FY23 Update: Aerosol Sampling for the Canister Deposition Field Demonstration

Spent Fuel and Waste Disposition

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ABSTRACT

This report describes the results of preliminary testing of aerosol monitoring equipment that will be used to continuously monitor the aerosol source term for the multi-year Canister Deposition Field Demonstration (CDFD). These data are required inputs for the development and validation of models for the deposition of dust and potentially corrosive salts on the surface of spent nuclear fuel (SNF) dry storage canisters. Surface salt loads correlate with the extent of corrosion damage on a metal surface, and potentially to the likelihood and timing of initiation of stress corrosion cracks.

Aerosols will be monitored at the CDFD site using three instruments. A Dekati® ELPI+ cascade impactor will be used for real-time monitoring of aerosol particle sizes. It will also collect dust in 14 size bins on impactor targets that can be chemically analyzed to determine the soluble salts present as a function of particle size. However, this instrument can only measure dried aerosols, with a diameter of <10 µm. The second instrument is a Topas laser particle size spectrometer, which provides real-time monitoring of aerosol particle sizes up to ~40 µm in size. It monitors both the ambient (potentially deliquesced) aerosol particle size distributions required for the dust deposition models and the distributions of the equivalent dried particles, allowing correlation with the Dekati® data. However, it does not discriminate between inert dust particles and salt aerosols, and it does not retain samples of the different particle sizes for later analysis. The third instrument that will monitor aerosols at the CDFD site is a Clean Air Status and Trends Network (CASTNET) tower, which uses a multiple canister system to collect weekly samples for analysis to total suspended aerosol particle compositions and atmospheric gas concentrations.

This status report describes work in FY23 to develop the capabilities for using these tools. In two training exercises, the cascade impactor and laser particle sizer were deployed in two different testing environments, one indoor and one outdoor. For the cascade impactor, the tests provided opportunities for the operators to familiarize themselves with impactor substrate preparation, and post-test sample removal and analysis. For the laser particle sizer, the tests were used to evaluate different instrument parameters, to determine the most appropriate settings for capturing transient events. Data and samples were collected for weeks to months for each test, and the results are presented here. In addition to the preliminary testing, contracts were developed with WSP Analytical Labs for sample preparation and analysis of the cascade impactor samples. The impactor tower from outdoor test was delivered to WSP and used to train the staff there in disassembly, sample extraction, sample analysis, and tower reassembly with new target substrates. These are tasks that WSP will be performing routinely for the CDFD project. The CASTNET system cannot be purchased or tested until an actual site has been selected for the CDFD test. Work for this FY has been restricted to preparation of contracts for purchasing the CASTNET tower, and for sample analysis, once the tower is in operation.

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ACRONYMS

AHSM	Advanced Horizontal Storage Module
BLM	Bureau of Land Management
CASTNET	Clean Air Status and Trends Network
CDFD	canister deposition field demonstration
DCSS	dry cask storage system
DI	deionized [water]
DOE	US Department of Energy
EPA	Environmental Protection Agency
FY	fiscal year
IC	ion chromatograph
ICP-OES	inductively-coupled plasma optical emission spectroscopy
ISFSI	independent spent fuel storage installation
NE	Office of Nuclear Energy
NUHOMS	NUTECH Horizontal Modular Storage
PNNL	Pacific Northwest National Laboratory
RH	relative humidity
SCC	stress corrosion cracking
SFWD	Spent Fuel and Waste Disposition Program
SNF	spent nuclear fuel
SNL	Sandia National Laboratories

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FY23 UPDATE: AEROSOL SAMPLING FOR THE CANISTER DEPOSITION FIELD DEMONSTRATION

This report fulfills milestone M3SF-23SN010207046 in the Stress Corrosion Cracking and Dry Storage Investigations - SNL work package (SF-23SN01020704). This work was sponsored under the Department of Energy's (DOE) Office of Nuclear Energy (NE) Spent Fuel and Waste Disposition (SFWD) campaign.

1 INTRODUCTION

For interim storage, spent nuclear fuel (SNF) is commonly stored in welded stainless steel canisters enclosed in passively ventilated overpacks. Over time, dust accumulates on the canister surfaces, and as the SNF cools, salts within that dust will deliquesce to form concentrated brines. If the salts contain aggressive species such as chloride, then the resulting brine can cause localized corrosion, and in weld regions with high residual stresses, stress corrosion cracking (SCC) can occur. Eventually, SCC cracks could penetrate the canister wall. The risk of corrosion and SCC is greatest in near-marine settings, where chloride-rich sea-salt aerosols are deposited on the canister surface. Developing an improved understanding of the occurrence and risk of SNF storage canister SCC is considered to be critical to demonstrating the safety of long-term dry storage of SNF (EPRI, 2011; Hanson et al., 2012; NRC, 2014; NWTRB, 2010; Teague et al., 2019).

Past work has shown that important parameters that affect the extent of corrosion damage and likelihood of canister SCC are the composition of the salts deposited on the canister surface; the composition and evolution of the deliquescent brines that form as the canister surface cools and the relative humidity increases; and the salt deposition rate, which determines evolution of the salt load (mass per unit area of deposited salts) over time. SNF dry storage canister surface deposits have been sampled at some independent spent fuel storage installation (ISFSI) sites and provide some information on the composition of deposited salts at different sites. However, the technical challenges to quantitatively sampling salts from the surface of a highly radioactive SNF dry storage canister mean that only qualitative data are available for salt loads. Moreover, in each case, only a small number of samples, representing very limited surface coverage, were collected. Hence, deposited salt compositions and salt deposition rates at different ISFSI geographic locations and at different canister surface locations are poorly understood. Coupled with a complete lack of data on aerosol source terms (atmospheric salt aerosol densities and particle size distributions), it is not currently possible to estimate salt deposition rates, at any canister surface location, at any ISFSI site.

In order to develop an understanding of dust and salt deposition on the surface of SNF dry storage canisters in near-marine environments, the DOE is starting a multi-year study at a near-marine location (Lindgren et al., 2020, 2021). As part of this study, three unused commercial 32PTH2 NUTECH Horizontal Modular Storage System (NUHOMS) welded stainless steel storage canisters will be placed into Advanced Horizontal Storage Modules (AHSMs) from Orano. To mimic the heat generated by radioactive decay in real SNF, internal heaters will be added to the three canisters. Two will be heated during the test, while the third will be unheated, but can be powered up used as a backup if one of the other systems fails. All three canisters and AHSMs will be extensively instrumented to monitor canister and storage module surface temperatures and airflow though the inlet and outlet vents of the AHSM (Lindgren et al., 2020, 2021).

Once the canisters are emplaced in the AHSM, canister heating will begin. Canister heat loads are being designed to simulate the canister surface temperatures corresponding to fuel heat loads of 0 kW, 10 kW, and 40 kW, and will not vary over the course of the multi-year experiment. Periodically (on a yearly or bi-yearly basis), the heaters will be turned off and the canisters will be allowed to cool. The canisters will be extracted from the overpacks and salt and dust surface deposits will be sampled by hand to determine the size and morphology of the deposited dust and salt particles, the composition of the soluble salts present, and the soluble salt load per unit area. These results will provide information on particle deposition rates as a function of canister surface location, orientation, and temperature (T). Salt analyses will also provide information on the effects of particle-gas conversion reactions—reactions between salt aerosols and atmospheric gases that can result changes in the composition and deliquescence properties of the particles, thus impacting the potential occurrence and timing of canister SCC. The process of hand-sampling surface deposits on the CDFD storage canisters is described in *FY23 Update: Surface Sampling Activities for the Canister Deposition Field Demonstration* (Knight et al., 2023) and is not discussed further here.

To understand dust and salt deposition at a mechanistic level, a second data set is required, which is a full characterization of the salt aerosol source term. This includes the particle densities and particle size distributions in the air entering the AHSM. Moreover, these data must be collected with sufficient temporal resolution to capture short-term, intense events (e.g., storms) that might significantly affect dust deposition. Once combined with the canister surface sampling data, this information will be used to calibrate and validate a salt deposition model for canister surfaces in passively ventilated AHSMs that is currently being developed at Pacific Northwest National Laboratories (Jensen et al., 2020; Jensen et al., 2022; Suffield et al., 2021). The characterization of the salt aerosol source term at the CDFD field site is the focus of this report.

One specific parameter that is required to parameterize the Pacific Northwest National Laboratory (PNNL) dust deposition model that is currently in development is the aerosol chloride concentration per unit volume of air as a function of the particle size. This cannot be measured directly but can be calculated from the following parameters, that can be derived from the following data, that will be collected by the cascade impactor and the laser particle sizer.

- Aerosol particle size distribution, from >10 um to <0.1 um, in several size fractions
- Aerosol particle density per unit volume of air ($\mu g/m^3$)
- Particle soluble salt compositions, as a function of particle size

Note that for the dust deposition model developed at PNNL, the required particle sizes are those of the aerosol particles that will enter the overpack. Depending upon the relative humidity (RH), these particles may be fully or partially deliquesced. Also, a large fraction of the dust particles will not be salt aerosols. Previous observations have shown that the majority of dust deposited on the SNF dry storage canisters is detrital mineral grains. At many ISFSI sites, these are silicate minerals such as quartz, feldspars, and clays. At other locations, where the surrounding bedrock is limestone or dolomite, carbonate minerals may dominate in the dust. Organic detritus such as pollen and stellate trichomes are also frequently abundant.

SNL will continuously monitor the environmental conditions and atmospheric aerosols at the CDFD site, to collect the necessary data to parameterize the PNNL dust deposition model. The site weather (T, RH, wind speed and direction, solar irradiance, and other parameters) will be monitored using a weather station and will provide necessary boundary conditions for the thermal and airflow models used in the dust deposition model. SNL will use three different instruments to continuously monitor aerosol compositions, particle densities, and particle size distributions at the CDFD site. The instruments include a Dekati® ELPI+ Cascade Impactor, a Topas Laser Aerosol Particle Size Spectrometer (LAP323). In addition, a filter pack system placed through the Clean Air Status and Trends Network (CASTNET) will be used to determine the composition of particles and the concentration of acid gas species in the atmosphere. Although not currently incorporated into the PNNL dust deposition model, atmospheric gases can react with salt aerosols, either prior to or after deposition onto canister surfaces. Such interactions can modify the composition of the salts and the composition and properties of the resulting deliquescent brines. These three instruments will provide specific, unique, and complimentary information that can used collectively to develop an in-depth understanding of the aerosol source term (aerosol particle densities and compositions, and particle size distributions) and atmospheric gas concentrations at the CDFD test site.

