# Appendix G

# SEM/EDS and ESEM/EDS Data for Test #3 Day-30 Gel

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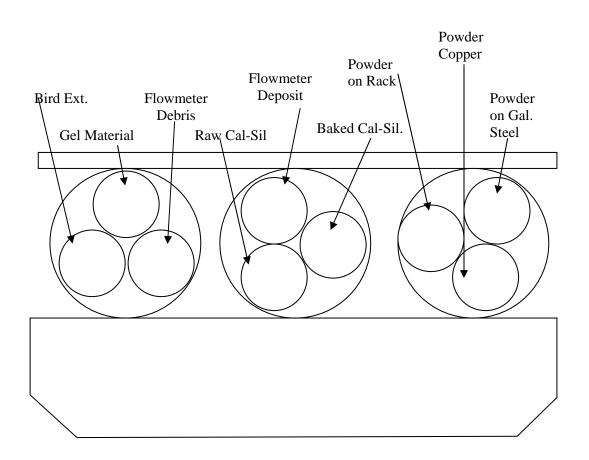
In ICET Test #3, one distinguished phenomenon is the presence of white gel-like precipitates in the testing solution. On the shutting down date of Test #3 deposits of the white gel-like precipitates were observed on the top of the birdcage. These precipitates may increase the containment sump screen head loss during a LOCA, therefore, it is necessary to investigate the morphology and composition of the white gel-like precipitate.

This appendix shows the ESEM/SEM/EDS and XRD/XRF results of the white gel-like precipitates. The precipitates were collected on the date Test #3 was shut down (May 5, 2005). For the SEM examination, the samples were dried in air before being coated with Au/Pd. EDS results provide a semi-quantitative elemental analysis of the sample compositions. Also, XRD results show the crystal structure of the white gel-like precipitates. Based on the XRD results, the composition of the white gel-like precipitates contained crystalline substances of sodium calcium hydrogen carbonate phosphate hydrate ( $Ca_8H_2(PO_4)_6\cdot H_2O\cdot NaHCO_3\cdot H_2O$ ) and lithium calcium hydrogen carbonate phosphate hydrate ( $Ca_8H_2(PO_4)_6\cdot H_2O\cdot Li_2CO_3\cdot H_2O$ ). In addition, XRF results indicate the chemical composition of the precipitates. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

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# **Transcribed Laboratory Log**

<u>Laboratory session from May 9, 2005.</u> Test #3 Day-30 Gel Material

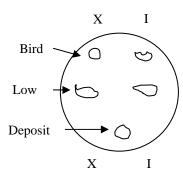


### **Gel Material**

| Image: | T3D30GelMaterial003 | $100 \times$ |                           | Figure G-1 |
|--------|---------------------|--------------|---------------------------|------------|
|        | T3D30GelMaterial004 | 1000 ×       |                           | Figure G-2 |
| EDS:   | T3D30Gel02          |              | Whole screen of image 004 | Figure G-3 |

# **Transcribed Laboratory Log**

### <u>Laboratory session from May 6, 2005.</u> Test #3 Day-30 Gel Material



### **Gel-like Material on Top of Birdcage**

| Image: | t3Gel08  | 1000 × |  | Figure G-4 |
|--------|----------|--------|--|------------|
| EDS:   | t3GelED4 |        | White gel has high C and low Si                | Figure G-5 |
|        | t3geled5 |        | Comparing t3bcexe2 from Appendix C4 & t3GelED4 | Figure G-6 |
|        | t3geled6 |        | EDS of gel material                            | Figure G-7 |
|        | t3geled7 |        | Comparing t3bcexe2 from Appendix C4 & EDS6     | Figure G-8 |

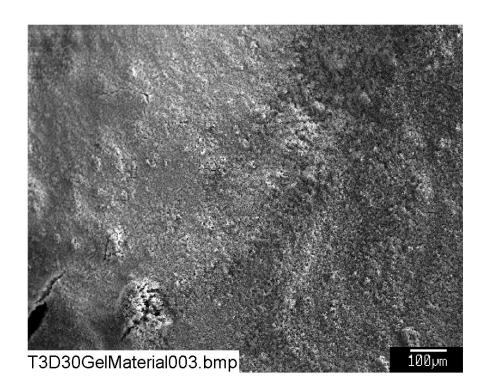


Figure G-1: SEM image magnified 100 times for a Test #3 Day-30 white gel-like material on the top of the birdcage. (T3D30GelMaterial003)

