THESIS

AN EVALUATION OF THE COLLECTION CHARACTERISTICS AND USABILITY FACTORS OF THREE NANOPARTICLE SAMPLERS

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ABSTRACT

AN EVALUATION OF THE COLLECTION CHARACTERISTICS AND USABILITY FACTORS OF THREE NANOPARTICLE SAMPLERS

Occupational exposure to nanoparticles is a concern to occupational hygienists because of the potential health effects of exposure, the lack of standardized sampling methods and regulatory guidance for exposure limits. Exposure assessments for nanoparticles should include analysis of particles with an electron microscope to allow for identification of particle size, shape and composition. This study is the first to use multiple aerosols to compare the particle size fractions collected by three handheld nanoparticle samplers designed to use transmission electron microscope grids for particle collection. These include the Tsai diffusion sampler (TDS), electrostatic precipitator (ESP), and thermophoretic personal sampler (TPS). Aerosols of sodium chloride, ISO fine test dust, and aluminum oxide were tested and the particle size fractions collected by the nanoparticle samplers were compared. The TDS collected more particles in a wider size range for the lowest concentration aerosol. The ESP sampled for much shorter than the others but collected the most particles for two out of three aerosols. The usability questionnaire assessed all steps involved in sampler usage and rated the features of each device. The TDS and TPS were best suited for full shift sampling and the ESP best for short term. The TDS was the most affordable and has the potential to collect larger particles on a secondary filter. Overall, the TPS was the easiest device to use. Study results indicated that all samplers successfully collected three types of aerosols, with smaller differences in the size fractions they collected and larger differences in the number of particles per surface area of their collection media.

ACKNOWLEDGEMENTS

I would like to thank my advisor Dr. Candace Tsai for putting a great deal of effort towards training me and pushing me to adhere to higher academic standards. I would also like to thank my parents for supporting me and working hard to give me advantages which facilitated my academic accomplishments.

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LIST OF ACRONYMS

ESP..Electro Static Precipitator
NIOSH.. National Institute of Occupational Safety and Health
NP...Nanoparticle
OEL...Occupational Exposure Limit
OPS..Optical Particle Sizer
RTI..Real Time Instrument
SD...Standard Deviation
SMPS..Scanning Mobility Particle Sizer
TC..Total Concentration
TEM...Transmission Electron Microscope
TDS...Tsai Diffusion Sampler
TPS...Thermophoretic Personal Sampler

CHAPTER 1: INTRODUCTION

The manufacture of nanoparticles (NPs) and goods containing NPs represent a potential threat to worker health if exposures are not properly assessed and controlled. Workers can be exposed in facilities that manufacture NPs or in the process of incorporating premade NPs into other goods. Examples of exposures can include when workers harvest carbon nanotubes from chemical vapor deposition furnaces, or when workers handle silver nanoparticles as they are added to clothing[1] for anti-microbial properties. Additional uses for nanoparticles include as: additives to plastic polymers, ultra-thin protective coatings, battery components, medicine delivery agents, and as extremely strong fibers. The global workforce which uses nanomaterials in manufacturing is expected to grow to approximately six million workers by 2020[2].

NPs suspended in the air can enter the respiratory tract where they can cause harmful effects. Additionally, they can be absorbed into the blood and relocate to other organs where toxic effects can be induced[3]. A relevant toxicological property of NPs is that these small particles have a large surface area to mass ratio, which likely contributes to increased toxicity. A main mechanism of NP toxicity is their capability to induce oxidative stress which can damage cell components such as DNA and mitochondria, with the oxidation of DNA being associated with cancer[4]. Carbon nanotubes have shown similarities in their toxicological effects to that of asbestos, which may be attributable to their similar physical shape. Health effects of exposure include granulomas and fibrosis in the lungs, and the development of mesothelioma has been found in animal studies[5]. Ingestion and dermal routes of exposure may also pose a risk to worker health. The largely unknown consequences of chronic exposure[6] and severity of potential health effects make an accurate exposure assessment especially important.

Since occupational exposure limits (OELs) are based on known health effects and epidemiological studies in this field are lacking, researchers at the National Institute of Occupational Safety and Health (NIOSH) have suggested that NP OELs could be grouped based on their mode of action[7], such as NP's whose main toxicological mechanism is the excess production of reactive oxygen species. NPs which share toxic effects can also share physiologic properties such as the similar aspect ratios of fibrous nanomaterials suspected of causing fibrotic lung disease. However, this is not always the case as there can be inconsistencies in the level of toxicity in nanomaterials with similar composition[7]. NIOSH has established recommended exposure limits for carbon nanotubes and titanium dioxide, but currently no enforceable limits have been promulgated by the Occupational Safety and Health Administration[8, 9]. In the United Kingdom a suggested standard for fibrous nanomaterial exposure is 0.01 fibers/mL, matching their standard for asbestos fibers[10].

Performing exposure assessments for NPs in the workplace should utilize both area and personal sampling. Area sampling devices can include real time instruments (RTIs) which are often large and difficult to transport. RTIs measure particle number concentration, size range, particle surface area, mass concentration and other metrics. A review of workplace exposure studies found that the most regularly used particle metrics by researchers were number concentrations and size distributions[11]. A variety of RTIs are available but many are not able to discriminate between incidental nanoparticles and those coming from a process of interest[11]. Smaller personal sampling devices may include filter based samplers and devices which collect particles on transmission electron microscope (TEM) grids. The use of filter based gravimetric personal samplers can give mass concentrations of nanometer and respirable sized particles, as well as allow for chemical composition analysis[12, 13]. A disadvantage of using filter based personal samplers to measure the mass concentration of NPs is that they are presently considered inadequate due to the sensitivity needed to detect the small mass of these particles[11]. NPs collected on TEM grids can be analyzed with an electron microscope to allow for particle size, morphology and chemical composition to be determined. TEM image analysis for NPs has been conducted by taking images of particles and using image software to measure particle size, clustering, and elemental composition through energy dispersive x-ray spectroscopy (EDX)[14, 15]. When smaller NPs cluster together it changes the size distribution of the aerosol and this can change where in the respiratory tract these particles deposit. Additionally, TEM analysis allows for the selection of which particles are tested for compositional analysis, as compared to bulk sample analysis that can be done with filter based samples.

A review of NP worker exposure studies in 2016 found that issues in performing exposure assessments included difficulty in collecting NP samples from the breathing zone, there not being a broadly accepted method for the analysis of particle images taken with a TEM, and that more research is needed on developing a method for analyzing NPs collected in occupational environments[2]. In the past, a modified version of method 7402 from the NIOSH Manual of Analytical Methods (NMAM) has been used to analyze TEM images of carbon nanotubes by noting their shape, dimensions, and clustering[16]. Progress has been made in creating a NMAM procedure designed specifically for carbon nanotubes and nanofibers, with a draft recently being released by NIOSH for public comment[17]. To conduct a more complete exposure assessment, detailed information should be gathered using both RTIs and electron microscopy[18], which provides more certainty the NPs of interest are the ones being sampled. Portable devices which can capture particles for characterization with an electron microscope are thus a valuable tool for describing NP exposure.

The usability of a sampling device can affect the quality of the data gathered and how often the device is used by practitioners in the field. Several usability rankings have been published for handheld medical devices and equipment[19-21]. Questionnaires were designed to assess all steps necessary in using the device and factors related to overall usability, like first use success and the most common problems encountered. Human factor criteria have been described to address effectiveness, learnability, errors, efficiency, and satisfaction[21]. A usability study for a cell phone described that the complexity of the system being evaluated is based on the amount of rules which the user needs to be proficient in to operate the instrument[22]. The questionnaire designed for this study focuses on criteria of importance to occupational safety practitioners like ease of use, reliability, and affordability. The effectiveness of each sampler at collecting various particles was also evaluated.

This study was designed to address the need to compare the performance and usability of samplers which could be used as part of a workplace exposure assessment of NPs. Sampling devices which are capable of personal sampling are relatively new and occupational health practitioners may have limited experience with them. The samplers evaluated for this project include: (1) the novel Tsai Diffusion Sampler (TDS), (2) an Electrostatic Precipitator[23] (ESP), and (3) a Thermophoretic Personal Sampler[24] (TPS). The TDS functions through diffusion, in which an aerosol containing airstream flows over the TEM grid and filter at 300 mL/min. Particles then deposit onto the grid as air moves across it, and onto the filter when air moves through it with the smallest particles diffusing the fastest. The ESP works through electrostatic precipitation. An electrostatic force is created when 6600 volts of current flows between two metal surfaces, and a flow of particle containing air is passed between these surfaces. The particles are charged when they pass through the corona and the field directs them to the negatively charged metal plate upon which a TEM grid rests[25]. The TPS collects particles by creating a thermophoretic force between a hot and cold plate. An 85-degree Celsius temperature gradient creates a force from the high temperature plate towards the cold plate where the TEM grid rests. This force is the result of the higher kinetic energy associated with the hotter gas molecules which transfer more momentum to particles, creating a net force away from the hot plate[25]. The ESP, TPS, and TDS have been used in several studies to characterize nanoparticles in the field and in the lab[23, 24, 26-31], but none have compared the collection performance of the samplers. For this study, the samplers were compared under lab conditions and utilizing various aerosols with different shape, size, and agglomeration. This was done to more fully describe the sampler performance by sampling aerosols with different properties. The aluminum oxide aerosol was representative of engineered nanomaterials, sodium chloride has been used extensively in aerosol research, and road dust was representative of natural NP containing aerosols. This information can help occupational health practitioners choose an appropriate sampling device for their needs and help identify future avenues for research and development relating to personal NP samplers.

CHAPTER 2: METHODOLOGY

2.1 Process

A total of nine experiments were conducted in a controlled environment to characterize the collection efficiencies of three personal NP samplers. This was accomplished by contrasting the differences among sampler size fractions collected and by comparing these to RTI size distributions. Three aerosols with varying morphology, agglomeration, and size were released into the center of a high efficiency particulate air filtered (HEPA) glovebox (Terra Universal, Fullerton CA, 35 in. x 24 in. x 25 in.) in an aerosol laboratory. Imaging software was used to measure particle diameters from the microscope images. These were used to create size distributions which were compared statistically by calculating two tailed p values from chi square analyses comparing the number of particles collected by the samplers in each size bin. Moreover, a questionnaire was developed to assess the usability and effectiveness of each sampler using a mixture of quantitative and qualitative measures. Finally, the strengths and weaknesses of the three samplers were summarized to give occupational health practitioners information regarding which device may best suit their needs.

All samplers, tubing, laboratory stands, mixers, generators, and inside surfaces of the glove box were cleaned using distilled water and cleanroom wipes before the start of each experiment. Three runs were conducted for each aerosol type for a total of nine experiments (3 aerosols × 3 repetitions each). Multiple runs were conducted to increase the particle sample sizes to give more confidence in statistical comparisons and to average out any differences in experimental conditions. All experiments were conducted with the glove box HEPA filtration system left on to provide low background particle counts and to help keep aerosol concentration levels stable throughout the experiment. Background concentration levels were recorded for several minutes, followed by the activation of the particle generators. After aerosol concentrations stabilized, the samplers were turned on and sampled for their allotted time, at which point the generators were turned off and samples retrieved for analysis.

2.2 Materials and Instrumentation

ISO fine test dust (12103-1 A2), referred to as road dust, contained 57% of particles by volume less than 11 µm in diameter and a density of 0.9 g/cm^3, and was dispersed using a Wright dust feeder (WDF II, Westwood NJ). Sodium chloride particles were released using a TSI (Shoreview, MN) particle generator (model 8026), which created an aerosol of particles with a density of 2.2 g/cm^3 and a count median diameter of 40 nm. Aluminum oxide (Al_2O_3) powder (Nanophase, Romeoville IL), with a mean particle diameter of 40 nm and a density of 4.0 g/cm^3, was dispersed using a compact digital mixer (Cole-Parmer, Vernon Hills IL) which stirred 200 mL of powder in a 500 mL büchner flask.

Two RTIs and three NP samplers were positioned equidistant from the aerosol source. RTIs were used to measure particles between 10 nm and 10 µm in diameter and included a Nanoscan Scanning Mobility Particle Sizer (SMPS, TSI 3910) and Optical Particle Sizer (OPS, TSI 3330) which both utilized one-minute sampling periods. Each RTI used a three-foot length of non-conductive tygon tubing to sample inside the glove box and tubes were taped together at the sample end. The personal NP samplers tested were the Thermophoretic Personal Sampler (TPS 100), the Electrostatic Precipitator (ESP nano), and the Tsai Diffusion Sampler (TDS). The TDS and ESP collected particles on TEM copper grids (400-mesh with carbon coating) and the TPS utilized 400-mesh carbon coated nickel TEM grids. The TPS operated at 5 mL/min and the ESP at 55 mL/min. The TDS used a Gil-Air 3 personal sampling pump (St. Petersburg FL) running at

300 mL/min and consisted of a 25mm slotted syringe filter cassette and polycarbonate 0.2µm pore size filter with a TEM grid in the center. The Gil-Air 3 pump was calibrated before and after each experiment.

Aerosols were generated and released pointing vertically at a height of 13cm in the center of the glove box for road dust and sodium chloride. Real time instruments and personal samplers were positioned 10cm above and 10cm away from the emission source and arranged from highest flow rate to lowest as shown in Figure 1.

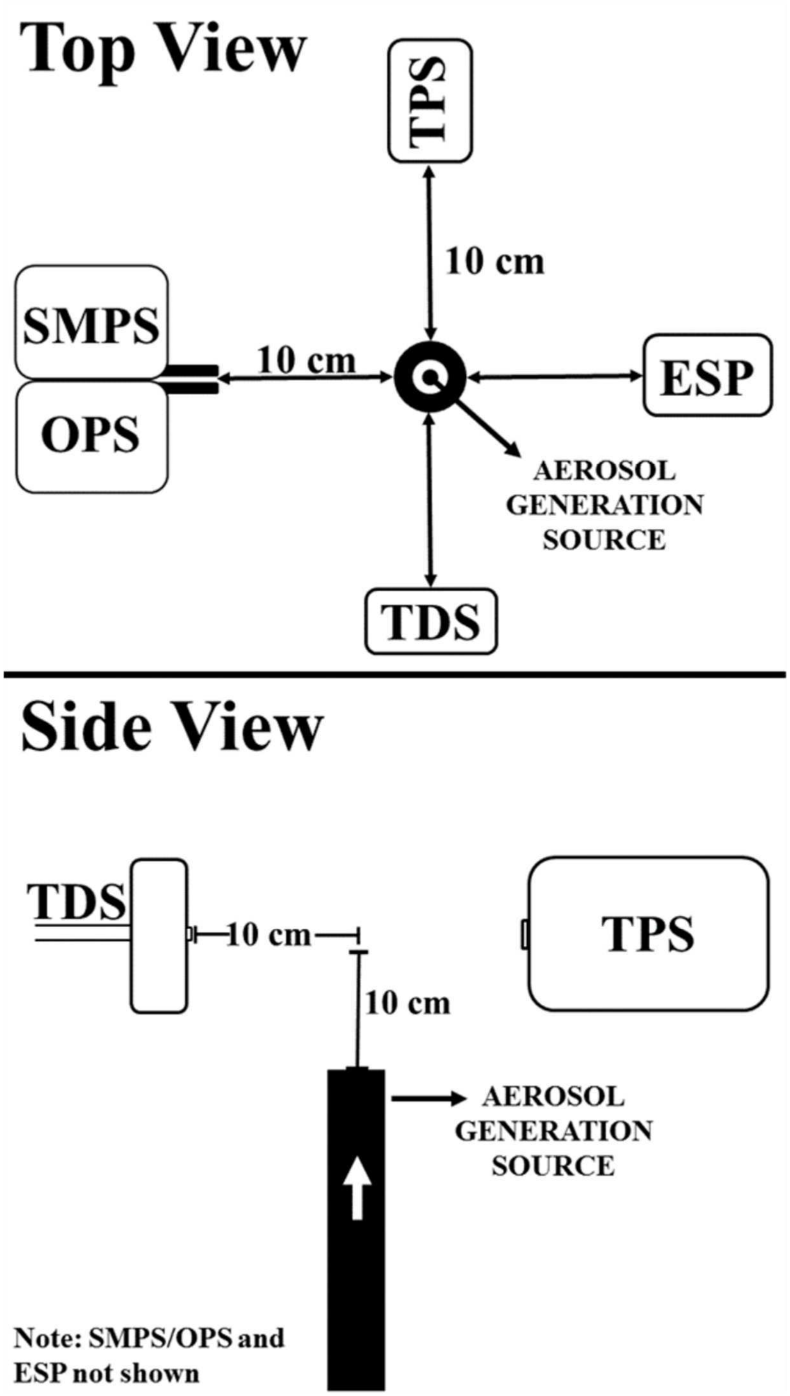


Figure 1 – Experimental setup for measuring aerosols in a HEPA filtered glovebox.