This status report describes work in FY23 to develop the capabilities for using these tools. In two training exercises, the cascade impactor and laser particle sizer were deployed in two different testing environments—one indoor and one outdoor. For the cascade impactor, the tests provided opportunities for the operators to familiarize themselves with impactor substrate preparation, and post-test sample removal and analysis. For the laser particle sizer, the tests were used to evaluate different instrument parameters, to determine the most appropriate settings for capturing transient events. Data and samples were collected for weeks to months for each test, and the results are presented here. In addition to the preliminary testing, contracts were developed with WSP for sample preparation and analysis of the cascade impactor samples. The impactor tower from outdoor test was delivered to WSP and used to train the staff there in disassembly, sample extraction, sample analysis, and tower reassembly with new target substrates. These are the tasks that WSP will be performing routinely for the CDFD project.

The CASTNET system cannot be purchased or tested until a site has been selected for the CDFD test. Work this FY has been restricted to the preparation of contracts for purchasing the CASTNET tower and for sample analysis, once the tower is in operation.

2 AEROSOL SAMPLING METHODS AND DISCUSSION

The two aerosol sampling instruments, the Dekati® ELPI+ Cascade Impactor and the Topas Laser Aerosol Particle Size Spectrometer (LAP323) were previously described in Schaller et al. (2022). That report documents the initial set-up of the aerosol sampling instruments, and initial testing to familiarize ourselves with the operation of the equipment. The general setup of the two instruments is shown in Figure 2.1.

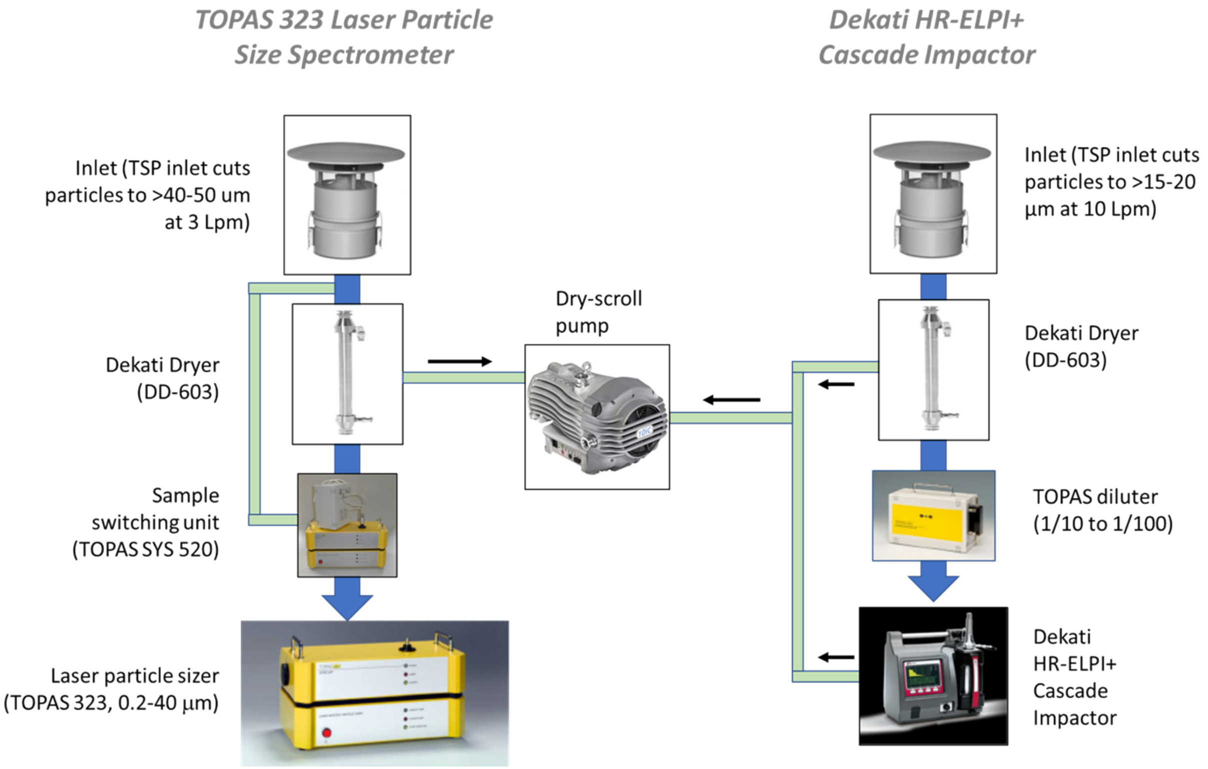

Figure 2.1. Set-up schematics for the laser particle sizer and cascade impactor.

The LAP323 is capable of measuring a much larger particle size range. Specific details regarding the LAP323 and its accessories have been previously provided (Bryan et al., 2021; Schaller et al., 2022). Briefly, the LAP323 is a ruggedized unit designed for environment monitoring and can size particles from 0.2 µm to 40 µm (0.2 to 5 µm or 0.7 to 40 µm) in 64-128 channels. The LAP323 is equipped with the Dekati® total suspended particle (TSP) inlet to cut very large particles. The LAP323 is operated at ~ 3 L/min, and therefore it is estimated by Dekati® that the inlet will reject particles > 40-50 um, which is sufficient to allow passage of all anticipated sea-salt aerosols. The LAP323 has its own internal pump, and so does not need a separate pump; however, the LAP323 inlet line has a Dekati® inline Nafion® membrane-based dryer, that will use the same vacuum pump as the EPLI+ cascade impactor. The LAP323 is equipped with an automated switching valve that allows for sampling of two different air streams. This will be used to sample the air stream both above and below the dryer. Using the switching unit in this way will provide a complete particle size distribution for both the ambient air and for the dried particles to estimate the shift in particle size distribution as deliquesced particles dry out. This is important because the dried particles allow a direct comparison to the particle size fractions collected by the cascade impactor (aerosols must be dried for that instrument), and sampling the deliquesced particles allows direct measurement of the actual aerosol source term particle size distribution, which is necessary for the PNNL model. The laser particle sizer will sample continuously at the CDFD site, collecting data in alternating 10-minute sampling increments from each of the two air pathways.

The Dekati® ELPI+ cascade impactor will provide particle size distributions for particles in the size range of 0.016 to 10 µm in several stages, and the stage targets can be analyzed to determine the composition as a function of particle size. The instrument has previously been described in detail (Bryan et al., 2021; Schaller et al., 2022). Briefly, the ELPI+ is a 14-stage impactor covering a size range from 0.016 µm to 10 µm, operating with a flow rate of 10 L/min. The impactor is equipped with a TSP inlet similar to that of the laser particle sizer; however, at the flow rate of 10 L/min, the inlet excludes particles >20 µm. The air stream passed through a Dekati® dryer prior to entering the cascade impactor, as potential condensation must be avoided in this instrument. The top 10 µm stage is collected, but not monitored electronically, therefore the weight of the 10 µm cut must be determined by the weight difference between the clean target and the target after use, at a known RH. Particles too small to be captured by the final target (<0.016 µm) are collected by a high-efficiency particulate air (HEPA) filter after the final target. This filter is fixed—it cannot be removed for sample retrieval and therefore no chemical analysis can be provided for these particles. Median particle sizes for the different stages are shown in Table 2.1.

Table 2.1 Median particle sizes for the cascade impactor stages.

Stage #	Median particle size
S-1	<0.016 µm
S-2	0.016 µm
S-3	0.030 µm
S-4	0.054 µm
S-5	0.094 µm
S-6	0.15 µm
S-7	0.25 µm
S-8	0.38 µm
S-9	0.60 µm
S-10	0.94 µm
S-11	1.6 µm
S-12	2.5 µm
S-13	3.6 µm
S-14	5.3 µm
S-15	>10 µm

The collection plates in each stage accept 25 mm polycarbonate collection targets which must be greased prior to loading on the compactor tower. These targets can be extracted for post-measurement chemical analysis to determine aerosol compositions. In this system, aerosol particles pass through a unipolar corona charger where a known positive charge is applied. In the impactor, the particles are size classified into 14 size classes onto the impactor collection plates. The charge deposited in each stage is measured, allowing real-time measurement of the particle number size distribution and concentrations. To convert to a mass distribution, a density must be assumed for the particles. Alternatively, the targets can be weighed at a known RH before and after use, as with a standard cascade impactor. The high-resolution collection mode of the instrument offers 100 particle size channels instead of 14 channels; the extra cuts are achieved through software and can be performed after the data is collected.

In FY23, the laser particle sizer and the cascade impactor were set up inside a building and initial samples and data sets were collected for an unspecified time interval. This test was used to familiarize the operators with the operation of the instruments, with data collection, formatting, and analysis, and with sample collection. Then, the instruments were moved to a sheltered external location at SNL, and atmospheric aerosols were monitored for a period of two weeks in a simulated field test. These two tests were used to develop operating procedures for the instruments, to determine space needs for the installation of the equipment at the CDFD site, and to plan for eventual field use.

The overall setup for both the indoor and outdoor test was the same and is illustrated in Figure 2.2. For both instruments, the vertical mast with the inlet vent was supported by a tripod. The dryers were located below the head of the tripod. For the laser particle sizer, two lengths of plastic tubing connected the mast to the sample switching unit, one splitting off above the dryer, and one from below the dryer. A single length of tubing connects the sample switching unit to the laser particle sizer. This setup allows sampling both the dried aerosols coming through the dryer, and the potentially deliquesced aerosols above the dryer. In the test, samples were collected with a 10-minute sampling interval, alternating between the two sampling positions above and below the dryer. For the cascade impactor, the bottom of the dryer is connected to the impactor tower, using a sliding fitting that allows easy removal of the impactor tower for sample retrieval. Note that the diluter shown in the schematic in Figure 2.1 is not in the sample string. This is because it must set at the factory and the actual degree of dilution must be determined based on analyses at the actual CDFD site. Hence, it will be added later, once the CDFD site has been chosen, and initial site-specific data have been collected. The data collected in the indoor and outdoor tests are discussed in the following sections for each instrument.

