

APPENDIX H

Sediment Analysis for Test #1

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H.1 Introduction

This technical addendum was prepared in response to a request for information submitted to the NRC by the nuclear utility industry through the Electric Power Research Institute (EPRI) regarding specific physical and chemical attributes of the debris types observed in ICET 1 conducted by LANL in the civil engineering department at UNM. The topics addressed here include (a) characterization of T1 sediment recovered from the floor of the ICET test tank with respect to moist/dry mass ratio and qualitative resuspension and settling behavior; (b) SEM images of T1 sediment with qualitative assessment of the fiber to particulate volume ratios, EDS determination of elemental composition, and total hydrated masses recovered from T1.

H.2 Characterization of T1 Tank Sediment

This section documents the qualitative characterization of T1 tank sediment performed on April 13, 2005, at UNM. Recall that T1 was terminated on December 21, 2004. A draft procedure for this examination was provided by EPRI on March 4, 2005, to satisfy two objectives: (1) determine the moist-to-dry mass ratio so that moist-sediment inventories reported after the test was completed can be converted to dry quantities and (2) qualitatively describe the propensity of sediment for resuspension and settling in the T1 tank solution.

H.2.1 Recommended Procedure for Sediment Examination

A recommended procedure was provided as follows. Italicized annotations have been added where needed for clarity.

A. Weighing

1. Obtain a small (approximately 1/2 teaspoon) sample of sediment from one of the four containers. *Sediment from each quadrant of the tank was collected and stored separately.* The sample should be obtained by first removing some of the material on the surface of the sediment in the container. *This process was proposed to avoid uneven moisture content in the event of surface drying.*
2. Weigh the sample without delay and record the weight.
3. Place the sample in an oven and allow to dry at approximately 220°F for 24 hours.
4. Weigh the dried sample and record the results. Label and save the sample.

B. Suspension

1. Obtain another small (approximately 1 teaspoon or less) sediment sample from one of the containers *and weigh while still moist.*
2. Place sediment into an approximately 50-ml sample bottle.

3. Add some decanted fluid from one of the end-of-T1 1-L archival samples, and fill the sample bottle containing sediment about half full. (Do not agitate the archival sample jar. The objective is to obtain decanted fluid without any of the precipitate/sludge that has settled in the archival solution.)
4. Using a glass or SS rod, agitate the sediment and fluid to break up the sediment material and attempt to resuspend it in the liquid. Make notes of how readily the sediment material breaks up and whether it resuspends.
5. Add more decanted fluid to fill the 50-ml sample bottle to about 90% full and place the cap on the bottle.
6. Vigorously shake the bottle to resuspend as much of the sediment as possible.
7. Set the bottle down and observe how much sediment immediately settles to the bottom of the bottle (linear measurement in millimeters on the bottom of the bottle).
8. Record the approximate length of time it takes for most of the material to settle and for the fluid to become visibly clear.
9. Measure how much sediment is in the bottom of the bottle (in millimeters).
10. Report the results, and label and retain the sample bottle with the sediment.

The intent of the draft procedure was followed as closely as possible, and additional measurements were added to track the settling of the fine particulate. However, a check list was not prepared to ensure explicit execution of each step. Deviations from the procedure are noted in the following narrative account of the observations.

- C. In addition to the requested procedures, time-dependent settling of very fine particulates was examined using a turbidity meter. A table of settling rate data is provided with an accompanying plot.

H.2.2 Part A: Wet/Dry Mass Comparison

All T1 sediment was characterized using material from the south quadrant of the ICET tank (87.4 g moist); however, the mass and visual appearance of sediment in each quadrant was consistent. The sample had been stored in a plastic container with a threaded lid. The sediment was still visibly moist, with uniform color and consistency. No free water was present, and no moisture condensation was present on the interior of the container. The aggregate sample of moist sediment appeared wrinkled (accordion folds) and grayish brown in color, much as wet cardboard that has been scraped from a surface. This appearance suggests that a relatively thin layer of material was removed from the bottom of the tank and that constituent fiber may have been present to help hold together the structural mat. Some patches of the debris clearly contained more sand-like

particles than others, and the location of particulates relative to the wrinkled surface suggest that larger amounts of particulate were located near the bottom of the bed.

Using tweezers, a small, penny-sized sample of moist sediment was extracted from the interior of the debris and placed on a tared filter paper and metal sample cup. The sample was weighed on the Mettler AE 200 mass balance located in the Environmental Engineering Laboratory at UNM. The moist sample (measured to be 1.665 g) was placed in an air convection drying oven at 100°F. After 10 minutes in the oven, the surface color changed to ash gray, indicating rapid drying (see Figure H-1 and Figure H-2). After drying overnight for approximately 29 hours, the sample had a dry weight of 0.6395 g, indicating a loss of approximately 62% of the original moist mass. Earlier repetitions of this measurement using larger quantities of sediment indicated losses of 51%. Thus, the ratio between dry sediment mass and moist sediment mass (M_{dry}/M_{wet}) is between 0.4 and 0.5. Similar measurements of T2 sediment exhibited a 48% mass reduction, which is again consistent with a dry-to-moist mass ratio of 0.5.