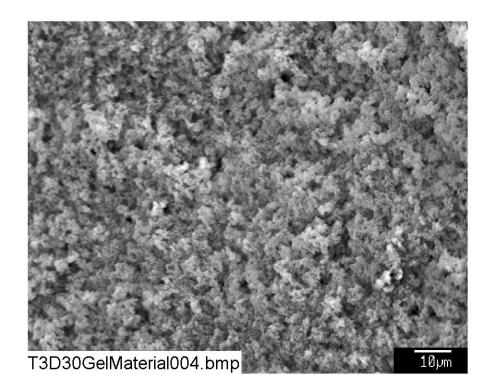


Figure G-2: SEM image magnified 1000 times for a Test #3 Day-30 white gel-like material on the top of the birdcage. (T3D30GelMaterial004)

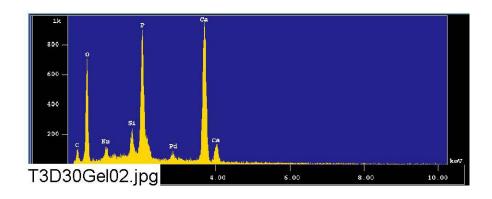


Figure G-3: EDS counting spectrum for the white gel-like material (whole image) shown in Figure G-2. (T3D30Gel02)

The results from the chemical composition analysis for T3D30Gel02 are given in Table G-1.

Table G-1. Chemical Compositions for T3D30Gel02, Figure G-3.

#### May 9 2005

Group : NRC

Sample : T3D30 ID# : 2 Comment : GelMaterial

Condition : Full Scale : 20KeV(10eV/ch,2Kch)

Live Time : 60.000 sec Aperture # : 1 Acc. Volt : 15.0 KV Probe Current : 1.606E-09 A

Stage Point : X=79.625 Y=59.260 Z=11.424 Acq. Date : Mon May 9 11:42:11 2005

|    | ment | Mode   | ROI (KeV)  | K-ratio(%) | +/-    | Net/Back | ground |
|----|------|--------|------------|------------|--------|----------|--------|
| C  | K    | Normal | 0.09- 0.46 | 0.6057     | 0.0005 | 338      | / 119  |
| -  | K    | Normal | 0.25- 0.77 | 12.2043    | 0.0032 | 4587     | / 68   |
| Na | K    | Normal | 0.81- 1.27 | 0.5675     | 0.0010 | 613      | / 50   |
| Si | K    | Normal | 1.50- 2.05 | 0.9391     | 0.0005 | 1366     | / 271  |
| P  | K    | Normal | 1.75- 2.38 | 8.4975     | 0.0055 | 7628     | / 107  |
| Ca | K    | Normal | 3.39- 4.30 | 17.1295    | 0.0038 | 12109    | / 26   |
|    |      |        |            |            |        |          |        |

Chi\_square = 42.7915

| Element | Mass*  | Atomic% | ZAF    | Z      | A      | F      |
|---------|--------|---------|--------|--------|--------|--------|
| C       | 4.355  | 7.8616  | 3.7318 | 1.0194 | 3.6611 | 0.9999 |
| 0       | 45.521 | 61.6928 | 1.9361 | 0.9721 | 1.9917 | 1.0000 |
| Na      | 1.639  | 1.5456  |        |        |        |        |
| Si      | 2.072  | 1.5994  | 1.1451 | 0.9756 | 1.1812 | 0.9937 |
| P       | 13.776 | 9.6435  |        |        |        |        |
| Ca      | 32.638 | 17.6571 | 0.9890 | 0.9947 | 0.9943 | 1.0000 |

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Total 100.000 100.0000 Normalization factor = 1.9265

TOTAL 100.000 100.0000

Normalization factor = 2.1120

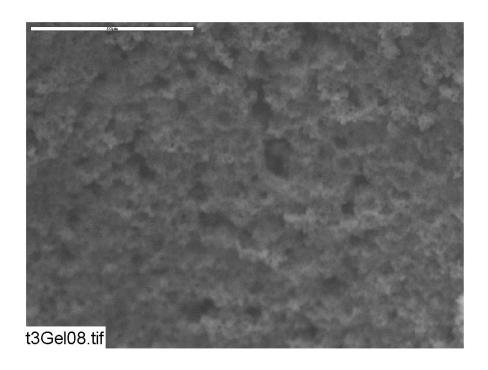


Figure G-4: ESEM image magnified 1000 times for a Test #3 Day-30 white gel-like material on the top of the birdcage. (t3Gel08)