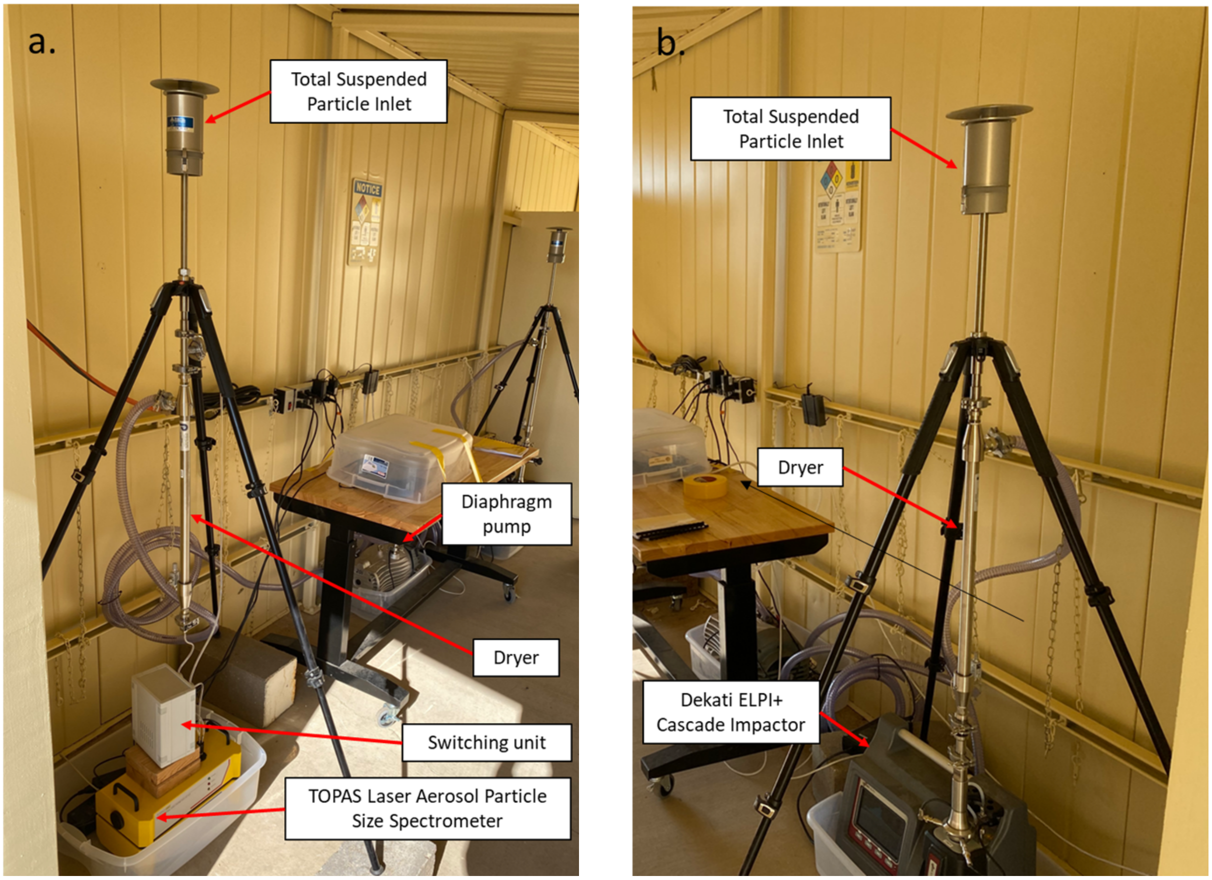

Figure 2.2. Actual Field set-up for laser particle sizer and cascade impactor.

2.1 Dekati® ELPI+ Cascade Impactor

Prior to collecting samples with the cascade impactor, a procedure was developed for preparing the substrates for each impactor stage for use and assembling the impactor tower, and for removing the substrates for later analysis after use. This procedure was developed for our use but was also shared with the WSP Analytical Lab that will prepare the impactor tower and extract and analyze the substrates during the actual CDFD test. It is not necessary to describe the procedure in detail here, but in general, it describes disassembly of the impactor tower, retrieval of the polycarbonate substrates from each stage, cleaning of the stages, preparation of the new polycarbonate substates by coating with a thin spray-on layer of Apiezon® grease, and re-assembly of the impactor tower, taking great care to assemble each stage in the correct order.

For the initial indoor test, the cascade impactor was set up in a large shop, and later moved to a second shop building. The impactor was run intermittently for several weeks, as the operator became familiar with the software, and different data collection parameters were tested. The initial results are discussed in Schaller et al. (2022) and are not repeated here. One major conclusion was that very few large particles were observed. This was attributed to the indoor location used for the test and was one reason why an outdoor site was chosen for the second field test.

Following the test, the impactor tower was disassembled and the mounted substrates were photographed. The jets for each stage of the impactor have a unique size and configuration, and that is reflected in the dust collected on the substrates. An example is shown in Figure 2.3, in which small accumulations of dust clearly correspond to each hole in the jet plate.

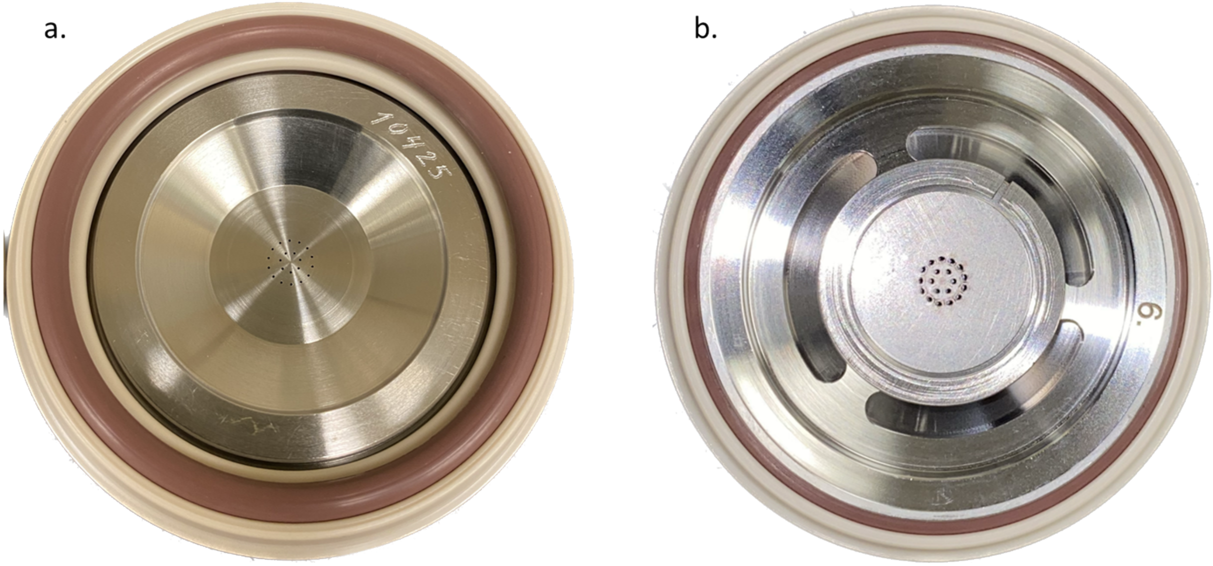

Figure 2.3. Comparison of impactor jet plate (a) and deposited dust on the target substrate (b) for Stage 6 of the cascade impactor.

After removal from the target mounts, the polycarbonate substates were also imaged using scanning electron microscopy (SEM), which provides a more detailed view of the dust distribution on each target. The mounted substrates and a magnified SEM image of the deposited dust for each of the targets is shown in Figure 2.4. It should be noted that the recommended dust load on any stage of the impactor should be less than 1 mg; the dust load was not monitored during this scoping test, and it is possible that this limit was exceeded for some impactor stages. Hence the observed dust loads and distributions may not be typical for normal operation.

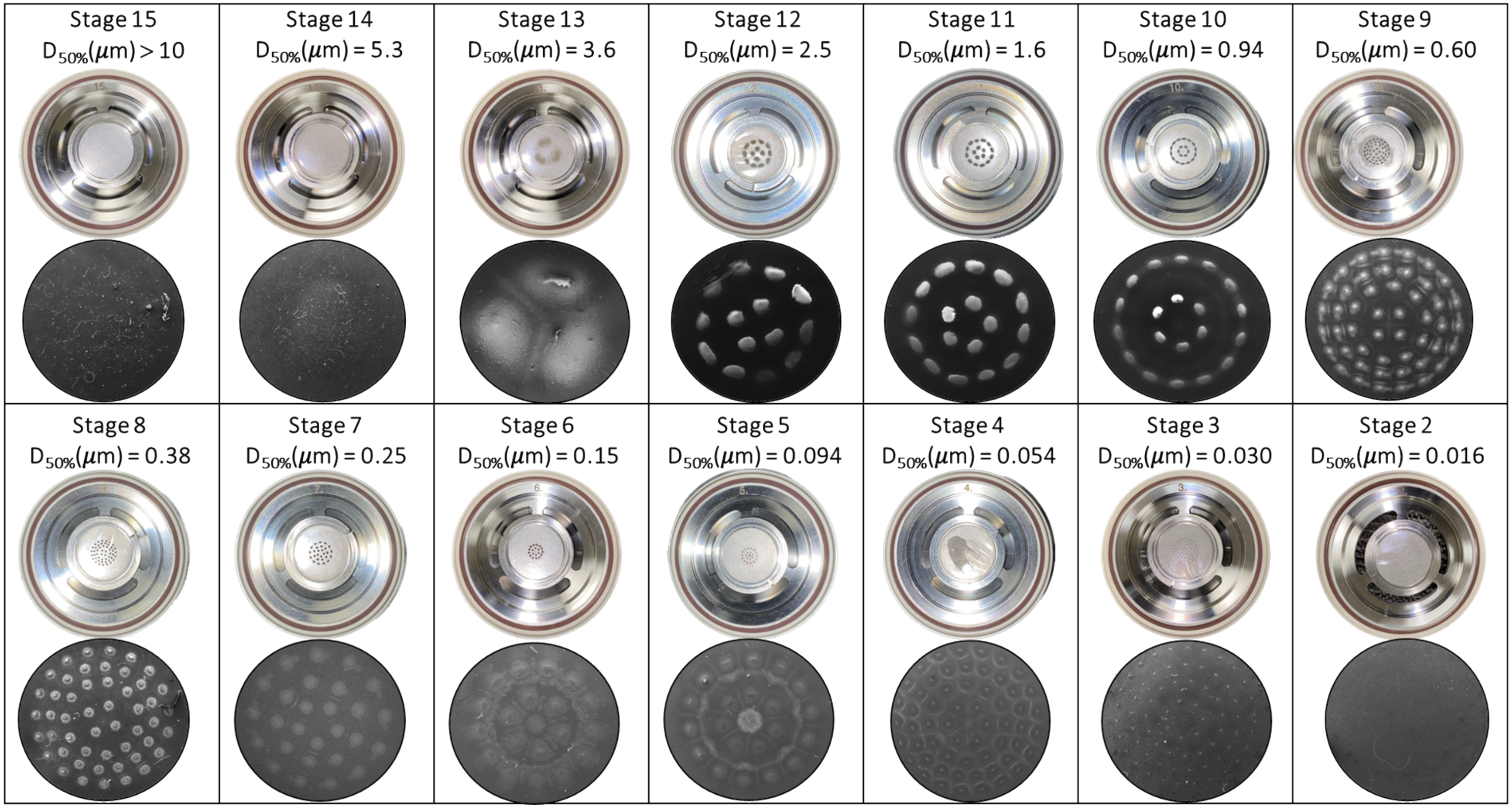

Figure 2.4. Photographs and SEM images of the dust on the substrates for each stage of the cascade impactor.

