Characterization of Dust on Solar Devices in Southern Nevada

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CHARACTERIZATION OF DUST ON SOLAR DEVICES IN SOUTHERN NEVADA

By

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Bachelor of Science – Chemistry

University of Nevada, Las Vegas

2012

A thesis submitted in partial fulfillment

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Dust can impact the efficiency of solar energy collection devices, and in some arid environments, dust can reduce solar energy efficiency up to 30%. Reducing the impact of dust is therefore critical in the expansion of solar technology throughout regions where solar energy is utilized. Characterization of suspended and settled particulate matter can assist in developing strategies for dust mitigation. With the characterization of suspended and settled particulate in remote, rural, and urban environments, more informed decisions can be made regarding the selection of coating material on solar panels as well as developing cleaning and maintenance procedures. Particulate matter that deposits on a solar surface can potentially interact with solar radiation, precipitation, or even directly with the surface material itself. These interactions could lead to the formation of coatings that reduce/block radiation and/or degrade the integrity of the surface. When you extrapolate these possibilities to a larger scale preliminary characterization of dust will play a vital role when planning the construction of a solar energy facility.

A variety of sampling techniques were employed to obtain particulate matter for characterization. These included direct collection of particulates from solar surfaces: via vacuum and wipe sample collection on panels, tacky dot adhesive slides and plain slides that were exposed at different intervals, desert vugs that are natural particulate collectors, as well as high volume air sampling for collection of suspended particulates. High volume air sampling was performed using glass fiber filters and 2 micron stainless steel screens.
Direct collection of settled particulates was performed by sampling from solar surfaces, vugs, and by collection on exposed glass surfaces. Collection onto glass surfaces was achieved by setting up a plain microscope slide, tacky dot slides, and panes of glass. The sampling methodology allowed for the collection of samples for analyses using various analytical methods that included Raman microspectroscopy, pyrolysis gas chromatography mass spectrometry, ion chromatography and inductively coupled plasma mass spectrometry. These various methods allow for identification of organic and inorganic components as well the mineral distribution of suspended and settled particulate material.
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CHAPTER 1

INTRODUCTION

1.1 Solar Energy in Nevada

1.1.1 Energy Production in Nevada

Electrical consumption is a necessary part of modern living in the United States with the means of electric energy generation varying from state to state. Nevada’s power consumption is roughly 132.8x10^6 MWh a year and will increase as the state’s population and renewable energy initiative continues to grow. In-state production doesn’t meet the needs of the state’s consumption rate and the deficit has to be imported from neighboring states. Of the energy that is produced in Nevada 73% of generated electricity is from natural gas powered plants, despite Nevada’s lack of natural resources (U.S. Energy Information Administration, 2016). Geographically, Nevada has the potential for expanded wind power, but the primary source of renewable energy comes from geothermal energy sources in which Nevada is second in the nation. The state is the nation’s leader in terms of energy potential from solar power according to the sun index (Nebraska Energy Office, 2010).

1.1.2 Solar Production in Nevada

In 1997 the Nevada Legislature required (NRS 704.7801) that by 2025, Nevada’s Energy Portfolio Standard would have 25% of electricity sales coming from renewable energy resources with 6% of that coming from solar technology by 2016 (State of Nevada Public Utilities Commission,
2016). With the goal of meeting future energy needs using renewable energy, Nevada is becoming one of the leading states in renewable energy production. In 2015, there was a 46% increase in investment of solar technology with a total of $833 million being invested in Nevada (Solar Energy Industries Association, 2016). Currently Nevada has a total of 1300 MW of solar energy installed with 2408 MW of additional solar capacity expected by 2020 (Solar Energy Industries Association, 2016). Nevada’s current solar production capacity provides enough power to supply over 200,000 homes. Business and large retailers are also switching to renewable energy, most notably the Mandalay Bay Resort Convention Center which utilizes 5 MW of solar power which offsets approximately 25% of the resort’s electrical demand (Solar Energy Industries Association, 2014). Reduction in the production cost of photovoltaics, increased tax incentives offered by the state, job creation, an average of over 3500 hours of yearly sunshine and available land for solar farms will continue to make Nevada a favorable location for solar production. With the majority of new energy production being based on solar technology, the need to address potential operating and maintenance concerns is a priority.

1.2 The Need for Particulate Characterization

1.1.3 Large Scale Solar Production

With the current and projected increases in solar production the need for a comprehensive look into external influences on solar production is a necessity. In 2012, the Bureau of Land Management released a report saying that there was 19 million acres of land that could be potentially available for solar development within 6 states in
the southwest (Bureau of Land Management, 2012). Of these 6 states, Nevada has the largest total area, 9,076,145 acres, of federal land that could be used for solar development (Bureau of Land Management, 2012). Nevada’s renewable energy commitment, specifically to solar energy, as well its large area of viable land for solar development make it an ideal location to study factors that impact solar energy. Additionally, Nevada’s installed solar capacity grew by 17% in 2015 and $833 million was invested in solar installations which is a 46% increase over 2014 (Solar Energy Industries Association, 2016). Nevada also houses large-scale production facilities utilizing various generation methods such as photovoltaic and solar thermal. The move towards large-scale production facilities was made the default approach in Nevada following a ruling by the Public Utilities Commission of Nevada regarding rooftop electric generation. Despite the state’s renewable portfolio standard goal of adding more solar production, the commission decided to drastically reduce the amount that is paid to rooftop solar owners with the issuing of its net metering rules and rates which are greatly beneficial to the state’s largest investor-owned utility, NV Energy. The portfolio energy credit trading program was originally set up to allow rooftop owners to sell their portfolio energy credits to the utility. A statement on NV Energy’s website completely summarizes the net metering policy: “Over the next 12 years, the basic service charge will increase once every three years. This increase will be accompanied by a related decrease in the energy charge that net metering customers pay for each unit of energy delivered by NV Energy and decrease in the credit that NV Energy provides for energy delivered by net metering customers to NV
Energy’s system” (NV Energy, 2016). NV energy has increased its renewable portfolio standard by approximately 13% over the past 5 years (Governor's Office of Energy, 2015). As it stands, the state’s net metering policy coupled with its renewable energy portfolio ensure large scale solar production facilities will continue to dominate Nevada.

With an increase in large-scale solar production facilities, another area that will have to be examined in greater details is the ecological impact that result from solar installation. Research suggests that the range of ecological impacts from large scale solar facilities can include: change in nutrient dynamics, barriers to geneflow, invasive plant species, biodiversity loss, biota displacement, habit fragmentation and loss, reduced visibility, release of soil borne pathogens, mortality and species loss, and water stress (Hernandez, et al., 2014). The research presented here could augment our understanding of ecological effects caused by solar production.

1.1.4 Impacts of Particulate Deposition
The impact of particulate deposition has yet to be fully determined but what is known is that dust or particulate deposition can lead directly to light attenuation. With the severity of particulate deposition depending on the region, light attenuation can lead to a drop in generating efficiency greater than 40% (Sayyah, Horenstein, & Mazumder, 2014). Particulate deposition is a sub class of the broader industry term of “soiling” that can range from dust to avian feces. There is dogma in the solar industry that asserts that the natural removal of these particles by rain, wind, and snow is sufficient without instituted
periodic cleaning (Cuddihy, 1980). This may be true in areas that have frequent precipitation but regions that have limited annual precipitation should be examined more closely. Research has also shown that the Van der Waals forces between a surface and small particle (<50 µm) can prevent removal by wind forces approaching hurricane velocities (Cuddihy, 1980). As a result “natural” cleaning by wind should not be assumed for small particles. This dogma may also be the reason that specific mineral identification of particulate deposition on panels has not been viewed as a necessity and therefore has not been widely examined. There are also activation mechanisms that can result in particulate matter becoming resistant to natural removal as precipitation events become less frequent. A study of calcium carbonate and its impact on photovoltaic performance found that the mineral does have an effect on short circuit current and maximum power (Darwish, Sopian, Alawadhi, Kazem, & Alghoul, 2016). Characterization of particulate matter would allow for the identification of water soluble salts and minerals that could lead to insoluble particle being anchored to the surface (Cuddihy, 1980). A detailed characterization of the particulate matter deposition is needed to fully assess possible interactions on the surface of the panel.
CHAPTER 2
BACKGROUND

2.1. Raman Theory

2.1.1. The Raman Effect

Raman scattering was first observed in 1928 by C.V. Raman after being predicted theoretically by Adolf Smekal in 1923 (Loader, 1970). The phenomenon is a result of a light source interacting with a molecule’s vibrational energy levels producing inelastically scattered light i.e. Raman scattered light. From this scattered radiation, detailed information can be obtained about the molecules’ vibrational and rotational energy levels (Loader, 1970). This Raman spectrum is unique for every molecule and can be used for accurate identification purposes as well as to follow changes in structure that a molecule may undergo during a chemical reaction. For a molecule to be Raman active it must be polarizable during an interaction with an electromagnetic field.

The principle of Raman scattering can be explained by looking at the energy difference between the scattering system and the incident radiation (Long, 1977). In this interaction, the incident radiation excites an electron in the ground state \( E_0 \), stokes scattering to a higher energy virtual state. The observation of Stokes Raman scattering is dependent on the population difference in vibrational energy levels of which most will be in the ground state at room temperature (Loader, 1970). The electron then relaxes to \( E_{0+1} \) emitting the excess energy in
the form of electromagnetic radiation. The difference in photon energies is best described through the expression:

\[ \Delta E = E_{\text{final}} - E_{\text{initial}} \]

The change in energy (\(\Delta E\)) is described in terms of a wavenumber (\(\tilde{\nu}_M\)) seen below and is generally expressed in terms of inverse centimeters \(\text{cm}^{-1}\).

\[ \Delta E = h c \tilde{\nu}_M \]

The Raman bands are characterized by the magnitude of the wavenumber shift relative to the excitation sources’ incident wavenumber. It’s these wavenumber shifts that are quantified using the high resolution grating and a charge coupled device (CCD) detector in the Raman instrument.

2.1.2. Raman Microspectroscopy

A bench top Raman microscope was selected for this study. This instrument is a combination microscope and Raman spectrophotometer with interchangeable excitation lasers, interchangeable spectral gratings, and analysis software. The instrument has a mobile stage that allows mapping of surfaces or particles spread on a microscopic slide. The ability to obtain microscopic images as well as obtain Raman spectra is useful.

2.2 Gas Chromatography Mass Spectrometry

Gas chromatography is a separation technique that when coupled with a mass spectrometry can be a very powerful tool. The separation technique
is a physical separation method in which a mobile phase, composed of a moving inert gas containing volatized analytes, passes through or over a heated stationary phase contained in the column. Solubility differences between the various components of the sample vapor in the stationary phase are the primary determinate for separation of the mixture into its components (Willard, Merrit Jr., Dean, & Settle Jr., 1988). The analytes then become ionized by 70 eV electron impact which creates molecular and fragment ions. The ions are then accelerated by various potentials into the ion trap. The ion trap is a combination of electrostatic and RF fields that “capture” ions in a stable orbit. The trap then separates ions by selective destabilization leading to a sequential mass scanning. Ions of the same mass are ejected from the trap to the detector due to variations in the electrostatic and RF field. The ions then impact the electron multiplier an electrical current is detected, amplified and recorded. The relative ion abundances or the spectrum can be utilized to identify the parent molecule. Pyrolysis with methylation using Tetramethylammonium hydroxide (TMAH) was utilized to degrade complex organic mixtures while simultaneously derivatizing the degradation products. The TMAH methylates many degradation and polar pyrolysis products that would not normally be suitable for GC/MS analysis.

2.3 Ion Chromatography

Ion chromatography is a separation technique that utilizes an ion exchanging stationary phase and a mobile aqueous phase containing a dilute ionic buffer of Na₂CO₃. The mode of Ion Chromatography used for this study was anion exchange. The sample is introduced into the injection loop which is used to inject a fixed sample volume into the
column. The role of the eluent is to compete with the analyte for the ion exchange sites on the column and contribute to the selectivity of the separation while transporting the anions through the system. The sample ions proceed through the IC column where they interact with the stationary phase that is composed of positive cations that are covalently bonded to the column’s polymeric stationary phase. These interactions allow for the separation of analytes based on their relative affinity for the stationary phase. The sample then goes through a suppressor which exchanges metal ions for protons while converting the carbonate buffer into either carbonic acid or CO₂, both of which have low conductivity. The analyte anions are connected to acids with high conductivity which result in a significant increase in sensitivity. The separated analytes will then enter the flow cell of the detector containing two electrodes to which an AC potential has been applied (SeQuant, 2007). Ions entering the cell will cause and increase in current that is proportional to an increase in conductivity that is a linear function of the ion concentration (SeQuant, 2007). Using an external standard calibration, target anions can be measured quantitatively.

2.4 Inductively Coupled Plasma Atomic Emission Spectrometry

Atomic emission spectroscopy works on the principle of electrons being thermally excited from a lower atomic orbital to a higher atomic orbital and then returning to a lower energy atomic orbital during which the excess energy is emitted in the form of a photon. Each element can be identified and quantified by its unique set of emission lines
corresponding to the respective energy levels of their atomic orbitals. Atomic Emission spectroscopy requires that a sample be converted into a free, gaseous atom. This atomization source usually doubles as the excitation source. The atomization that was chosen was inductively coupled plasma which forms by ionizing a stream of argon gas. The plasma reaches greater temperatures than flame sources, up to 8000 °C, and generates better atomization and a higher excited state population (Wentworth, 1922). The emitted light is then dispersed by a monochromater to the two dimensional CCD array where the desired analyte emission wavelengths are recorded.
CHAPTER 3
MATERIALS AND METHODS

3.1 Sample Collection

Sampling was performed on a variety of surfaces, and from cracks and crevices. Sampling included direct sampling from vugs, solar panels, and improvised collection devices and by high volume air sampling. This allows for comparison of particulates suspended in the air, to those found in the top layer of soil, to those collected from solar devices. Direct collection was a term that was used to describe the collection of settled ambient dust. This included collection of settled particles directly from solar panels, top soils at solar sites, desert vugs, and dust that settled directly onto microscope slides.

A pane of glass was also set up for collection at an urban location in Southern Nevada for 10, 20, 30, 60, and 100 days. The pane was cleaned prior to each set up using soap and water followed by methanol. The pane was secured using two standard lab stands and lab clamps. The angle of the pane was set to approximately 0° because attempts to set the pane at 45° resulted in the pane falling over on windy days. This is most likely due to the larger cross section of the pane at 45° and lack of weight at the base of the stand. After the pane sat for the allotted sampling period, a wipe sample was taken using a 55mm diameter glass fiber filter manufactured by VWR (Radnor, PA). This sampling method is the same as the method used for sample collection from the solar panels at various locations. This approach allowed for the evaluation of rural versus urban samples as well as collection from different solar sites.
in Southwest Nevada. The solar panel wipes taken from the City of Las Vegas Solar Facility were labeled Solar Panel A and B. These panels are shown below in Figure 1 with Panel A being smaller and lower to the ground.

![Image of Solar Panel A (smaller) and B (larger) City of Las Vegas](image)

Figure 1: Image of Solar Panel A (smaller) and B (larger) City of Las Vegas

The panels at the Copper Mountain 1 Solar Facility were all photovoltaic panels but were labeled by relative age of the section. The samples labeled Copper Mountain 9 year, Top soil 10MW, and Reference Panel were all taken in the orange region below in Figure 2. The 7 year panel was taken within the yellow region and the near tower sample was collected with the red square also seen below.
The UNLV rooftop pane also allows for direct correlation between the mineral identification via Tacky Dot and plain slides, organic constituents and anion/cation concentrations from a single location given that they are sampled at the exact same time and location. A direct collection sampling apparatus was not set up at any solar facility due to access to a facility as well its remote location.

Top soil samples were also collected throughout various locations throughout Southern Nevada. A collection vessel, 50mL polystyrene conical centrifuge tube manufactured by VWR (Radnor,PA), was placed perpendicular to the top soil and a paint brush was used to sweep the soil into the container. Only a few millimeters of the topsoil was collected since the surface is probably the fraction of the soil that will become suspended into the air.

