In-Depth Survey Report

EVALUATION OF ENGINEERING CONTROLS FOR MANUFACTURING NANOFIBER SHEETS AND YARNS

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Abstract

This field study addresses the evaluation of exposures and control measures used in a nanomanufacturing facility. The purpose of this study was to investigate the ability of engineering controls to mitigate exposure to engineered nanomaterials during nanomanufacturing. Direct-reading instruments were used to measure the baseline levels of airborne particles and monitor activities in this facility. Integrating all instruments on a mobile sampling cart allowed for the monitoring of spatial and time variation of nanoparticles from processes or tasks.

At this study site, carbon nanotube products were synthesized in furnaces contained within ventilated enclosures. During normal operation, enclosure doors were often kept open to allow access and reduce heat built up by these hot processes. In addition, the low enclosure exhaust flow rates resulted in release of fugitive nanoparticles into the workplace. High concentrations of nanoparticles released during furnace maintenance and product harvesting were observed because both tasks required partially and fully opening furnace chambers. Compared to background (~ 1.0×10^5 #/cm^3), the task of furnace maintenance caused a twofold increase in nanoparticle concentration. Product harvesting also resulted in the release of nanoparticle concentrations at least one order of magnitude (~ 3.0×10^5 #/cm^3) higher than background (~ 2.0×10^4 #/cm^3). Two ventilation systems referred to as exhaust ventilation systems (EVSs) were installed in the furnace room. The EVSs, with integral filters connected to the furnace enclosures, were used in the production area to remove fugitive nanoparticles. Two identical Fast Mobility Particle Sizers were used to assess the filtration performance of the EVSs. One EVS serving two furnaces had higher filtration efficiency (95.80% on average) than the other EVS serving seven furnaces (92.73% on average). The filter inefficiency could be the result of bypass from damaged filter media, faulty seals, and/or sheet metal leaks.

Measurements from the office and laboratory areas also suggested that most nanoparticles below 100 nanometers found in non-production areas likely migrated from the production area. According to the TEM analysis and report done by the University of Massachusetts Lowell (UMass Lowell) researchers, most nanoparticles released from the production processes were not engineered nanomaterials, but soot particles. A second visit by the UMass Lowell team showed that nanoparticle concentrations in the non-production areas decreased after improvement of facility ventilation. Those test results are also summarized in a separate report.

This in-depth field survey provides important observations and measurements regarding ventilated enclosures used during nanomanufacturing. Nanoparticle emissions can be reduced by optimizing the design and operation parameters of the engineering controls and by maintaining a preferred pressurization scheme inside the facility. Maintaining the CNT production area under negative pressure relative to the rest of the plant can be done by setting a flow differential of 5%, but no less than 1,416 liter per minute (i.e., 50 cubic feet per minute), between supply and exhaust flow rates. Using higher exhaust flow rates and keeping enclosures closed should reduce particle emissions. Better containment design to accommodate heat and access for workers can also improve the control efficiency of enclosures. Because hazards and risks related to health effects from exposure to engineered nanomaterials are still unclear, effective control of nanoparticle emissions in the workplace is highly recommended.

Introduction

Background

The purpose of this study, funded by the NIOSH Nanotechnology Research Center (NTRC), is to investigate the effectiveness of control measures used by nanomaterial manufacturers. Potential risks associated with nanoparticle exposure have been identified from toxicological research on engineered nanomaterials [Buzea *et al.*, 2007; International Organization for Standardization, 2008; European Agency for Safety and Health at Work, 2009; Safe Work Australia, 2009a]. Workplace controls have been recommended to prevent or minimize exposure to engineered nanomaterials [Safe Work Australia, 2009b]. The use of engineering controls such as enclosures, fume hoods, glove boxes/bags, cleanrooms, laminar flow clean benches, and local exhaust ventilation in nanomanufacturing workplaces has been reported [ICON, 2006]. However, more detailed information concerning the effectiveness of engineering controls for mitigating exposure to nanomaterials during manufacturing and handling is needed. The results from this study and other planned evaluations in the nanomanufacturing industry could lead to the development of better recommendations for using engineering controls in these workplaces.

This site survey was conducted by NIOSH researchers collaborating with the University of Massachusetts Lowell (UMass Lowell). The study was mainly focused on evaluating the performance of control measures employed by the manufacturer. Real-time measuring instruments were used to monitor nanoparticle emissions from the tasks and processes and to assess the filtration efficiency of the general exhaust ventilation system. Transmission electron microscopy (TEM) sampling was conducted by the UMass Lowell research team to provide qualitative analysis of particulates in the workplace atmosphere. The TEM results are summarized in a separate report prepared by UMass Lowell.

Potential Health Effects of Carbon Nanotubes

The facility that was surveyed manufactures products consisting of long carbon nanotubes (CNTs). A review report from the Institute of Occupational Medicine has identified many similarities between asbestos and high aspect ratio nanoparticles (HARN) [Tran *et al.*, 2008]. Asbestos fibers have been classified by the International Agency for Research on Cancer as carcinogenic for humans. This suggests that HARN (e.g., CNTs) could have similar characteristics to pathogenic fibers. From animal *in vivo* exposure

studies and cell-culture-based *in vitro* experiments, toxicological research on CNTs has shown that these nanomaterials at high doses can contribute to fibrotic lung response, inflammation, and granulomas, and can induce oxidative stress and cellular toxicity. On the basis of conclusions drawn by these toxicological studies, we can readily identify several findings:

(1) **Cytotoxicity order**: Asbestos, recognized as carcinogenic to humans, has fewer toxicological effects than single-walled CNTs (SWCNTs) but is more toxic than multi-walled CNTs (MWCNTs) [Jia *et al.*, 2005; Murr *et al.*, 2005; Tian *et al.*, 2006; Inoue *et al.*, 2008].

(2) **CNT purification**: Purified CNTs are more toxic than their unrefined counterparts [Carrero-Sánchez *et al.*, 2006; Wick *et al.*, 2007]. Moreover, the cytotoxicity of purified MWCNTs can be increased to be more toxic than asbestos [Muller *et al.*, 2005].

(3) **Surface area and surface chemistry of CNTs**: Tian et al. have found that The material with the smallest surface area (SWCNTs in this case) is more toxic than other tested materials [Tian*, et al.*, 2006]. Their results also give a good explanation for the effect of CNT purification: the refining process changes the aggregation state of CNTs and then modifies the surface chemistry.

(4) **CNT structure**: Long MWCNTs exhibit asbestos-like hazards, but short and tangled MWCNTs show no significant toxicity [Poland *et al.*, 2008]. The presumption of the risk associated with long CNTs is that macrophages cannot completely engulf (or phagocytose) long fibers to clear them from tissues; however, effective phagocytosis is completed for short or tangled CNTs to clear them through the lymphatic system [Kostarelos, 2008].

Good summary reports of risk assessment of CNTs are available to the public [Kobayashi *et al.*, 2009; Safe Work Australia, 2009a]. A more detailed and updated list of risk studies for CNTs can be found on the website of NCEO [Nanotechnology Citizen Engagement Organization, 2011].

Due to the high demand and wide market applications for CNTs, the rate or incidence of adverse effects on occupational safety and health from exposure to CNTs could potentially increase. In 2008, the U.S. Environmental Protection Agency (EPA) formally issued a notice to CNT manufacturers to

show its intention to consider CNTs as new chemicals and therefore potentially subject to regulation under the Toxic Substances Control Act (TSCA). Meanwhile, NIOSH provided interim guidance about specific medical screening for workers exposed to engineered nanoparticles including SWCNTs [NIOSH, 2009].

Workers may be exposed to CNTs by way of inhalation, dermal contact, ingestion, or injection during manufacturing and handling of the nanomaterials. Appropriate engineering controls can reduce emissions, providing protection for workers. In addition, wearing personal protective equipment (PPE) in the workplace is strongly recommended.

Published Regulations

There is currently no specific regulatory occupational exposure limit (OEL) for engineered nanomaterials. The Occupational Safety & Health Administration (OSHA) Permissible Exposure Limit (PEL) and the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Value (TLV) for carbon black is 3.5 mg/m^3 on time weighted average (TWA) [OSHA; ACGIH, 2011]. In the British Standards Institution guide [BSI, 2007], benchmark exposure levels (BELs) of 0.01 fiber/mL for insoluble fibrous nanomaterials (such as carbon nanotubes and nanowires) have been recommended. For CNTs, NIOSH is preparing a criteria document with a recommended exposure limit (REL) of 7 $\mu g/m^3$ [NIOSH, 2010].