For aluminum oxide, the samplers were attached to tygon tubing which was placed down into the neck of the büchner flask, and 12cm above where the stirrer agitated the powder. Previous testing indicated this was the best method to obtain sufficiently high and stable aluminum oxide concentrations. Particle number concentrations were monitored using the RTIs until they stabilized, followed by the activation of the samplers. The TDS and TPS ran for 40 minutes while the ESP sampled for 50 seconds based on recommended sampling times printed on the back of the device. After all samplers completed their sampling period the particle generators were turned off, the sampler inlets capped, and the ventilation system left on for several minutes to purge contaminants from the glove box. The samplers were then removed and samples collected for analysis.

2.3 Electron Microscope Imaging and Analysis

A transmission electron microscope was used to image the particles collected by the samplers. TEM grids were directly analyzed by a TEM (JOEL Model 2100F, Peabody, MA, USA) at 200 kV equipped with a digital Gatan Ultrascan camera. Energy Dispersive X-ray spectroscopy (EDX) was conducted to confirm that the elemental composition of particles collected by each sampler matched that of the respective aerosol. Particle images were taken following this standardized procedure: (1) Low resolution photos (80x) of the center and four corners of the grid, (2) images were then taken (500x) of individual grid spaces that show the range of low to high particle counts, (3) a grid space which was generally representative of the particle count of the rest of the grid was chosen for detailed imaging, and (4) the grid space was methodically traversed and particles were imaged (6000-8000x) until 300 particles are counted.

Fiji[32] software was used to analyze TEM images through the following process. High magnification images of individual particles or clusters of particles were converted to pure black and white by adjusting the image threshold. This caused particles to be shaded in black with the background being converted to white. In some cases, the contrast between the particle and the background was not sufficient to be automatically recognized by the program. If this was the case, the boundary of the particles had to be drawn in manually using a pencil tool before the particles could be automatically highlighted. Next the software utilized a distance scale provided by the electron microscope to measure the surface area of each particle. The program output data indicated the number of particles with the measured surface area of each particle. Additionally, an image was generated which showed the outlines of every particle measured and assigned each particle a number. This image was checked to ensure that all particles were accounted for and that the borders were outlined correctly. Finally, this surface area was converted to the diameter of a circle with the equivalent surface area in an excel spreadsheet (particle area in nanometers=x, $SQRT[x/\pi]^2$). This diameter data was compiled to create size distributions showing the size fractions collected by each NP sampler. The statistical difference in the number of particles collected by the samplers in a certain particle size bin was compared by calculating chi square values and two-tailed p values to determine the significance of those differences. The data from three runs for each aerosol were averaged to create one data set for the comparisons reported in this study.

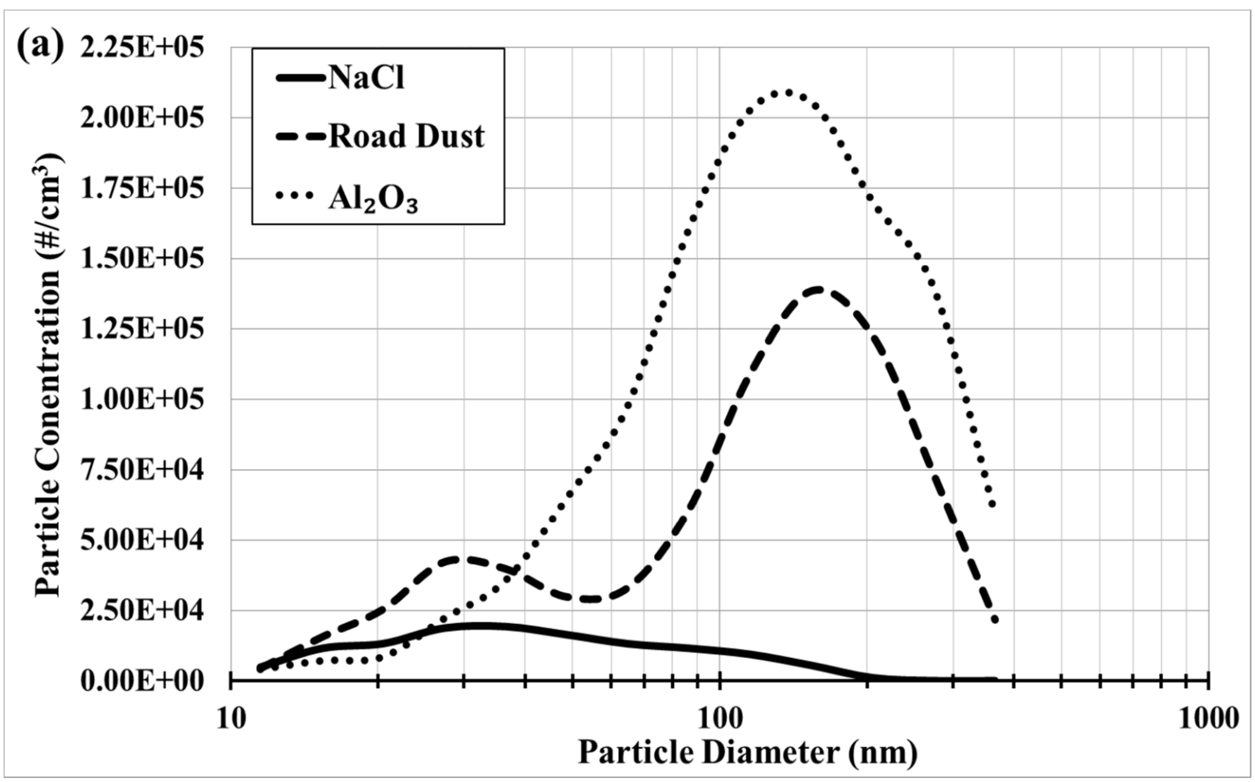
2.4 Usability Analysis

A Questionnaire was designed to evaluate the ease of use and effectiveness of each of the three NP samplers using both quantitative and qualitative measures. The questionnaire covered five categories including: (1) the device interface, (2) the process of sampling, (3) durability and reliability, (4) effectiveness at collecting particles, and (5) affordability. This survey was completed by the student researchers. The scoring system is based on a four-point scale which rates the perceived room for improvement in every category. A ranking of four indicates no room for improvement, while three through one indicate room for little, moderate, or large improvement respectively. A total of 27 items were evaluated in the survey and the scores in each category were averaged to compare sampler performance. All items were positively worded so that a higher score indicated better performance in that category. Conclusions were then made about the strengths and weaknesses of each sampler. The TDS does not have a digital interface like the ESP and TPS, but because the TDS requires the use of a sampling pump to operate, the functionality of the pump used in this study was evaluated. Accordingly, some scores for the TDS will differ based on the features of the pump chosen. Evaluating the sampling pump for direct comparison with the ESP and TPS is justified because a sampling pump must be used to operate the TDS and the pump chosen in this study is representative of ones commonly used in industrial hygiene practice.

The device interface category consisted of items relating to the buttons, the screen, the menu, and the complexity of preparing the device for sampling. The process of sampling and the steps taken immediately after were accounted for in the ease of placing a grid in the holder and loading the device, and the suitability of the device to be used for personal and area sampling based on the adjustability of sampling parameters and size of the device. Additionally, the process of removing grids from the device and maintaining the sampler were addressed in that category. Durability and reliability were measured by the ruggedness of the sampler and its storage container, changes in the device functionality over several uses, and the need for maintenance. The effectiveness rating was based on the number of particles deposited per grid space for each aerosol. Lastly, affordability was measured by the cost of the device and the materials needed to sample with it. Justifications for the scores given are also listed in the questionnaire to give context for the appropriateness of the score and address the possible appearance of bias. The finished survey with scored items and explanations are listed in the supplemental materials section.

CHAPTER 3: RESULTS

Particle statistics measured by the SMPS and OPS were normalized for size bin width ($dN/dlogD_p$). Background particle concentrations immediately before experiments were a maximum of between 3 and 74 particles per cubic centimeter ($\#/cm^3$) for the SMPS size range of 10 to 420 nanometers (nm) and 0.1 to 4 $\#/cm^3$ for the OPS size range of 0.3 to 10 micrometers (µm). A comparison of the total concentrations (TC) measured by the (a) SMPS and (b) OPS are shown in Figure 2. Examples of TEM images used in calculating sampler size distributions are shown below in Figure 3. The particles shown in these images are representative of the particles which were used in this study.



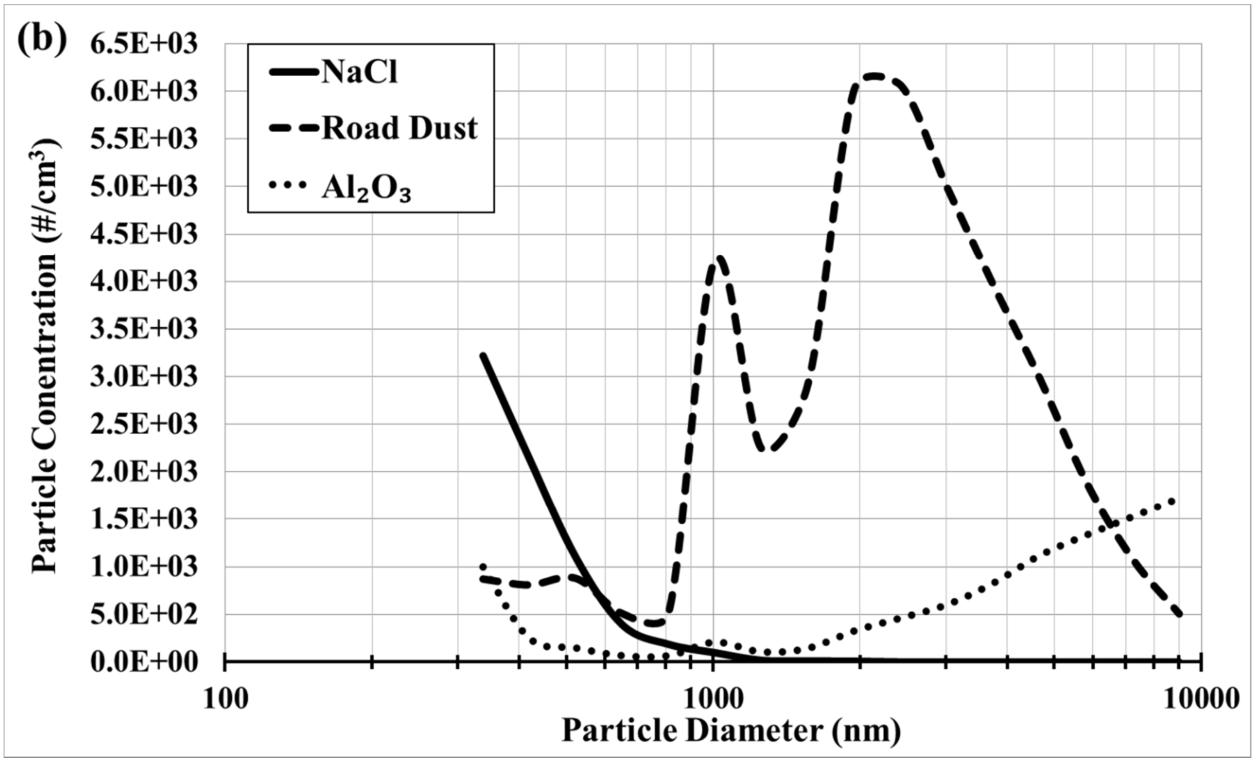
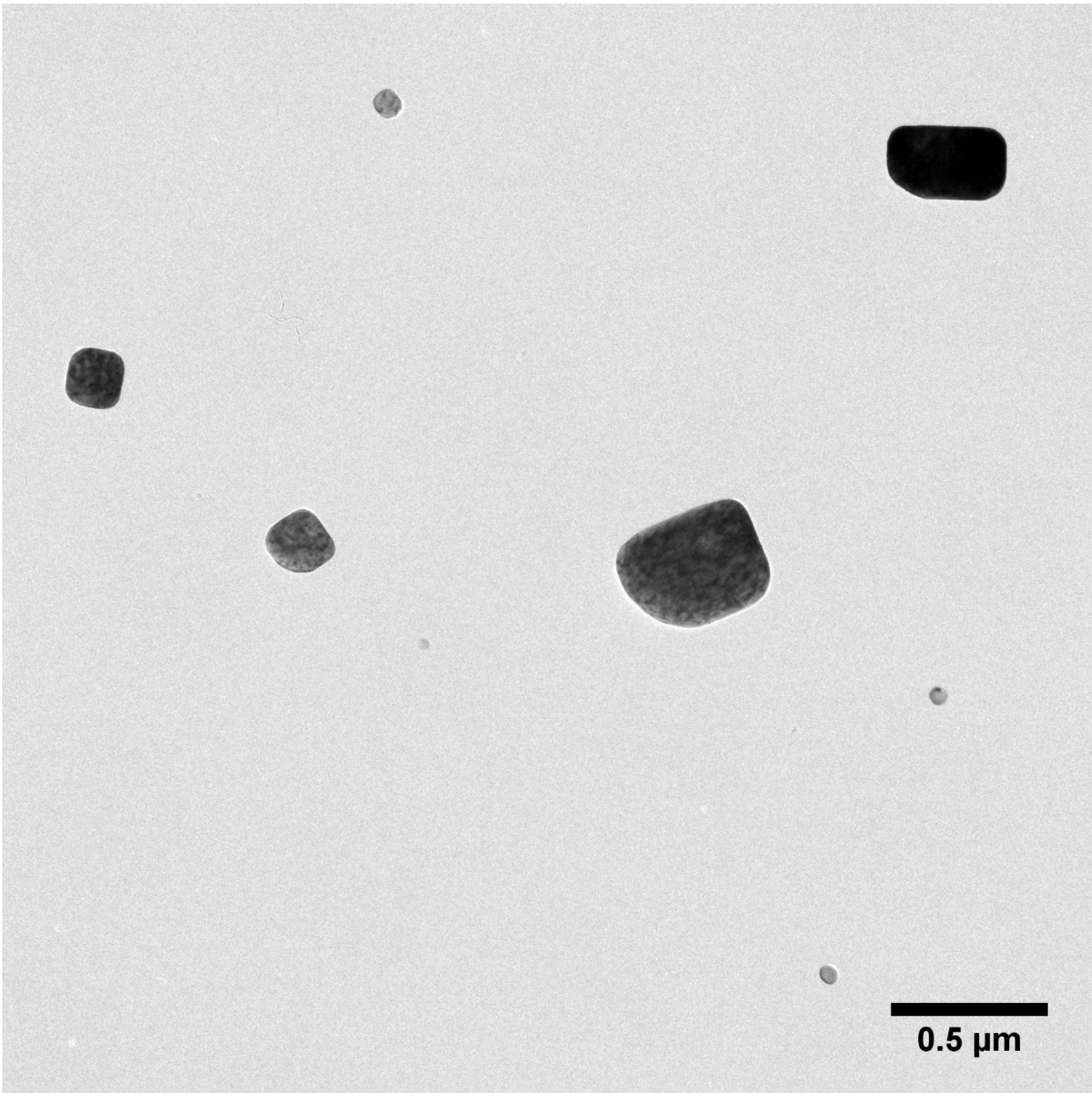
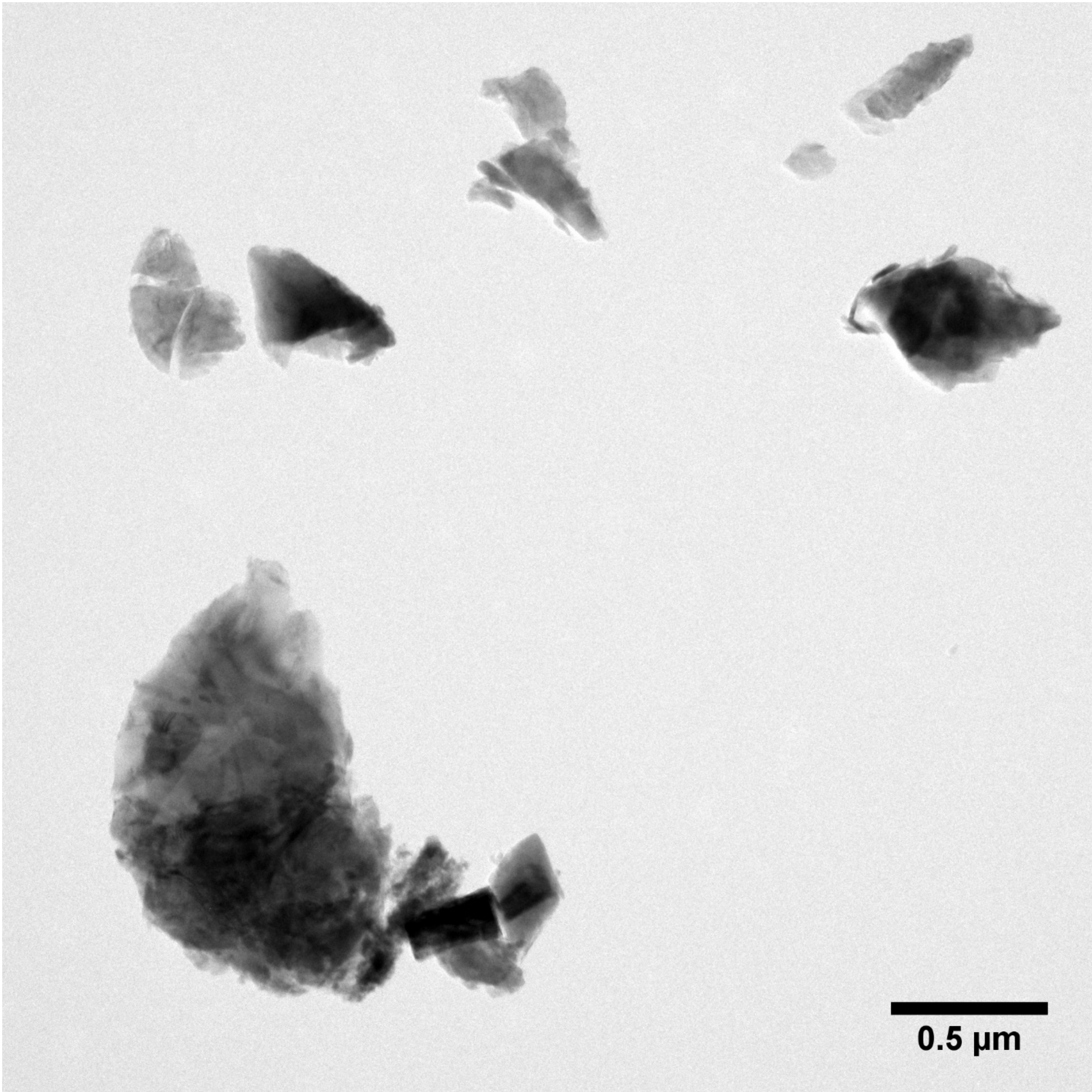