Other samples were collected from geological formation known as vugs, seen below in figure 3. A vug is a cavity in a rock that given the
right position can serve as an excellent particulate collector. The vugs were collected to obtain long time accumulations of suspended particulate versus the samples that would be collected at other locations. The vug samples were collected at a desert location in the southwest part of the Las Vegas Valley. A 50mL polystyrene conical centrifuge tube manufactured by VWR (Radnor, PA), was placed near the opening of the vug and the contents were brushed or scooped out using a metal spatula depending on the orientation of the vug.

![Figure 3: Image of Vug 1 in Southwest Las Vegas Valley](image)

A major component of Raman imaging of minerals was accomplished utilizing a Tacky Dot slide that was manufactured by Structure Probe, Inc (West Chester, PA); the product has since been discontinued. The Tacky Dot slide was utilized because imaging dust in “bulk” presented many issues. Depth of field and quantification were the main factors in the decision in utilizing the Tacky Dot slide. A large depth of field makes it difficult to focus on multiples particles. Depth of field is
the distance between the nearest and furthest objects that appear in focus and given the distribution of size in dust that was collected, depth of field issues were apparent between multiple particles. The Tacky Dot array deposited the particulates in a single layer, most of the time, which reduced but did not eliminate the depth of field issues. The Tacky Dot also utilized a grid system which made identification and quantification easier. The grid also made it possible to keep track of regions that had already been measured. A Tacky Dot slide is a microscope slide that is arranged in a grid with adhesive dots having a diameter of either 200µm or 150µm; see below in figure 4.

Figure 4: Tacky Dot Slide

These microscope slides were used to mount the particulate samples collected from vugs, solar panels, and the top soil layer. A soil sample was mounted to the Tacky Dot slide by placing approximately 0.250 mg of sample, sieved at 60µm, into a 50 mL centrifuge tube. The Tacky Dot slide is then placed into the centrifuge tube and sealed. The tube is then gently rotated and flipped end-over-end for approximately 2 minutes. The Tacky Dot is then removed from the centrifuge tube and placed into a
slide box to prevent contamination. The exposed Tacky Dot slide is given a period of approximately 6 hours for the adhesive to set.

The Tacky Dot slides were also used to directly collect particulate matter at an urban location in Southern Nevada for 10, 20, 30, 60, and 100 days. A slide was secured to the roof using a standard lab stand and clamp with the angle being set to approximately 45°. Tacky Dot slides were labeled prior to being set up for collection and upon collection were placed in a microscope slide case.

A plain glass slide manufactured by VWR (Radnor, PA) was also exposed using the same procedure. This was to compare the collection of particulate matter on surfaces similar to solar devices versus the adhesive Tacky Dot slide. These slides received no pretreatment and were set up for collection at an urban location in Southern Nevada for 10, 20, 30, 60, and 100 days. They were placed in the same location as the Tacky Dot slides using a standard lab stand and clamp at approximately a 45° angle.

3.2 Instrumentation and Experimental Setup

3.2.1 Raman Microspectroscopy

All Raman microspectroscopy measurements performed on a DXR™xi Raman imaging microscope manufacture by Thermo Scientific™ (Waltham, MA) with the excitation laser and objective lenses being obtained from the same manufacturer. Measurements were generally carried out using a 10X objective and a 532nm excitation laser. The software provided by the manufacturer was utilized for imaging and analyses. In preparation for Raman Imaging, samples were placed on the microscope stage and focused
using the 10x objective with the eye-piece also 10x, thus giving a total magnification of 100x. The first step in obtaining Raman imaging is the creation of the “mosaic.” A Mosaic is a feature of the software that “stiches” together individual images (~873 x 530µm) to form a single large image. The mosaic allows for the creation of mapped regions in which spectral data can be collected and particle size measurements can be recorded. The regions for Raman imaging were chosen randomly with 10 different locations for each sample. A mosaic was created for each location. The laser power for each sample was adjusted between 5.0 and 7.5 mW depending on fluorescence of the sample. High sample fluorescence can lead to the suppression of the Raman signal or to over exposure of the charge coupled device (CCD). The exposure time of each sample was set between 320 to 280 Hz depending on the signal to noise ratio. Frequency for this instrument is the amount of time it takes the laser to raster across a segment of a region. This means that a lower frequency will result in an increased exposure time. Given the compositional variation of the sample this range was chosen as a balance between weak scattering minerals and strong scattering minerals. Longer exposure times generally yielded a higher signal-to-noise ratio but would have had to be evaluated and adjusted on a particle-to-particle basis to prevent overexposure. This approach was not pursued due to large sample volume. Longer exposure times increase the time required for imaging. The number of scans performed varied per sample from 80 to 120 scans per region and impacted the overall background of the sample with the higher number of scans usually producing a higher signal-to-noise ratio. The number of scans that was selected was primarily dependent on run time.
The reason for not choosing to a large number scans is that it did not yield a noticeably better spectrum for the longer run time and the extra disc space required to store the data could not be justified. A high number of scans also tended to lead to saturation of the CCD. This was an issue that was encountered with high intensity scattering minerals that would show signs of what is termed “noise clipping.” If this was caught early during a run the exposure time or laser power would be reduced, otherwise the particle was labeled “no signal” because an identifiable spectra could not be obtained. The pixel size of each region varied from 3 to 4µm and this was also based on runtime. Decreasing the pixel size did not improve the resolution of the spectral data.

A total of four different apertures are available on the microscope including: 25-µm confocal pinhole, 25-µm slit, 50-µm confocal pinhole, and 50-µm slit. An aperture is used to achieve spectral resolution by controlling the amount of Raman scattered light that passes onto the detector (Spectroscopy, 2016). In the case of a confocal pinhole aperture, the out of focus light is eliminated before reaching the detector. The 25-µm confocal pinhole aperture was used for each run to collect the strong Raman scattering signal while reducing the overall background signal. Parameters and their primary effect can be seen below in Table 1.
For each sample, 10 regions were randomly selected and spectral data was collected. After all regions were imaged and spectral data was acquired, the sample would be processed. Data analysis was performed by doing the following: first the image of the measurements’ region was transferred to editing software (Microsoft Paint™) where the particles were assigned a numerical identification number. A Raman spectra was then collected for each particle and it was labeled according to its identification which was determined using multiple Raman mineral databases. The first was made in the lab using a mineral collection purchased from Carolina (Burlington, NC) from which a sample was pulverized, placed on a microscope slide, and spectral data was collected. A database published by the University of Arizona and Caltech known as RRUFF library was used as a secondary source for Raman mineral spectra. Spectral data was downloaded from their database and converted into a library within the DXRxi software. The third mineral database was obtained from the vendor and was used as the primary source for identification.

Spectral data for all samples were collected using the 532 nm laser with the parameters listed below in Table 2.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Primary Effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laser Power</td>
<td>Signal</td>
</tr>
<tr>
<td>Aperature</td>
<td>Background Reduction</td>
</tr>
<tr>
<td>Exposure Time</td>
<td>Signal</td>
</tr>
<tr>
<td># of Scans</td>
<td>Smoothing</td>
</tr>
<tr>
<td>Pixel Size</td>
<td>Resolution</td>
</tr>
</tbody>
</table>

Table 1: Raman Parameters and Effects
If no characteristic Raman signal was identified then the particle was labeled “No Signal.” Particle size was manually measured using the “measure feature length” tool of the software and recorded next to the numerical value associated with the particle. Particles perceived to be under 10µm were not analyzed due to the limited resolution of the processing software. Spectra that were collected that had a graphitic-like signature, a band around 1600 cm⁻¹, was labeled as such. Particles that possessed two characteristic Raman spectra of two different minerals and the particles that were graphitic-like were categorized as “mixed spectra”. It was investigated on whether the signal was a result of two different particles, with only one visible, or if the particle was composed of two different minerals.

3.2.2. Pyrolysis Gas Chromatography Mass Spectrometry (GC-MS)

Pyrolysis GC-MS was performed on a Varian CP-3800 GC and Saturn 2200 MS/MS manufactured by Varian, Inc. (Palo Alto, CA). The pyrolysis was performed with a CDS Analytical (Oxford, PA) pyroprobe 2000. Samples were prepared by adding ~35mg aliquot for soil samples or ~10 mg of the panel wipe into a 2 mm diameter quartz tube manufactured by CDS Analytical (Oxford, PA). The quartz tube was then packed with quartz wool manufactured by VWR (Radnor, PA) followed by the addition of 20 µL of the derivatization agent, 25% Tetramethyl ammonium hydroxide (TMAH). The sample was inserted into the pyroprobe, dried for 30s at 90°C before
being heated to 600°C for 10s followed by a 10:1 split ratio injection into the GC. The column Rtx-5ms with the dimensions of 30m x 0.25mm and a phase thickness of 0.25µm was manufactured by Restek (Bellefonte, PA). The oven temperature program for all runs was an initial temperature of 40°C for 6 min, then a ramp up at a rate of 10°C/min to 280°C where temperature was maintained for an additional 10 minutes. Helium was utilized as the carrier gas at 1mL/min and analytes were identified using the NIST’s 2007 standard library.

3.1.3. Ion Chromatography

Ion Chromatography was performed using a model 883 Basic IC plus manufactured by Metrohm (Riverview, FL). Samples were injected using a 20 µL injection loop and a Metrosep A Supp 5 250/4.0 column for separation. Flow rate was set to 0.700ml/min with the column pressure usually around the 1400 psi. Samples were prepared by adding 250mg or ~2cm wipe sample to a 10 mL test tube manufactured by VWR (Randor, PA). 5 mL of deionized water was added, the sample was vortexed for 15s and stored at room temperature for 24 hours. After 24 hours the sample was vortexed for 15s and filtered through a 0.2µm Acrodisc® filter manufactured by Pall Corporation (Port Washington, NY) and injected into the IC. A four-point calibration was performed on the following anions: Fluoride, Chloride, Bromide, Nitrate, Phosphate and Sulfate. The manufacture’s software was used for peak identification and data collection. The calibration curve and anion concentrations were calculated using Microsoft Excel™.
3.1.4. Inductively Coupled Plasma Optical Emission Spectroscopy (ICP_OES)

Cation analysis was performed using an ICPE-9000 manufactured by Shimadzu (Tokyo, Japan). Samples were digested based upon U.S. E.P.A. method 3050B (United States Environmental Protection Agency, 1996). An aliquot of 500 mg or the remaining mass of sample was placed into a teflon digestion tube manufactured by Savillex (Eden Prairie, MN) and 5 mL of 1:1 HNO₃ was added. The sample was then covered and refluxed on a digital block heater manufactured by VWR (Radnor, PA) at 95°C for 15 minutes. The samples were then cooled and 2.5 mL of concentrated HNO₃ was added, the sample was covered, placed back on the block heater, and refluxed at 95°C for 30 minutes. The samples were removed from heat and cooled to near room temperature. After cooling 1 mL of DI water and 1.5 mL of 30% H₂O₂ was added. The samples were covered and refluxed for 120 minutes at 95°C. After digestion samples were cooled to room temperature, brought to volume (50 mL), filtered through 0.45µm nylon filters, and placed into 50ml polystyrene tubes both manufactured by VWR (Radnor, PA). Calibration and sample measurements were performed using the instrument software then transferred to Microsoft Excel™ for data processing.
CHAPTER 4
RESULTS AND DISCUSSION

4.1 Mineral Characterization

4.1.1 Introduction

Mineral identification by Raman spectroscopy is fairly common and the technique is widely used in academia, pharmaceutical, and materials industries. Raman microspectroscopy is generally done on simple mixtures, pure sample, or polished surface where all particles being examined are the same size and focal depth. These minerals are usually large particles, 0.5 to 1 mm coarse sand, and fairly uniform in composition, whereas the particulates that we are examining are characterized as silt, 3.9 to 62.5 µm according to the Udden-Wentworth Scale (Wentworth, 1922). Difficulties arose as we started imaging and gathering spectra on our first preliminary samples. The complex composition of the matrix being examined posed many problems that were not initially anticipated. Giving the wide range of particle size, 6.5 to >100 µm, depth of field issues posed many challenges when imaging and collecting good high signal to noise ratio spectra. Initially we attempted to use the correlation function of the software to map particle distributions. This would have allowed a set area to be measured with a standard spectrum to be applied, as a reference, resulting in the correlation to various mineral spectra in a region. Particles would appear in a gradient from red to blue showing high correlation to low correlation respectively. This correlation function appeared to be
calculated by the software, primarily on area of the experimental spectra above an assumed baseline and yielded both false positives and negatives. This was speculated after observing the same mineral spectra at two very different intensities not showing up on the correlation feature of the software. This failure was probably also a result of the various particle sizes and various depths of field where the same minerals could be found. The software does not distinguish a raise in the baseline from a peak. Another issue encountered was the presence of graphitic-like coating that was present on many particles. Particles exhibiting this phenomenon would have the characteristic peaks of a known mineral as well as a broad feature around 1600 cm⁻¹ as seen below in figure 10 and 11. It was thought that these graphitic-like structures were a result of the burning of humic materials, commonly found in top soil, by the raman lasers. This was shown by examining pure humic acid and observing the effect of burning as shown below in Figure 5 with the spectra being seen in Figure 6.
Approximately 5 mL of a humic acid solution and 250 mg of a strong Raman scattering mineral calcite were combined and a spectra was obtained. The spectra was very similar to what we will describe as mixed spectra below, see Figure 7. The degree of broadening caused by the graphitic-like coating does vary between samples as seen between Figure 7 and 8.
These factors were crucial in deciding to use the manual data analysis method as discussed previously.

Particles exhibiting what we have described as mixed spectra are labeled as such. The particle being imaged could actually have a carbon coating as illustrated in the spectra above. The other possible explanation is that a strong Raman scattering mineral is below or imbedded in the particle being imaged as illustrated below in Figure 12.

4.1.2 Analysis by Raman Microspectroscopy
Several common minerals were identified and from the direct slide sampling included carbonates, quartz, gypsum, anatase, along with a few less common minerals that will be presented in the results below. Comparisons of particulate deposition of rural versus regional, adhesive versus plain slide, and variation of sample time will also be presented.

The Tacky Dot slide from the 10-day sampling period is presented as an example. This slide had a total of 69 particles that were analyzed over an approximate area of 0.47 µm² in which only 30.3% of particles yielded an identifiable Raman spectra. The results are summarized below in Table 3.

<table>
<thead>
<tr>
<th>Total Particles</th>
<th>Carbonate</th>
<th>Graphitic</th>
<th>Mixed spectra</th>
</tr>
</thead>
<tbody>
<tr>
<td>69</td>
<td>15.9%</td>
<td>10.1%</td>
<td>4.3%</td>
</tr>
</tbody>
</table>

Table 3: Tacky Dot 10-Day Results

The mixed spectra were identified as graphitic-like with carbonate along with carbonate with quartz seen in Figure 9.
The Tacky Dot slide from the 20-day sampling period had a total of 132 particles that were analyzed over an approximate area of 0.42 µm² in which only 13.4% of particles yielded an identifiable Raman spectra. The results are summarized below in Table 4.

<table>
<thead>
<tr>
<th>Total Particles</th>
<th>Carbonate</th>
<th>Quartz</th>
<th>Graphitic</th>
<th>Mixed spectra</th>
</tr>
</thead>
<tbody>
<tr>
<td>132</td>
<td>2.3%</td>
<td>1.2%</td>
<td>7.6%</td>
<td>2.3%</td>
</tr>
</tbody>
</table>

Table 4: Tacky Dot 20-Day Results

The mixed spectra were identified as graphitic-like with carbonate along with graphitic-like and quartz.

The original Tacky Dot sample for this 30-day sampling period was damaged upon removal from sampling apparatus. The results below are for a second sampling period. The Tacky Dot slide from the 30-day B sampling period had a total of 366 particles that were analyzed over an approximate area of 0.46 µm² in which only 22.4% of particles yielded an identifiable Raman spectra. The results are summarized below in Table 5.

<table>
<thead>
<tr>
<th>Total Particles</th>
<th>Carbonate</th>
<th>Quartz</th>
<th>Graphitic</th>
<th>Anatase</th>
<th>Mixed spectra</th>
</tr>
</thead>
<tbody>
<tr>
<td>366</td>
<td>5.5%</td>
<td>1.1%</td>
<td>11.5%</td>
<td>0.5%</td>
<td>3.8%</td>
</tr>
</tbody>
</table>

Table 5: Tacky Dot 30-Day Results

The mixed spectra were identified as graphitic-like with carbonate, graphitic-like and quartz.