A larger sediment sample was similarly removed from the interior of the aggregate debris for use in the settling tests. Some effort was made to obtain a representative fraction of both the particulate “mud” and the wrinkled surface layer. The sample was placed directly into a pretared, clean glass vial for testing to avoid any sample losses that might occur during transfer between containers. The sediment material dropped cleanly to the bottom, with very little deposit on the side of the glass vial. The measured moist mass was 4.203 g (see Figure H-3).



Figure H-1. T1 sediment container from south quadrant (right) and a 1.67-g sample for drying (left).



Figure H-2. T1 sediment container from south quadrant of tank (close-up).



Figure H-3. Sediment sample (4.2 g) extracted for settling tests.

H.2.3 Part B: Qualitative Resuspension and Settling Characteristics

The information reported in this section is largely qualitative; however, the basic attributes of container volume, sample mass, and sediment layer thickness may be useful for inferring particle-size information. Some additional desired information will need to be extracted from the photometric evidence that is supplied here. For physical reference, vials of two sizes with similar cylindrical shapes were employed. The volumes of each vial were determined using a calibrated syringe to fill them with distilled water up to the beginning of the glass neck. The small vials have a volume of 32 ml, and the large vial has a volume of 65 ml. The large vial has an inside diameter of approximately 1 in. (2.54 cm) and an outside diameter of approximately 1 1/8 in. (2.86 cm), as measured across the bottom using a steel rule with 1/32-in. (0.79-mm) graduations (shown in later photos). Thicknesses of the glass walls were estimated visually. The small vials have an inside diameter of approximately 7/8 in. (2.22 cm) and an outside diameter of approximately 15/16 in. (2.38 cm).

The recommended procedure requested that settling behavior be observed in supernatant liquid archived from the end of T1. At this time, approximately ten to twelve 1-L bottles of test solution remained. One partially empty bottle (labeled #18 12-21-04) was selected for use. Visible precipitate was present in the bottom of each bottle, so care was taken not to disturb the settled material. Archival solutions are stored at room temperature, and all resuspension/settling tests were conducted at room temperature.

Approximately 30 ml of supernate was removed from the top of bottle #18 using a syringe to avoid disturbing the precipitant at the bottom. This test solution was added to the sediment by tipping the large vial nearly horizontally and slowly injecting the liquid to avoid disturbing the sediment. Little to no evidence of disruption was observed in either the reservoir or the test sample, as indicated by the uniform color (pale yellow), and no visible suspended or swirling matter was observed. The large test vial was approximately half-full with T1 solution and immersed debris (see Figure H-4).

A separate sample of test solution was extracted by syringe to fill a small test vial. The baseline turbidity of this sample was measured to be 2.2 NTU (nephelometric turbidity units) using the Hach ratio turbidimeter present in the ICET laboratory (UNM Civil Engineering hydraulics laboratory). After shaking the vial, turbidity increased to 2.4 NTU.

The potential for resuspension of the immersed sediment material was investigated by sequentially sweeping or picking at the surface of the debris with a glass stirring rod and noting the results. Each interrogation became increasingly invasive until finally the vial was swirled and then eventually shaken. Observations for each level of disturbance are described in the following paragraphs.



Figure H-4. T1 archive solution and large vial used for resuspension/settling tests.

A gentle sweep over the surface of the immersed debris released significant quantities of fine particulate that swirled like “wisps of smoke” in the liquid. This verbal description was applied by two independent observers upon seeing the sample for the first time. A typical eddy that was generated using very little energy released a component of larger particles that fell back to the bed within 15 seconds, and a portion of very fine, visibly indistinguishable particles remained suspended for the duration of these examinations.

After two or three stronger sweeps through the surface layers of the sediment, a uniform brown haze was visible in the top 2 cm of solution above the sediment. However, because the vial had not yet been uniformly mixed, the dirty layer sat beneath the “clean” solution with a well-defined interface at the mixing boundary. Gently rocking the vial side to side like a pendulum tended to settle the debris pile more uniformly and to separate some of the larger particles to the bottom. These exams left the impression that the sediment present in T1 was very loosely aggregated. It may be that the constituent soil and concrete dust added to the test as latent debris simulant would behave in much the same manner. Extremely fine particulates are easily released from the sediment sample with almost any degree of small agitation; thus, the fiber fraction, confirmed to be present by SEM examination, and any chemical constituents (if present) do not appear to enhance the cohesiveness of the sediment layer collected from T1 (see Figure H-5 and Figure H-6).