Manufacturing Facility and Control Measures

The CNT sheets and yarns were manufactured in the furnace room (**Figure 1**). Two doors on the north side of the furnace room were used to access the non-production areas such as laboratories and offices; the door on the west side led to the loading dock. There was no strict rule to close these doors during manufacturing. Doors were kept open sometimes to cool off the furnace room because of the high temperatures generated by the production furnaces and to allow easy access between areas.

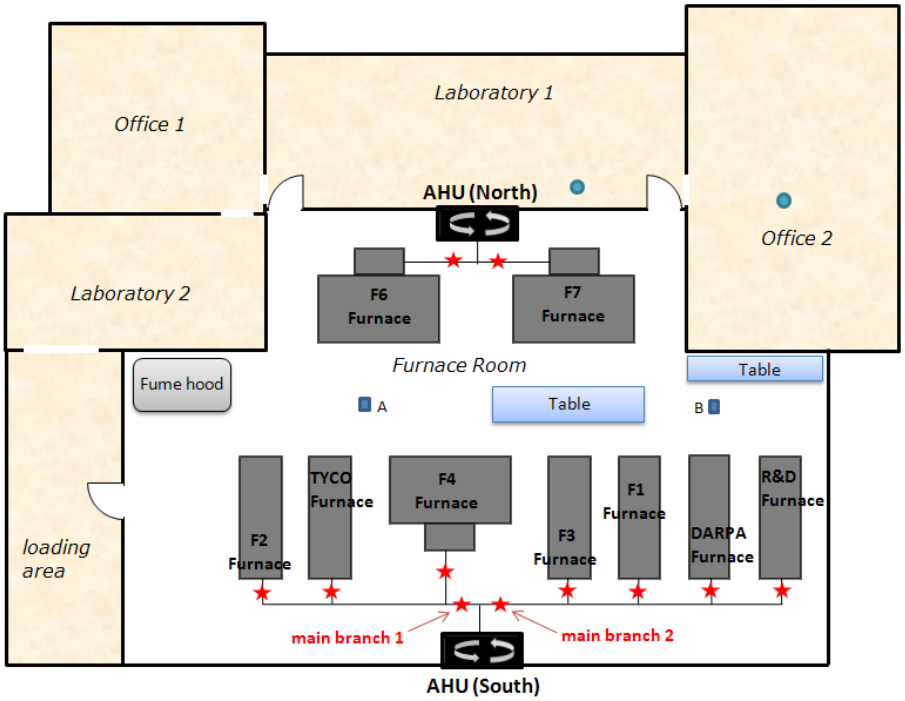


Figure 1: Layout of the facility for site survey. Square dots in the furnace room and circular dots in other rooms show the locations for area monitoring. Star symbols indicate the locations where duct velocities and/or air temperatures were measured.

Product Manufacturing and Harvesting

The company installed nine furnaces in the furnace room; some were used for production, and others were for research activities only. The manufacturing processes were controlled by computer programs. Workers monitored the operating parameters from the computer monitors and observed production of CNT yarns through view windows on the furnaces. Workers used steel rods at the view windows to remove unwanted products inside the furnaces. If necessary, workers opened the view window and inserted a rod into the furnace to perform this task. Like CNT yarn furnaces, view windows and rods were installed on the furnaces for producing CNT sheets. Removing the unwanted products at the CNT sheet furnaces was performed without opening the view windows, but product harvesting required fully opening both the enclosures and the furnaces. Product harvesting was performed by workers manually opening the enclosure and furnace door and then collecting the product with a roller.

Engineering Controls

Two exhaust ventilation systems (hereinafter called the North EVS and the South EVS) were installed in the furnace room (**Figure 1**). Both EVSs were integrated with prefilters and main filters to remove airborne nanoparticles from exhaust air taken from the furnace enclosures. The fan speed of EVSs can be adjusted by a frequency inverter. Normally, the exhaust fan was operated at 45 Hz. Full speed can be reached by increasing the operating frequency to 60Hz.

Every furnace was contained by an enclosure whose exhaust was connected with EVSs to contain nanomaterial emissions from the furnaces (**Figure 2**). Furnaces F6 and F7 were served by the North EVS, and the other seven furnaces were served by the South EVS. Some enclosure doors were normally kept closed during production processes except for cleaning chambers and harvesting final products. Others were open for easy access and to abate excess heat from the furnaces.

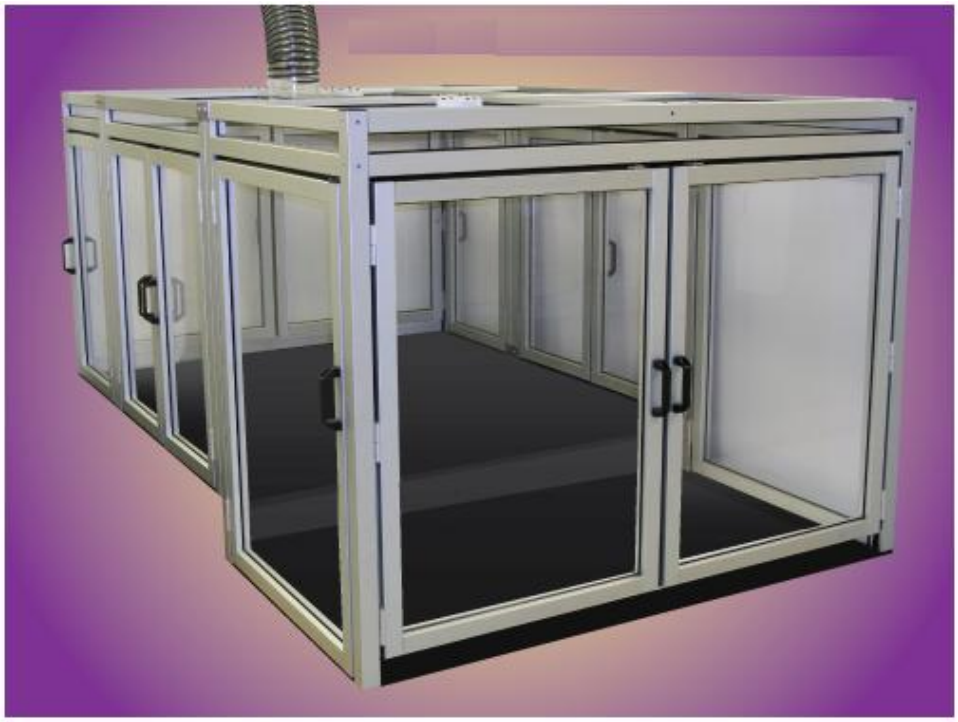


Figure 2: A ventilated enclosure similar to this one was used to contain production furnaces to mitigate particle emissions in the workplace (Modified and reprinted from Flow Sciences Inc.).

Methodology

Area monitoring was conducted at locations A and B (see **Figure 1**) representing the sites with lower and higher traffic flow and production activities, respectively. The data can be used to check general air quality of the furnace room and to help identify sources releasing engineered nanomaterials during process monitoring. In addition, the airborne particle concentrations in the non-production areas were measured by the instruments to characterize possible migration of particles from production areas to non-production areas of the plants.

EVSs play an important role in removing contaminants generated from furnaces and tasks in the furnace room. Testing of these exhaust systems can verify their ability to handle the existing production capacity. The testing was done by inspecting air movements and currents of enclosures and EVSs. It was also useful to assess the filtration efficiency of EVSs to improve the control effectiveness.

Instrumentation

Direct-reading instruments were used in the survey to evaluate the effectiveness of enclosures and EVSs used in this facility [Brouwer *et al.*, 2004; Demou *et al.*, 2008; Peters *et al.*, 2009]. They can be operated in real-time mode to provide continuous measurements of concentrations that can be correlated with the specific production equipment and work processes. Measurements of number, size, mass, and surface area concentrations of nanomaterials are needed because there are no established exposure criteria [Mark, 2007]. The instruments used to measure particle concentrations in this survey were the Fast Mobility Particle Sizer (FMPS) spectrometer, Aerodynamic Particle Sizer (APS) spectrometer, and DustTrak aerosol monitor.

