



Selenium Sediment Quality Sampling for the Las Vegas Wash and Tributaries: 2006 Sampling

*(In Support of the Bioassessment Monitoring Plan for Las Vegas Wash and Tributaries,
Las Vegas Wash Coordination Committee)*

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December 2007

Prepared by
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Prepared for
Southern Nevada Water Authority

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ABSTRACT

The Las Vegas Wash (Wash) receives water and groundwater from a variety of sources, including storm water, urban runoff, shallow groundwater, and treated wastewater from the entire Las Vegas Valley. To evaluate the potential for contaminant accumulation in the Las Vegas Wash sediments, sampling of sediments was conducted along the Wash and selected tributaries. A physicochemical characterization of the collected sediments was completed, including particle size distribution, specific surface area, mineralogy, and morphology. The sediments were also analyzed for selenium, one of the elements of most concern in the Las Vegas area, specifically, and the U.S. Southwest, in general. Appropriate sampling techniques were employed to obtain representative samples and to include fine particle size fractions. The measured selenium concentrations were evaluated relative to published values of concern associated with sediments. In general, particle surface area was associated primarily with particles in the micrometer range. The sediments were composed mostly of quartz, feldspars, amphiboles, carbonates, biotite, and a zeolite mineral. Electron microscopy analysis confirmed these results. Sediment specific surface areas ranged from 5.96 to 16.03 m²/g. The aqueous concentrations of selenium in the supernatant ranged from approximately 4 ppb in the Las Vegas Bay sample to 8.1 ppb in the Nature Preserve sample. The selenium sediment concentrations ranged from 1.7 ppm in the LV Wash 0.8 sample to 4.3 ppm in the Duck Creek sample. Most of the selenium sediment values fall between the suggested values of concern and effect, 1 and 4 ppm, respectively. These results suggest a more complex relationship between selenium sediment concentration and aqueous concentrations in the Las Vegas Wash and tributaries. Future additional sampling and analysis would be required to monitor concentration trends as a function of time, location, and management approaches.

ACKNOWLEDGEMENTS

The contributions of Tracy Boettcher, Rebekah Harris-Burr, and Racheal Johnsen during the planning, sample collection, and sample analysis phases of this project are gratefully acknowledged. Support for this work was provided by the Southern Nevada Water Authority under Contract SNWA 3200-19101-500-R3134. The support of Peggy Roefer, and Xiaoping Zhou is gratefully acknowledged. The skillful word processing work of Debi Noack is greatly appreciated.

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CONTENTS

ABSTRACT	iii
ACKNOWLEDGEMENTS	iii
LIST OF FIGURES	vi
LIST OF TABLES	vi
INTRODUCTION	1
METHODS	2
Sampling Locations	2
Designation of Sample Nomenclature	2
Sample Collection Methods.....	2
Decontamination Procedures.....	5
Quality Assurance/Quality Control	5
Analytical Methods.....	5
RESULTS AND DISCUSSION	6
Physicochemical Sediment Characterization.....	6
Particle Size Distribution	6
Specific Surface Area.....	8
Mineralogical Composition.....	12
Particle Morphology and Composition	15
Selenium Analysis	24
Assessment of Selenium Concentrations in Sediments.....	29
SUMMARY AND CONCLUSIONS	31
REFERENCES	32
APPENDIX A: Particle Size Distribution	35
APPENDIX B: Mineralogical Composition.....	43
APPENDIX C: Particle Morphology and Elemental Composition	51

LIST OF FIGURES

1. Map of the Las Vegas Wash area showing the location of the sampling sites. 3

2. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Las Vegas Bay sample..... 7

3. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Las Vegas Wash 10.75 mile sample..... 9

4. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Duck Creek sample..... 10

5. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Nature Preserve sample. 11

6. Semiquantitative mineralogical composition of sediment samples. 15

7. General view of the Las Vegas Bay duplicate sample. 16

8. Scanning electron micrograph and EDX spectrum of the Las Vegas Bay sample (magnification 3,000x). 17

9. Scanning electron micrograph and EDX spectrum of the Las Vegas Bay duplicate sample (magnifications 250x and 800x). 18

10. Scanning electron micrograph and EDX spectrum of the Las Vegas Bay duplicate sample (magnification 1,100x)..... 19

11. Scanning electron micrograph and EDX spectrum of the Las Vegas Wash 0.8 mile sample (magnification 400x)..... 20

12. Scanning electron micrograph and EDX spectrum of the Las Vegas Wash 0.8 mile sample (magnifications 600x and 1,200). 21

13. Scanning electron micrograph and EDX spectrum of the Las Vegas Wash 6.05 mile sample (magnification 600x)..... 22

14. General view of the Las Vegas Wash 10.75 mile sample.. 23

15. General view of the Nature Preserve sample.. 23

16. Concentrations of soluble selenium, selenium associated with oxides, and total selenium ($\mu\text{g/g}$). 27

LIST OF TABLES

1. Location of sampling sites..... 3

2. Nomenclature used for sample description. 4

3. Sediment specific surface area (m^2/g), as determined by nitrogen adsorption. 12

4. Semiquantitative mineralogical composition of sediment samples. 14

5. Selenium concentrations of aqueous samples ($\mu\text{g/L}$)..... 26

6. Concentrations of soluble selenium, selenium associated with oxides, and total selenium ($\mu\text{g/g}$) in sediment samples. 27

INTRODUCTION

The Las Vegas Wash (Wash) is the final collector of water from the entire Las Vegas Valley watershed, including storm water, urban runoff, shallow groundwater, and treated wastewater from three wastewater treatment plants in the Las Vegas Valley. The variety of inflows into the Las Vegas Wash has the potential for accumulation of both inorganic and organic pollutants in the sediments.

To determine baseline conditions regarding contaminant accumulation in the Las Vegas Wash and tributaries, a comprehensive monitoring plan was developed by the Las Vegas Wash Coordination Committee (LVWCC). The Bioassessment Monitoring Plan for the Las Vegas Wash and Tributaries was developed to provide a “snapshot” for the concentrations of contaminants of concern in the Las Vegas Wash and tributaries. The media that are being sampled include water, sediments, bird eggs, aquatic species, and plants. The sampling conducted here focused on one of these media, sediments.

The overall objective of the sediment sampling program was to provide a baseline dataset for the concentration of inorganic and organic pollutants in sediments along the entire Las Vegas Wash and selected tributaries. The concentrations in the sediments would then be correlated to the concentrations of the same constituents in the other media, to establish whether there is a reason for concern, based on concern levels established for wildlife. These general objectives were broken down into the following more specific objectives.

- To conduct a one-time sediment sample collection along the Las Vegas Wash and selected tributaries to obtain a baseline dataset of concentrations of organic and inorganic contaminants of concern in sediments.
- To compare the concentrations for all constituents analyzed with generally accepted levels of concern for wildlife, based on recommendations of the U.S. Fish and Wildlife Service and other published documents.
- To compare the concentrations for the same constituents as a function of location along the Las Vegas Wash and selected tributaries to determine if there are any geographical areas of concern.
- In the case the results suggest a level of concern for wildlife, to provide guidance with respect to determining the level and pathway of exposure and help reduce or mitigate this exposure.

The original sampling to meet these objectives was conducted in 2003 (Papelis 2004). Renewed sampling was conducted in 2006 to obtain a new set of results and to compare the new dataset to the first dataset to determine any possible concentration trends. As part of the sampling effort, a complete physicochemical characterization of the sediments was conducted. The physicochemical characterization was considered vital to the interpretation of the occurrence and distribution of potential contaminants in the Las Vegas Wash area. The entire suite of constituents was analyzed again; however, only the sediment characterization and selenium results are discussed here. All other constituents were analyzed by an external laboratory and the results are reported elsewhere. Following this introduction, sampling and analytical methods are described, followed by presentation and discussion of results.

METHODS

Sampling Locations

Sediment sampling was conducted along the entire Las Vegas Wash and selected tributaries in the locations listed below. The sampling locations were essentially the same, to the extent possible, to the initial sampling locations (Papelis 2004) These six sampling locations included:

- LVB. This site is located in the Las Vegas Bay Delta (as of April 2006).
- LW 0.8. This site is located underneath the Northshore Road bridge, downstream from Lake Las Vegas and is very close to the endpoint of the Las Vegas Wash.
- LW 6.05. This site is located upstream from the Pabco Road erosion control structure and downstream of all three municipal wastewater treatment facilities.
- LW 10.75. This site is located below the confluence of Flamingo Wash, Las Vegas Creek historic channel, and Sloan Channel. The site is located upstream of all three municipal wastewater treatment facilities.
- DC. This site is located in Duck Creek below Broadbent Road and has potentially high contaminant concentrations because of urban runoff.
- NP. This site is located at the Nature Preserve at the Clark County Wetlands Park.
- All sampling locations are shown on the Las Vegas Wash map shown in Figure 1. The exact GPS coordinates for each sampling site are given in Table 1.

Designation of Sample Nomenclature

A nomenclature system was created for identifying the source of each sample, the method of collection, and the analysis to be conducted (Table 2). Examples of the nomenclature used are also given in Table 2. The nomenclature was designed so that one string of alphanumeric characters would be sufficient to fully describe the sample origin, sample processing, and subsequent analyses. Sample identifiers were applied to each sample using bottle-specific labels, which were then covered with clear tape to avoid label destruction from moisture.

Sample Collection Methods

Samples were collected at the banks of each sampling location, at the bank/stream interface. Locations were chosen to avoid stagnant pools. Soil samples were collected in accordance with requirements published by the US Environmental Protection Agency (EPA) (U.S. Environmental Protection Agency 1995).

Samples collected for semi-volatile organic analyses (SVOAs), metals and other inorganics, herbicides, and pesticides were taken from representative locations immediately adjacent to the bank/stream interface. To obtain a more representative sample, at each sampling site, a total of five samples were collected; one from a central location in the center of a square and four more from the corners of the square. (U.S. Environmental Protection Agency 1995) The edge of the square was approximately 10 meters, or less, where appropriate, depending on the width of the Las Vegas Wash at the particular sampling location.

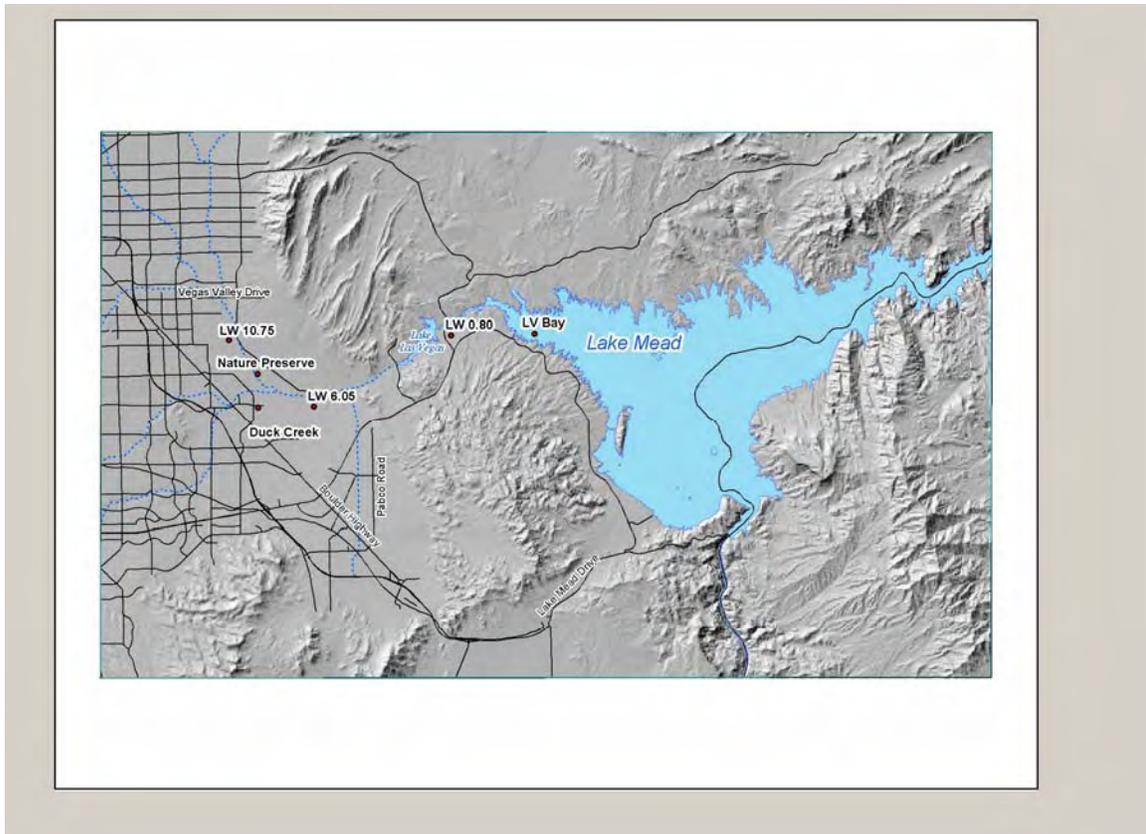


Figure 1. Map of the Las Vegas Wash area showing the location of the sampling sites.

Table 1. Location of sampling sites.

Site	Distance (mi)	Site Label	Location		Elevation (ft)
Las Vegas Bay	0	LV00.0002	36° 07.419 N	114° 51.833 W	1,137
Las Vegas Wash	0.8	LW00.8002	36° 07.334 N	114° 54.287 W	1,239
Las Vegas Wash	6.05	LW06.0502	36° 05.295 N	114° 59.211 W	1,528
Las Vegas Wash	10.75	LW10.7502	36° 07.854 N	115° 02.086 W	1,663
Duck Creek		DC00.0002	36° 05.517 N	115° 01.264 W	1,598
Nature Preserve		NP00.0002	36° 06.451 N	115° 01.480 W	1,637

Table 2. Nomenclature used for sample description.

<p>AA = Two-character designation for site</p> <p>LV – Las Vegas Bay LW – Las Vegas Wash DC – Duck Creek</p>
<p>bb.bb = Distance along the Wash – designated in miles from Las Vegas Bay</p>
<p>cc = Sampling trip number</p>
<p>w =Location (within the site)</p> <p>1 – subsite 1 2 – subsite 2 3 – subsite 3 4 – subsite 4 5 – subsite 5 6 – composite of five sites</p>
<p>x = Analysis</p> <p>1 – VOAs 2 – SVOAs/pesticides 3 – metals and other inorganics 4 – herbicides</p>
<p>y = Source</p> <p>1 – Grab 2 – Composite 3 – Core 4 – Replicate 5 – Equipment Blank</p>
<p>z = Laboratory</p> <p>1 – Lab #1 (mail laboratory chosen to perform the analysis) 2 – Lab #2 (alternate laboratory chosen for QA/QC purposes) 3 – DRI</p>
<p>Examples:</p> <p>LW10.7501-1133: LW10.75 site, first time sampling at a location in the Wash 10.75 miles upstream, subsite 1,VOA analysis using core sample, analyzed by DRI.</p> <p>LV00.0003-6321: LVB site, third time sampling at the mouth of the Las Vegas Bay, 00.00 miles upstream, composite sample, metal and inorganic analysis using composite, analyzed by Lab #1.</p>

Samples were collected from the upper approximately 15 cm (6 inches) of sediment, and composited in a stainless steel bowl using a stainless steel spoon. A sufficient volume of soil was taken for filling three 250 mL sampling jars. The threads of the vials were cleaned with disposable paper towels before the vial was sealed. Sample labels, chain of custody seals, and clear tape were then applied to the vial before storing in an ice-filled cooler.

These sampling methods were designed to allow sampling of a large range of particle size fractions, including the finer material. Collecting the finer material is particularly important because the smaller the particle size, the larger the specific surface area of the particles (Gregg and Sing 1982). Increased surface area is associated with increased potential for interaction between sediments and organic or inorganic constituents. It is therefore expected that in terms of the worst case scenario, the highest concentrations of analyzed constituents would be associated with the finer particles.

Decontamination Procedures

All materials that contacted soil or sediment (mixing bowls, spoons, etc.) were decontaminated at each site after sampling using standard procedures (EPA document #540/P-91-006, Sampling Equipment Decontamination, SOP #2006). Decontamination procedures used a series of washtubs. The first tub contained tap water and Liquinox soap. The second tub contained deionized water. Sampling instruments were then sprayed down with ethanol, and then cleaned a final time using deionized water. After air-drying, sampling instruments were wrapped in paper towels and stored in the vehicle until used at the next site. Only personnel wearing latex gloves handled sampling instruments and sampling vials.

Quality Assurance/Quality Control

Standard quality assurance/quality control (QA/QC) procedures were followed throughout sample collection, shipment, and analysis. Equipment blanks were collected, following equipment decontamination, at sampling sites by pouring deionized water over sampling equipment and collecting the rinsate in respective vials or jars. Chain of custody procedures were followed throughout the entire sample collection and analysis. All samples from all six sampling sites were analyzed at Del Mar Analytical Laboratories. Another complete set of samples was used by DRI for the sediment characterization and selenium analysis. Finally, another set of samples (from the Las Vegas Bay sampling site) was also independently analyzed at DRI laboratories to check the reproducibility of results.

The EPA certified laboratory (Del Mar Analytical) follows EPA approved QA/QC procedures, including use of standard operating procedures, regularly maintained and calibrated instruments, use of matrix spikes, duplicates, spike recovery, calculation of method detection and reporting levels, laboratory fortified blanks, and chain-of-custody documentation.

Analytical Methods

The specific analytical procedures for sediment characterization will be discussed in more detail in the following section. For selenium, both a total digestion procedure was used (EPA Method 3050B), as well as a sequential extraction procedure, designed to estimate the fraction of selenium considered as soluble or easily exchangeable, as well as the fraction of selenium associated with oxides (mostly iron oxides). More detailed information is provided in the selenium analysis section. All aqueous or extracted selenium samples were analyzed

by Graphite Furnace Atomic Absorption Spectroscopy (GFAAS), using a Perkin Elmer 4110 ZL spectrophotometer with Ziemann background correction. The selenium analytical procedures included analysis of duplicates and spike recovery.

RESULTS AND DISCUSSION

The sediment characterization results will be discussed first, followed by the selenium analysis results.

Physicochemical Sediment Characterization

Particle Size Distribution

It is well known that the association of metals and other contaminants with soils and mineral surfaces is a function of the total number of reactive sites available. The total number of reactive sites is directly proportional to the specific surface area of soils, i.e., the surface area per unit mass (Stumm 1992). In addition, the specific surface area of soils is inversely proportional to the particle size of soil grains or sediments (Gregg and Sing 1982). It is therefore expected that finer particles are likely to be associated with the highest concentrations of organic and inorganic constituents.

A conscientious effort was therefore made during sampling to include the finer particle size fractions in the collected sediments. Large particles, although they account for the overwhelming majority of sample mass, they contribute very little to the specific surface area of the sample. On a per-mass basis, therefore, millimeter and larger particles are not expected to be a significant source of either organic or inorganic contaminants. Thus, in order to capture most of the potentially available concentration of selenium in these samples, we focused our attention the smaller size fractions, as will be discussed below. A characteristic particle size distribution curve is shown in Figure 2, which shows the volume frequency percentage and surface area population (m^2/g) of Las Vegas Bay sediments vs. particle diameter. The particle size distribution for all samples was obtained using a Micromeritics Saturn DigiSizer 5200. All analyses were conducted in duplicate.

It can be seen clearly by inspection of Figure 2a, that the particle size distribution by volume is centered around approximately $100\ \mu\text{m}$, although particles range in size from approximately $800\ \mu\text{m}$ down to approximately $0.1\ \mu\text{m}$. The upper end size cut off can be easily explained, because larger particles were removed from all samples by sieving. As pointed out above, particles larger than $1\ \text{mm}$ contribute very little to the surface area of the sample and therefore to the fraction of any constituent associated with these larger particles. Based on Figure 2a, it is clear that particles of sufficiently small size were present with which chemical constituents of potential concern would be presumably associated. As mentioned above, however, specific surface area increases dramatically as particle size decreases. This is manifested in Figure 2b, which shows the surface area contribution of the different size fractions.

It can clearly be seen by inspection of Figures 2a and 2b that although by volume the mode (highest number in the distribution) of the particle size distribution lies around $100\ \mu\text{m}$, and a relatively small percentage of volume is associated with particles smaller than $10\ \mu\text{m}$, particles smaller than $10\ \mu\text{m}$ essentially account for the total surface area associated with these particles, and the majority of the surface area is actually associated with particles

smaller than 2 μm , generally considered to make up the clay fraction (Bohn et al. 1985). This observation is not surprising, given that clay particles are known to have large surface areas, up to 900 m^2/g for smectite clays (Van Olphen and Fripiat 1979). It should be noted, however, that the estimate of specific surface area shown in Figure 2, as a function of particle size, is obtained assuming spherical nonporous particles.

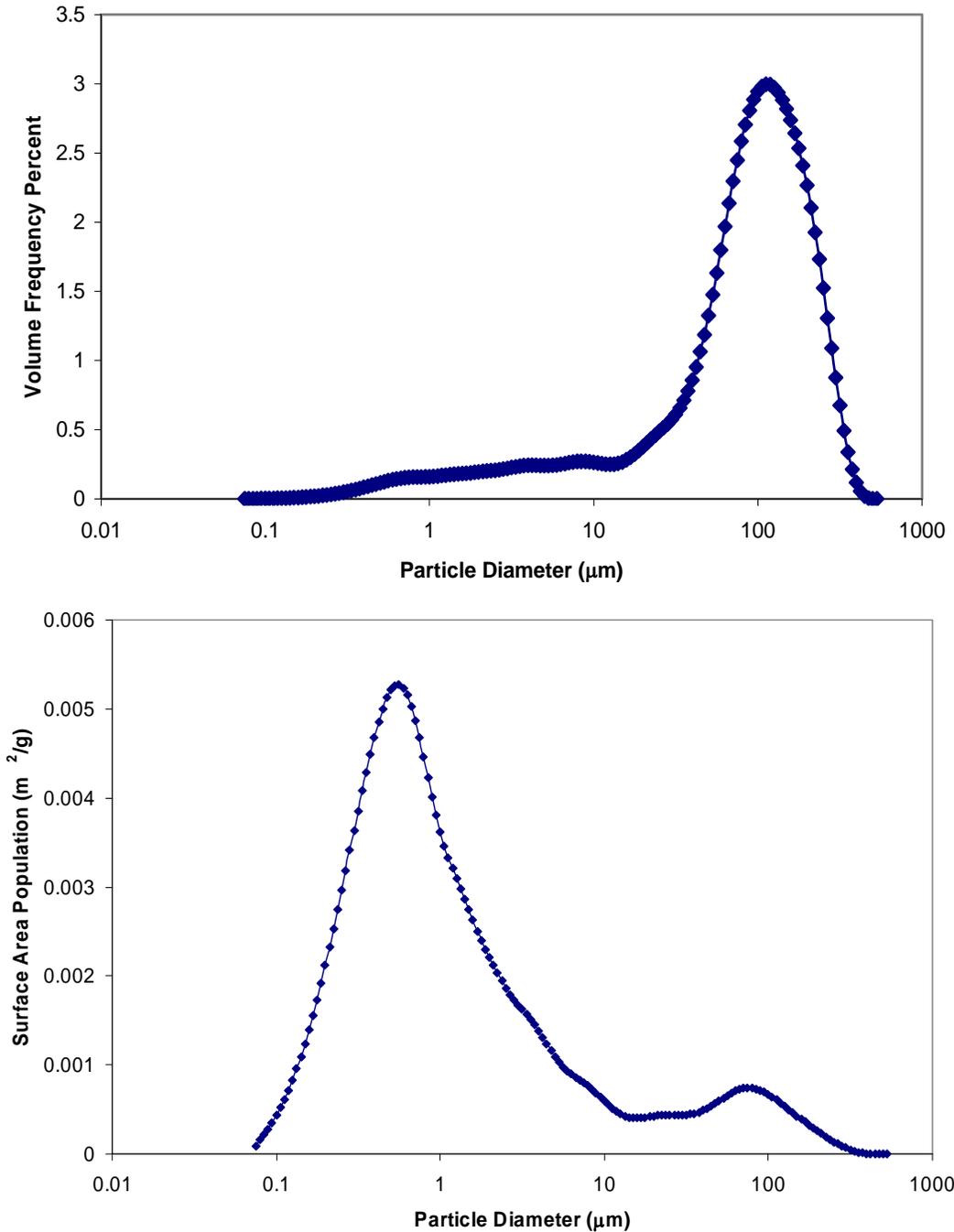


Figure 2. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Las Vegas Bay sample.

Particle size distributions for other samples are shown in Figures 3 through 5 for samples Las Vegas Wash 10.75, Duck Creek, and Nature Preserve, respectively. The particle size distribution results for all samples are included in Appendix I, Figures A-1 to A-7. Although the particle size distribution of the Las Vegas Bay sample shown in Figure 2 was the most common, other samples exhibited slightly different distributions. For example, the volume frequency percent distribution for the Las Vegas Wash 10.75 sample was clearly bimodal, with modes around 100 and 10 μm . Similarly, the volume frequency percent distribution for the Duck Creek sample clearly showed a shoulder around 10-20 μm (Figure 4a), and the volume distribution of the Nature Preserve sample, shown in Figure 5a, showed a narrow peak around 200 μm and a much broader peak around 10 μm .

It is worth noting, however, that despite the differences in volume frequency distributions, the surface area population distribution was very similar for all samples and the mode lied somewhere between 0.1 and 1.0 μm . This is not too difficult to justify, given that the specific surface area of particles is inversely proportional to particle size, and therefore the finer particles account for the vast majority of the total surface area, regardless of the distribution of the larger particles that account for the majority of volume in a sample. The conclusion of the particle size distribution analysis, therefore, is the confirmation of the assumption that the finer particles, certainly below 100 μm , and most likely below 10 μm , are responsible for the physicochemical behavior of these sediments.

Specific Surface Area

It is widely accepted that estimation of the solid surface area is required to model any type of interfacial process, including association of metalloids, such as selenium, with sediments (Davis and Kent 1990). An important parameter is the specific surface area, or surface area per unit mass of a solid. Increased specific surface area, and therefore increased concentration of reactive sites, is expected to lead to increased reaction rates, increased dissolution of a solid phase, or increased ion sorption at the mineral-water interface (White and Brantley 1995). It is therefore reasonable to assume that sediments and mineral particles with higher specific surface area might be associated with higher selenium concentrations.

All specific surface area measurements were performed with a Micromeritics ASAP 2010 automatic physisorption analyzer with multi-gas option. Because of the relatively high surface area of the sediment samples, nitrogen was used as the sorption gas for all samples. The specific surface area was then estimated using the BET method (Brunauer et al. 1938; Gregg and Sing 1982). All specific surface area results are included in Table 3. For each sample, the BET surface area estimate (m^2/g), the micropore volume (cm^3/g) and micropore area (m^2/g) and the corresponding average pore diameter (\AA) are included. The external surface area is given as the difference between the total BET surface area and the area associated with micropores and therefore truly represents the area associated with the true external surface, as well as macro- and mesopores. Macropores are defined as having diameters larger than 50 nm, mesopores are defined as pores with diameters between 2 and 50 nm, and micropores are defined as pores with diameters smaller than 2 nm (Gregg and Sing 1982).

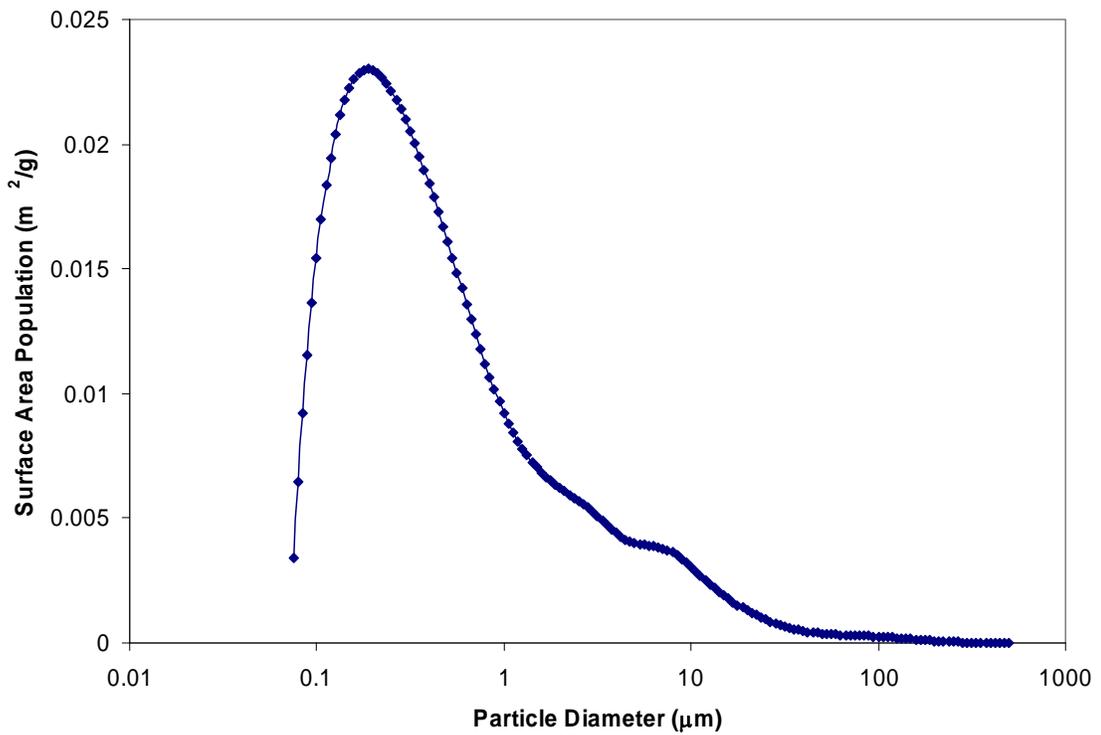
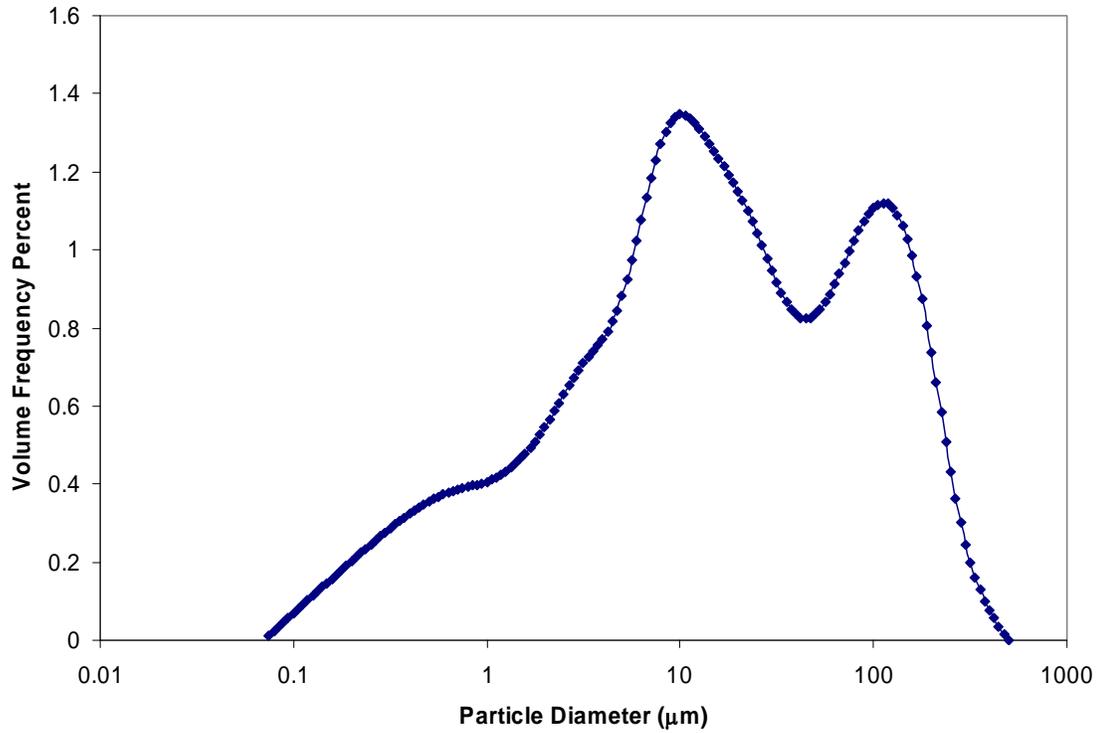


Figure 3. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Las Vegas Wash 10.75 mile sample.

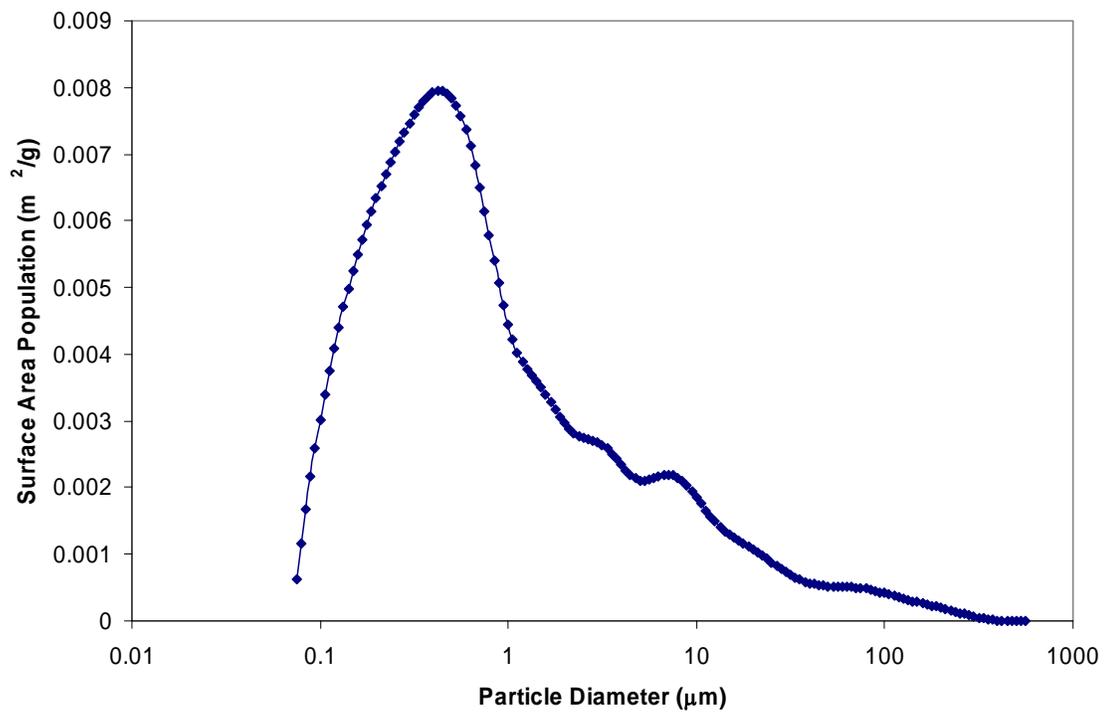
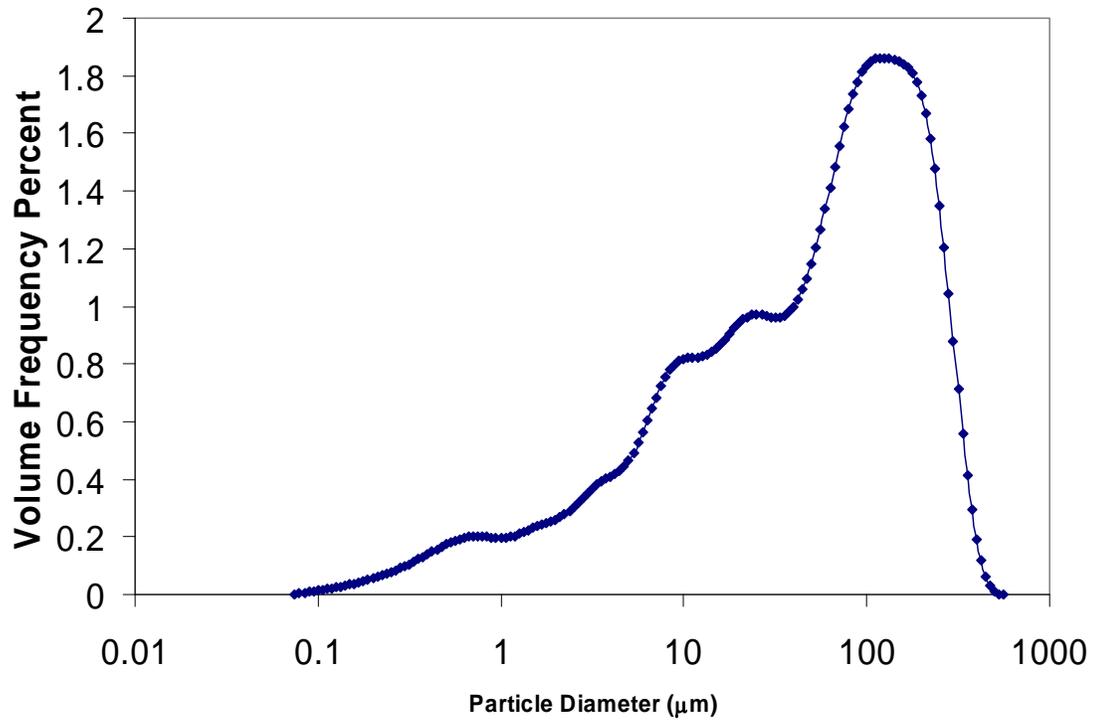


Figure 4. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Duck Creek sample.

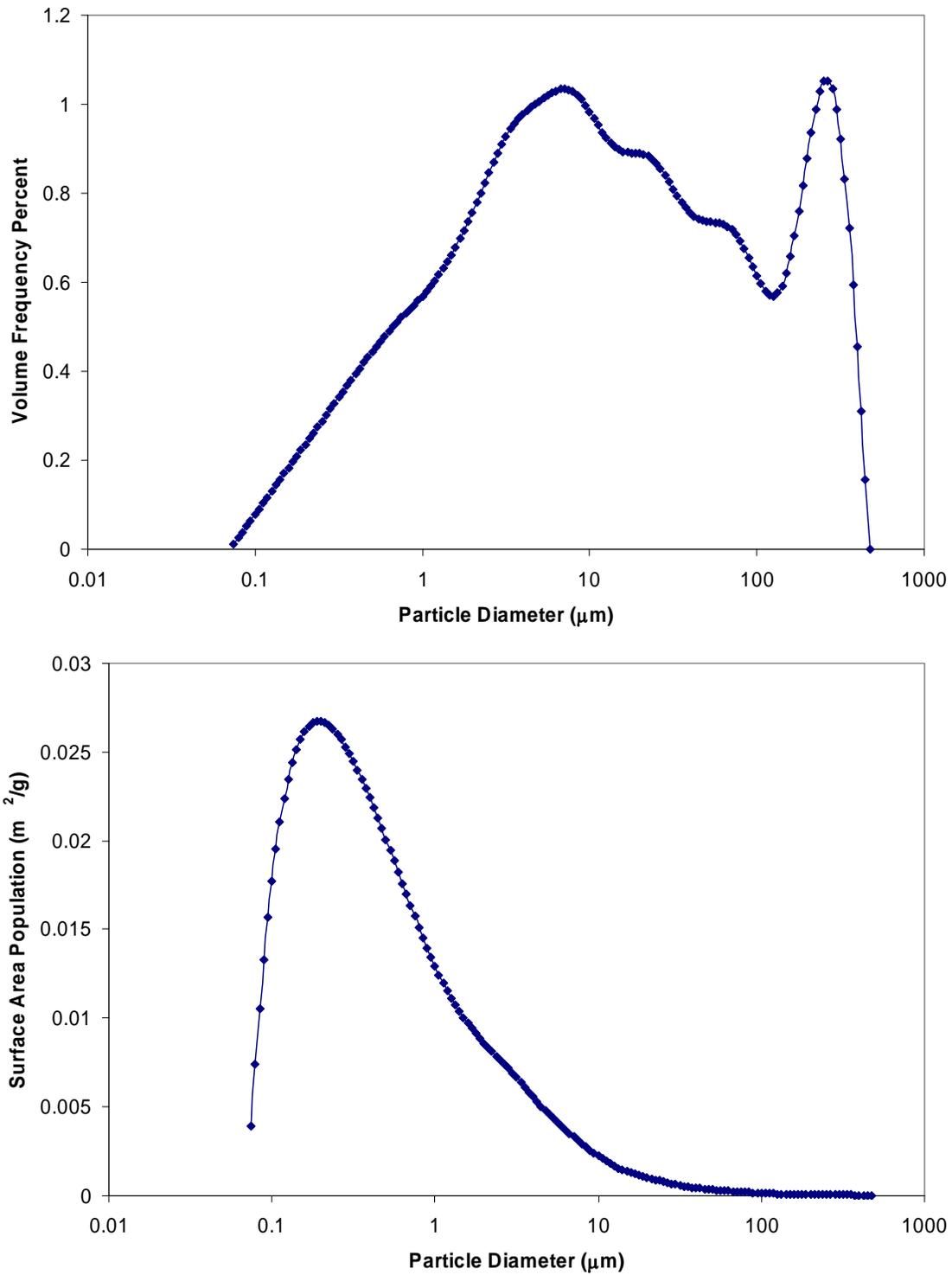


Figure 5. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Nature Preserve sample.