Figure H-5. T1 sediment in T1D30 T1 test solution after gently probing surface layers of debris. Note the brown haze of suspended particulate below the relatively clean solution.

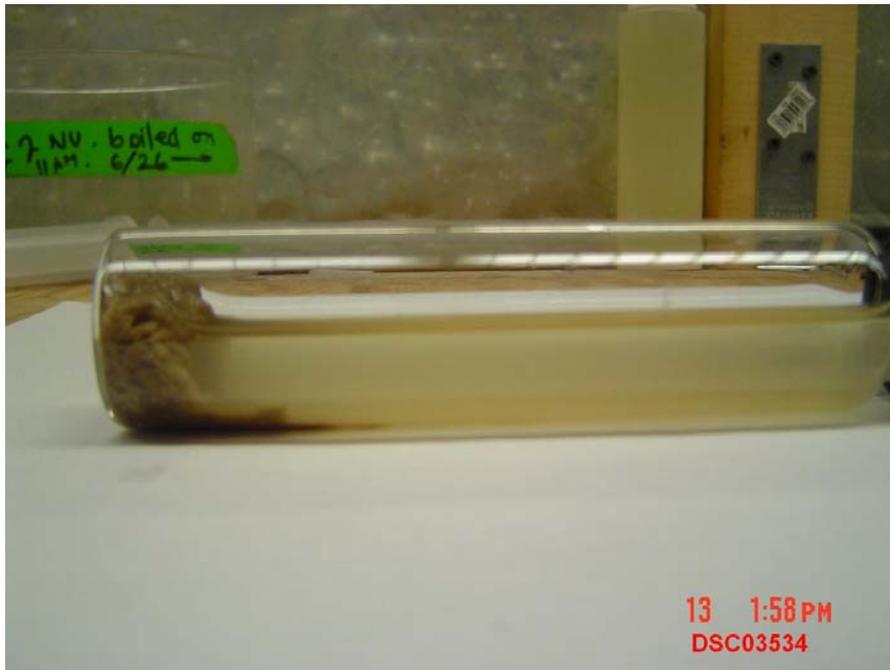


Figure H-6. Horizontal sediment vial showing mechanical behavior of a slumping bed before shaking.

When swirling the vial to the point of uniformly mixing the brown color, the solids behave like a suspension of river-bed silt. After significant agitation is stopped, the solids immediately stratify into well defined layers by size and color. At the bottom of the vial were found (a) the larger visible grains, followed by (b) a layer of dark brown “mud.” The largest portion of the vial in the middle, (c), was uniformly brown in color and appeared to be homogeneous in mixture. Near the top was (d), a layer of relatively clean semitransparent solution with a well-defined boundary separating it from the homogeneous mixture in the middle of the vial. The bottom sediment layer and the top clean layer grow with settling time as the middle layer shrinks through deposition and settling. Figure H-7 shows the vial after vigorous swirling and approximately 5 minutes of settling.

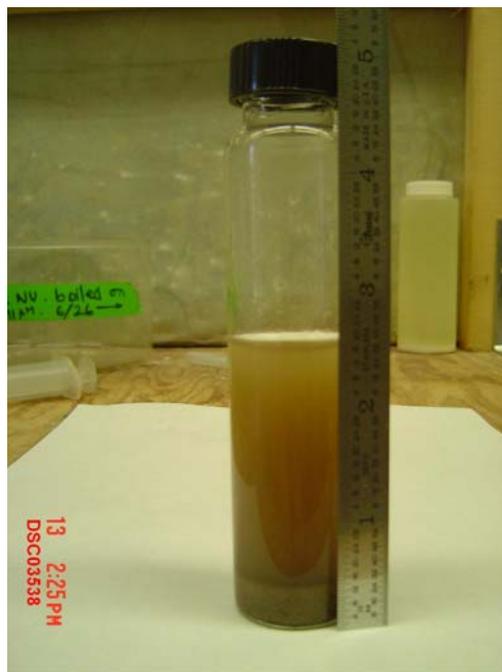


Figure H-7. Large sediment vial after thorough mixing and 5 minutes of settling.

Manually shaking the vial to the point of air entrainment and bubble formation had the somewhat surprising effect of floating fibrous material to the top of the column. Small clumps, networks, and strands of very fine fibers were visible in the foam residue on the glass around the top of the mixture.