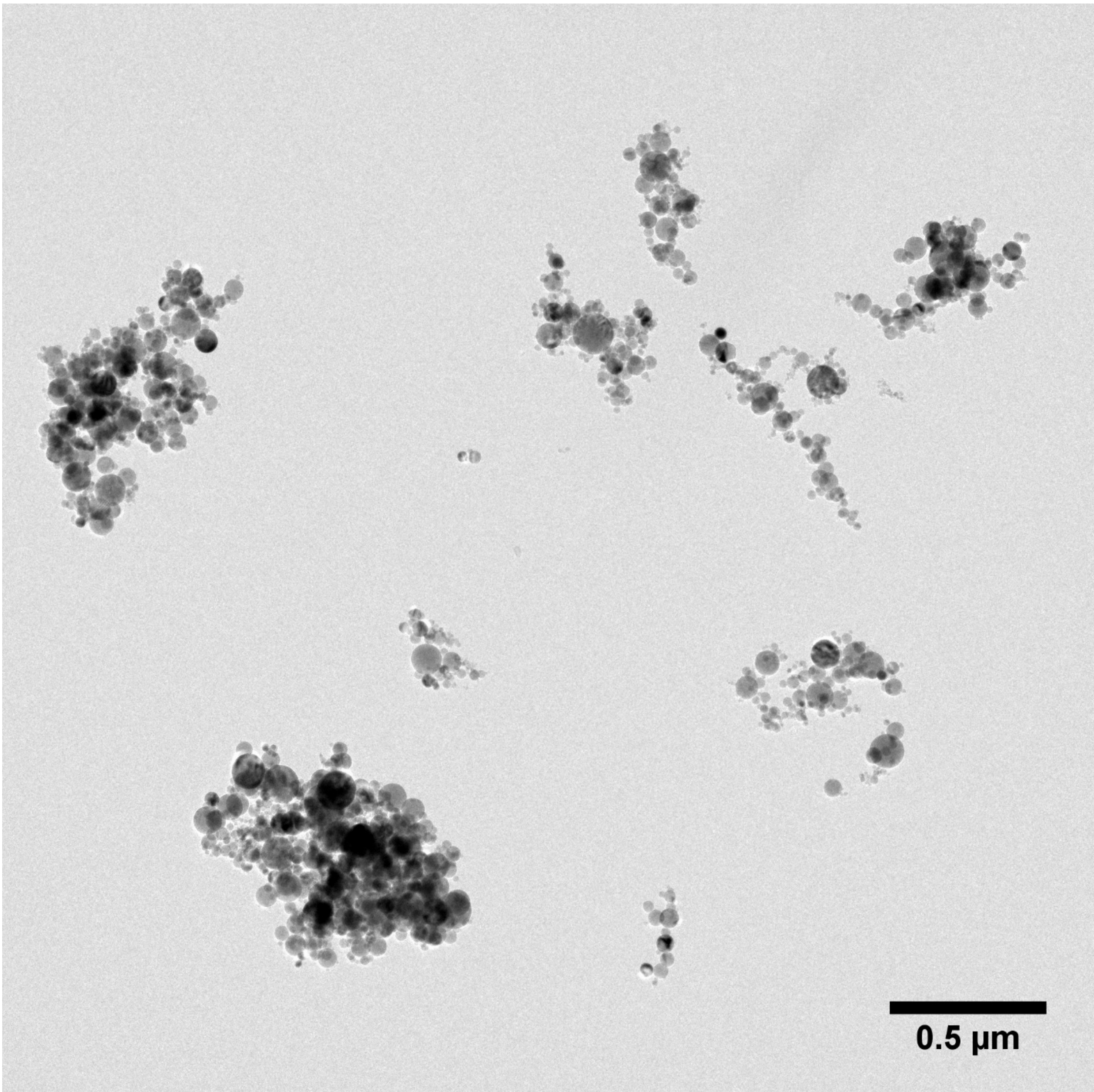
Figure 2 – Total particle number concentrations measured by real time instruments.
Note: The SMPS size range of 10–420 nm is shown in (a) and the OPS range of 0.3-10 μm is shown in (b). Sodium chloride (NaCl), road dust (RD) and aluminum oxide (Al_2O_3) are abbreviated. The standard deviations (SDs) for the total concentrations (#/cm3) measured by the SMPS in each size bin, between the three runs for each aerosol were 1,270 (NaCl), 37,114 (RD), and 15,203 (Al2O3). SDs for the OPS were 43 (NaCl), 443 (RD), and 150 (Al2O3). These values describe how similar the number concentrations of each particle size were between the repetitions conducted for each aerosol.



(a) NaCl



(b) Road Dust

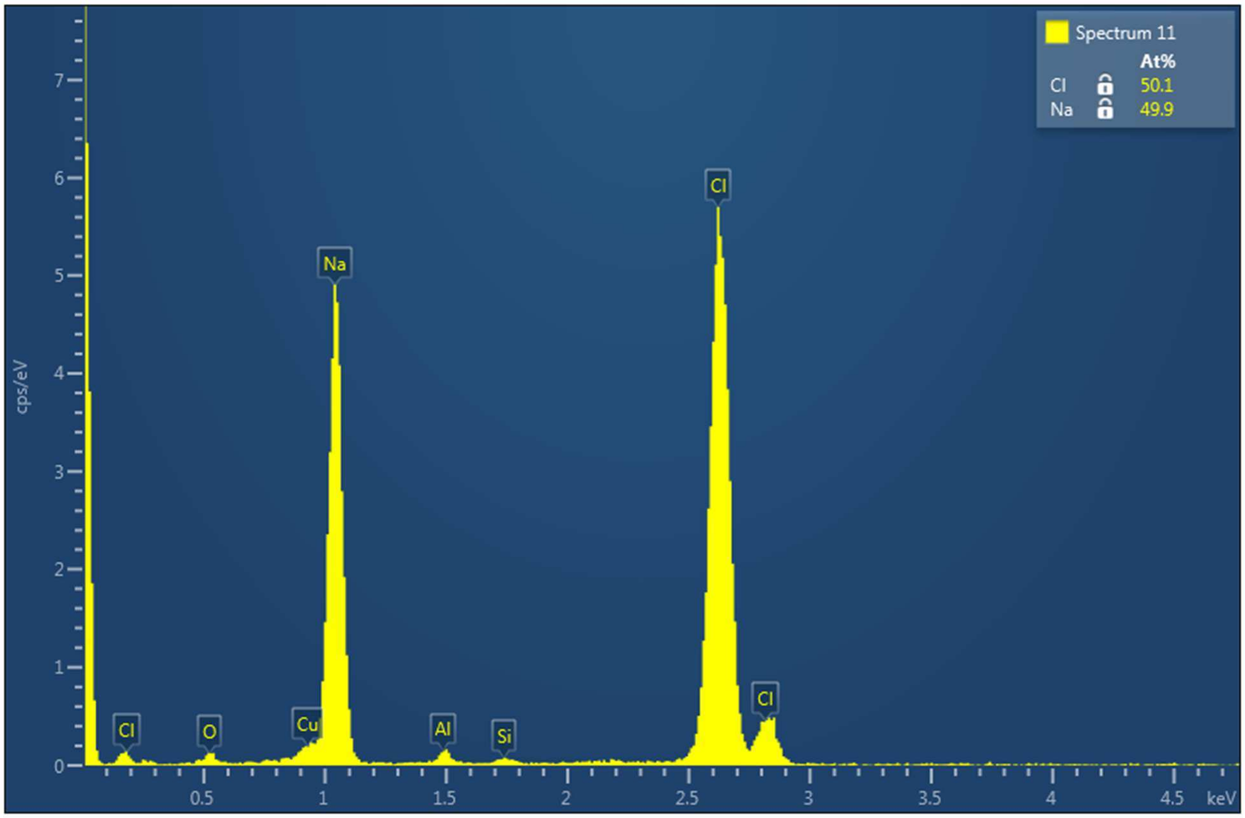


(c) Al_2O_3

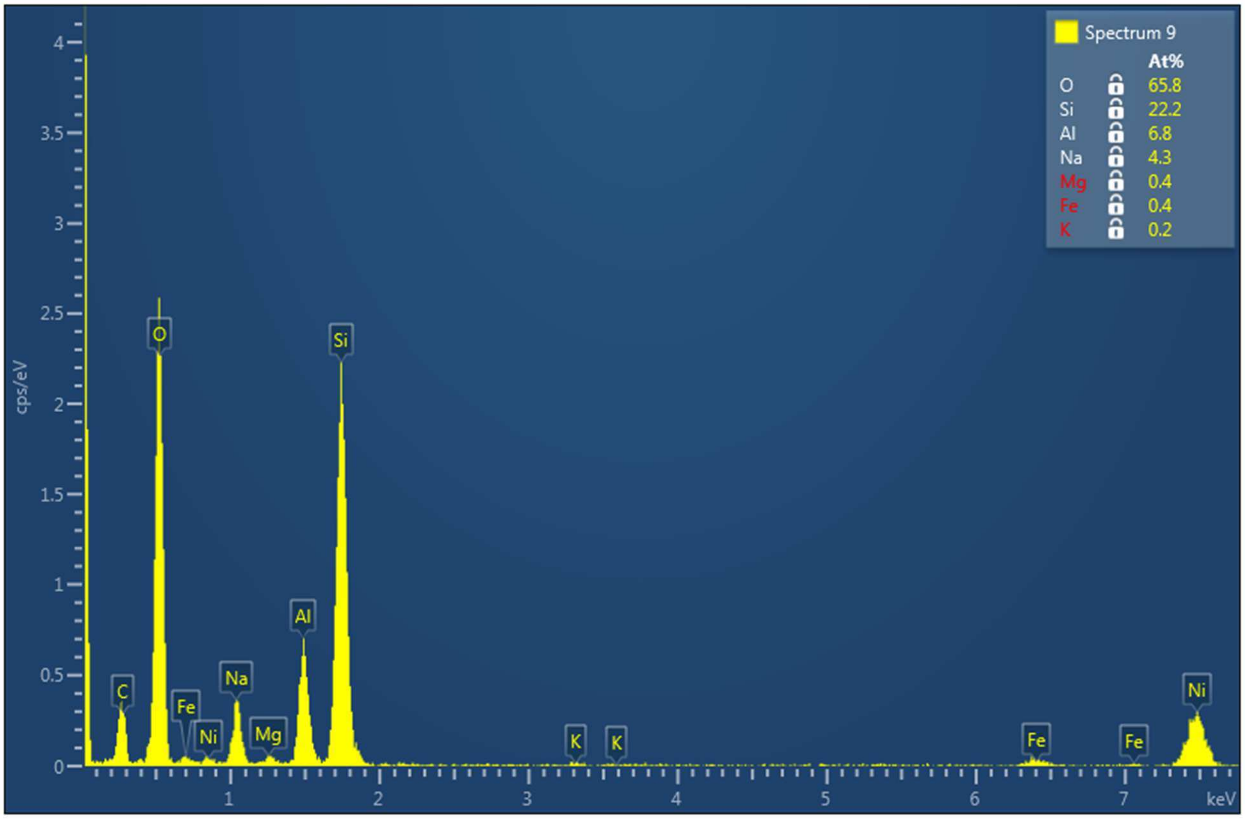
Figure 3 – TEM images of different particle types used in FIJI analysis.

Note: NaCl (a) did not show agglomeration, while Road Dust (b) and Al_2O_3 (c) displayed significant agglomeration.

The elemental composition of particles collected by each sampler was evaluated through EDX and results are shown below in Figure 4. EDX results indicated that particles which were representative of those used to construct sampler size distributions, had compositions which matched the aerosols created for this experiment. This gives confidence that the particles collected by the samplers came from the contamination source of interest and were not background or extraneous particles.

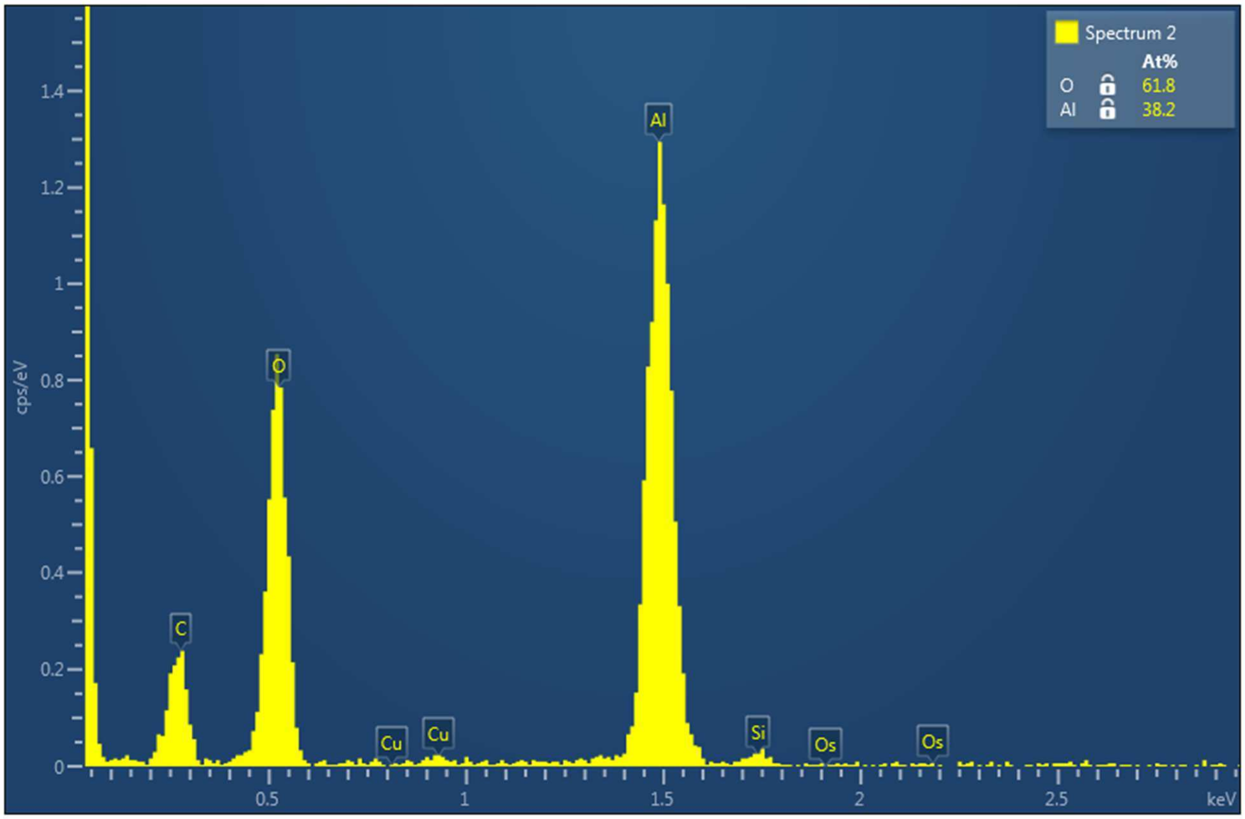


(a) NaCl sample collected by the TDS.