The Tacky Dot slide from the 60-day sampling period had a total of 312 particles that were analyzed over an approximate area of 0.48 µm² in
which only 22.4% of particles yielded an identifiable Raman spectra. The results are summarized below in Table 6.

<table>
<thead>
<tr>
<th>Total Particles</th>
<th>Carbonate</th>
<th>Quartz</th>
<th>Graphitic</th>
<th>Anatase</th>
<th>Mixed spectra</th>
<th>Other minerals</th>
</tr>
</thead>
<tbody>
<tr>
<td>312</td>
<td>4.2%</td>
<td>1.0%</td>
<td>13.1%</td>
<td>2.2%</td>
<td>1.3%</td>
<td>0.6%</td>
</tr>
</tbody>
</table>

Table 6: Tacky Dot 60-Day Results

The mixed spectra were identified as graphitic-like with carbonate.

The other minerals identified were Orthoclase along with an unknown spectra both shown below in Figure 10 and 11.
The Tacky Dot slide from the 100-day sampling period had a total of 312 particles that were analyzed over an approximate area of 0.42 µm² in which only 11.9% of particles yielded an identifiable Raman spectra. The results are summarized below in Table 7.

<table>
<thead>
<tr>
<th>Total Particles</th>
<th>Carbonate</th>
<th>Quartz</th>
<th>Graphitic</th>
<th>Anatase</th>
<th>Rutile</th>
</tr>
</thead>
<tbody>
<tr>
<td>312</td>
<td>1.0%</td>
<td>1.0%</td>
<td>8.7%</td>
<td>1.0%</td>
<td>0.3%</td>
</tr>
</tbody>
</table>

Table 7: Tacky Dot 100-Day Results

The plain slide from the 10-day sampling period was not collected at the same time as the 10-day tacky dot sample. Since the sampling was to be performed simultaneously with the tacky dot it was not analyzed.

The plain slide from the 20-day sampling period had a total of 272 particles that were analyzed over an approximate area of 2.5 µm² in which only 20.6% of particles yielded an identifiable Raman spectra. The results are summarized below in Table 8.

<table>
<thead>
<tr>
<th>Total Particles</th>
<th>Carbonate</th>
<th>Quartz</th>
<th>Graphitic</th>
<th>Anatase</th>
<th>Mixed Spectra</th>
<th>Other Minerals</th>
</tr>
</thead>
<tbody>
<tr>
<td>272</td>
<td>4.0%</td>
<td>0.7%</td>
<td>6.3%</td>
<td>1.1%</td>
<td>0.7%</td>
<td>1.5%</td>
</tr>
</tbody>
</table>

Table 8: Plain Slide 20-Day Results

The mixed spectra were identified as graphitic-like with carbonate along with carbonate and antimony (however unlikely) shown in Figure 14. The other minerals included an unidentified spectra, inquigueite, and calcium oxalate hydrate (CaC₂O₄) shown below in Figures 12-15 in their respective orders.
Figure 12: Plain Slide 20-Day Carbonate and Antimony (Standard References in Blue and Green)

Figure 13: Plain Slide 20-Day Unidentified

Figure 14: Plain Slide 20-Day Inquiqueite (Standard Reference in Blue)
The plain slide from the 30-day sampling period had a total of 402 particles that were analyzed over an approximate area of 2.5 \( \mu \text{m}^2 \) in which only 29.8% of particles yielded an identifiable Raman spectra. The results are summarized below in Table 9.

<table>
<thead>
<tr>
<th>Total Particles</th>
<th>Carbonate</th>
<th>Quartz</th>
<th>Graphitic</th>
<th>Anatase</th>
<th>Rutile</th>
<th>Mixed Spectra</th>
<th>Other Minerals</th>
</tr>
</thead>
<tbody>
<tr>
<td>402</td>
<td>10.2%</td>
<td>1.5%</td>
<td>10.7%</td>
<td>0.5%</td>
<td>0.2%</td>
<td>5.2%</td>
<td>1.5%</td>
</tr>
</tbody>
</table>

Table 9: Plain Slide 30-Day Results

The mixed spectra were identified as graphitic-like with carbonate, graphitic and anatase, carbonate and anatase (Figure 17), along with carbonate and rutile (Figure 18). The other minerals included three
unidentified spectra (Figures 19, 20, and 21), gypsum (Figure 22), and alunogen \((\text{Al}_2(\text{SO}_4)_3\cdot17\text{H}_2\text{O})\) (Figure 23) spectra can be seen below.

Figure 17: Plain Slide 30-Day Carbonate and Anatase (Standard Reference in Blue and Green)

Figure 18: Plain Slide 30-Day Carbonate and rutile (Standard Reference in Blue and Green)

Figure 19: Plain Slide 30-Day Unidentified spectra
Figure 20: Plain Slide 30-Day Apatite and Unidentified spectra (Standard Reference in Blue)

Figure 21: Plain Slide 30-Day Unidentified spectra

Figure 22: Plain Slide 30-Day Gypsum (Standard Reference in Blue)
A second plain slide was set up for a 30-day sampling period because the original tacky dot slide was damaged during removal from sampling apparatus. The plain slide sample labeled 30-day B corresponds to the Tacky dot sample 30-day B. The plain slide 30-day B had a total of 402 particles that were analyzed over an approximate area of 2.5 µm² in which only 41.8% of particles yielded an identifiable Raman spectra. The results are summarized below in Table 10.

<table>
<thead>
<tr>
<th>Total Particles</th>
<th>Carbonate</th>
<th>Quartz</th>
<th>Graphitic</th>
<th>Anatase</th>
<th>Mixed Spectra</th>
<th>Other Minerals</th>
</tr>
</thead>
<tbody>
<tr>
<td>407</td>
<td>12.3%</td>
<td>1.7%</td>
<td>14.5%</td>
<td>1.5%</td>
<td>8.6%</td>
<td>3.2%</td>
</tr>
</tbody>
</table>

Table 10: Plain Slide 30-Day B Results

The mixed spectra were identified as graphitic-like with carbonate, graphitic and quartz, unknown with anatase (Figure 24), carbonate and anatase along with graphitic-like ankerite (Ca(Fe,Mg,Mn)(CO₃)₂) (Figure 32). The other minerals included three unidentified spectra (Figures 25, 26, 29, and 31), microcline (KAlSi₃O₈) (Figure 27), brookite (TiO₂) (Figure 28), gypsum, titanite (CaTiSiO₅) (Figure 30), and apatite (NaAlSi₃O₈) (Figure 33) spectra can be seen below.
Figure 24: Plain Slide 30-Day B Unidentified and Anatase (Standard Reference in Blue)

Figure 25: Plain Slide 30-Day B Unidentified

Figure 26: Plain Slide 30-Day B Unidentified
Figure 27: Plain Slide 30-Day B Microcline (Reference Spectra in Blue)

Figure 28: Plain Slide 30-Day B Brookite (Reference Spectra in Blue)

Figure 29: Plain Slide 30-Day B Unidentified
Figure 30: Plain Slide 30-Day B Titanite (Reference Spectra in Blue)

Figure 31: Plain Slide 30-Day B Unidentified and Apatite (Reference Spectra in Blue)

Figure 32: Plain Slide 30-Day B Graphitic-like and Ankerite (Reference Spectra in Blue)
The plain slide from the 60-day sampling period had a total of 199 particles that were analyzed over an approximate area of 2.4 µm² in which only 23.2% of particles yielded an identifiable Raman spectra. The results are summarized below in Table 11.

<table>
<thead>
<tr>
<th>Total Particles</th>
<th>Carbonate</th>
<th>Graphitic</th>
<th>Anatase</th>
<th>Rutile</th>
<th>Mixed Spectra</th>
<th>Other Minerals</th>
</tr>
</thead>
<tbody>
<tr>
<td>199</td>
<td>3.0%</td>
<td>16.7%</td>
<td>0.5%</td>
<td>0.5%</td>
<td>1.0%</td>
<td>1.5%</td>
</tr>
</tbody>
</table>

Table 11: Plain Slide 60-Day Results

The mixed spectra were identified as graphitic-like with carbonate. The other minerals identified were three particles of copper(II)phthalocyanine, which is not actually a mineral, and the spectra shown below in Figure 34 with standard spectra in Figure 35.
The plain slide from the 100-day sampling period had a total of 320 particles that were analyzed over an approximate area of 2.4 µm² in which only 23.0% of particles yielded an identifiable Raman spectra. The results are summarized below in Table 12.

<table>
<thead>
<tr>
<th>Total</th>
<th>Carbonate</th>
<th>Graphitic</th>
<th>Anatase</th>
<th>Mixed Spectra</th>
<th>Other Mineral</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particles</td>
<td>8.1%</td>
<td>10.3%</td>
<td>0.3%</td>
<td>3.4%</td>
<td>0.9%</td>
</tr>
</tbody>
</table>

Table 12: Plain Slide 100-Day Results

The mixed spectra were identified as graphitic-like with carbonate, graphitic-like with quartz along with graphitic-like with anatase. The other minerals were identified as two particles of nitratine (NaNO₃)
(Figure 36), and talc (Mg₃Si₄O₁₀(OH)₂ (Figure 37), the spectra are shown below.

![Figure 36: Plain Slide 100-Day Nitratine (Reference Spectra in Blue)](image1)

![Figure 37: Plain Slide 100-Day Talc (Reference Spectra in Blue)](image2)

The data collected from the various sampling periods were then put into two graphs in an attempt to visualize trends. The data consisting of particle counts of each mineral or mineral type was normalized to the total number of particles in that sample. The totals from each sample type are present separately. The plain slide collectors for all days showed a high relative abundance of graphitic-like and carbonate particles as seen in Figure 38. This is also seen in the normalized data (Figure 39) where the mixed spectra particles from the 30-Day and 30-Day B appear the most abundant. This is most probably due to the high
presence of graphitic-like particles which result in most mixed spectra particles having a graphitic component.

![Figure 38: Plain Slide Totals](image1)

The Tacky Dot Slides’ mineral composition (Figure 40) was primarily graphitic with most samples having over twice the number of graphitic
particles compared to other identifiable spectra. A decrease in the fraction of particles with identifiable spectra is observed after the 30-day sampling period while the total number of particles decreases slightly as seen in Figure 42. Normalization highlights the total percentage of graphitic particles compared to other minerals as seen in Figure 41. The outlier is the 10-day sampling period in which a larger percentage of carbonate particles is observed. Environmental factors could have contributed to this including prevailing winds and dust generated by nearby construction.

![Tacky Dot Totals](image)

Figure 40: Tacky Dot Totals
Total particle collection counts varied widely between the two different sample types labeled TD and PS for Tacky Dot and plain slide respectively. The 20- and 60-day period had a 51% and 36% difference, respectively, while the 100-day sample had 2% difference.
Particles with identifiable Raman spectra were more prevalent on the plain slide sample compared to the Tacky Dot as shown below in Figures 43, 44, and 45. This is also demonstrated in the normalized data with a 50% difference in abundance between carbonate and mixed spectra particles as well as a 32% difference in the number of overall identifiable particles. This could be because of retention discrimination of the different sampling surfaces.

![Figure 43: Total Mineral Identification by Slide Type](image-url)
Figure 44: Total Mineral Identification by Slide Type (Plain Slide 30-Day Removed)

The mineral identification totals for the different slide type and sampling periods are shown in Figures 46 and 47. The sample identifications were abbreviated PS and TD for plain slide and tacky dot respectively. We can see that there is a plateau in the total number of particles and identifiable particles that occur after 30 days. This trend
could be due to various factors such as deterioration of the tacky dot adhesive as well as weather patterns and will have to be investigated further.

Figure 46: Total Mineral Count by Day

Figure 47: Total Mineral Count by Day (Normalized)
4.2 Organic Composition of Dust

4.2.1 Analysis using Pyrolysis GC-MS

Gas Chromatography Mass Spectrometry (GC-MS) was utilized to compare overall total ion chromatograms (TIC) of samples as well as ion chromatograms for several target ions that generally correspond to hydrocarbons (57), saturated fatty acids (74), methylated carbohydrates (101 and 129), and lignin phenols (165). A sample total ion chromatogram and target ion chromatogram are present below in Figure 48 along with peak identification in Table 13.
Table 13: Compound Identification Key for TIC in Figure 51

<table>
<thead>
<tr>
<th>Peak</th>
<th>Identification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>C14 Fatty Acid Methyl Ester (Methyl tetradecanoate)</td>
</tr>
<tr>
<td>2</td>
<td>9H-Purin-6-Amine, N,N,9-trimethyl</td>
</tr>
<tr>
<td>3</td>
<td>C15 Fatty Acid Methyl Ester (Isomer)</td>
</tr>
<tr>
<td>4</td>
<td>C15 Fatty Acid Methyl Ester (Isomer)</td>
</tr>
<tr>
<td>5</td>
<td>C15 Fatty Acid Methyl Ester (Isomer)</td>
</tr>
<tr>
<td>6</td>
<td>C16 Fatty Acid Methyl Ester (Isomer i.e Pentadecanoic acid)</td>
</tr>
<tr>
<td>7</td>
<td>C16:1 (i.e 11-Hexadecenoic Acid, Methyl Ester)</td>
</tr>
<tr>
<td>8</td>
<td>C16 Fatty Acid (i.e Hexadecanoic Acid, Methyl Ester)</td>
</tr>
<tr>
<td>9</td>
<td>C17 Fatty Acid Methyl Ester Isomers</td>
</tr>
<tr>
<td>10</td>
<td>Unknown (Possible Fatty Acid Derivative)</td>
</tr>
<tr>
<td>11</td>
<td>C18:1 (Octadecenioc Acid, Methyl Ester Isomers)</td>
</tr>
<tr>
<td>12</td>
<td>C18 (Octadecenioc Acid, Methyl Ester)</td>
</tr>
<tr>
<td>13</td>
<td>C18:2 (Octadecenioc Acid, Methyl Ester Isomers)</td>
</tr>
<tr>
<td>14</td>
<td>C19:1 (Nonadecenioc Acid)</td>
</tr>
<tr>
<td>15</td>
<td>C19 (Nonadecanoic Acid)</td>
</tr>
<tr>
<td>16</td>
<td>Alkene (Possibly C35H70)</td>
</tr>
<tr>
<td>17</td>
<td>C20 (Eicosanoic Acid, Methyl Ester)</td>
</tr>
<tr>
<td>18</td>
<td>α-D-Glucopyranoside, Phenyl 2,3,4,6-tetra-O-methyl-</td>
</tr>
</tbody>
</table>

Compound identification was confirmed by comparison to a NIST standard library as seen below in Figure 49 for Hexadecanoic acid.

The compound classes were selected because they are common plant and soil organic carbon constituents. The data is meant to provide a qualitative overview of the organic composition of various samples.

The TICs for the two desert vug samples, shown below in Figure 50, have a similar profile with the Vug 2 sample having a larger overall signal. This is reasonable given that the samples were taken in close...
proximity of each other but orientated differently with respect to the ground. Vug 1 was orientated perpendicular and Vug 2 was parallel. When these are compared to Vug 3 taken approximately 222 km away, there is a reduced number of peaks in Vug 3, as well as an overall lower intensity as seen in Figure 51. The difference in the TICs of the Southern Nevada vugs and Southern Utah vug can be attributed to the composition of the nearby topsoil.

Figure 50: Vug 1 (Top) and 2 (Bottom) Total Ion Chromatograms
Figure 51: Vug 1(Top), 2(Middle) and 3(Bottom) Total Ion Chromatograms

The TICs from dust that was vacuumed off of the panels and topsoil from the nearby ground were very similar as shown in Figure 52. The ion chromatograms for the target ions at 57, 74, 101, 129, and 165 were examined to provide further correlation between the two samples as shown in Figure 53 and 54. The total and target TICs show that the particulate matter on the panel is very similar to that of the nearby topsoil. A similar trend is also seen from a panel wipe that was collected as shown in Figures 55 and 56.
Figure 52: City of Las Vegas Panel Vacuum (Top) and Top Soil (Bottom) Total Ion Chromatograms
Figure 53: City of Las Vegas Panel Vacuum Ion Chromatograms (Target Ions) 57 (Top), 74, 101, 129, and 165 (Bottom)
Figure 54: City of Las Vegas Top Soil Ion Chromatograms (Target Ions) 57 (Top), 74, 101, 129, and 165 (Bottom)
Figure 55: City of Las Vegas Solar Panel A Total Ion Chromatogram
The TICs and target ion chromatograms from the Copper Mountain Solar Facility are similar to the 7-year panel wipe, 9-year panel wipe, and topsoil sample. By examining the TICs from each sample you can see the overall chromatograms for the two panels and the topsoils are similar and the target ion chromatograms for each sample are also similar. The similar chromatograms strengthen the postulate that the organic composition of the dust on the solar panels is very similar to that of the nearby soil as illustrated in Figures 57, 58, 59, and 60.