Table 3. Sediment specific surface area (m^2/g), as determined by nitrogen adsorption.

Sample	BET Surface Area (m^2/g)	Average Pore Diameter (Å)	Micropore Volume (cm^3/g)	Micropore Area (m^2/g)	External Surface Area (m^2/g)
Las Vegas Bay	7.92	54.6	0.000719	1.70	6.22
Las Vegas Bay (Dupl.)	8.27	53.9	0.000937	2.15	6.12
LV Wash 0.8	8.49	60.8	0.000706	1.69	6.80
LV Wash 6.05	16.03	66.2	0.001057	2.61	13.42
LV Wash 10.75	10.66	86.3	0.000176	0.66	10.00
Duck Creek	5.96	81.5	0.000084	0.36	5.61
Nature Preserve	12.20	69.1	0.000709	1.79	10.41

As expected, the majority of the surface area was not associated with micropores, although for the Las Vegas Bay sample the micropore area accounted for 20-25% of the total surface area. The smallest fraction of micropore to total surface area was found in samples LV Wash 10.75 and Duck Creek, approximately 6%. With or without micropores, however, it is reasonable to assume that some porosity was present in these sediment samples, based on the total surface area, which ranged from $5.96 \text{ m}^2/\text{g}$ for the Duck Creek sample to $16.03 \text{ m}^2/\text{g}$ for the LV Wash 6.05 sample. Even assuming that the majority of the surface area is associated with particles having diameters between 0.1 and $1.0 \text{ }\mu\text{m}$, the corresponding predicted surface area would probably be lower than the measured surface area (Papelis et al. 2003). It is therefore reasonable to assume that some porosity exists in various degrees in these samples. Given that the particle size distribution for the different sediment samples was not dramatically different, it is reasonable to assume that the differences in surface area might be reflecting differences in mineralogical composition.

Mineralogical Composition

The mineralogy of the sediment samples was determined by X-ray diffraction (XRD) using a PANalytical X'PERT Pro X-ray Diffraction Spectrometer. Spectra were collected in the 5 to $60^\circ 2\theta$ range using $0.03^\circ 2\theta$ steps and $\text{Cu K}\alpha$ radiation ($\lambda = 1.54060 \text{ Å}$). Although it was not expected to detect major mineralogical differences between sediment samples collected along the Wash, the mineralogical composition of a sample is a factor that must be considered to account for any differences in interactions between chemical constituents and mineral surfaces (Davis and Hayes 1986; Stumm 1987). For this work, the major mineral constituents were determined and a semiquantitative mineralogical analysis is presented for each sample. It should be noted, however, that detailed analysis for clay minerals can be very complex (Jenkins and Snyder 1996; Moore and Reynolds Jr. 1997; Massa 2000) and therefore the results referring to clay mineralogy of the samples should be viewed cautiously. Additional work would need to be completed to identify specific clay mineral phases accurately.

The semiquantitative mineralogical composition of all samples is shown in Table 4 and graphically represented in Figure 6. Not surprisingly, the major mineral phases were common minerals, such as quartz (α -SiO₂) and feldspars, a group of common tectosilicates. In these samples, orthoclase (KAlSi₃O₈) was identified in the Las Vegas Bay sample, while in every sample a mineral of the solid solution series plagioclase was also always present. Plagioclase is the solid solution series between the end members albite (NaAlSi₃O₈) and anorthite (CaAl₂Si₂O₈). Based on the semiquantitative analysis, the minimum quartz composition was 32% in the Nature Preserve sample and up to 47% in the LV Wash 6.05 sample (Table 4). The minimum feldspar concentration (combined orthoclase and plagioclase minerals) was 14% in the LV Wash 6.05 sample, while the maximum, 37%, was encountered in the Las Vegas Bay sample. An additional common silicate mineral found in all samples, in typical concentrations of 4-6%, was hornblende (Ca₂(Mg,Fe,Al)₅(Si,Al)₈O₂₂(OH)₂), belonging to the double-chain silicate group amphiboles. Only in the Nature Preserve sample was no hornblende identified.

In addition to quartz and the common silicate minerals feldspars and amphiboles, carbonates were the other group of minerals present in all samples. Specifically, calcium carbonate (calcite, CaCO₃) and calcium magnesium carbonate (dolomite, CaMg(CO₃)₂) were present in all samples. This is not surprising, given the prevalence of carbonate rocks in the Las Vegas valley. Together, quartz, feldspars, amphiboles, and carbonates accounted for the vast majority of all samples, ranging from 84% in the Nature Preserve sample to 94% in the Las Vegas Bay sample.

The rest of each sample was associated either with the widespread rock forming clay mineral biotite (K(Mg,Fe)₃(Si₃Al)O₁₀(OH,F)₂) or in some samples (LV Wash 6.05, Duck Creek, and Nature Preserve) with the zeolite mineral merlinoite ((K,Na)₅(Ba,Ca)₂(Si₂₃Al₉)O₆₄•24H₂O). In addition, two ammonium compounds, NH₄SH and (NH₄)₃(SnF₆)F were identified in the LV Wash 10.75 sample. These appear to be products of decomposition of organic matter. Finally, the common mineral gypsum (calcium sulfate dihydrate, CaSO₄•2H₂O) was identified in the Nature Preserve sample. It is a little surprising that gypsum, a common salt in soils around Lake Mead and the Las Vegas Wash, was not found in additional samples. The most likely explanation for the absence of gypsum from the other samples hinges on the high solubility of this mineral. Given that all samples were collected as wet samples, in contact with water either from the Las Vegas Wash, Duck Creek, or Lake Mead, it is reasonable to assume that highly soluble minerals, such as gypsum, were in much lower concentrations and possibly below the detection limit of the technique, as used.

The mineralogical composition results are consistent with the specific surface area measurements presented above, and the particle morphology and composition presented in the following section. For example, samples with the highest percentages of biotite and merlinoite, the highest surface area minerals, were expected to have the highest specific surface areas. This was definitely observed in the case of the Nature Preserve and LV Wash 6.05 samples, the samples with the highest specific surface areas, 12.20 and 16.03 m²/g, respectively. The only exception was the Duck Creek sample, which, unexpectedly, had the lowest surface area of all samples, 5.96 m²/g, even though it contained both biotite and merlinoite.

Table 4. Semiquantitative mineralogical composition of sediment samples.

Sample	Quartz	Dolomite	Calcite	Orthoclase	Plagioclase	Hornblende	Biotite	Merlinoite	Ammonium Comp.	Gypsum
Las Vegas Bay	38	7	3	0	33	13	6	0	0	0
Las Vegas Bay (Dupl.)	37	11	4	11	26	4	6	0	0	0
LV Wash 0.8	46	17	4	0	21	5	8	0	0	0
LV Wash 6.05	47	10	7	0	14	6	11	4	0	0
LV Wash 10.75	41	11	12	0	17	4	5	0	10	0
Duck Creek	36	2	13	0	32	5	4	7	0	0
Nature Preserve	32	11	8	0	33	0	6	7	0	3

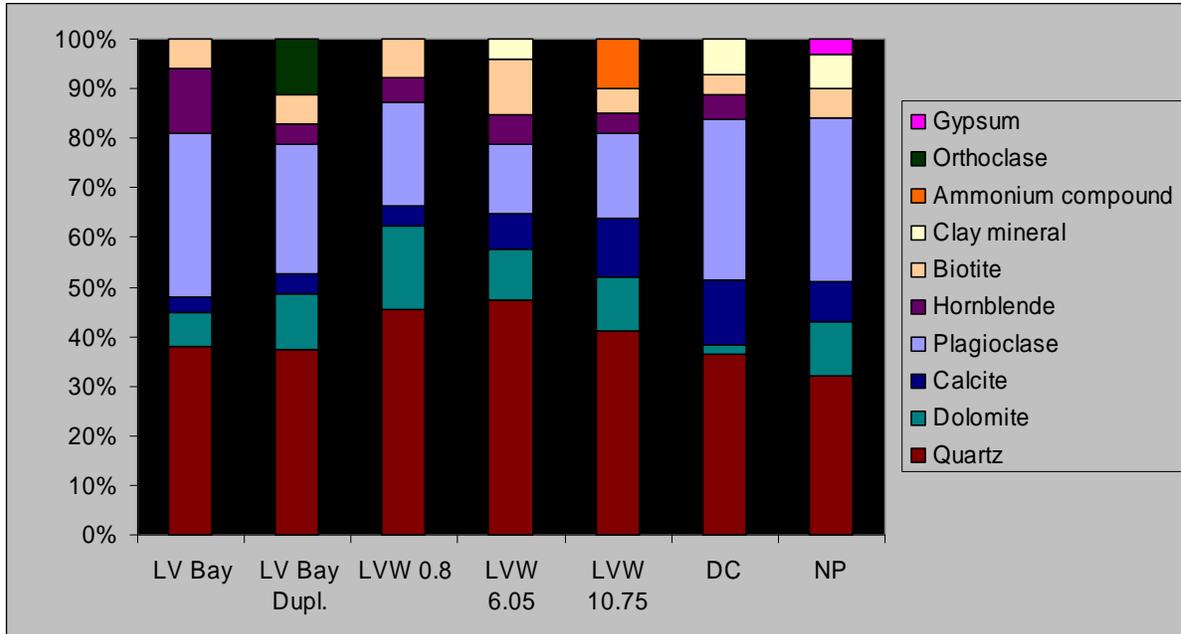


Figure 6. Semiquantitative mineralogical composition of sediment samples.

Particle Morphology and Composition

Electron microscopy can be used for a direct observation of a surface. The main advantages of Scanning Electron Microscopy (SEM) over optical microscopy are the increased magnification levels that can be achieved through electron microscopy, as well as the enhanced depth of field (Perry 1990; Goldstein et al. 1992). Magnification of 3,000 to 5,000 times is routinely achievable using modern electron microscopes. In addition, examination with an Energy Dispersive X-ray attachment (EDX) can provide a fast, at least semiquantitative, analysis of a surface. Finally, in the backscattering mode, modern electron microscopes can provide elemental maps, showing the spatial distribution of individual types of atoms of a specimen. In this work, a JEOL JSM-5610 scanning electron microscope was used with an Oxford Link EDX attachment. For all samples, several scanning electron micrographs at different magnifications were obtained, and the corresponding composition was determined by EDX. The particle morphology and elemental composition for specific characteristic samples are discussed below, while figures showing the particle morphology, EDX spectra, and elemental composition of all samples and all areas of interest and magnification are collected in Appendix III and shown in Figures A-15 to A-51.

Individual samples and EDX spectra at different magnifications for all sediment samples are shown in Figures 7 through 15. An SEM image of the Las Vegas Bay duplicate sample is shown in Figure 7. This micrograph is consistent with both the particle distribution data and surface area estimates. For example, there are particles ranging from larger than 200 μm down to the micrometer range and while the larger particles would account for the majority of volume, the finer ones certainly account for the specific surface area of this sample. As all samples contained quartz and calcite, it was relatively easy to identify these minerals in the Las Vegas Bay sample and they are shown in Figure 8. In addition, biotite

was present in this sample and a micrograph showing this mineral and the corresponding EDX spectrum is shown in Figure 9. Following quartz, feldspars were the most common minerals in these samples and an example showing the combination of these minerals is shown in Figure 10.

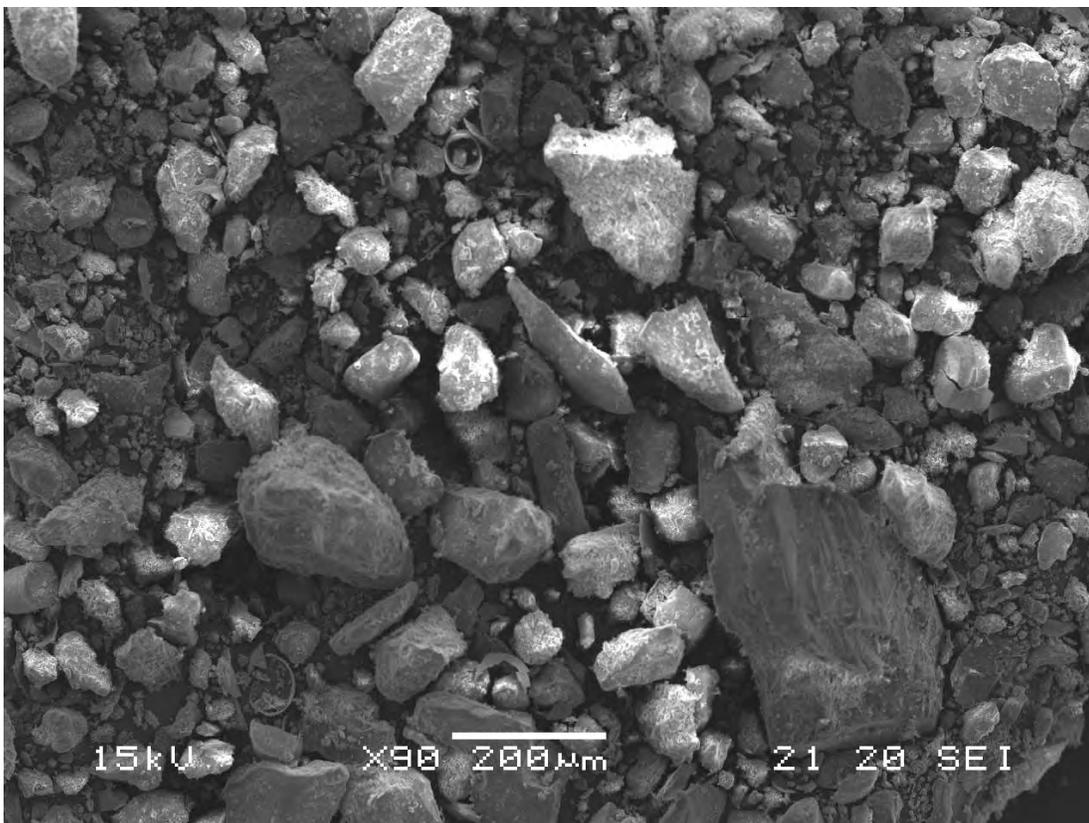


Figure 7. General view of the Las Vegas Bay duplicate sample. Magnification 90x.

Based on the EDX spectrum, a combination of calcite, quartz, and dolomite present in sample LV Wash 0.8 is shown in Figure 11, while mostly pure calcite in the same sample is shown in Figure 12. A SEM micrograph of the LV Wash 6.05 sample showing a feldspar crystal is shown in Figure 13. A general view micrograph of the LV Wash 10.75 sample is shown in Figure 14. Again, the particle size range observed in the micrograph is consistent with the particle size distribution and specific surface area data. Similar results, for a similar magnification, can also be observed for the Nature Preserve sample, shown in Figure 15.

Unfortunately, SEM combined with Energy Dispersive X-ray (EDX) detectors, is not sensitive enough to detect minor and trace elements, like selenium. As will be discussed in the following section, the measured selenium concentrations on the sediments were in the low part per million (ppm) range, whereas elemental composition, as determined by EDX, is given in terms of percentages. To obtain ppm level detection limits, we would have to use techniques like microprobe (EPMA). Still, the SEM technique is valuable as a tool, not just to take often stunning pictures, but also as confirmation of the particle size distribution, mineralogical composition, and surface area measurements. It is one of as many as possible techniques we try to use to corroborate the findings.

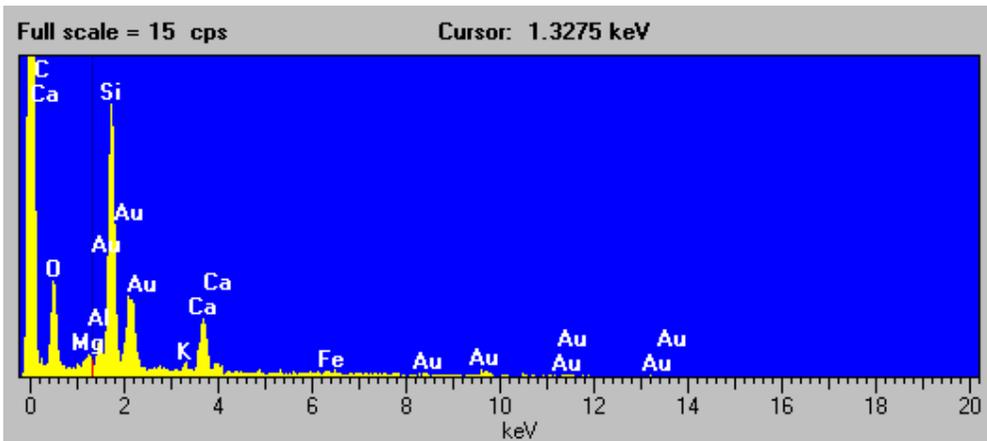
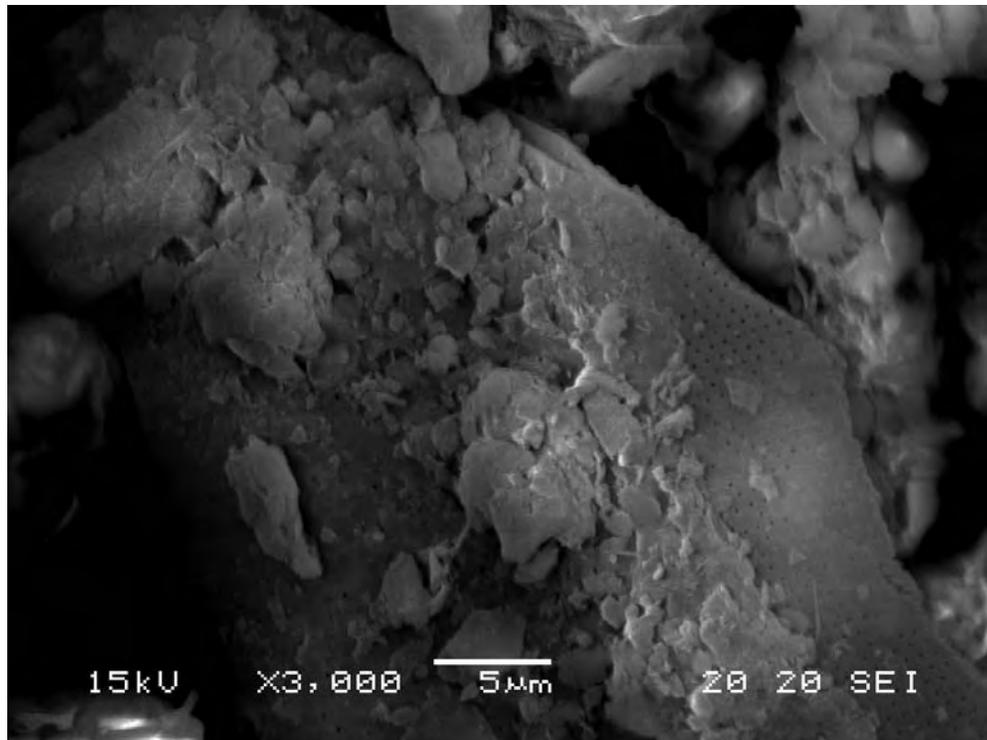


Figure 8. Scanning electron micrograph and EDX spectrum of the Las Vegas Bay sample (magnification 3,000x). Spectrum composition consistent with the presence of quartz and calcite in this sample.

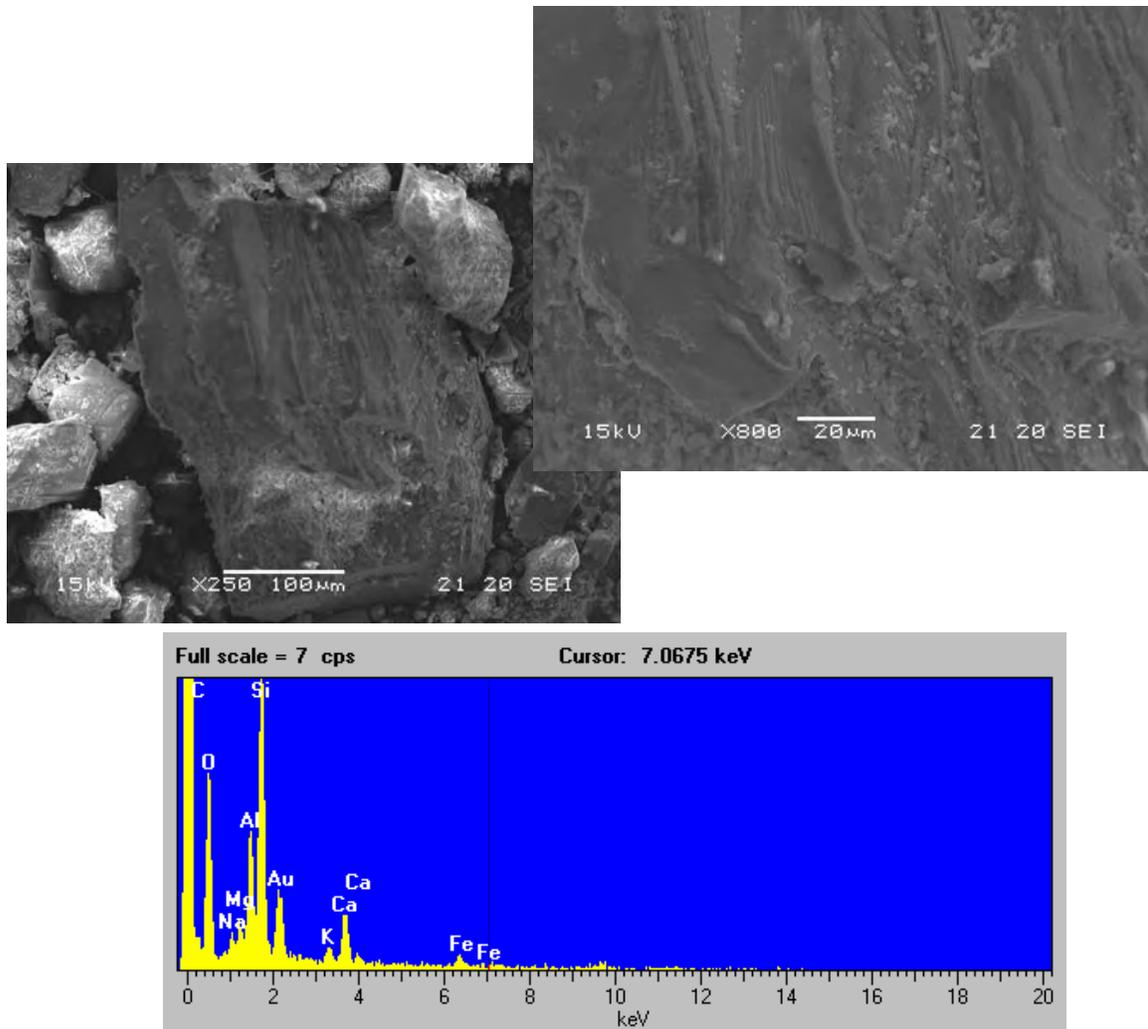


Figure 9. Scanning electron micrograph and EDX spectrum of the Las Vegas Bay duplicate sample (magnifications 250x and 800x). Spectrum consistent with the presence of biotite in this sample.

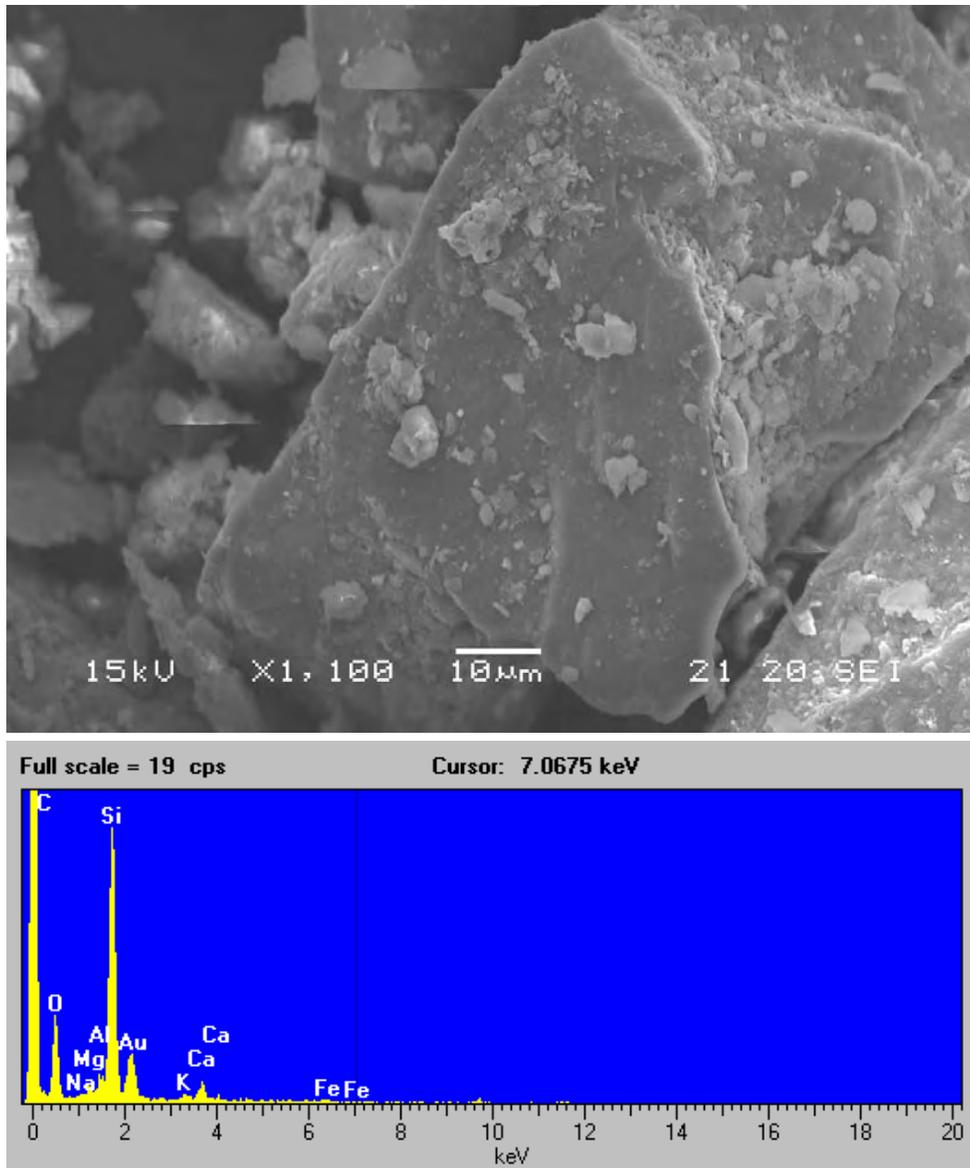


Figure 10. Scanning electron micrograph and EDX spectrum of the Las Vegas Bay duplicate sample (magnification 1,100x). Spectrum consistent with the presence of mostly quartz and some orthoclase in this sample.

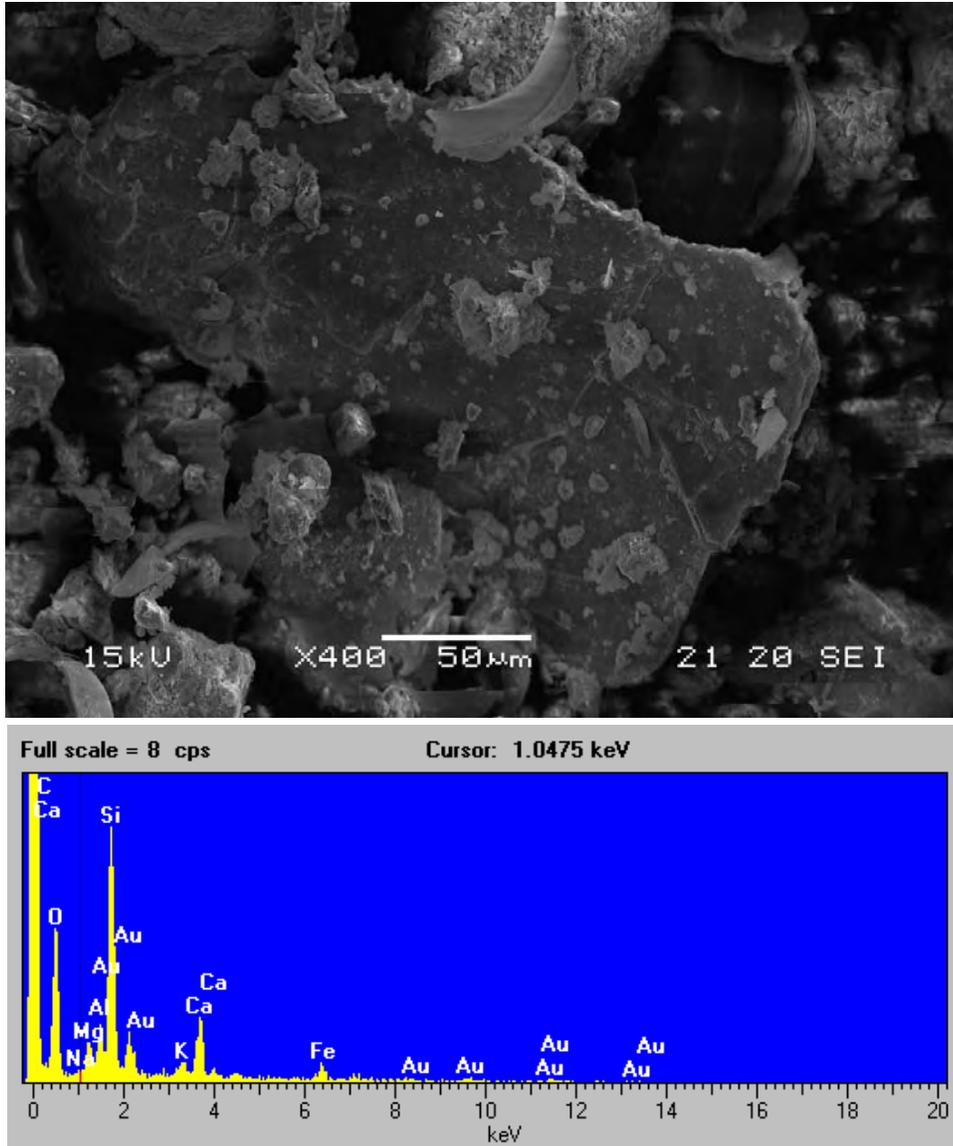


Figure 11. Scanning electron micrograph and EDX spectrum of the Las Vegas Wash 0.8 mile sample (magnification 400x). Spectrum consistent with the presence of mostly quartz, calcite, and dolomite in this sample.

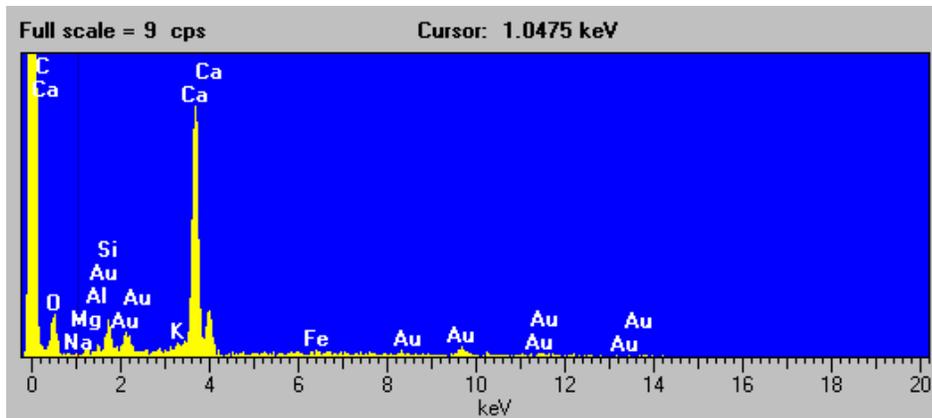
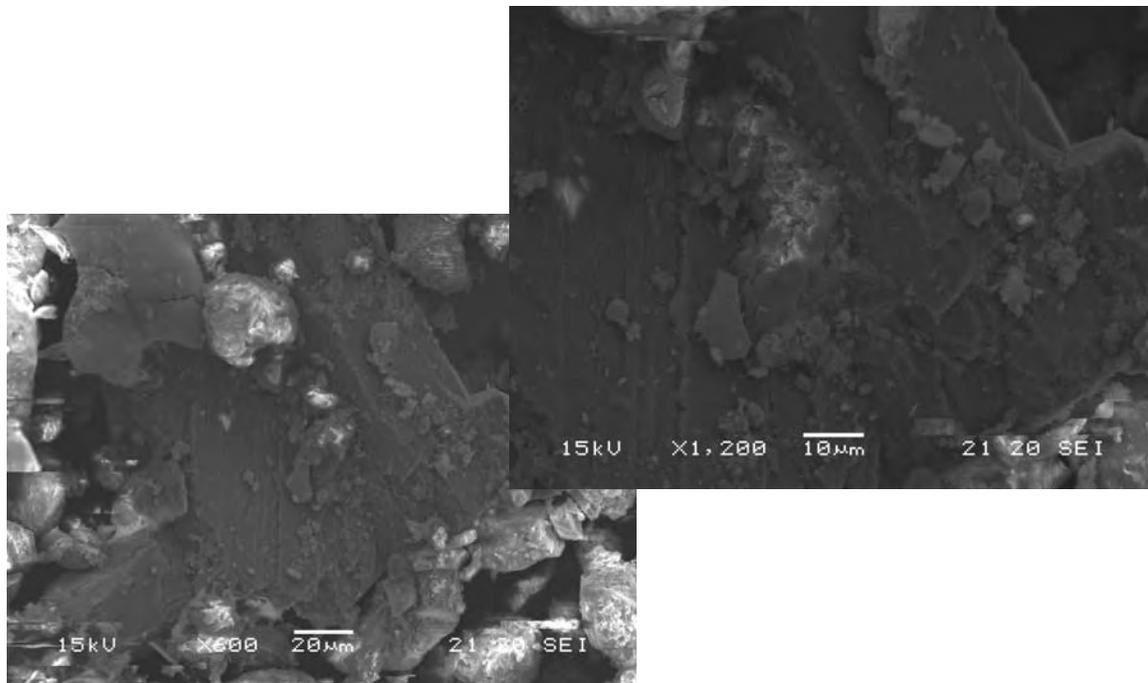


Figure 12. Scanning electron micrograph and EDX spectrum of the Las Vegas Wash 0.8 mile sample (magnifications 600x and 1,200). Spectrum consistent with the presence of calcite in this sample.

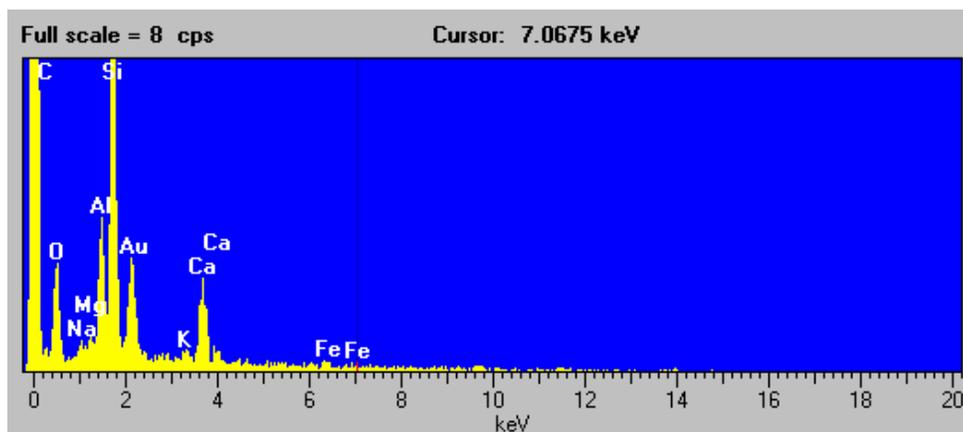
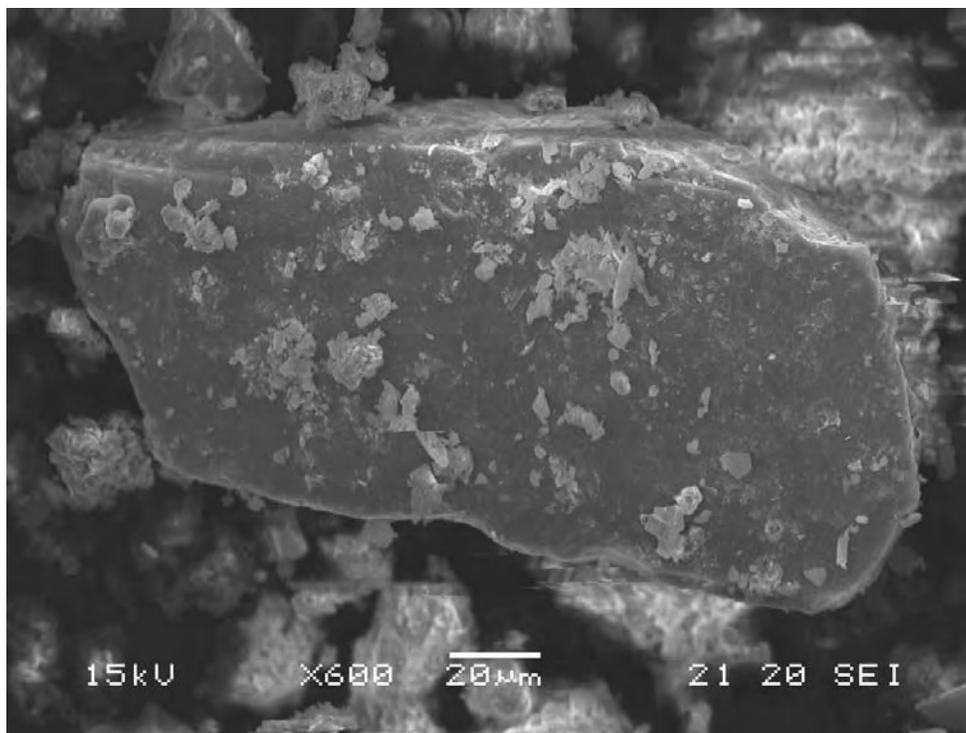


Figure 13. Scanning electron micrograph and EDX spectrum of the Las Vegas Wash 6.05 mile sample (magnification 600x). Spectrum consistent with the presence of plagioclase in this sample.