Next, the large test vial was filled to the neck with additional T1 archival solution, and the agitation tests were repeated. Additional liquid diluted the stratified layers to make the internal structures more visible, but the qualitative separation described previously was the same. At the deposition interface between layers (b) and (c), the material appeared to collect in a loosely aggregated bed that was almost “fluffy” in appearance. The grayish color of this material is suggestive of the original description of sediment in

the storage container as wet shredded fiber board. Figure H-8 shows the full vial after approximately 5 minutes of settling.



Figure H-8. Large sediment vial after filling, thorough mixing, and 5 minutes of settling.

H.2.4 Part C: Time-Dependent Turbidity Measurements

Steps 7–9 of the recommended settling procedure do not provide specific criteria for desired settling times or fluid clarity. Furthermore, although an apparently large portion of the sediment mass was observed to settle on the time scale of minutes, a significant portion appeared to be settling on a time scale of hours. Given the limited time available for this examination, an alternative method was applied using time-dependent turbidity measurements to characterize settling rates.

The large vial filled with solution and sediment was shaken and allowed to settle for 11 minutes, as described previously. A syringe was used to extract approximately 32 ml of solution (about half) from the homogeneous middle layer of the vial without disturbing the aggregate layers near the bottom. This volume was sufficient to fill a single small test

vial for direct turbidity measurement. Upon thoroughly shaking the small vial, the measured turbidity exceeded the 200-NTU range of the instrument.

Approximately one half of the shaken contents of the small vial was poured into a second small vial and diluted with tap water. It was noted before shaking that the clean tap water remained stratified above the suspension of sediment. Figure H-9 shows the first dilution vial before and after shaking. The turbidity of clean tap water was measured to be 0.8 NTU. The turbidity of the diluted suspension still exceeded 200 NTU, so the dilution process was repeated a second time and a third time before the turbidity could be successfully measured. At this point, suspended solids were visible only as a slight discoloration, with an initial turbidity of 110 NTU, as shown in Figure H-10.

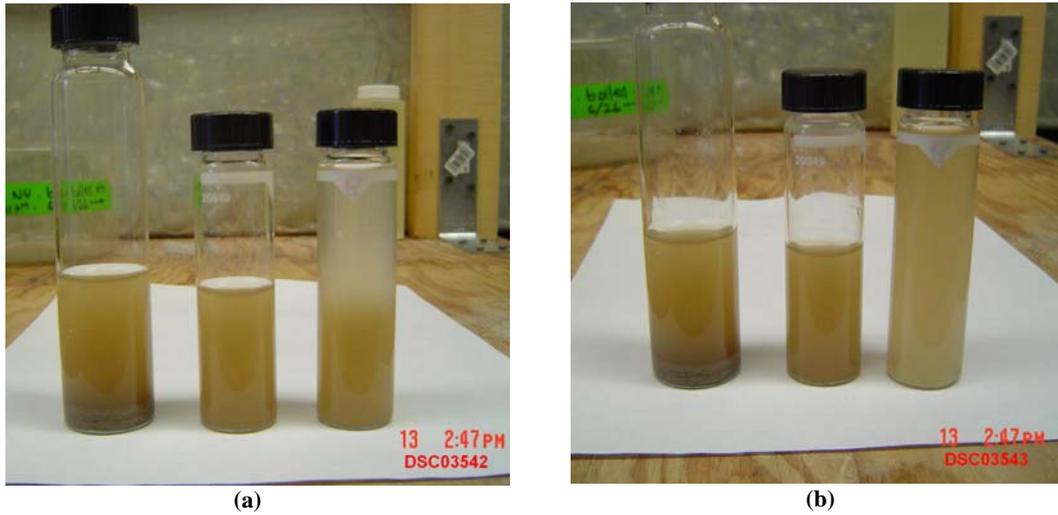


Figure H-9. Dilution vial to the right before (a) and after (b) shaking.



Figure H-10. Successive stages of dilution just before measuring turbidity. From left to right, the first two vials are original concentration followed by the first, second, and third dilution, respectively.

The time-dependent turbidity of the successively diluted sample was monitored periodically for 60 minutes without removal from the turbidimeter, and again after 17 hours of settling. Figure H-11 shows the test vials after approximately 40 minutes of settling time, and Figure H-12 shows the test vials again after approximately 17 hours. Material sufficient to cover the bottom of each vial was noted in every sample. The depth of debris in the original large vial after settling overnight was measured to be 7.94 mm in a water column 8.10 cm in height. The turbidity of the last diluted sample had decreased to 17.6 NTU. Time-dependent turbidity data are tabulated in Table H-1 and are illustrated in Figure H-13.

At the conclusion of this examination, the contents of the first small turbidity vial (undiluted) were returned to the large sample vial and archived as requested. The mass fractions present in the remaining vials were not determined but are judged to be small. This information could be recovered at any time by filtering, drying, and weighing the contents of the large vial to compare the initial mass introduced with the mass recovered after the tests.