It is notable that in the first three stages, few particles were collected, and most appear to be fibrous. It is not clear if these are naturally occurring material (e.g., plant fibers), or anthropogenic particles (e.g., lint). These fibers are much larger than the cutoff that should have been implemented by the inlet head, possibly indicating that the inlet is not effective at excluding this type of particle. Alternatively, the fibers may represent contaminants that were trapped in the components of the inlet string during assembly and that should not be observed once the system has been fully assembled and installed. It might be worthwhile to use compressed air to blow dust off all components when the system is reassembled.

After being imaged, the samples were leached by transferring substrates to pre-weighed screw-cap sample tubes and adding 10 ml of deionized (DI) water. The samples were placed on a shaker table and agitated for 5 minutes. Subsequently, the leachate was filtered through a 0.45 µm syringe filter to remove insoluble particulates. After leaching the targets from the indoor test, ion chromatography (IC) analyses were performed at SNL with a Dionex ICS-1100 RFIC ion chromatograph. The anionic analytes were F^-, Cl^-, Br^-, NO_2^-, NO_3^-, SO_4^{-2}, and PO_4^{-3}. However, signals for Br^- and NO_2^- were never detected and are not included in any analysis. Anionic analyses were performed with a 4 mm Dionex Ionpac AS-23 RFIC column, AG-23 guard column, and a Dionex AERS 500 suppressor. Cationic analytes were Li^+, Na^+, NH_4^+, K_+, Mg^{+2}, and Ca^{+2}. Ionic IC analyses were performed with a Dionex Ionpac CS-12A column and CG-12A guard column, and a CERS 500 suppressor, all 4 mm in diameter. Blanks were run after every sample to minimize contamination. Six standards and a blank were made by dilution of stock Dionex IC anion and cationic standards. Sample concentrations were estimated using a subset of the standards, excluding those which were higher than necessary to constrain the sample concentration. This was done because the calibration curves were fitted using the least squares method which over-weights higher-concentration standards, potentially resulting in larger errors for values in the lower part of the concentration range.

The samples collected during the two-week outdoor test were not imaged. Instead, the entire tower was packaged and sent to WSP Analytical Lab for analysis. WSP will be analyzing the cascade impactor samples for the actual CDFD test, and the outdoor test was an opportunity to familiarize them with removing, analyzing, and replacing the substrates in the impactor tower; these are tasks that WSP will be performing routinely for the CDFD test. A SNL staff member accompanied the impactor tower and taught WSP personnel the necessary procedure. WSP leached the substrates in DI water, and then analyzed the anions using IC (specifications), and the cations using a Perkin Elmer inductively-coupled plasma optical emission spectroscopy (ICP-OES) instrument. NH_4^+ was measured using a segmented flow analyzer (SEAL AA500). It should be noted that WSP routinely analyzes very small samples for the Environmental Protection Agency (EPA) and is highly experienced in this type of work.

The soluble ion concentrations in the deposited dust on each stage, for the indoor and outdoor tests, are tabulated in Table 2.2 and Table 2.3, and shown in Figure 2.5 and Figure 2.6, respectively. For both tests, the soluble salts are concentrated in the middle stages, from approximately 0.1 to 2 µm. In both cases, the dominant solutes are sulfate and ammonium, both of which peak at the intermediate particle sizes. Plotting the concentrations in micro-Equivalents (µEq) (Figure 2.7 and Figure 2.8) show that the ammonium and sulfate values in µEq are identical for stage 9 (0.6 µm median diameter) and smaller, indicating that ammonium sulfate is the dominant phase present in both the indoor and outdoor samples in this particle size range. At larger particle sizes, other solutes become important. Potassium and nitrate show similar bimodal distributions, and KNO_3 may be one nitrate salt phase that is present. However, there is insufficient potassium to account for all the nitrate, so other nitrates must be present.

The association of specific solutes with different particle sizes is shown in Figure 2.9 for both the indoor and outdoor tests. The same solutes are enriched in the same particle sizes, regardless of the test location. However, when the solutes are plotted on the same vertical scales for the two tests (Figure 2.10), it is apparent that the relative proportions of the species vary greatly. The ammonium and sulfate concentrations are broadly similar for the indoor test and the outdoor test, but this does not imply that the same aerosol particle densities were present in both cases, since the indoor test intermittently collected data over several months, while the outdoor test was run continuously for 2 weeks. If ammonium and sulfate are excluded, the concentrations of all other species are much lower in the outdoor test. Perhaps the oddest distribution is shown by chloride, which is much higher in the indoor test but only occurs over a very narrow particle size range. Chloride is very low in the outdoor test but is measurable in several size fractions over the entire particle size range. The differences in the relative proportions of the analytes between the two tests may be related to the sampling locations—perhaps higher levels of anthropogenically-produced aerosols are present at the indoor site. However, the two sites were also sampled at different times of the year. The indoor data was collected sporadically over several months in the summer and fall, while the outdoor test represented two weeks of continuous sampling in the winter. Therefore, the observed differences in aerosol compositions may be due to seasonal variations.

Table 2.2 Indoor test: cascade impactor soluble ion concentrations per stage (µg/sample).

Stage #	**Median diameter**	**Na^+**	**K^+**	**NH_4^+**	**Ca^{+2}**	**Mg^{+2}**	**Cl^-**	**NO_3^-**	**SO_4^{-2}**	**Total**
2	**0.016 µm**	n.d.	n.d.	1.102	n.d.	n.d.	n.d.	0.284	2.834	4.221
3	**0.030 µm**	0.114	0.523	1.234	0.517	n.d.	n.d.	2.138	3.534	8.060
4	**0.054 µm**	0.270	0.833	0.853	0.426	n.d.	n.d.	3.292	2.372	8.046
5	**0.094 µm**	0.227	0.666	3.787	0.301	n.d.	n.d.	2.696	10.261	17.939
6	**0.15 µm**	n.d.	0.295	8.470	n.d.	n.d.	n.d.	2.106	23.336	34.206
7	**0.25 µm**	n.d.	n.d.	13.793	n.d.	n.d.	n.d.	0.501	37.578	51.872
8	**0.38 µm**	1.756	3.124	13.701	0.583	n.d.	0.742	7.577	38.832	66.315
9	**0.60 µm**	4.248	5.717	6.814	2.603	0.195	1.896	15.398	21.846	58.716
10	**0.94 µm**	9.057	4.851	0.826	3.718	0.562	5.023	13.352	14.709	52.098
11	**1.6 µm**	12.245	4.320	0.555	5.098	0.888	7.524	13.059	18.679	62.369
12	**2.5 µm**	9.613	0.485	0.496	3.412	0.688	n.d.	n.d.	n.d.	14.693
13	**3.6 µm**	3.615	0.061	0.181	2.771	0.010	n.d.	2.727	7.396	16.759
14	**5.3 µm**	0.101	n.d.	0.219	0.678	n.d.	n.d.	0.630	2.018	3.647
15	**<10 µm**	0.729	n.d.	0.045	0.404	n.d.	n.d.	0.597	n.d.	1.775

Table 2.3 Outdoor test: cascade impactor soluble ion concentrations per stage (µg/sample).

Stage #	**Median diameter**	**Na^+**	**K^+**	**NH_4^+**	**Ca^{+2}**	**Mg^{+2}**	**Cl^-**	**NO_3^-**	**SO_4^{-2}**	**Total**
2	**0.016 µm**	0.042	0.080	0.021	0.031	0.001	0.055	n.d.	n.d.	0.336
3	**0.030 µm**	0.023	0.022	0.152	0.034	0.002	n.d.	n.d.	0.418	0.757
4	**0.054 µm**	0.049	0.058	0.422	0.048	0.003	n.d.	0.130	1.130	1.946
5	**0.094 µm**	0.057	0.135	1.384	0.085	0.013	0.008	0.473	3.208	5.469
6	**0.15 µm**	0.069	0.250	2.957	0.070	0.005	n.d.	0.969	6.664	11.090
7	**0.25 µm**	0.090	0.250	5.157	0.115	0.009	n.d.	0.998	13.906	20.631
8	**0.38 µm**	0.098	0.235	10.790	0.199	0.019	0.003	0.869	33.791	46.110
9	**0.60 µm**	0.194	0.107	5.275	0.393	0.038	0.109	0.237	17.860	24.319
10	**0.94 µm**	0.211	0.057	0.745	0.756	0.054	n.d.	0.184	4.447	6.560
11	**1.6 µm**	0.349	0.078	0.127	1.496	0.089	n.d.	0.521	1.435	4.201
12	**2.5 µm**	0.348	0.081	0.130	1.820	0.086	0.005	0.608	1.071	4.255
13	**3.6 µm**	0.254	0.095	0.165	1.952	0.083	0.080	0.449	0.772	3.956
14	**5.3 µm**	0.119	0.045	0.146	0.630	0.021	0.114	0.139	0.475	1.795
15	**<10 µm**	0.062	0.017	0.069	0.133	0.007	0.036	0.052	0.007	0.489

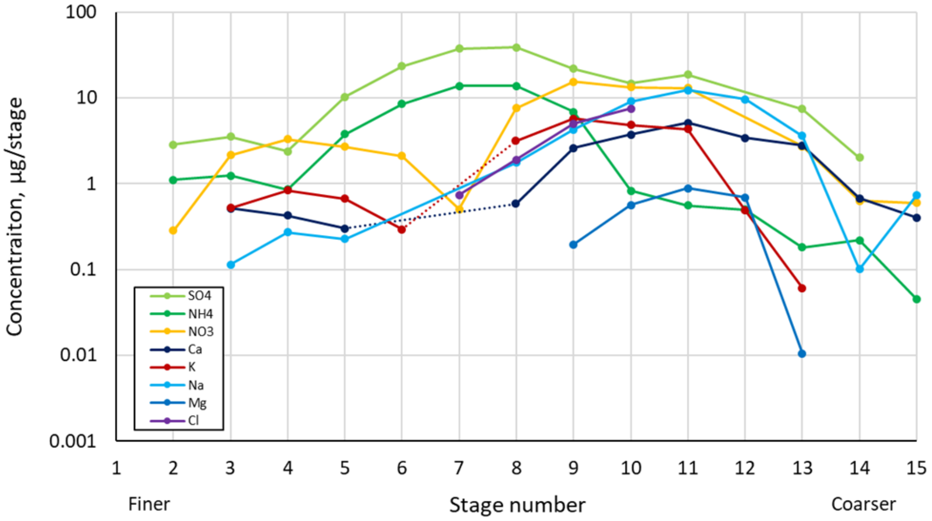

Figure 2.5. Soluble ion concentrations in µg/stage, indoor test.