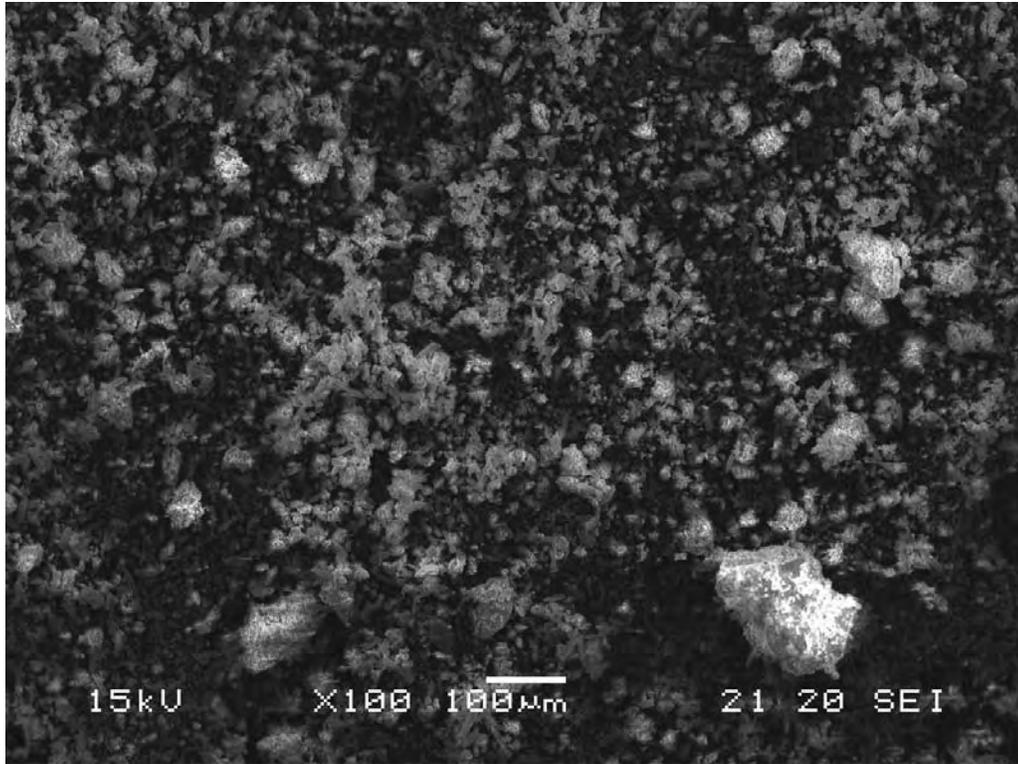


Figure 14. General view of the Las Vegas Wash 10.75 mile sample. Magnification 100x.

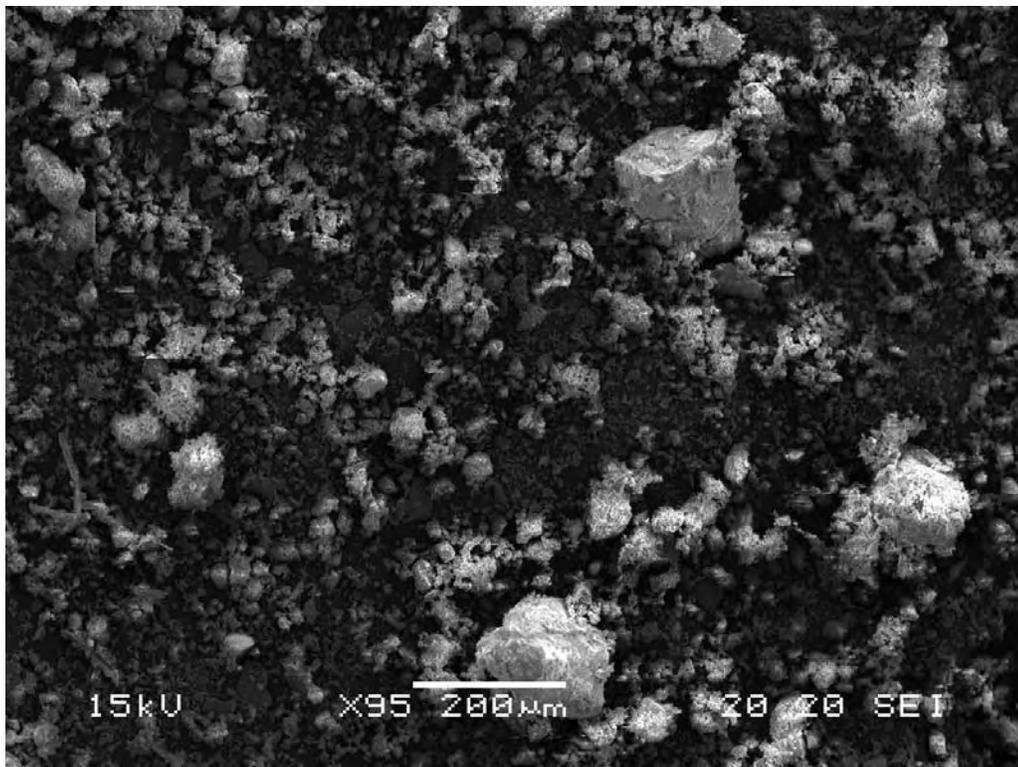


Figure 15. General view of the Nature Preserve sample. Magnification 95x.

Selenium Analysis

Selenium analysis included analysis for aqueous selenium in the supernatant of the samples, as well as analysis of the selenium strictly associated with the sediments. All analyses were performed with a Graphite Furnace Atomic Absorption Spectrometer (GFAAS). As was pointed out earlier, metals and metalloids distributed across two phases, such as the aqueous and solid phase, eventually will reach equilibrium, given enough time. In a dynamic system, such as the Las Vegas Wash, Lake Mead, or the Duck Creek, the aqueous selenium concentration is a function of the selenium concentration already present in the water, as well as the interaction of the water with sediments along the flow path. Selenium concentrations may therefore vary both spatially and temporally.

During routine water quality analyses, a water sample is collected and analyzed for selenium. It is unlikely that the water sampled is in equilibrium with the sediments present in the particular location, but the aqueous selenium value obtained is most likely a snapshot of a rather complex selenium transport process. During the sediment sampling, wet sediment was collected, including pore waters. The sediments were then presumably allowed to reach equilibrium with the pore water, until aqueous and sediment phases were separated for analysis. Specifically, the sediments were centrifuged and the clear supernatant was analyzed for selenium, while the remaining sediments were dried and processed for further sediment characterization and analysis. It is therefore instructive to consider the relationship between aqueous and sediment concentrations, as they probably better reflect the equilibrium relationship between aqueous selenium and selenium bound to sediments at these specific locations.

It is well known that selenium exists in different oxidation states with considerably different mobilities in the environment (Hayes et al. 1987; Deverel and Millard 1988; Hayes et al. 1988; Brown et al. 1989; Pickering et al. 1995; Tokunaga et al. 1997). The two most common oxidation states, +IV and +VI, correspond to the oxyanions selenite (SeO_3^{2-}) and selenate (SeO_4^{2-}), respectively. Selenate is much more mobile compared to selenite (Hayes et al. 1988), having similar transport characteristics to the major anion sulfate. Under oxidizing conditions, selenate is expected to be the thermodynamically stable form, although redox reaction rates may substantially affect the relative ratio of the two forms in an aqueous environment.

For this work, only total aqueous selenium is reported, although separate analyses by other laboratories (Clark County Water Reclamation District and Southern Nevada Water Authority) indicate that the majority of selenium in aqueous samples in the Las Vegas Wash and tributaries is in the oxidized form, selenate. Unfortunately, distinction between different oxidation states for selenium associated with solid phases is a much more involved process and the only possibility for such analyses involves spectroscopic techniques such as x-ray absorption spectroscopy (XAS) or x-ray photoelectron spectroscopy (XPS). These techniques can be both expensive – for example, XAS can only be performed at a few synchrotron facilities around the country – and in addition have a relatively high detection limit, typically substantially higher – sometimes orders of magnitude higher – compared to values found in typical sediments, even in sediments with elevated concentrations.

Instead of determining the oxidation state of selenium associated with sediments, therefore, other techniques have been developed in the past to help understand the association

of selenium with specific sediment fractions, as well as the total amount of selenium on sediments. The two techniques involve either a total selenium extraction method (EPA method 3050B, U.S. Environmental Protection Agency 1996) or a series of sequential extractions. The EPA method is designed to determine the total, so-called environmentally available selenium concentration.

Method 3050B uses a two step hot water bath digestion with concentrated nitric acid, followed by 30% hydrogen peroxide. One gram of sediment sample is placed inside a glass beaker that is in a water bath (95°C) and heated without boiling. Aliquots of concentrated nitric acid are added to the sample until no more brown fumes are given off. The solution is then heated without boiling for an additional two hours. Next, aliquots of 30% hydrogen peroxide are added (up to 10 mL) while the sample is again heated. The acid-peroxide digestate is then heated, without boiling, for an additional two hours and diluted to a final volume of 100 mL, before analysis. Knowing the total mass of sediments used for the extraction, from the selenium concentration in the digestate, we can calculate the concentration of selenium on the sediment samples, usually reported as µg/g, or ppm.

Sequential extraction methods have been used in the past to investigate the interaction of metals and metalloids with specific components of soils (Patterson and Passino 1990; Allen 1995; Allen et al. 1995; Tessier and Turner 1995). For this work, the method of Tokunaga et al. (1991) was used, as modified by Zhang and Moore (1996). In the original method, the distinction between selenium bound to amorphous and crystalline phases was made. In the modified version, however, all oxides are treated simultaneously. The individual fractions include selenium assumed to be soluble (easily exchangeable), adsorbed, associated with carbonates, associated with oxides, or associated with organic matter. The specific extractants and procedure used to extract each fraction are reported in detail in the original publication (Zhang and Moore 1996). After each extraction step, the extract is centrifuged and the supernatant is analyzed. For this work, the soluble selenium (extracted with 0.25M KCl) and the selenium associated with oxides (extracted with 4.0M HCl) were only determined. The analysis of other fractions proved to be problematic and unreliable, most likely because of interferences from other ions present in the sample in high concentrations (sulfate being one of them).

The results of selenium concentrations in the aqueous samples are shown in Table 5 in µg/L (ppb). The lowest concentration was found in the Las Vegas Bay sample, averaging approximately 4 ppb, between the two duplicate samples. All other samples exceeded 5 ppb, the standard based on chronic wildlife exposure (Schnoor 1996). The next lowest concentration sample was LV Wash 6.05, 5.32 ppb, while the other samples were higher, approximately 7 to 8 ppb. As can be seen from the duplicate analyses, the data were reproducible and reliable. In summary, these concentrations generally exceed the standards for chronic wildlife exposure to selenium.

It should be kept in mind that these are concentrations found in the supernatant of the collected sediment samples and they do not necessarily reflect selenium concentration in any of these locations along the Wash. These values only reflect the concentration of selenium in at least pseudoequilibrium with these particular sediments. These results will be further discussed in connection with sediment concentration results; it is, however, worth noting that the lowest concentration was observed in the Las Vegas Bay (Lake Mead) sample, while some of the highest concentrations were observed upstream (LV Wash 10.75) and the Nature

Preserve. It is possible that these trends can be at least in part attributed to dilution effects. The samples obtained from Lake Mead were presumably in contact with higher quality water, lower in selenium concentration, resulting in lower selenium in the supernatant as well, while the samples higher upstream were presumably in contact with higher selenium concentration waters.

Table 5. Selenium concentrations of aqueous samples ($\mu\text{g/L}$).

Sample	Replicate 1	Replicate 2	Average Concentration
Las Vegas Bay	4.06	4.42	4.24
Las Vegas Bay (Dupl.)	3.53	4.02	3.78
LV Wash 0.8	6.87	6.93	6.90
LV Wash 6.05	5.14	5.50	5.32
LV Wash 10.75	7.86	8.06	7.96
Duck Creek	6.42	6.76	6.59
Nature Preserve	8.09	N/A	8.09

The concentration of selenium in the sediments collected is shown in Table 6 and graphically in Figure 16. The three columns represent the soluble (easily exchangeable) selenium, selenium associated with the oxide fraction, and total environmentally available selenium estimated using EPA method 3050B. In general, for all samples, the easily soluble fraction appears to be a very small fraction of the total selenium. This is not surprising, given the high salinity of the Las Vegas Wash and surrounding waters, similar to the composition of the solution used in the extraction step. In other words, any selenium likely to be extracted by exposure to similar water has already been extracted. The oxide fraction was much more substantial for all samples and accounted for approximately 20 to 50% of the total environmentally available selenium. It is clear, however, that a substantial fraction of the total selenium must be incorporated in other sediment fractions, such as the adsorbed, carbonate, and organic fractions, which were not analyzed individually. This is not surprising, given the relatively high percentage of carbonate minerals in these samples, as discussed earlier.

It is useful to consider the sediment selenium results as a function of the analytical method used. The first method, EPA method 3050B, is referred to as “total environmentally available” selenium. It is not a method meant to determine the truly total selenium in the sediments, as would have been done if the entire sediment mass were dissolved. In other words, it does not include selenium bound in the matrix of aluminosilicate minerals, such as quartz and feldspars. These minerals are never dissolved during the EPA 3050B method. Other than the fraction incorporated in aluminosilicate minerals, the other fractions included in the total are certainly environmentally available, albeit at different degrees, under relatively common environmental conditions.

Table 6. Concentrations of soluble selenium, selenium associated with oxides, and total selenium ($\mu\text{g/g}$) in sediment samples.

Sample	Soluble Selenium (KCl Extraction)	Oxide Fraction (HCl Extraction)	Total Selenium (EPA 3050B Method)
Las Vegas Bay	0.01	0.51	2.71
Las Vegas Bay (Dupl.)	0.06	0.64	2.57
LV Wash 0.8	0.06	0.77	1.68
LV Wash 6.05	0.10	0.59	3.54
LV Wash 10.75	0.12	0.99	3.99
Duck Creek	0.03	0.93	4.27
Nature Preserve	0.09	0.96	3.18

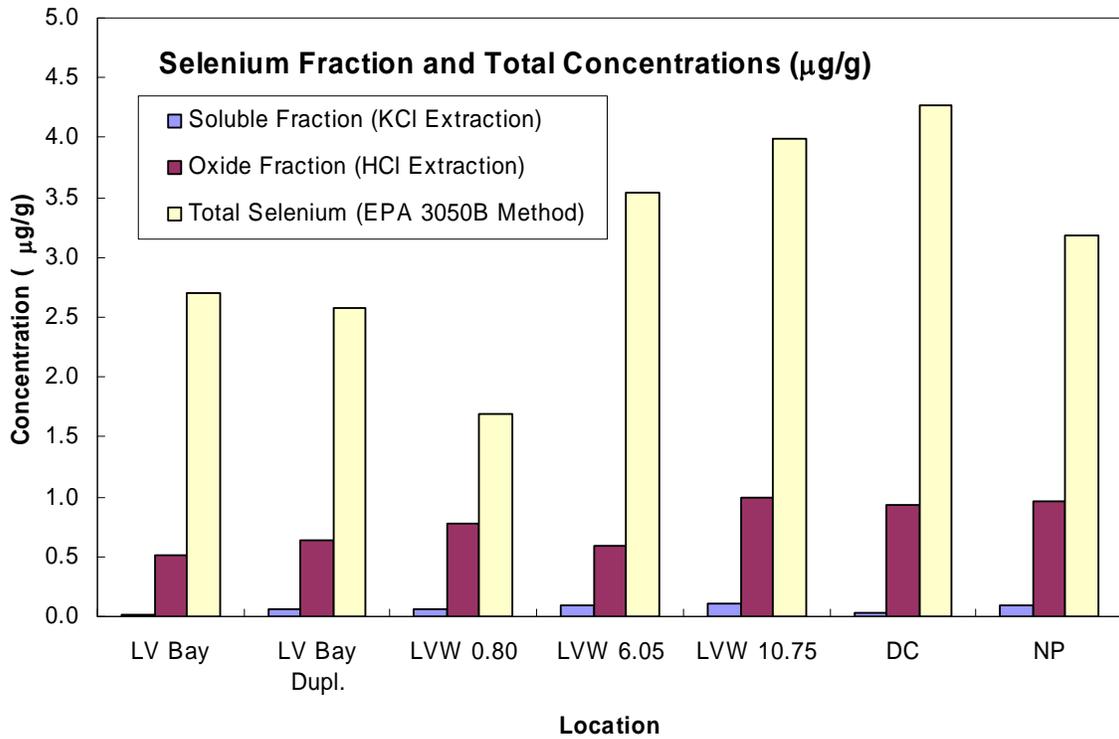


Figure 16. Concentrations of soluble selenium, selenium associated with oxides, and total selenium ($\mu\text{g/g}$).

These fractions include easily soluble, adsorbed, oxide, carbonate, and associated with organic matter. Clearly, the easily soluble fraction is the most readily available and any contact with water will essentially remove it. It is likely that this fraction accounts to a large extent for the aqueous selenium concentration measured in the Wash and tributaries. Other fractions, although not as readily available, would still contribute to the total selenium load. For example, carbonate minerals are not too insoluble and participate in adsorption reactions. Changes in the partial pressure of CO₂ can easily affect the solubility of these minerals. The solubility of iron oxides (the most common oxides) is highly dependent on redox conditions. Under anoxic conditions -- frequently encountered in sediments -- iron oxides would dissolve, releasing any associated metals. The sorbed fraction is pH dependent. Since selenium sorbs on mineral phases in the form of anions like selenite and selenate, higher pH, not lower, would tend to mobilize selenium. Finally, the selenium associated with the organic fraction could likely be available to biota.

It is important to compare the selenium results in the aqueous and sediment phases. It is worth noting that the sediments from the Las Vegas Bay had some of the lowest sediment concentrations (approximately 2.6 ppm) and the lowest supernatant concentration (approximately 4 ppb). It might, therefore, be reasonable to assume that sediments in the Las Vegas Bay have been in contact with relatively cleaner Lake Mead water, and that the easily exchangeable selenium has already been removed from the sediments.

These trends cannot be generalized, however. For example, the LV Wash 0.8 sample had the lowest sediment concentration, 1.68 ppm, even though the aqueous selenium concentration for this sample was similar to the concentrations of the other samples, 6.90 ppb. The highest sediment concentration was found in the Duck Creek sample, 4.27 ppm, while the rest of the samples had concentrations between 2 and 4 ppm.

These trends cannot be attributed to random variations based on location alone, but rather they must be attributed to anthropogenic inputs and the local hydrogeology. As stated earlier, inputs from anthropogenic sources through seeps and shallow groundwater, along the Wash are possible. For example, Duck Creek has consistently high selenium concentrations and the fact that the sediment concentration was also the highest in this sample should not be surprising. There is no doubt that there is a relationship between the source of water, with its associated water quality, the drainage area for this water, and the concentration of selenium in the water and sediments of a particular area. Additional investigations would be required to evaluate the degree of interaction of selenium in these waters with the sediments in contact with tributaries and the Las Vegas Wash.

Other than the Duck Creek sample, the LV Wash 10.75 sample had the next highest sediment concentration, 3.99 ppm. This sample had also the second highest selenium concentration in the supernatant, 7.96 ppb. Relatively lower flow rate in the Las Vegas Wash at this location may explain the higher concentration values. It should be remembered, however, that soil samples previously analyzed from the Las Vegas Wash and Nature Preserve areas, above the groundwater table, had routinely selenium concentrations above 4 and as much as 14 ppm. It is therefore reasonable to assume that the aqueous selenium concentrations measured in the Las Vegas Wash and tributaries are strongly correlated to the selenium concentrations in the sediments of that area.

Finally, the selenium concentrations could possibly be correlated to the mineralogy of the samples, as determined by XRD. It appears that there is a correlation between mineralogical composition, BET surface area, and selenium concentration. The only exception being the Duck Creek sample, which had the lowest BET surface area but the highest Se concentration. This apparent anomaly might be related to additional anthropogenic inputs to the Duck Creek from urban runoff or other uses.

Assessment of Selenium Concentrations in Sediments

As pointed out in the introduction, the objective of this study was to obtain a snapshot of the concentration of organic and inorganic constituents associated with sediments along the Las Vegas Wash. The ultimate goal, of course, is to establish whether there is a reason for concern, based on the overall environmental quality of the Las Vegas Wash area. Of particular significance is the assessment of the data obtained with respect to potential adverse effects to wildlife. The data collected, therefore, must be examined relative to values established by regulatory agencies which are designed to protect wildlife.

This is a difficult and complex task, for several reasons. Unfortunately, there is no single value that can be used as an absolute criterion to evaluate the health of an ecosystem. Several different methods have been proposed by several different regulatory agencies in their effort to establish evaluation criteria (Power and McCarty 1998; Ecological Planning and Toxicology Inc. 1999). Decisions are further complicated by the plethora of inorganic and organic constituents that need to be considered, different species with different susceptibilities to a particular chemical that need to be protected, as well as deciding what an appropriate target level should be for metal concentrations in soils.

The target values are often given different names and have different definitions. In addition, the assumptions used to derive these values, such as safety factors, are typically controlled by the experimental conditions used to arrive at these values. To complicate matters more, the same terms are often defined differently in different studies (Tuttle and Thodal 1998). Finally, specific environmental and geochemical conditions, such as pH and the geochemical matrix in a specific environment, can influence substantially the toxicity of a metal, so that a single value could not be applicable in different environments.

For example, according to Ecological Planning and Toxicology Inc. (1999), Soil Protection Values (SPVs), which are also known by many other variants, such as “precautionary soil values,” “soil quality objectives,” etc. are defined such that at concentrations below the SPV, one can be relatively certain that there will be no adverse effects on wildlife, whereas at values above the SPV there may be adverse effects, depending on a number of parameters. Under the sediment criteria proposed by NOAA, an Effects Range Low (ER-L) and an Effects Range High (ER-H) is established. These values are determined so that at values below ER-L effects on the test organism have never been observed, whereas at values above ER-H, effects are always observed. Obviously, between the ER-H and ER-L values, effects on the test organism are sometimes observed.

A different nomenclature is adopted by (Tuttle and Thodal 1998) and includes “concern” and “effect” levels. Although the definition of these two levels depends on individual studies, often “concern” implies the minimum concentration of observable effects, while “effect” is associated with severe or much more likely adverse health effects. In this discussion, the measured selenium concentrations will be evaluated based on the concern and effect levels listed in Tuttle and Thodal (1998) or the SPV listed in Ecological Planning and

Toxicology Inc. (1999), where applicable. It should be remembered, however, that none of these values can be taken as an absolute value; instead they are meant to provide guidance with respect to potential effects on wildlife and possibly an indication that further studies are warranted.

The concern and effect levels for selenium are 1 and 4 ppm, respectively. The reported SPVs range from 0.81 up to 100 ppm; however, the geometric mean is 5.08. The two values, therefore, are not too different. It should also be noted that the guidelines referring to “concern” and “effect” levels of selenium in soils, refer to total selenium concentration, for example, as measured by EPA method 3050B, which was used in this study.

The highest selenium value measured was 4.27 ppm at Duck Creek. This value is just above the effect value. A number of additional soil samples, however, mainly from the Nature Preserve area, have been analyzed over the last five years (Papelis, Unpublished data) and these concentrations range from 4.1 to 13.8 ppm, for several samples. Although only one sample, at Duck Creek, had a sediment concentration higher than the effect level of 4 ppm, all other sediment samples had selenium concentrations between the concern and effect levels, with the LV Wash 10.75 sample essentially at the effect level and the LV Wash 6.05 sample not much lower (3.54 ppm). The concentration of selenium in the sediments along the Las Vegas Wash, therefore, deserves further evaluation.

Clearly, it is important to compare the current results with the results of selenium in soils obtained during the previous sampling study in 2003. Unfortunately, in the case of selenium it will be hard to make the comparison. The reason is that, unfortunately, the outside laboratories that performed all the analyses during the first (2003) sampling used a technique with a 5 ppm detection limit. This is higher than the effect level of 4 ppm and therefore only the Las Vegas Bay sample had a reportable concentration of 6.7 ppm. That was the sample with the highest selenium concentration in 2003. All other samples were reported as less than 5 ppm. In the current sampling study (2006), the Las Vegas Bay sample (2.71 ppm) definitely did not have the highest concentration. The highest concentration was at Duck Creek, 4.27 ppm. So, in terms of maximum concentration there appear to be differences between the two sampling events. However, for all sites, the results from the second sampling are below 5 ppm, so the results are consistent with the first sampling study (except for Las Vegas Bay, all values below 5 ppm). The differences regarding the Las Vegas Bay samples may be related to Lake Mead level fluctuations between the two sampling events.

SUMMARY AND CONCLUSIONS

To determine baseline conditions regarding potential contaminant accumulation in the Las Vegas Wash and select tributaries, sampling and analysis of sediments from four sites along the Wash, in addition to Duck Creek and the Nature Preserve, was conducted. Samples were collected at the banks of each sampling location, in accordance with requirements published by the US EPA. Sediments were analyzed for metals, nutrients, perchlorate, volatiles (BTEX), semivolatiles, pesticides, PCBs, and herbicides. In this report, we limit the discussion on the physicochemical characterization of the sediments, as well as sediment analysis for selenium.

The particle size distribution was typical for sediments and followed either a modal or a bimodal distribution. Regardless of the particle distribution, however, the vast majority of the surface area appeared to be associated with particles of micrometer dimensions. The particle mineralogy revealed that sediments were composed mostly of quartz, feldspars, an amphibole group mineral (hornblende), the carbonate minerals calcite and dolomite, as well as biotite and a zeolite type mineral. The specific surface area of the sediments was relatively high, ranging from 5.96 m²/g for the Duck Creek sample to 16.03 m²/g for the LV Wash 6.05 sample. The relatively high surface areas were consistent with the measured particle size distribution and mineralogical composition and could be a factor in the observed high selenium concentration. The sediment morphology and composition, as determined by scanning electron microscopy, was consistent with the measured particle size distribution and sediment mineralogy.

Selenium concentrations were measured both in the supernatant as well as the sediments. The supernatant selenium concentrations ranged from approximately 4 ppb in the Las Vegas Bay sample to 8.1 ppb in the Nature Preserve sample. The total environmentally available selenium on the sediments was estimated using EPA method 3050B, as well as sequential extractions. The total selenium sediment concentrations ranged from 1.7 ppm in the LV Wash 0.8 sample to 4.3 ppm in the Duck Creek sample. A very small fraction appeared to be associated with easily exchangeable, soluble selenium, while 20 to 50% was associated with oxides. The rest of selenium appears to be associated with carbonate and the organic fraction, or exist as an adsorbed phase.

Although a relationship appears to exist between the aqueous and sediment concentrations in these samples, this relationship does not appear to be a simple one, i.e., higher dissolved selenium concentrations do not necessarily correspond to the highest sediment concentrations. There is obviously a dynamic relationship between sediment composition and selenium loading, as well as water quality and the hydrogeology of the area. The selenium concentrations obtained were compared to published values designed to indicate whether these concentrations in sediments could be a cause for concern. Considerations of these values suggest that in most samples the sediment selenium concentrations were in the range suggesting concern, while in two cases, samples LV Wash 10.75 and Duck Creek, the sediment concentrations were at or above the effect level of 4 ppm. These results suggest that selenium distribution and mobility in the Las Vegas Wash and surrounding wetlands and tributaries should be further monitored and investigated.

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APPENDIX A: Particle Size Distribution

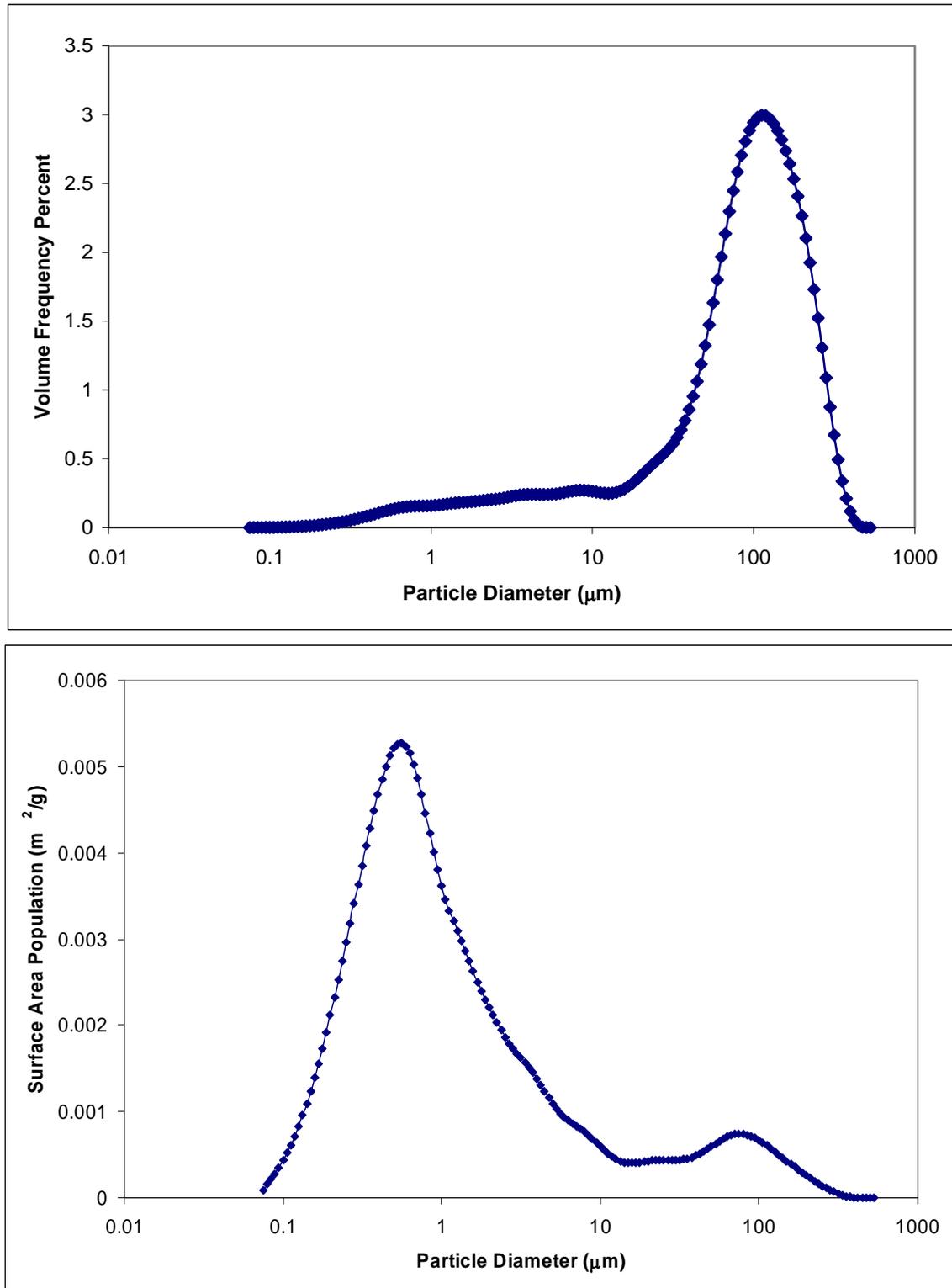


Figure A-1. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Las Vegas Bay sample.

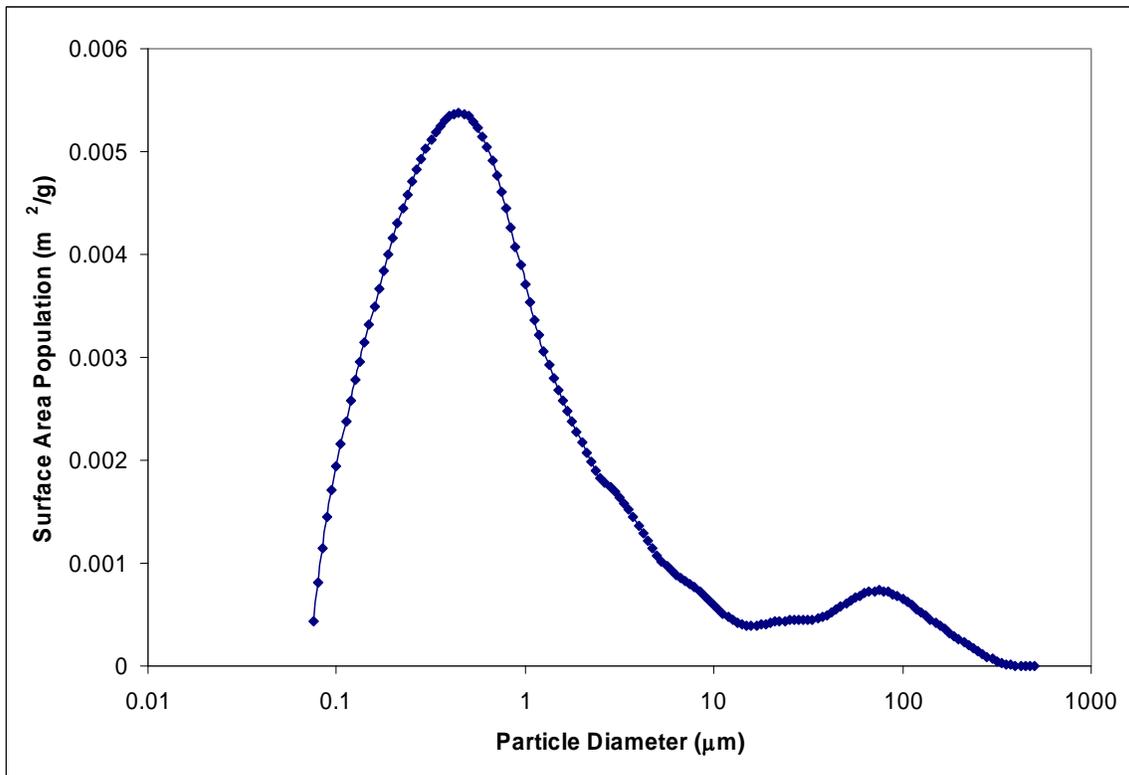
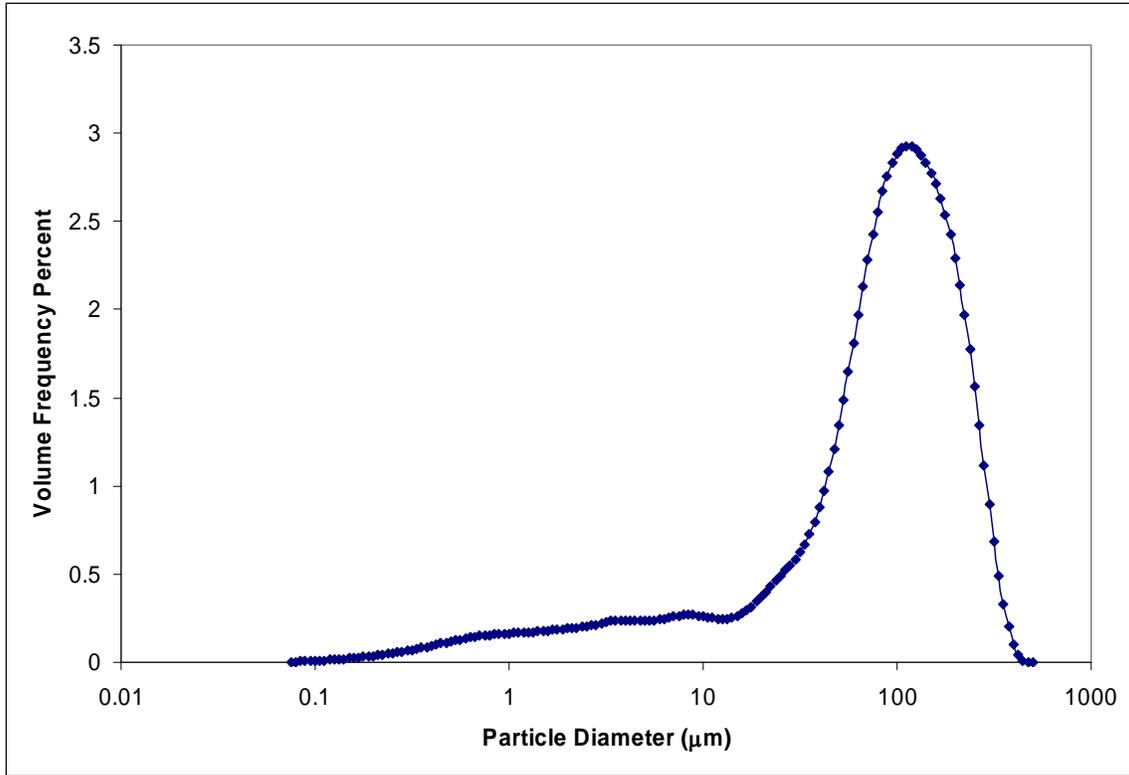


Figure A-2. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Las Vegas Bay duplicate sample.