An FMPS (Model 3091, TSI Inc.) was used to identify particle emissions in this in-depth study. The FMPS uses a corona charger to positively charge particles and simultaneously determines number size distributions with an array of electrometers. It is capable of measuring particle sizes ranging from 5.6 to 560 nanometers (nm) with a resolution time of 1 second. Real-time measurement allows the determination of fluctuations of size/number distributions of released nanoparticles in the nanomanufacturing workplace. The FMPS has been previously used in field studies for exposure assessment [Bello *et al.*, 2009; Tsai *et al.*, 2009].

Aged nanoparticles, originally released from any large-scale nanoparticle manufacturing process, tend to agglomerate to become larger sized particle clusters. Using a light-scattering technique, an APS (Model 3022, TSI Inc.) was used to measure aerodynamic diameters ranging from 0.5 to 20 micrometers (µm). Like the FMPS, the sampling frequency of the APS can be as short as 1 second. Therefore, the measurement results from the APS and FMPS can provide a full spectrum of airborne particle size and number distributions in work areas.

Mass concentration is traditionally used as a metric for exposure assessment. A real-time laser photometer, the DustTrak DRX aerosol monitor (Model 8533, TSI Inc.), was used to measure mass concentrations of contaminants for this survey. It can measure particles ranging from 0.1 to 10 µm at concentration ranges between 0.001 and 150 mg/m^3 and can display mass fractions in the modes of PM_1, $PM_{2.5}$, PM_{10} (respirable particulate matter), and total mass concentration.

Real-time direct-reading instruments (FMPS, APS, and DustTrak) and a laptop computer were integrated by NIOSH researchers on a mobile sampling cart as shown in **Figure 3**. The same models of the FMPS and APS were brought by the UMass Lowell researchers to facilitate data collection during the field survey. In addition to collecting the data of particle concentrations, face velocities and duct flow rates of enclosures were measured by a velocity meter (VelociCalc Plus hot wire anemometer, Model 8386, TSI Inc.) and a pitot tube.

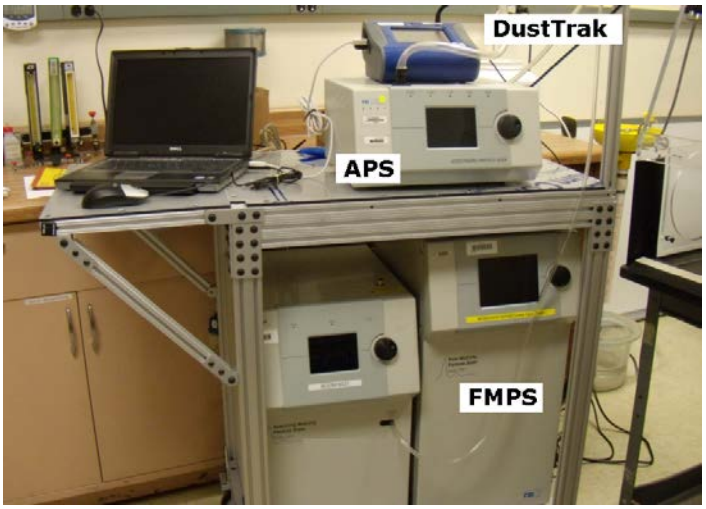


Figure 3: Sampling instruments (including the FMPS, APS, and DustTrak) to monitor aerosol emissions.

Sampling Plan

Process Monitoring

Nanoparticle concentrations were monitored during typical worker tasks. Operators were seated in front of some CNT yarn furnaces to monitor the processes. For some furnaces, workers kept the enclosure doors open most of the time to observe the products being processed inside the furnaces and to allow excess heat from production to escape the enclosures and go into the workplace. Steel rods at the view windows of furnaces were used occasionally to accumulate unwanted products inside furnaces during production. If necessary, workers would open the enclosure doors and the view windows to perform maintenance tasks such as furnace cleaning (by using steel rods to remove the unwanted accumulated products) during production. To open a view window, workers needed to first disconnect the tube between the process chamber and the view window. Product harvesting from CNT sheet furnaces is another task that requires opening the enclosure and furnace doors, often for approximately 10 minutes.

During this survey, several production furnaces including DARPA, F1, F3, F4, and F6 were monitored during regular operation. As shown in **Figure 4**, particle concentrations were measured for 2 minutes at the worker's breathing zone, at the view window of furnaces (potential particle source), inside the furnace enclosure, and around the monitored enclosure (for process background). When the furnace chamber was opened for cleaning, however, the sampling point was specifically kept near the view window to monitor nanoparticle emissions.

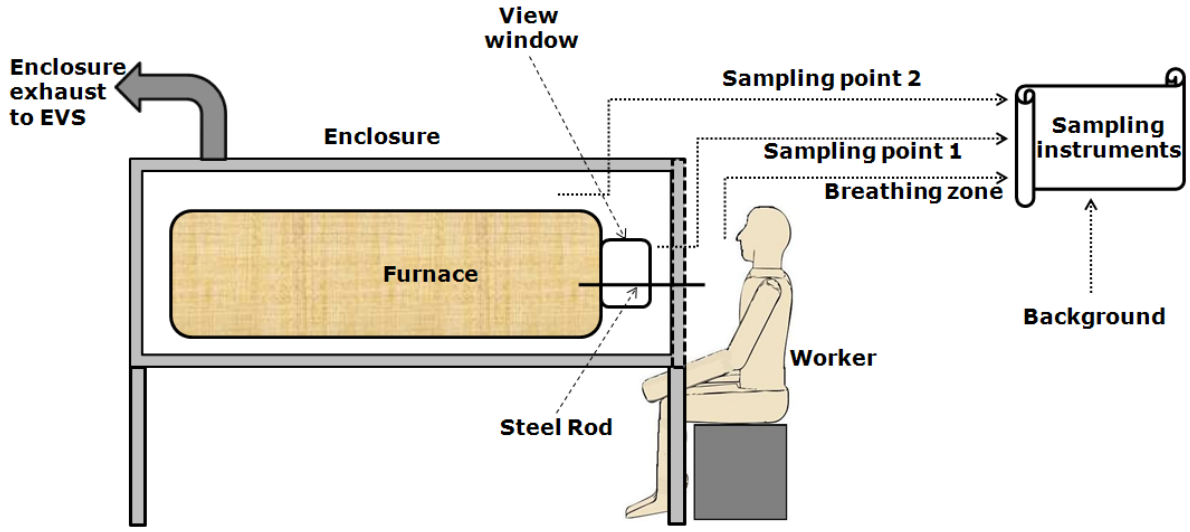


Figure 4: Sampling locations during process monitoring. Sampling point 1 was located at the view window to assess the process as a source of nanoparticles. Sampling point 2 was located inside the furnace enclosure to provide information on the source.

Filtration Efficiency Evaluation

Pre-filters and main filters were installed in both production area EVSs, and the pressure difference across the filters was routinely monitored according to the facility manager. The primary filters used in the EVSs were 24 x 24 x 11.5-inch, 95% efficiency pleated filters manufactured by Flanders (Washington, NC). These filters are rated at resistance of 1.0-inch water gauge (w.g.) at a flow rate of 200 cubic feet per minute (cfm). Two sampling ports were located at positions upstream and downstream of the primary filter to evaluate the filtration efficiency (**Figure 5**). Dual FMPSs were used to measure upstream and downstream concentrations at the same time for 5 minutes. The same 5-minute measurement cycle was repeated by switching FMPSs to monitor different sampling ports of the EVS to help eliminate any instrument bias. The EVS efficiency (*E*) was calculated by

$$E = \frac{N_{upstream} - N_{downstream}}{N_{upstream}} \times 100\% \quad [1]$$

where *N* refers to the average or the sum of total number concentrations over the 5-minute cycle.