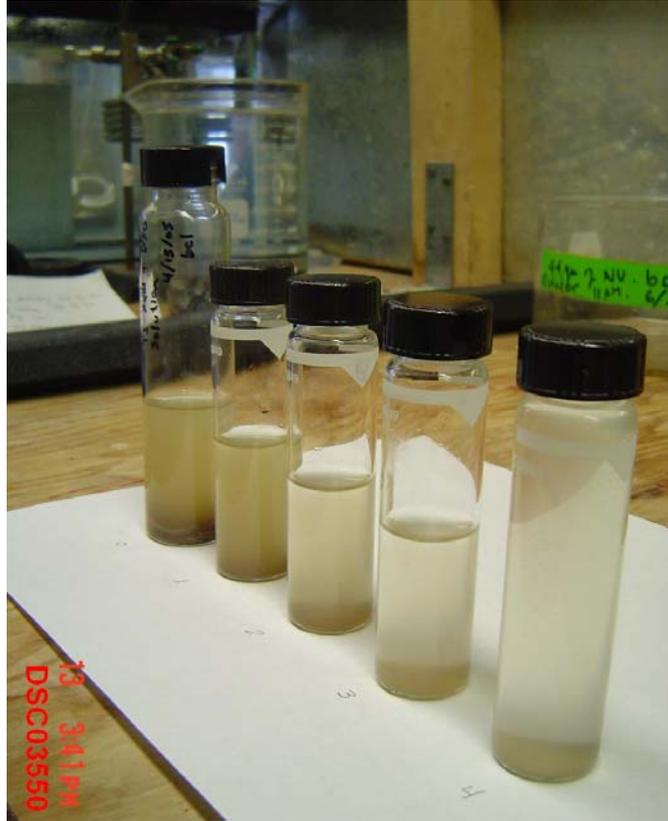


Figure H-11. Sediment vials after successive dilution and 40 minutes of settling.



Figure H-12. Sediment vials after successive dilution and approximately 17 hours of settling.

Table H-1. Time-Dependent Turbidity Data for Settling of T1 Sediment

Time (minutes)	Turbidity (NTU)
0	110
2.5	105
5	103
11	100
17	95
35	79
40	77

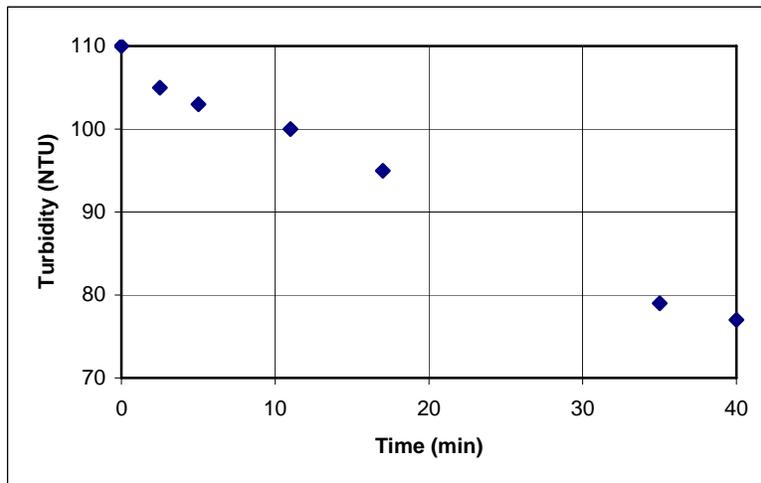


Figure H-13. Time-dependent settling behavior of T1 sediment as indicated by turbidity measurements.

H.3 Sediment Images, EDS Composition, and Quantities

Each ICET test is initiated with a background loading of latent debris composed of crushed concrete and dirt. In T1, this material was observed to settle completely on the floor of the tank over the course of several days. This particulate, in combination with fugitive fiberglass strands, form the basic substrate of the sediment layer recovered from the tank at the end of each test. In addition, this bed may serve as a repository for chemical products that are either formed in the bed or deposited on top via settling over the course of the tests. Among all of the sample types collected during ICET, tank sediment is the most heterogeneous in terms of both physical configuration and elemental composition. After draining the tank and manually recovering the sediment, 339 g was collected following T1. This is the total mass as measured when thoroughly drained by gravity of free water but while still moist.

Figure H-14 illustrates the complexity of sediment collected from T1. This view suggests that a significant amount of fiberglass is present in the debris. Qualitative visual estimates of the fiber fraction might range from 60% to 75% fiber by volume. Note that visual assessments can compare only the volume ratios and not the mass ratios. The fiber present at the bottom of the ICET tank represents fiberglass that has escaped the SS mesh bags that were constructed to hold the primary volume of this debris type. In the containment pool, the fiber-to-particulate ratios might vary greatly by location and may not resemble the ratios suggested by this image.