(b) Road Dust sample collected by the TPS.

Note: TPS used a nickel grid. ISO test dust specifications list ingredients by percentage in the order of silica and aluminum oxide, followed by oxides of iron, calcium, potassium and sodium.

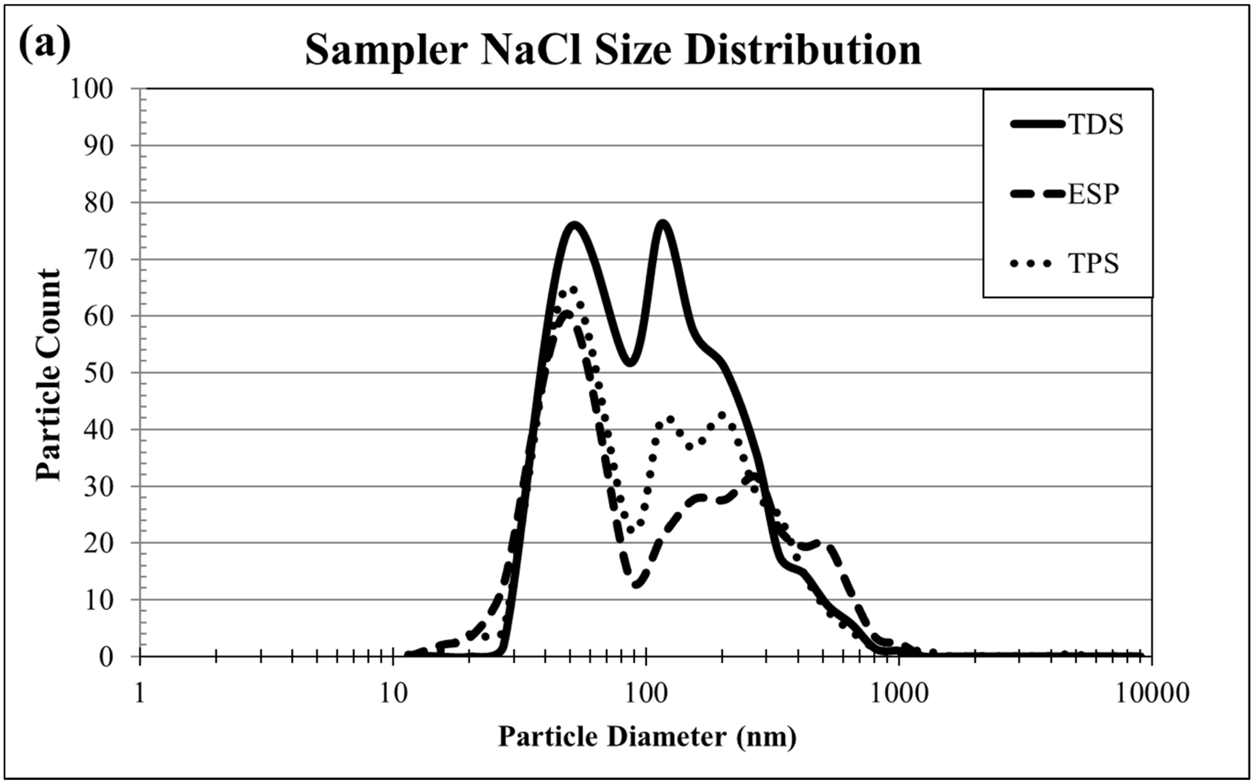


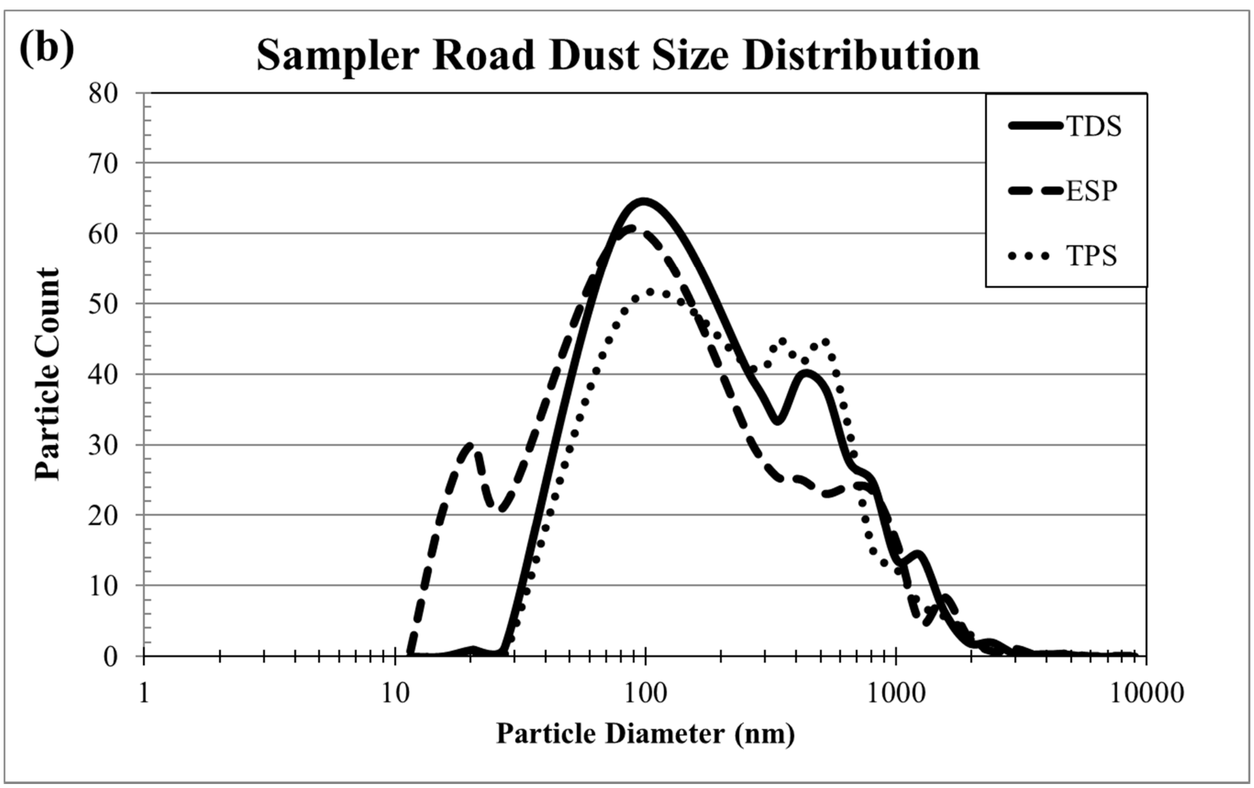
(c) Al_2O_3 sample collected by the ESP

Figure 4 – EDX Analysis of TEM Images.

Note: All TEM grids used pure carbon coating so some carbon is seen on the EDX graphs.

A comparison of the size fractions collected by each NP sampler is shown below in Figure 5 and represent data averaged over all three runs conducted for each aerosol type.


(a)
Sampler NaCl Size Distribution
Particle Count
Particle Diameter (nm)
TDS
ESP
TPS
0
10
20
30
40
50
60
70
80
90
100
1
10
100
1000
10000


(b)
Sampler Road Dust Size Distribution
Particle Count
Particle Diameter (nm)
TDS
ESP
TPS
0
10
20
30
40
50
60
70
80
1
10
100
1000
10000

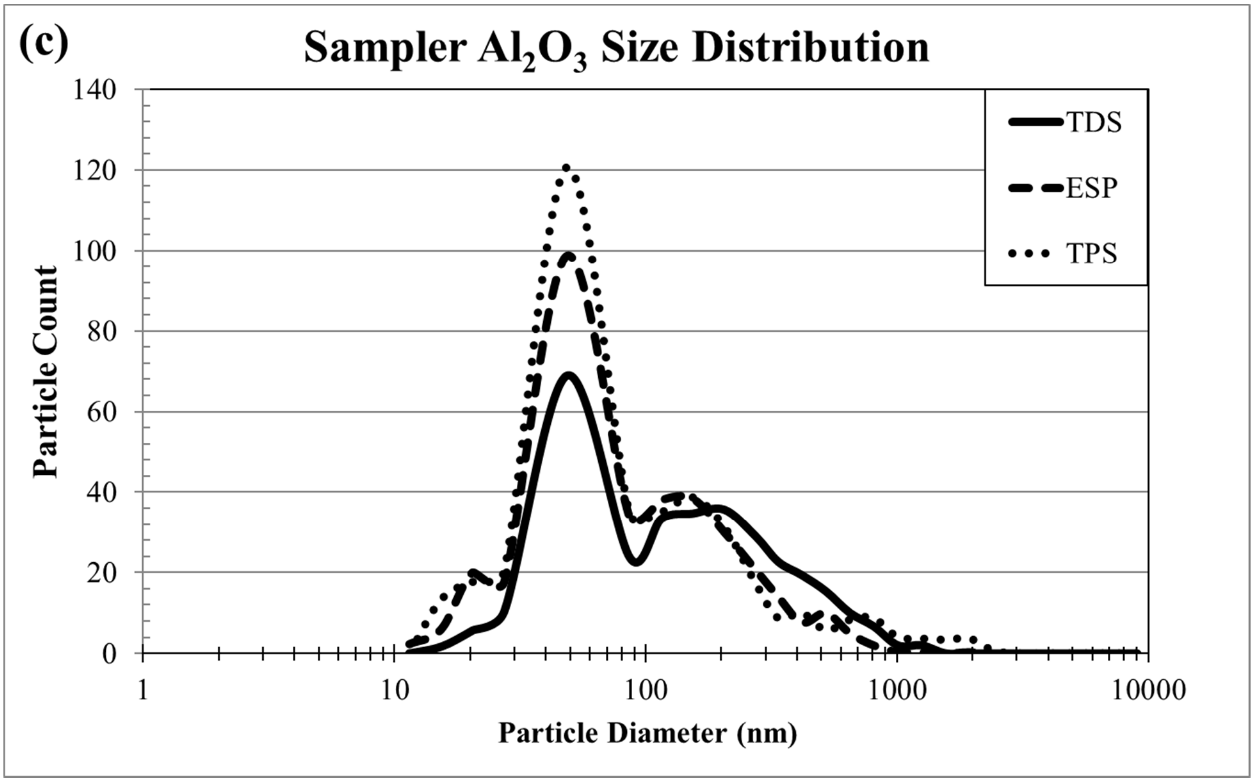
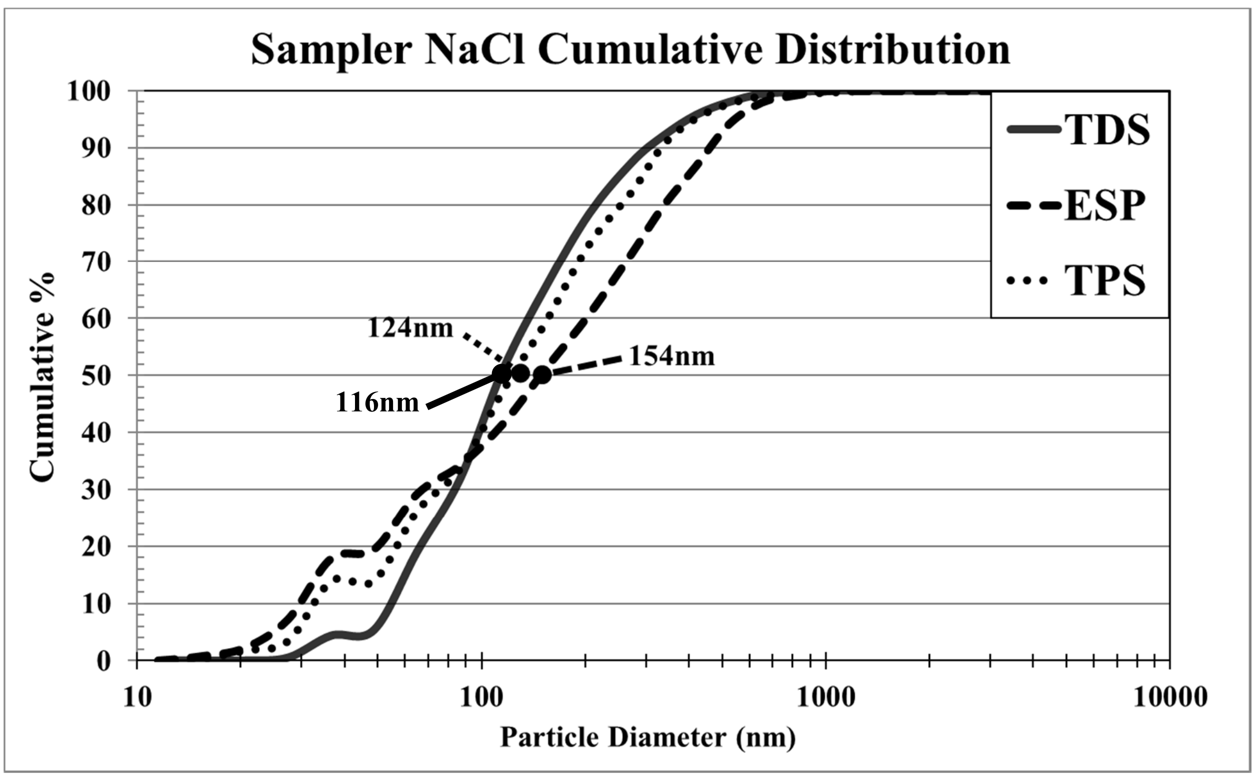
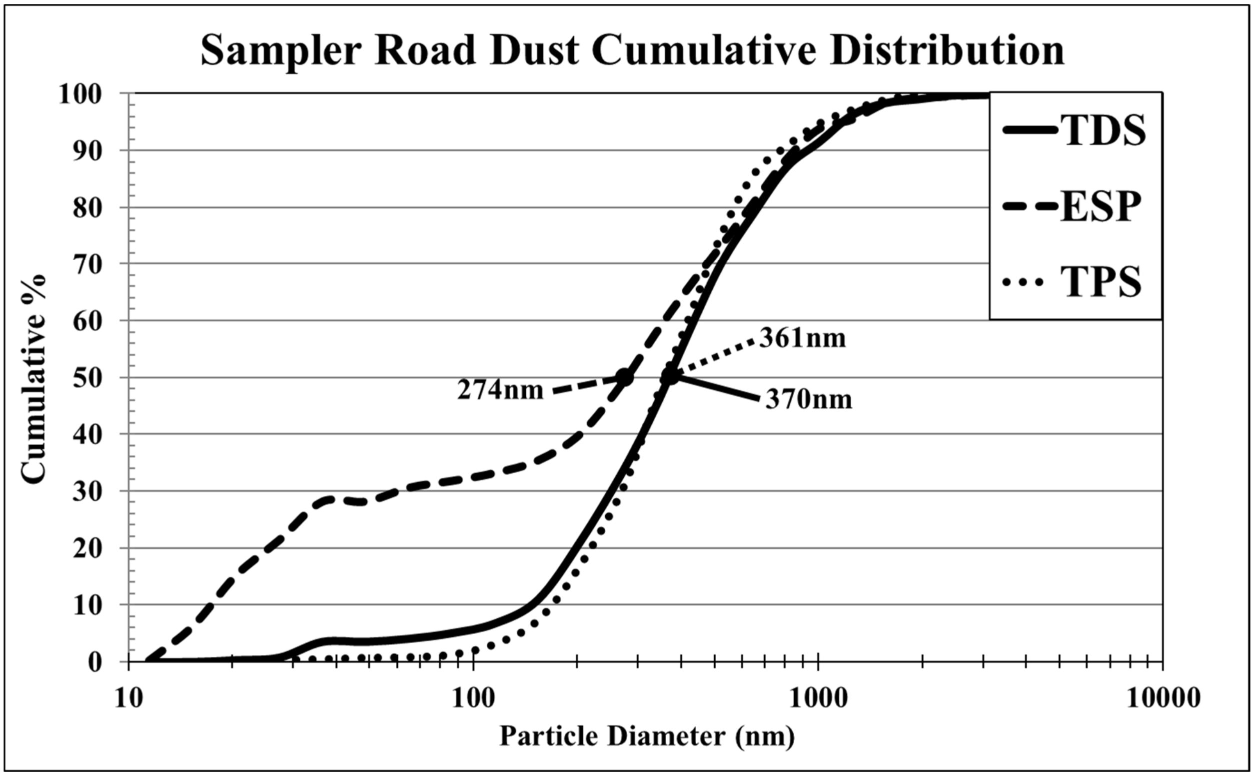


Figure 5 – Sampler size distributions from TEM image analysis for all aerosols.