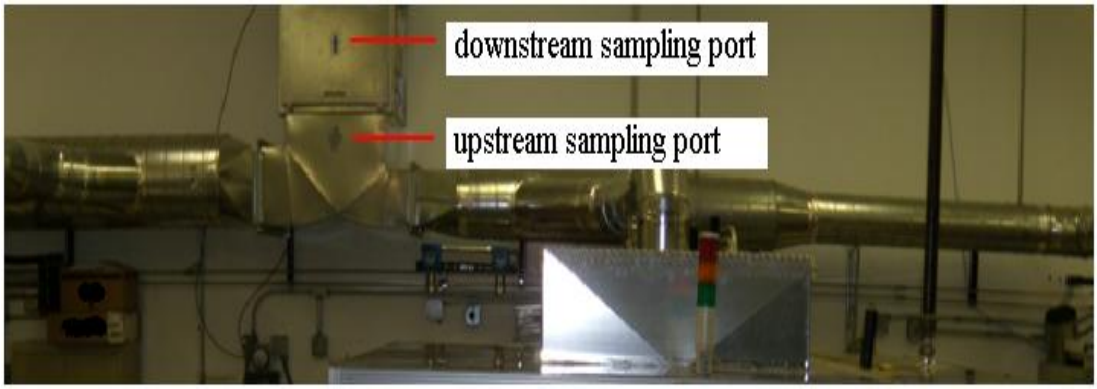


Figure 5: Exhaust ventilation system (EVS) where the locations of upstream/downstream sampling ports are shown.

Air Flow Measurements

A thermal anemometer was used to make face capture velocity measurements across the opening of each ventilated enclosure using. For this study, the open face of each ventilated enclosure was divided into six equal area square grids. Measurements were taken at the center of each grid and perpendicular to the plane of the opening. The air temperature in each duct was also measured with the same hotwire anemometer by inserting the probe through pre-drilled holes in each duct and waiting for the air temperature reading to stabilize.

A pitot tube was used to measure velocity pressure in each duct. Two 10-point orthogonal traverses were performed per duct to determine average duct air velocity [ACGIH, 2007]. The traverses were performed in the duct that connected each ventilated enclosure with the main duct and again between the main duct and each EVS. Air velocity in the duct was calculated from the velocity pressures, and volumetric flow rate through each duct was determined by multiplying the average velocity by the cross-sectional area of the duct.

Results and Discussions

second for real time monitoring to characterize process emissions. In general, the data obtained from the APS and DustTrak show no significant variation because the processes and tasks generated very fine particles that are not in the APS or DustTrak size range. Therefore, the data discussed in the following sections were those obtained from the FMPSs (hereinafter called FMPS 1 and FMPS 2). The variation of particle concentrations is expressed in terms of 15-second moving averages to smooth out short-term fluctuations from data collection at every second and to highlight long-term trends.

Area Monitoring

The total number concentrations and the particle size distributions from area monitoring are summarized in **Table 1** and **Figure 6**, respectively. Large fluctuations of area concentrations were found in the furnace room, because of the high frequency of staff movement and production activities. The lowest average area concentration was found on September 22 during instrument setup and initial facility sampling. During this sampling period, the door on the west side of the production area (**Figure 1**) next to the loading dock was kept open. The area concentrations on September 23 and 24 were higher when all the room doors were kept closed. The area data measured on September 23 by both FMPSs at different locations were comparable. The evaluation of EVS filtration efficiency was planned on September 24. To understand instrumental bias, two FMPSs were put side-by-side at location B to measure area concentrations on September 24. However, the average total number concentration obtained from FMPS 2 was found to be 31.8% higher than that from FMPS 1.

In general, particle size distributions shown in **Figure 6** were polydisperse with maxima at 10 and 20 nm, except for the distribution obtained from FMPS 2 on September 24. Particles > 30 nm contributed to the higher total number concentration obtained from FMPS 2 on September 24. It was also shown that the area concentrations could change noticeably between different working periods. For example, a higher average concentration in the afternoon was measured at location A on September 23 (**Table 1**). The area monitoring data in the furnace room showed that production activities generated high number concentrations (overall average ~ 7.98×10^4 #/cm^3).

Table 1: Summary of area monitoring data as measured by the FMPSs in this survey (see **Figure 1** for sample locations in the facility).

Date	**09/22/2010**	**09/23/2010**			**09/24/2010**	
Instrument	**FMPS 1**	**FMPS 1**		**FMPS 2**	**FMPS 1**	**FMPS 2**
Sampling location	**A**	**A**		**B**	**B**	**B**
Sampling time period	15:46 ~ 17:18	9:10 ~ 10:00	12:00 ~ 13:30		07:35 ~ 09:40	
Average total number concentration [#/cm^3]	4.19E4	6.11E4	8.98E4	9.08E4	5.08E4	6.50E4
Standard deviation [#/cm^3]	1.21E4	9.39E3	1.46E4	1.45E4	1.63E4	1.79E4

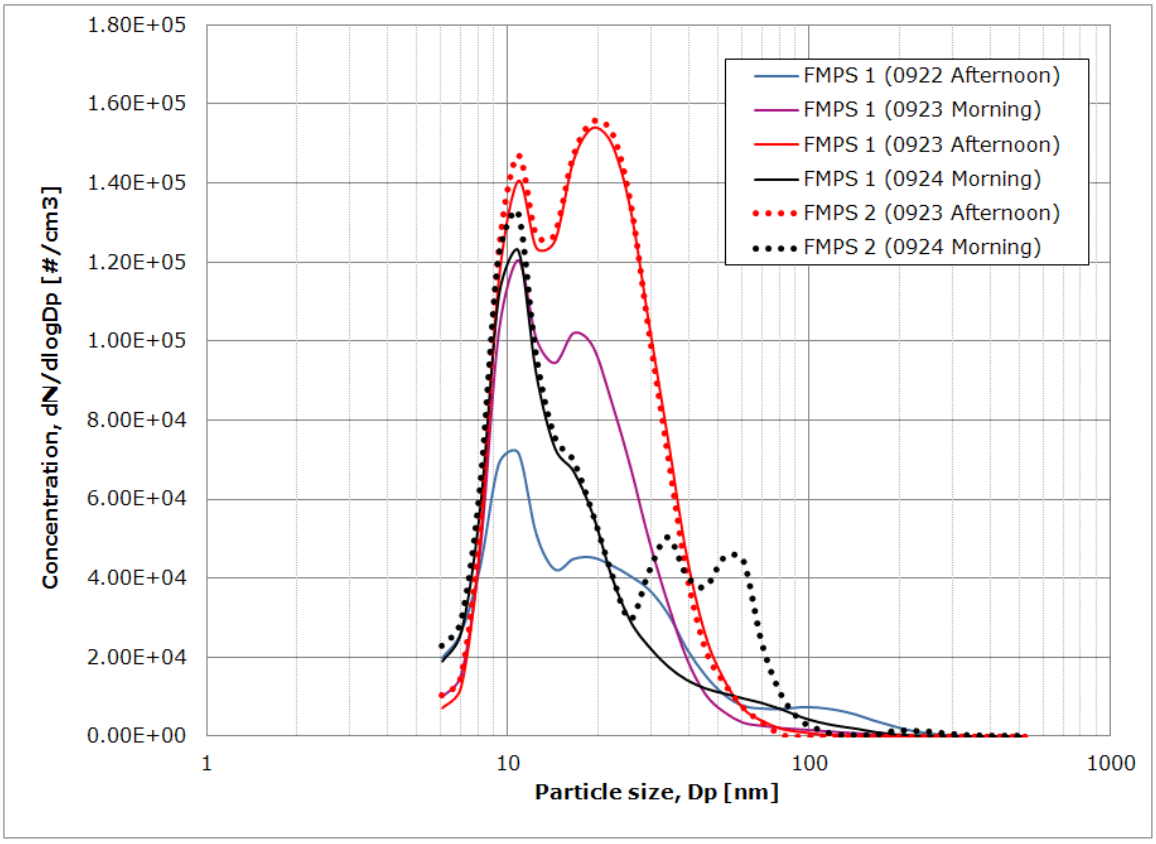


Figure 6: Average particle size distributions measured by two FMPS spectrometers for area monitoring on different dates and times.

Process Monitoring

To identify particle emissions and migration, production furnaces were monitored during regular operation and maintenance. Five-minute sampling periods were performed at different locations shown in **Figure 4** for regular operation. Maintenance tasks, however, were monitored throughout the entire length of the activity.

The sampling results for the DARPA, F1, F3, F4, and F6 furnaces during regular operation are summarized in **Table A-1** of the Appendix. No sampling was done on other furnaces, because they were only used in R&D activities. It was noted that (1) the concentrations near the worker breathing zone and background were similar, and (2) the concentrations inside enclosures were lower than those outside enclosures for most furnaces except Furnace F6. Higher concentrations inside the enclosure of Furnace F6 can be attributed to the better enclosure (less open area). The findings indicated that reducing furnace enclosure openings along with improved ventilation can lower workplace particle emissions.