It is interesting to note that the different TICs from the City of Las
Vegas sample showed a larger organic signal than the Copper Mountain 1 samples.

Figure 57: Copper Mountain 1 Topsoil (Top), 9-year Panel (Middle), and 7-year Panel (Bottom) Total Ion Chromatograms
Figure 58: Copper Mountain 1 Topsoil Ion Chromatograms (Target Ions) 57 (Top), 74, 101, 129, and 165 (Bottom)
Figure 59: Copper Mountain 1 9-year Panel Wipe Ion Chromatograms (Target Ions) 57 (Top), 74, 101, 129, and 165 (Bottom)
4.3 Anion Composition

4.3.1 Analysis by Ion Chromatography

Anion identification by ion chromatography is a standard analysis in environmental monitoring of soils. The anions selected for this study were fluoride, chloride, bromide, nitrate, phosphate and sulfate. The data presented is meant to provide a qualitative overview of normalized anion distributions throughout the various matrices. The data is qualitative because while an external calibration was performed exact masses were of the filters and particulates were not recorded. To
facilitate comparisons graphical representations of the various anions all have been normalized to the sulfate concentration. Below are the qualitative results of the non-normalized data as well as the normalized data separated into various sections based on sample location. The concentrations are presented in parts per million (mg/L) for the non-normalized data.

The samples with varying exposure times were collected using a glass pane set at 0° on the roof of the chemistry building. These samples showed no significant variation or trend in anion distribution. The only discernable trend was an increase in sulfate concentration with increased exposure times. There are many factors that could explain the lack of any trends between the various samples but the primary one would have to be the varying weather during sample collections. Wind and precipitation were not recorded during sampling periods but may have been a factor in varying anion concentration. Table 14 shows the qualitative anion concentrations while figure 61 shows the data normalized to sulfate.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Fluoride</th>
<th>Chloride</th>
<th>Bromide</th>
<th>Nitrate</th>
<th>Phosphate</th>
<th>Sulfate</th>
</tr>
</thead>
<tbody>
<tr>
<td>30 Day</td>
<td>0.269</td>
<td>4.481</td>
<td>ND</td>
<td>9.518</td>
<td>ND</td>
<td>2.310</td>
</tr>
<tr>
<td>60 Day</td>
<td>ND</td>
<td>1.687</td>
<td>ND</td>
<td>2.111</td>
<td>ND</td>
<td>4.894</td>
</tr>
<tr>
<td>100 Day</td>
<td>0.137</td>
<td>1.417</td>
<td>1.591</td>
<td>8.462</td>
<td>ND</td>
<td>6.260</td>
</tr>
</tbody>
</table>

Table 14: UNLV Roof Wipes Qualitative Anion Concentrations (ppm)
High volume air sampling was performed on the roof of the UNLV’s chemistry building at an approximate rate of 600 L/min. That brings the total volumes sampled for 5 hours, 8 hours, and 24 hours to approximately 180k m$^3$, 280k m$^3$, and 864k m$^3$ respectively. As stated previously a ~2cm piece of filter was used for anion extraction. The qualitative anion concentrations (Table 15) as well as normalized data are presented below in figures 62 and 63.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Fluoride</th>
<th>Chloride</th>
<th>Bromide</th>
<th>Nitrate</th>
<th>Phosphate</th>
<th>Sulfate</th>
</tr>
</thead>
<tbody>
<tr>
<td>High Volume 5hr</td>
<td>0.161</td>
<td>1.873</td>
<td>ND</td>
<td>6.436</td>
<td>ND</td>
<td>6.130</td>
</tr>
<tr>
<td>High Volume 8hr</td>
<td>0.109</td>
<td>2.745</td>
<td>1.558</td>
<td>13.354</td>
<td>1.672</td>
<td>18.542</td>
</tr>
<tr>
<td>High Volume 24hr</td>
<td>0.952</td>
<td>6.397</td>
<td>1.660</td>
<td>41.677</td>
<td>1.672</td>
<td>48.954</td>
</tr>
</tbody>
</table>

Table 15: UNLV Roof High Volume Anion Concentration (ppm)
The panel wipes collected at the City of Las Vegas solar facility showed similar anion concentrations with slight variations between the two different solar panels, Solar Panel A and Solar Panel B. The topsoil and the particulates that were vacuumed off the panel had similar anion concentrations in fluoride, chloride, and nitrate. These samples also
had a different sulfate to nitrate ratio than the panel wipes suggesting that nitrate deposition may be from the atmosphere. The measured anion concentrations can be seen in Table 16.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Fluoride</th>
<th>Chloride</th>
<th>Bromide</th>
<th>Nitrate</th>
<th>Phosphate</th>
<th>Sulfate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solar Panel B 03/22/16</td>
<td>0.020</td>
<td>2.424</td>
<td>ND</td>
<td>4.184</td>
<td>ND</td>
<td>3.824</td>
</tr>
<tr>
<td>Solar Panel A 03/22/16</td>
<td>0.019</td>
<td>3.044</td>
<td>ND</td>
<td>5.621</td>
<td>ND</td>
<td>6.289</td>
</tr>
<tr>
<td>Solar Panel B 05/28/16</td>
<td>ND</td>
<td>2.161</td>
<td>ND</td>
<td>5.171</td>
<td>1.444</td>
<td>5.946</td>
</tr>
<tr>
<td>Solar Panel A 05/28/16</td>
<td>ND</td>
<td>2.369</td>
<td>ND</td>
<td>6.182</td>
<td>1.461</td>
<td>7.163</td>
</tr>
<tr>
<td>City of Las Vegas Ground</td>
<td>ND</td>
<td>9.029</td>
<td>ND</td>
<td>1.708</td>
<td>ND</td>
<td>157.069</td>
</tr>
<tr>
<td>Panel Vacuum City LV</td>
<td>ND</td>
<td>9.477</td>
<td>1.835</td>
<td>1.736</td>
<td>1.825</td>
<td>174.675</td>
</tr>
</tbody>
</table>

Table 16: City of Las Vegas Anion Sample Concentrations (ppm)

The solar panel A and B samples from the same date have the same relative anion concentrations which is illustrated better when normalized to sulfate as shown in figures 64 and 65. It is interesting to note the appearance of phosphate in the 05/28/2016 samples. This could possibly be attributed to a nearby park lawn that may have been fertilized since the sampling date of 03/15/2016. There was no apparent correlation between the ground and vacuum sample to that of the panel wipes. Sulfate and chloride levels were significantly higher in the vacuum and ground samples while the nitrate levels were lower. Nitrate levels on the panels may originate from atmospheric deposition and could originate from air pollution but further studies would be needed to make that determination. The high sulfate levels in the vacuum could be attributed to the amount of sample used for the particulate collection, 250mg, as opposed to the ~2cm wipe for the panels.
The samples collected at Copper Mountain 1 solar facility showed similar anion concentrations with slight variations between the different sections of solar panels. The topsoil within in the solar panel field, CM Topsoil 10MW, and the topsoil from near the plant, CM Topsoil
Near Tower, showed different anion concentrations. A summary of the measured anion concentrations can be seen in the Table 17.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Fluoride</th>
<th>Chloride</th>
<th>Bromide</th>
<th>Nitrate</th>
<th>Phosphate</th>
<th>Sulfate</th>
</tr>
</thead>
<tbody>
<tr>
<td>CM REF Panel</td>
<td>0.077</td>
<td>1.381</td>
<td>1.584</td>
<td>3.804</td>
<td>ND</td>
<td>2.900</td>
</tr>
<tr>
<td>CM 7yr</td>
<td>0.109</td>
<td>3.107</td>
<td>1.582</td>
<td>8.272</td>
<td>ND</td>
<td>5.820</td>
</tr>
<tr>
<td>CM 9yr</td>
<td>0.142</td>
<td>1.506</td>
<td>1.929</td>
<td>4.423</td>
<td>ND</td>
<td>3.000</td>
</tr>
<tr>
<td>CM Topsoil 10MW</td>
<td>ND</td>
<td>3.117</td>
<td>1.601</td>
<td>3.336</td>
<td>ND</td>
<td>2.108</td>
</tr>
<tr>
<td>CM Topsoil Near Tower</td>
<td>0.030</td>
<td>0.785</td>
<td>1.608</td>
<td>ND</td>
<td>ND</td>
<td>7.374</td>
</tr>
</tbody>
</table>

Table 17: Copper Mountain 1 Anion Sample Concentration (ppm)

Normalization to sulfate reveals similar anions distributions between the different panels in terms of nitrate, phosphate, and fluoride. Variation arises in chloride and bromide from the two topsoil samples as shown in Figure 66. Phosphate was not detected at this location.

The topsoil sampled from within the panel field and the topsoil from near the tower (~800m away) are different in terms of normalized distribution. However, their measured bromide levels are very similar despite the differences seen in the normalized data (Figure 67).
The 3 different sections of panels all seem to have similar anion distributions when normalized as shown below in figure 69. The copper mountain 7-year panel has a higher chloride, nitrate, and sulfate concentration than the other samples as shown in figure 70.
Figure 69: Copper Mountain 1 Panels (Normalized)

Figure 70: Copper Mountain 1 Panel Wipes Measured Concentration (ppm)
4.4 Metal Composition

4.3.2 Analysis by ICP-AES

Metal determination by Inductively Coupled Plasma Atomic Emission spectroscopy is a standard analysis for medium to high concentration (5ppb to 10ppm) metals identification in environmental monitoring of soils. The metal species selected were aluminum, calcium, iron, potassium, magnesium, sodium, phosphorus, and sulfur. Calcium and magnesium were the dominant target species being the most abundant in common minerals found in soils. The other metals were also selected because they were in the same standard calibration solution. The data presented is meant to provide an overview of metals’ trends and distributions throughout the various matrices. The various metal concentrations all have been normalized to the calcium concentration and are graphically presented below. The non-normalized and the normalized distribution concentration data were organized based on sample location or sampling surface (i.e solar panels, vugs, etc.) and are presented below. The concentrations are presented in milligram per gram (mg/g) for the non-normalized data and a summary is presented in Table 18.
Table 18: Metals Concentrations (mg/g)

The samples collected from the City of Las Vegas Solar Facility correlated with the previous results in that there was little variation in composition between the two different types of panels examined as shown in Figure 71.

Composition from the ground and particulate vacuumed off the panels have slightly varying concentrations as seen in Figure 72. The variation in
the three samples could result from panel height, orientation and depth of top soil sampled. When the data is normalized to calcium (Figure 73) the distributions of each are almost identical with the exception of sulfur on panel B. The lack of correlation between the ICP’s sulfur concentration and the IC’s sulfate concentration is noted. A possible explanation is fractionation and non-homogenous sampling before digestions causing an uneven distribution of particles.

Figure 72: City of Las Vegas Ground and Vacuum Samples (mg/g)
Comparison of the normalized data highlights that the panel wipes have a higher relative abundance of other metals compared to the topsoil and vacuumed sample, with the exception of magnesium, as shown in Figure 74.

The Copper Mountain samples had varying concentrations ranging from the reference panel being the lowest and the 7-year panel being the
highest as seen below in Figure 75. Because the reference panel is regularly cleaned, it is understandable that it has the lowest concentration of metals. It is unclear on why the newer section (~7 years) would have a higher metals concentration than the older section (~9 years). A few possibilities could be that there is higher maintenance vehicle traffic near the newer section or that the older section was cleaned after construction of the newer section.

![Copper Mountain 1 Panel Wipes](Image)

**Figure 75: Copper Mountain 1 Panels (mg/g)**

Normalization of these samples to calcium reveals that other metals are relatively higher in the older section while the reference panel remains the lowest, with the exception of sodium as shown below in Figure 76.
The southwest desert samples, Figure 77, were collected from various locations around Southern Nevada. The sample Vug 2 was collected in the southwest part of the Las Vegas valley while dust sample 2 was collected from topsoil along state route 160, roughly 10km from Vug 2. Dust sample 6 was collected northwest of Las Vegas in Pahrump, NV, roughly 68km away. When Vug 2 and dust sample 6 are normalized to calcium both have very similar distribution (Figure 78), probably due to the fact that both of these sample are from suspended particulate collectors. These are collectors where dust can deposit and not be readily removed by weather events while the dust sample 6 was collected from the topsoil. Dust sample 2, a topsoil sample, had almost triple the amount of calcium compared to the Vug 2 and dust sample 6.
A comparison of all solar panel wipe samples collected shows similar sodium, potassium, and sulfur levels with other metals varying significantly as shown below in Figure 79.
The 30- and 100-day direct collect samples from UNLV’s roof showed similarities in iron, sulfur, and magnesium with significant differences in the remaining metals are presented below in Figures 80 and 81.

Figure 80: UNLV Roof mg/g
Figure 81: UNLV Roof (Normalized)
CHAPTER 5
CONCLUSIONS

The objective for utilization of Raman microspectroscopy was to determine the mineral composition of the suspended particulate as well as to possibly demonstrate long distance transport. In this study, many common local minerals were identified that included: dolomite, carbonate, rutile, quartz, gypsum, rutile, and anatase. Raman microspectroscopy also revealed the presence of a graphitic-like substance coating many of the particles. This resulted in the observation of mixed spectra involving the graphitic-like substance and strong Raman scattering minerals. We attempted to remove the organic coating by treatment with UV radiation and H₂O₂ but were unsuccessful. The graphitic spectrum is thought to result from sp-2 and sp-3 hybridized carbon bonds stacking non-uniformly which resulted in dispersion and formed a broad spectra around 1600 cm⁻¹ (Ferrari & Robertson, 2001). This spectrum differs from pure graphite which has an intense peak at 1579 cm⁻¹ and secondary peak at 1349 cm⁻¹. The graphitic-like spectra that coated particles accounted for the majority of the measurable spectra found in nearly every sample. The carbonates (mainly dolomite and calcite) were the second most prevalent minerals identified and this correlated with ICP-AES measurements which showed abundant calcium and magnesium. There was no correlation between mineral abundance and sampling time for any of the minerals or for the total particle count. This could be attributed to the many variables that contribute to surface accumulation and surface loss over a long-term exposure.
Pyrolysis GC-MS indicated organic matter in the dust reflected organic constituents in the nearby soil. This was shown by comparisons of each sample’s TIC with results from nearby soil and was the main supporting evidence for the assumption that the main contributor of the dust on the panels is the nearby topsoil. Targeted ion chromatograms (57, 74, 101, 129, and 165) further supported this correlation with the nearby topsoil’s organic material being consistent with the common ions found in organic constituents of plant fragments. Organic matter may also contribute to particle adhesions to solar surfaces but further research is needed to corroborate this assumption. Additional studies will also provide more insight into whether the practice of using plants to anchor the nearby soil should be utilized or not. The data also allowed for the rural versus urban particulate matter on panels to be compared and differentiated. The urban samples had a higher pyrolysis signal and increased number of compounds present. Differing rural compositions were also highlighted in the Vug 2 and 3 samples showing that organic compositions will vary significantly between samples that are relatively close geographically (222km).

The utilization of ion chromatography demonstrated further correlation between the composition of the nearby topsoil and particulates on solar panels. This correlation as seen in the panel vacuum samples and the topsoil sample from the City of Las Vegas solar facility. A strong correlation was not observed between the topsoil sample and the panel wipe samples but this may be attributed to sample size that was used for digestion as well as analytic loading on the wipe. The wipe samples were primarily ultra-fine particulates. The solar panel
A and B wipes were very similar in composition and both had a similar level of phosphate which appeared in the second sampling on 05/28/2016. This is another example of the many variables that are present when performing sampling in an uncontrolled environment. The Copper Mountain 1 panel samples were similar in composition with the 7-year section being slightly higher than the 9-year section and reference panel. It is interesting to note that the topsoil sample taken within the Copper Mountain 1’s 10 MW section was significantly different in composition than a sample taken outside of the array, roughly 800m away.