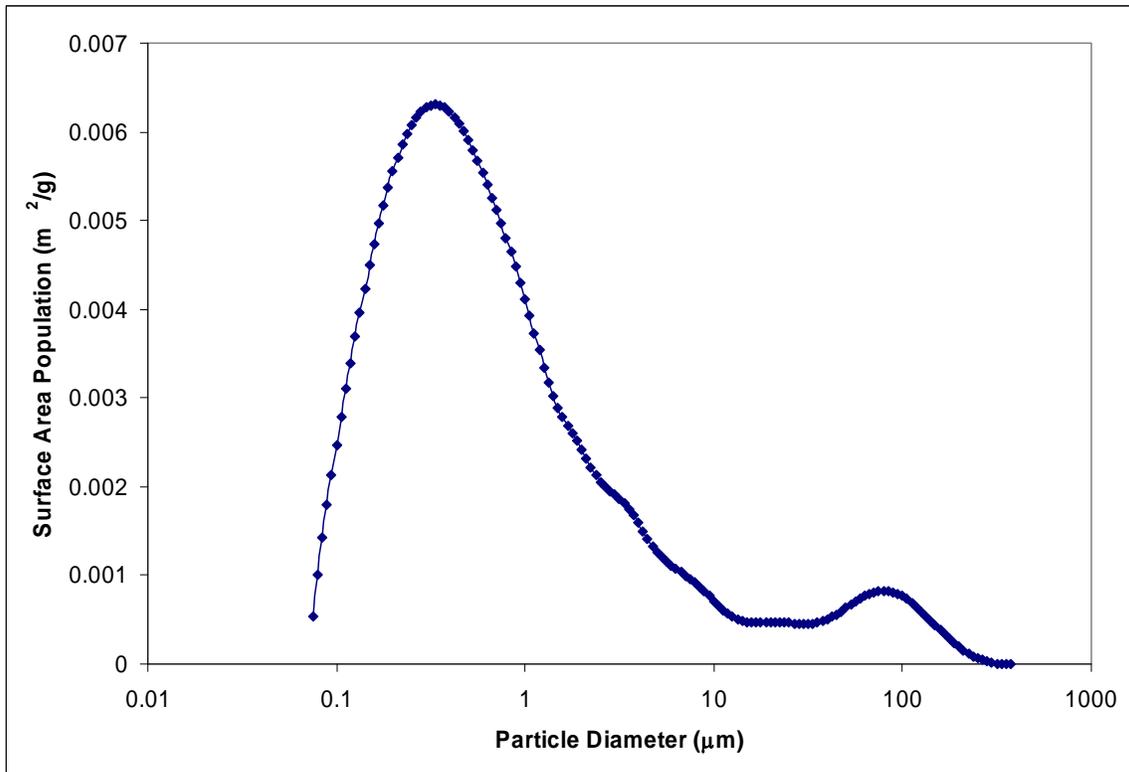
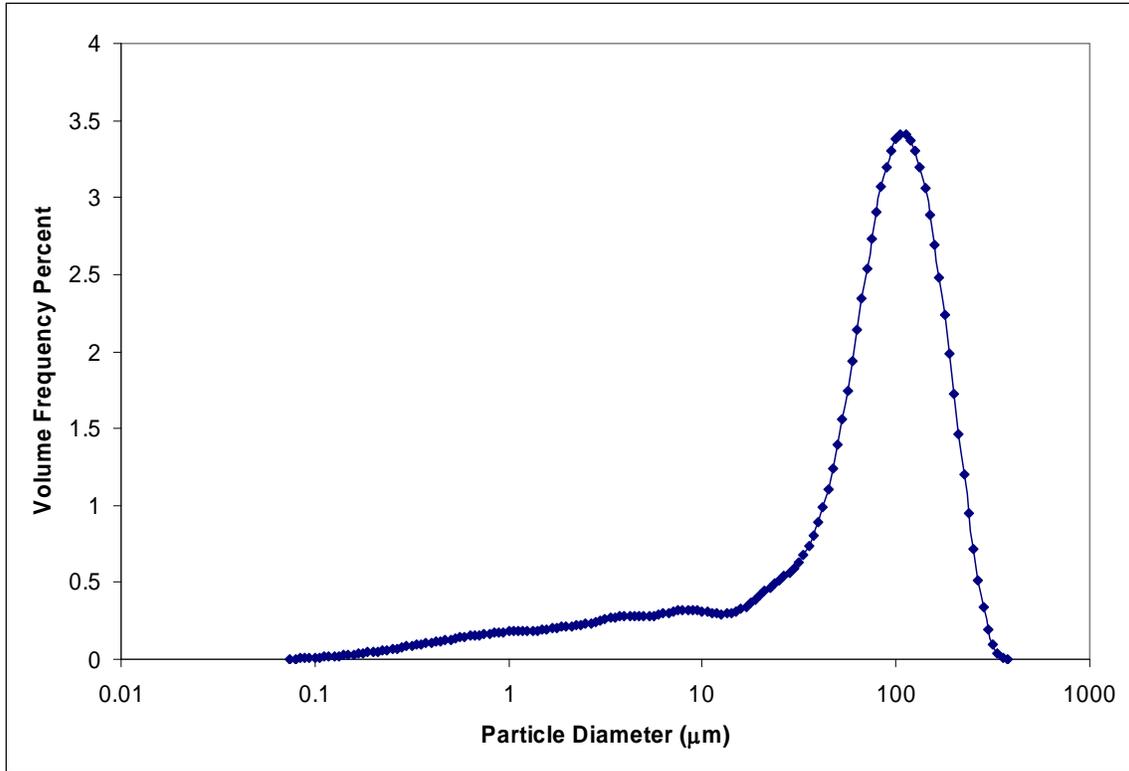


Figure A-3. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Las Vegas Wash 0.8 mile sample.

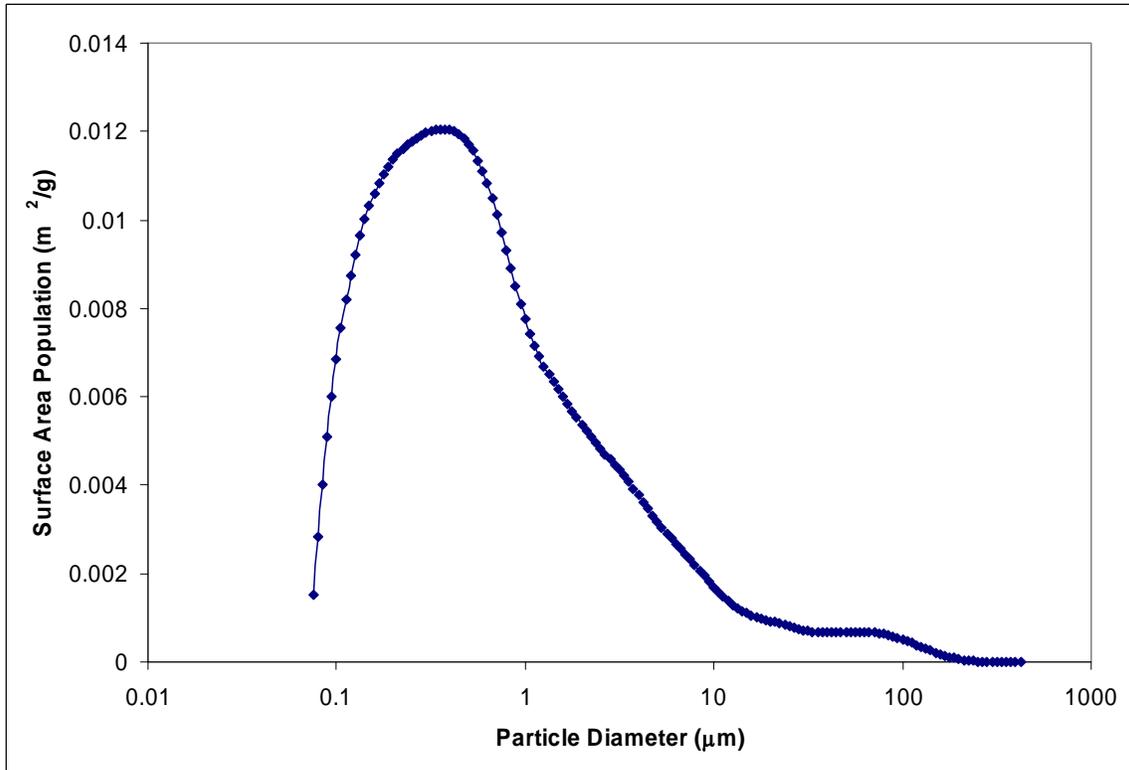
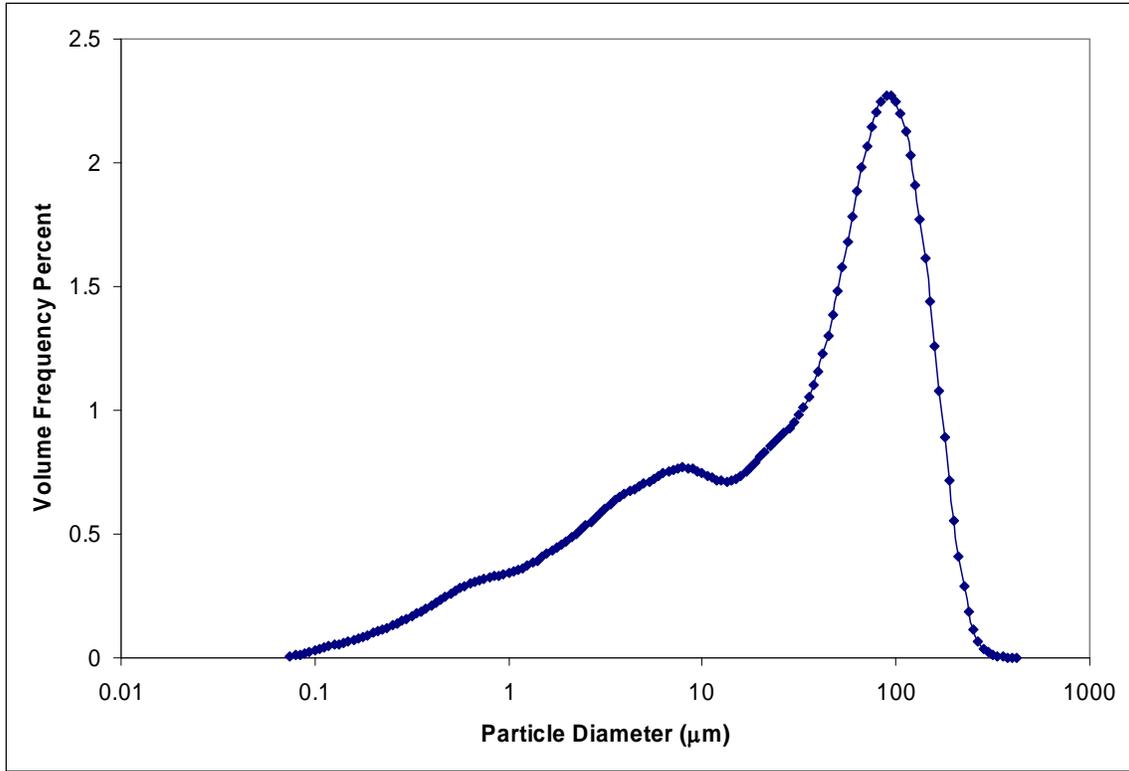


Figure A-4. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Las Vegas Wash 6.05 mile sample.

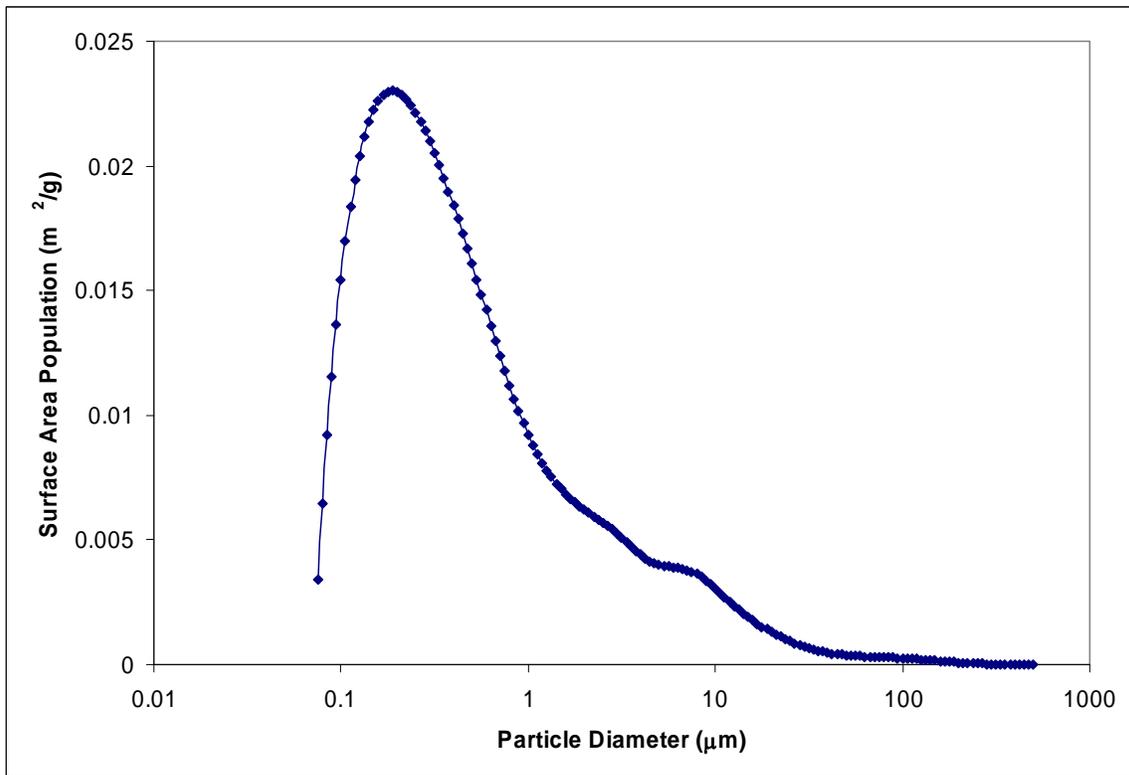
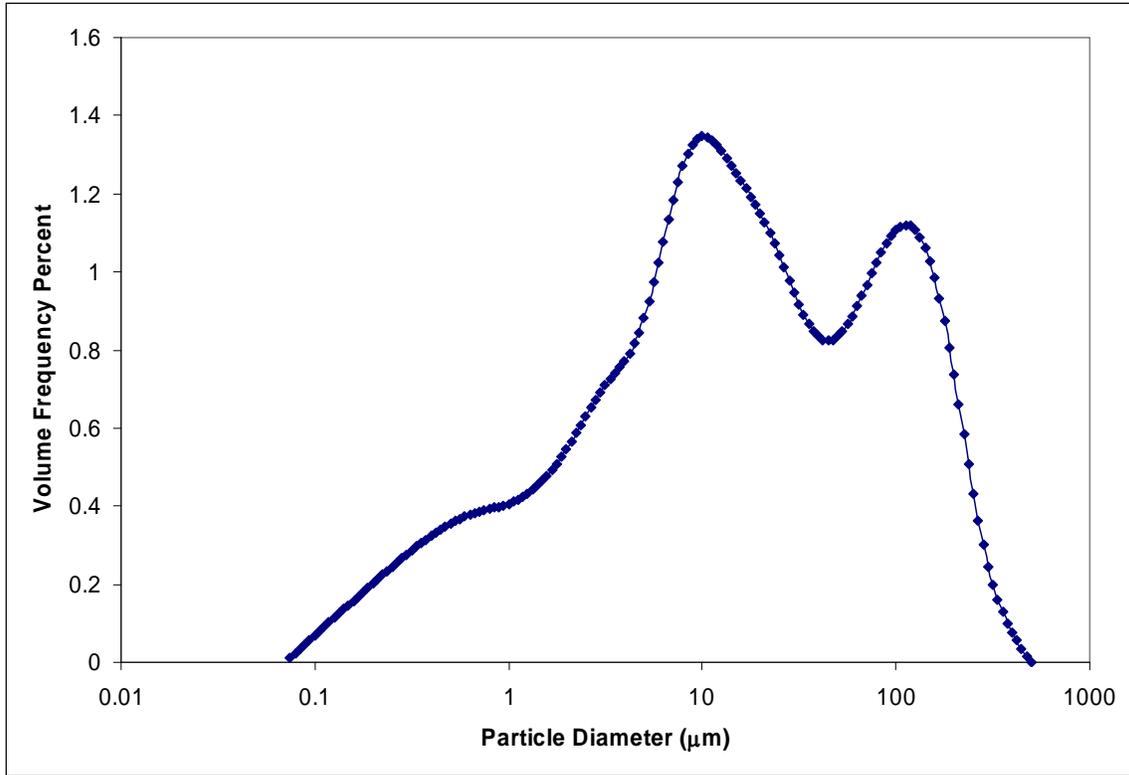


Figure A-5. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Las Vegas Wash 10.75 mile sample.

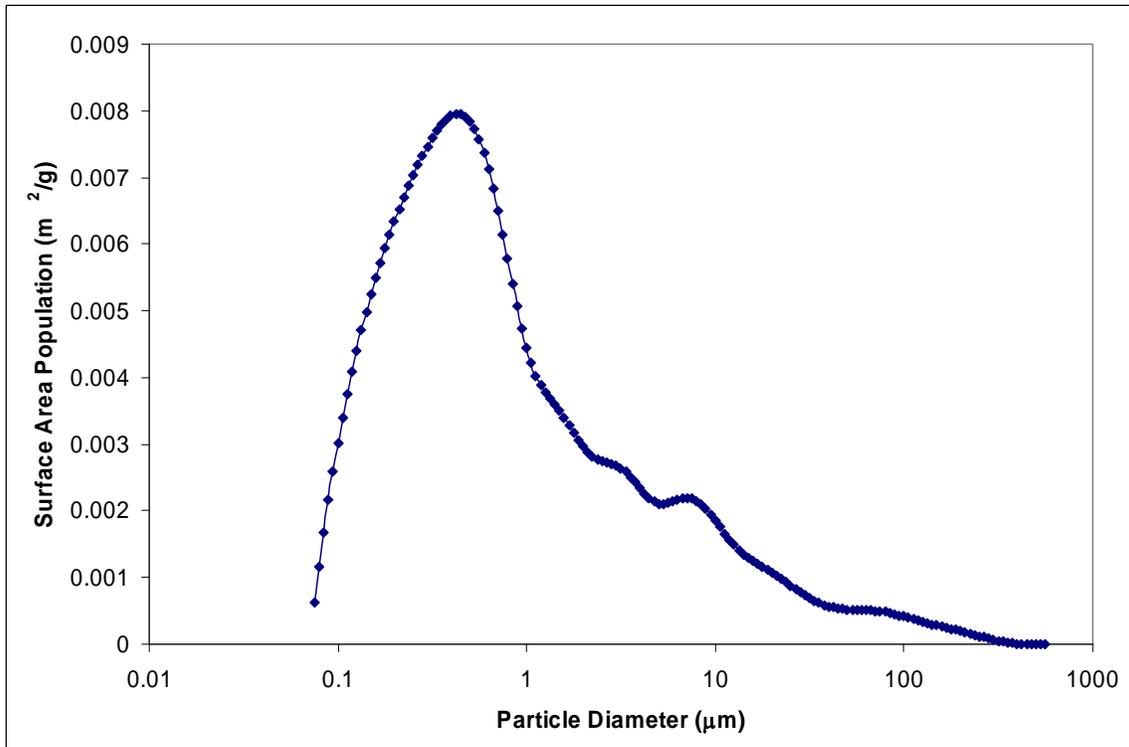
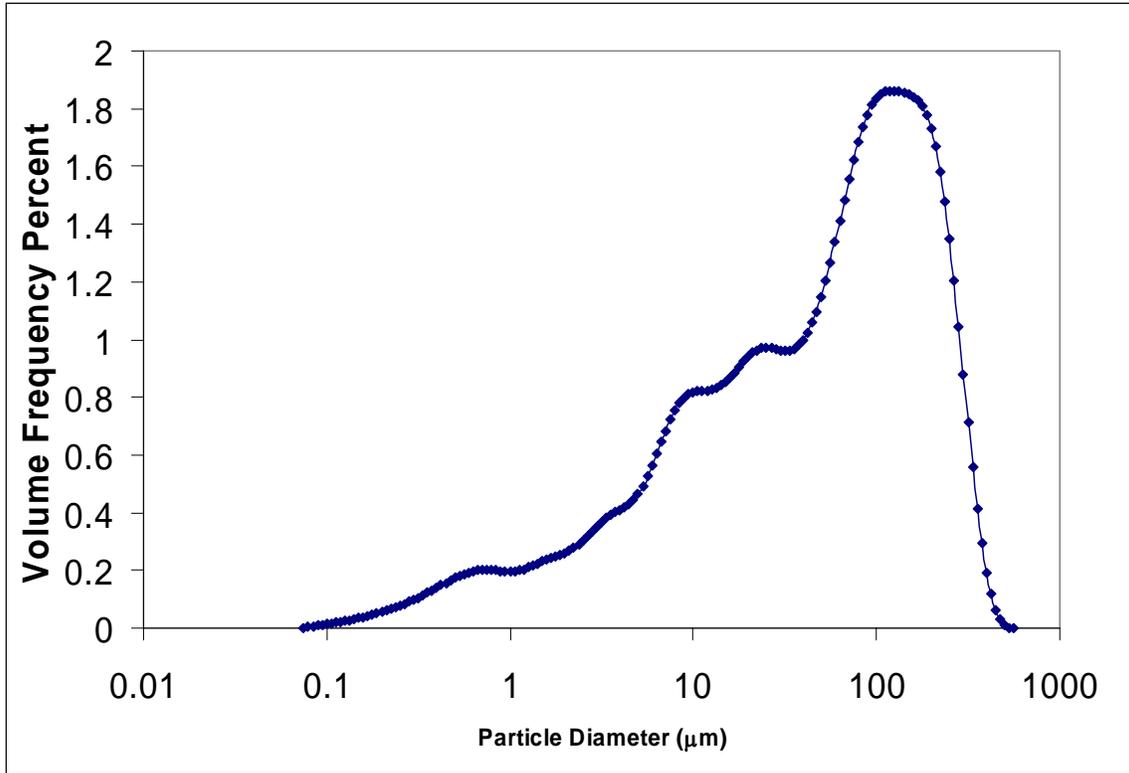


Figure A-6. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Duck Creek sample.

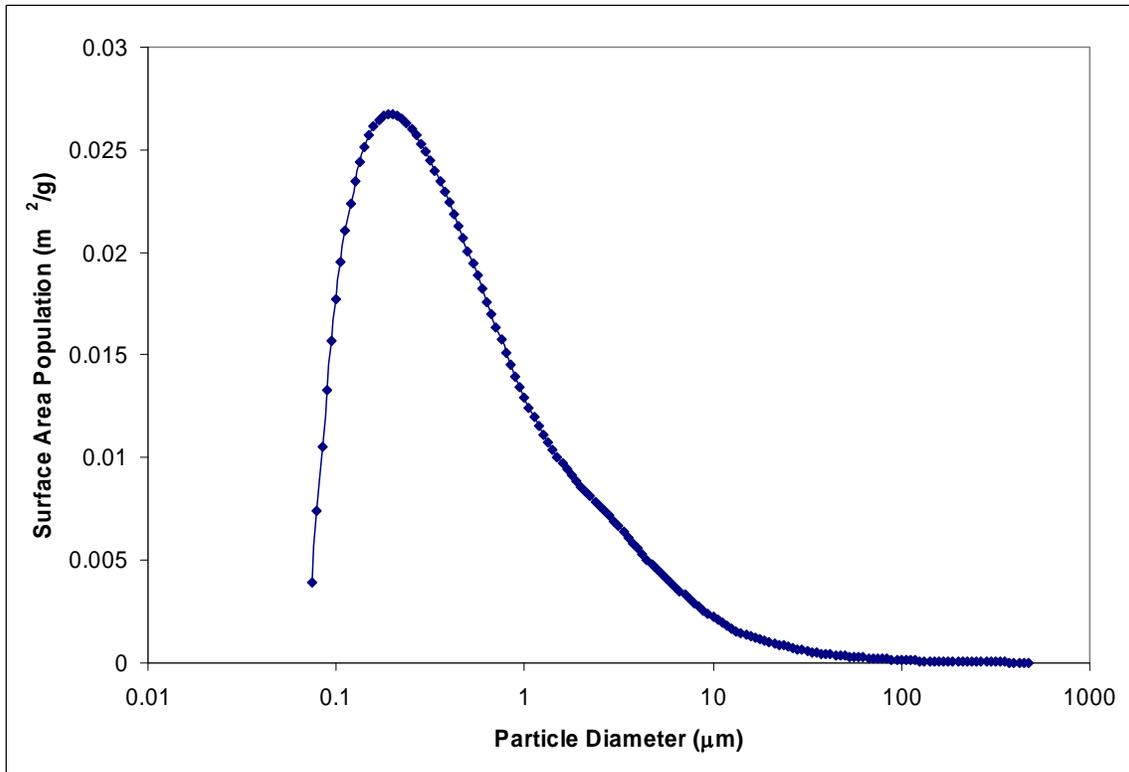
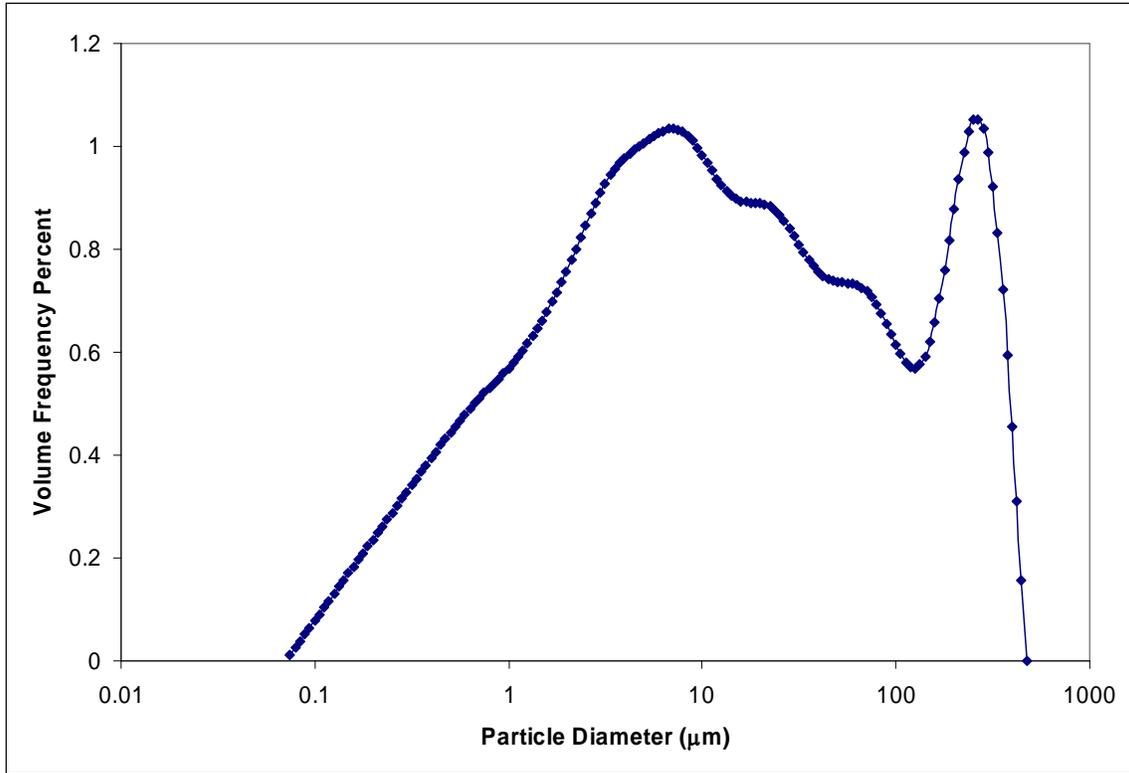
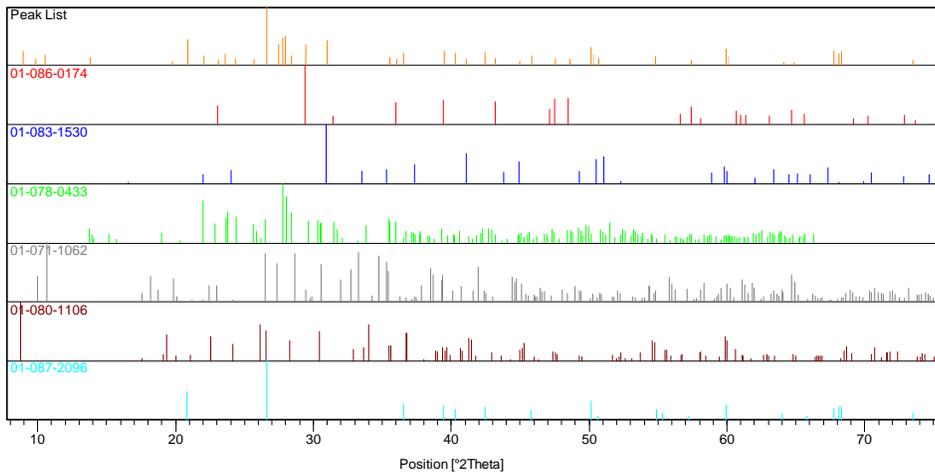
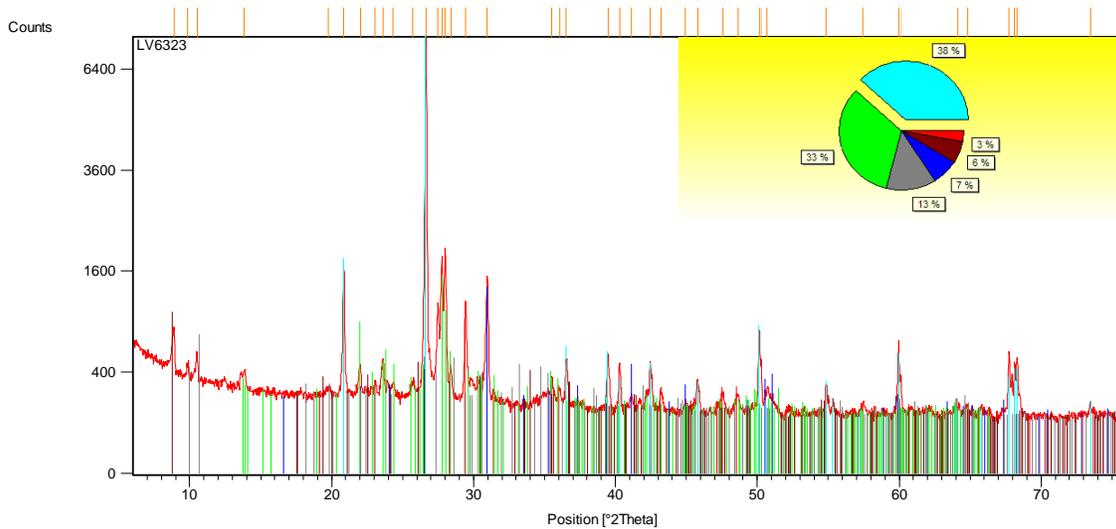


Figure A-7. (a) Particle volume frequency vs. diameter and (b) particle surface area population vs. diameter for the Nature Preserve sample.

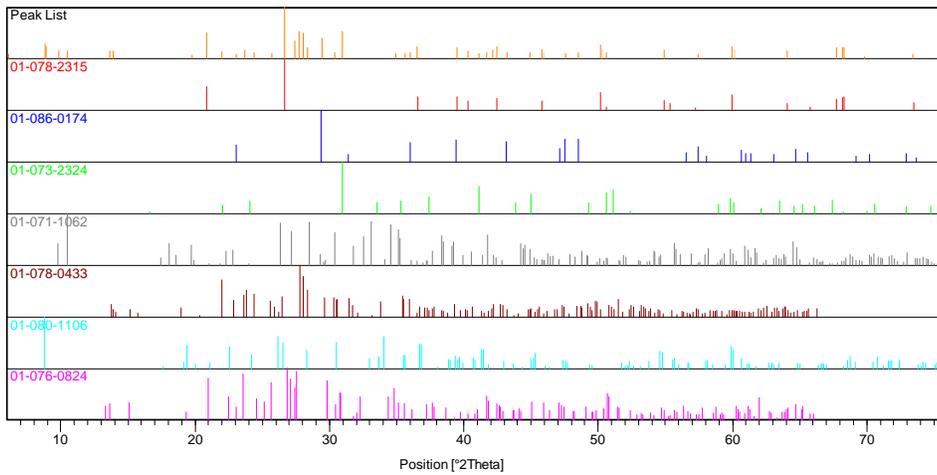
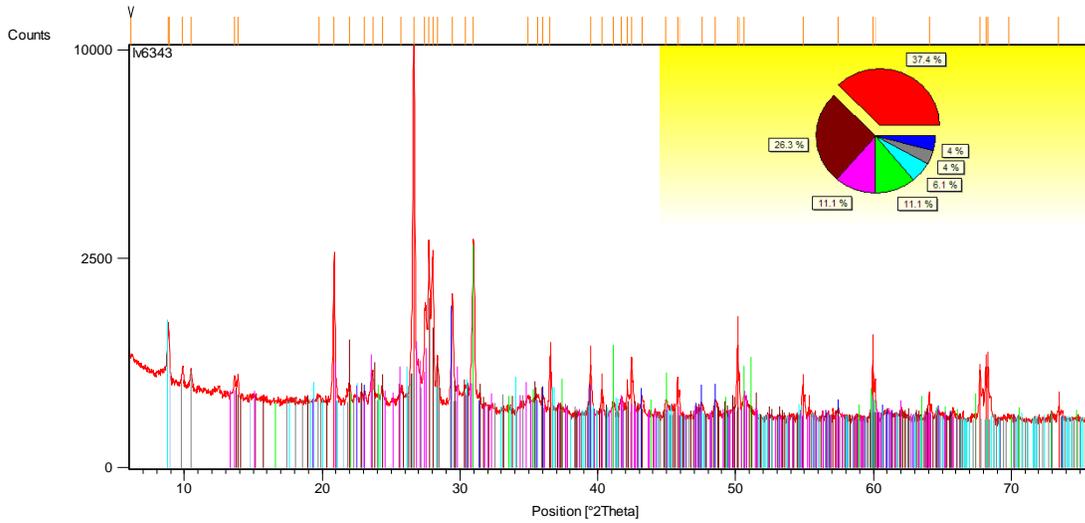
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APPENDIX B: Mineralogical Composition



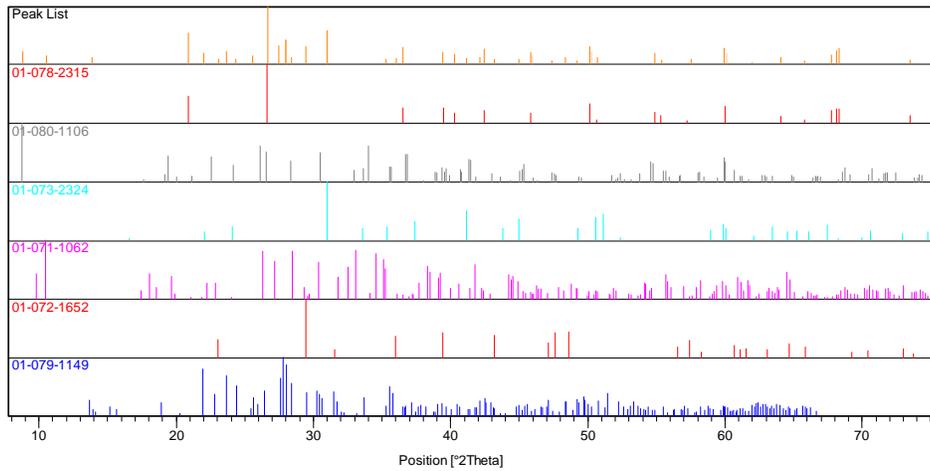
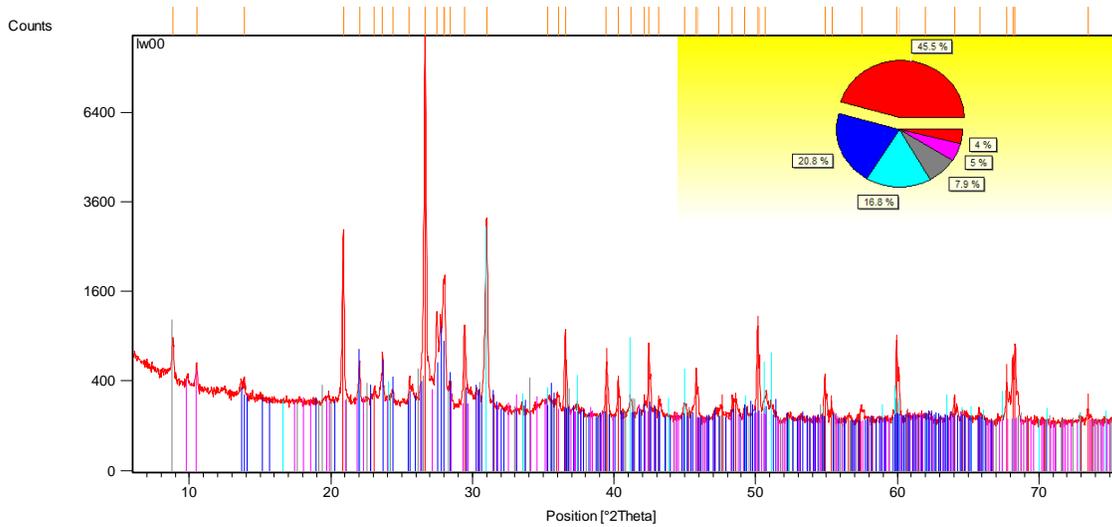
Ref. Code	Score	Compound Name	Chemical Formula
01-086-0174	55	Calcite, syn	Ca (C O3)
01-083-1530	31	Dolomite	Ca Mg (C O3)2
01-078-0433	35	Labradorite	Na0.45 Ca0.55 Al1.55 Si2.45 O8
01-071-1062	Unmatched Strong	Hornblende	(K.3 Na.6) (Ca1.7 Mg.3) (Mg3 Fe Fe.5 Al.3 Ti.2) Al1.6 Si6.4O22.5 (O H)1.5
01-080-1106	18	Biotite	K Fe Mg2 (Al Si3 O10) (O H)2
01-087-2096	75	Quartz low	Si O2

Figure B-1. X-ray diffraction spectrum and fitting results for the Las Vegas Bay sample.



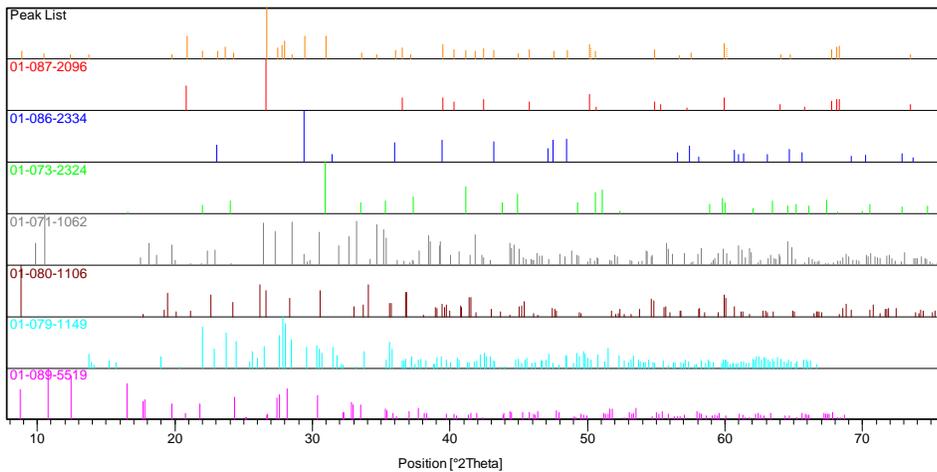
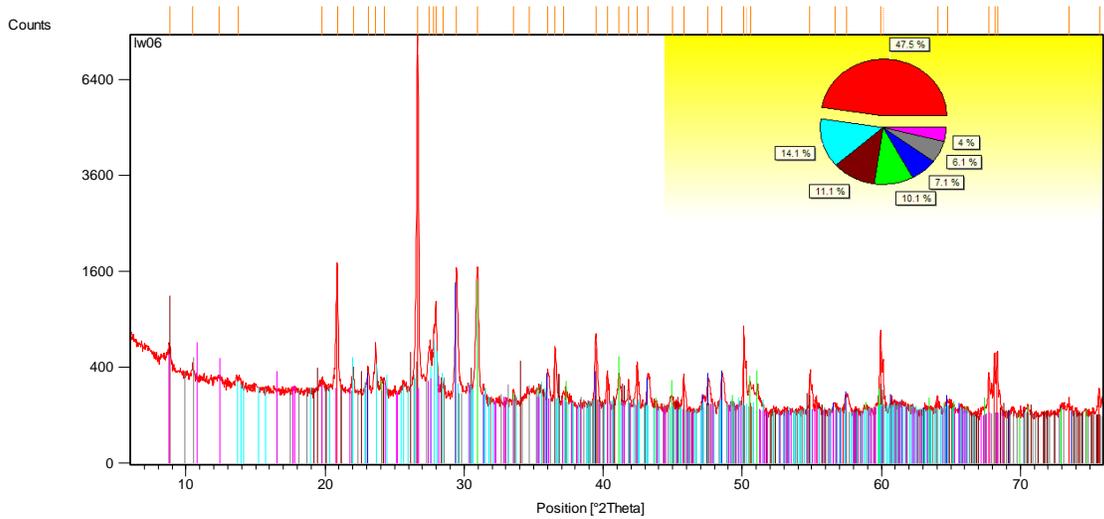
Ref. Code	Score	Compound Name	Chemical Formula
01-078-2315	73	Quartz	Si O ₂
01-086-0174	44	Calcite, syn	Ca (C O ₃)
01-073-2324	28	Dolomite	Ca Mg (C O ₃) ₂
01-071-1062	19	Hornblende	(K _{0.3} Na _{0.6}) (Ca _{1.7} Mg _{0.3}) (Mg ₃ Fe Fe _{0.5} Al _{0.3} Ti _{0.2}) Al _{1.6} Si _{6.4} O _{22.5} (O H) _{1.5}
01-078-0433	36	Labradorite	Na _{0.45} Ca _{0.55} Al _{1.55} Si _{2.45} O ₈
01-080-1106	21	Biotite	K Fe Mg ₂ (Al Si ₃ O ₁₀) (O H) ₂
01-076-0824	32	Orthoclase	(K _{0.931} Na _{0.055} Ca _{0.009} Ba _{0.005}) (Al _{0.97} Si _{3.03} O ₈)

Figure B-2. X-ray diffraction spectrum and fitting results for the Las Vegas Bay duplicate sample.