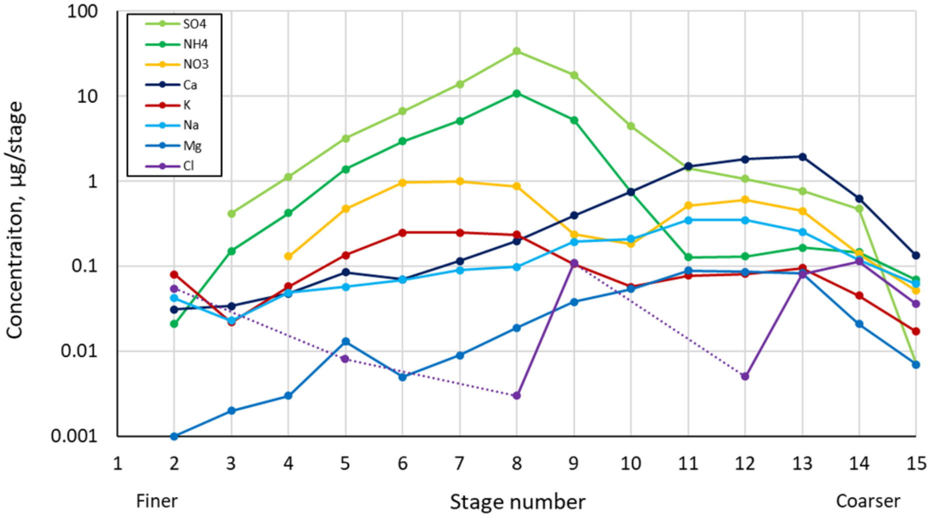

Figure 2.6. Soluble ion concentrations in µg/stage, outdoor test.

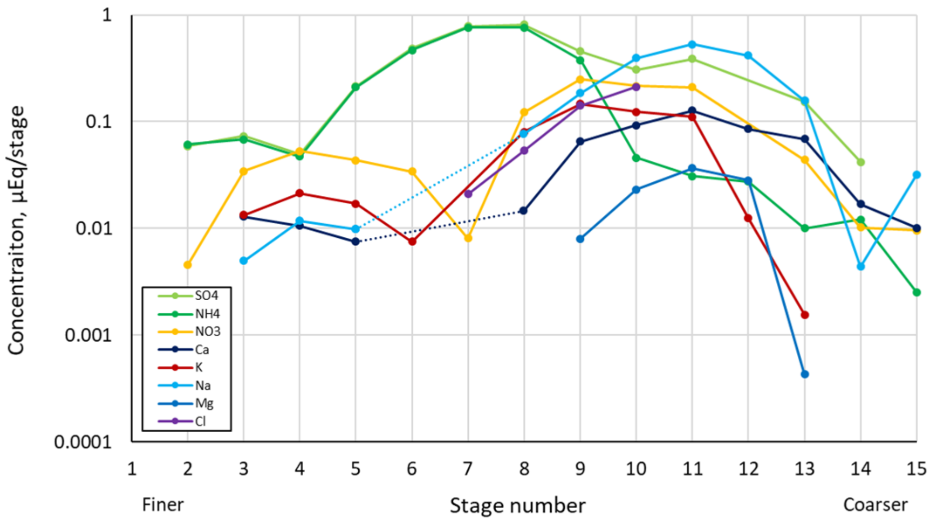

Figure 2.7. Soluble ion concentrations in µEq/stage, indoor test.

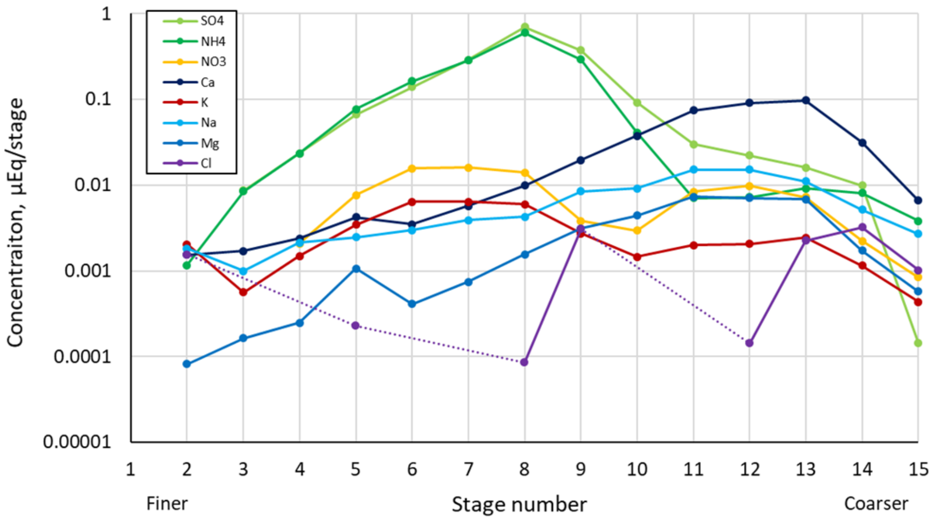

Figure 2.8. Soluble ion concentrations in µEq/stage, outdoor test.

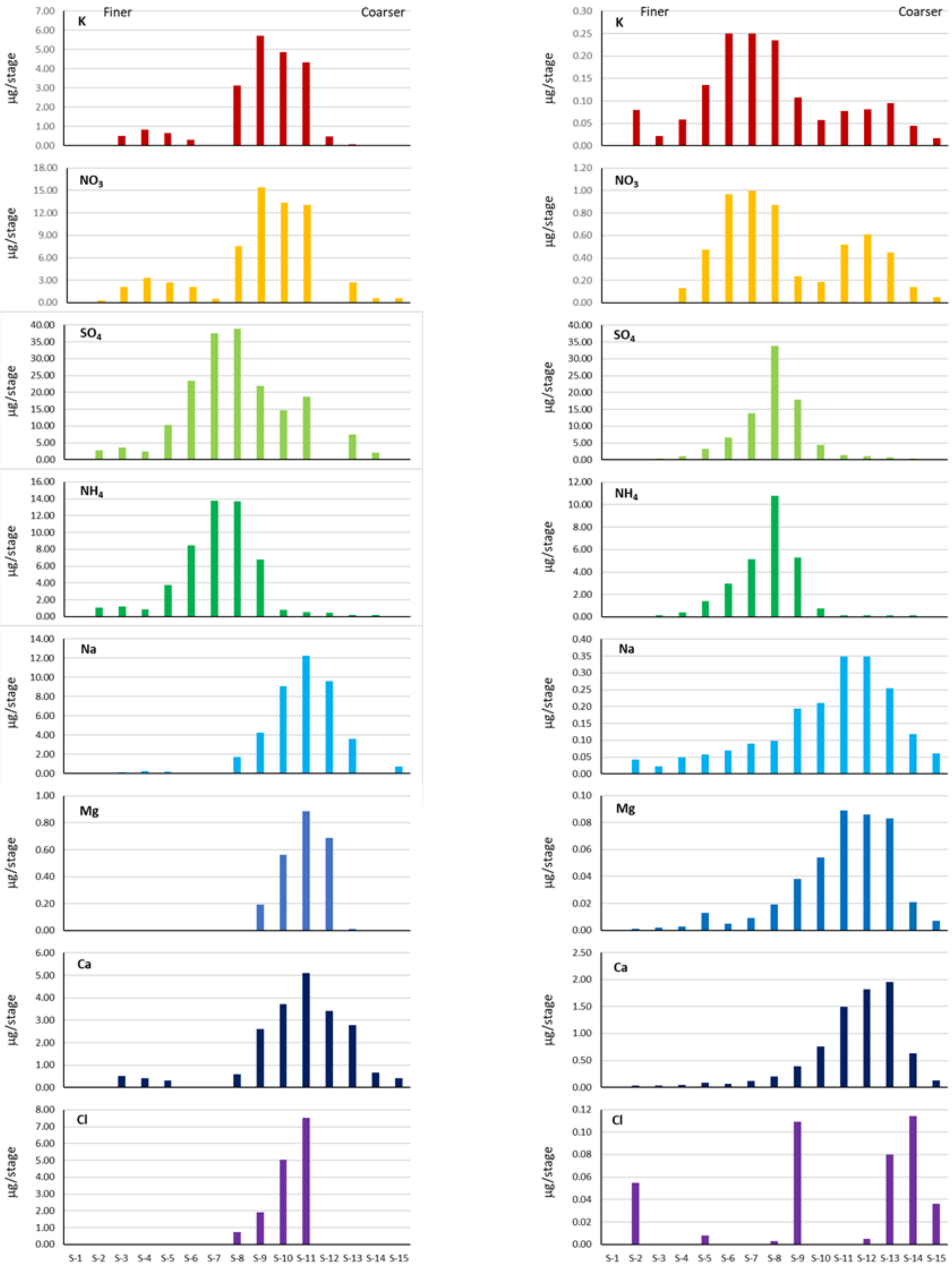

Figure 2.9. Soluble ion concentrations (μEq/stage), as a function of stage number. Left: indoor test; Right: outdoor test.