Note: Median diameters were 116 nm, 124 nm, and 154 nm for the TDS, TPS and ESP respectively.



Note: Median diameters were 361-370 nm for TDS and TPS, and 274 nm for the ESP.

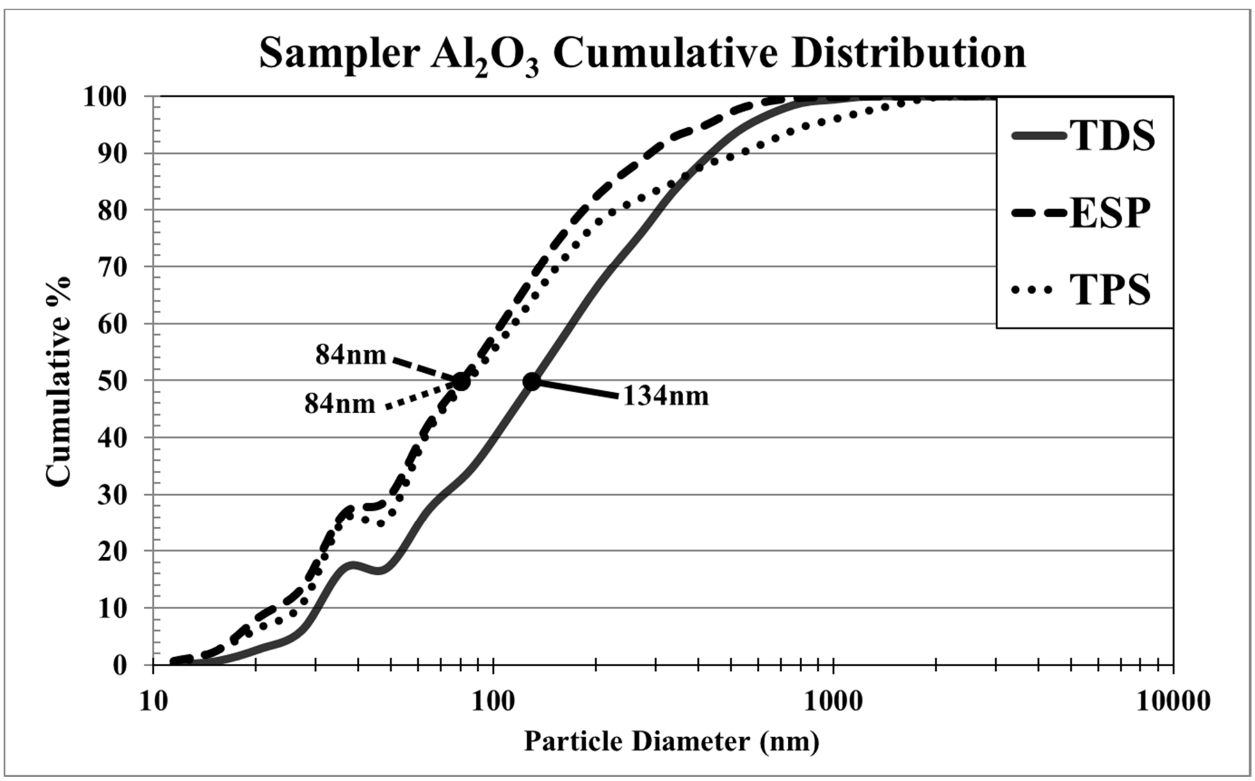


Figure 6 – Sampler cumulative distributions from TEM image analysis for all aerosols.
Note: Median diameters were 84nm for the ESP and TPS, and 134nm for the TDS.

Measurements presented from RTIs and NP samplers are an average taken from three experiments for each aerosol. An important trend shown in Figure 5 above is that the samplers did not collect many particles larger than 2000 nm. This shows that these samplers were not able to capture all aerosol particles on TEM grids which pose a threat to worker health. Particles larger than 2 µm can still deposit in the respiratory tract and cause health effects, so these NP samplers must also be used with devices which can capture particles larger than 2µm for characterization. However, the TDS is an exception because it was designed to simultaneously collect larger particles on a polycarbonate filter. Above in Figure 6, cumulative size distributions are shown for the samplers for each aerosol and the median particle diameters are also given below each figure.

3.1 Sodium Chloride

The total concentration was 15,539 $\#/cm^3$ for the SMPS and 690 $\#/cm^3$ for the OPS. The SMPS mode diameter of 36.5 nm was smaller than the geometric mean (GM) diameter of 41 nm. The OPS displayed the same trend with a mode of 337 nm and a GM of 417 nm (Table 1).
The number of particles imaged for analysis included 1194 for the TDS, 835 for the ESP, and 904 for the TPS. The GM particle diameters collected by the samplers were between 120 and 134 nm, as compared to an SMPS mean of 41 nm. The SD of the diameters of particles collected by the TDS was 122 nm, 176 nm for the ESP, and 182 nm for the TPS (Table 1). All three samplers had two mode diameters, one at 48.7 nm and the other between 115 and 205 nm. The TDS collected an equal number of particles at both modes, which contrasted with the ESP and TPS collecting fewer particles at their larger modes (Figure 5a). The ESP median diameter (d_{50}) was 206 nm with the other samplers having theirs at 154 nm. This indicates that the ESP collected fewer smaller particles (10-200 nm) than the other samplers which collected half of their distribution under 154 nm. For particles 49 nm and smaller the ESP and TPS collected 14% and 19% of their total

distribution as compared to the TDS which collected 5%. For larger particles, the TDS and TPS performed similarly collecting 92% and 90% of particles 337 nm or smaller. The ESP differed in collecting 80% of particles below that size, indicating it collected a larger proportion of its particles above 337 nm as compared to the other samplers. On two out of the three runs, all samplers collected no particles between 37 and 49 nm, despite the SMPS measuring the highest number concentration in that size range. At 48.7nm the TDS collected more particles than the ESP ($p<0.38$) and TPS ($p<0.37$). The most significant difference between the samplers was at 115 nm, with the ESP collecting 7.5% of total particles at that size as compared to the TDS collecting 19% ($p<0.00002$). A smaller difference was found between the TDS and TPS at 115 nm ($p<0.07$). Cumulative distributions from the samplers among all aerosol types are shown in Figure 6.

Table 1 - Particle concentrations and counts from real time instruments and samplers.
Note: values represent an average of three runs.

Real Time Instruments		NaCl	RD	Al_2O_3
SMPS	Geo. Mean (nm)	41	106	133
	Mode (nm)	37	154	154
OPS	Geo. Mean (nm)	417	2236	2983
	Mode (nm)	337	2421	9016
Samplers				
TDS	Geo. Mean (nm)	120 (122)	349 (363)	125 (178)
	Mode (nm)	116	87	49
	Median (nm)	112	367	129
ESP	Geo. Mean (nm)	134 (176)	224 (365)	84 (135)
	Mode (nm)	87	87	49
	Median (nm)	139	250	83
TPS	Geo. Mean (nm)	122 (182)	362 (273)	101 (282)
	Mode (nm)	87	87	49
	Median (nm)	125	369	83

3.2 Road Dust

The SMPS total concentration for road dust was 126,795 #/cm^3 and 3,839 #/cm^3 for the OPS. The three-run average particle concentration SD was largest for this aerosol type, with the SMPS measuring 37,114 #/cm^3 and 443 #/cm^3 for the OPS. The SMPS and OPS modes at 154 nm and 2.4 μm were comparable to their respective GM diameters of 106 nm and 2.2 μm (Table 1). The number of particles analyzed was 923 for the TDS, 950 for the ESP and 897 for the TPS. Median particle diameters for the samplers were all smaller than the GM diameter, with the ESP having the largest difference. Particle sizes also varied the most with this aerosol, with SDs of 363, 365, and 273 nm for the TDS, ESP, and TPS respectively (Table 1). The median diameters for the TDS and TPS was 337 nm, and was 274 nm for the ESP. The largest visual difference in the cumulative distributions shown in Figure 6 was for the ESP at a size range below 150 nm. Overall, the road dust cumulative distribution showed the largest visual variation in the size fractions the samplers collected as compared to the other aerosols. The smaller median diameter for the ESP illustrates it captured more smaller particles. More specifically, the ESP collected 30% of its the total particle distribution at 87 nm or smaller, as compared to the TPS collecting 1% of its total particles below 87 nm. The TDS collected 3% of total particles below 116 nm for road dust. All the samplers displayed a mode of 87 nm with the ESP having an additional peak between 10 and 49 nm. At the mode diameter, the largest difference between the samplers was between the TDS and ESP ($p<0.63$). The most significant difference between the samplers occurred at 20.5 nm, the ESP collected 9.5% of its particles at this size with the TDS and TPS collecting less than 0.4% ($p<0.0000002$). The ESP collected 7.9% of particles at 419 nm as compared to 13% ($p<0.037$) and 13.9% ($p<0.014$), for the TDS and TPS respectively.

3.3 Aluminum Oxide

The SMPS total concentration was 164,605 #/cm^3, with the OPS measuring 887 #/cm^3. The OPS GM diameter of 3 μm was markedly lower than the mode of 9 μm (Table 1).
The number of particles collected for analysis was 963 for the TDS, 1035 for the ESP, and 842 for the TPS. The median diameter for all samplers were between 83 nm and 129 nm and their

mode diameter was 49 nm. The TDS had a cumulative distribution which was consistently skewed larger than the other two samplers which had similar distributions. At 49 nm the TDS collected 17% of its particles at that size or smaller. Which compares to the TPS collecting 26% and the ESP collecting 29%. At a larger particle size of 337 nm a different pattern emerged of the TDS and TPS collecting 83-84% of particles at that size or smaller. The ESP however cumulatively collected 93% at that size. This illustrates the TDS and TPS collected more particles above 337nm as compared to the ESP which only collected 7% of total particles above that size. The GM diameters for all samplers were between 84 nm to 125 nm. At 20.5 nm the ESP and TPS collected nearly the same number of particles but there was a larger difference between the TDS and ESP ($p<0.009$) because the TDS collected fewer particles at that size. The TDS collected 21.5% of particles at 48.7 nm as compared to the ESP and TPS collecting 28.6% ($p<0.03$) and 31.3% ($p<0.003$). Among all aerosols, the samplers captured the most comparable size distributions with aluminum oxide.

3.4 Usability

The usability analysis was conducted using a questionnaire addressing sampler interface, procedures required for sampling, post-sampling activities, durability and reliability, effectiveness, and affordability. The full questionnaire is shown in Table S1 in Appendix A. For each item in the questionnaire, a justification for the score was given and a suggested improvement to address the deficiency was proposed. The questionnaire categories each contained between two and seven individual items. All items were scored on a one to four scale with four indicating no need for improvement in that category. The average score for each category and the breakdown of the percentage of scores falling into each scoring grade is listed in Table 2.

Table 2 – Sampler usability and effectiveness measures.

Usability Category	TDS	ESP	TPS
Interface	3.0	3.8	3.3
Sampling	3.2	3.0	3.8
Durability/Reliability	4.0	3.4	3.8
Effectiveness	3.3	3.7	3.3
Affordability	4.0	3.0	3.0
Average score	**3.5**	**3.4**	**3.4**
Score Distribution			
4	59%	44%	66%
3	22%	41%	30%
2	15%	11%	4%
1	4%	4%	0%
Particles per grid space			
NaCl	194	47	50
Road Dust	128	231	113
Al_2O_3	97	345	213

Note: A score of 4 indicated no room for improvement, and a score of 1 specified large room for improvement. Particles per grid space are an average of three runs for each particle type.

The device interface category addressed the buttons and switches, the visibility of the text on the screen, and how easy it was to learn the menu options. The pump used with the TDS required some dexterity to manipulate a small power switch under a rubber membrane and a tiny calibration screw. Pushing any button on the ESP would turn on the device, allowing it to be turned on inadvertently which happened during an experiment. The menu for the ESP was easy to navigate and required few inputs. The TPS menu was also easy to navigate but required several inputs before sampling could begin, such as environmental conditions and device settings. The increased amount of inputs the TPS required resulted in a lower score in the interface category. The screen on the TPS has black text and in low light conditions was difficult to see. The ESP had a screen with green text on a black background that provided better contrast under all lighting conditions. The ESP earned the highest score in this category and the TDS and ESP were tied a few points below.

The sampling category encompassed the process of preparing the device for sampling, the activities conducted during sampling, and the actions required post-sampling. The TPS earned the highest score in this category. Issues with the TPS in this domain were that it had raised edges which made cleaning the device more difficult and that the performance consequence of adjusting the hot and cold plate temperatures was not explained in the manual. The main drawbacks of the TDS were: (1) the pump needing to be manually turned off after the sampling period, (2) delicateness required for removing the TEM grid from the filter, (3) the need of cleaning the filter cassette, and (4) the lower quality storage case that came with the sampling pumps. Two of these issues can be remedied by using a higher quality sampling pump, and the commercially available version of the TDS may use a more user-friendly attachment to secure and release the TEM grid. The central issue with the ESP was the difficulty in placing TEM grids onto the sample holder. This required the user to center the grid exactly under three sides of tape. The tape must also be flattened to be flush against the plastic grid holder. If a small edge of the tape sticks up, the tape will be sheared back and the grid damaged when the sample holder slides into the tight internal cavity of the ESP. On several occasions, it took more than one attempt to successfully load the ESP with a TEM grid. Furthermore, the ESP was limited in sampling duration and allowed for a collection period of between 1-500 seconds. This would not permit for a full-shift sample if the shift is longer than about 8 mins. Removing the sample holder from the ESP requires a large amount of force as the two have a tight fit, and this can result in a sudden jerk once the holder releases from the device. The ESP does not come with rechargeable batteries and requires the user to unscrew the top cover to replace the batteries. The storage cases for the samplers were rated and the TPS case offered the best protection, with the ESP and TDS pump case being less user friendly and offering less protection.

The relative durability of the samplers was evaluated through items relating to the sampler being vulnerable to external and internal damage and if maintenance was required. The ESP and TPS were rated equally in terms of their durability because the shells to the devices resisted scratching and chipping during their use and because their screens were protected with a plastic window. The vulnerability to fall damage was evaluated, and the TDS sampler was rated to be inherently more resistant to fall damage than other two samplers because it has no electronics and is made from a single lightweight material. The reliability of the sampler in holding the TEM grid in place was assessed. The TDS and TPS had no issues when removing the sample holder after an experiment, the grids did not move from their original position. Use of the ESP caused problems in this regard because of the tight fit of the sample holder into the ESP. Sometimes the grid would be damaged while pushing the sample holder into the ESP or when pulling it out. The cause of this issue is the tape used to secure the TEM grid, and the small clearance between the sample holder and the chamber it slides into.

The overall affordability of the samplers was compared by contrasting their initial purchase cost and the costs of the supplies required to sample. The TPS and ESP both cost between $6000 and $7000. The TDS sampler requires a sampling pump and a special 25 mm cassette. The cassette costs $11 each, and a sampling pump that operates up to 3 liters per min was $800. This puts the package cost of the TDS with sampling pump at $811, with some variation depending on the cost of the sampling pump chosen. The recurring costs of the samplers include batteries for the ESP, TEM grids for all three samplers, and 25 mm filters for the TDS.