The utilization of ICP-AES for metals identification assisted in showing correlation between samples and is consistent with the Raman mineral data. The metals analysis data showed high calcium and magnesium concentrations which correlates with the large number of carbonate, mainly calcite and dolomite, particles that were observed in the Raman data. The City of Las Vegas solar facility panel wipes were nearly identical in metals composition and have some correlation with the ground and panel vacuum samples. The Copper Mountain 1 panel samples were similar in composition with the 7-year section sample again being the outlier. Normalization of all the samples from that location didn’t reveal any strong correlation. The Vug 2 sample and dust sample 6 had nearly identical metals distribution despite being sampled 68km apart and the Spring Mountain range. A sample collected 10km away from Vug 2, located near a highway was significantly different in composition and concentration. The two UNLV roof top samples that were measured had significant differences and no correlation was found between the two samples.
The methods utilized in this study aimed to give a broad overview of composition. These measurements are the initial step towards a more complete characterization of particulate matter deposition on solar surfaces. The characterization data presented here can be utilized in determining the planning and operation costs of building large-scale solar facilities. This research could also be utilized by those interested in creating coating materials to help mitigate the effects of particulate matter on solar panels. This data could also be used to support environmental models that examine the effects of panel washing on the local ecology. Future work will include a more detailed and controlled sampling of solar facilities as well as monitoring weather patterns and their effects on panel soiling. Characterizing the surfaces of old used panels by the methods mentioned above as well as scanning electron microscopy should also be explored.
REFERENCES


APPENDIX A: RAMAN MICROSCOPY DATA

Tacky-dot 10 Day

Region 1

Diameter 244.7 µm

Number of particles 6

No Signal 1(13.0) 2(45.6) 3(14.2) 5(17.8) 6(11.1)

Carbonate and Quartz 4(20.2)

Region 2
Diameter 243.3 µm

Number of particles 12

No Signal 1(9.2), 3(6.1), 4(7.2), 5(4.5), 6(10.8), 8(9.0), 9(6.6), 10(9.2), 11(11.4), 12(6.6)

Carbonate 2(32.4), 7(36.0),

Region 3

Diameter 242.9 µm

Number of particles 8
No signal 1 (20.7), 3 (12.8), 4 (10.5), 5 (17.1), 6 (9.6)

Carbonate 2 (20.9), 8 (25.1)

Graphitic-like and Carbonate 7 (35.4)

Region 4
Diameter 250.1 µm

Number of particles 7

No Signal 1(28.4), 2(10.6), 3(21.4), 4(55.9), 5(13.2)

Graphitic-like and Carbonate 6(52.4),
Carbonate and Quartz 7(15.3)

Region 5

Diameter 244.0 µm

Number of particles 6

No Signal 1(14.0), 6(23.9)

Graphitic-like 2(17.8)
Graphitic-like and Carbonate 3(40.0)

Carbonate 4(27.5), 5(34.0)

Region 6

Diameter 236.6 µm

Number of particles 5
No Signal 1(19.8), 3(10.2), 4(30.5), 5(14.1)

Graphitic-like 2(38.5)

Region 7

Diameter 245.7 µm

Number of particles 9
No signal 1(9.8), 2(28.0), 3(10.2), 4(17.3), 5(12.2), 7(13.4), 8(13.4), 9(14.9)

Graphitic-like 6(51.2)

Region 8

Diameter 238.9 µm

Number of particles 7
No Signal 1(32.2), 2(28.2), 5(23.9), 6(7.8), 7(4.9)

Graphitic-like 3(30.1)

Carbonate 4(40.9)

Region 9
Diameter 253.9 µm

Number of particles 3

No signal 1(32.6), 2(45.5),

Carbonate 3(38.4)

Region 10

Diameter 244.9 µm

Number of particles 6
No signal 5(13.7), 6(113.1)

Graphitic-like and Carbonate 1(18.9)

Carbonate 3(23.7)

Graphitic-like 2(18.5), 4(33.9)
**Tacky-dot 20 Day**

Region 1

Diameter 232.9 µm

Number of particles 19

No signal

1(43.3), 2(14.2), 3(10.0), 4(10.1), 5(29.0), 6(9.9), 7(7.4), 8(15.3), 9(9.7), 10(8.1), 11(12.3), 13(9.3), 14(43.4), 15(16.8), 16(13.8), 17(8.0), 19(8.5)

Graphitic-like 12(14.2)
Graphitic-like and Carbonate 18(20.7)

Region 2

Diameter 233.0 µm

Number of particles 16

No signal

1(12.9), 2(5.4), 3(8.1), 4(4.8), 5(5.7), 6(7.7), 7(23.2), 8(6.0), 9(7.7), 10(6.2), 11(5.4), 12(11.1), 13(36.2), 14(4.9), 15(12.7), 16(4.9)
Region 3

Diameter 231.3 µm

Number of particles 5

No signal 1(14.6), 3(38.8), 4(15.1), 5(8.0)

Carbonate 2(58.6)

Region 4

Diameter 230.5 µm
Number of particles 7

No signal 1(8.6),2(5.5),3(8.1),4(26.9),7(41.0)

Carbonate 5(14.9)

Quartz 6(39.7)
Region 5

Diameter 229.6 µm

Number of particles 18

No signal

1(16.1), 2(24.2), 3(12.7), 4(10.9), 5(31.9), 6(13.6), 7(12.3), 10(18.7), 11(10.1), 12(29.3), 13(6.1), 14(16.9), 15(7.4), 16(7.8), 18(6.7)

Quartz 8(59.9)
Graphitic-like 9(19.4),17(21.4)

Region 6

Diameter 232.5 µm

Number of particles 12

No Signal 2(12.7),3(11.4),4(26.9),5(8.5),7(11.4),8(7.1),
9(27.9),10(7.6),12(10.4)
Carbonate 1\( (11.8) \)

Graphitic-like 6\( (22.3) \), 11\( (13.0) \)

Region 7

Diameter 239.6 µm

Number of particles 21
No Signal

1(7.2), 2(8.3), 3(6.5), 4(14.3), 5(12.3), 6(13.5), 7(7.8), 8(7.6), 9(6.0), 10(12.0), 11(13.3), 13(10.6), 14(43.7), 15(13.4), 16(13.), 17(9.4), 19(7.4), 20(6.2), 21(9.3)

Graphitic-like 12(14.9), 18(20.5)

Region 8

Diameter 233.2 µm

Number of particles 9
No Signal  2(7.0), 3(40.2), 5(34.4), 6(12.4), 7(17.6), 8(13.4)

Graphitic-like  1(23.2), 9(10.7)

Graphitic-like and Quartz  4(24.2)

Region 9
Diameter 232.5 µm

Number of particles 13

No signal 1(8.9), 2(11.7), 3(9.2), 4(6.5), 5(3.9), 6(8.1), 8(8.3), 9(9.5), 10(13.4), 11(7.8), 12(11.9), 13(9.9)

Graphitic-like and Quartz 7(161.2)

Region 10

Diameter 231.9 µm

Number of particles 12
No Signal

1(14.2), 2(47.8), 3(15.2), 4(11.0), 5(27.5), 6(27.5), 7(8.3), 8(8.9), 9(10.6),
10(12.6), 11(48.7)

Graphitic-like 12(23.4)

**Tacky-dot 30 Day**

Region 1

Diameter 247.2 µm

Number of particles 22
No signal 1(11.6), 2(7.7), 3(33.2), 4(20.7), 5(10.0), 7(6.1), 8(7.1), 10(44.1), 11(6.7), 12(12.8), 13(11.9), 15(15.4), 16(7.6), 17(5.8), 18(57.7), 19(40.9), 20(33.0), 21(8.9), 22(8.0),

Graphitic-like 6(54.4), 9(16.4), 14(41.8),

Region 2

Diameter 245.7 µm

Number of particles 34
No Signal 1(11.8), 3(10.7), 5(22.3), 6(37.2), 10(22.4), 11(34.3), 12(25.5), 13(7.0), 14(8.4), 15(15.7), 16(9.6), 17(9.5), 18(7.1), 19(18.6), 20(35.8), 21(6.0), 22(11.8), 24(28.2), 25(7.2), 26(6.4), 27(10.1), 30(12.2), 31(11.5), 32(5.4), 33(6.3),

Quartz 2(11.7),
Graphitic-like and Carbonate 4(26.2),34(20.5)

Graphitic-like and Carbonate 7(28.5)

Graphitic-like and Quartz 8(32.3)

Graphitic-like 9(23.8),23(6.7)

Graphitic-like and Carbonate 28(39.2)
Anatase 29(35.1)

Region 3

Diameter 247.6 µm

Number of particles 40

No Signal

2(16.1), 3(14.9), 4(11.2), 5(7.2), 6(8.3), 9(12.0), 10(22.0), 11(12.7), 12(25.2), 13(16.9), 14(5.4), 15(7.8), 16(56.7), 17(6.6), 18(10.2), 19(8.3), 20(4.6), 21(6.3), 22(16.9), 23(4.7), 24(4.3), 25(5.6), 26(11.0), 28(11.5), 29(7.7), 30(17.3), 31(33.9), 33(5.6), 34(7.4), 36(14.1), 37(9.9), 39(9.7), 40(6.2)
Graphitic-like 1(30.2), 8(26.1),

Anatase 7(27.4),

Quartz 27(34.1), 38(30.9)

Carbonate 32(32.9),

Graphitic-like and Carbonate 35(19.2)
Region 4
Diameter 241.5 µm
Number of particles 20

No Signal 1(7.7), 2(29.4), 3(23.8), 6(10.7), 8(25.9), 10(13.3), 11(28.8), 12(11.4), 15(16.9), 16(9.3), 17(18.9), 18(7.2), 19(6.3), 20(11.3)

Graphitic-like 5(33.8), 7(48.1),

Graphitic-like and Carbonate 3(20.1), 14(28.2)
Carbonate 9(30.6), 13(26.7)

Region 5

Diameter 248.1 µm

Number of particles 33

No signal

1(6.7), 2(14.0), 3(6.1), 4(11.5), 5(7.2), 7(26.5), 10(7.6), 11(18.1), 12(12.5), 13(17.0), 14(10.2), 15(21.1), 17(28.4), 18(13.3), 19(7.9), 20(26.7), 21(23.2), 22(12.2), 23(15.6), 26(22.1), 27(9.1), 28(13.1), 29(29.9), 30(10.0), 31(33.1), 32(13.3), 33(22.5),
Graphitic-like 16(9.1), 24(20.1),

Carbonate 6(24.9), 9(24.4)

Quartz 8(35.3)

Unidentified 25(74.4),

Region 6

Diameter 217.0 µm

Number of particles 21
No Signal

1(17.6), 2(13.5), 3(41.1), 4(20.4), 5(14.7), 6(30.8), 7(28.3), 8(20.8), 9(28.4), 11(26.9), 12(14.4), 15(23.7), 16(17.8), 17(18.5), 18(24.0), 20(41.0),

Graphitic-like 10(33.1), 14(69.4), 21(15.8)

Carbonate 13(17.3), 19(45.4)

Region 7

Diameter 246.3 µm

Number of particles 80
No Signal

3(11.3), 4(9.8), 5(6.3), 7(18.6), 9(14.3), 10(12.4), 14(19.8), 16(25.2), 17(20.3), 19(20.2), 22(29.8), 23(18.2), 24(25.1), 25(24.5), 26(5.1), 27(7.6), 28(7.1), 29(7.0), 30(12.5), 31(16.5), 32(14.3), 33(15.9), 34(15.7), 35(11.5), 36(8.8), 37(18.4), 38(15.9), 39(11.4), 40(8.9), 41(5.6), 42(9.0), 43(9.8), 45(11.9), 46(18.1), 47(10.8), 49(8.3), 50(7.3), 53(11.2), 55(22.0), 58(15.6), 59(14.9), 60(13.7), 61(18.4), 62(24.3), 63(14.9), 64(21.4), 65(22.3), 66(15.2), 67(13.0), 68(12.8), 69(16.4), 70(8.1), 71(10.8), 72(13.2), 73(23.3), 74(17.3), 75(16.6), 76(14.7), 77(6.4), 79(30.0)

Graphitic-like

2(20.6), 8(34.6), 12(24.1), 13(22.3), 15(37.0), 20(30.5), 44(14.5), 48(18.4), 51(8.9), 52(7.5), 54(11.6), 56(13.6), 57(14.2), 78(30.7),
Carbonate 1(34.1), 6(18.1), 18(25.7),

Carbonate 11(18.0), 21(31.9), 80(28.6)

Region 8

Diameter 231.9 µm

Number of particles 63
No signal

1(20.9), 2(5.6), 3(7.1), 4(7.8), 5(11.0), 6(8.7), 10(7.1), 12(27.0), 13(16.9), 14(22.3), 16(22.1), 17(17.7), 18(10.0), 19(6.9), 25(36.6), 26(9.1), 27(7.9), 28(16.0), 29(4.4), 30(11.7), 31(13.8), 32(11.6), 33(12.6), 34(12.7), 35(9.7), 37(9.0), 38(14.4), 39(7.2), 40(14.9), 41(13.7), 42(7.8), 43(10.7), 44(14.2), 46(7.0), 47(8.9), 48(10.5), 49(8.4), 50(18.2), 51(14.7), 53(21.7), 55(7.9), 56(7.5), 57(15.4), 58(11.0), 59(16.0), 61(12.4), 62(21.7), 63(13.9)

Graphitic-like 7(7.3), 9(15.5), 11(17.6), 22(32.6), 24(24.9), 45(12.7),

Quartz 8(17.1),
Carbonate 15(18.6), 22(16.5),

Graphitic-like and Carbonate 20(14.7)

Graphitic-like and Carbonate 23(19.8),

Carbonate 36(13.3), 52(12.3), 60(14.4),
Graphitic-like and Anatase 54(33.6)

Region 9

Diameter 246.4 µm

Number of particles 20

No Signal

1 (21.6), 3 (20.2), 4 (29.7), 5 (16.1), 6 (41.9), 7 (45.3), 8 (9.1), 9 (21.3), 10 (9.6), 11 (10.9), 13 (7.9), 15 (38.4), 16 (14.0), 17 (13.9), 20 (8.0)
Carbonate 2(42.4)

Carbonate 18(25.2), 19(16.1)

Graphitic-like 12(20.5), 14(13.2)

Region 10

Diameter 245.7 µm

Number of particles 33
No Signal
1(10.5), 2(15.4), 3(11.9), 7(32.1), 8(15.7), 9(13.2), 10(27.8), 11(14.0), 12(14.7), 13(21.7), 18(19.2), 19(16.8), 20(11.1), 21(11.4), 22(8.2), 23(29.2), 24(17.0), 25(17.4), 27(7.4), 28(14.6), 29(31.5), 30(16.0), 31(15.0), 32(8.3), 33(13.5)

Graphitic-like and Carbonate 4 (16.9), 5(12.8), 6(29.2),

Graphitic-like 14(16.1), 15(11.8), 16(19.7), 17(27.7), 26(58.2)

Tacky-dot 60 Day
Region 1

Diameter 248.5 µm

Number of particles 46

No Signal 1(33.8), 2(25.2), 3(19.1), 4(16.7), 5(31.3) slight signal, 7(8.4), 8(7.2), 10(7.6), 11(11.4), 12(15.7), 13(11.1), 14(10.9), 15(9.6), 16(9.8), 17(8.2), 18(9.3), 19(5.6), 20(6.7), 21(8.2), 22(11.6), 24(9.6), 25(6.0), 26(6.6), 28(11.7), 29(16.7), 30(10.1), 32(9.2), 33(10.9), 34(23.9), 35(6.6), 36(5.0), 37(6.4), 39(11.0), 41(12.8), 42(9.7), 43(16.0), 44(13.9), 45(9.6)

Graphitic-like 31(11.1), 40(19.1),
Orthoclase 6(19.5),

Carbonate 9(21.1), 45(12.0),

Carbonate and Graphitic-like 23(23.3),

Carbonate 27(14.2),
Unidentified 38 (34.6)

Region 2

Diameter 231.9 µm

Number of particles 33

No Signal

2 (18.3), 3 (12.4), 4 (19.1), 5 (20.6), 6 (18.0), 7 (28.4), 8 (5.1), 9 (7.8), 10 (16.0), 11 (14.0), 13 (21.3), 14 (11.0), 16 (31.8), 18 (35.5), 19 (21.7), 21 (13.5), 22 (15.5), 23 (26.1), 24 (18.5), 25 (13.8), 26 (12.3), 27 (13.2), 29 (44.7), 30 (35.6), 31 (39.6), 32 (25.2), 33 (9.5), 125
Graphitic-like 1 (28.1), 17 (28.9), 20 (29.7), 28 (24.5),