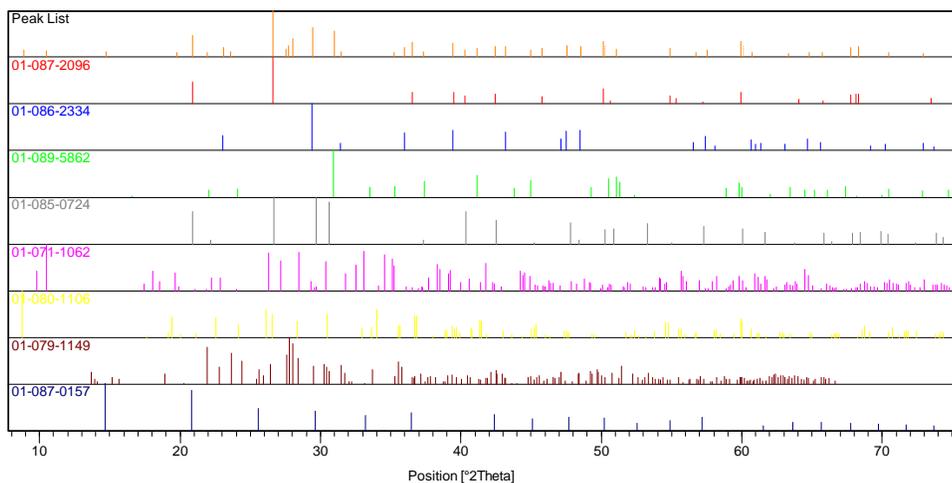
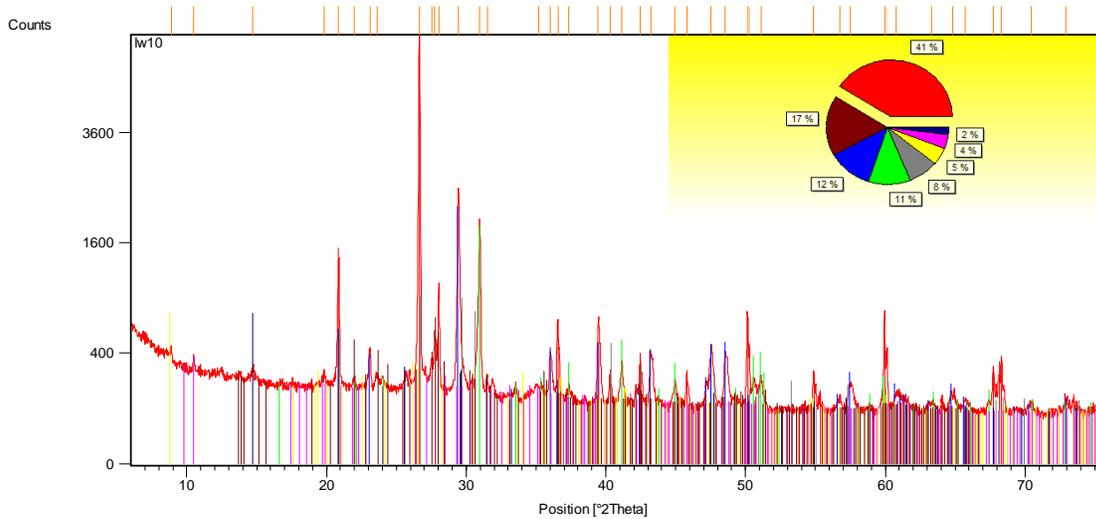
Ref. Code	Score	Compound Name	Chemical Formula
01-078-2315	79	Quartz	Si O ₂
01-080-1106	19	Biotite	K Fe Mg ₂ (Al Si ₃ O ₁₀) (O H) ₂
01-073-2324	35	Dolomite	Ca Mg (C O ₃) ₂
01-071-1062	16	Hornblende	(K ₃ Na ₆) (Ca _{1.7} Mg ₃) (Mg ₃ Fe Fe ₅ Al ₃ Ti ₂) Al _{11.6} Si _{6.4} O _{22.5} (O H) _{1.5}
01-072-1652	47	Calcite	Ca C O ₃
01-079-1149	32	Andesine	Na _{4.99} Ca _{4.91} (Al _{11.488} Si _{2.506} O ₈)

Figure B-3. X-ray diffraction spectrum and fitting results for the Las Vegas Wash 0.8 mile sample.



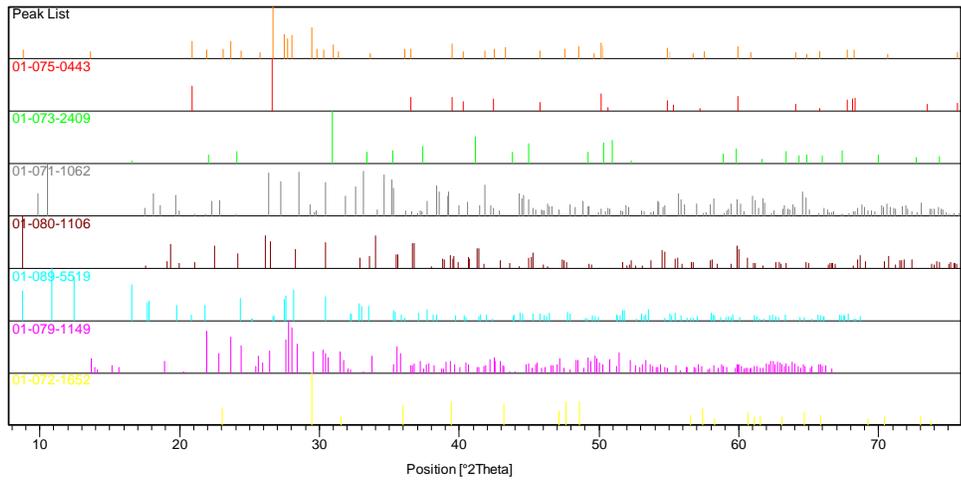
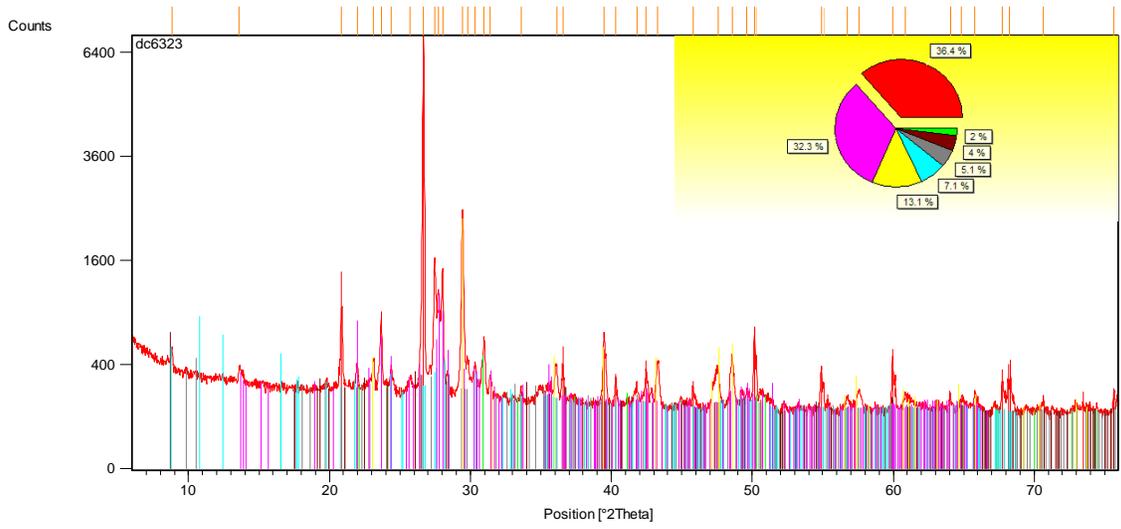
Ref. Code	Score	Compound Name	Chemical Formula
01-087-2096	76	Quartz low	Si O ₂
01-086-2334	59	Calcite	Ca (C O ₃)
01-073-2324	44	Dolomite	Ca Mg (C O ₃) ₂
01-071-1062	14	Hornblende	(K ₃ Na ₆) (Ca _{1.7} Mg ₃) (Mg ₃ Fe Fe ₅ Al ₃ Ti ₂) Al _{1.6} Si _{6.4} O _{22.5} (O H) _{1.5}
01-080-1106	18	Biotite	K Fe Mg ₂ (Al Si ₃ O ₁₀) (O H) ₂
01-079-1149	26	Andesine	Na _{4.99} Ca _{4.91} (Al _{1.488} Si _{2.506} O ₈)
01-089-5519	11	Merlinoite, syn	(C ₈ H ₂₀ N) _{0.8} H _{0.37} K _{5.53} (Al _{6.7} Si _{25.3} O ₆₄) (H ₂ O) _{15.62}

Figure B-4. X-ray diffraction spectrum and fitting results for the Las Vegas Wash 6.05 mile sample.



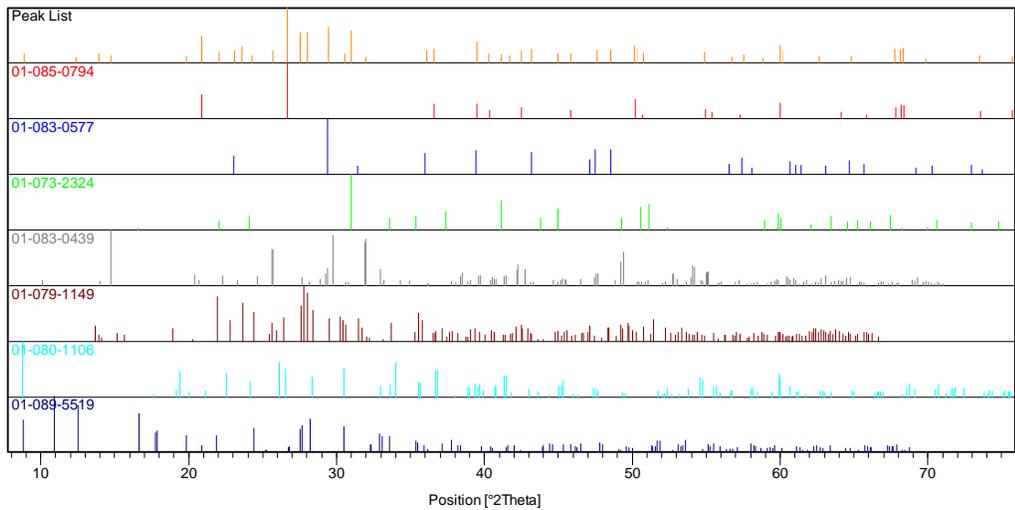
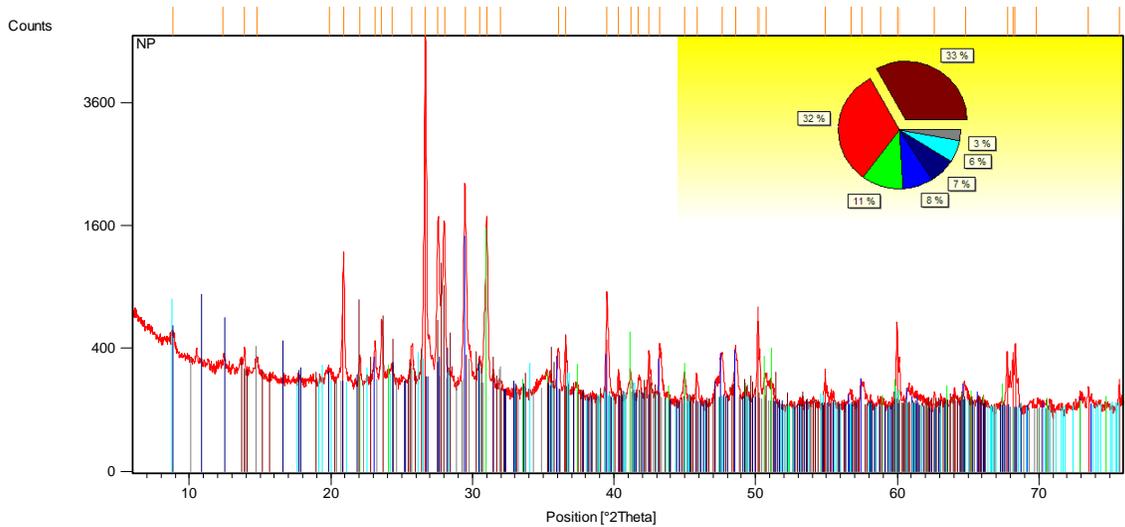
Ref. Code	Score	Compound Name	Chemical Formula
01-087-2096	73	Quartz low	Si O ₂
01-086-2334	65	Calcite	Ca (C O ₃)
01-089-5862	51	Dolomite	Ca Mg (C O ₃) ₂
01-085-0724	26	Ammonium Hydrogen Sulfide	(N H ₄) S H
01-071-1062	12	Hornblende	(K _{0.3} Na _{0.6}) (Ca _{1.7} Mg _{0.3}) (Mg ₃ Fe Fe _{0.5} Al _{1.3} Ti _{0.2}) Al _{1.6} Si _{6.4} O _{22.5} (O H) _{1.5}
01-080-1106	13	Biotite	K Fe Mg ₂ (Al Si ₃ O ₁₀) (O H) ₂
01-079-1149	27	Andesine	Na _{0.499} Ca _{0.491} (Al _{1.488} Si _{2.506} O ₈)
01-087-0157	33	Ammonium Tin Fluoride	(N H ₄) ₃ (Sn F ₆) F

Figure B-5. X-ray diffraction spectrum and fitting results for the Las Vegas Wash 10.75 mile sample.



Ref. Code	Score	Compound Name	Chemical Formula
01-075-0443	71	Quartz	Si O ₂
01-073-2409	29	Dolomite	Ca Mg (C O ₃) ₂
01-071-1062	9	Hornblende	(K ₃ Na ₆) (Ca _{1.7} Mg ₃) (Mg ₃ Fe Fe ₅ Al ₃ Ti ₂) Al _{11.6} Si _{6.4} O _{22.5} (O H) _{1.5}
01-080-1106	11	Biotite	K Fe Mg ₂ (Al Si ₃ O ₁₀) (O H) ₂
01-089-5519	13	Merlinoite, syn	(C ₈ H ₂₀ N) _{0.8} H _{0.37} K _{5.53} (Al _{6.7} Si _{25.3} O ₆₄) (H ₂ O) _{15.62}
01-079-1149	31	Andesine	Na _{4.99} Ca _{4.91} (Al _{11.488} Si _{2.506} O ₈)
01-072-1652	39	Calcite	Ca C O ₃

Figure B-6. X-ray diffraction spectrum and fitting results for the Duck Creek sample.



Ref. Code	Score	Compound Name	Chemical Formula
01-085-0794	73	Quartz	Si O ₂
01-083-0577	54	Calcite	Ca (C O ₃)
01-073-2324	39	Dolomite	Ca Mg (C O ₃) ₂
01-083-0439	21	Calcium Sulfate Hydrate	Ca (S O ₄) (H ₂ O) _{0.5}
01-079-1149	27	Andesine	Na _{0.499} Ca _{0.491} (Al _{1.488} Si _{2.506} O ₈)
01-080-1106	16	Biotite	K Fe Mg ₂ (Al Si ₃ O ₁₀) (O H) ₂
01-089-5519	12	Merlinoite, syn	(C ₈ H ₂₀ N) _{0.8} H _{0.37} K _{5.53} (Al _{6.7} Si _{25.3} O ₆₄) (H ₂ O) _{15.62}

Figure B-7. X-ray diffraction spectrum and fitting results for the Nature Preserve sample.

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APPENDIX C: Particle Morphology and Elemental Composition

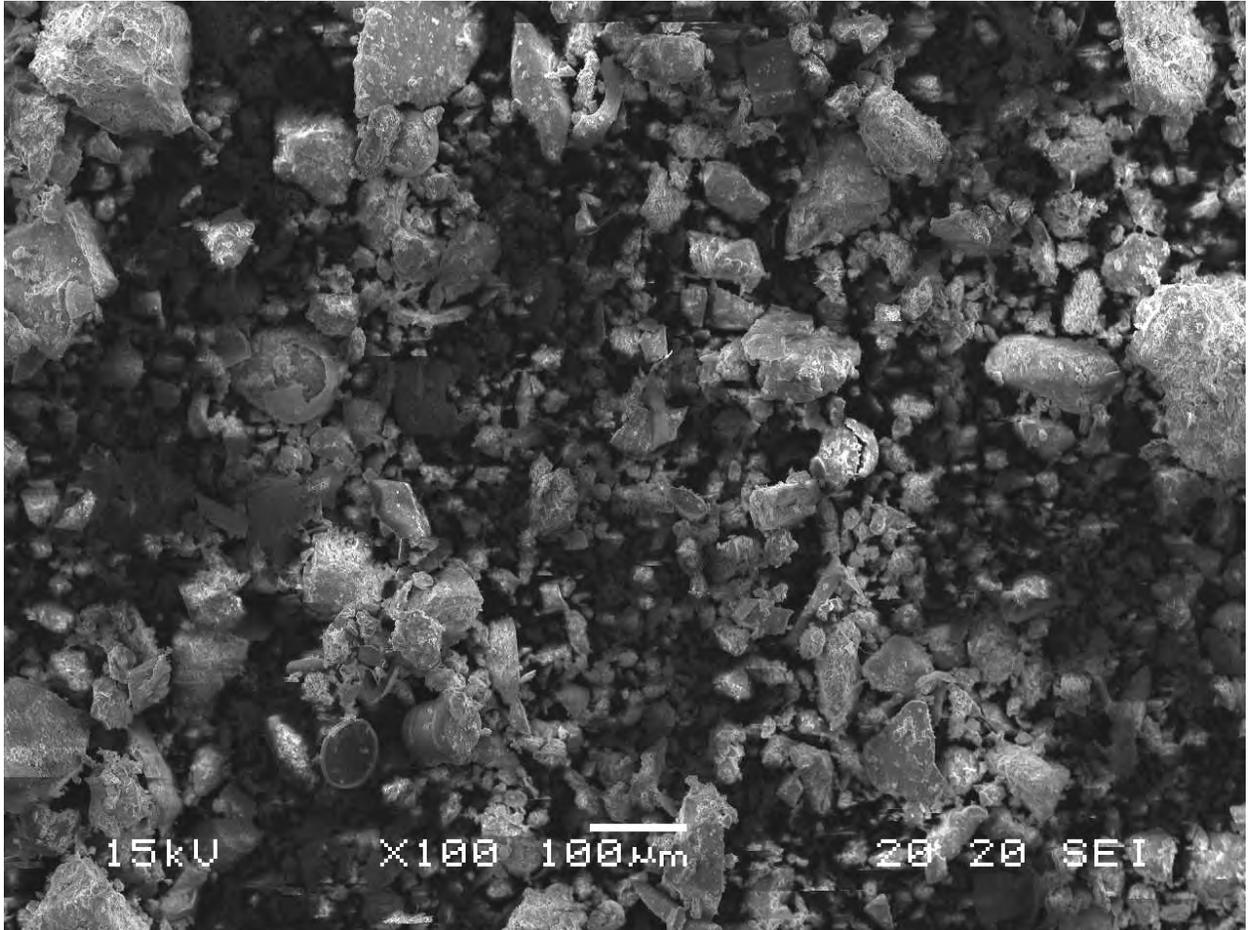


Figure C-1. General view of the Las Vegas Bay sample. Magnification 100x.

SEMQuant results. Listed at 10:24:33 AM on 2/27/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

3 peaks possibly omitted: 0.02, 2.14, 9.60 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	53.80	68.67
Na K	ED	0.15*	0.13*
Mg K	ED	4.11	3.45
Al K	ED	3.74	2.83
Si K	ED	26.00	18.90
K K	ED	1.76	0.92
Ca K	ED	8.93	4.55
Fe K	ED	1.52*	0.55*
Total		100.00	100.00

* = <2 Sigma

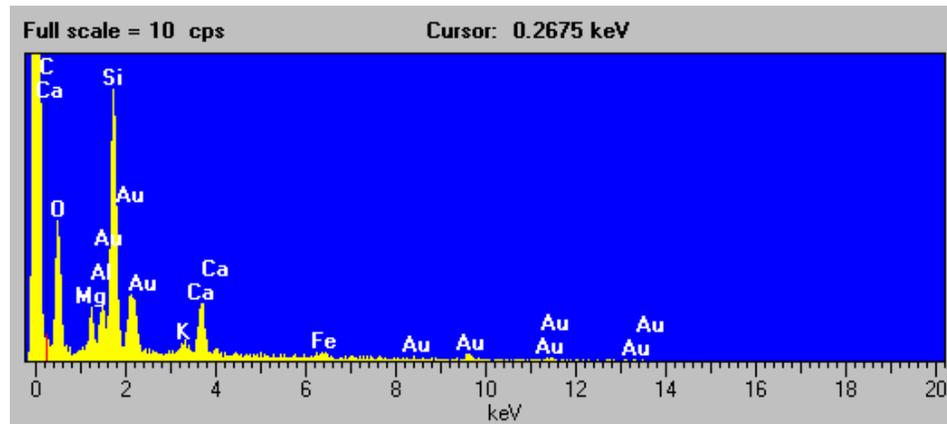
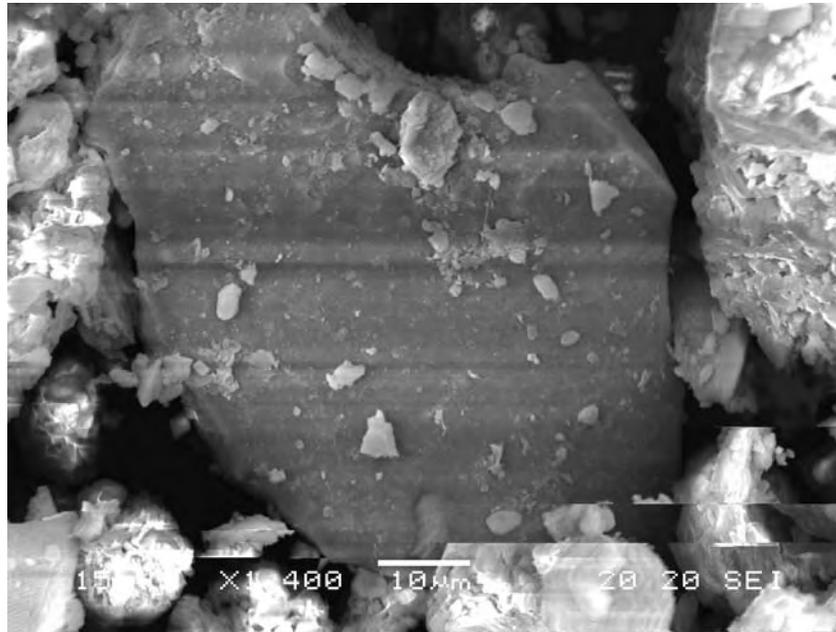


Figure C-2. Scanning electron micrograph, EDX spectrum, and elemental composition of the first particle of the Las Vegas Bay sample (magnification 1,400x).

SEMQuant results. Listed at 10:34:51 AM on 2/27/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (4 iterations).
 Analysed all elements and normalised results.

1 peak possibly omitted: 0.02 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	53.40	70.65
Na K	ED	-0.46*	-0.42*
Mg K	ED	7.64	6.65
Al K	ED	2.17	1.70
Si K	ED	9.33	7.03
K K	ED	0.83	0.45
Ca K	ED	24.68	13.03
Fe K	ED	2.40	0.91
Total		100.00	100.00

* = <2 Sigma

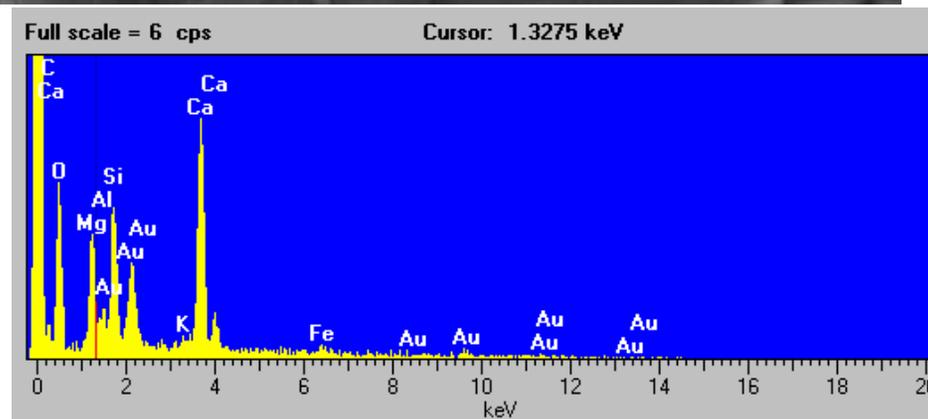
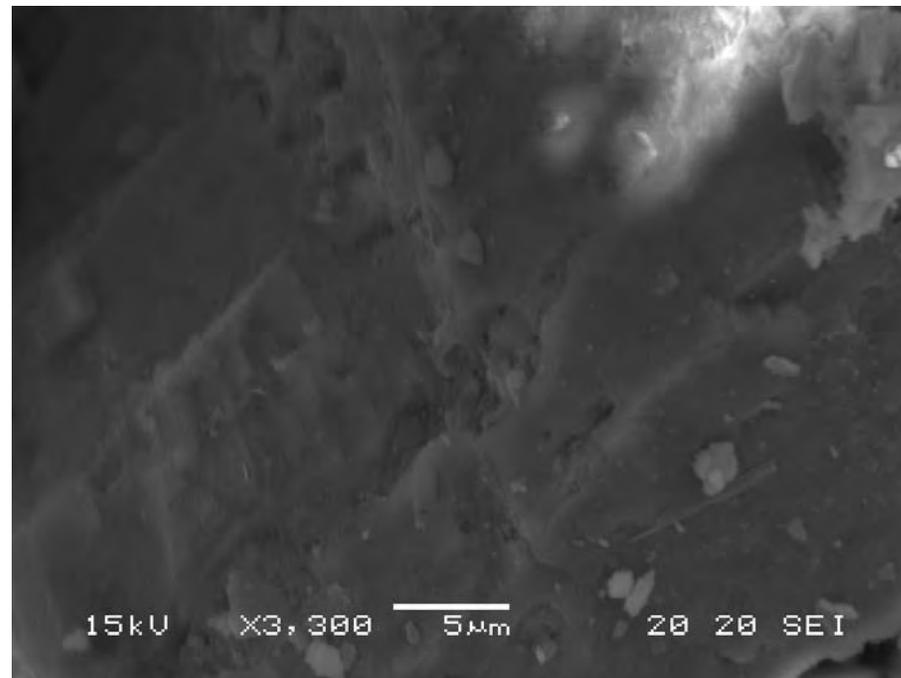


Figure C-3. Scanning electron micrograph, EDX spectrum, and elemental composition of the second particle of the Las Vegas Bay sample (magnification 3,300x).

SEMQuant results. Listed at 10:46:17 AM on 2/27/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic	Atomic
Type	%	%		
O K	ED	51.36	66.73	
Na K	ED	-0.12*	-0.10*	
Mg K	ED	1.63	1.39	
Al K	ED	1.76	1.36	
Si K	ED	31.93	23.63	
K K	ED	1.68	0.89	
Ca K	ED	11.76	6.10	
Fe K	ED	0.00*	0.00*	
Total		100.00	100.00	

* = <2 Sigma

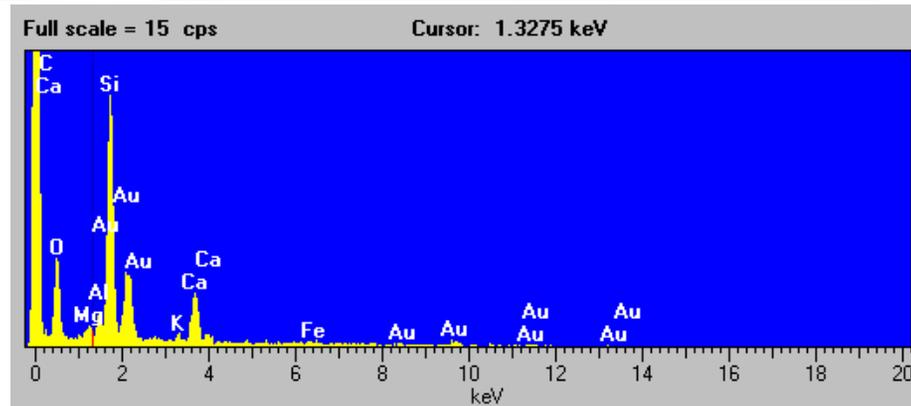
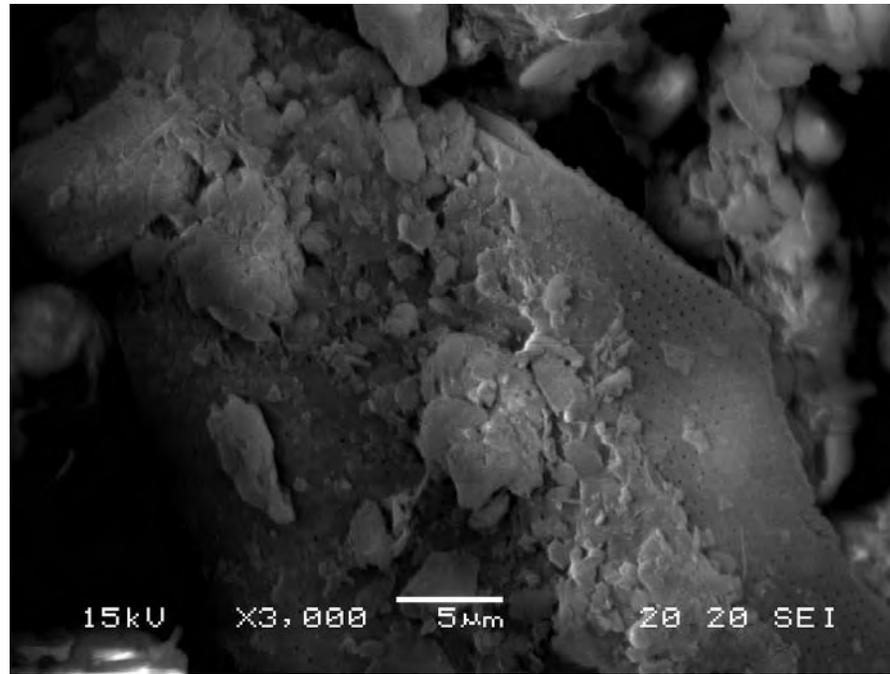


Figure C-4. Scanning electron micrograph, EDX spectrum, and elemental composition of the third particle of the Las Vegas Bay sample (magnification 3,000x).

SEMQuant results. Listed at 10:57:20 AM on 2/27/07

Operator: TracyB

Client: Tracy Boettcher

Job: Job number 51

Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (4 iterations).

Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93

Na K Albite 02/12/93

Mg K MgO 01/12/93

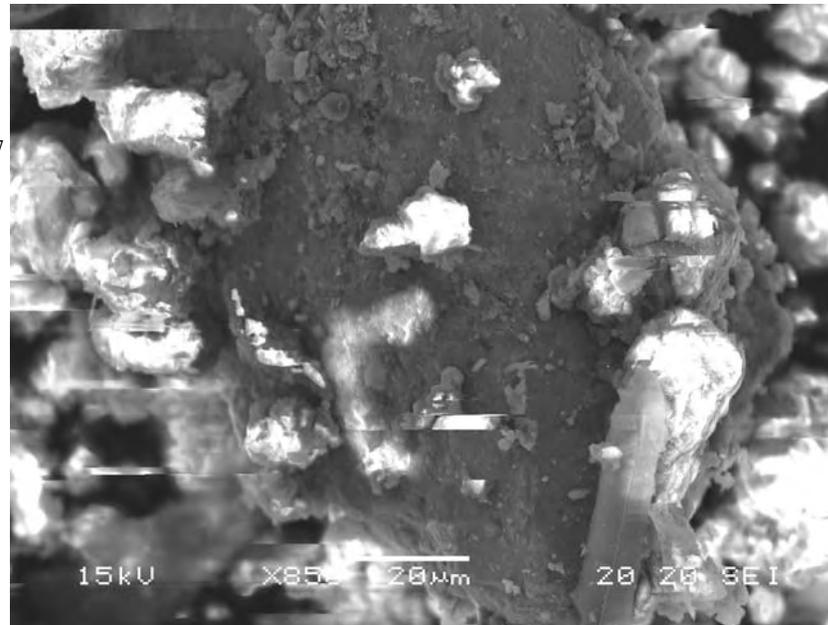
Al K Al₂O₃ 23/11/93

Si K Quartz 01/12/93

K K MAD-10 02/12/93

Ca K Wollas 23/11/93

Fe K Fe 01/12/93



Elmt	Spect.	Element	Atomic Type	%	%
O	K	ED		53.41	71.39
Na	K	ED		-0.10*	-0.09*
Mg	K	ED		2.34	2.06
Al	K	ED		3.16	2.50
Si	K	ED		16.02	12.20
K	K	ED		1.07	0.58
Ca	K	ED		14.18	7.56
Fe	K	ED		9.93	3.80
Total				100.00	100.00

* = <2 Sigma

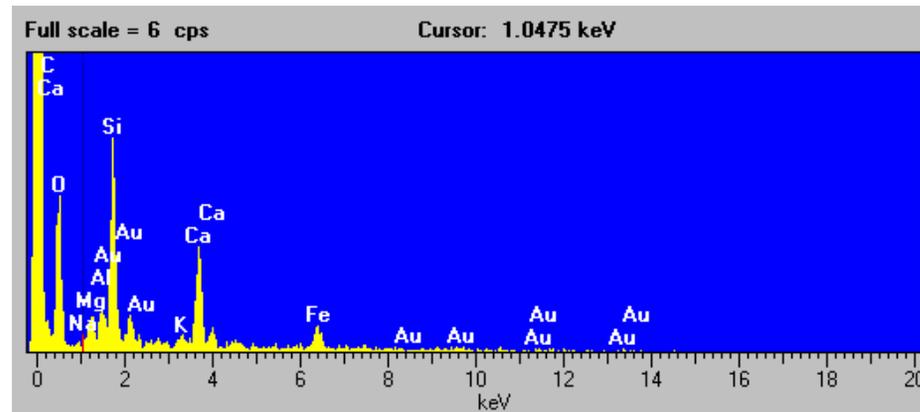


Figure C-5. Scanning electron micrograph, EDX spectrum, and elemental composition of the fourth particle of the Las Vegas Bay sample (magnification 850x).

SEMQuant results. Listed at 11:11:40 AM on 2/27/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic	Atomic
Type	%	%		
O K	ED	52.43	69.66	
Na K	ED	-0.58*	-0.54*	
Mg K	ED	4.53	3.96	
Al K	ED	2.26	1.78	
Si K	ED	19.13	14.48	
K K	ED	2.30	1.25	
Ca K	ED	12.11	6.42	
Fe K	ED	7.81	2.97	
Total		100.00	100.00	

* = <2 Sigma

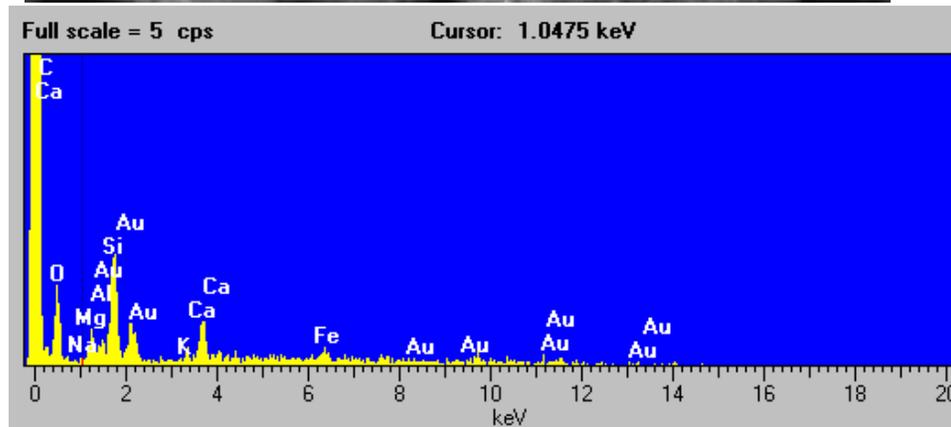
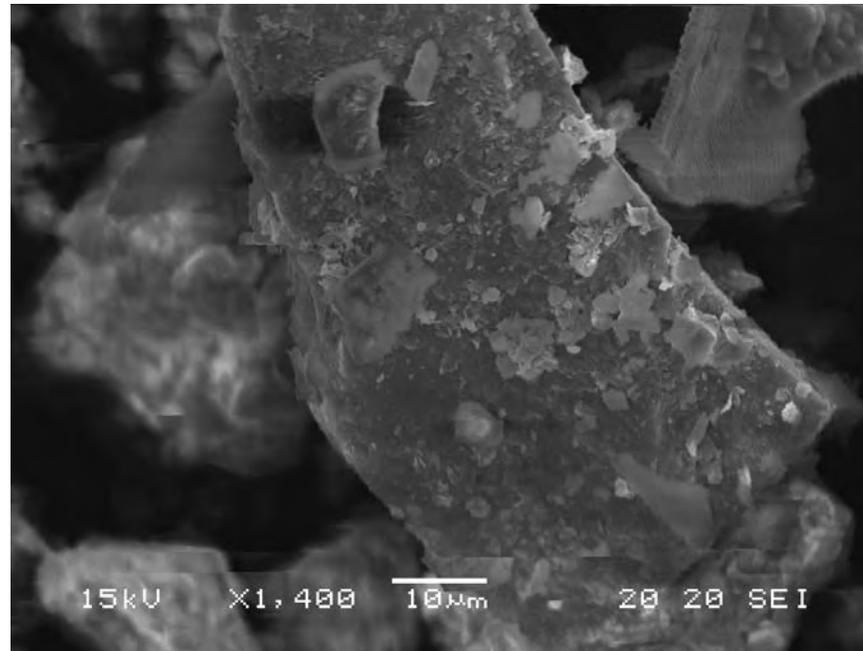


Figure C-6. Scanning electron micrograph, EDX spectrum, and elemental composition of the fifth particle of the Las Vegas Bay sample (magnification 1,400x).