In addition, cleaning the furnace chamber during production can result in the release of nanoparticles into the workplace. The maintenance performed for the DARPA Furnace released nanoparticles at levels up to two times higher than the background concentration ~ 1.0×10^5 #/cm^3 (**Figure 7**). The front and side enclosure doors of the DARPA Furnace were usually kept open during production. The nanoparticle concentration increased once the worker opened the view window, and it reached the maximum after the worker inserted a rod into the view window for cleaning unwanted byproducts. The particle concentration did not fall to its normal level immediately after the instrument was moved away from the DARPA Furnace for background checks. It took nearly 3 minutes to reach regular background levels. This time lag suggests that the nanoparticles generated by the maintenance task were not effectively contained by the enclosure and were dispersed into the workplace air. This finding was confirmed by the monitoring data from Furnace F1 discussed later.

Average size distributions measured by FMPS 1 during different sampling periods before and after maintenance was performed on the DARPA Furnace are summarized in **Figure 8**. The maintenance task generated very fine particles around 10 nm. The average 10-nm particle concentration during maintenance (~ 3.2×10^5 #/cm^3) increased three times higher than the background levels prior to maintenance (~ 1.1×10^5 #/cm^3).

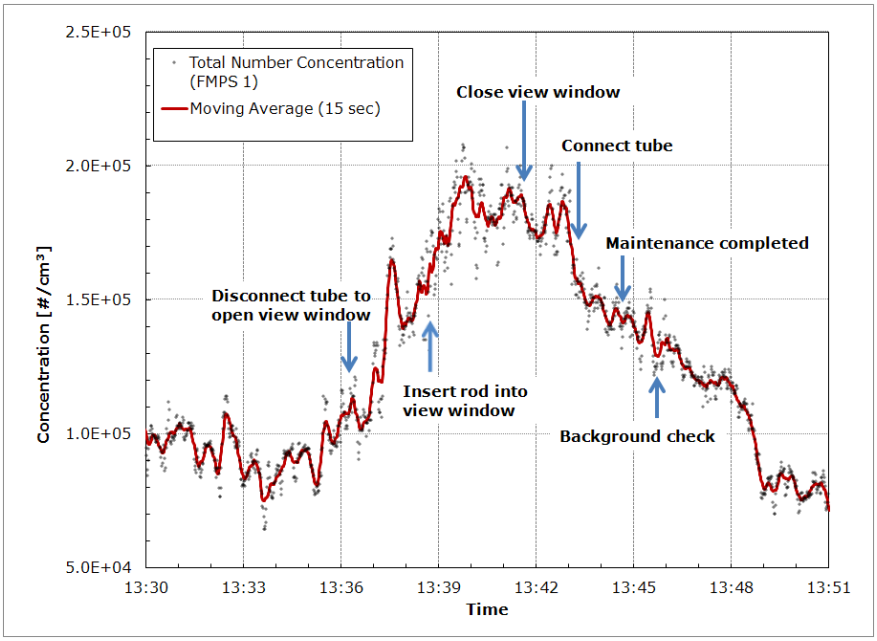


Figure 7: Process monitoring by FMPS 1 for the DARPA Furnace during maintenance on 09/23/2010.

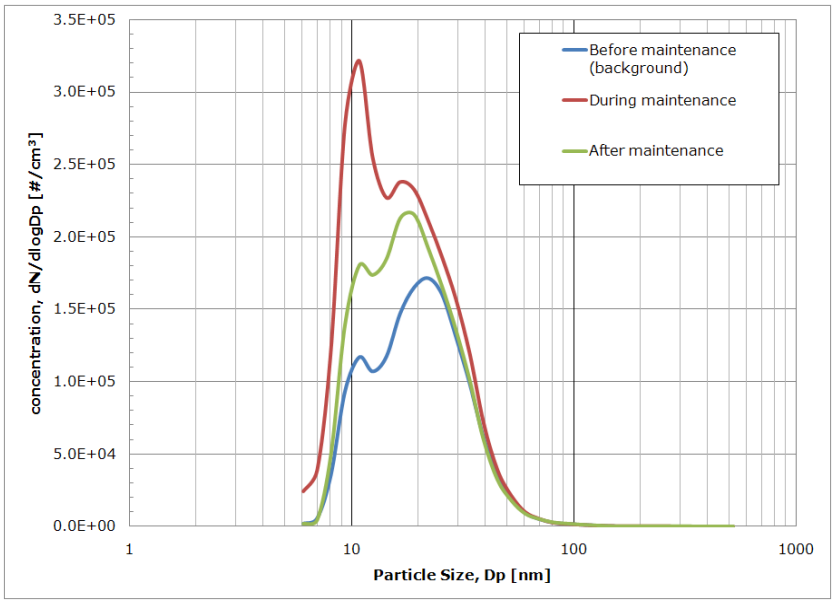


Figure 8: Average size distributions during the maintenance task performed at the DARPA Furnace monitored by FMPS 1 on 09/23/2010. The size distribution after maintenance was calculated by the data between 13:45 (starting background check) and 13:49 (reaching the normal background level) as shown in **Figure 7**.

Monitoring Furnace F1 (**Table A-1**) on September 23 showed higher concentrations inside the F1 enclosure while the enclosure was kept open. During this sampling, maintenance tasks were performed on the neighboring furnace, DARPA. The sampling of the F1 furnace was halted, and the instruments were moved to the DARPA Furnace to monitor particle emissions. The sampling of Furnace F1 was resumed after the maintenance at the DARPA Furnace was finished. Increased background concentrations were found by FMPS 1 after it was moved back to F1 to continue sampling at 15:24 (**Figure 9**). Similar information was found from the average size distributions presented in **Figure 10**. The sampling results were likely influenced by the maintenance tasks done for the adjacent furnace. This finding indicated that the enclosures did not effectively control particle emissions during some specific tasks, such as opening the enclosures for checking and maintaining the furnace. The FMPS 2 was used in parallel with the FMPS 1 to monitoring Furnace 1. Similar results were found from the FMPS 2, and are shown in **Figure A-1** and **Figure A-2**.

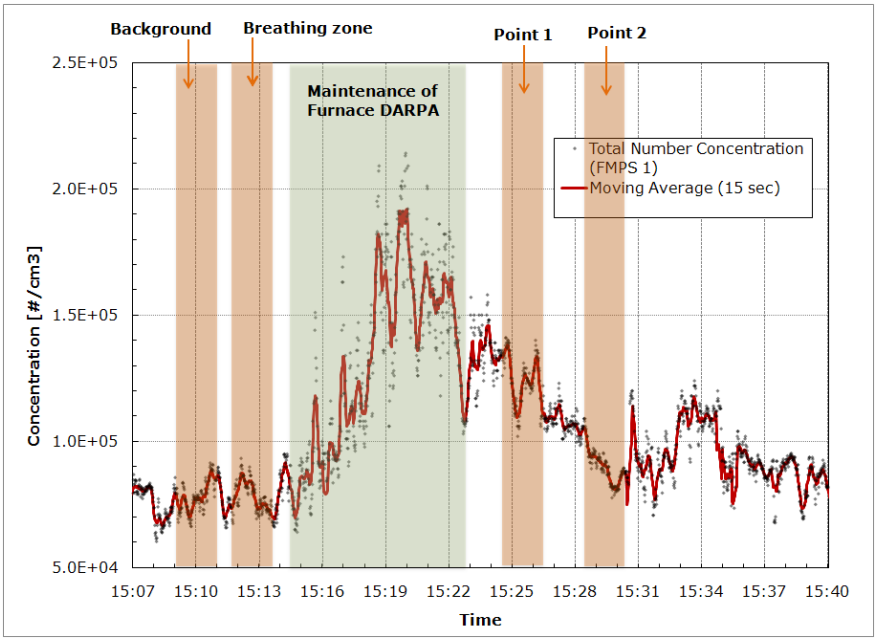


Figure 9: Enclosure test by FMPS 1 for Furnace F1 on 09/23/2010. The sampling procedures were marked by colored columns to highlight sampling durations and locations. The sampling was halted by moving the sampling ports into the DARPA Furnace to check particle emissions from its 6-minute maintenance operation. The enclosure test was resumed by finishing two measurements inside Furnace F1.