Anatase 12 (12.7), 15 (8.9)

Region 3

Diameter 248.0 µm

Number of particles 21

No Signal

1 (15.1), 2 (17.1), 3 (10.0), 4 (22.0), 6 (11.3), 7 (8.8), 8 (11.7), 9 (12.8), 10 (13.0), 14 (6.6), 15 (5.5), 16 (6.7), 18 (6.8), 19 (26.2), 20 (5.5)
Graphitic-like 11(25.2), 17(21.7),

Carbonate 5(20.7)

Region 4

Diameter 251.0 µm

Number of particles 29

No Signal

1(5.9), 2(8.7), 4(10.8), 7(70.3), 8(23.9), 9(18.5), 12(7.5), 14(9.2), 15(10.9), 17(8.6), 19(8.6), 20(8.9), 21(3.9), 22(5.0), 23(4.7), 24(8.0), 25(8.4), 26(6.7), 27(5.5), 28(4.1), 28(7.0)
Graphitic-like $6(21.6), 10(11.2), 11(13.1), 18(6.6)$,

Carbonate $3(11.7), 5(14.1)$,

Anatase $23(6.1)$

Graphitic-like and Carbonate $16(20.5)$

Region 5

Diameter 248.3 µm

Number of particles 39

128
No Signal

1(13.7), 2(16.2), 3(17.0), 4(9.1), 5(9.5), 6(23.8), 7(13.4), 8(21.6), 9(14.4),
10(8.5), 11(10.8), 12(8.0), 14(12.9), 17(9.0), 18(25.3), 19(13.6), 20(9.6), 21
(14.0), 23(33.1), 24(24.9), 25(15.1), 26(32.6), 27(27.1), 28(31.0), 29(23.8),
31(4.5), 32(38.2), 33(11.7), 34(8.6), 35(15.2), 36(21.9), 37(17.4), 38(9.6), 3
9(12.3)

Graphitic-like 15(14.0), 16(27.2), 30(37.9),

Anatase 13(19.6), 22(7.7), 40(12.5)

Region 6

129
Diameter 246.0 µm

Number of particles 33

No Signal
2(14.8), 3(28.5), 4(20.6), 5(13.9), 6(19.8), 7(27.0), 8(19.4), 9(14.3), 10(18.0), 11(17.1), 13(10.3), 15(7.7), 20(13.6), 21(13.0), 22(8.9), 24(11.2), 25(7.9), 31(6.1), 32(9.4),

Graphitic-like
1(43.9), 11(10.0), 14(6.8), 16(15.4), 17(13.3), 18(23.4), 19(46.4), 23(10.7), 26(18.4), 28(8.2), 30(7.0),
Anatase 33(2.7),

Quartz 27(16.4), 29(18.2),

Region 7

Diameter 247.4 µm

Number of particles 13

No Signal

1(12.2), 2(6.8), 3(21.5), 4(14.6), 5(8.8), 6(7.9), 7(13.6), 8(15.1), 9(14.0), 10(5.0), 13(20.4)
Graphitic-like 10(21.4),

Graphitic-like and Carbonate 11(24.7),

Region 8

Diameter 241.7 µm

Number of particles 14

No Signal

2(13.2), 3(6.8), 4(14.1), 5(19.7), 6(10.1), 7(10.9), 8(26.1), 10(11.3), 11(19.1), 12(50.1), 14(25.3),
Graphitic-like 1 (24.6),

Carbonate 9 (24.0)

Carbonate 13 (10.4),

Region 9

Diameter 252.4 µm

Number of particles 58
No Signal

1 (13.9), 2 (10.4), 3 (10.6), 4 (6.2), 5 (11.7), 6 (22.3), 7 (8.6), 8 (8.4), 9 (7.5), 10 (7.4), 11 (10.7), 12 (8.4), 13 (18.7), 14 (9.6), 15 (9.9), 16 (16.8), 17 (8.4), 18 (12.1), 22 (18.3), 24 (17.5), 26 (7.4), 28 (8.5), 29 (9.2), 30 (5.2), 31 (11.8), 32 (13.7), 33 (9.7), 34 (28.2), 35 (12.7), 36 (14.6), 37 (7.3), 38 (6.6), 39 (17.5), 40 (25.5), 41 (7.9), 42 (18.8), 43 (11.0), 44 (18.3), 45 (16.6), 46 (16.0), 47 (17.1), 48 (8.9), 49 (11.0), 50 (12.0), 51 (15.7), 52 (21.8), 53 (11.1), 55 (12.9), 56 (9.9), 57 (18.2), 58 (8.9)

Graphitic-like 19 (13.5), 20 (14.1), 21 (15.3), 23 (27.7), 25 (16.8), 54 (31.0),

Carbonate 27 (20.4),
Region 10

Diameter 245.6 µm

Number of particles 26

No Signal

2(15.4), 5(27.8), 6(19.8), 8(17.6), 9(28.3), 10(25.0), 11(12.8), 13(16.8), 14(14.1), 15(13.4), 16(17.0), 17(21.8), 22(12.1), 23(60.2), 25(21.7),

Graphitic-like 1(22.5), 3(27.0), 7(18.3), 12(58.7), 19(11.1), 20(9.4), 27(18.3)

Carbonate 4(21.7),
Carbonate 18(28.0), 21(27.0), 24(24.9)

Quartz 26(38.2), 12 (Calcium and Anatase and Graphitic-like present in different regions but overall amorphous).

**Tacky Dot Slide 100 Day**

Region 1

Diameter 239.5

Number of particles 30
No Signal

1(17.0), 2(11.8), 3(16.7), 4(9.2), 5(8.7), 6(5.7), 7(12.1), 8(14.8), 9(8.7), 10(41.7), 13(19.7), 14(21.8), 15(18.7), 16(16.1), 17(32.6), 18(8.0), 19(9.2), 20(15.2), 21(9.1), 22(12.3), 23(15.6), 24(9.8), 25(11.3), 26(10.9), 27(36.4), 28(7.2), 29(8.7), 30(8.2),

Amorphous Carbon 11(44.7), 12(36.5),

Region 2

Diameter 232.8

Number of particles 24
No signal

1 (17.2), 2 (13.3), 3 (11.2), 4 (8.4), 5 (17.3), 7 (22.0), 8 (16.4), 9 (19.7), 10 (14.7), 12 (10.3), 13 (12.5), 14 (10.2), 15 (9.7), 16 (30.4), 17 (15.9), 18 (11.7), 19 (7.2), 20 (22.9), 22 (12.2), 23 (8.9), 24 (10.6),

Amorphous Carbon 6 (21.4), 21 (9.6),

Rutile 11 (21.3)

Region 3

Diameter 233.6

138
Number of particles 22 (skipped #9)

No Signal

1(22.4), 2(12.0), 3(16.2), 4(11.5), 5(7.5), 6(10.7), 7(16.1), 8(9.9), 10(15.3),
11(18.0), 12(7.5), 13(8.9), 14(12.1), 15(11.2), 16(16.4), 17(24.9), 18(8.2),
19(22.6), 20(10.7), 21(14.6), 22(18.8), 23(18.0)

Amorphous Carbon

Region 4

Diameter 230.4

Number of particles 37
No Signal

2 (25.6), 3 (28.6), 4 (16.3), 5 (18.3), 6 (11.7), 7 (9.3), 8 (14.5), 9 (13.8), 10 (9.6), 11 (10.0), 12 (11.2), 13 (17.3), 14 (20.2), 15 (16.2), 16 (10.6), 17 (17.8), 19 (16.3), 20 (8.4), 21 (10.6), 22 (6.9), 22 (13.2), 24 (22.0), 25 (14.7), 26 (15.8), 27 (21.4), 28 (21.3), 29 (20.8), 30 (8.0), 31 (11.4), 32 (7.3), 33 (25.6), 34 (20.3), 35 (15.3), 36 (11.1), 37 (19.1)

Amorphous Carbon 1 (24.7), 18 (22.3),

Region 5

Diameter 233.5 um

Number of particles 39
No Signal

1(10.5), 2(20.4), 3(31.3), 4(16.8), 5(9.9), 6(11.6), 7(10.4), 8(11.4), 12(9.9),
13(11.1), 14(16.0), 15(14.5), 16(11.5), 17(11.6), 18(9.0), 19(17.9), 20(13.2),
21(10.4), 22(16.7), 23(13.4), 24(15.9), 26(16.2), 27(10.5), 28(15.9), 30(14.0),
31(12.7), 32(11.5), 33(10.7), 34(9.0), 35(12.5), 36(12.0), 38(13.0), 39(17.4),

Amorphous Carbon 9(10.0), 10(9.8), 11(9.0), 25(14.9), 29(15.3),

Anatase 37(18.1),
Region 6

Diameter 231.7

Number of particles 14

No Signal

1 (13.3), 2 (28.7), 3 (12.9), 4 (7.1), 5 (18.4), 6 (7.6), 7 (9.9), 8 (14.9), 9 (10.8), 10 (18.7), 11 (11.2), 12 (25.0), 13 (12.3), 14 (8.1)

Region 7

Diameter 229.7

Number of particles 50
No Signal

\(2(9.8), 3(13.2), 4(11.9), 5(12.4), 6(24.2), 7(8.3), 9(20.3), 10(12.4), 12(16.1), 13(8.4), 14(9.3), 15(25.8), 16(10.7), 20(18.3), 21(9.5), 22(19.8), 23(10.3), 24(11.9), 25(21.3), 26(12.6), 27(32.3), 28(27.0), 29(9.5), 30(29.8), 31(6.9), 32(10.7), 33(22.4), 34(24.5), 35(23.8), 36(15.7), 37(8.9), 38(11.2), 39(14.4), 40(9.1), 41(6.9), 42(11.3), 43(4.4), 44(12.8), 46(12.6), 47(11.3), 48(14.6), 49(10.1), 50(16.3)\)

Amorphous Carbon 1(16.4), 8(15.3), 17(11.0), 18(25.4), 19(12.5)

Calcium Carbonate 11(18.6), 45(17.5)
Region 8

Diameter 225.9um

Number of particles 26

No Signal
1(7.4), 2(10.4), 3(25.9), 4(9.6), 5(10.5), 6(16.0), 7(7.1), 8(11.5), 9(6.9), 10(8.1), 11(9.2), 12(9.8), 13(8.6), 14(7.6), 15(13.1), 17(16.7), 18(34.8), 19(16.1), 20(10.3), 21(8.6), 22(19.1), 24(5.0), 25(4.1), 26(8.3)

Amorphous Carbon 16(34.5),

Region 9

Diameter 233.4

Number of particles 37
No signal

2(11.0), 3(7.2), 4(16.1), 5(12.0), 6(14.8), 7(15.9), 8(12.9), 13(22.2), 14(9.5), 15(16.7), 16(10.5), 17(13.7), 18(19.9), 19(6.1), 20(40.6), 21(15.9), 22(6.4), 24(8.8), 25(10.6), 26(13.0), 27(12.9), 28(7.6), 29(8.2), 30(8.5), 31(9.3), 33(10.7), 34(12.4),

Graphitic-like 1(12.3), 9(20.4), 10(12.1), 32(24.2),

Anatase 11(8.6), 23(7.7)
Anatase 12(15.4),

Quartz 35(29.6), 36(11.2), 37(11.8)

Region 10

Diameter 224.2

Number of particles 33
No Signal

2(13.0), 5(9.1), 6(6.6), 7(11.1), 8(34.3), 9(19.4), 10(24.5), 11(14.5), 12(10.2), 13(10.5), 14(13.2), 15(16.6), 17(14.0), 18(31.1), 19(10.8), 20(10.1), 22(10.2), 25(10.0), 26(10.5), 27(11.4), 29(9.3), 30(10.3), 31(10.6), 32(9.7), 33(10.2),

Graphitic-like 1(20.7), 3(16.8), 4(10.2), 21(16.9), 23(21.6), 28(24.1),

Carbonate 24(12.0),
**Plain slide 20 Day**

Region 1

Area 500 x 500 µm

Number of particles 42

No Signal

1(15.2), 2(25.3), 3(22.7), 4(8.7), 5(14.6), 9(9.6), 10(24.2), 11(11.1), 12(8.1), 13(13.9), 14(7.9), 15(8.4), 17(6.7), 18(15.7), 18(7.6), 21(15.6), 22(13.2), 23(26.7), 25(10.9), 26(44.0), 27(7.6), 28(36.0), 29(13.5), 30(24.2), 31(18.3), 32(34.2), 33(22.8), 34(12.8), 35(9.6), 36(14.9), 37(39.6), 38(15.9), 39(27.3), 40(13.6), 41(19.5), 42(9.3)
Graphitic-like 6(14.6)

Graphitic-like 7(18.9)

Anatase 16(22.9)

Carbonate 5(11.0), 20(13.0),
Carbonate and Antimony 24(12.4)

Region 2

Area um 500 X 500 µm

Number of particles 21

No Signal

1(15.6), 2(12.5), 3(7.2), 5(12.2), 6(7.2), 7(14.1), 8(21.5), 9(7.1),
11(18.9), 12(21.1), 14(117.1), 15(15.6), 16(17.4), 17(38.6), 18(11.2), 19(30).
Graphitic-like 4(8.7)

Carbonate 10(9.8)

Quartz 13(12.6)

Region 3

Area um 500 x 500 µm

Number of particles 12
No Signal

1(25.3), 2(20.5), 3(12.9), 4(12.7), 5(19.7), 6(6.7),
7(13.1), 8(10.4), 9(6.5), 10(20.4), 11(6.4)

Region 4

Area um 500 x 500 µm

Number of particles 26
No Signal

1(19.3), 2(55.7), 3(7.8), 4(29.7), 5(8.0), 6(16.2), 7(6.6), 8(13.0), 9(6.8), 10(32.1), 11(19.2), 12(19.4), 13(23.6), 14(14.6), 16(10.4), 19(17.9), 20(7.7), 21(12.4), 22(24.2), 23(6.2), 24(41.4), 25(18.4), 26(10.3)

Unidentified 15(10.9)
Graphitic-like 17(76.3)

Inquiqueite 18(22.3)

Region 5

Area 500x500 µm

Number of particles 25 (2 no signal)

No Signal

1(19.5), 4(16.9), 5(10.6), 6(11.2), 7(13.5), 9(9.0), 10(11.1), 11(27.7), 12(15.3), 13(22.7), 14(29.9), 15(18.1), 16(15.7), 17(13.5), 18(22.3)
18(16.6), 19(20.9), 21(11.0), 22(21.0), 23(11.6), 24(15.9), 25(12.2),

Graphitic-like 3(41.5), 8(13.5), 26(43.2)

Carbonate 20(10.2)

Region 6

Area 500x500 µm

Number of particles 19
No Signal

$1(16.0), 2(16.6), 3(14.7), 4(25.7), 6(13.0), 7(41.1), 9(8.7), 11(10.3), 12(26.7), 13(8.9), 14(10.5), 15(14.0), 16(14.0), 17(7.8), 18(9.4), 19(9.5), 20(37.2), 21(24.8)$

Graphitic-like $5(29.0), 10(18.8)$
Carbonate 8 (23.9)

Region 7

Area 500x500 µm

Number of particles 26

No Signal

1 (15.9), 2 (31.0), 3 (14.9), 4 (13.4), 5 (28.4), 6 (15.9), 7 (24.1), 8 (11.0), 11 (14.4), 12 (9.2), 14 (33.3), 16 (40.3), 17 (29.2), 18 (34.5), 19 (22.8), 20 (17.9), 21 (15.0), 22 (12.4), 23 (11.1), 24 (12.4), 25 (11.5), 26 (62.3)

Carbonate 8 (21.1)
Graphitic-like 10(11.9)

Anatase 13(40.5),

Quartz 15(42.3)

Region 8

Area 500x500 µm

Number of particles 25
No Signal

1(12.0), 2(32.1), 3(8.0), 4(10.5), 5(11.3), 6(8.4), 7(12.4), 8(18.3), 9(25.3), 10(12.2), 11(14.1), 12(7.9), 13(25.7), 15(6.5), 17(10.3), 18(13.7), 19(19.6), 20(10.0), 22(17.1), 25(16.1)