The ESP and TDS used copper TEM grids while the TPS uses more expensive nickel TEM grids. The TDS has the additional expense of the 25 mm filters but the cost per unit is under $1. The TDS was given the highest score for affordability.

The effectiveness of the samplers was evaluated by comparing the relative particle density collected on the TEM grids. The number of particles collected per grid space was averaged for each aerosol type and is shown in Table 2, with more detailed information available below in Figure 7.

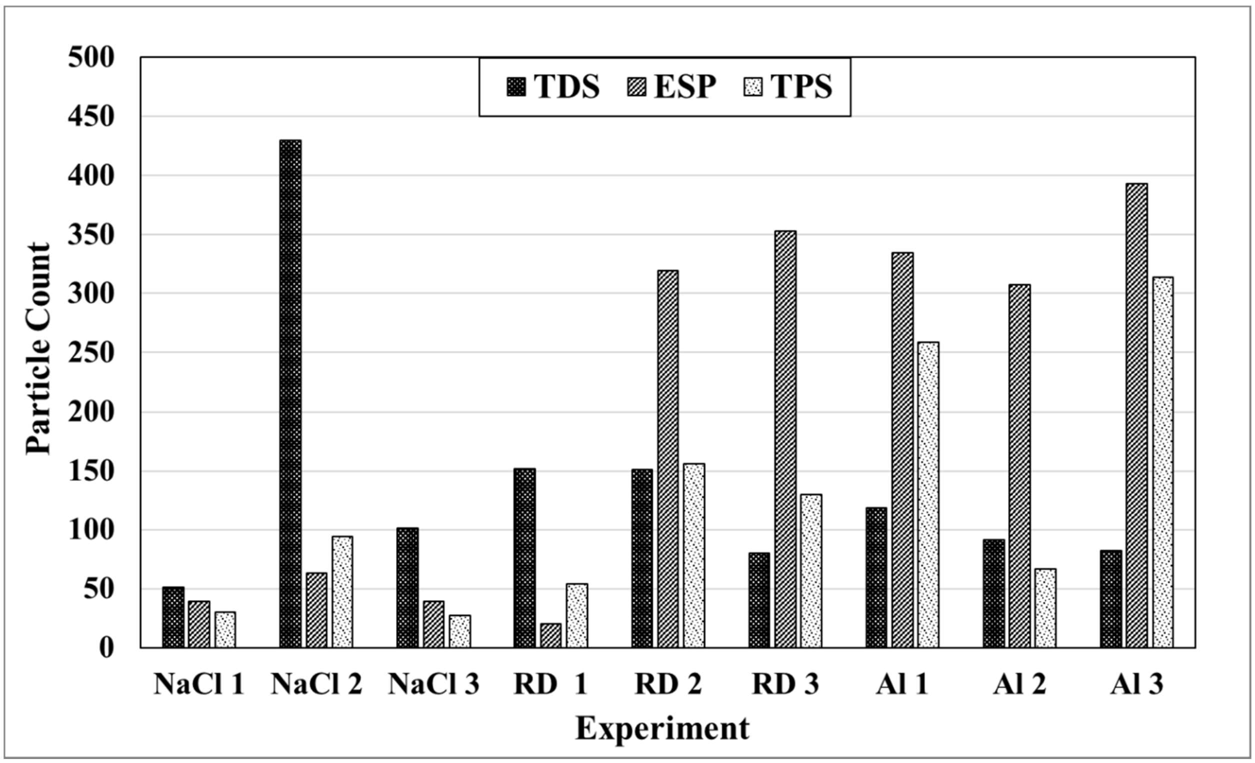


Figure 7- Average Particles Per Grid Space.
Note: The number of particles imaged per grid space was counted for all grids that were completely imaged, and data is shown for each of the 9 experiments conducted.

The TDS was the most effective sampler in collecting sodium chloride particles with the highest particle count per grid space. The ESP and TPS were 25% less effective at collecting this aerosol as compared to the TDS. The ESP had the highest collection efficiency with road dust, followed by the TPS and TDS which collected about 50% fewer particles per grid space. The highest particle count per grid space was collected by the ESP with aluminum oxide. The TPS had its highest collection efficiency with aluminum oxide and the TDS had its lowest.

CHAPTER 4: DISCUSSION

All samplers collected particles under identical conditions. The differences in the size fractions they collected reflect differences in the mechanisms the samplers use to capture particles onto the TEM grid. Moreover, the number of particles imaged for each sampler within or across aerosol types were not equal. Therefore, the number of particles per grid space and the differences in the average size distributions were compared.

The TDS was equally effective in collecting sodium chloride particles at both 48.7 and 115.5 nm, while the ESP and TPS captured about half as many particles at the 115.5 nm size. This indicated the method of particle capture used by the TDS showed a significant difference in collection efficiency at that size range. The diffusion coefficient (equation 1), which describes the rate at which particles diffuse (m/s), is inversely proportional to particle diameter[25].

Equation 1: $$D = \frac{kTC_c}{3\pi\eta d_p}$$

where;

d_p = particle diameter (meters)

This indicates that the TDS should have both a smaller median diameter and particle cutoff size (d_{100}). The electrostatic force applied to particles in the ESP creates a particle velocity (equation 2) which is also inversely proportional to the particle diameter[25].

Equation 2: $$V_e = \frac{neEC_c}{3\pi\eta d_p}$$

where;

e = charge on each particle (coulombs)

E = electric field (Volt/meter)

As a result, the ESP should capture smaller particles with the most efficiency. The thermophoretic force generates a particle velocity (equation 3) in the TPS which is directly proportional to particle diameter[25].

Equation 3: $$V_{th} = \frac{\rho \lambda d_p \nabla T C_c}{3\pi \eta T}$$

where;

∇T = thermal gradient between plates (kelvin)

This indicates the TPS should capture larger particles with greater efficiency.

The sodium chloride cumulative distribution data indicate the TDS did collect the most smaller particles (<116 nm) and the TPS collected the largest particles up to 1.25 µm. However, the ESP had the largest median diameter, indicating it collected the fewer smaller particles than the TDS. In an earlier study the ESP collected sodium chloride particles with the highest efficiency between 30 and 100 nm, and above 300 nm [23]. Particle count size distributions from this experiment revealed an ESP mode diameter in the smaller range around 50 nm. The TPS has been used in several previous studies [28-31], but none systematically used TEM imaging to characterize the size fractions collected. As compared to the other aerosols used in this study, sodium chloride had the lowest SMPS total concentration and mode particle diameter.

The samplers collected road dust at a mode diameter close to that of the SMPS mode. The TDS and TPS had comparable particle size distributions, however the ESP captured more particles under 36.5 nm. In addition, all sampler size distributions had the largest particle SDs with road dust. Cumulative size distributions revealed the ESP had the smallest median diameter which follows with the theory. The TDS had the largest median diameter, collected fewer smaller particles, and had a larger particle cutoff diameter around 4.6 µm; all of which did not follow with the expected results based on the theory of operation. The TPS also had the smallest cutoff diameter around 2.4 µm.

The TDS, ESP, and TPS collected comparable size fractions of aluminum oxide, with the most significant difference being the TDS collected a smaller percentage of aluminum oxide particles at the mode diameter. The sampler cumulative distribution indicated the TDS had the largest median diameter, again disagreeing with the theory of operation since it collected fewer smaller particles. The particle cutoff diameters did reveal that the TDS and ESP had a smaller cutoff and the TPS had the largest, this result did follow with sampler theory. Across all samplers and particle types, a maximum of 1% of particles were larger than 1.9 µm. If there is a need to characterize particles larger than this, the 25-mm filter used with the TDS could be analyzed with a scanning electron microscope. Then the size, shape, and elemental composition of those larger particles could be identified.

The effectiveness of the samplers at collecting each aerosol was measured by the number of particles imaged per grid space, for grids that were completely imaged. By this measure the ESP was remarkably effective by collecting the highest particle deposition distributions for road dust and aluminum oxide, but was the least effective among all the samplers for sodium chloride. This is likely the result of low concentrations of sodium chloride and the sampling time of 50 seconds. The TDS collected the most sodium chloride particles which were in the lowest SMPS concentration and mean diameter. The TDS collected the lowest particle deposition distribution with aluminum oxide, but the ESP and TPS did best with this particle type.

For usability, the TDS received deductions in the sampling score due to drawbacks of the sampling pump and the carefulness required to remove a TEM grid secured with tape. The TDS did allow flexibility by allowing the user to choose an appropriate sampling pump. The ESP is the least complex and most effective device to use. Its main shortcoming was being difficult to load with a TEM grid, followed by having limited sampling periods which make it unsuitable for work shift sampling. The TPS was easy to sample with. Its main weaknesses included the quantity of menu items and the lack of screen contrast.

CHAPTER 5: CONCLUSION

The analysis of usability and particle collection used in this study was in both qualitative and quantitative terms, and can be used together to describe the usefulness of each sampler for different applications. The TDS is relatively inexpensive, more durable, and more versatile because it can collect both larger particles on a polycarbonate filter and smaller particles on a TEM grid. The ESP was very effective at collecting particles during a short period and in higher quantity. The TPS excelled at making the process of sampling easy and repeatable. It had a clip for ease of personal sampling, and was flexible in allowing for the adjustment of sampling parameters which also meant a more complex menu. The TDS required the use of a sampling pump which needs calibration, adding to the complexity of the sampling process. The ESP was difficult to load for sampling due to the method of securing TEM grids to the holder. The TPS requires environmental temperature and humidity readings and a short warm up period before sampling can start, which increases the effort needed for sampling. Overall the TDS is better suited for short and long-term sampling. The ESP is best suited for short term sampling and identifying sources of particles. The TPS is better for long term sampling. The TDS is unique in its capability to capture nanometer and micrometer sized particles and would be well suited for sampling environments with several sources of contamination and processes which generate poly-dispersed aerosols.

Analysis of particles with electron microscopy is a costly and time-consuming endeavor. Therefore, it is important that a sampler be as effective, versatile, and affordable as possible. An evaluation of the usability of a personal nanoparticle sampler has not been published in the past. Additionally, previous experimenters have not used TEM imaging to characterize the particle size fractions collected by these samplers and compared their performance. In the future, more research is needed to compare the performance of these samplers in more practical environments, to investigate how the physio-chemical properties of an aerosol affect the size fractions collected by these samplers, and to determine why the RTI peak concentrations did not match the sampler modes in consistent ways. Based on the usability evaluation, minor changes can be made in the design of each sampler to make it easier to use. Further information will be published on the particle collection characteristics of a 3D printed version of the TDS in preparation for its commercial development.

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APPENDIX A

Table S1 – Full usability questionnaire.
Note: Scores range from 1-4 with 4 indicating no need for improvement in that area and 1 indicating room for large improvement. Scores marked with * denote items that were scored for the specific sensidyne pump used in conjunction with the TDS sampling cassette. Use of another pump would significantly affect the respective score.

	TPS Score	TDS Score	ESP Score	Justification TPS	Justification TDS	Justification ESP	Improvement TPS	Improvement TDS	Improvement ESP
Interface									
Turning on/off the device	**4**	**3***	**3**	On/Off switch easy to operate without looking	Sampling pump has sliding on and off knob which is very small and covered by a rubber membrane. Manipulating it with gloves on sometimes required more than one attempt.	ESP has no on button, you just press any button to turn it on. This could lead to the sampler turning on inadvertently while handling it	N/A	Make the switch taller so it is easier to slide with gloved hands (improvement for the pump)	Make center button only one which can activate device and have to hold it down for 3 seconds to turn on or off. This will minimize instances of device being turned on inadvertently.
The buttons provided tactile feedback and could be manipulated while using nitrile gloves	**4**	**2***	**4**	Buttons have adequate separation, provide tactile feedback, and are easy to see	Besides on/off only other control is a small screw used to adjust flow rate. To make a 10 mL/min flow rate change, you must adjust the screw with an imperceivablly small turn.	Buttons have adequate separation, provide tactile feedback, and are easy to see	N/A	Make a screw that clicks and changes the flow rate in discrete units instead of a continuum. Make the screw bigger so the head doesn't strip out as easily	N/A
Visibility of text on the screen	**2**	**4***	**4**	TPS screen has black text on a screen similar to that of a graphing calculator. Amount of contrast between text and background is lacking in low light environments and when viewed at a sharp angle.	Screen on side displays sampling time in minutes, screen is easy to see	Green text on black background has excellent contrast	Illuminate the screen, or use a different screen which has better contrast between text color and screen.	N/A	N/A

Preparing the device for sampling was easy to learn (including navigating the menu, not including placing sample in device)	**3**	**3***	**4**	You must collect temp and humidity data first. Then the TPS has 9 menu steps to go through before you can sample. You must enter the environmental conditions, the cold and hot plate temperatures, then sample duration.	The TDS needs to be prepared for sampling by calibrating the sampling pump which uses an adjustment screw. Must also clean the tubing before each use, which requires distilled water and tubing needs to be completely dry before use.	Just enter the sampling duration and start sampling	Collecting environmental data is important for proper functioning, could integrate humidity and temperature sensors into the device and have it automatically enter those settings	Purchase more expensive pump with easier calibration. Sampling conducted after this project utilized a gil-air plus personal sampling pump. Digital interface allowed for easy calibration and sampling times with automatic shut-off, screen also has illumination. This addressed the main shortcomings of sampling with the gil air 3.	There are two additional menu items after menu option 3 which is when you begin sampling. These are voltage settings and never had to be adjusted.
<u>Sampling</u>									
Placing a TEM grid in the sample holder	**4**	**4**	**1**	Sample holder has a magnet which keeps the nickel grid in place, very easy to center it in the correct position	When the TDS is commercially available it will come with a TEM grid pre-attached to the 25mm filter. The user will not have to place a TEM grid into the sample holder.	Successfully placing a TEM grid in the holder is difficult even after practice. The tape does not adhere very well to the smooth plastic of the grid holder which can allow the tape to slide away from the center of the grid holder. It is very difficult to place the grid in the center of the the tape's edges. The tape must not overlap beyond the periphery of the grid otherwise it will cover the area where particles are collected.	N/A	N/A	Make a small depression in the sample holder the exact size of the TEM grid so it's easier to consistently place in position. Provide TEM grids pre-attached to tape so the user only has to place the grid onto the grid holder.Do not have reusable tape, have single use tape. Use an attachment method other than tape