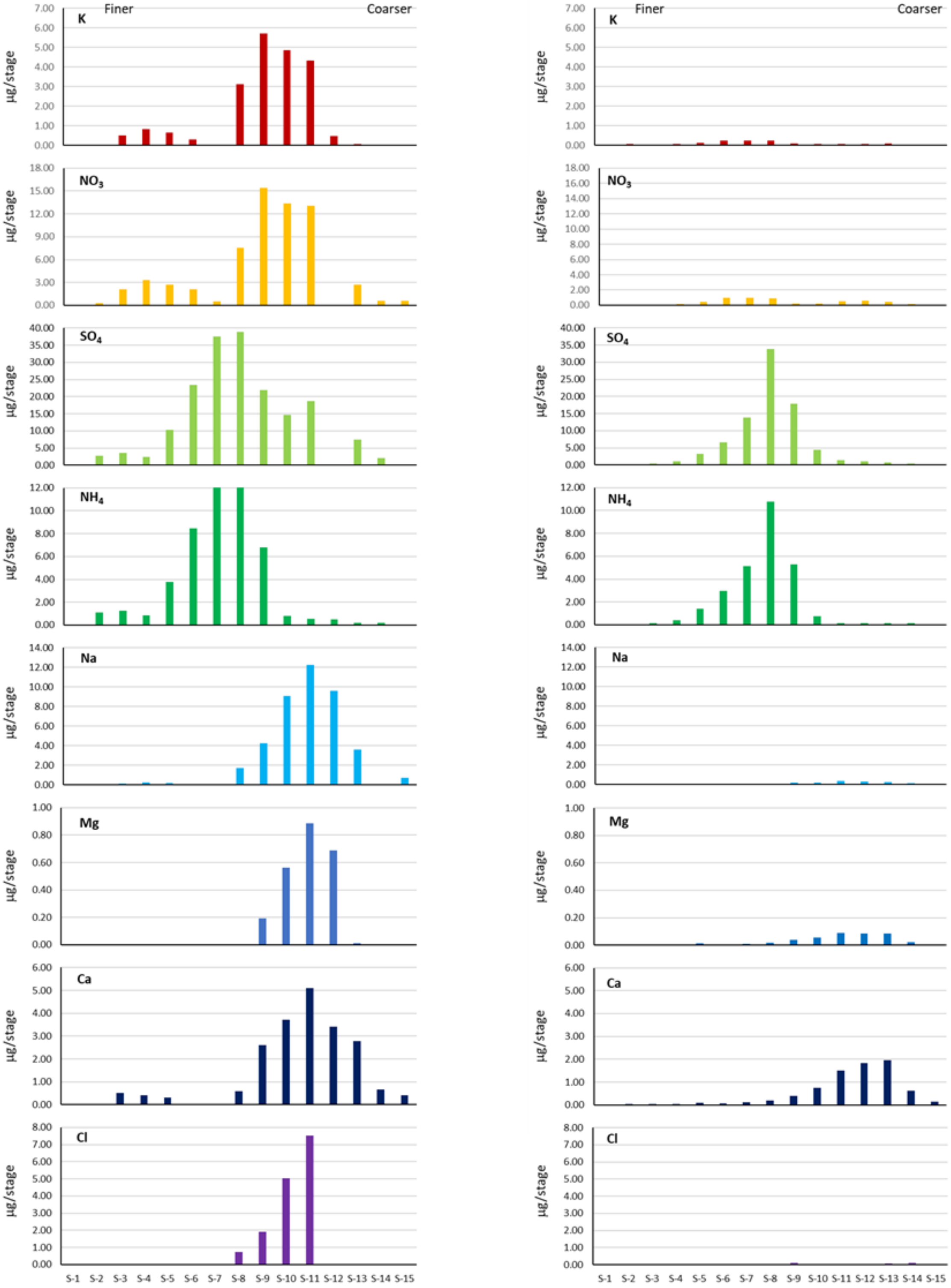

Figure 2.10. Soluble ion concentrations (μEq/stage), as a function of stage number. Left: indoor test; Right: outdoor test. Each solute is shown on the same scale.

One major goal of the indoor and outdoor tests was to become familiar with installation, maintenance, and use of the cascade impactor. An additional goal was to obtain an impactor tower containing used target substrates, to provide to WSP to train them to extract the substrates for analysis and to replace them with clean substrates. The extracted samples also provided SNL an opportunity to evaluate WSP as an analytical lab. Based on these tests, we are confident that SNL can operate the cascade impactor in a field setting, and that WSP can perform the required analyses and will provide excellent data. A contract for analysis of the CDFD samples has been placed with WSP.

2.2 Topas Laser Particle Size Spectrometer

The Topas LAP 323 complements the cascade impactor. The impactor can only collect and size that are less than 10 µm in aerodynamic equivalent diameter when dry, and less than ~20 µm when deliquesced; any larger aerosols are excluded by the TSP inlet. If weather data is also monitored and the RH can be estimated, the actual diameter of the original deliquesced aerosol can be estimated from the impactor data on the dried aerosols. However, at some sites, dried sea-salt aerosols up to 10-15 µm have been observed in dust from canister surfaces; if deliquesced, these could easily exceed the cutoff imposed by the cascade impactor inlet. The laser particle size spectrometer is designed to directly measure aerosol particles >40 µm in diameter. Moreover, at the slower flow rate of the later particle spectrometer (3 L/min), the TSP inlet only cuts particles > 40-50 microns in size. Hence, the laser particle sizer can directly measure a much larger range of aerosol particle sizes, covering the entire likely range for even deliquesced sea-salt aerosols. The LAP323 is equipped with an automated switching valve that allows for sampling the air streams both above and below the dryer. The laser particle sizer will sample continuously at the CDFD site, collecting data in alternating 10-minute increments from each of the two air pathways.

Specific details regarding the LAP323 and its accessories have been provided previously (Bryan et al., 2021; Schaller et al., 2022). Initial tests have been used to develop operating procedures and an understanding of the system (e.g., the relative sampling efficiency of the two flow pathways for different particle sizes). Unlike the ELPI+, there is no preparation prior to data collection. Also, no samples are collected for analysis, because the LAP323 simply provides real-time particle size monitoring. Testing in FY22 focused on determining the sampling rate and understanding the efficiency of the dryer. The sampling rate is an instrument parameter and is the total amount of time the instrument collects and counts particles in any given counting interval. Collecting for longer periods of time provides a great particle count, but at the expense of time resolution. However, if the collection time is too short, the counting statistics may not be sufficient to accurately describe the characteristics of the aerosol population. For example, a 10-minute acquisition time will collect for 10 minutes from one of the flow-paths and report the total counts of particles during that period, and then sample the other flow-path for the same length of time. The 10-minute acquisition time could potentially capture short-term weather events that could affect aerosol particle densities. In contrast, if a one-hour acquisition time is used, the instrument will continuously count particles for 1 hour and report the distribution and total particles over that interval, and then switch to the other flow-path. If a weather event (e.g., wind gust) is brief, changes in aerosol particle density or particle size distribution during that event will be averaged over the entire duration of the sampling period. Moreover, transient weather events that are less than 2 hours long could be captured by only one of the flow paths. This could lead to dramatic misinterpretations with respect to conditions of deposition for the CDFD experiment.

Data collected by the laser particle sizer for the indoor test are summarized in Schaller et al. (2022) and suggested that alternating sampling intervals of 10-minute duration are sufficient to obtain good counting statistics. The data were also used to estimate the relative sampling efficiency of the two airflow pathways. The proportion of aerosol particles that passed through the dryer, relative to those that bypassed it. Two processes could affect the apparent sampling efficiency. First, if the particles are deliquesced, then they would dry out in the dryer pathway, but not in the bypass pathway. This would not affect the overall particle count but would affect the number of particles of a given size. The second effect has to do with the geometry of the two pathways. The dryer pathway is a straight vertical path, but the bypass T's off from the sample string at a right angle, and the sharp curve may affect the efficiency of particle transmission for the bypass pathway. The relative humidity at the SNL site in Albuquerque New Mexico is routinely very low—far too low for deliquescence to significantly affect the aerosol particle size distributions. Hence in the experiments here at SNL, it is assumed that particle size changes due to deliquescence/dry-out are minimal, and that differences in the upstream and downstream count and particles distributions can be attributed to different flow path geometries.

Data from the indoor test were collected over a range of collection times and instrument parameters. The most relevant data were those collected at the same instrument settings as the outdoor test—sampling intervals of 10 minutes, alternating between the airstreams upstream and downstream of the dryer. These data are shown in Figure 2.11. Note that these data only represent 100 minutes of total counting time for each flow path, so counting statistics are relatively poor. This is especially true for particles larger than 4 µm, where few, if any, particles were counted in each size bin. However, it is apparent that the sampling efficiency varies with the two flow pathways. For particles less than 0.4 microns, the upstream pathway that bypasses the dryer has slightly higher particle counts than the dyer pathway. However, at larger particle sizes, it is the downstream pathway through the dryer that shows higher particle counts. Nevertheless, the overall shapes of the two particle size distributions are very similar.

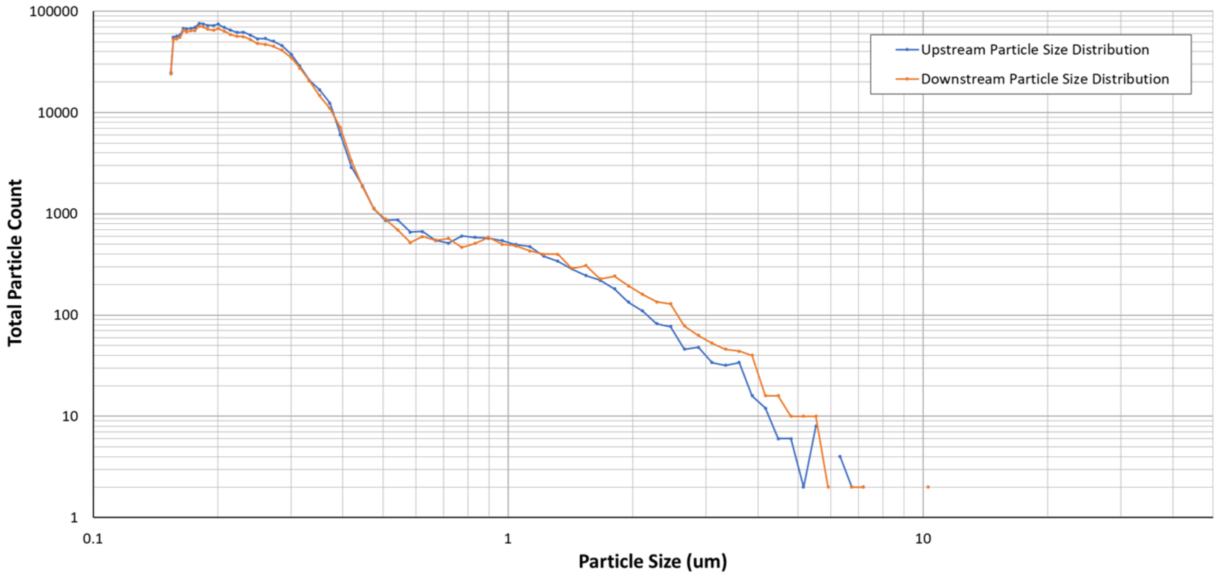

Figure 2.11. Aerosol particle size distributions collected during the indoor test.

Regardless of the pathway, only a small number of larger aerosol particle sizes were collected, so the counting statistics are very poor. Hence, it was not possible to estimate the relative sampling efficiency for the two flow paths for the larger particles. One incentive for the outdoor test was to try and collect more large particles to better estimate the most effective sampling interval and sampling efficiency for those particle sizes.

As noted previously, the outdoor test took place in the winter (late December-early January), and the instruments operated for two weeks. The measured particle size distributions for the outdoor test are shown in Figure 2.12 and Figure 2.13. These data were collected in 10-minute intervals, but for the total experiment duration of two weeks. Hence, the overall particle counts are much higher than shown in Figure 2.11. In this case, the counting statistics are much better, and the data show that the particle size distributions from the upstream and downstream sampling locations are very similar. The only significant difference occurs in the size range from 1-5 microns, where, similar to the indoor test case, the downstream counts are again slightly higher than the upstream counts. Sufficient larger particles were collected to show that the number of particles counted is similar for the two flow paths for sizes up to at least 10 µm—at larger particle sizes, the counts were still too low (less than 10 total particles) to provide adequate counting statistics.