Figure H-15 through Figure H-17 present a set of increasing magnification images that successively focus on a clump of particulate material. This set of images highlights the wide range of particle sizes present in the ICET sediment layer, ranging from 30 μm down to submicron particles attached to individual fibers. The close-up provided in Figure H-17 suggests that some of the larger observed particles actually may be agglomerates of smaller constituents.

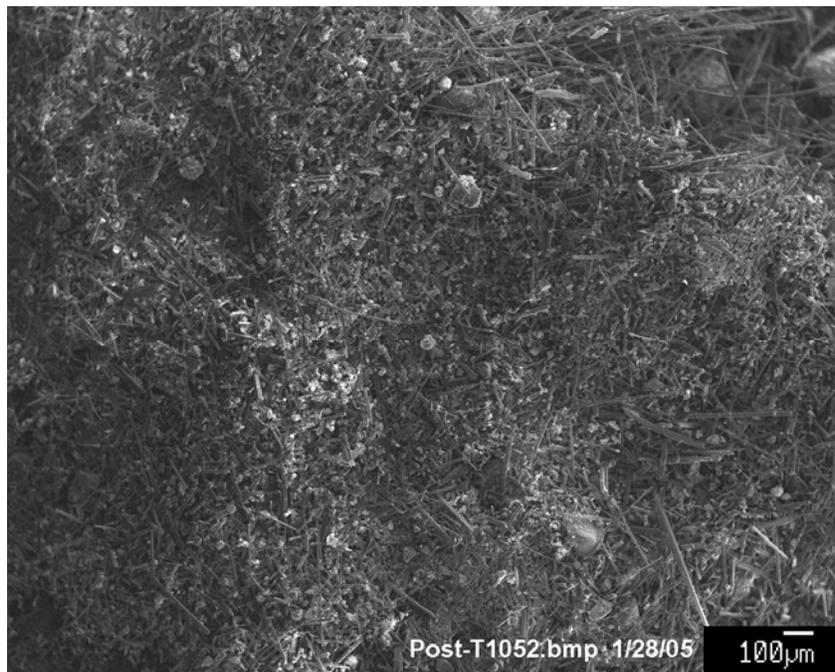


Figure H-14. T1D30 tank sediment showing combination of fiber and particulate, magnified 43 times.

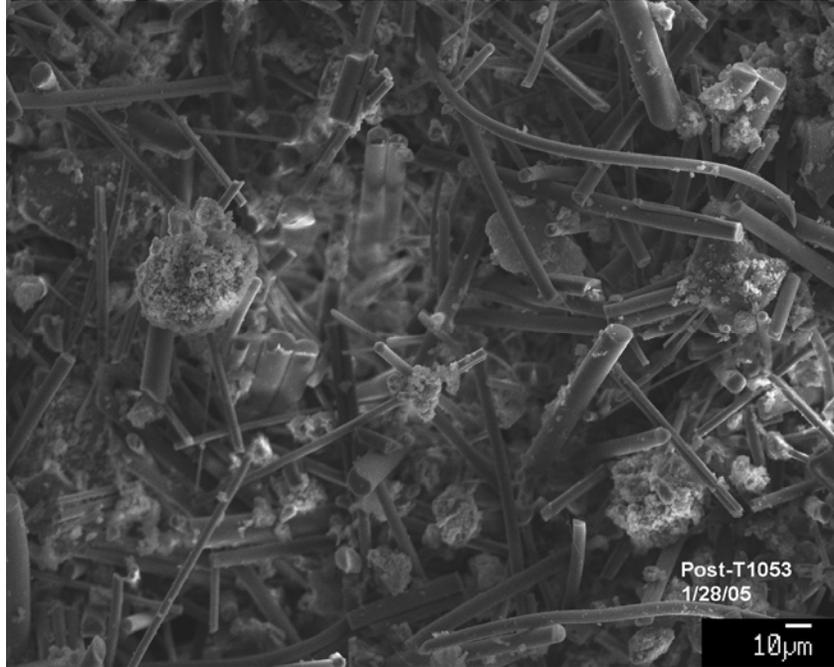


Figure H-15. T1D30 tank sediment focused on two large particles in Figure H-14, magnified 370 times.