Graphitic-like 14(13.8), 21(26.9)
Anatase 16(16.2)

Carbonate 23(21.9),

Calcium Oxalate Hydrate 24(40.1)

Region 9

Area 500x500 µm

Number of particles 37 (no #5)
No signal

1 (23.6), 2 (13.2), 5 (14.7), 6 (8.4), 7 (9.9), 8 (5.6), 9 (9.2), 10 (5.4), 11 (10.3), 12 (12.1), 13 (12.0), 14 (8.3), 15 (8.5), 16 (16.4), 17 (16.1), 18 (11.8), 19 (9.0), 20 (10.4), 21 (47.1), 23 (8.3), 24 (11.4), 25 (16.1), 26 (24.6), 29 (20.2), 30 (18.5), 31 (26.6), 32 (23.0), 33 (17.7), 34 (9.5), 35 (17.1), 36 (13.4)

Carbonate 3 (25.9), 22 (14.4), 28 (23.8),
Graphitic-like 27(42.0), 37(42.8)

Region 10

Area 500 x 500 µm

Number of particles 39

No Signal

1(9.5), 2(10.5), 3(31.8), 5(20.7), 6(12.2), 7(8.9), 8(23.7), 9(9.1), 10(7.1), 11(11.5), 12(21.2), 13(11.2), 15(14.5), 16(16.4), 18(6.6), 19(13.9), 20(8.6), 21(16.7), 22(6.9), 23(33.4), 24(7.7), 25(6.5), 26(18.3), 27(22.6), 29(11.0), 30
(22.2), 31(17.4), 32(14.2), 33(30.6), 34(23.3), 36(13.1), 38(8.1), 39(14.3)

Carbonate 4(41.2),

Graphitic-like 14(36.6), 28(20.4), 35(21.2),

Carbonate 17(18.8),

Graphitic-like and Carbonate 37(18.5),
Plain slide 30 Day

Region 1

Area 500 x 500 µm

Number of particles 23

No signal 1(59.1), 5(16.9), 6(14.3), 7(10.5), 8(11.3), 9(14.4), 11(12.4),
12(8.5), 14(39.6), 16(32.7), 17(16.6), 18(18.2), 19(22.6), 20(26.0), 21(12.6)

Graphitic-like 2(18.7), 4(68.1), 12(10.2),
Graphitic-like and Carbonate 3(39.7)

Carbonate 10(28.1), 15(19.1), 23(15.6)

Unidentified 13(10.4)

Unidentified 2 and Apatite 22(18.1)

Region 2
Area 500 x 500 µm

Number of particles 24

No Signal

3(12.0), 4(60.9), 5(9.0), 6(12.6), 8(12.7), 9(24.9), 11(14.6), 13(9.9), 14(12.8), 15(6.5), 16(52.2), 17(22.9), 19(45.8), 20(61.1)

Graphitic-like 1(27.6), 7(21.8), 10(9.8), 12(23.3), 23(63.1)
Carbonate 2(28.3), 18(24.2)

Region 3

Area 500 x 500 µm

Number of particles 28

No Signal

8(7.7), 9(21.9), 11(6.9), 12(10.7), 14(12.6), 17(19.9), 18(11.0), 19(28.8), 20(6.1), 21(15.6), 22(27.1), 23(11.7), 25(9.6), 26(9.6), 27(8.5), 28(14.0)
Anatase and Carbonate 1(34.2)

Carbonate 3(41.0), 4(25.9), 6(19.8), 10(31.3), 16(18.1)

Anatase 5(25.4)

Graphitic-like 7(38.0), 24(10.2)
Graphitic-like and Carbonate 2(27.0), 13(27.4)

Rutile 15(28.8)

Region 4

Area 500 x 500 µm

Number of particles 62
No signal

1 (12.9), 2 (8.8), 3 (17.8), 4 (9.4), 5 (106.9), 6 (12.2), 7 (7.7), 8 (21.2), 9 (23.6), 10 (24.3), 11 (19.9), 12 (7.0), 14 (13.0), 15 (7.6), 16 (14.7), 17 (18.2), 18 (14.4), 19 (41.9), 20 (14.3), 21 (8.1), 22 (9.8), 23 (14.3), 24 (8.1), 25 (9.3), 26 (11.7), 27 (9.0), 28 (7.3), 29 (23.3), 30 (8.8), 31 (4.9), 32 (17.1), 34 (10.8), 35 (4.2), 36 (6.6), 37 (8.7), 38 (11.63), 39 (23.8), 40 (11.1), 41 (26.9), 42 (89.1), 43 (9.8), 44 (54.5), 45 (5.2), 46 (10.0), 47 (4.1), 48 (16.2), 49 (11.0), 50 (6.3), 51 (6.8), 52 (8.4), 55 (21.4), 56 (10.4), 57 (54.8), 58 (7.8), 59 (7.7), 60 (10.1), 61 (8.4), 62 (26.6)

Carbonate 13 (11.7), 33 (18.0),

Graphitic-like 53 (6.8) 54 (6.2)
Region 5

Area 500 x 500 µm

Number of particles 42

No Signal

1(19.9), 2(14.0), 5(37.0), 9(13.8), 10(19.5), 11(7.9), 13(16.3), 14(22.4), 15(14.4), 16(8.5), 17(11.3), 18(18.2), 19(16.4), 22(8.8), 23(19.4), 27(6.2), 28(6.9), 30(7.5), 32(9.5), 33(7.4), 34(10.0), 36(22.7), 38(17.3), 41(12.8), 42(8.2)

Gypsum 3(16.6)
Quartz 4(20.1), 12(16.8), 28(16.8)

Graphitic-like and Carbonate 6(22.7), 7(12.8),

Anatase 8(22.2)

Carbonate 20(20.9), 21(14.3), 23(24.7), 29(16.0), 39(8.9), 40(18.7),

172
Graphitic-like 25(27.9), 26(15.0), 37(28.6)

Carbonate 31(9.2), 35(19.3)

Region 6

Area 500 x 500 µm

Number of particles 68
No signal

1 (14.9), 2 (20.4), 3 (34.0), 4 (24.5), 5 (15.0), 6 (8.9), 10 (24.2), 11 (20.3), 12 (13.0), 14 (8.2), 15 (9.4), 16 (9.8), 17 (17.1), 18 (15.8), 19 (9.0), 20 (9.2), 21 (12.0), 22 (9.4), 23 (12.7), 24 (8.9), 25 (12.0), 27 (11.6), 28 (21.3), 29 (14.7), 31 (22.3), 33 (13.8), 34 (18.7), 35 (8.0), 37 (12.1), 38 (16.7), 39 (14.6), 40 (16.2), 41 (11.8), 42 (18.6), 43 (29.9), 44 (10.2), 45 (12.0), 46 (33.1), 48 (31.1), 49 (25.8), 50 (13.5), 51 (12.1), 52 (26.1), 54 (16.3), 58 (17.8), 59 (13.3), 63 (10.5), 65 (35.3), 66 (41.9), 67 (50.5)
Graphitic-like
7(22.0), 8(11.4), 9(16.3), 13(26.4), 32(35.8), 36(28.4), 57(15.8), 60(32.3), 61(74.2),

Carbonate 26(15.3), 47(22.5), 53(15.3), 62(10.2), 68(25.2)

Unidentified 30(47.0)

Graphitic-like and Carbonate 55(21.5),
Graphitic-like and Carbonate 64(23.1)

Region 7

Area 500 x 500 µm

Number of particles 38

No Signal

2(15.3), 3(15.7), 4(10.2), 5(7.1), 6(11.1), 7(187.7), 8(15.5), 9(10.3), 10(24.1), 11(22.4), 14(15.8), 15(10.3), 16(15.8), 17(12.1), 18(8.1), 20(14.0), 21(15.9), 22(13.9), 23(11.5), 24(29.4), 25(11.9),
26(5.1), 27(11.0), 28(7.2), 29(11.6), 30(7.5), 31(15.2), 32(10.6),
33(8.1), 35(13.5), 37(16.4), 38(15.9)

Graphitic-like 1 (9.9), 19(35.9), 34(13.3), 36(33.7)

Carbonate 9(23.0)

Graphitic-like and Anatase 13(20.9)

Region 8

Area 500 x 500 µm

Number of particles 53
No Signal

1 (13.5), 2 (22.7), 6 (11.3), 7 (23.8), 8 (22.5), 10 (31.3), 14 (40.3), 16 (27.2), 17 (25.0), 18 (12.8), 19 (33.0), 20 (22.3), 21 (56.2), 23 (33.4), 24 (20.5), 25 (38.4), 26 (32.4), 27 (48.1), 30 (13.7), 31 (19.4), 36 (11.1), 38 (31.1), 39 (79.0), 41 (10.3), 44 (28.3), 45 (26.2), 46 (17.9), 47 (14.5), 48 (26.0), 49 (18.8), 50 (15.2), 51 (10.4), 53 (12.1),

Carbonate and Graphitic-like

3 (16.8), 22 (47.9), 29 (39.3), 40 (40.7), 42 (21.2), 54 (16.6)
Graphitic-like

4(24.4), 9(47.7), 12(15.8), 13(30.0), 15(16.6), 32(20.7), 43(20.4), 52(23.1),

Carbonate 5(20.0), 11(18.5), 33(70.0), 35(14.4)

Quartz 28(123.5), 34(43.9),

Graphitic-like and Carbonate 37(147.3),
Region 9

Area 500 x 500 µm

Number of particles 29

No Signal

2 (30.1), 3 (30.8), 4 (13.1), 5 (37.5), 6 (20.4), 7 (10.1), 8 (9.0), 9 (13.3), 11 (6.8), 12 (5.6), 13 (9.3), 14 (25.1), 15 (15.3), 16 (15.1), 18 (6.5), 19 (12.1), 20 (21.2), 24 (7.7), 26 (18.9),

Graphitic-like 21 (15.8), 27 (21.6)
Graphitic-like and Carbonate 17(9.0), 22(13.7),
23(15.8), 29(13.8)

Carbonate and Rutile 25(22.0)

Carbonate 1(29.4), 10(17.9), 28(10.2)

Region 10

Area 500 x 500 µm

Number of particles 35
No Signal

1 (61.6), 4 (11.0), 5 (10.4), 6 (13.1), 7 (10.5), 14 (39.2), 15 (21.4), 18 (30.8), 19 (26.5), 21 (17.0), 23 (30.8), 24 (7.4), 25 (26.0), 26 (15.1), 27 (21.0), 29 (25.6), 30 (26.4), 33 (8.1), 34 (4.8), 35 (8.6)

Carbonate 2 (30.3), 13 (22.3)

Graphitic-like 3 (24.2), 8 (23.1), 16 (23.1), 28 (31.2), 31 (17.4)
Plain slide 30 Day B (second sampling)

Region 1

Area 500 x 500um

Number of particles 81
No Signal

1(15.8), 3(16.3), 6(17.1), 7(18.1), 8(17.3), 9(35.5) Albite (14%), 11(19.9), 12 (34.6), 15(18.1), 16(15.8), 18(14.1), 20(9.3), 21(19.9), 24(15.5), 27(8.8), 28 (10.6), 27(11.5), 30(20.4), 31(17.0), 34(11.8), 39(13.3), 48(13.6), 51(11.1), 52(12.0), 53(22.3), 54(13.6), 56(22.3), 58(21.4), 59(19.6), 63(18.2), 64(26.6), 66(27.9), 71(11.5), 76(23.6), 77(17.4), 79(23.6)

Graphitic-like

10(33.2), 12(34.7), 14(38.1), 22(10.2), 25(15.0), 32(17.3), 41(15.0), 47(25.9), 55(20.6), 68(15.5), 77(17.4), 79(23.6)

Quartz 2(12.7), 17(10.2), 43(18.6)
Carbonate
4 (11.7), 19 (26.7), 23 (12.0), 37 (14.9), 44 (20.9), 45 (47.5), 50 (13.4), 69 (12.6),
72 (12.1), 75 (16.4), 78 (9.8), 80 (23.4),

Quartz and Graphitic-like 5 (68.5),

Graphitic-like and Carbonate
26 (16.3), 42 (16.0), 57 (18.9), 61 (21.8), 62 (17.8),
Unidentified 33(18.8)

Microcline 46(23.9),

Graphitic-like and Carbonate 60(19.9)

Unidentified and Anatase 65(38.7),

Brookite 67(10.5) 73(23.5), 81(27.0)
Region 2

Area 500 x 500 µm

Number of particles 44
No signal

3(31.4), 4(8.5), 5(15.6), 6(11.7), 7(11.1), 8(13.0), 10(11.1), 11(13.0), 13(29.7), 14(5.4), 15(6.6), 17(11.2), 18(9.1), 21(12.2), 22(13.3), 28(8.5), 29(9.7), 30(8.6), 31(6.1), 33(12.8), 36(5.9), 37(10.6), 38(14.4), 39(15.6), 42(8.9), 43(7.9), 44(7.2)

Graphitic-like 2(35.1), 9(12.0), 12(8.4), 24(9.3), 25(7.5), 32(39.8),

Graphitic-like and Carbonate 1(55.7), 16(17.9),
Graphitic-like and Carbonate 19(17.1), 20(14.7), 26(53.0),

Quartz 23(11.4), 40(10.6),

Unidentified 27(48.6),

Carbonate 34(12.4), 35(21.1), 41(17.4),
Region 3

Area 500 x 500 µm

Number of particles 24

No Signal

1(20.7), 5(14.0), 7(18.3), 9(41.0), 16(11.1), 18(11.6), 19(14.9), 23(34.0),

Graphitic-like 4(19.6), 12(27.3), 14(19.6),

Gypsum and Graphitic-like 2(42.9)
Gypsum 3 (30.7)

Graphitic-like and Carbonate 6 (17.9), 21 (12.3), 22 (10.6), 24 (27.0)

Carbonate 8 (20.0)

Unidentified 10 (23.6)
Carbonate 11(12.9), 17(11.4),

Graphitic-like and Anatase 15(16.6),

Carbonate and Anatase 13(80.8),

Region 4

Area 500 x 500 µm

Number of particles 42
No Signal

2 (31.0), 3 (13.3), 4 (8.2), 5 (7.3), 6 (7.2), 8 (5.9), 9 (18.0), 11 (9.5), 12 (13.1), 13 (5.7), 14 (6.9), 15 (10.0), 17 (17.9), 19 (21.7), 22 (12.1), 23 (5.9), 24 (5.9), 25 (16.6), 26 (8.0), 27 (8.1), 28 (5.8), 29 (12.4), 30 (14.4), 31 (9.9), 32 (9.3), 33 (8.1), 36 (16.8), 37 (14.9), 38 (3.8), 40 (6.3), 41 (6.1)

Graphitic-like

1 (8.9), 7 (12.3), 10 (13.4), 16 (11.9), 18 (14.0), 20 (22.7), 34 (8.1), 39 (17.5), 42 (38.2)

Graphitic-like and Carbonate 21 (16.0),
Carbonate 35(14.9),

Region 5

Area 500 x 500 µm

Number of particles 44

No Signal

1(21.8), 5(18.4), 6(8.3), 7(7.8), 9(9.4), 10(7.8), 13(9.1), 15(26.2), 16(4.2), 17(11.1), 18(11.6), 19(16.9), 22(14.5), 27(8.9), 29(7.3), 31(9.4), 33(10.0), 34(23.3), 38(20.4), 39(16.3), 42(10.1), 42(7.6), 43(31.4), 44(8.0),

Graphitic-like
Graphitic-like and Carbonate 3(18.3), 23(30.9), 36(14.2), 41(9.8),

Anatase 12(37.4),

Carbonate 20(8.5), 21(21.0), 30(29.6), 32(6.5), 40(24.3),
Graphitic-like and Quartz 26(51.4),

Carbonate 35(25.8)

Region 6

Area 500 x 500 µm

Number of particles 30

No signal

3(9.7), 6(7.8), 10(21.3), 13(11.4), 15(31.7), 17(13.4), 18(15.8), 19(20.3), 21
(8.6), 22(6.4), 23(11.9), 24(8.2), 26(14.6), 27(15.9), 28(26.6), 30(16.4)

Graphitic-like 2(17.7), 5(12.2), 8(25.1), 9(19.4), 12(23.3),

Carbonate 1(16.4), 20(6.8), 29(15.9),

Gypsum 4(8.8),

Carbonate 7(13.1), 14(11.1), 25(8.9),
Titanite 11(12.2)

