density, and TSS.

14. Stop pump.

15. Place coupon racks and fiberglass holders into tank.

16. Verify locations of coupon racks and fiberglass holders.

17. Lift tank lid into position on top of tank.

18. Start pump and adjust pump speed to 25 gpm.

19. Open valve V-7 (tank spray supply) to direct water to nozzles and adjust valves V-7 (tank spray supply) and V-8 (recirculation supply) until nozzle flowmeter is reading 3.5 gpm. Verify total flow is still 25 gpm and adjust variable frequency drive (VFD) if necessary.

20. Record date and time at which nozzle flow started. This is time t = 0 for the test.

21. Start chemical metering pump from TSP Solution Batch 1 at a rate of 0.0417 gpm (158 mL/min). The objective here and in step #22 is to add a total of 10 gallons of TSP solution in 4 hours.

22. After 2 hours, switch the chemical metering pump to TSP Solution Batch 2.

23. Take a measurement of hydrogen concentration. At 2-hour increments, repeat the hydrogen concentration measurement. If the concentration reaches 10% of the flammability limit, purge the tank atmosphere. This needs to be repeated until the hydrogen concentration has been determined to be below the flammability limit, and then the frequency of hydrogen concentration measurements is to be re-evaluated.

24. At t = 4 hours, stop the chemical metering pump and close valve V-7 (tank spray supply).

25. Immediately after closing valve V-7 (at t = 4 hours), take water grab sample for analysis for the parameters listed below. Record the time of sample collection.

    a. pH

    b. temperature

    c. turbidity

    d. viscosity

    e. chloride

    f. total suspended solids (TSS)

    g. dissolved oxygen (DO)

    h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved

26. At t = 24 hours, t = days 2, 3, 4, 5, 6, 7, 9, 11, 13, 15, 18, 21, 24, 27, and 30 take water grab sample for analysis for the parameters listed below. Record the time of sample collection.

    a. pH

    b. turbidity

    c. viscosity

    d. temperature

    e. total suspended solids (TSS)

    f. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved. An exception is that B, Li, K, Pb, and chloride analyses will be performed only at t = days 15 and 30. Also, dissolved oxygen will be measured at day 30.

27. During each daily water sample collection, look inside tank (through windows) and record observations.

28. At t = 24 hours, and weekly thereafter and at the end of the test, collect 100 mL water sample for particulate size distribution and density analysis, to be performed at AALI. The particulate size ranges to be used will be as close as possible to those called out in the test plan: (in microns), 1-10, 11-25, 26-50, 51-75, 76-100, and > 100 microns.

29. At 14 days $\leq$ t $\leq$ 16 days and at the end of the test, collect a sacrificial fiberglass sample to be inspected and examined with SEM.

30. At 24 hours, at 14 days $\leq$ t $\leq$ 16 days and at the end of the test, run 1L of water through a nucleopore filter. The filter will be taken for SEM analysis as specified in ICET-PI-007.

31. Shut down pump

32. Indicate end of test on the data acquisition system and shut down the data acquisition software

33. Proceed directly to Instruction sheet for Post-Test Operations.

K2.6 ATTACHMENTS

K2.6.1 ATTACHMENT A. DAILY LOG SHEET

Table K2-2. Daily Log Sheet

Integrated Chemical Effects Test (Test #2)

Date: _____ Time of sample collection: _____

Sample taken by: _____ Verified by: _____

Sample bottle identification:

Assaigai (total): _____

Assaigai (filtered): _____

UNM (total): _____

UNM (filtered): _____

Control system readings:

Temperature: _____ Flow: _____ pH: _____

Analyses:

Volume filtered for TSS: _____ pH: _____

Temperature: _____ Dissolved oxygen: _____

Turbidity: _____ Viscosity: _____

Water Level: _____ Water Added: _____

Other: _____ Other: _____

Fiberglass or other samples taken: _____

Comments:

☐ Continued on back

K2.7 MATERIAL CHECKLIST

_____ boric acid, 15.14 kg

_____ lithium hydroxide (0.1 N solution), 118 mL

_____ hydrochloric acid (6 N solution), 432 mL

_____ TSP, 3786 g evenly mixed in two 5-gallon containers

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

_____ top-loading balance

_____ weigh pan for 2 kg aliquots of boric acid

_____ 250 mL graduated cylinder

_____ 500 mL graduated cylinder

_____ totalizing flow meter

_____ sample containers (see Chemical Sampling Instruction)

_____ analytical equipment (see Chemical Sampling Instruction)

_____ pre-assembled coupon racks

_____ pre-assembled fiberglass baskets

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

_____ computer disks for backup of Labview data

_____ Masterflex peristaltic pump and tubing

_____ demineralized water production system

K3 ICET TEST #2 POST-TEST OPERATIONS

K3.1 INTRODUCTION

K3.1.1 PURPOSE

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

K3.1.2 SCOPE

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

K3.1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.b, February 9, 2005
- ASTM Standard G 4-01
- ASTM Standard G 31-72
- ICET-PI-002, Coupon Receipt, Preparation, Inspection, and Storage, November 19, 2004
- ICET-PI-011, Rev. 0, Test Operations, Test #2, February 3, 2005
- ICET-PI-005, Rev. 1, Chemical Sampling and Analysis, February 3, 2005
- Laboratory safety guidelines
- ICET Project Safety Plan