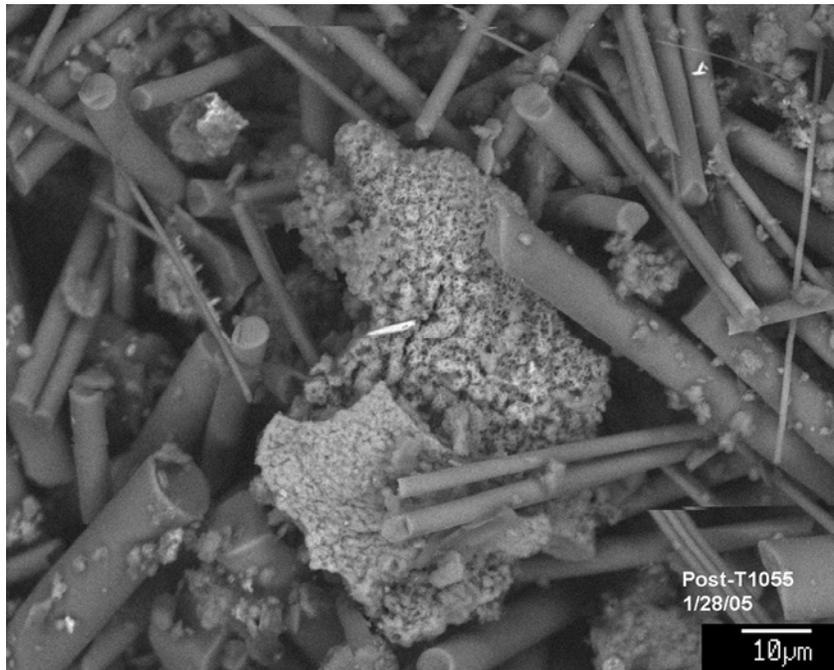


Figure H-16. T1D30 sediment on a typical fuzzy particle, as shown in Figure H-15, magnified 1000 times.

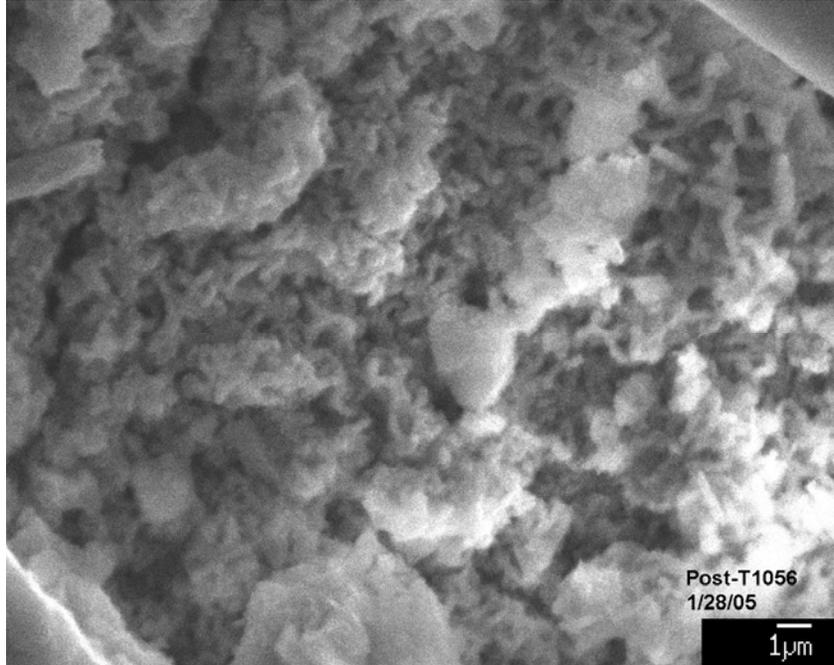


Figure H-17. T1D30 sediment magnified 5000 times on the particle, as shown in Figure H-16.

Figure H-18 and Figure H-19 illustrate the variety of elemental compositions observed in the tank sediment. Note that a sputter coating of gold (Au) and palladium (Pd) is applied during SEM sample preparation; thus, these elements are always present in the spectrum. Given the presence of crushed concrete aggregate and common dirt present in the tank, it is not surprising to find a wide variety of mineral constituents.