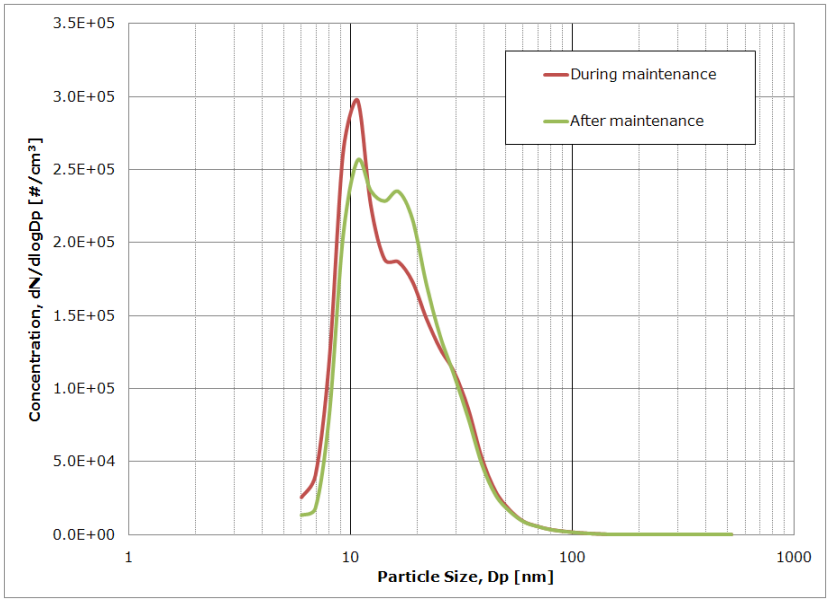


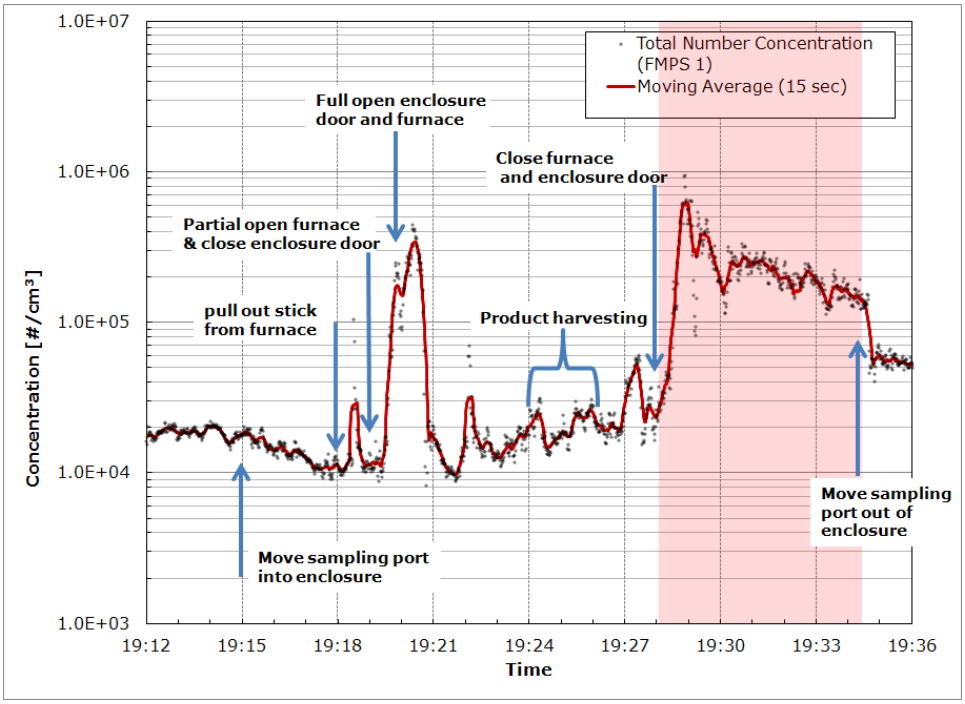
Figure 10: Average size distributions from the maintenance tasks performed at the DARPA Furnace monitored by FMPS 1 during process monitoring for Furnace F1 on 09/23/2010. The size distributions after maintenance were calculated with the data collected between 15:22 (stopping maintenance monitoring of the DARPA Furnace) and 15:24 (before sampling at point 1 inside the F1 enclosure) from **Figure 9**.

Product Harvesting

Product harvesting from Furnace F4 was monitored by the FMPS 1 on September 23 (**Figure 11-a**). High spikes of nanoparticles were identified by the FMPS after opening and closing the enclosure and furnace doors during product harvesting. Particle concentrations increased to the level at least one order of magnitude higher than background (~ $2.0x10^4$ #/cm^3) when both the enclosure door and furnace were fully open for product harvesting. The background concentration remained at a higher level (~ $5.0x10^4$ #/cm^3) even after completing the task. Particle size distributions during different stages are presented in **Figure 11-b**. Large quantities of nanoparticles around 10 nm were released during product harvesting.

After product harvesting, the sampling port was kept inside the enclosure to monitor the temporal concentration variations when the enclosure door was closed (see the marked area in **Figure 11-a**). The particle concentrations were decreased linearly from ~ $6.2x10^5$ #/cm^3 to ~ $1.3x10^5$ #/cm^3 in 6 minutes. The data showed that the F4 enclosure removed 79% of air contaminants in 6 minutes under normal operation (i.e., 45 Hz fan speed).

(a)



(b)

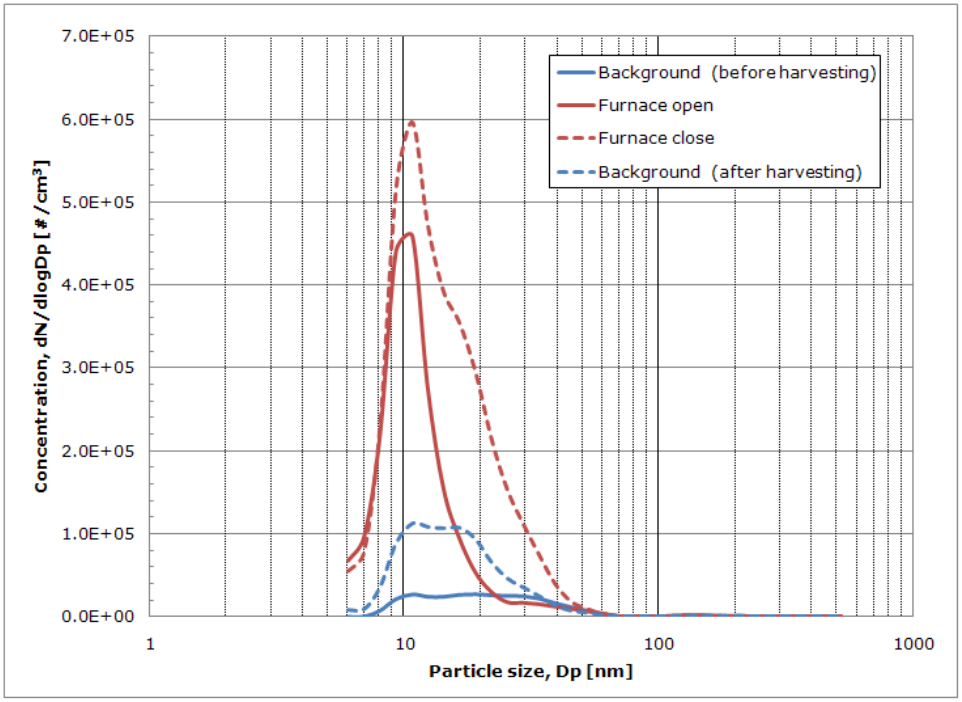


Figure 11: Process monitoring by FMPS 1 on 9/23/2010 for product harvesting from Furnace F4; (a) particle concentration over entire process, and (b) average particle size distributions during different stages of F4 product harvesting.

EVS Efficiency Evaluation

Dual FMPSs were used to measure the upstream and downstream concentrations of both EVS systems to evaluate their filtration efficiency. The electrometers of both FMPSs were re-zeroed before taking measurements, because the area monitoring in the morning of September 24 showed differences between the two instruments' response. A five-minute background checks for the instruments indicated that the average total number concentration from the FMPS 1 (~ 1.19×10^5 #/cm^3) was comparable to that from the FMPS 2 (~ 1.09×10^5 #/cm^3).