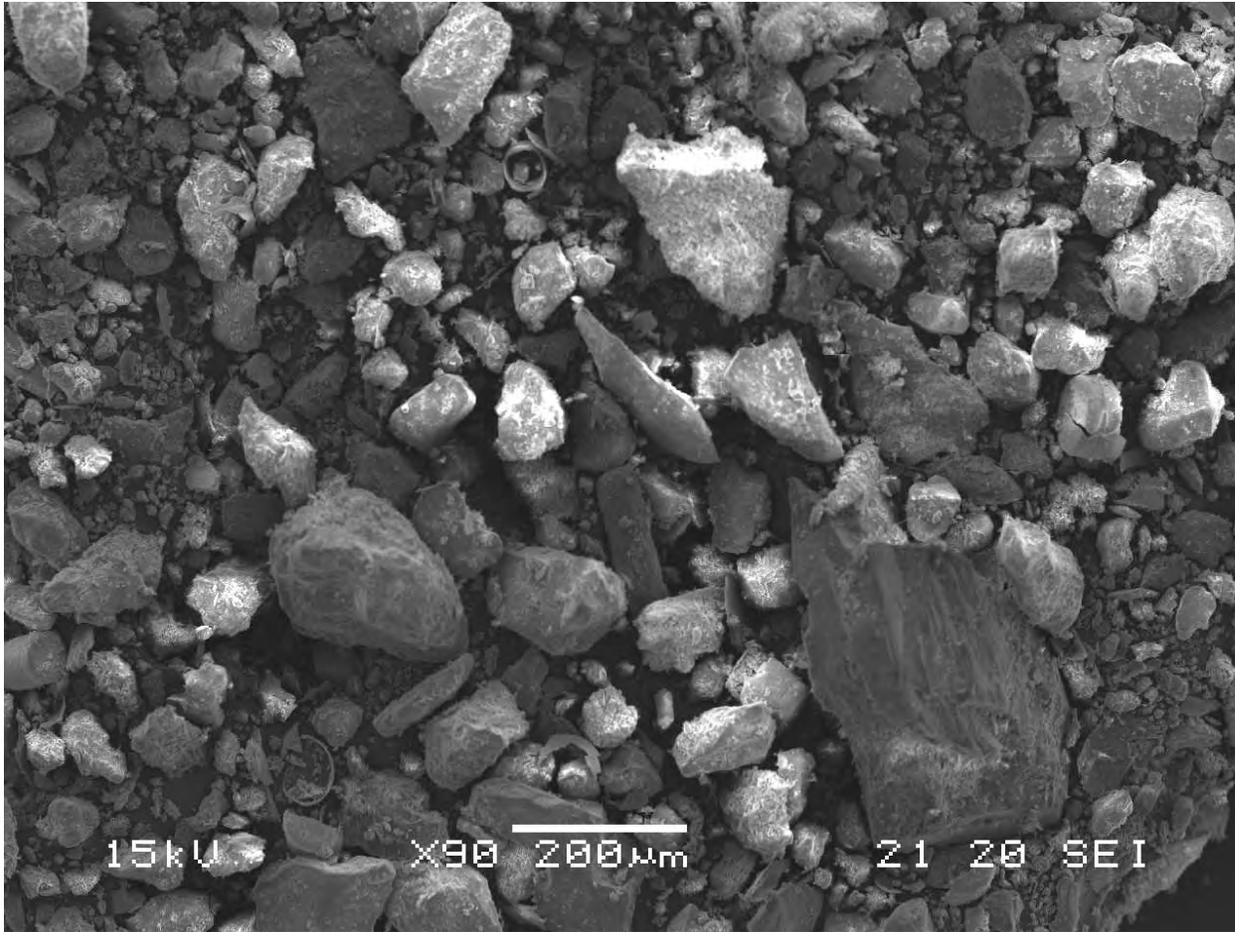


Figure C-7. General view of the Las Vegas Bay duplicate sample. Magnification 90x.

SEMQuant results. Listed at 10:19:50 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic Type	%	%
O	K	ED		53.68	68.66
Na	K	ED		1.41	1.26
Mg	K	ED		1.60	1.34
Al	K	ED		8.13	6.16
Si	K	ED		23.01	16.76
K	K	ED		2.11	1.10
Ca	K	ED		7.04	3.60
Fe	K	ED		3.02	1.11
Total				100.00	100.00

* = <2 Sigma

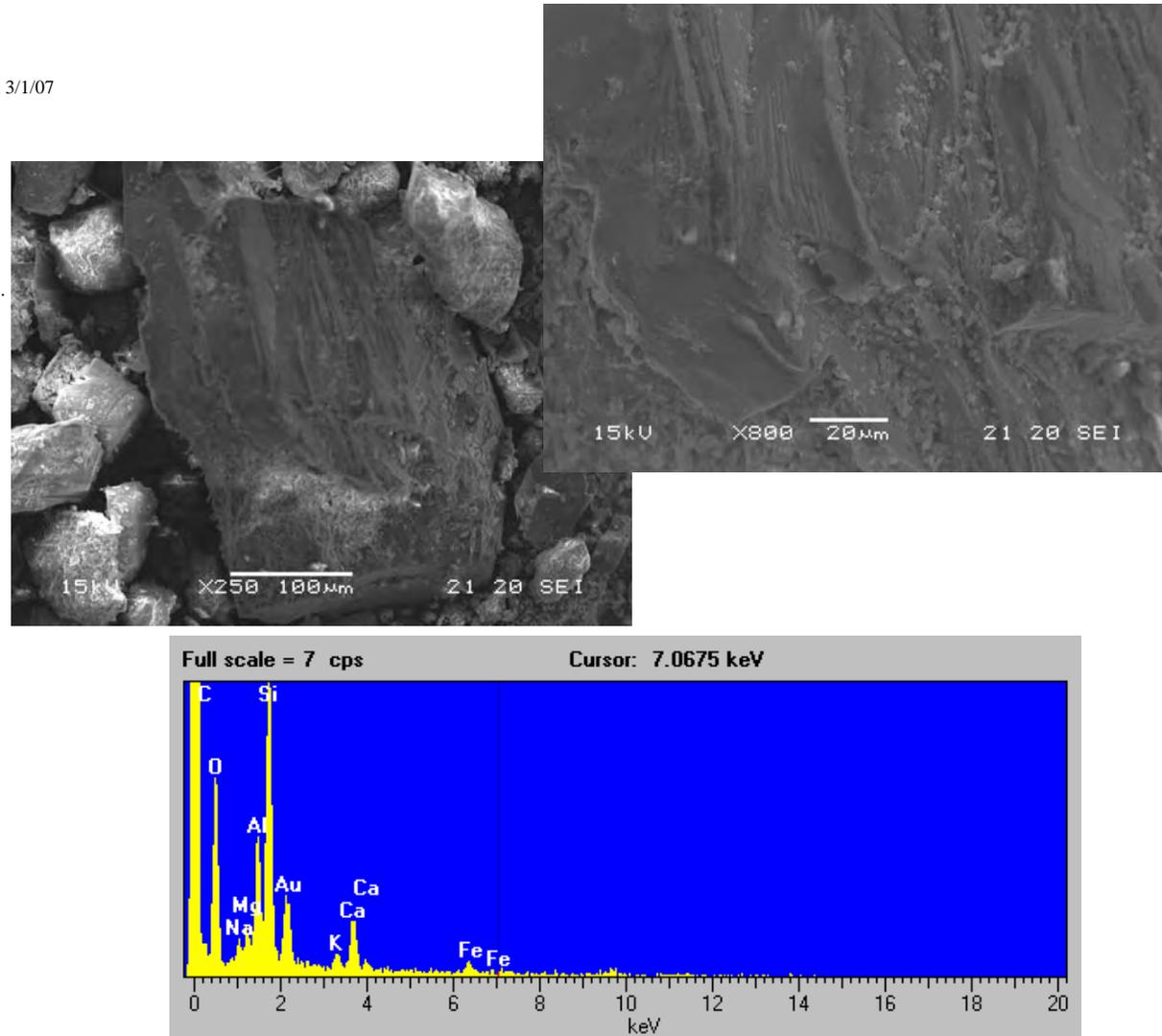


Figure C-8. Scanning electron micrograph, EDX spectrum, and elemental composition of the first particle of the Las Vegas Bay duplicate sample (magnifications 250x and 800x).

SEMQuant results. Listed at 10:29:49 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	50.09	64.84
Na K	ED	0.40*	0.36*
Mg K	ED	1.16	0.99
Al K	ED	0.60*	0.46*
Si K	ED	40.31	29.72
K K	ED	1.39	0.74
Ca K	ED	4.45	2.30
Fe K	ED	1.60*	0.59*
Total		100.00	100.00

* = <2 Sigma

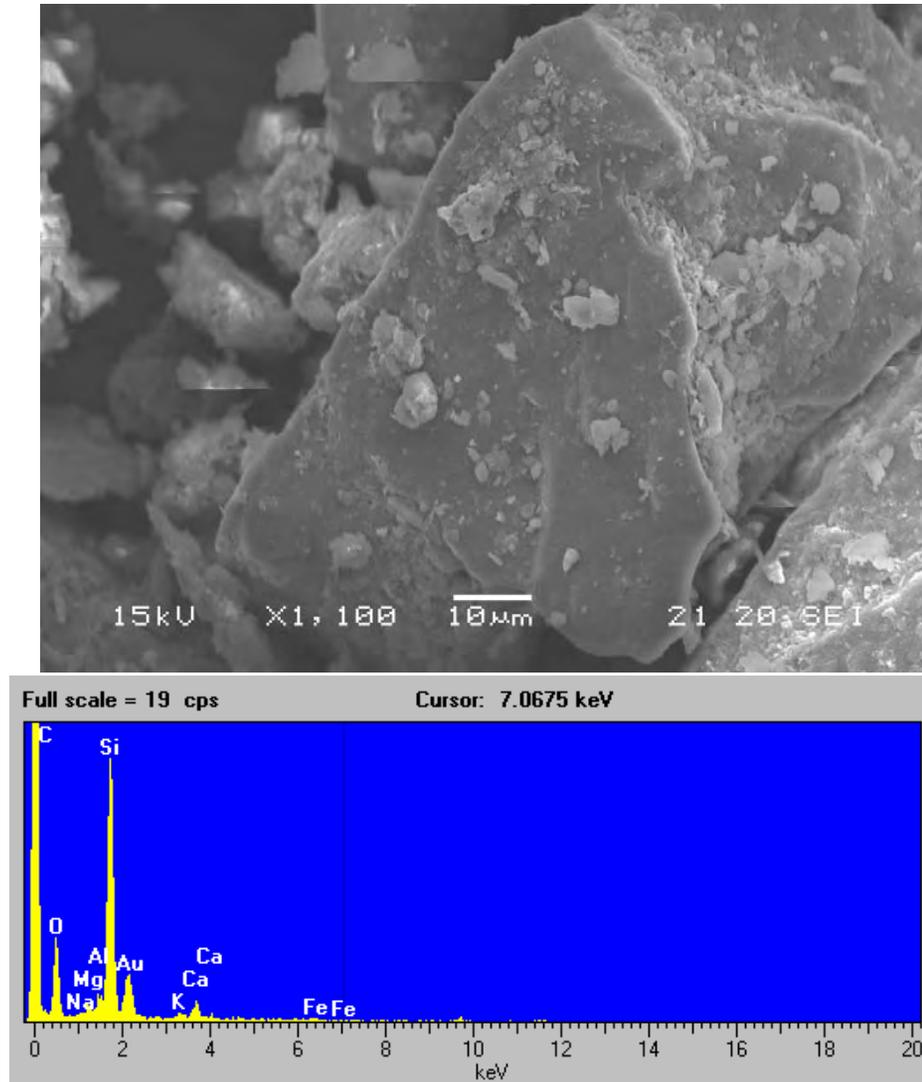


Figure C-9. Scanning electron micrograph, EDX spectrum, and elemental composition of the second particle of the Las Vegas Bay duplicate sample (magnification 1,100x).

SEMQuant results. Listed at 10:38:32 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

3 peaks possibly omitted: 0.02, 2.14, 9.62 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	53.44	68.35
Na K	ED	1.05*	0.93*
Mg K	ED	1.88	1.58
Al K	ED	2.16	1.64
Si K	ED	30.23	22.03
K K	ED	1.48	0.78
Ca K	ED	7.76	3.96
Fe K	ED	2.00*	0.73*
Total		100.00	100.00

* = <2 Sigma

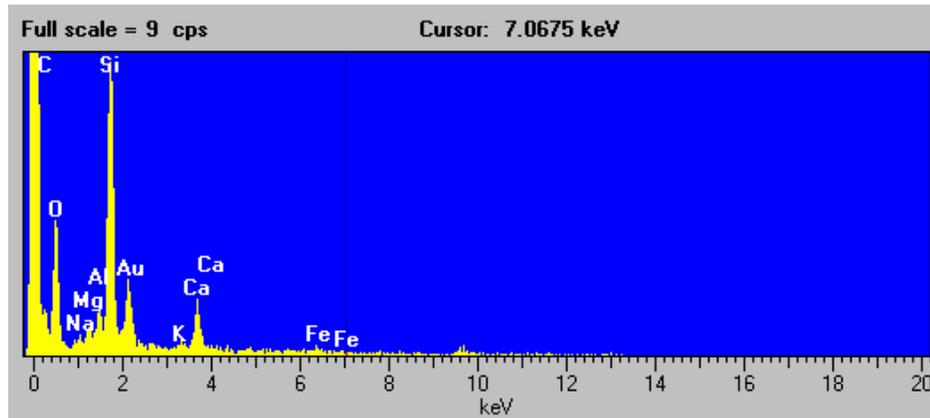
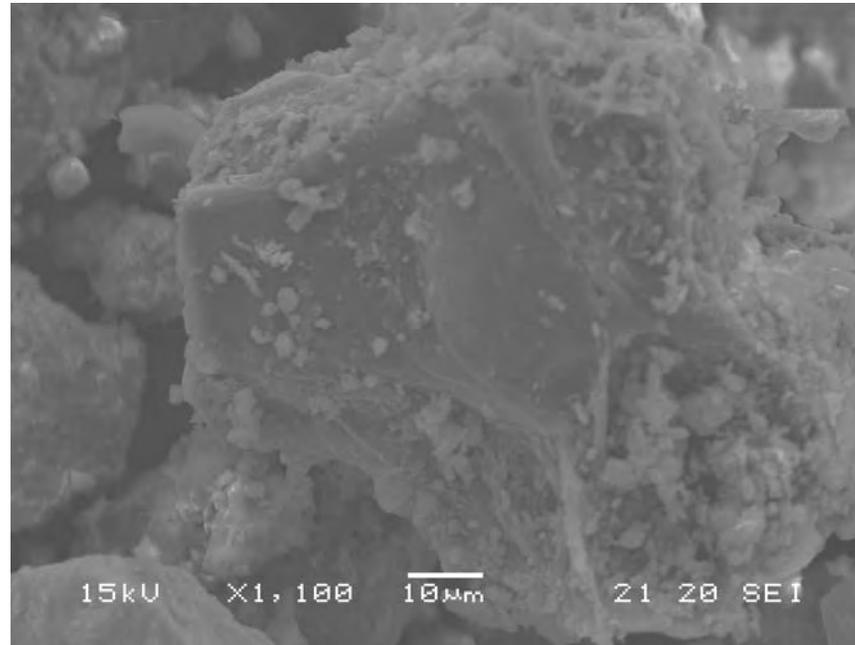


Figure C-10. Scanning electron micrograph, EDX spectrum, and elemental composition of the third particle of the Las Vegas Bay duplicate sample (magnification 1,100x).

SEMQuant results. Listed at 10:42:28 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	53.10	66.97
Na K	ED	0.16*	0.14*
Mg K	ED	0.70	0.58
Al K	ED	-0.36*	-0.27*
Si K	ED	43.16	31.01
K K	ED	0.36*	0.19*
Ca K	ED	2.43	1.22
Fe K	ED	0.45*	0.16*
Total		100.00	100.00

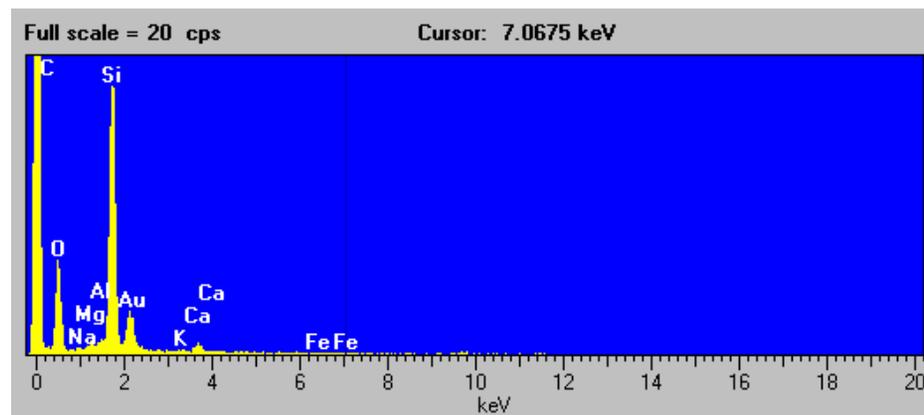
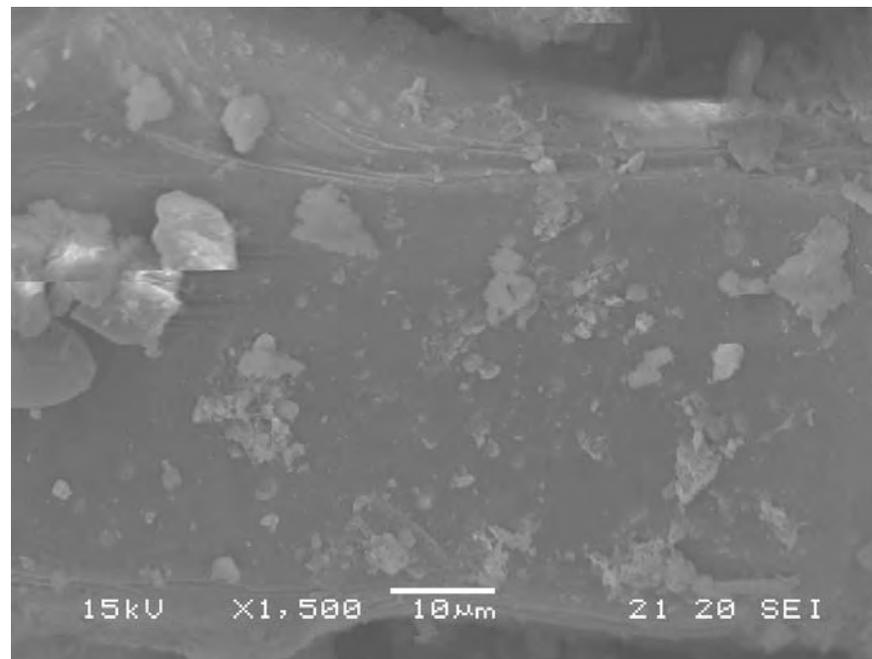


Figure C-11. Scanning electron micrograph, EDX spectrum, and elemental composition of the fourth particle of the Las Vegas Bay duplicate sample (magnification 1,100x).

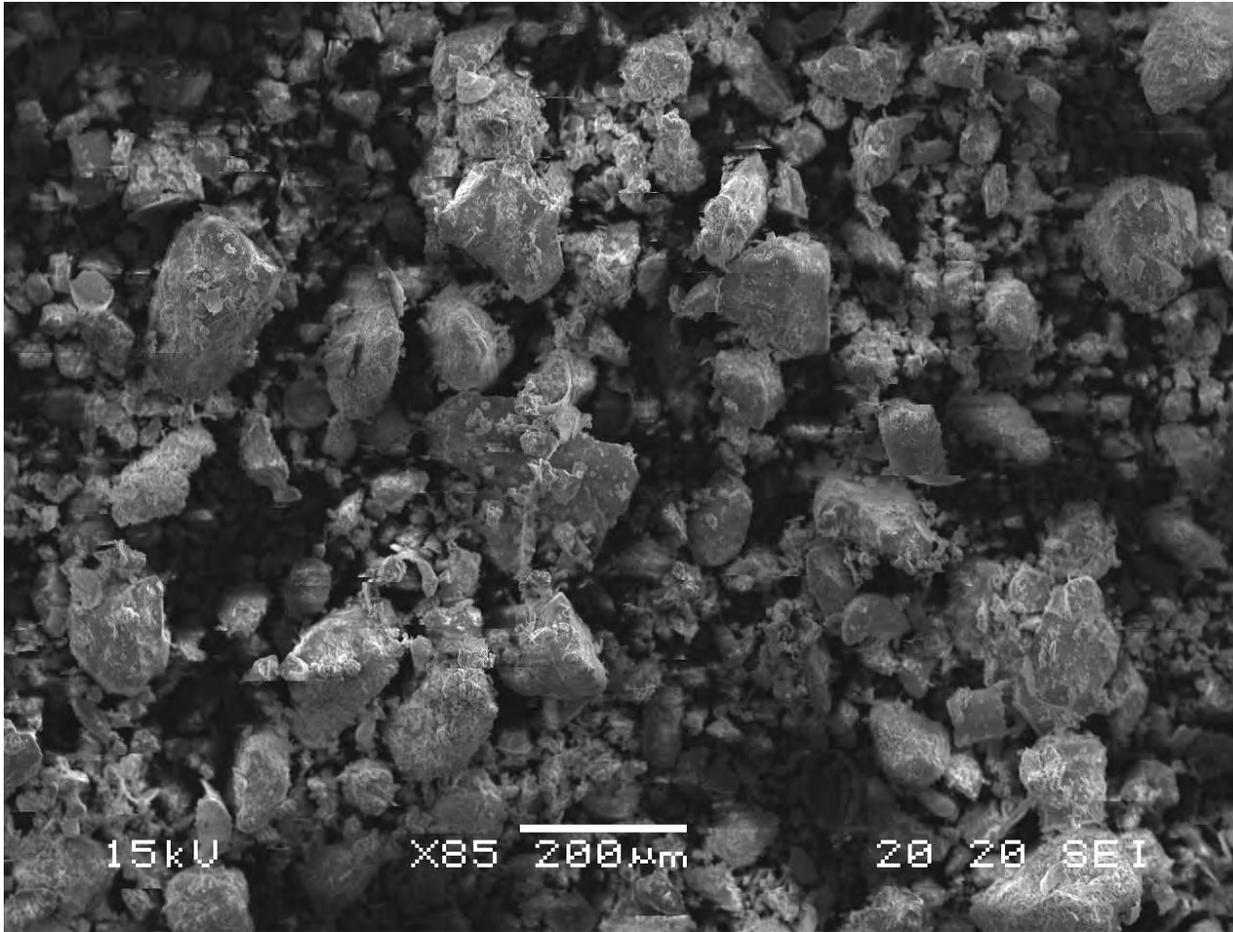


Figure C-12. General view of the Las Vegas Wash 0.8 mile sample. Magnification 85x.

SEMQuant results. Listed at 11:53:37 AM on 2/27/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al2O3 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	55.33	69.99
Na K	ED	0.44*	0.39*
Mg K	ED	2.49	2.07
Al K	ED	3.10	2.33
Si K	ED	28.16	20.29
K K	ED	1.43	0.74
Ca K	ED	6.42	3.24
Fe K	ED	2.64	0.95
Total		100.00	100.00

* = <2 Sigma

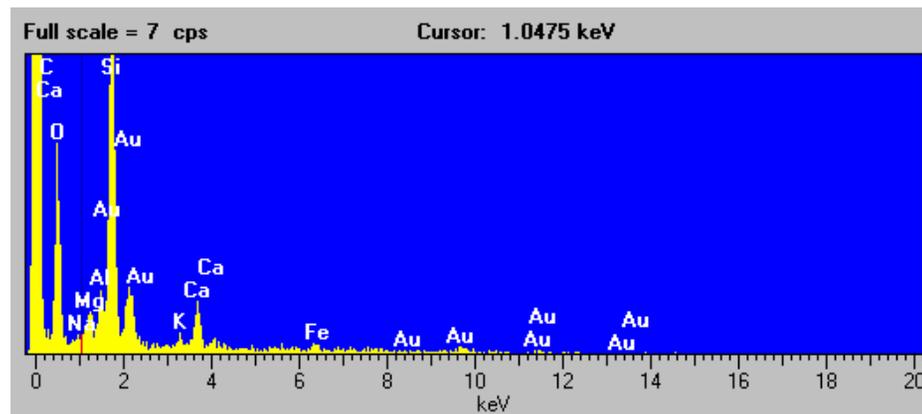
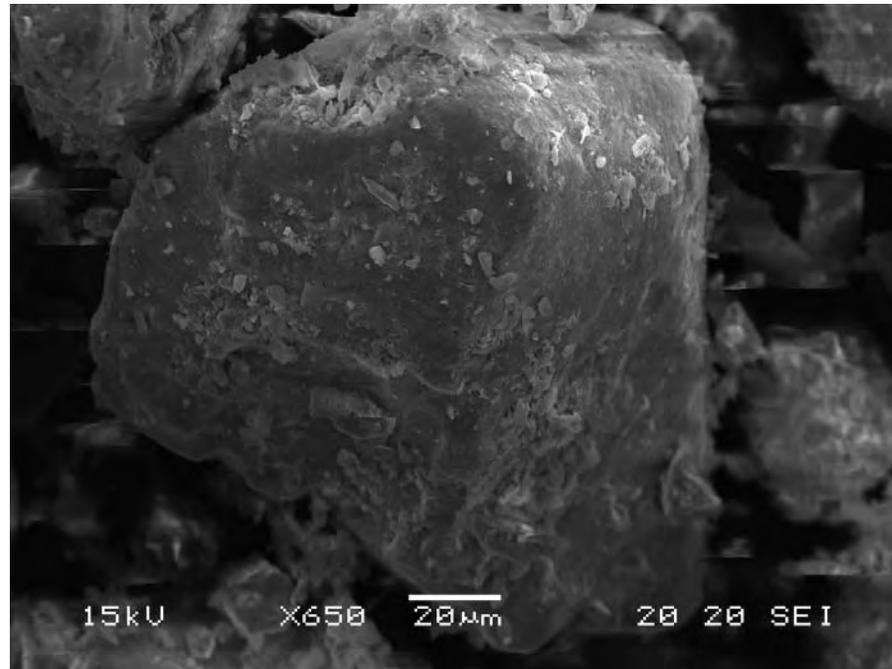


Figure C-13. Scanning electron micrograph, EDX spectrum, and elemental composition of the first particle of the Las Vegas Wash 0.8 mile sample (magnification 650x).

SEMQuant results. Listed at 11:59:25 AM on 2/27/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

1 peak possibly omitted: 0.02 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic	Atomic
Type	%	%		
O K	ED	52.87	69.21	
Na K	ED	0.31*	0.28*	
Mg K	ED	3.06	2.64	
Al K	ED	3.53	2.74	
Si K	ED	22.37	16.68	
K K	ED	1.91	1.03	
Ca K	ED	9.78	5.11	
Fe K	ED	6.16	2.31	
Total		100.00	100.00	

* = <2 Sigma

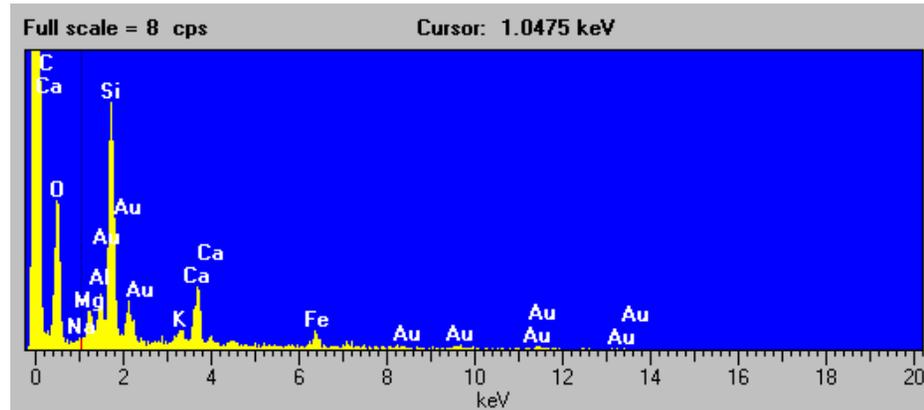
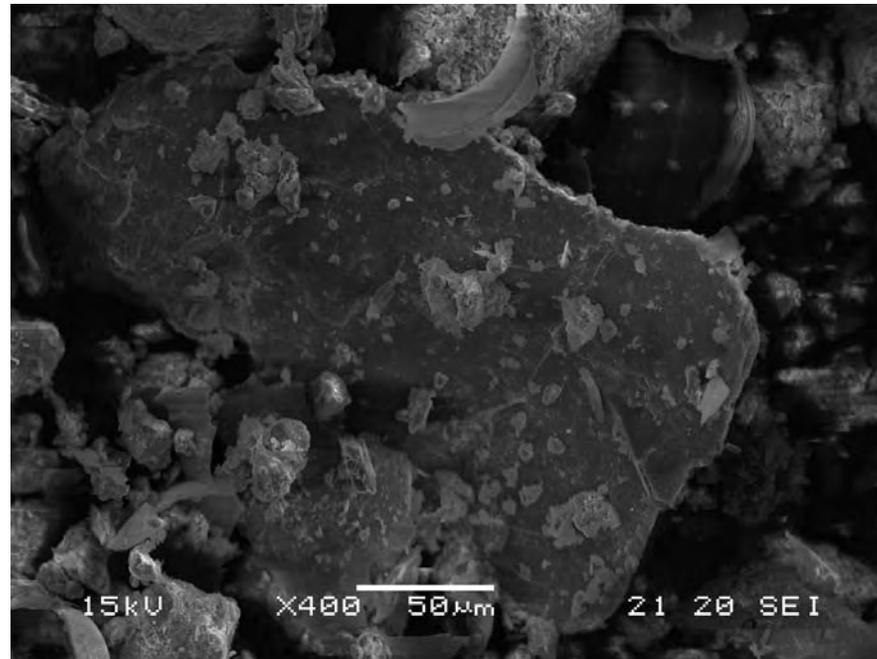


Figure C-14. Scanning electron micrograph, EDX spectrum, and elemental composition of the second particle of the Las Vegas Wash 0.8 mile sample (magnification 400x).

SEMQuant results. Listed at 12:05:52 PM on 2/27/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic	Atomic
Type	%	%		
O K	ED	46.09	61.24	
Na K	ED	-0.48*	-0.44*	
Mg K	ED	2.16	1.88	
Al K	ED	10.87	8.56	
Si K	ED	32.26	24.42	
K K	ED	4.41	2.40	
Ca K	ED	1.03	0.55	
Fe K	ED	3.66	1.39	
Total		100.00	100.00	

* = <2 Sigma

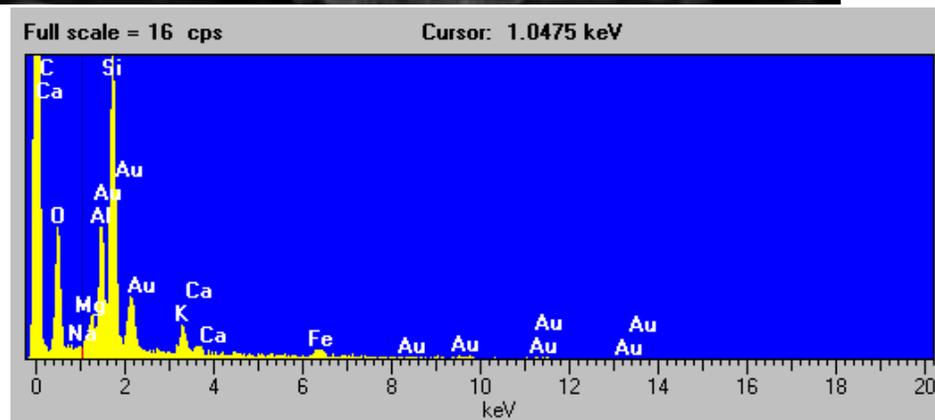
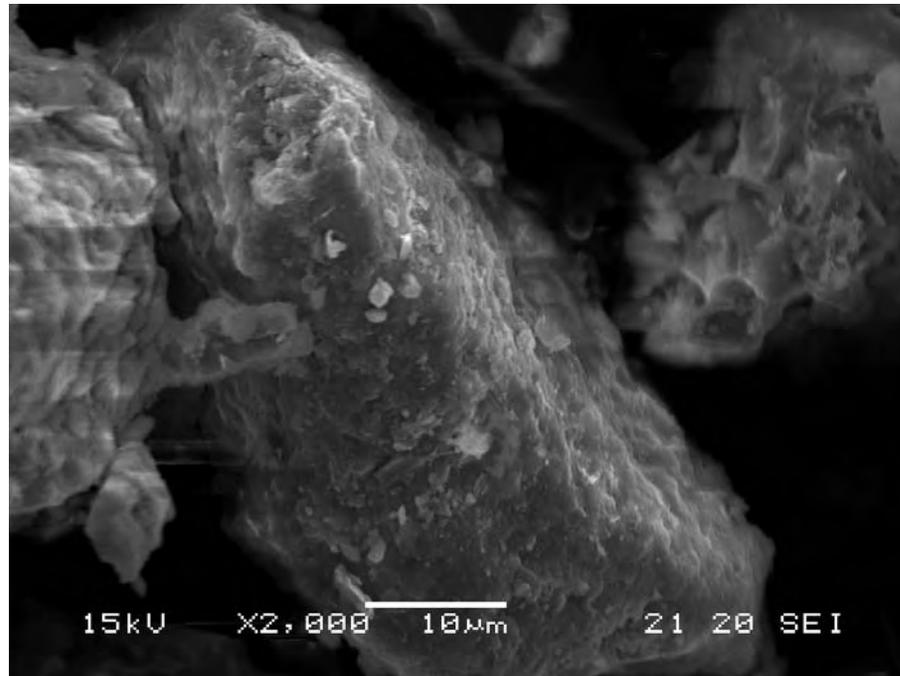


Figure C-15. Scanning electron micrograph, EDX spectrum, and elemental composition of the third particle of the Las Vegas Wash 0.8 mile sample (magnification 2,000x).

SEMQuant results. Listed at 12:10:10 PM on 2/27/07

Operator: TracyB

Client: Tracy Boettcher

Job: Job number 51

Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (3 iterations).

Analysed all elements and normalised results.

3 peaks possibly omitted: 0.02, 2.14, 9.64 keV

Standards :

O K Quartz 01/12/93

Na K Albite 02/12/93

Mg K MgO 01/12/93

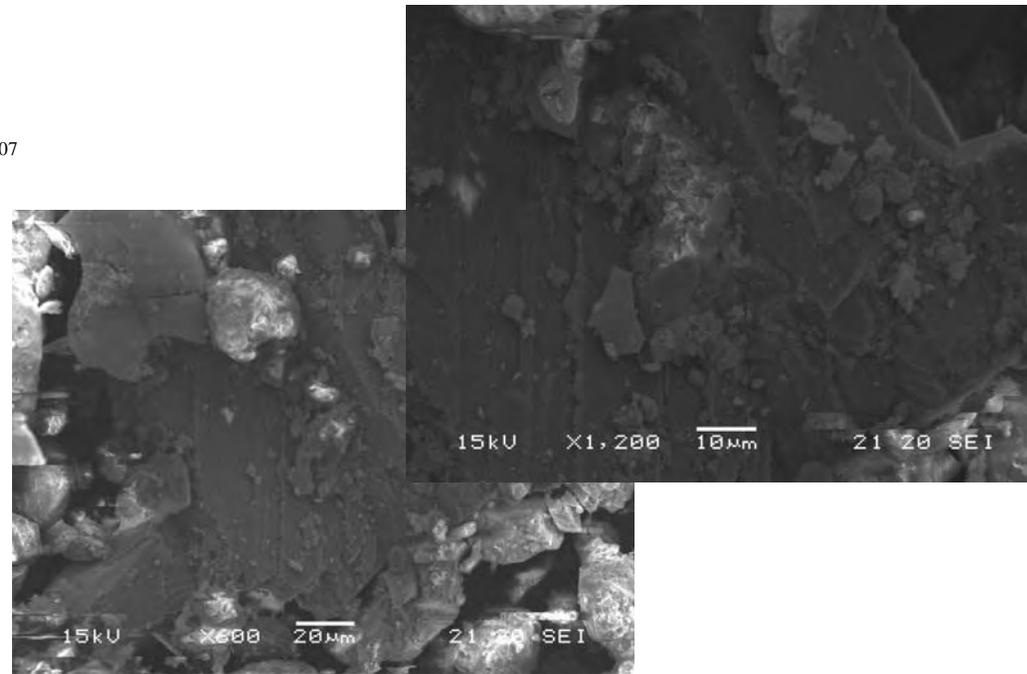
Al K Al₂O₃ 23/11/93

Si K Quartz 01/12/93

K K MAD-10 02/12/93

Ca K Wollas 23/11/93

Fe K Fe 01/12/93



Elmt	Spect.	Element	Atomic Type	%	%
O	K	ED		43.69	65.42
Na	K	ED		-0.11*	-0.12*
Mg	K	ED		0.57	0.57
Al	K	ED		0.55*	0.49*
Si	K	ED		3.29	2.80
K	K	ED		1.58	0.97
Ca	K	ED		48.81	29.17
Fe	K	ED		1.61*	0.69*
Total				100.00	100.00

* = <2 Sigma

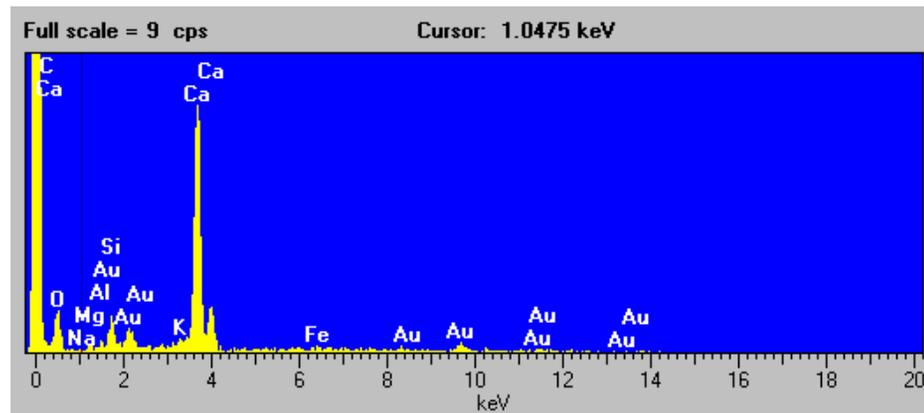


Figure C-16. Scanning electron micrograph, EDX spectrum, and elemental composition of the fourth particle of the Las Vegas Wash 0.8 mile sample (magnifications 600x and 1,200).