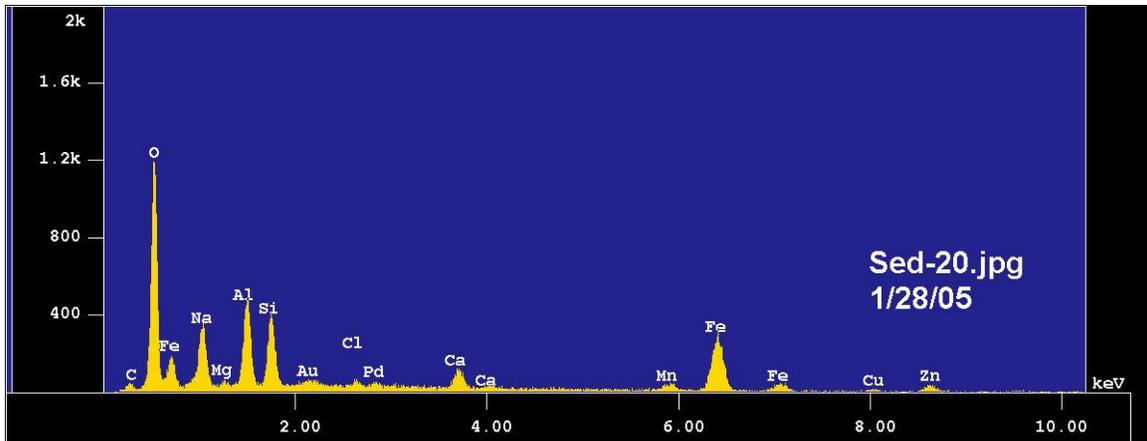


Figure H-18. EDS spectrum for the particle shown in Figure H-17.

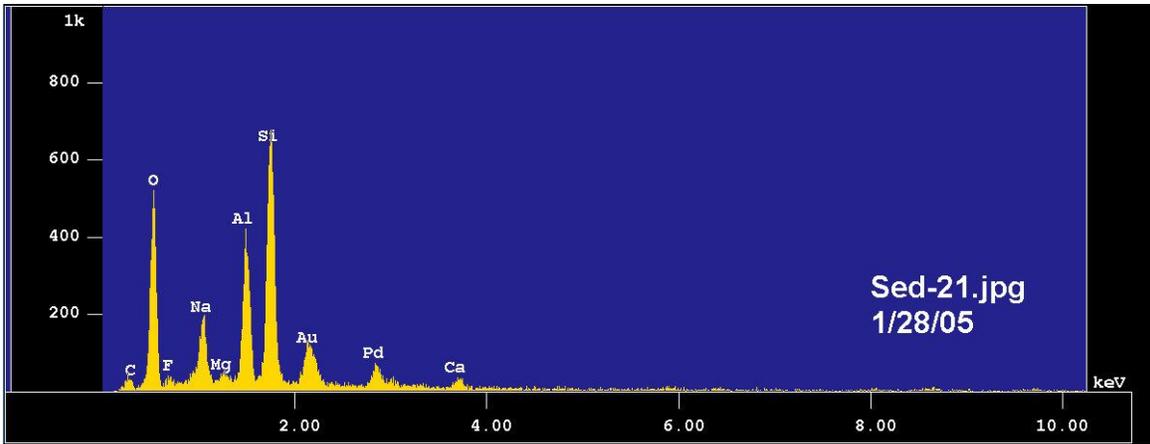


Figure H-19. EDS spectrum for another representative particle present in T1D30 tank sediment.

Figure H-20 illustrates another typical T1 sediment sample containing minerals such as quartz and possible chemical products. Points EDS-23 and EDS-24 marked in the figure exhibit dominant proportions of oxygen, sodium, silicon, and aluminum, consistent with the composition of white precipitant observed in T1 water samples when cooled (see Figure H-21). It is impossible to determine whether these flakes originated in the sediment bed or were dislodged from other surfaces while draining the tank.



Figure H-20. T1D30 tank sediment showing the presence of fiber, minerals (quartz) and possibly chemical products (EDS-23 and EDS-24), magnified 40 times.

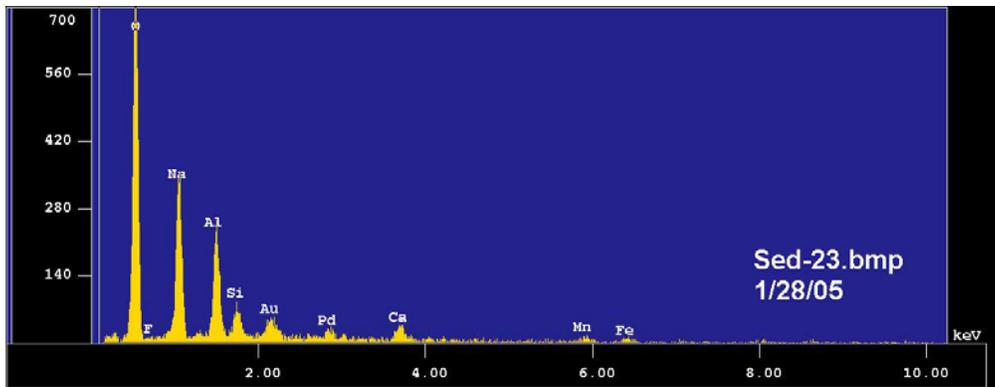


Figure H-21. EDS spectrum of point EDS-23 shown in Figure H-20 containing oxygen, sodium, aluminum, and silicon ratios similar to T1 precipitant.