Placing the sample holder into the device	**4**	**4**	**3**	Sample holder is shaped like a trapezoid, can only insert one way and has very little wiggle room	The filter with TEM grid must be placed in the casette, and the two halves screwed together until tight. Then the cassette is attached to the tube connecting it to the sampling pump.	The sample holder must be pushed into a tight space which can sometimes result in the tape and TEM grid being scraped off or pushed out of position. We had to push up on the bottom surface of the external end of the holder while sliding it in, so that the front end was pivoted down and its top surface did not make contact with anything.	N/A	N/A	Slightly reduce the dimensions of the sample holder so that the top surface where the TEM grid is has more clearance and is less likely to be sheared off.
The device is not cumbersome and can be held with one hand	**4**	**4**	**4**	You can easily hold it with one hand and it weighs 0.7 lb	The cassette is small and light, the sampling pump was designed to be worn in the field. The device weighs 1.3 lb	The device is slightly larger than the TPS but can be held with one hand and weighs 2lb	N/A	N/A	N/A
The device can sample from a worker's breathing zone	**4**	**4**	**3**	The TPS has a clip which can be attached to a shirt or vest	The TDS uses a 25mm cassette and standard sampling pump which are easily attached to the worker, identical to what is widely used in industrial hygiene to sample PBZ	Must be hand held, has no clips or other attachment points	N/A	N/A	Attach a clip to the device or design a harness for the ESP which allows for attachment to the worker
The device is capable of area sampling because it can sample for a variety of time periods	**4**	**4***	**2**	The device can sample over a wide range of times.	The device can sample over a wide range of times	The device can only sample up to 500 seconds	N/A	N/A	Allow for sampling over longer periods
The device collection parameters can be adjusted to avoid under/over loading	**3**	**3***	**3**	Hot and Cold plate temperatures can be adjusted, and the manual gives a range of recommended values.	The sampling air flow rate was adjustable on the pump, allowing for different volumes of	The device has 5 settings for sampling duration to account for different particle	Include information in the manual about how changing hot and cold plate temperatures can	changing flow rate can affect particle collection, can include information on the effect of	Include information about changing certain voltage values, and what effect it will have

the TEM grid (not including sampling time).				However the manual does not indicate how adjusting these values will affect the collection performance. Default settings were used for all experiments.	air to flow past collection media during a certain sampling period.	concentrations. There were other voltage settings but no indication in the manual about if they should be changed and how this would affect performance	change particle collection, i.e. a larger temperature difference will increase particle collection?	increasing or decreasing flow on particle collection.	on particle collection.
Post Sampling									
The device automatically stops after the sampling period is complete	**4**	**2***	**4**	The device automatically stops after the sampling period ends	The device must manually be turned off, but many other models will turn off automatically	The device automatically stops after the sampling period ends	N/A	Use a sampling pump that turns off automatically	N/A
Removing the sample holder	**4**	**4**	**3**	Removing the sample requires a screw to be inserted and the holder pulled out with a small amount of force	Removing the sample requires the user to remove the filter and TEM grid by unscrewing the cassette and using tweezers	Requires considerable force to pull the sample holder from the device, have to apply upwards pressure to the bottom of the holder so the top is pivoted down slightly, and the TEM grid doesn't make contact with the top of the sampler chamber	N/A	N/A	Change the shape of the sample holder or cavity it enters in so that the top surface where the TEM grid is has more clearance
Removing the sample from the holder	**4**	**2**	**3**	There are divets around the TEM grid allowing tweezers to easily grab the edge of the grid	The edge of the grid must be pulled perpendicular to the direction of the tape so the tape comes off without exerting much force on the grid.	Removing the grid requires the tape on both sides of the grid to be pulled up by pushing a tweezer under the tape. Then the grid can be removed	N/A	The use of a special glue on the back which would balance the needs of keeping the grid securely attached and allowing for easy removal	Have a wedge like tool to remove the tape, using tweezers can result in damaging the tape. Have one use tape so that damaging the tape when removing the TEM grid is inconsequential.
There are inlet covers to prevent contamination of sample	**4**	**1**	**4**	There is a plug for when the device isnt being used, and when a sample is in there is a screw in handle that can serve as a plug when needed	With the cassette, only the inlet was the correct diameter for common plugs for 25mm cassettes, the outlet was too small and so the device was placed in a clean zip	There are red caps that fit over the sample containers and the device	N/A	include special tape to cover cassette inlet/outlet, or plugs that fit. The TDS will come with plugs when it is commercially available	N/A

					lock bag to prevent sample contamination				
Replenishing the power source (rechargeable battery vs changing battery)	**4**	**4***	**3**	Device comes with charger, displays voltage level when turned on and before sampling	Device comes with charger	Batteries must be changed by removing the top of the ESP and 4 screws	N/A	N/A	Make battery rechargeable
Cleaning the outside surfaces of the device (TPS has raised surfaces that create hard to reach corners, ESP is very smooth with no recesses)	**3**	**3**	**4**	TPS has raised ends which create corners where particles can collect and require extra care to clean	Sampling pump is easy to clean, the cassette used is more difficult since there are not many flat surfaces, requires extra effort to clean	Flat surfaces very easy to clean	Redesign housing to be flush and smooth for easy cleaning	Cassettes should be designed to minimize edges and grooves which are more difficult to clean and can trap contamination	N/A
The storage case is user-friendly (loud to close, too large, water resistant, good handle)	**4**	**2***	**2**	High quality pelican case that has a high level of crush and liquid resistantance	Sampling pump 3 pack came with plastic case that is moderately crush resistant, not sealed with rubber but loose metal to metal, liquid can get inside	High quality plastic case that is crush and liquid resistant. Has three latches that are extremely loud when they snap closed	N/A	Will need to purchase a more water resistant case if the case travels frequently	Redesign latches to not be so loud, consider 2 latches instead of 3 to make frequent use more convenient
<u>Durability and Reliability</u>									
The device was resistant to damage from routine usage	**4**	**4**	**4**	No problems were encountered when the devices were clamped tightly with laboratory stands	No problems were encountered when the devices were clamped tightly with laboratory stands	No problems were encountered when the devices were clamped tightly with laboratory stands	N/A	N/A	N/A
The display screen was protected	**4**	**4**	**4**	The display screen had a protective transparent cover	The display screen had a protective transparent cover	The display screen had a protective transparent cover	N/A	N/A	N/A
The device is vulnerable to fall damage	**3**	**4**	**3**	Device could not be dropped, but it is more likely to sustain damage affecting its ability to function properly than if the TDS was dropped	The plastic cassette is intrinsically more robust since it has no electrical parts, is made of a single material which is light weight, and has no moving parts.	Device could not be dropped, but it is more likely to sustain damage affecting its ability to function properly than if the TDS was dropped	Samplers worn in the field by workers are practically guaranteed to be dropped at least once in their operational life time. The extent of	N/A	Samplers worn in the field by workers are practically guaranteed to be dropped at least once in their operational life time. The extent of

							the device's ability to withstand a fall is unknown, so it was only rated relatively to the TDS.		the device's ability to withstand a fall is unknown, so it was only rated relatively to the TDS.
The device did not require manufacturer maintenance during the course of the experiments	**4**	**4**	**4**	The devices worked properly	The devices worked properly	The devices worked properly	N/A	N/A	N/A
The sample holder securely held the TEM grids in place	**4**	**4**	**2**	The magnet held the TEM grid in place, no problems post sampling with grid position being off	The tape held the TEM grid in place, no problems post sampling with grid position being changed	When inserting or removing the sample holder the tape was sheared off due to tight clearances. Or the tape sometimes lost its stickiness and the edge got caught as it was slid into or out of the device	N/A	N/A	Use new tape every time to prevent edges from bending upwards and being caught during insertion and removal of sample holder. Use a non-tape method of securing grid. Increase the clearance between the top of the sample holder and the cavity it slides into, to prevent tape/grid from shearing off.
Effectiveness									
Relative amount of NaCl particles collected per grid space	**3**	**4**	**3**	collected less than TDS, very similar to ESP,	Collected most, and was highest count for this sampler among particle types	collected less than TDS, very similar to TPS	N/A	N/A	N/A
Relative amount of RD particles collected per grid space	**3**	**3**	**4**	Collected less than ESP, similar to TDS	Collected less than ESP, similar to TPS	Collected most	N/A	N/A	N/A
Relative amount of Al particles collected per grid space	**4**	**3**	**4**	Collected 2nd most but was still high, and was highest count for this sampler among particle types	Collected less than ESP/TPS	Collected most, highest count among particle types	N/A	N/A	N/A
Affordability									

The initial cost of the device	**3**	**4**	**3**	$6k	Amount you spend depends on the quality of the sampling pump you decide to use. Ours was $800 per pump	$6-7k	Price will likely decrease over time as more units are sold	N/A	Price will likely decrease over time as more units are sold
The cost of the supplies needed to operate the device	**3**	**4**	**3**	Requires the use of more expensive nickel grids ($130 for 50)	The sampler requires a 25mm cassette and filter with pre-attached TEM grid.We used 400 mesh cu grid with carbon coating ($120 for 50)	Requires a cu grid with carbon coating which costs $2.40 each plus the cost of C batteries (fairly inexpensive)	N/A	N/A	N/A

APPENDIX B

Table S2 – Statistics of particle size sampler comparisons

Sodium Chloride		ParticleSize		Total
		48.7nm	not 48.7nm	
Sampler	TDS	75	323	398
	ESP	60	218	278
Total		135	541	676

48.7nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	.768[a]	1	0.381		
Continuity Correction[b]	0.606	1	0.436		
Likelihood Ratio	0.764	1	0.382		
Fisher's Exact Test				0.381	0.218
Linear-by-Linear Association	0.767	1	0.381		
N of Valid Cases	676				

a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.

b. Computed only for a 2x2 table

Sodium Chloride		ParticleSize		Total
		48.7nm	not 48.7nm	
Sampler	TDS	75	323	398
	TPS	65	236	301

Total		140	559	699	
48.7nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	.809a	1	0.368		
Continuity Correction[b]	0.647	1	0.421		
Likelihood Ratio	0.806	1	0.369		
Fisher's Exact Test				0.391	0.21
Linear-by-Linear Association	0.808	1	0.369		
N of Valid Cases	699				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

Sodium Chloride		ParticleSize		
		115nm	not 115nm	Total
Sampler	TDS	76	322	398
	ESP	21	257	278
Total		97	579	676

115nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	17.740[a]	1	0.000		

Continuity Correction[b]	16.813	1	0.000		
Likelihood Ratio	19.015	1	0.000		
Fisher's Exact Test				0.000	0.000
Linear-by-Linear Association	17.714	1	0.000		
N of Valid Cases	676				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

Sodium Chloride		ParticleSize		
		115nm	not 115nm	Total
Sampler	TDS	76	322	398
	TPS	42	259	301
Total		118	581	699

115nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	3.229a	1	0.072		
Continuity Correction[b]	2.873	1	0.090		
Likelihood Ratio	3.277	1	0.070		
Fisher's Exact Test				0.083	0.044

Linear-by-Linear Association	3.225	1	0.073		
N of Valid Cases	699				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

Road Dust		ParticleSize		Total
		20.5nm	not 20.5nm	
Sampler	ESP	30	287	317
	TDS	1	307	308
Total		31	594	625

20.5nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	27.679[a]	1	0.000		
Continuity Correction[b]	25.774	1	0.000		
Likelihood Ratio	34.684	1	0.000		
Fisher's Exact Test				0.000	0.000
Linear-by-Linear Association	27.634	1	0.000		
N of Valid Cases	625				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

Road Dust	ParticleSize	Total

		20.5nm	not 20.5nm	
Sampler	ESP	30	287	317
	TPS	1	298	299
Total		31	585	616

20.5nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	26.833[a]	1	0.000		
Continuity Correction[b]	24.957	1	0.000		
Likelihood Ratio	33.821	1	0.000		
Fisher's Exact Test				0.000	0.000
Linear-by-Linear Association	26.789	1	0.000		
N of Valid Cases	616				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

Road Dust		ParticleSize		
		86.6nm	not 86.6nm	Total
Sampler	TDS	64	244	308
	ESP	61	256	317
Total		125	500	625

86.6nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)

Pearson Chi-Square	.230a	1	0.631		
Continuity Correction[b]	0.144	1	0.704		
Likelihood Ratio	0.230	1	0.631		
Fisher's Exact Test				0.689	0.352
Linear-by-Linear Association	0.230	1	0.631		
N of Valid Cases	625				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

Road Dust		ParticleSize		
		86.6nm	not 86.6nm	Total
Sampler	TDS	64	244	308
	TPS	50	249	299
Total		114	493	607

86.6nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	1.637a	1	0.201		
Continuity Correction[b]	1.382	1	0.240		
Likelihood Ratio	1.641	1	0.200		

Fisher's Exact Test				0.213	0.120
Linear-by-Linear Association	1.634	1	0.201		
N of Valid Cases	607				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

Road Dust		ParticleSize		
		419nm	not 419nm	Total
Sampler	ESP	25	292	317
	TDS	40	268	308
Total		65	560	625

419nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	4.361[a]	1	0.037		
Continuity Correction[b]	3.831	1	0.050		
Likelihood Ratio	4.392	1	0.036		
Fisher's Exact Test				0.049	0.025
Linear-by-Linear Association	4.354	1	0.037		
N of Valid Cases	625				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					

b. Computed only for a 2x2 table

Road Dust		ParticleSize		
		419nm	not 419nm	Total
Sampler	ESP	25	292	317
	TPS	42	257	299
Total		67	549	616

419nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	6.024[a]	1	0.014		
Continuity Correction[b]	5.405	1	0.020		
Likelihood Ratio	6.068	1	0.014		
Fisher's Exact Test				0.019	0.010
Linear-by-Linear Association	6.014	1	0.014		
N of Valid Cases	616				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

Road Dust		ParticleSize		
		86.6nm	not 86.6nm	Total
Sampler	ESP	64	244	308
	TPS	50	249	299
Total		114	493	607

86.6nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	1.637a	1	0.201		
Continuity Correction[b]	1.382	1	0.240		
Likelihood Ratio	1.641	1	0.200		
Fisher's Exact Test				0.213	0.120
Linear-by-Linear Association	1.634	1	0.201		
N of Valid Cases	607				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

Aluminum Oxide		ParticleSize		
		20.5nm	not 20.5nm	Total
Sampler	TDS	6	315	321
	ESP	20	325	345
Total		26	640	666

48.7nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	6.839a	1	0.009		

	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Continuity Correction[b]	5.832	1	0.016		
Likelihood Ratio	7.244	1	0.007		
Fisher's Exact Test				0.009	0.007
Linear-by-Linear Association	6.828	1	0.009		
N of Valid Cases	666				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

Aluminum Oxide		ParticleSize		
		48.7nm	not 48.7nm	Total
Sampler	TDS	69	252	321
	ESP	99	246	345
Total		168	498	666

48.7nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	4.571a	1	0.033		
Continuity Correction[b]	4.197	1	0.041		
Likelihood Ratio	4.593	1	0.032		
Fisher's Exact Test				0.040	0.020

Linear-by-Linear Association	4.564	1	0.033		
N of Valid Cases	666				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

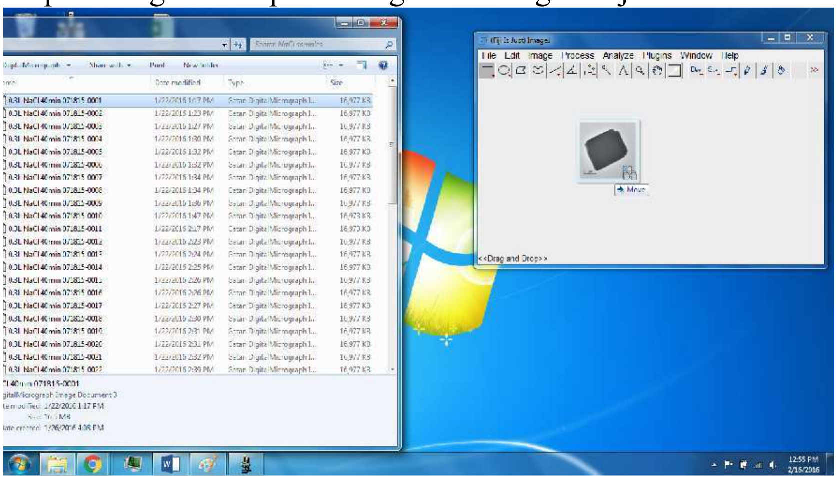
Aluminum Oxide		ParticleSize		Total
		48.7nm	not 48.7nm	
Sampler	TDS	69	252	321
	TPS	121	264	385
Total		190	516	706

48.7nm	Value	df	Asymptotic Significance (2-sided)	Exact Sig. (2-sided)	Exact Sig. (1-sided)
Pearson Chi-Square	8.781a	1	0.003		
Continuity Correction[b]	8.283	1	0.004		
Likelihood Ratio	8.884	1	0.003		
Fisher's Exact Test				0.004	0.002
Linear-by-Linear Association	8.769	1	0.003		
N of Valid Cases	706				
a. 0 cells (0.0%) have expected count less than 5. The minimum expected count is 55.52.					
b. Computed only for a 2x2 table					

APPENDIX C

FIJI Image Analysis Instructions
Step 1: Open ImageJ/Fuji

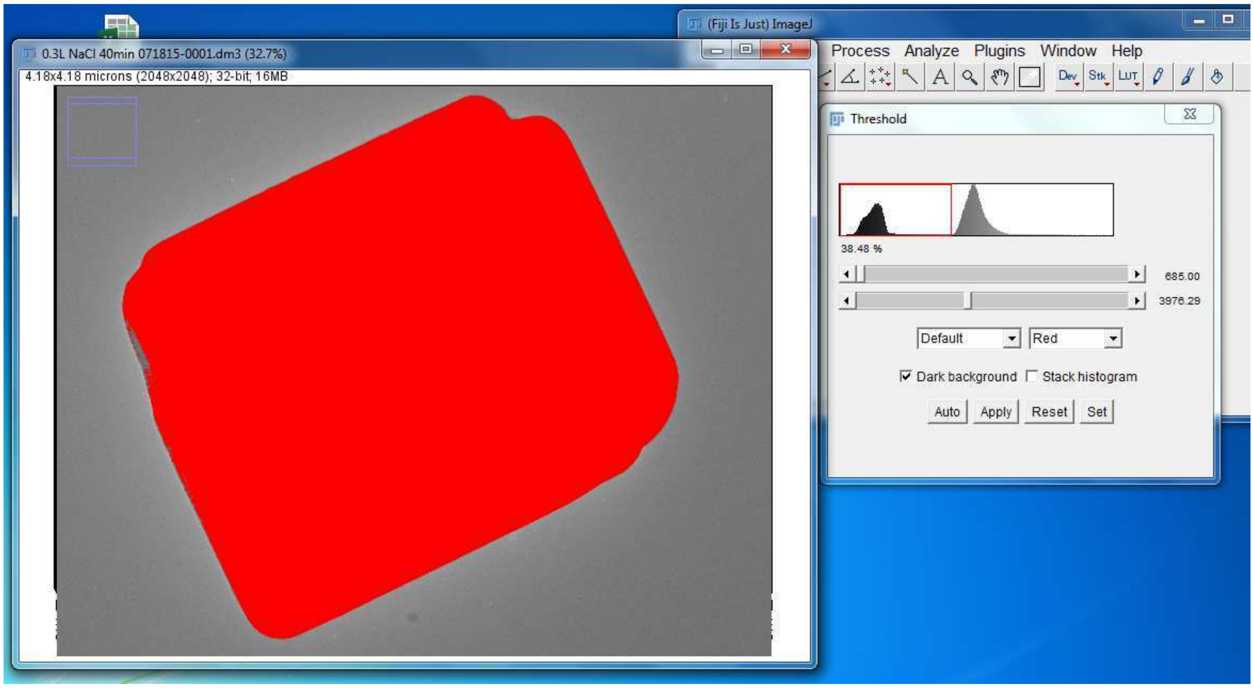
Step 2: Drag and drop the image into ImageJ/Fuji



Step 3: Identify how many particles are in your image and where they are located so that all are accounted for when you adjust the threshold.