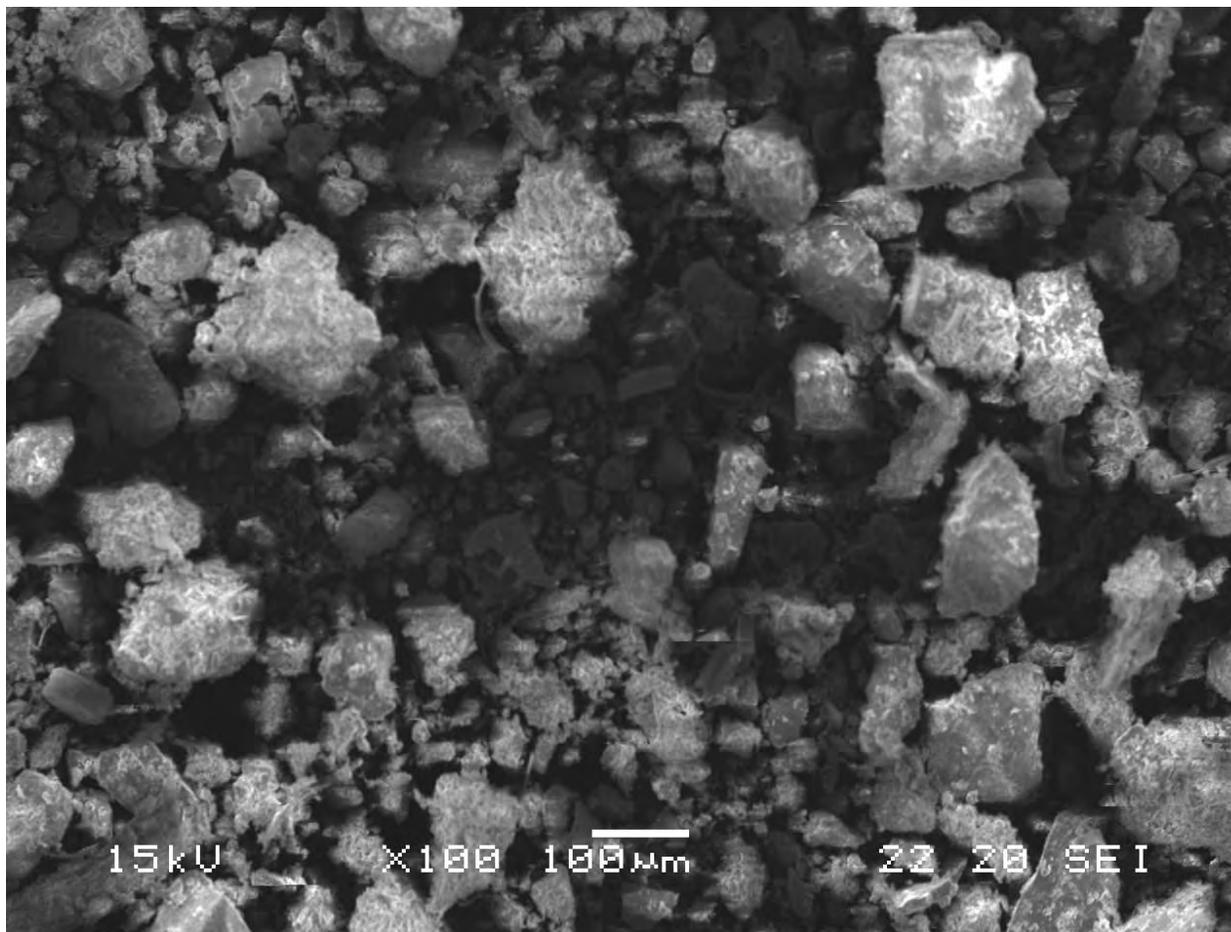


Figure C-17. General view of the Las Vegas Wash 6.05 mile sample. Magnification 100x.

SEMQuant results. Listed at 10:58:54 AM on 3/1/07

Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic	Atomic
	Type	%	%	
O	K	ED	55.79	70.21
Na	K	ED	-0.52*	-0.45*
Mg	K	ED	1.73	1.43
Al	K	ED	1.52	1.13
Si	K	ED	33.35	23.90
K	K	ED	0.98	0.50
Ca	K	ED	4.90	2.46
Fe	K	ED	2.25	0.81
Total			100.00	100.00

* = <2 Sigma

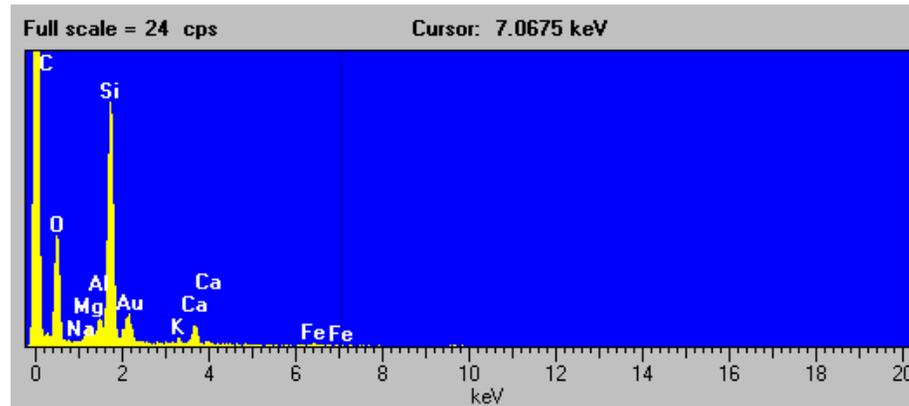
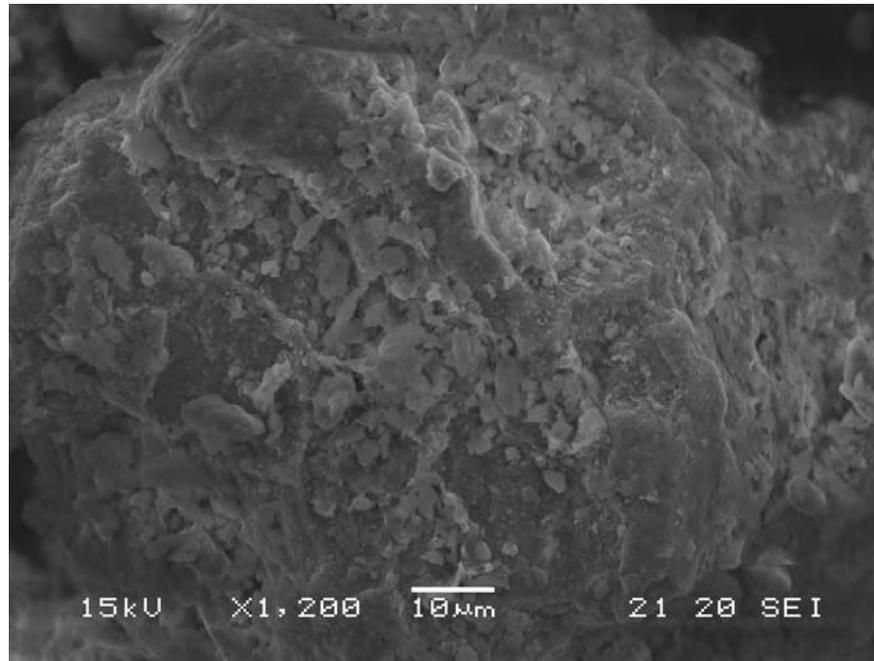


Figure C-18. Scanning electron micrograph, EDX spectrum, and elemental composition of the first particle of the Las Vegas Wash 6.05 mile sample (magnification 1,200x).

SEMQuant results. Listed at 11:06:52 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

4 peaks possibly omitted: 0.02, 2.14, 4.50,
 9.64 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic Type	%	%
O	K	ED		56.16	71.76
Na	K	ED		-0.51*	-0.45*
Mg	K	ED		5.32	4.48
Al	K	ED		6.79	5.15
Si	K	ED		18.15	13.21
K	K	ED		3.37	1.76
Ca	K	ED		1.16	0.59
Fe	K	ED		9.55	3.50
Total				100.00	100.00

* = <2 Sigma

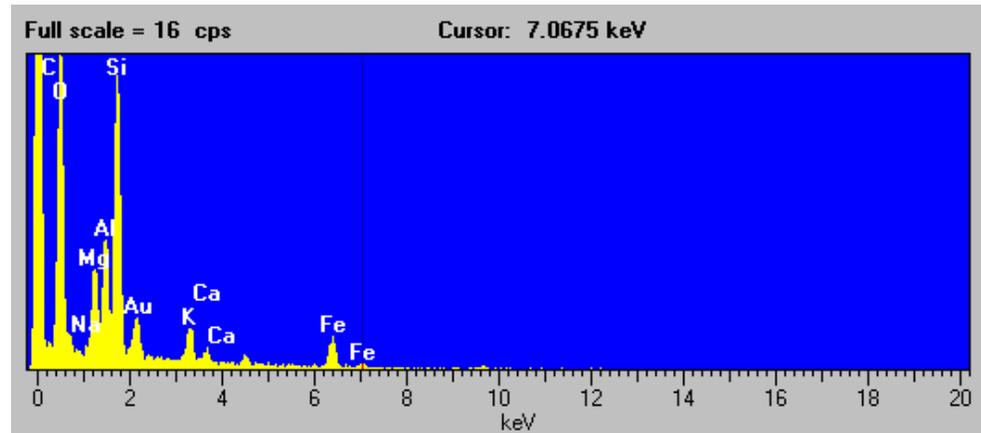
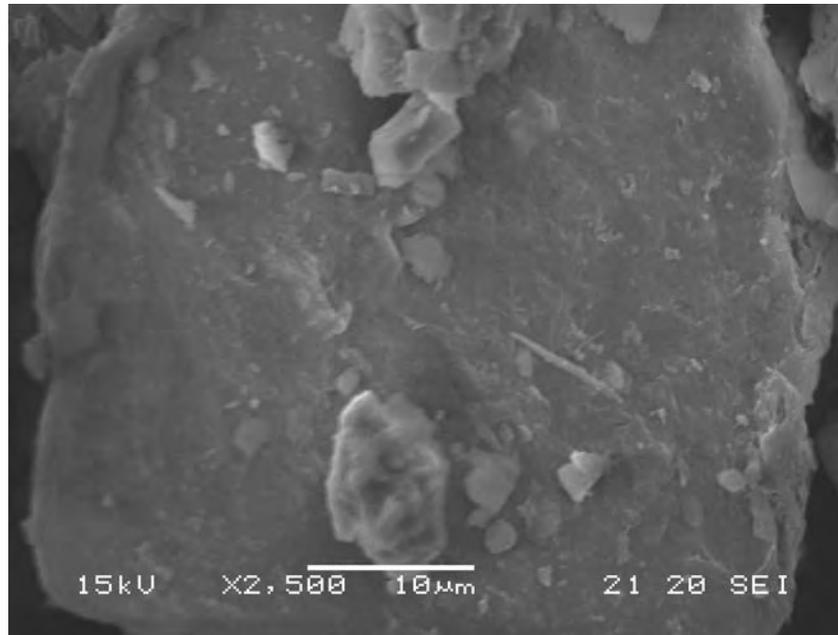


Figure C-19. Scanning electron micrograph, EDX spectrum, and elemental composition of the second particle of the Las Vegas Wash 6.05 mile sample (magnification 2,500x).

SEMQuant results. Listed at 11:13:12 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

1 peak possibly omitted: 0.02 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	41.56	57.61
Na K	ED	1.37	1.32
Mg K	ED	0.78*	0.72*
Al K	ED	9.62	7.91
Si K	ED	29.77	23.51
K K	ED	2.25	1.27
Ca K	ED	11.89	6.58
Fe K	ED	2.75	1.09
Total		100.00	100.00

* = <2 Sigma

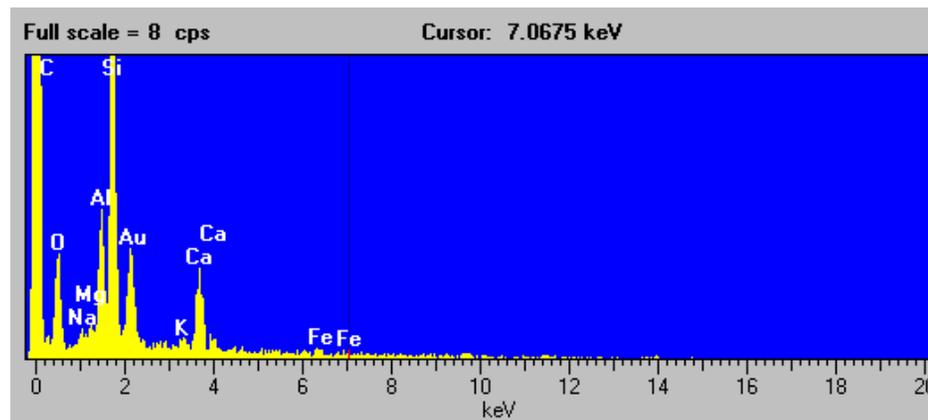
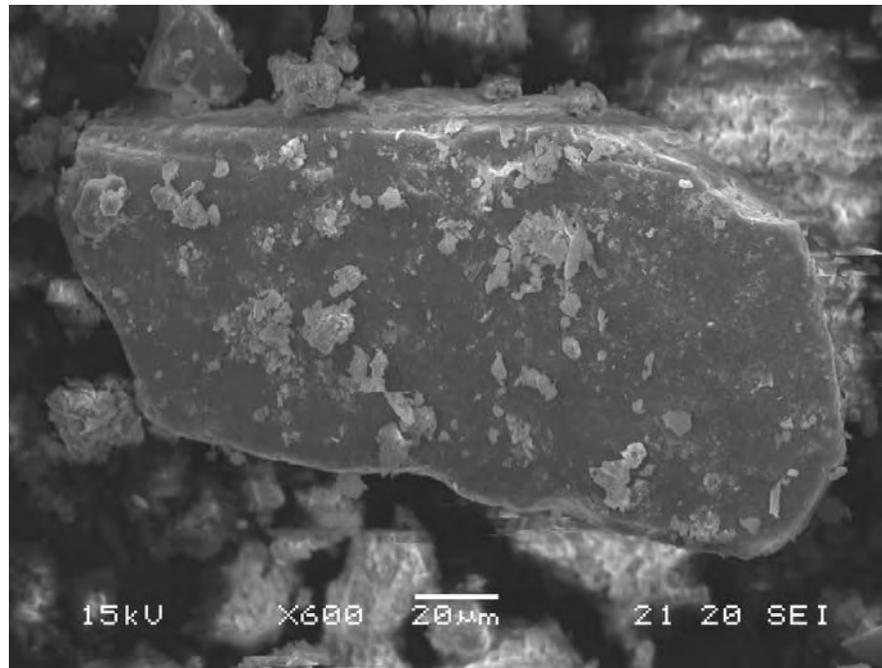


Figure C-20. Scanning electron micrograph, EDX spectrum, and elemental composition of the third particle of the Las Vegas Wash 6.05 mile sample (magnification 600x).

SEMQuant results. Listed at 11:22:20 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic	Atomic
Type	%	%		
O K	ED	48.01	65.42	
Na K	ED	-0.90*	-0.85*	
Mg K	ED	2.46	2.21	
Al K	ED	4.68	3.78	
Si K	ED	22.97	17.83	
K K	ED	6.90	3.85	
Ca K	ED	10.24	5.57	
Fe K	ED	5.63	2.20	
Total		100.00	100.00	

* = <2 Sigma

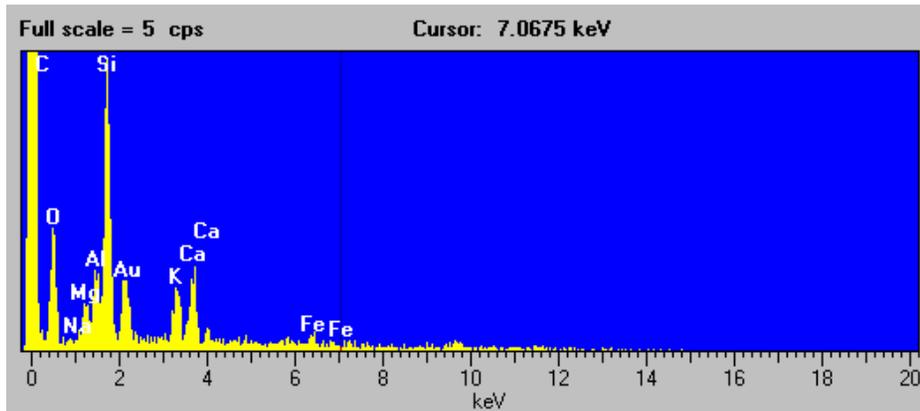
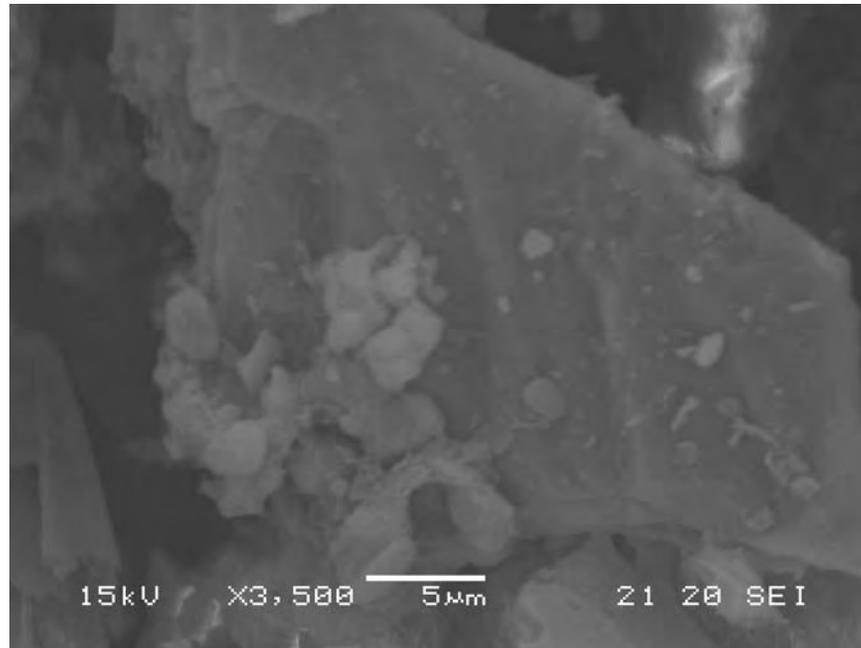


Figure C-21. Scanning electron micrograph, EDX spectrum, and elemental composition of the fourth particle of the Las Vegas Wash 6.05 mile sample (magnification 3,500x).

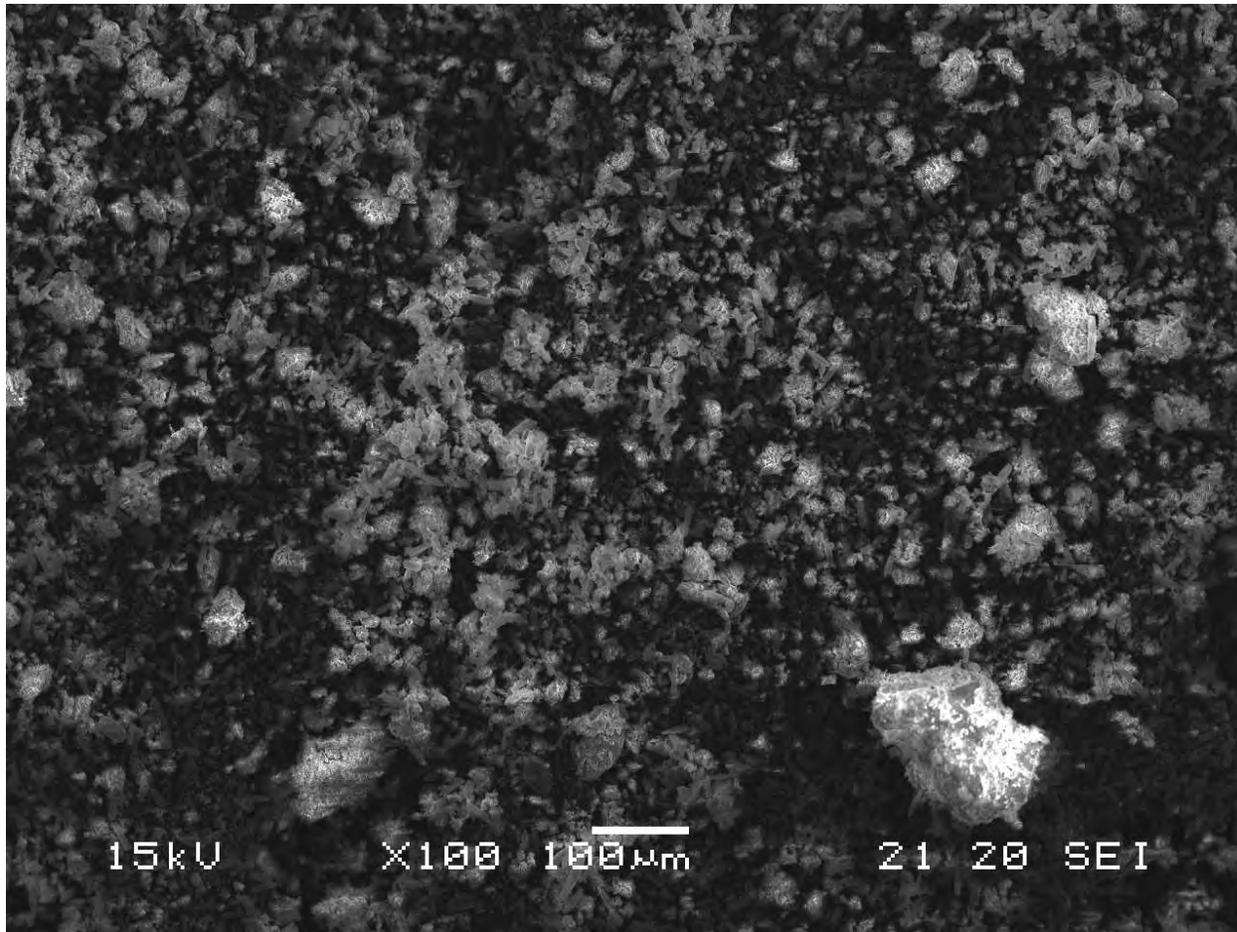


Figure C-22. General view of the Las Vegas Wash 10.75 mile sample. Magnification 100x.

SEMQuant results. Listed at 11:33:05 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (4 iterations).
 Analysed all elements and normalised results.

1 peak possibly omitted: 0.02 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic	Atomic
Type	%	%		
O K	ED	61.50	75.88	
Na K	ED	-0.17*	-0.15*	
Mg K	ED	2.39	1.94	
Al K	ED	3.63	2.66	
Si K	ED	17.39	12.23	
K K	ED	3.02	1.53	
Ca K	ED	11.46	5.64	
Fe K	ED	0.78*	0.27*	
Total		100.00	100.00	

* = <2 Sigma

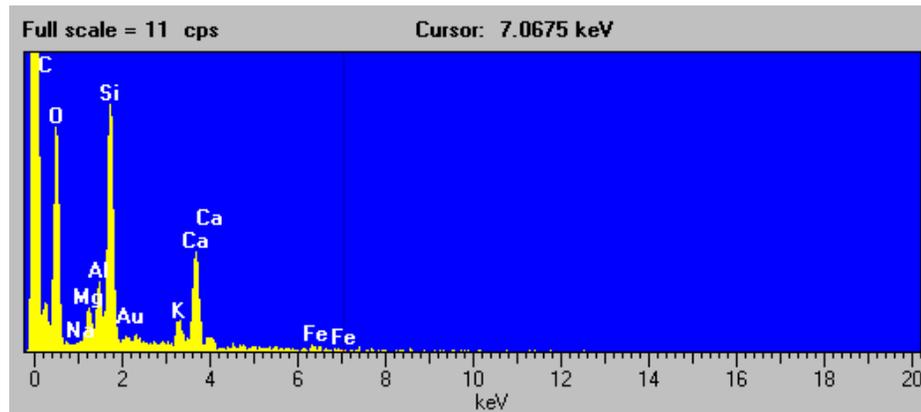
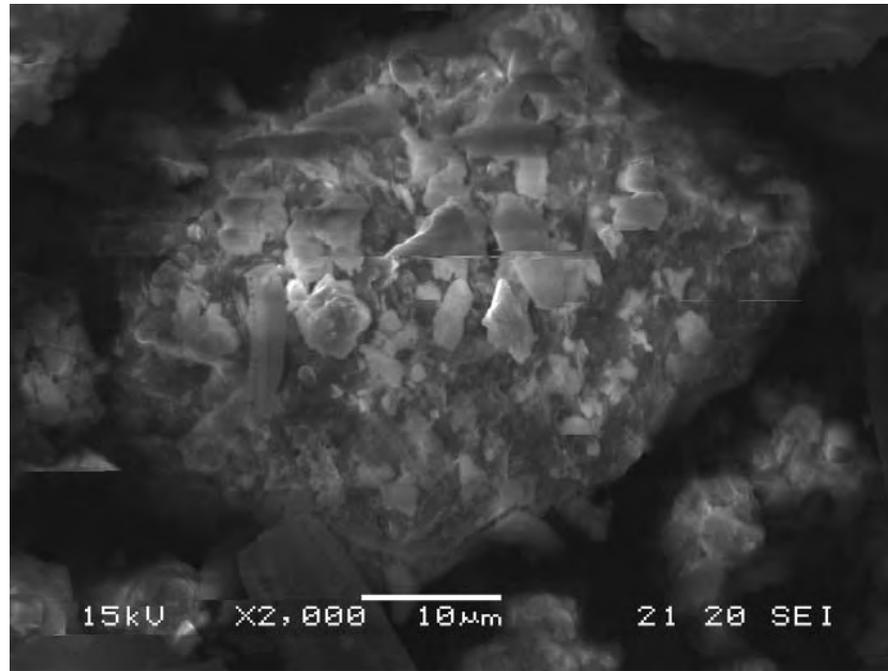


Figure C-23. Scanning electron micrograph, EDX spectrum, and elemental composition of the first particle of the Las Vegas Wash 10.75 mile sample (magnification 2,000x).

SEMQuant results. Listed at 11:39:35 AM on 3/1/07

Operator: TracyB

Client: Tracy Boettcher

Job: Job number 51

Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (4 iterations).

Analysed all elements and normalised results.

1 peak possibly omitted: 0.02 keV

Standards :

O K Quartz 01/12/93

Na K Albite 02/12/93

Mg K MgO 01/12/93

Al K Al₂O₃ 23/11/93

Si K Quartz 01/12/93

K K MAD-10 02/12/93

Ca K Wollas 23/11/93

Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic	Atomic
	Type	%	%	
O	K	ED	61.43	74.70
Na	K	ED	-0.47*	-0.40*
Mg	K	ED	1.38	1.11
Al	K	ED	1.48	1.07
Si	K	ED	29.00	20.09
K	K	ED	0.82	0.41
Ca	K	ED	5.96	2.89
Fe	K	ED	0.40*	0.14*
Total			100.00	100.00

* = <2 Sigma

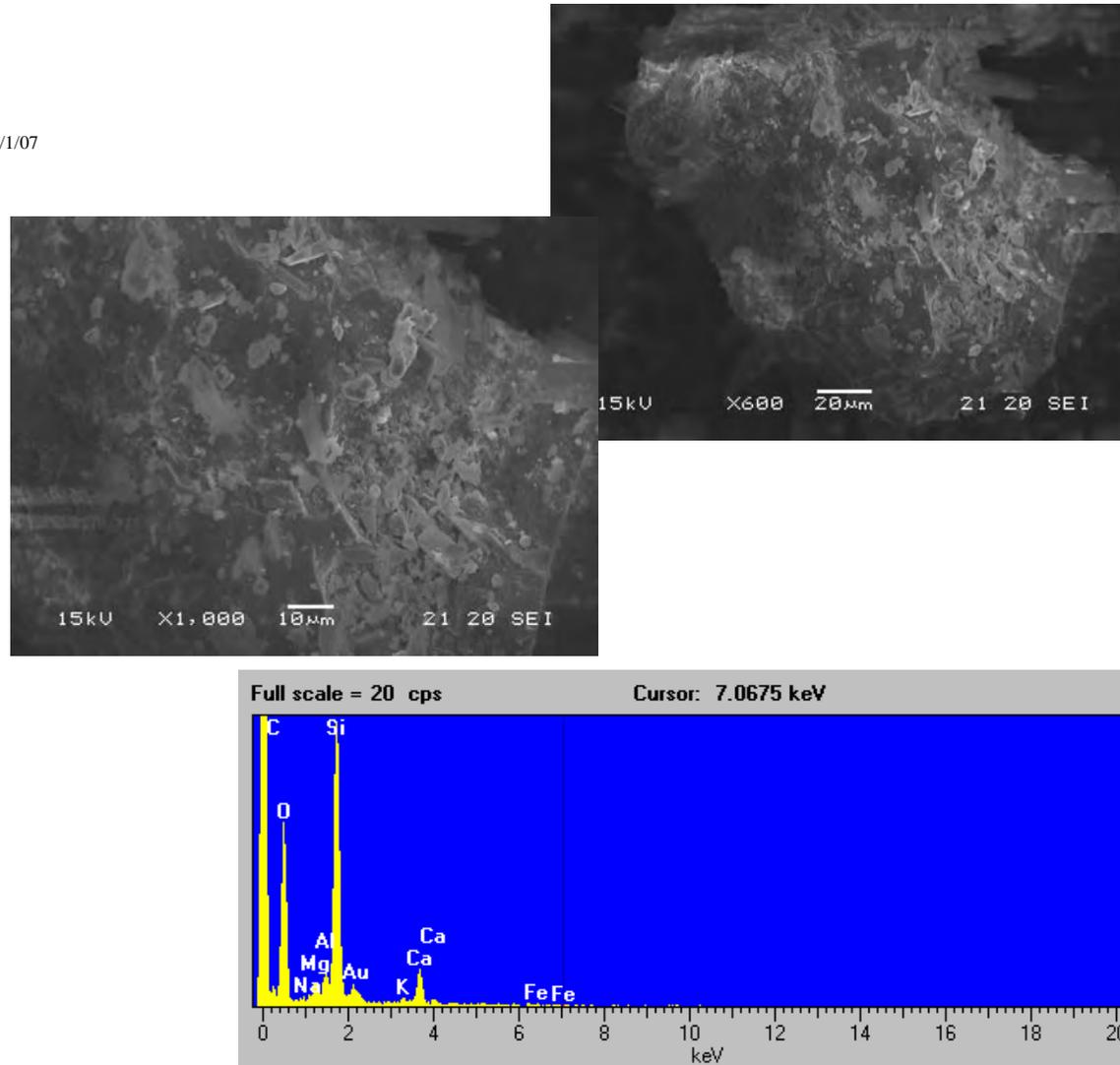


Figure C-24. Scanning electron micrograph, EDX spectrum, and elemental composition of the second particle of the Las Vegas Wash 10.75 mile sample (magnifications 600x and 1,000x).

SEMQuant results. Listed at 11:50:38 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (4 iterations).
 Analysed all elements and normalised results.

1 peak possibly omitted: 0.02 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al2O3 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	59.09	72.22
Na K	ED	2.46	2.09
Mg K	ED	1.78	1.43
Al K	ED	5.94	4.30
Si K	ED	24.51	17.06
K K	ED	1.36	0.68
Ca K	ED	3.68	1.80
Fe K	ED	1.19	0.42
Total		100.00	100.00

* = <2 Sigma

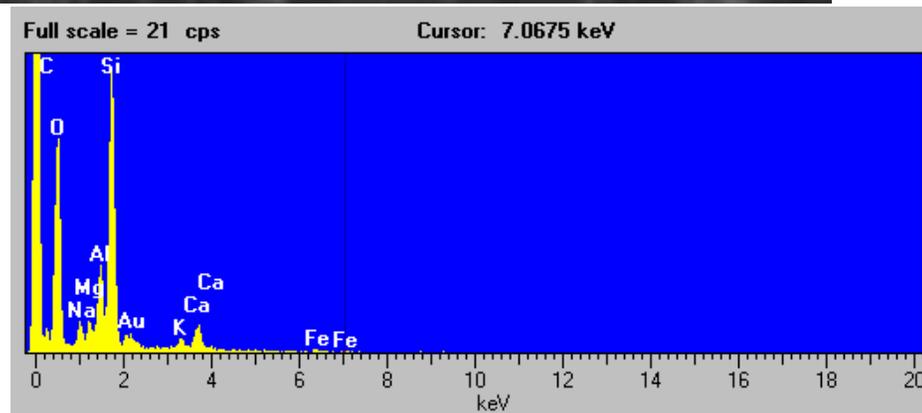
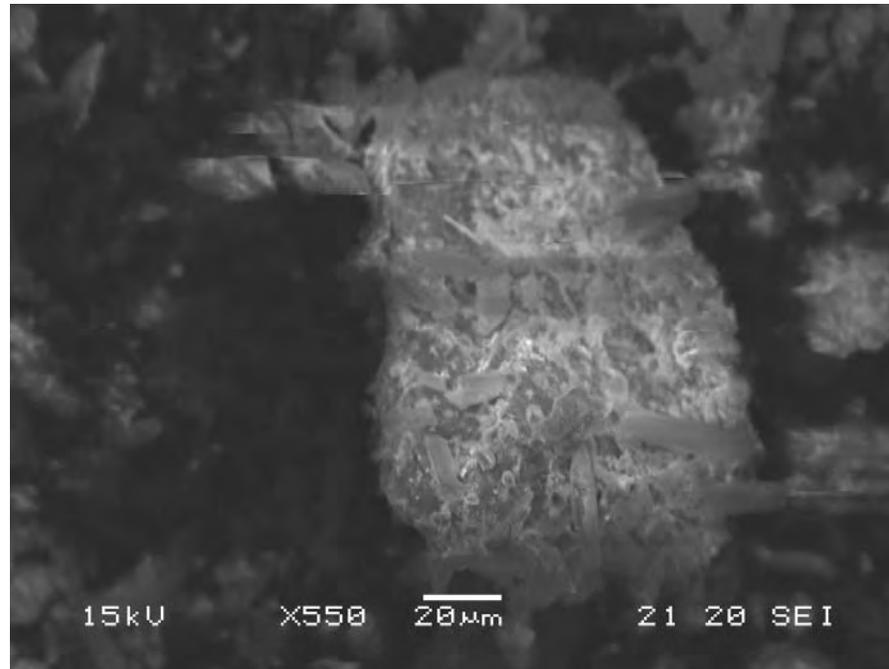


Figure C-25. Scanning electron micrograph, EDX spectrum, and elemental composition of the third particle of the Las Vegas Wash 10.75 mile sample (magnification 550x).

SEMQuant results. Listed at 12:01:50 PM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (5 iterations).
 Analysed all elements and normalised results.

1 peak possibly omitted: 0.02 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	62.80	77.66
Na K	ED	0.31*	0.27*
Mg K	ED	2.22	1.80
Al K	ED	1.63	1.19
Si K	ED	13.04	9.18
K K	ED	1.28	0.65
Ca K	ED	18.67	9.22
Fe K	ED	0.06*	0.02*
Total		100.00	100.00

* = <2 Sigma

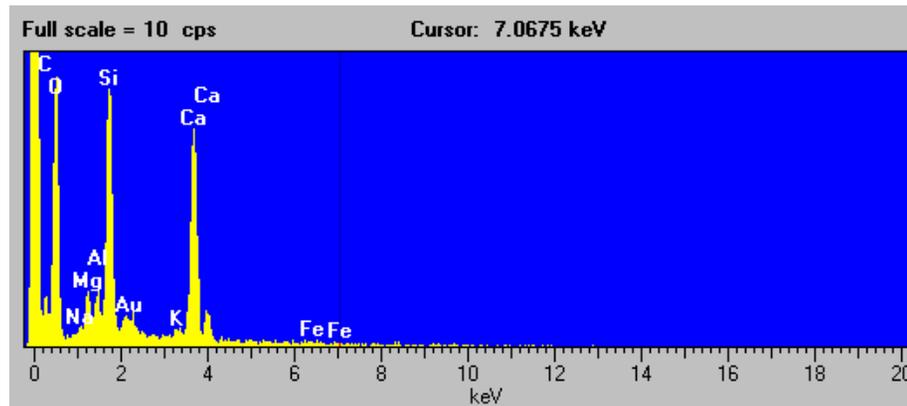
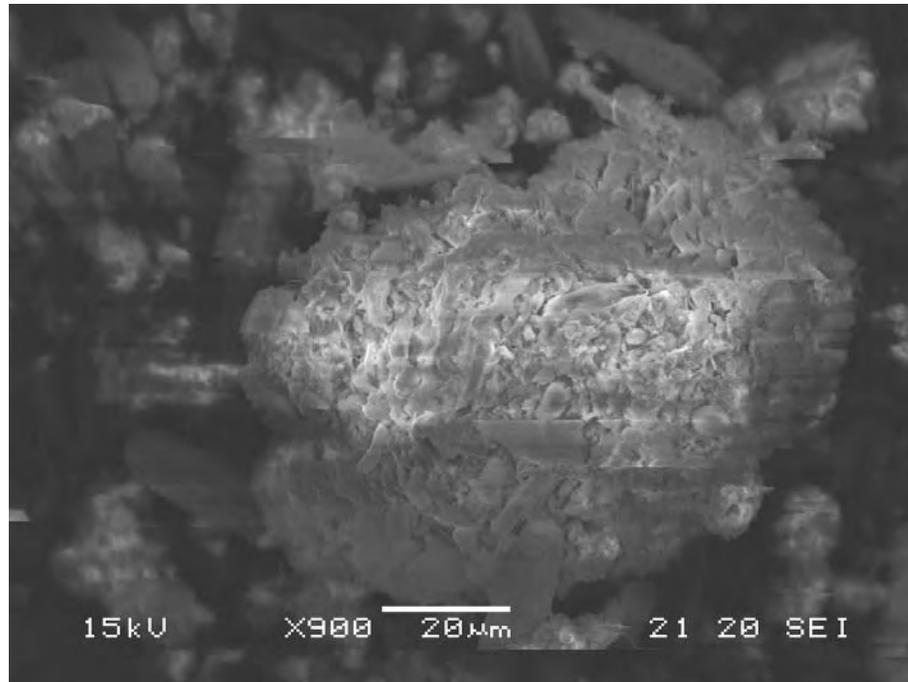


Figure C-26. Scanning electron micrograph, EDX spectrum, and elemental composition of the fourth particle of the Las Vegas Wash 10.75 mile sample (magnification 900x).

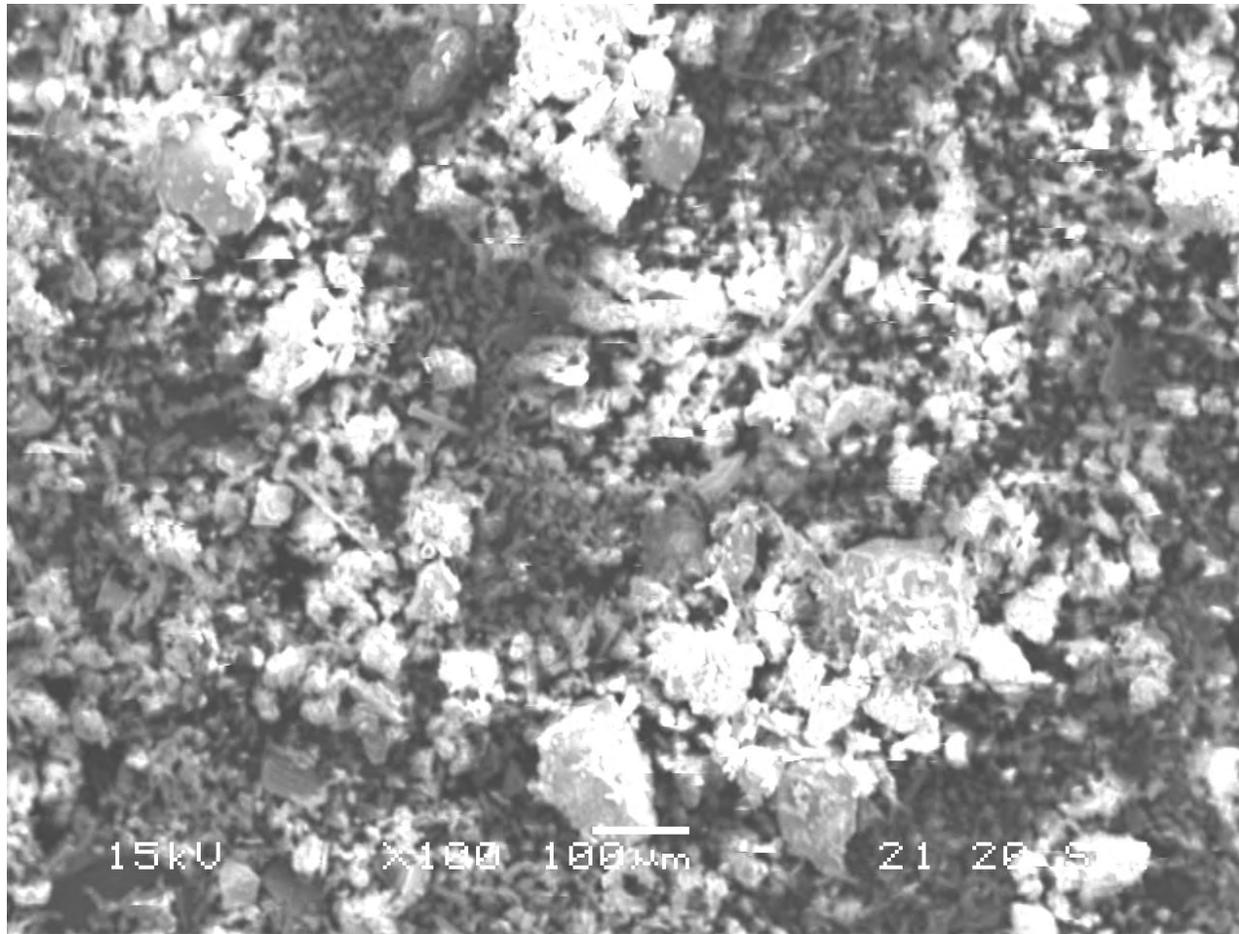


Figure C-27. General view of the Duck Creek sample. Magnification 100x.