Five-minute sampling results are summarized in **Table 2**, where SUM and AVG represent the summation and the mean values of total number concentrations from 5-minute measurements. The results of EVS efficiency from every test were calculated from equation [1]. The data of EVS efficiencies were calculated based on intra-instrument (efficiency is calculated with readings from different instruments) and inter-instrument (efficiency is calculated with readings from same instruments) measuring results.

The results from intra-instrument data (i.e., Test 1 and Test 2 data) showed that the filtration efficiency of the North EVS (95.80% on average) was better than that of the South EVS (92.73% on average). The test data can be recalculated from the inter-instrument basis assuming two FMPSs were used independently. The inter-instrument data indicated that the efficiency of North EVS (95.28% on average) was still better than that of South EVS (90.02% on average). Because the particle concentrations were not constant and consistent over the sampling periods, the efficiency results on the inter-instrument basis are shown here for reference only.

Researchers should be cautious when using a single particle sizer to evaluate EVS efficiency. For our case, the FMPS 2 data in **Table 2** showed that the performance of the South EVS (96.86%) was better than that of the North EVS (90.69%). The average size distributions from upstream and downstream measurements were inspected (**Figure A-3** and **Figure A-4**). It was noted that high concentrations of large particles from upstream of South EVS was detected by FMPS 2 (**Figure A-4**) so that the filtration efficiency of South EVS was boosted. However, the high particle counts could be the transient nature of processes when high concentrations were generated during the specific time period.

Table 2: Test results of EVS efficiency evaluation on 09/24/2010.

System Efficiency (%)	Total Number Concentration		Test 1 (11:41 ~11:46)		Test 2 (11:50 ~ 11:55)	
			FMPS 1	**FMPS 2**	**FMPS 1**	**FMPS 2**
North EVS	Up-stream	SUM	X	16805200	18967500	X
		AVG	X	55831	63015	X
	Down-stream	SUM	26613	X	X	1563877
		AVG	88	X	X	5196
	Intra-instrument		**99.84 (Test 1)**		**91.75 (Test 2)**	
	Inter-instrument		**99.86* (FMPS1)**		**90.69* (FMPS2)**	
South EVS	**Total Number Concentration**		**Test 1 (12:03 ~ 12:08)**		**Test 2 (12:10 ~ 12:15)**	
			FMPS 1	**FMPS 2**	**FMPS 1**	**FMPS 2**
	Up-stream	SUM	101000000	X	X	224565000
		AVG	335548	X	X	746063
	Down-stream	SUM	X	7041940	16998100	X
		AVG	X	23395	56472	X
	Intra-instrument		**93.03 (Test 1)**		**92.43 (Test 2)**	
	Inter-instrument		**83.17* (FMPS1)**		**96.86* (FMPS2)**	

* Inter-instrument data shown here for reference only.

Remarks: (1) The pressure drops of both EVSs were at 0.7 in–H_2O during testing.
(2) North EVS was connected with Furnaces F6 and F7; South EVS connected with Furnaces R&D, DARPA, TYCO, F1, F2, F3, and F4.

Office / Laboratory Area

The total number concentrations in non-production areas monitored by FMPS 1 were 8,513 particles/cm^3 in Laboratory 1 and 8,758 particles/cm^3 in Office 2. The average size distributions in these areas were polydisperse with maxima at 10, 16, 30, and 140 nm (**Figure 12**). Without any significant nanoparticle sources, regular indoor size distributions of ultrafine particles are usually concentrated around 100 nm [Franck *et al.*, 2003; Zhu *et al.*, 2005]. The area monitoring in the furnace room has shown that particle size distribution in the production area was polydisperse with maxima at 10 and 20 nm (**Figure 6**). Therefore, most of the nanoparticles below 100 nm found in the non-production areas could be contaminants that migrated from the production room.

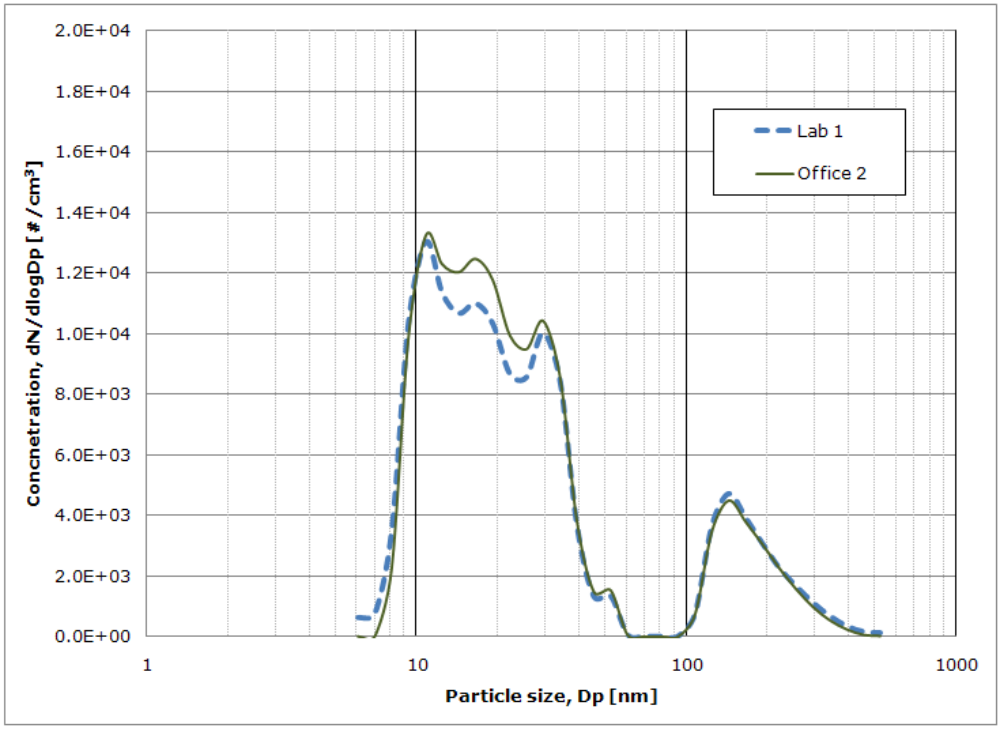


Figure 12: Average size distributions in the areas of Laboratory 1 and Office 2 monitored by FMPS 1 on 09/24/2010.

Examination of Airflow Pattern around Production Furnaces

All the furnaces were operated full opened or with only the sides open (**Figure 2**), except for F6 and F7. **Table 3** summarizes the average face capture velocity in feet per minute (fpm) and open area dimensions for each ventilated enclosure. Duct velocity measurements were determined by averaging the velocity measurements of each orthogonal pitot tube traverse and then converted to flow by multiplying by the cross-sectional area of the duct. Because the exhaust flow rate can be adjusted by a frequency inverter, duct flow rates from the duct exhausting the ventilated enclosure around each furnace were measured at the regular speed (45 Hz) and the maximum speed (60 Hz) and summarized in**Table 4**. Therefore, the flow rates of EVSs were 1,436 cubic feet per minute (cfm) and 1,457 cfm for North EVS and South EVS during normal operation (45 Hz), and 2,010 cfm and 2,229 cfm during full speed operation (60 Hz). (See **Figure 1** for exhaust duct and main duct layout.) Both EVSs have the same capacity.

Table 3: Average face capture velocity measurements at the opening to each ventilated enclosure. A dash indicates that the enclosure door was kept closed during regular operation.

Furnace (dimensions of opening: height x width, inch)	**Average velocity (fpm) with only the front open**	**Average velocity (fpm) with front and side open**
R&D (27 x 41)	19	16
DARPA (33 x 45)	–	27
F1 (33 x 41)	29	27
F3 (33 x 41)	30	28
F4 (39 x 81)	–	22
TYCO (14 x 42)	40	–
F2 (14 x 42)	40	–
F6 (37 x 140)	–	–
F7 (37 x 140)	–	–

Table 4: Measurement results of average duct velocity (fpm) and flow rate (cfm) at two frequency settings.