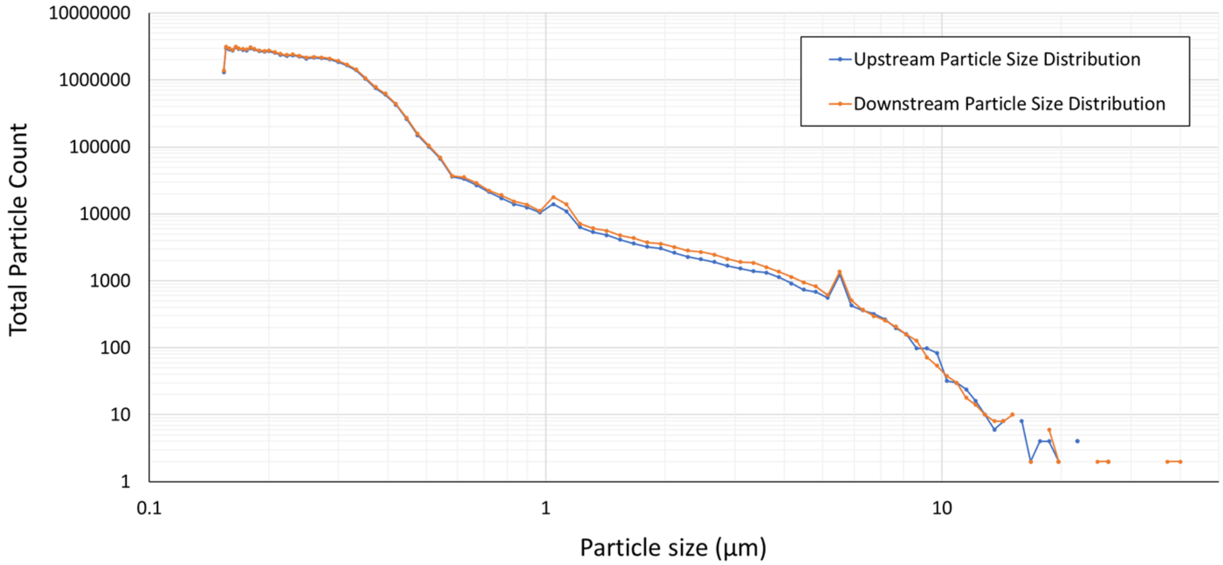

Figure 2.12. Aerosol particle size distributions (total particle count vs. particle size) collected during the outdoor test.

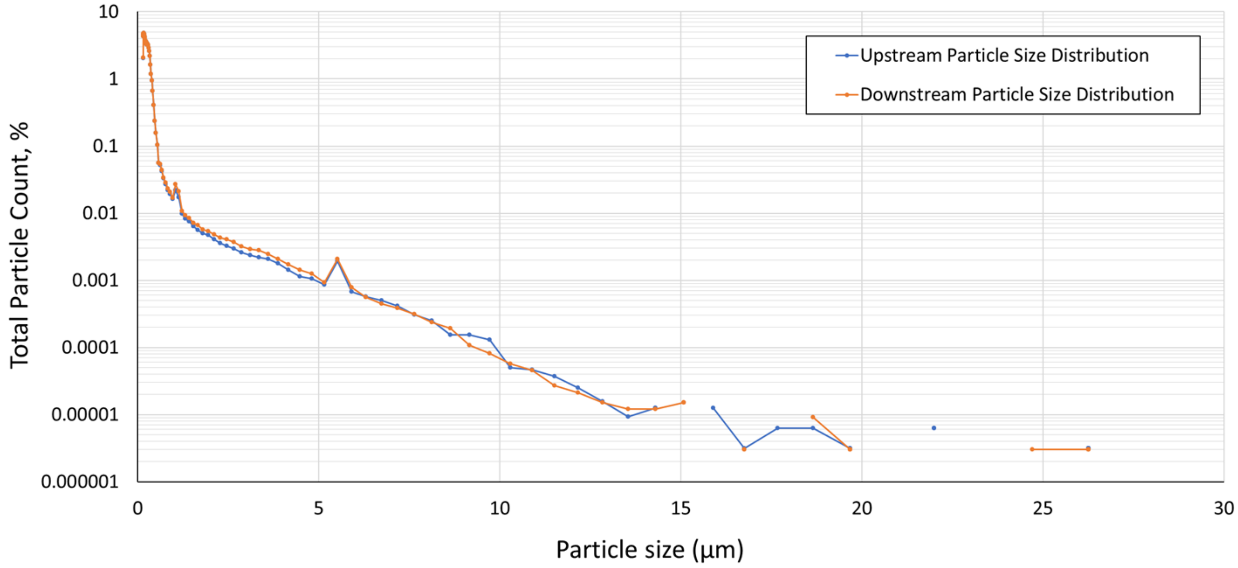

Figure 2.13. Aerosol particle size distributions (% of total particles vs. particle size) collected during the outdoor test.

The sampling efficiency of the upstream flow path relative to the downstream flow path, for both the indoor and outdoor test, is shown in Figure 2.14. The data for the indoor test are highly scattered because the short counting time (100 minutes for each flow path) resulted in poor counting statistics. However, there appears to be slight decrease in sampling efficiency between 1 and 6 µm. The data for the outdoor test show much less scatter because of the long counting times (each flow path was counted for 1 week total), and confirm that the upstream bypass flow path has as much as 30% lower counts in the 1-6 µm particle size range. The relative counting efficiency for the upper path appears to rise to near-100% for particles above 6 µm, but poor counting statistics make this a tentative conclusion. It should be noted however, that the counting efficiency of the dryer itself is not 100%, relative to an inlet string without a dryer. Data provided by Topas indicate that the dryer efficiency drops to about 90% for particles larger than 1µm (Schaller et al., 2022).

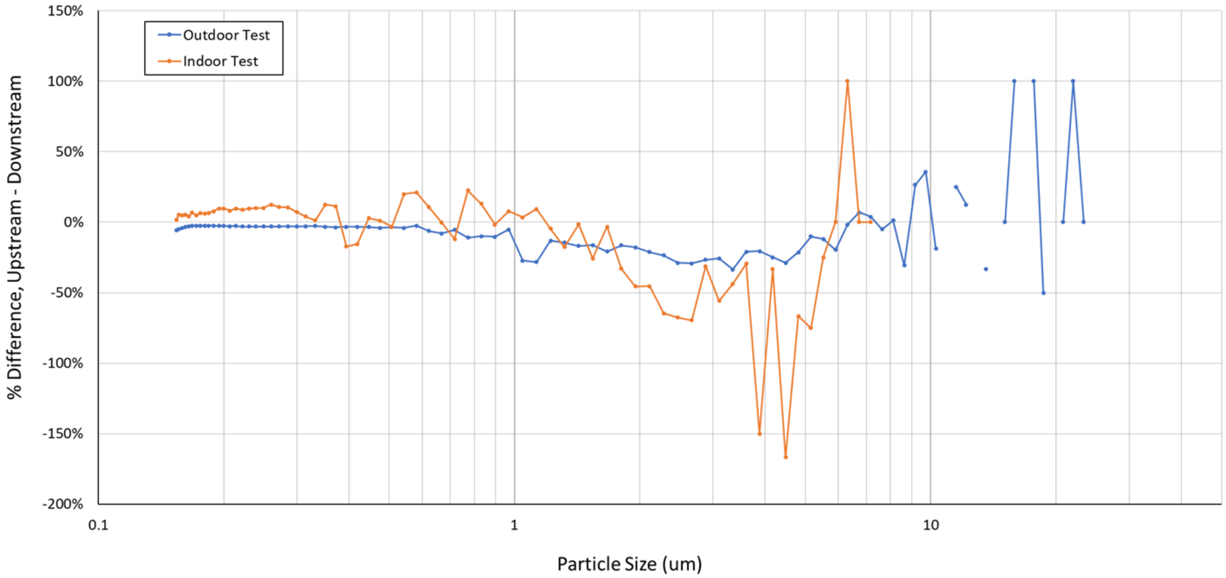

Figure 2.14. Relative sampling efficiency for the upstream flow path, outdoor and indoor tests.

Finally, the data from the outside test allow us to evaluate the appropriateness of the selected 10-minute sampling interval. This interval was selected to be sufficiently long to provide good counting statistics, while being short enough for both paths to capture weather transients, so that the results for the deliquesced and dried aerosols can be directly compared. This can be evaluated by comparing the total particle counts for the two flow paths as a function of time over the duration of the test. These results are shown in Figure 2.15, and it is apparent that in nearly all cases, the same transient events are observed in each flow path. Moreover, in most cases, the number of particle counts are similar. The same data are shown in Figure 2.16, with particle size information included. These data also show that in cases where there are significant differences in total particle counts for the two flow paths, they appear to short-term events involving mostly very small particles. This is hard to explain, as it might be expected that larger particles would settle out of the air column quickly and would be more affected by sharp wind gusts or other short-term weather transients.

These data suggest that, for the conditions sampled (i.e., Albuquerque during a 2-week period in the winter), a 10-minute sampling interval is effective for capturing transients. At least initially, this sampling interval will be used in the CDFD test.

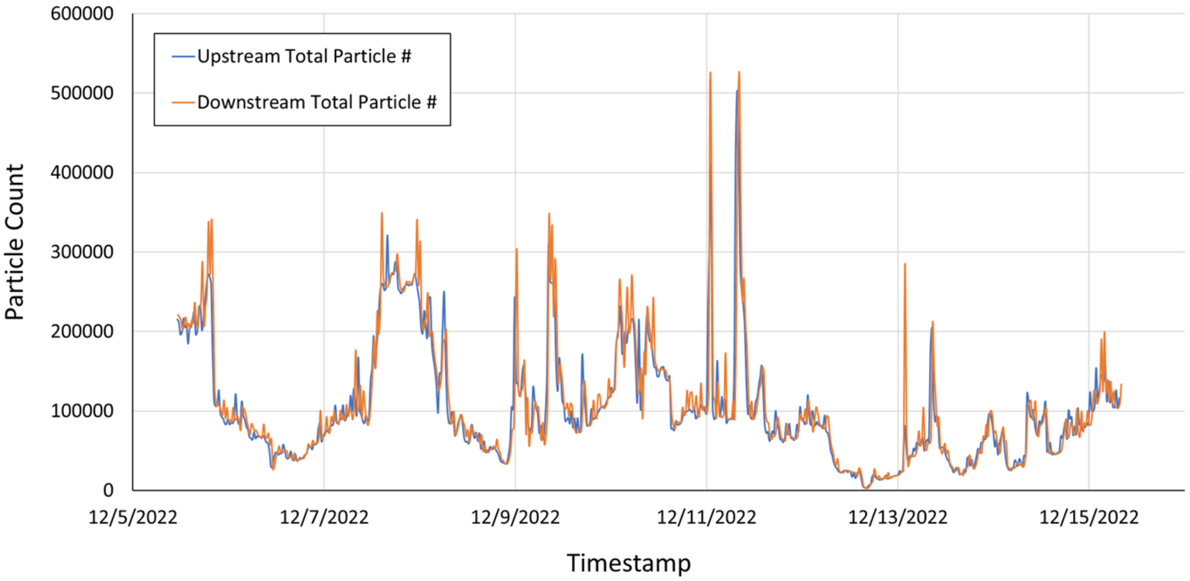

Figure 2.15. Comparison of total aerosol particle counts for the upstream and downstream flow paths, during the outdoor test.