Graphitic-like and Carbonate 16(10.7)

Region 7

Area 500 x 500 µm

Number of particles 41

No signal

1(6.3), 5(12.5), 6(9.5), 9(9.9), 11(8.4), 12(20.1), 17(22.8), 18(14.8), 19(8.4)
21(22.3), 22(9.6), 24(6.4), 25(15.9), 26(6.8), 27(6.8), 30(14.1), 32(14.3),
34(13.3), 35(11.1), 37(28.8), 39(20.6), 41(16.6)
Graphitic-like

3(12.8), 7(20.2), 8(7.2), 28(23.7), 29(11.1), 36(10.6), 38(12.6)

Carbonate 2(8.2), 20(28.6),

Carbonate 5(6.2), 16(6.4), 23(13.5),

Quartz 10(23.7),
Graphitic-like and Carbonate 13(10.3), 14(22.9),

Graphitic-like and Carbonate 15(14.5)

Anatase 31(16.3), 33(30.4), 40(10.9)

Region 8

Area 500 x 500 µm

Number of particles 38
No Signal

5(19.6), 6(18.2), 8(9.3), 9(12.1), 11(15.7), 14(8.4), 15(9.9), 16(9.5), 17(8.2), 18(9.9), 19(12.5), 20(11.1), 23(11.3), 24(18.7), 25(18.3), 26(32.9), 27(18.1), 29(42.9), 31(19.6), 33(14.2), 34(18.5), 36(12.2), 38(15.7),

Graphitic-like 1(16.6), 2(21.2), 3(45.7), 21(63.5), 22(17.5), 28(30.9)

Unidentified and Apatite 4(31.1)
Carbonate 7(20.7), 12(15.8)

Carbonate 10(8.7), 13(29.2)

Anatase 30(20.0)

Graphitic-like and Ankerite 32(29.5)
Graphitic-like and Quartz 35(15.2),

Amorphous Carbon and Carbonate 37(22.2)

Region 9

Area 500 x 500 µm

Number of particles 39
No Signal

1(18.5), 2(7.8), 3(27.9), 4(37.4), 5(29.0), 6(11.8), 7(41.0), 8(13.0), 9(9.3), 10(8.2), 11(12.7), 12(7.6), 12(28.1), 13(7.3), 14(17.0), 15(11.8), 17(16.4), 19(12.9), 20(16.1), 21(7.1), 22(12.6), 23(12.0), 24(13.0), 29(17.9), 30(20.5), 31(28.2), 32(8.5), 33(31.2), 34(19.5), 35(8.1), 36(17.6), 37(27.1), 38(22.9), 39(47.5)

Graphitic-like 16(44.0), 25(20.1), 28(11.7)

Anatase 18(11.9)

204
Quartz 26(30.9)

Albite 27(58.6)

Region 10

Area 500 x 500 µm

Number of particles 24
No Signal

1(38.8), 2(69.9), 7(11.5), 9(7.6), 14(8.5), 17(11.8), 18(16.2), 19(24.7)

Graphitic-like 4(22.0), 15(12.8), 16(11.4), 20(22.6), 21(23.0), 24(13.9)

Carbonate 3(17.5), 8(18.7), 11(9.9), 12(11.1), 23(12.6),

Graphitic-like and Carbonate 5(34.4), 22(31.4),
Plain slide 60 Day

Region 1

Area 212 x 519 µm

Number of particles 41

No Signal

1(28.0), 2(25.2), 4(36.8), 5(62.0), 9(35.5), 10(31.4), 11(18.8), 13(29.7), 14(33.0), 15(16.6), 17(18.7), 18(58.4), 20(47.3), 21(40.4), 23(46.5), 26(26.2), 27(37.1), 28(30.0), 29(32.1), 31(24.0), 32(17.3), 33(19.1), 34(30.8), 35(24.0), 37(70.9), 39(22.3), 41(24.2)
Graphitic-like
6(30.9), 7(23.9), 8(29.0), 12(12.3), 19(22.9), 22(35.6), 24(65.5), 30(86.8), 3
6(31.9), 38(54.3), 40(23.7)

Graphitic-like and Carbonate 3(41.2),

Carbonate 16(15.2), 25(12.1),

Region 2

Area 500 x 500 µm

Number of particles 18
No Signal

1(32.3), 2(10.2), 3(19.2), 4(13.2), 6(8.9), 7(39.5), 8(21.4), 9(8.0), 10(24.0), 11(44.5), 12(17.9), 13(44.3), 16(14.6), 18(15.7)

Graphitic-like 5(7.4), 14(14.5), 15(11.7), 17(29.3)

Region 3

Area 500 x 500 μm

Number of particles 11
No Signal

1(10.3), 2(16.8), 3(12.9), 4(11.0), 5(12.1), 6(11.5), 7(7.4), 8(44.4), 9(13.4),
10(23.3), 11(18.8)

Region 4

Area 500 x 500 µm

Number of particles 24
No Signal 1(18.0),
2(14.8), 4(56.7), 5(20.5), 6(10.2), 7(23.0), 9(56.5), 10(22.9), 11(16.0), 12(15.2), 13(41.1), 14(16.8), 15(20.3), 16(25.3), 17(17.5), 18(15.5), 19(16.9), 20(16.8), 22(14.0), 23(9.2), 24(13.5)

Graphitic-like 3(26.3), 8(20.2), 21(26.7)

Region 5

Area 500 x 500 µm

Number of particles 16
No Signal

\(2(15.2), 3(22.3), 4(29.4), 5(6.8), 6(7.1), 8(10.3), 9(5.4), 10(11.9), 11(18.8), 12(12.3), 13(14.3), 14(14.5), 15(8.1), 16(19.1),\)

Graphitic-like 7(16.8),

Carbonate 1(32.0),

Region 6

Area 500 x 500 µm

Number of particles 12
No Signal

1(15.2), 2(11.0), 3(7.4), 4(7.9), 5(12.7), 6(25.1), 7(23.6), 8(8.8), 9(7.9), 10(9.6), 11(10.5), 12(15.3)

Region 7

Area 500 x 500 µm

Number of particles 17
No Signal 1(21.0),3(22.5),5(43.0),

Graphitic-like
2(30.7),4(22.0),7(17.8),8(29.5),9(25.6),10(21.8),11(32.5),12(27.0),13(38.6),15(26.1),16(33.3),

Carbonate 6(26.0),14(22.2)
Carbonate 7(17.5),

Graphitic-like and Carbonate 17(25.4)

Region 8

Area 500 x 500 µm

Number of particles 24

No Signal

1(29.3), 2(10.7), 3(7.7), 4(15.4), 5(34.8), 6(19.5), 7(25.8), 8(25.0), 9(14.6), 10(22.0), 11(25.3), 12(42.0), 13(32.3), 14(34.1), 15(24.2), 16(18.5), 17(12.1), 18(14.7), 19(23.9), 20(22.9), 21(21.5), 22(14.0), 23(30.6), 24(7.5)
Region 9

Area 500 x 500 µm

Number of particles 15

No Signal

1(22.6), 2(16.3), 3(10.6), 4(13.5), 5(6.6), 6(26.2), 7(11.2), 8(17.3), 9(5.7),
10(7.1), 11(15.0), 12(14.6), 13(17.1)

Graphitic-like 14(53.6), 15(12.5),

Anatase 16(6.9)
Region 10

Area 500 x 500 µm

Number of particles 21

No Signal

1(33.4), 2(13.9), 3(17.8), 4(21.4), 5(8.9), 6(20.7), 7(18.3), 8(22.7), 9(12.0), 10(11.8), 11(18.9), 12(7.9), 13(12.0), 14(19.0), 15(57.9), 20(11.2)

Graphitic-like
copper(II)phthalocyanine 16(11.7),17(29.1)19(18.0)

Rutile 18(6.3)

Direct Collection Plain Slide 100 Day

Region 1

Area 500 x 500um

Number of particles 60
No Signal

2 (24.9), 3 (12.0), 4 (22.1), 5 (45.0), 7 (15.4), 8 (9.1), 9 (23.9), 12 (16.0), 13 (9.2), 14 (17.4), 15 (13.7), 16 (43.1), 17 (12.0), 18 (13.9), 19 (22.1), 21 (11.9), 23 (44.2), 24 (22.6), 25 (16.3), 26 (14.2), 27 (11.6), 28 (33.6), 29 (10.5), 30 (30.9), 31 (38.3), 32 (23.4), 33 (31.0), 34 (17.2), 35 (41.8), 36 (16.3), 37 (26.9), 38 (23.2), 42 (15.7), 44 (12.5), 45 (8.9), 46 (15.6), 47 (28.4), 48 (19.0), 49 (13.3), 50 (10.3), 51 (9.7), 52 (9.7), 53 (12.8), 54 (10.4), 55 (9.2), 56 (12.6), 59 (9.4), 60 (8.9)

Graphitic-like 10 (19.5), 11 (28.5), 43 (16.2),

Carbonate 1 (10.7), 13 (9.2), 39 (15.9), 40 (18.8), 41 (18.3), 57 (22.7), 58 (10.0)
Region 2

Area 500 x 500um

Number of particles 18

Graphitic-like and Carbonate 6(31.5),20(9.2),22(28.6)

No Signal

1(35.7),3(34.1),7(17.2),8(18.2),9(12.9),10(12.8),11(13.9),12(20.7),14(220)
14.0), 15(19.5), 16(39.2), 17(16.5),

Amorphous Carbon 2(32.4), 18(16.3),

Nitratine 4(23.3),

Carbonate 5(10.9), 6(48.7), 13(30.9),

Region 3

Area 500 x 500µm

Number of particles 17
No Signal

2(23.3), 3(9.6), 6(14.2), 7(7.8), 8(21.5), 9(30.6), 11(28.1), 12(11.9), 15(13.3), 16(10.1),

Graphitic-like 4(45.4), 17(44.9),

Graphitic-like and Carbonate 1(53.3), 14(32.3),
Carbonate 13(14.5),

Talc 5(17.2),

Unknown 10(13.7),

Region 4

Area 500 x 500um

Number of particles 28
No signal

1(15.0), 3(14.1), 4(20.0), 7(22.0), 8(25.1), 9(10.4), 10(14.2), 11(20.2), 12(16.9), 13(21.0), 14(18.1), 15(13.6), 16(13.6), 18(23.7), 19(14.7), 20(10.5), 24(14.1), 25(29.3),

Graphitic-like 2(27.4), 6(47.9), 23(23.1), 26(18.2),

Oversaturated 5(37.0)

Carbonate 17(25.4), 21(20.0), 27(12.1),
Graphitic-like and Carbonate (weloganite) 22(25.1)

Carbonate (Weloganite) 28(31.6),

Region 5

Area 500 x 500um

Number of particles 27
No Signal

1(12.9), 2(10.9), 5(9.2), 7(24.1), 8(11.0), 9(12.1), 10(10.1), 11(10.2), 12(19.8), 13(17.7), 15(14.3), 17(7.6), 18(23.7), 19(10.4), 20(7.7), 21(20.3), 22(13.2), 23(11.7), 24(11.7), 25(13.1), 26(7.7), 27(29.4), 28(31.8),

Graphitic-like

Carbonate (Ankerite) 3(29.3), 4(18.5)
Graphitic-like and Anatase 6(29.2),

Carbonate (Spurrite) 14(26.7),

Graphitic-like and Carbonate 16(42.7),

Region 6

Area 500 x 500um

Number of particles 24
No Signal

1(13.5), 2(24.4), 3(13.3), 4(14.5), 5(11.0), 6(13.1), 8(8.3), 9(8.1), 11(23.2), 13(11.5), 14(20.4), 15(18.5), 16(13.8), 17(31.8), 18(28.8), 19(19.1), 20(22.0), 21(20.2), 22(12.1), 23(14.9), 24(10.9),

Graphitic-like 10(11.0), 12(24.2),

Region 7

Area 500 x 500um

Number of particles
No Signal

2(34.1), 4(13.5), 6(9.3), 7(11.2), 9(13.5), 10(8.5), 11(10.0), 12(9.5), 13(20.3), 16(10.0), 17(12.1), 20(27.3), 21(11.3), 22(11.3), 23(10.2), 25(15.2), 27(11.1), 28(12.8), 29(14.6), 30(11.8), 31(14.5),

Graphitic-like 1(18.2), 5(19.3), 8(14.4), 18(26.9), 24(22.9), 26(16.7),

Quartz and Graphitic-like 3(41.2),
Carbonate (Spurrite) 14 (20.4),

Carbonate (Ankerite) 15 (37.3)

Carbonate and Graphitic-like 19 (19.7),

Region 8

Area 500 x 500um

Number of particles 31
No Signal

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Graphitic-like 3(16.3), 18(23.8), 31(9.9),

Carbonate 2(19.1), 16(13.9), 30(15.2),
Region 9

Area 500 x 500um

Number of particles 57

No Signal
1(11.9), 2(49.5), 3(11.6), 4(29.3), 5(29.5), 7(26.2), 8(15.9), 9(47.4), 11(15.9), 15(11.8), 16(25.3), 17(10.0), 18(16.0), 19(21.7), 20(11.9), 21(15.5), 22(10.3), 23(17.4), 24(32.1), 25(12.9), 26(37.5), 28(14.7), 29(15.9), 30(10.6), 31(16.3), 32(11.5), 34(19.5), 35(13.7), 37(13.6), 40(17.9), 42(14.1), 43(18.5), 44(18.4), 45(22.1), 47(18.3), 48(34.3), 49(14.8), 50(15.3), 51(16.5), 52(13.4), 54(21.2), 55(10.5), 56(12.9), 57(10.0),

Graphitic-like
12(18.2), 13(17.2), 14(14.1), 27(38.1), 36(30.7), 38(20.0), 41(18.2), 46(32.8), 53(13.3),
Region 10

Area 500 x 500um

Number of particles 27
No Signal

2(11.3), 3(17.5), 4(15.0), 6(17.2), 7(13.1), 8(15.8), 9(9.9), 11(21.3), 12(10.9), 13(12.9), 14(10.4), 15(20.1), 16(10.8), 17(58.5), 18(11.6), 19(13.8), 20(30.4), 21(19.5), 22(17.5), 24(18.3), 25(8.5), 26(26.2), 27(33.4),

Graphitic-like 5(16.0), 10(18.8),

1(18.6),
Graphitic-like and Carbonate 23(27.1),
APPENDIX B: ION CHROMATOGRAPHY DATA AND CALIBRATION

### Ion Chromatography Calibration Concentrations Sample Set 1

<table>
<thead>
<tr>
<th>Conc</th>
<th>Fluoride Peak Area</th>
<th>Chloride Peak Area</th>
<th>Bromide Peak Area</th>
<th>Nitrate Peak Area</th>
<th>Phosphate Peak Area</th>
<th>Sulfate Peak Area</th>
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<td>0</td>
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### Ion Chromatography Raw Sample Set 1

<table>
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<tr>
<th>Sample ID</th>
<th>Fluoride</th>
<th>Chloride</th>
<th>Bromide</th>
<th>Nitrate</th>
<th>Phosphate</th>
<th>Sulfate</th>
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<tbody>
<tr>
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<td>2.0328</td>
<td>ND</td>
<td>0.0223</td>
<td>ND</td>
<td>21.3626</td>
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<td>0.1172</td>
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### Ion Chromatography Calculated Concentration Sample Set 1

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<tr>
<th>Sample ID</th>
<th>Fluoride</th>
<th>Chloride</th>
<th>Bromide</th>
<th>Nitrate</th>
<th>Phosphate</th>
<th>Sulfate</th>
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### Ion Chromatography Calibration Concentrations Sample Set 2

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<th>Concentration (ppm)</th>
<th>Fluoride</th>
<th>Chloride</th>
<th>Bromide</th>
<th>Nitrate</th>
<th>Phosphate</th>
<th>Sulfate</th>
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<tr>
<td>0</td>
<td>0.0206</td>
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<td>1.5772</td>
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237
### Ion Chromatography Raw Sample Set 2

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<th>Nitrate</th>
<th>Phosphate</th>
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### Ion Chromatography Calculated Concentrations Sample Set 2

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<th>Bromide</th>
<th>Nitrate</th>
<th>Phosphate</th>
<th>Sulfate</th>
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</tbody>
</table>
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