K3.2 PREREQUISITES

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

K3.2.1 TRAINING REQUIREMENTS

- Laboratory Safety Guidelines
- ICET Project Safety Plan

K3.2.2 EQUIPMENT REQUIREMENTS

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

K3.3 DOCUMENTATION REQUIRED

Documentation related to test parameters, chemical water analyses, coupon and fiberglass examinations, and daily test operations are outlined elsewhere. In this instruction, the steps

required to remove samples from the test apparatus and to make it ready for the next test are outlined. In addition, observations as to the test apparatus' condition are obtained and recorded here.

K3.4 HAZARDS

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

K3.5 INSTRUCTIONS

1. On the last day of testing, collect water samples and perform analyses as outlined in ICET-PI-011 and ICET-PI-005.
2. Remove 10L of water from the test apparatus and store at test temperature, for future analyses
3. Shut off the recirculation pump.
4. Remove the small fiberglass samples for SEM examination.
5. Leave one heater on and continue to monitor tank water temperature.
6. Isolate and drain the test apparatus piping.
7. Remove the tank lid.
8. Before removing coupon racks or insulation samples, examine and take photographs and notes of the inside of the tank, the coupons and racks, and the insulation samples.
9. Remove the six non-submerged coupon racks to a staging area for drying and post-test examinations (refer to ICET-PI-002).
10. Take additional photographs of the inside of the tank.
11. Take swipes from several locations on the inside tank wall, and place the samples in plastic holders that are marked with the sample location.
12. Drain the tank slowly, down to the level that uncovers the submerged rack, but keeping the water level above the heater.
13. Remove the submerged coupon rack to the staging area.
14. Repeat steps # 10 and 11.
15. Turn off the heater.
16. Completely drain the tank, taking precautions so that the sediment on the bottom of the tank is not disturbed any more than necessary.
17. When the tank is drained, repeat step # 10. Note especially the locations and orientations of the remaining samples.
18. Remove the remaining insulation samples to the staging area to dry.
19. Ensure that all samples removed from the tank are clearly marked as to their location and orientation within the tank.
20. After all samples have been removed, repeat step # 10.

21. Inspect the interior of the tank, noting any observations.
22. Note the presence of any sediment. Carefully remove as much sediment as possible, noting any unique aspects of it, such as location. Place the sediment in plastic containers with lids, marking the location of the sediment in the tank.
23. Remove the tank drain screen and remove the insulation sample for future analysis.
24. Rinse the tank with tap water and drain the water.
25. Fill the system with 250 gallons of tap water and circulate water through the spray nozzles and recirculation headers for at least 60 minutes. Repeat with de-mineralized water.
26. Store water that was drained from the tank until it is cleared for discharge.
27. If any signs of deterioration are observed on the inside of the test apparatus tank, remove selected insulation on the tank. Inspect the stainless steel tank for any abnormalities.

K3.6 ATTACHMENTS

No forms are attached to this document.

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(See instructions on the reverse)

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

11. ABSTRACT *(200 words or less)*

A 30-day test was conducted in the Integrated Chemical Effects Test (ICET) project test apparatus. The test simulated the chemical environment present inside a pressurized-water-reactor containment water pool after a loss-of-coolant accident. The initial chemical environment contained 14.54 kg of boric acid and 0.663 g of lithium hydroxide (LiOH). Trisodium phosphate (3.786 kg), hydrochloric acid (211 mL), and additional boric acid (600 g) were added beginning at 30 minutes and lasting until 4 hours into the test. Materials tested within the constant temperature (60°C) environment included representative amounts of submerged and unsubmerged aluminum, copper, concrete, zinc, carbon steel, and fiberglass insulation samples. Representative amounts of concrete dust and latent debris were also added to the test solution. The test solution reached a pH of 7.3, and the test solution turbidity steadied out at approximately 1 NTU after 5 days. No precipitates were observed in the solution, but large amounts of white deposits (nominally 0.125 to 0.250 in. in diameter) were observed on the submerged galvanized steel, aluminum, and inorganic zinc-coated steel coupons. Calcium, magnesium, silica, and sodium were prevalent in the Newtonian solution.

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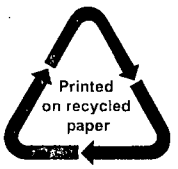

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