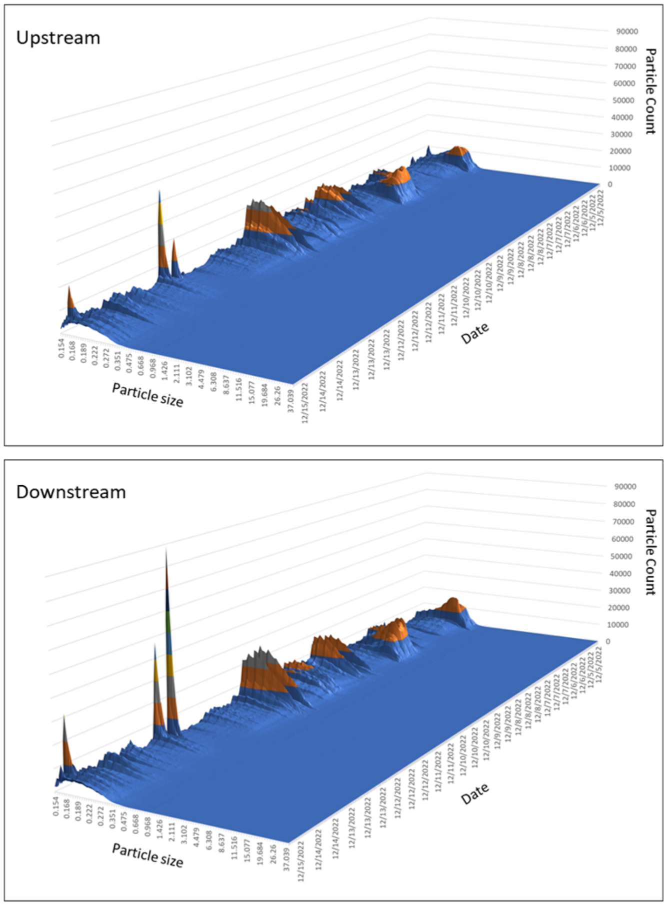

Figure 2.16. 3-D comparison of total aerosol particle counts and particle sizes for the upstream and downstream flow paths, during the outdoor test.

2.3 CASTNET Filter Packs

As discussed previously, CASTNET is a long-term atmospheric monitoring program that is managed and operated by the U.S. EPA in cooperation with the National Parks Service (NPS), Bureau of Land Management (BLM), and many local partners. CASTNET sites measure the ambient concentrations of atmospheric gases and aerosol particulate concentrations and compositions on a weekly basis. These analyses provide information regarding the composition (no size discrimination) of atmospheric particulates and acid gases via analysis of the filter pack collection system; however, the system provides no size discrimination. The common sample analytes for the CASTNET system are bulk cations (Ca^{2+}, Na^+, Mg^{2+}, K^+, NH_4^+), bulk anions (Cl^-, NO_3^-, SO_4^{2-}), acid gases ($HNO_{3(g)}$, $H_2SO_{4(g)}$, $SO_{2(g)}$), and ozone. Ammonia can also be measured. Being a part of the CASTNET network will allow for sample collection, analysis, and maintenance to be performed by CASTNET partners. Filter pack analysis will be provided by WSP Analytical Labs, with a special subcontract to the University of Wisconsin State Hygienic Laboratory to analyze for atmospheric ammonia through the through the Ammonium Monitoring Network (AMoN). These data will provide highly accurate information regarding composition of the atmosphere near the any potential CDFD test site. For the CASTNET system, contracts have been written and will be placed once the site has been finalized.

One critical piece of information that CASTNET can provide is atmospheric acid gas compositions and concentrations. This information is important because the deposited salts can react with atmospheric gases to form different salt assemblages, which will impact the overall composition and deliquescence properties of the salts present on the canister surface.

2.4 Future Work

The two preliminary tests carried out by SNL provide the information required for the field use of these aerosol sampling instruments. In June 2023, the cascade impactor and laser particle sizer will be deployed at a potential CDFD field site, and aerosol data and samples will be collected. The site in consideration is a more humid environment than the SNL site in New Mexico, and the data produced may show that different instrument parameters are required to appropriately characterize the aerosols at the candidate site. The field deployment will provide data on aerosol particle size distributions and compositions at the candidate site, information that is required to determine the appropriateness of the site for hosting the CDFD project. This field test will be an excellent opportunity to demonstrate the capabilities of the instrumentation, and to evaluate if any changes are necessary to obtain the information necessary for parameterization of an aerosol deposition model for SNF dry storage canisters in their overpacks.

3 CONCLUSIONS

This report describes the results of preliminary testing of aerosol monitoring equipment that will be used to continuously monitor the aerosol source term for the multi-year CDFD project. These data are required inputs for the development and validation of models for the deposition of dust and potentially corrosive salts on the surface of SNF dry storage canisters. Surface salt loads correlate with the extent of corrosion damage on a metal surface, and potentially to the likelihood and timing of initiation of stress corrosion cracks.

Aerosols will be monitored at the CDFD site using three instruments. A Dekati® ELPI+ cascade impactor will be used for real-time monitoring of aerosol particle sizes. It will also collect aerosols in 14 size bins on impactor targets that can be chemically analyzed to determine the soluble salts present as a function of particle size. However, this instrument can only measure dried aerosols, with a diameter of <10 µm. The second instrument is a Topas laser particle size spectrometer, which provides real-time monitoring of aerosol particle sizes up to ~40 µm in size. It monitors both the ambient (potentially deliquesced) aerosol particle size distributions required for the dust deposition models and the distributions of the equivalent dried particles, allowing correlation with the Dekati® data. However, it does not discriminate between inert dust particles and salt aerosols, and it does not retain samples of the different particle sizes for later analysis. The third instrument that will monitor aerosols at the CDFD site is a filter pack associated with CASTNET, which uses a multiple canister system to collect weekly samples for analysis to total suspended aerosol particle compositions and atmospheric gas concentrations.

This status report describes work in FY23 to develop the capabilities for using these tools. In two training exercises, the cascade impactor and laser particle sizer were deployed in two different testing environments, one indoor and one outdoor. For the cascade impactor, the tests provided opportunities for the operators to familiarize themselves with impactor substrate preparation, and post-test sample removal and analysis. For the laser particle sizer, the tests were used to evaluate different instrument parameters, to determine the most appropriate settings for capturing transient events. Data and samples were collected for weeks to months for each test, and the results are presented here. In addition to the preliminary testing, contracts were developed with WSP for sample preparation and analysis of the cascade impactor samples. The impactor tower from outdoor test was delivered to WSP and used to train the staff there in disassembly, sample extraction, sample analysis, and tower reassembly with new target substrates. These are tasks that WSP will be performing routinely for the CDFD project. The CASTNET system cannot be purchased or tested until a site has been selected for the project. Work for this FY has been restricted to preparation of contracts for purchasing the CASTNET tower, and for sample analysis, once the tower is in operation.

In June 2023, the two instruments will be deployed at a potential CDFD field site and aerosol data, and samples will be collected. The field deployment will provide data on aerosol particle size distributions and compositions at the candidate site and may be useful in informing the future direction of the CDFD campaign.

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Sandia National Laboratories

APPENDIX E

NFCSC DOCUMENT COVER SHEET[1]

Name/Title of Deliverable/Milestone/Revision No.: M3SF-23SN010207046: FY23 Update: Aerosol Sampling for the Canister Deposition Field Demonstration

Work Package Title and Number: SF-23SN01020704: SCC and Dry Storage Investigations – SNL

Work Package WBS Number: 1.08.01.02.07

Responsible Work Package Manager: Charles R. Bryan/ Charles R. Bryan 5/24/2023
(Name/Signature) Date Submitted

Quality Rigor Level for Deliverable/Milestone[2]	☐ QRL-1 ☐ Nuclear Data	☐ QRL-2	☒ QRL-3	☐ QRL-4 Lab QA Program[3]

This deliverable was prepared in accordance with QA program which meets the requirements of: Sandia National Laboratories

☒ DOE Order 414.1 ☐ NQA-1 ☐ Other

This Deliverable was subjected to:

☒ Technical Review ☐ Peer Review

Technical Review (TR)	**Peer Review (PR)**
Review Documentation Provided	**Review Documentation Provided**
☐ Signed TR Report or,	☐ Signed PR Report or,
☐ Signed TR Concurrence Sheet or,	☐ Signed PR Concurrence Sheet
or,	
☒ Signature of TR Reviewer(s) below	☐ Signature of PR Reviewer(s)
below	

Name and Signature of Reviewers

Carlos Jove-Colon 5/23/2023

NOTE 1: *Appendix E should be filled out and submitted with the deliverable. Or, if the PICS:NE system permits, completely enter all applicable information in the PICS:NE Deliverable Form. The requirement is to ensure that all applicable information is entered either in the PICS:NE system or by using the NFCSC Document Cover Sheet.*

- *In some cases there may be a milestone where an item is being fabricated, maintenance is being performed on a facility, or a document is being issued through a formal document control process where it specifically calls out a formal review of the document. In these cases, documentation (e.g., inspection report, maintenance request, work planning package documentation or the documented review of the issued document through the document control process) of the completion of the activity, along with the Document Cover Sheet, is sufficient to demonstrate achieving the milestone.*

NOTE 2: *If QRL 1, 2, or 3 is not assigned, then the QRL 4 box must be checked, and the work is understood to be performed using laboratory QA requirements. This includes any deliverable developed in conformance with the respective National Laboratory / Participant, DOE or NNSA-approved QA Program.*

NOTE 3: *If the lab has an NQA-1 program and the work to be conducted requires an NQA-1 program, then the QRL-1 box must be checked in the work Package and on the Appendix E cover sheet and the work must be performed in accordance with the Lab's NQA-1 program. The QRL-4 box should not be checked.*