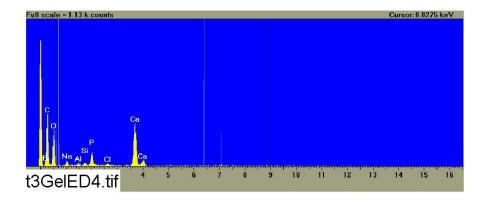


Figure G-5: EDS counting spectrum for the white gel-like material shown in Figure G-4. (t3GelED4)

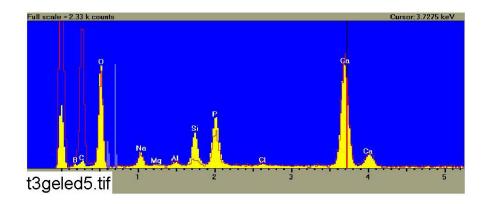


Figure G-6: Comparison of EDS counting spectra for Figure G-5 (red, the gel-like materials shown in Figure G-4) and Figure C4-5 (yellow, the large deposits taken from the birdcage exterior shown in Figure C4-4). . (t3geled5)

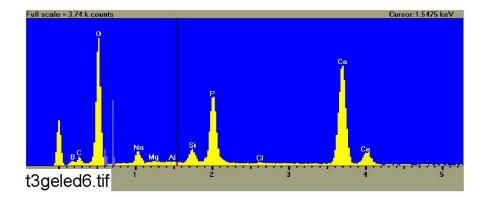


Figure G-7: Another EDS counting spectrum for the white gel-like material shown in Figure G-4. (t3geled6)

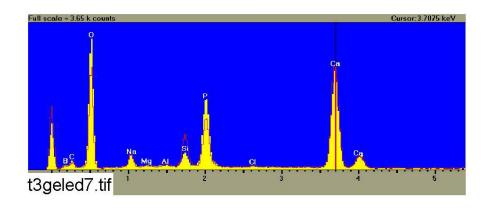


Figure G-8: Comparison of EDS counting spectra for Figure G-7 (yellow, the gel-like materials shown in Figure G-4) and Figure C4-5 (red, the large deposits taken from the birdcage exterior shown in Figure C4-4). (t3geled7)

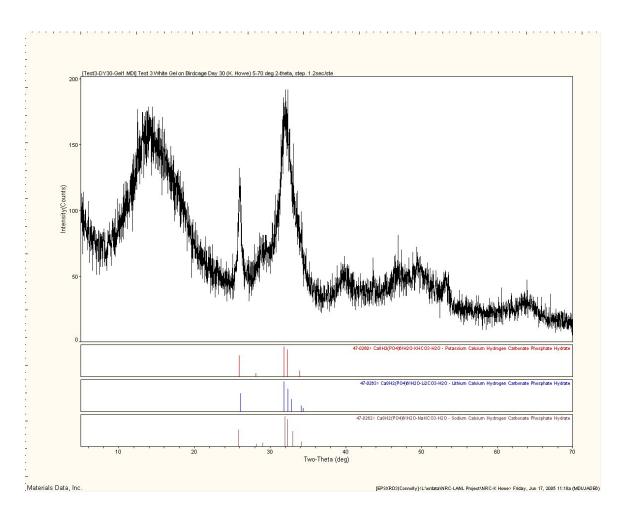


Figure G-9: XRD Results for Test #3 Day-30 White Gel-like Material.

Table G-2. Dry Mass Composition of a Test #3 Day 30 White Gel-like Sample by XRF Analysis

| Sample<br>ID | SiO <sub>2</sub> | TiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | FeO  | MnO  | MgO  | CaO   | Na <sub>2</sub> | K <sub>2</sub> O |      | H <sub>2</sub> O(+)<br>CO <sub>2</sub> | $P_2O_5$ |       | H2O(+)CO2<br>/DF (10) &<br>Cover. To % |
|--------------|------------------|------------------|--------------------------------|--------------------------------|------|------|------|-------|-----------------|------------------|------|--|----------|-------|--|
| Test #3      |                  |                  |                                |                                |      |      |      |       |                 |                  |      |  |          |       |  |
| Day 30       |                  |                  |                                |                                |      |      |      |       |                 |                  |      |  |          |       |  |
| White        |                  |                  |                                |                                |      |      |      |       |                 |                  |      |  |          |       |  |
| Gel          | 5.26             | 0.02             | 0.63                           | 0.07                           | 0.00 | 0.00 | 0.25 | 35.01 | 2.39            | 0.06             | 4.75 | 19.24                                  | 27.09    | 94.77 | 1.0196                                 |