Step 4: Adjust the threshold so you can analyze the photo for particles.
Go to Image -> Adjust -> Threshold

Step 5: Set the boxes to Default and Red, and then adjust the two slider bars so that the particles are red and the background is gray/white

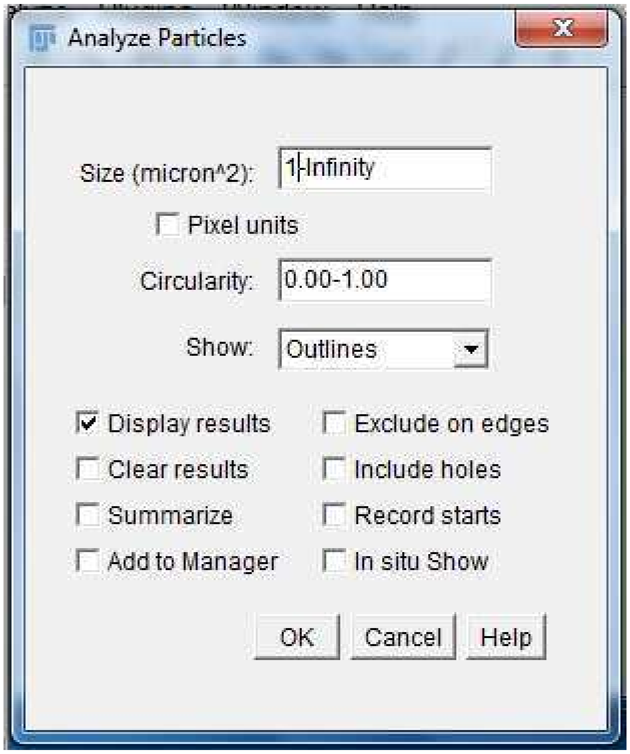


Click Apply and in the next dialog box unclick the "Set background pixels to NaN" and press Ok. It should now show the particle as black and the background as white, close the Threshold dialog box.
*If it is not possible to adjust the threshold so that the particles are easily distinguishable from the background, you will have to manually edit the particles (draw their boundaries) and delete all background shading. See Difficult to threshold particles on page 3.

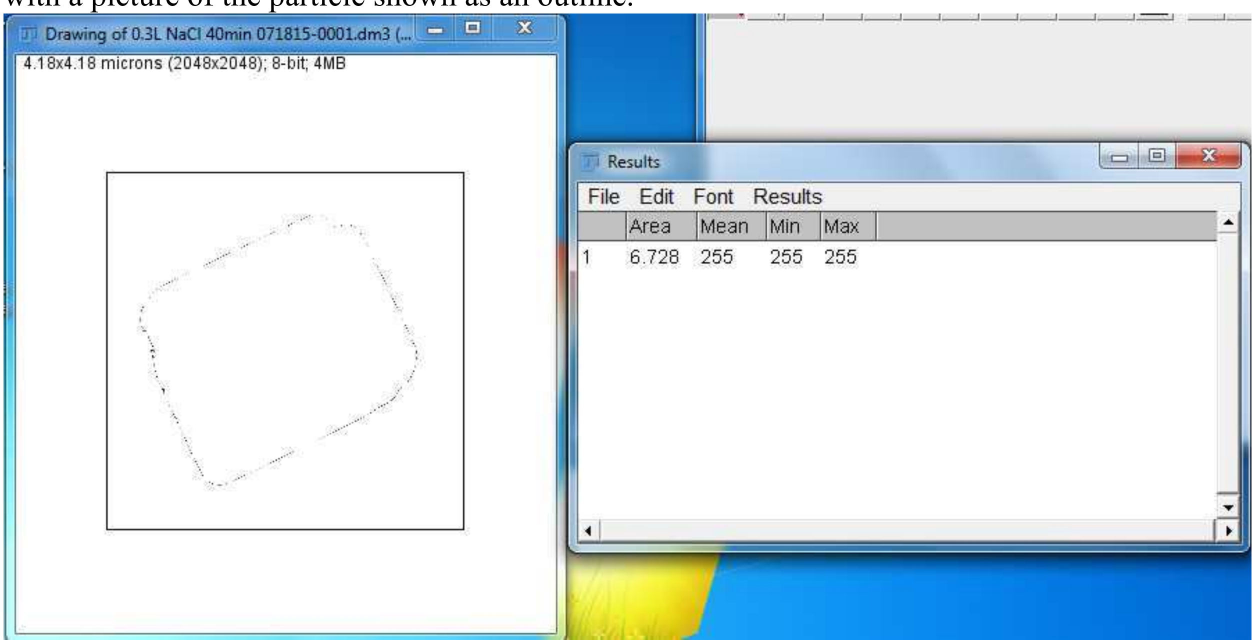
Step 6: Click Analyze -> Analyze Particles , the analyze particles dialog box should open

Select "Show: Outlines", and check "Display results"

Set the minimum size range based on your rough estimate of how big the particles are (use the scale bar in the image), leave the upper size limit to infinity. Sometimes the minimum may be 50 , other times 0.0001 .



Press OK and a Results box should pop up showing the number of particles and their area, along with a picture of the particle shown as an outline.



Be sure to note from the scale bar whether the area units are in micrometers or nanometers.

Zoom in (using the magnifying glass button on the menu bar under Plugins) on the particles shown in the outline picture to make sure one particle wasn't recognized as two smaller ones, and that some background darkness wasn't measured as a particle. Each particle will have its own small red number in the center.

Make sure the number of particles it identified is correct, if not you will have to adjust the size range and re-analyze.

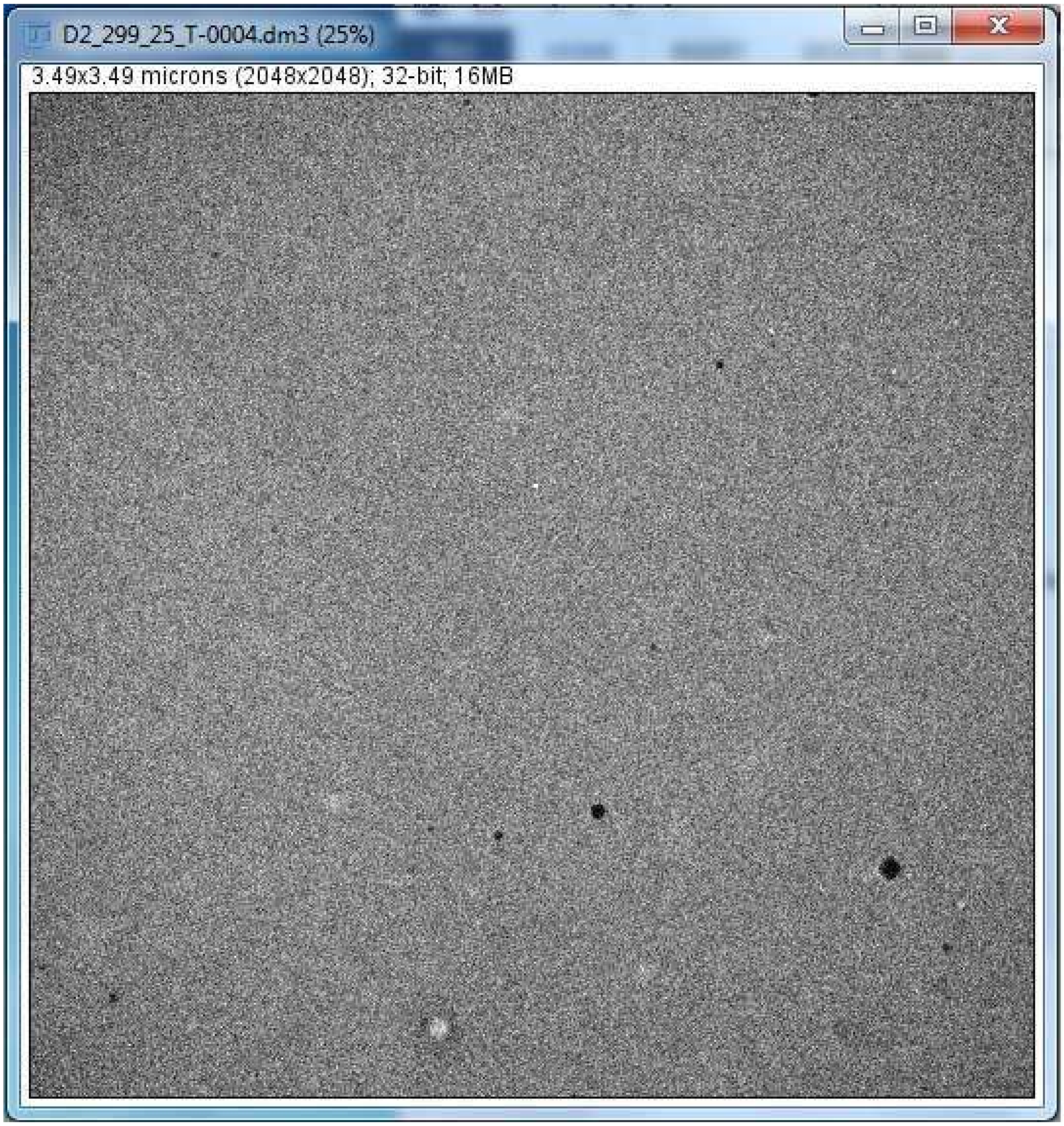
To do this first close the results dialog box and do not save, then close the outline photo created.

Then go to Analyze -> Analyze Particles and make the minimum cutoff size smaller if all the particles were not accounted for. If you got more particles than existed in the photo, make the minimum cutoff size bigger so that small background specs are not counted. You may have to do this several times in a row before getting the size window just right so that the correct number of particles are identified. Following step 3 is very important to this process so that you account for all of the particles.

You can then copy and paste data from the results box directly into excel.

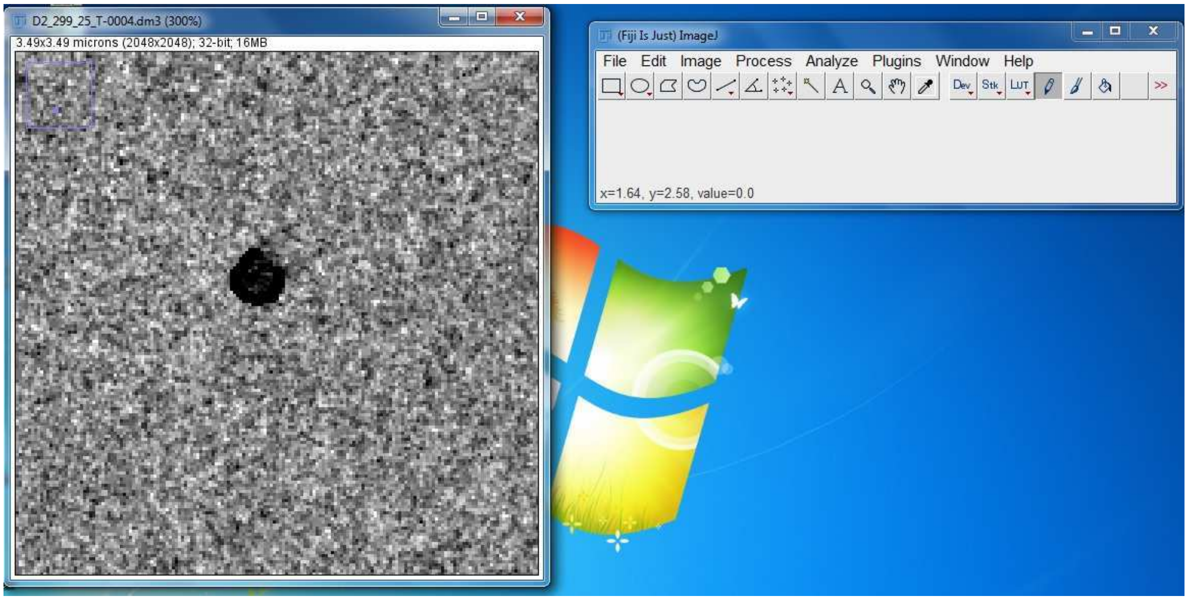
Difficult to threshold images

When the background is dark and the particles are small (image below), adjusting the threshold will not work well enough to analyze all the particles because of the lack of contrast between the background and particle.

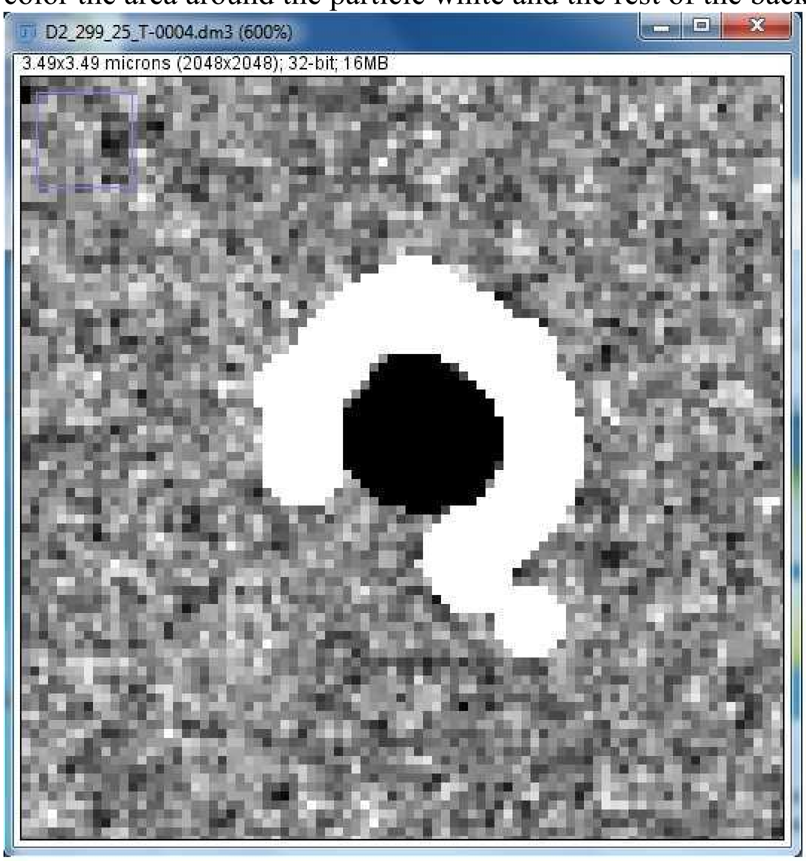


Step 1: Identify which black dots are particles and which are not. It helps to zoon in by clicking the magnifying glass button on the tool bar.

Step 2: Zoom in closely to each particle, set the paint color to black, right click on the pencil or paintbrush to set the pixel size, and carefully color in the entire particle. You must be careful not to make the edges of the particles bigger or smaller.



Step 3: Set the color to white by using the color picker (medicine dropper) button, then delicately color the area around the particle white and the rest of the background of the image.



Step 4: After all the background is white and the particles are colored in black, setting threshold will be very easy and you can analyze the particles.