SEMQuant results. Listed at 9:35:05 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

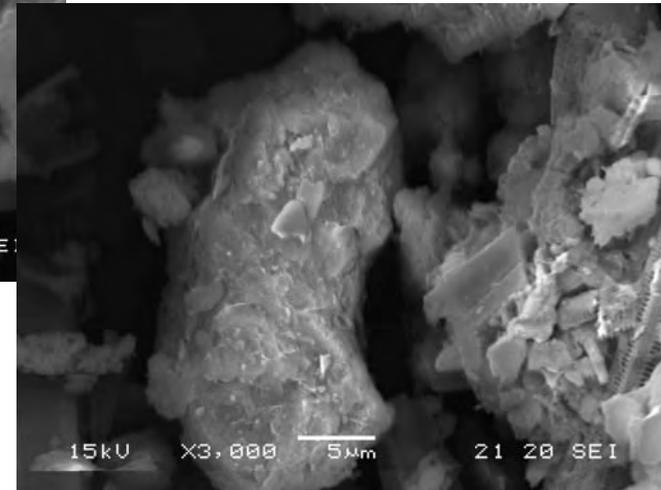
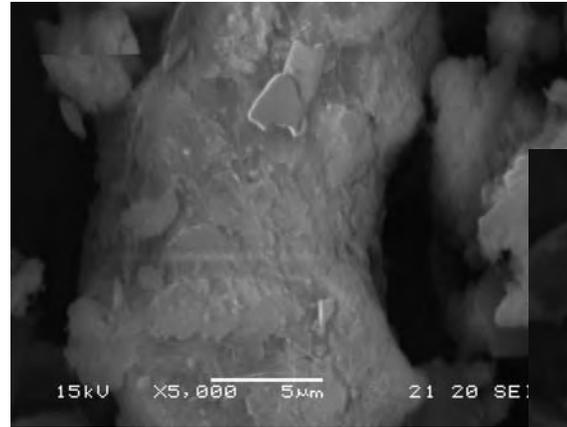
System resolution = 114 eV

Quantitative method: ZAF (4 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93



Elmt	Spect.	Element	Atomic Type	%	%
O	K	ED		58.49	75.19
Na	K	ED		0.51*	0.46*
Mg	K	ED		0.81	0.69
Al	K	ED		2.37	1.81
Si	K	ED		11.70	8.57
K	K	ED		1.89	0.99
Ca	K	ED		23.28	11.95
Fe	K	ED		0.94*	0.35*
Total				100.00	100.00

* = <2 Sigma

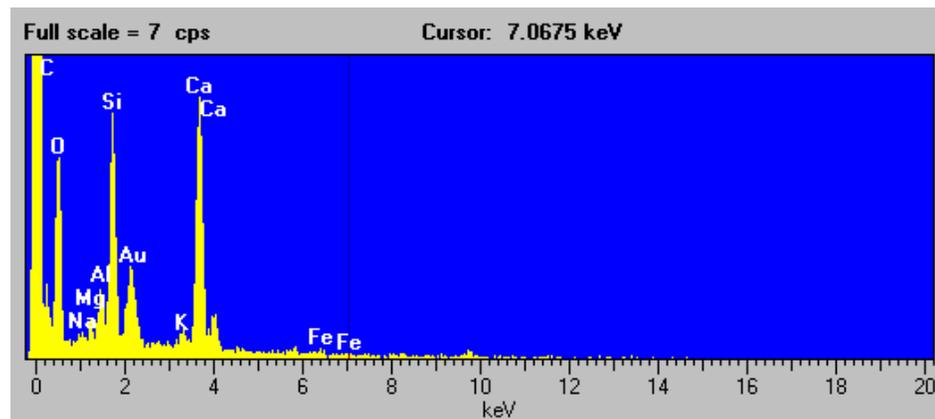


Figure C-28. Scanning electron micrograph, EDX spectrum, and elemental composition of the first particle of the Duck Creek sample (magnifications 3,000x and 5,000x).

SEMQuant results. Listed at 9:45:57 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

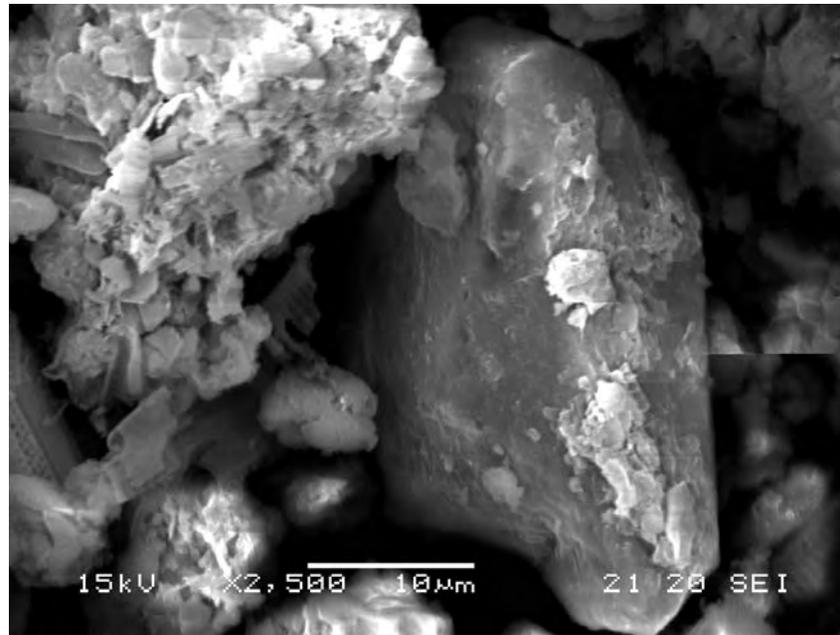
System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93



Elmt	Spect.	Element	Atomic Type	%	Atomic %
O	K	ED		41.22	59.36
Na	K	ED		0.19*	0.19*
Mg	K	ED		2.31	2.19
Al	K	ED		4.61	3.94
Si	K	ED		24.24	19.88
K	K	ED		4.10	2.41
Ca	K	ED		14.77	8.49
Fe	K	ED		8.57	3.54
Total				100.00	100.00

* = <2 Sigma

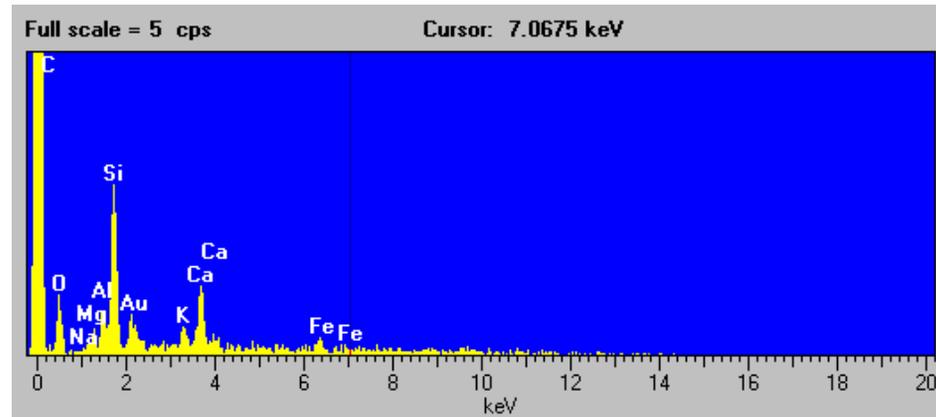


Figure C-29. Scanning electron micrograph, EDX spectrum, and elemental composition of the second particle of the Duck Creek sample (magnification 2,500x).

SEMQuant results. Listed at 9:53:19 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (4 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al2O3 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic	Atomic
	Type	%	%	
O	K	ED	54.54	69.41
Na	K	ED	0.72	0.63
Mg	K	ED	0.37*	0.31*
Al	K	ED	4.13	3.11
Si	K	ED	28.49	20.65
K	K	ED	3.78	1.97
Ca	K	ED	7.07	3.59
Fe	K	ED	0.91*	0.33*
Total			100.00	100.00

* = <2 Sigma

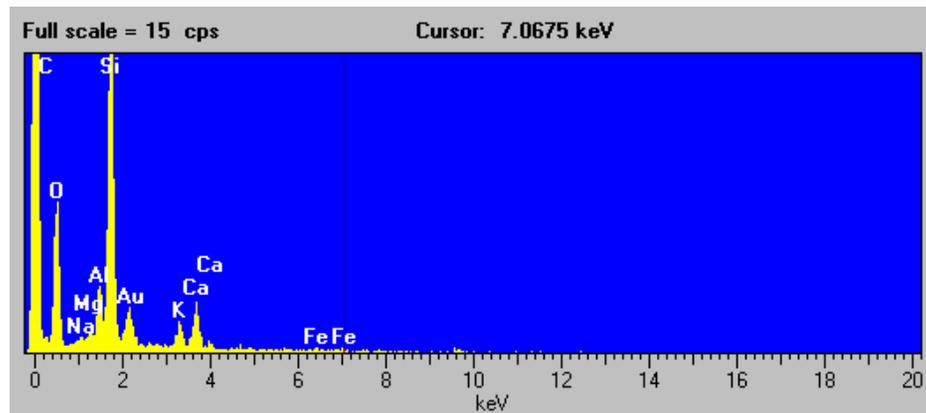
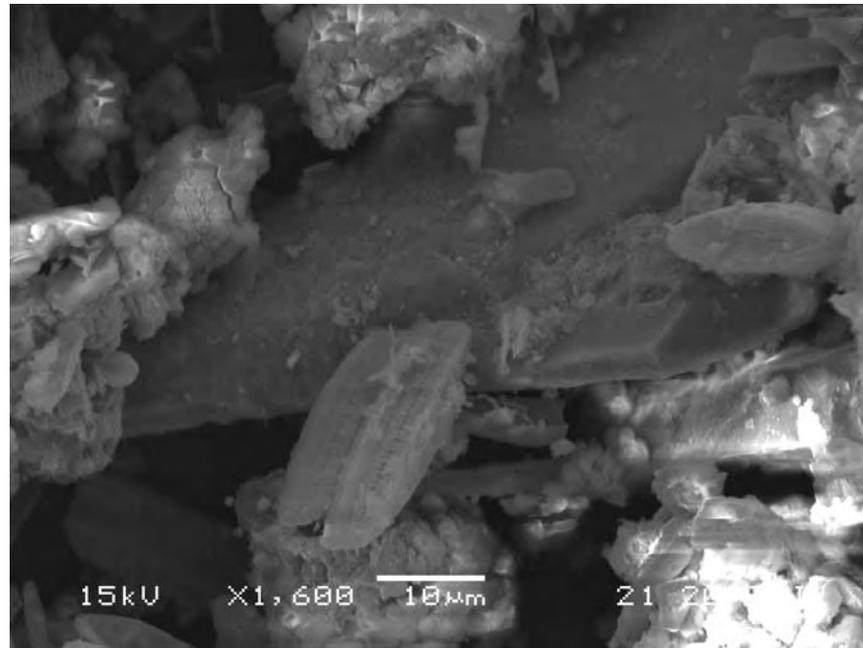


Figure C-30. Scanning electron micrograph, EDX spectrum, and elemental composition of the third particle of the Duck Creek sample (magnification 1,600x).

SEMQuant results. Listed at 10:04:53 AM on 3/1/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	53.21	68.53
Na K	ED	0.45*	0.41*
Mg K	ED	1.38	1.17
Al K	ED	1.14	0.87
Si K	ED	31.39	23.03
K K	ED	1.06	0.56
Ca K	ED	8.51	4.37
Fe K	ED	2.86	1.06
Total		100.00	100.00

* = <2 Sigma

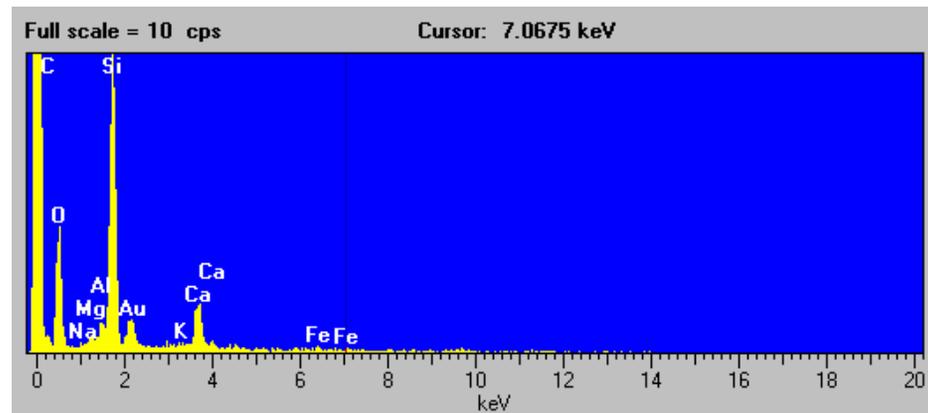
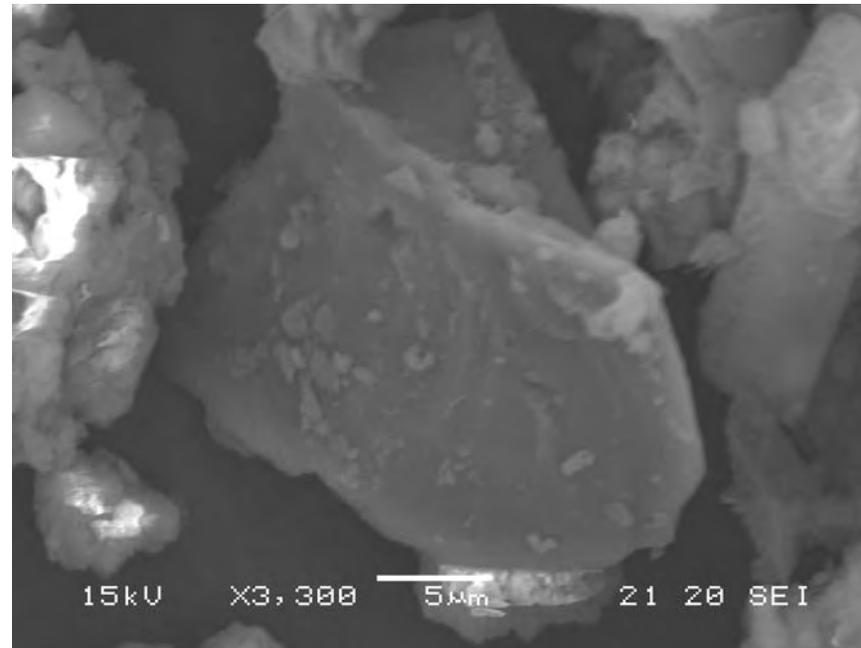


Figure C-31. Scanning electron micrograph, EDX spectrum, and elemental composition of the fourth particle of the Duck Creek sample (magnification 3,300x).

SEMQuant results. Listed at 10:09:27 AM on 3/1/07

Operator: TracyB

Client: Tracy Boettcher

Job: Job number 51

Spectrum label:

System resolution = 114 eV

Quantitative method: ZAF (3 iterations).

Analysed all elements and normalised results.

1 peak possibly omitted: 0.02 keV

Standards :

O K Quartz 01/12/93

Na K Albite 02/12/93

Mg K MgO 01/12/93

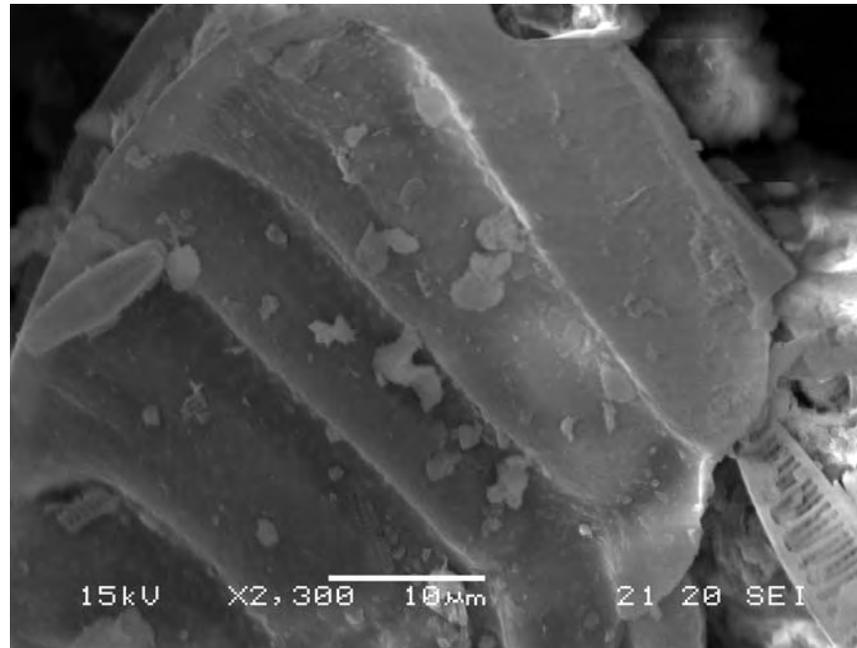
Al K Al₂O₃ 23/11/93

Si K Quartz 01/12/93

K K MAD-10 02/12/93

Ca K Wollas 23/11/93

Fe K Fe 01/12/93



Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	47.13	62.02
Na K	ED	-0.02*	-0.02*
Mg K	ED	0.36*	0.31*
Al K	ED	0.25*	0.20*
Si K	ED	44.53	33.38
K K	ED	0.97*	0.52*
Ca K	ED	6.96	3.66
Fe K	ED	-0.19*	-0.07*
Total		100.00	100.00

* = <2 Sigma

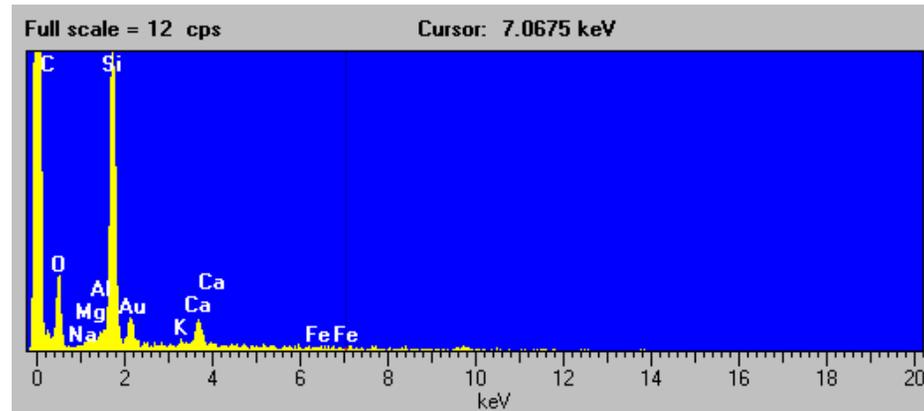


Figure C-32. Scanning electron micrograph, EDX spectrum, and elemental composition of the fifth particle of the Duck Creek sample (magnification 2,300x).

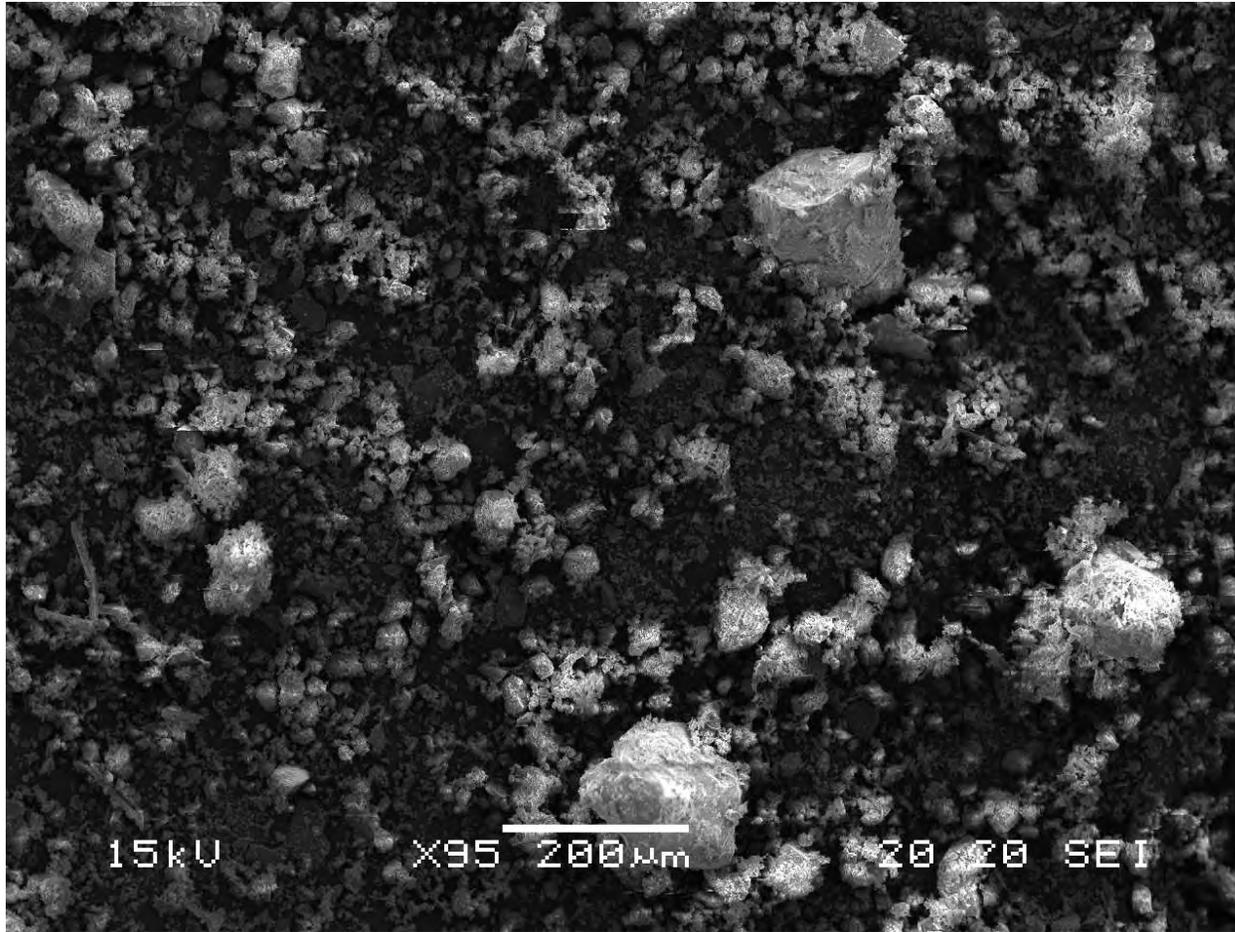


Figure C-33. General view of the Nature Preserve sample. Magnification 95x.

SEMQuant results. Listed at 11:25:17 AM on 2/27/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (4 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	57.90	74.24
Na K	ED	-0.41*	-0.36*
Mg K	ED	3.03	2.55
Al K	ED	3.19	2.42
Si K	ED	13.60	9.93
K K	ED	1.86	0.98
Ca K	ED	17.88	9.15
Fe K	ED	2.96	1.09
Total		100.00	100.00

* = <2 Sigma

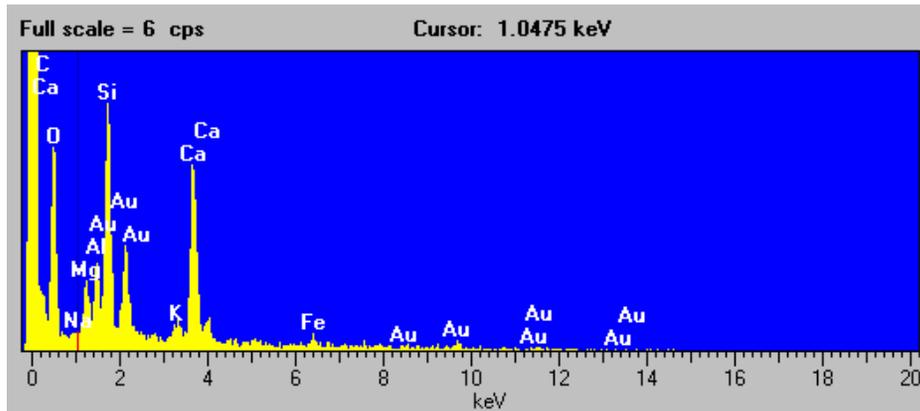
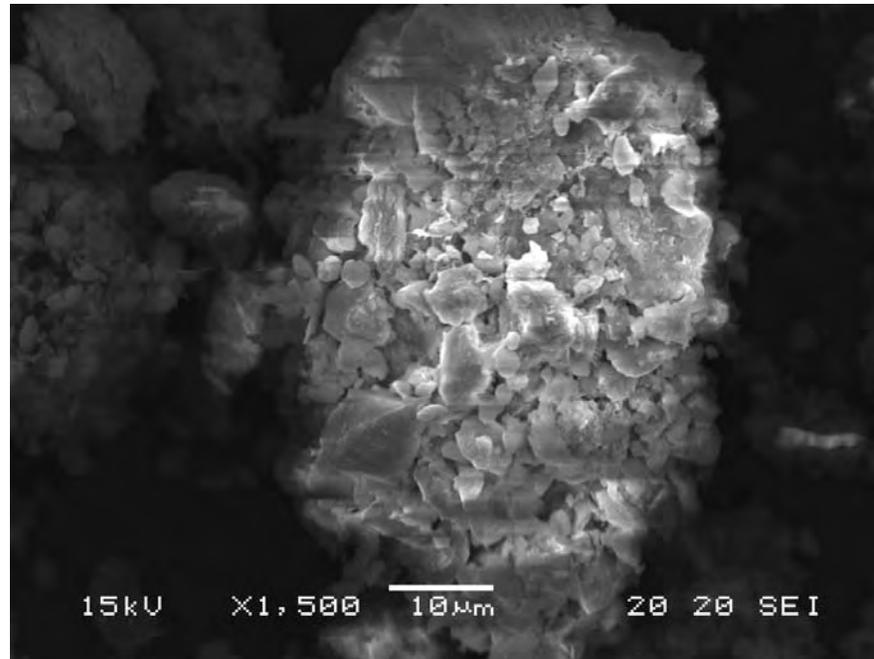


Figure C-34. Scanning electron micrograph, EDX spectrum, and elemental composition of the first particle of the Nature Preserve sample (magnification 1,500x).

SEMQuant results. Listed at 11:31:10 AM on 2/27/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (2 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	23.10	38.84
Na K	ED	-0.22*	-0.26*
Mg K	ED	0.62*	0.68*
Al K	ED	7.51	7.49
Si K	ED	29.69	28.44
K K	ED	21.13	14.54
Ca K	ED	7.92	5.32
Fe K	ED	10.26	4.95
Total		100.00	100.00

* = <2 Sigma

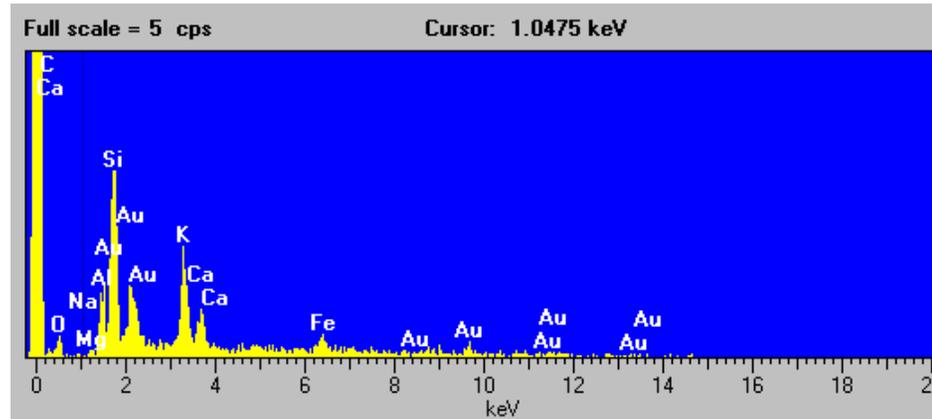
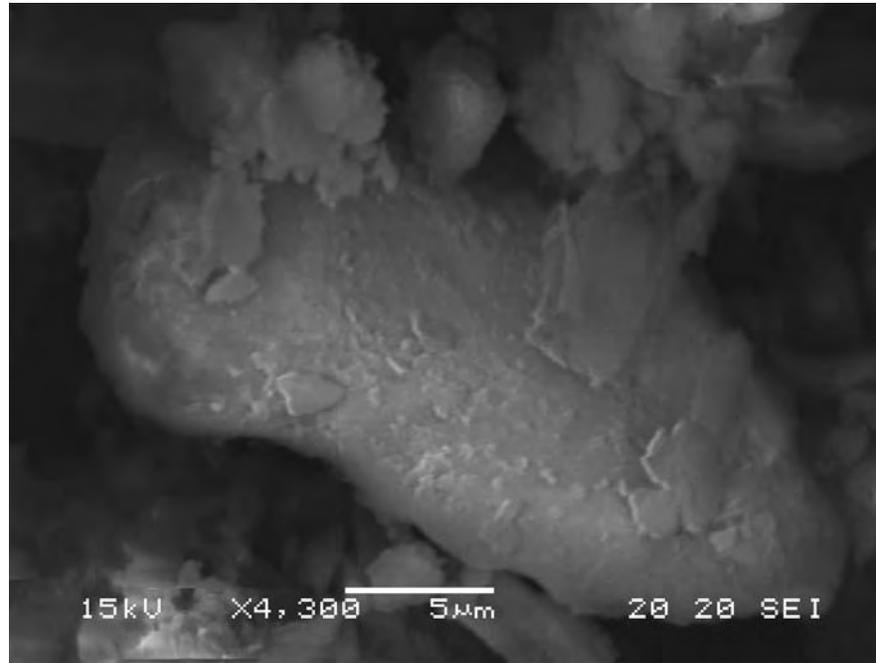


Figure C-35. Scanning electron micrograph, EDX spectrum, and elemental composition of the second particle of the Nature Preserve sample (magnification 4,300x).

SEMQuant results. Listed at 11:39:31 AM on 2/27/07
 Operator: TracyB
 Client: Tracy Boettcher
 Job: Job number 51
 Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (3 iterations).
 Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93
 Na K Albite 02/12/93
 Mg K MgO 01/12/93
 Al K Al₂O₃ 23/11/93
 Si K Quartz 01/12/93
 K K MAD-10 02/12/93
 Ca K Wollas 23/11/93
 Fe K Fe 01/12/93

Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	57.24	71.96
Na K	ED	-1.01*	-0.89*
Mg K	ED	9.85	8.15
Al K	ED	9.63	7.18
Si K	ED	13.11	9.39
K K	ED	0.33*	0.17*
Ca K	ED	0.87	0.44
Fe K	ED	9.99	3.60
Total		100.00	100.00

* = <2 Sigma

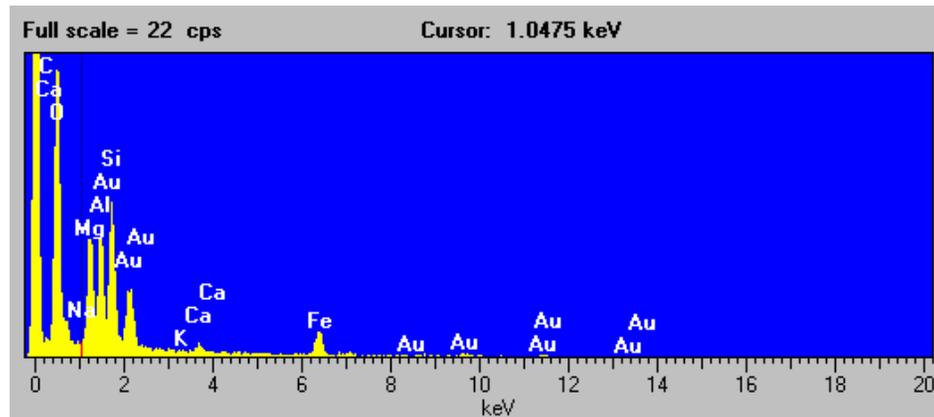
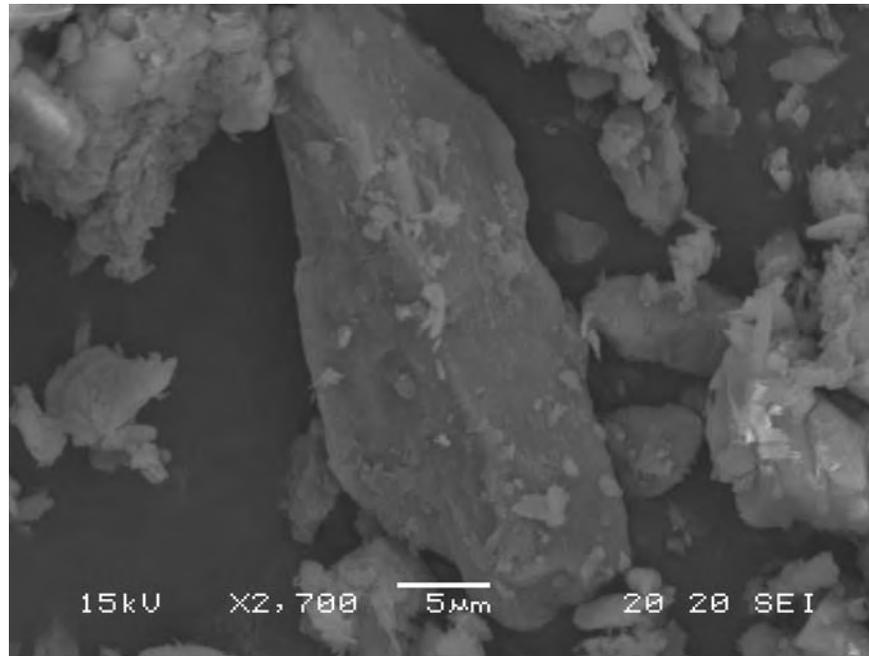


Figure C-36. Scanning electron micrograph, EDX spectrum, and elemental composition of the third particle of the Nature Preserve sample (magnification 2,700x).

SEMQuant results. Listed at 11:43:47 AM on 2/27/07

Operator: TracyB

Client: Tracy Boettcher

Job: Job number 51

Spectrum label:

System resolution = 115 eV

Quantitative method: ZAF (3 iterations).

Analysed all elements and normalised results.

2 peaks possibly omitted: 0.02, 2.14 keV

Standards :

O K Quartz 01/12/93

Na K Albite 02/12/93

Mg K MgO 01/12/93

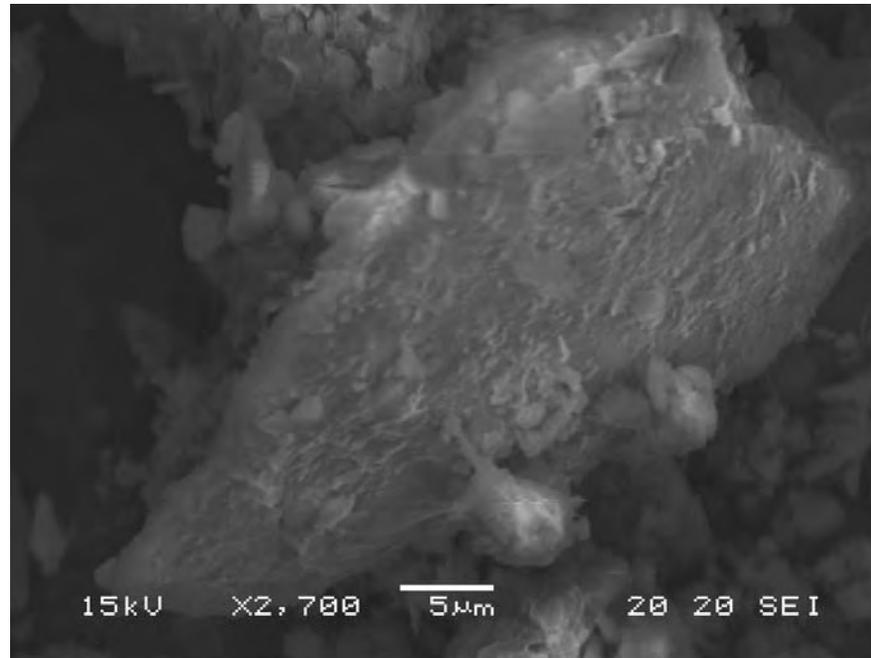
Al K Al₂O₃ 23/11/93

Si K Quartz 01/12/93

K K MAD-10 02/12/93

Ca K Wollas 23/11/93

Fe K Fe 01/12/93



Elmt	Spect.	Element	Atomic
Type	%	%	
O K	ED	52.06	68.32
Na K	ED	-0.04*	-0.03*
Mg K	ED	5.75	4.97
Al K	ED	4.83	3.76
Si K	ED	19.89	14.87
K K	ED	2.58	1.38
Ca K	ED	7.60	3.98
Fe K	ED	7.34	2.76
Total		100.00	100.00

* = <2 Sigma

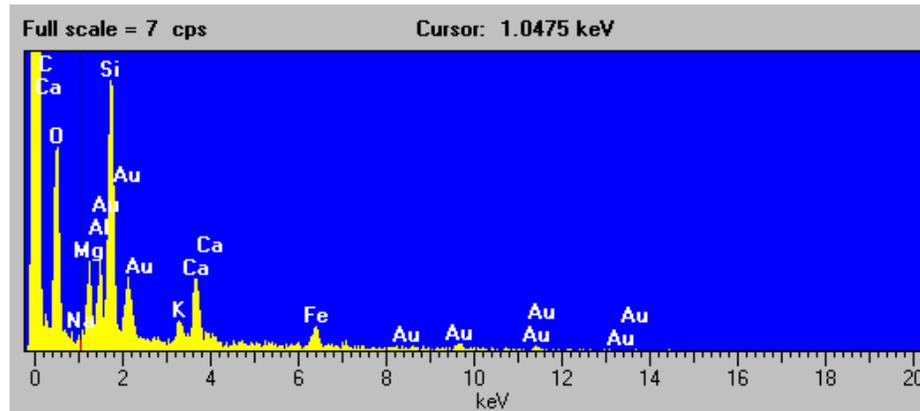


Figure C-37. Scanning electron micrograph, EDX spectrum, and elemental composition of the fourth particle of the Nature Preserve sample (magnification 2,700x).