EVS	Measurement location (see **Figure 1**)		Variable frequency drive at 45 Hz		Variable frequency drive at 60 Hz (maximum)	
			fpm	**cfm**	**fpm**	**cfm**
North EVS	Furnace	F2	622	217	NA	NA
		F4	458	250	NA	NA
		TYCO	710	248	NA	NA
	Main branch 1 (14-in diameter duct, 97 ^{o}F)		556	594	823	880
	Furnace	F1	673	235	NA	NA
		F3	584	204	NA	NA
		DARPA	804	281	NA	NA
		R&D	511	279	NA	NA
	Main branch 2 (18-in diameter duct, 91 ^{o}F)		477	842	763	1349
	Total flow rate		–	**1436**	–	**2229**
South EVS	**F6** (14-in diameter duct, 99 ^{o}F)		542	579	762	814
	F7 (14-in diameter duct, 83 ^{o}F)		821	878	1118	1196
	Total flow rate		–	**1457**	–	**2010**

Conclusions and Recommendations

Ventilated Enclosures and Air Handling Units

Ventilation measurements (exhaust air volume and capture velocities) were taken at each of the furnace enclosures. The volume of air exhausted from each enclosure was generally too low as indicated by low capture velocities and thermal issues. Average capture velocities were lower than 50 fpm at the opening of each ventilated enclosure around each furnace. OSHA specifies that adequate hood face velocity should be 60−100 linear feet per minute [CFR 1910.1450 App A]. The ACGIH Industrial Ventilation Manual (Table 6-2) recommends 75−100 fpm for processes with little motion [ACGIH, 2010]. Given the activity and thermal drafts in the production areas, higher air velocities at all hood openings are warranted to provide good containment.

It is also important to consider the hot processes in the ventilated enclosure when determining the optimal exhaust flow rate and design configuration. Each furnace was operated with the enclosures open to control the excess heat produced by the furnace. This practice severely compromised the ability of the EVSs to create a negative pressure sufficient to ensure adequate containment. Background air drafts in workplaces routinely range up to 50 fpm requiring exhaust airflows above this level for reasonable containment effectiveness. In addition, the buoyant updraft from the hot air produced from the furnace can easily exceed the exhaust flow rate. This can cause air to leak out of the top cracks or out of the top portion of a front or side opening. Exhaust air flow rates should be increased to address the buoyancy effects from the hot air and to maintain good capture velocities at the face of the enclosure. Additionally, the top of each enclosure was parallel with the top of each furnace with only a couple inches between the two parallel planes. The top of the enclosure could be shaped more like a canopy receiving hood leading to the duct take off to help utilize the buoyant effect and direct air into the duct. The ACGIH Industrial Ventilation Manual provides additional design considerations for hot processes in enclosing hoods [ACGIH, 2010].

The test results based on FMPS measurement have shown that the filtration efficiency of the North EVS was better than that of the South EVS. The performance of the EVSs can be enhanced by optimizing furnace capacity and filter replacement policy. The filter pressure drop should be monitored regularly and included in the preventative maintenance plan to ensure proper performance. The sizing of the exhaust fans and filters will need to be reconsidered if changes to the overall enclosure exhaust flow rates are

increased. Exposure assessments should be conducted after any process or production change to ensure adequate containment and control.

Manufacturing Facility

The direct-reading instrument data showed that nanoparticles are released from the ventilated enclosures during normal operations, maintenance, and product harvesting. Low enclosure capture velocities and the practice of keeping enclosure doors open are the primary contributors to process-based nanoparticle emissions. Though most of the fugitive nanoparticles concentrated at 10 nm were not engineered nanomaterials (based on the TEM results provided by the UMass Lowell), they result in high background concentrations in the production area and may pose risk to workers' health. Therefore, a negative air pressure differential should be maintained in the production area with respect to adjacent rooms/areas. This will help reduce the potential escape of CNTs and exposure to office and other workers. To maintain a slight negative pressure, the room supply air volume should be slightly less than the exhaust air. A general guide is to set a 5% flow difference between supply and exhaust flow rates but no less than 50 cfm [ACGIH, 2010].

Recommendations

The following recommendations are provided to prevent worker exposure to CNTs and to provide a safer and healthier work environment:

1) Better containment design to accommodate heat and access for workers.

- Furnace enclosures should be kept closed as much as possible to provide improved containment. When doors are opened, the exhaust system cannot provide sufficient airflow to maintain a negative enclosure pressure and adequate capture velocity.
- Furnace enclosures and exhaust airflow rates should be designed to handle excess heat so that the enclosure integrity is not compromised during routine operation. The opening of enclosure doors should only be allowed during preparation for operation, product harvesting, and furnace maintenance.
- As new facilities are being designed, evaluate the design of the enclosure using guidance from consensus organizations such as ACGIH, American National Standards Institute (ANSI), American

Society of Heating, Refrigerating, and Air-Conditioning Engineers (ASHRAE), and American Industrial Hygiene Association (AIHA). Guidance on the design of exhaust hoods for hot processes is covered in the ACGIH Ventilation Manual [ACGIH, 2010].

2) Exhaust flow rates should be increased to maintain adequate capture at all hood openings. This may require a redesign of the EVSs to ensure adequate capture velocities at each furnace operation.

- Exhaust airflow rates should be increased to handle excess heat from the furnaces and provide a target capture velocity of 100 fpm at each furnace opening.

3) Use makeup air to reduce drafts, maintain exhaust system performance, and develop and implement a pressurization scheme.

- Replacement air should be provided to the CNT production area at a flow rate slightly lower than the overall flow rates of both EVSs. The lack of replacement air may cause uncontrolled drafts, reduction in exhaust system performance, and problems with the opening/closing of doors in the facility.

- Ensure that ventilation systems maintain the CNT production area under negative pressure relative to the rest of the plant. This will prevent contaminants in the air of the production area from spreading to the rest of the plant. Ensure that air from this room is not recirculated to other areas of the facility and is exhausted directly to the outdoors in accordance with any environmental regulations.

- Production areas should not share ventilation systems with office areas.

4) Ensure that exhaust air discharge stacks are located away from air intakes, doors and windows. According to ASHRAE, outdoor air intakes shall be located at least 25 feet from potential sources of air contaminants such as exhaust outlets of ventilating systems [ASHRAE, 2011]. A properly-designed exhaust stack can help prevent re-entry of contaminated air into the building.

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Acknowledgements

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Appendixes

Table A-1: Summary of enclosure efficiency check.

	Date	9/23/2010		9/242010	
Furnace	Instrument	FMPS 1	FMPS 2	FMPS 1	FMPS 2
DARPA*	Breathing zone	9.38E4±1.40E4	9.86E4±6.62E3	8.87E4±5.38E4	8.36E4±7.00E3
	Sampling point 1	8.11E4±5.90E3	8.47E4±8.82E3	8.13E4±2.31E3	7.83E4±3.63E3
	Sampling point 2	7.02E4±3.00E3	6.73E4±3.41E3	7.38E4±3.02E3	8.75E4±7.81E3
	Background	8.70E4±5.24E3	9.37E4±5.56E3	8.90E4±2.30E3	9.34E4±2.04E3
F1*	Breathing zone	8.00E4±3.64E3	8.36E4±2.75E3	7.79E4±6.36E3	7.92E4±4.80E3
	Sampling point 1	7.89E4±3.50E3	7.71E4±4.37E3	1.21E5±1.04E4	1.18E5±9.38E3
	Sampling point 2	8.11E4±1.03E5	7.81E4±1.08E4	9.09E4±7.49E3	8.96E4±6.12E3
	Background	1.52E5±2.88E4	1.49E4±2.38E4	7.82E4±5.98E3	8.09E4±6.70E3
F6	Breathing zone	3.30E4±1.76E3	2.90E4±1.85E3	--	--
	Sampling point 1	4.23E4±4.02E3	3.78E4±3.65E3	--	--
	Sampling point 2	5.79E4±3.50E3	5.20E4±3.23E3	--	--
	Background	4.16E4±1.05E3	3.80E4±5.67E3	--	--
F4	Breathing zone	5.35E4±1.21E4	4.93E4±1.15E4	--	--
	Sampling point 1	3.33E4±3.60E3	3.10E4±3.37E3	--	--
	Sampling point 2	4.76E4±2.73E3	4.37E4±2.67E3	--	--
	Background	5.54E4±7.40E3	4.97E4±7.79E3	--	--
F3	Breathing zone	--	--	8.18E4±9.60E3	1.08E5±1.74E4
	Sampling point 1	--	--	1.06E5±7.87E3	1.10E5±2.09E4
	Sampling point 2	--	--	9.98E4±4.63E3	1.22E5±1.30E4
	Background	--	--	9.72E4±6.14E3	1.28E5±1.64E4

*Enclosures of Furnaces DARPA and F1 were tested twice on 9/23/2010, but no test was conducted for these two furnaces on 9/24/2010.

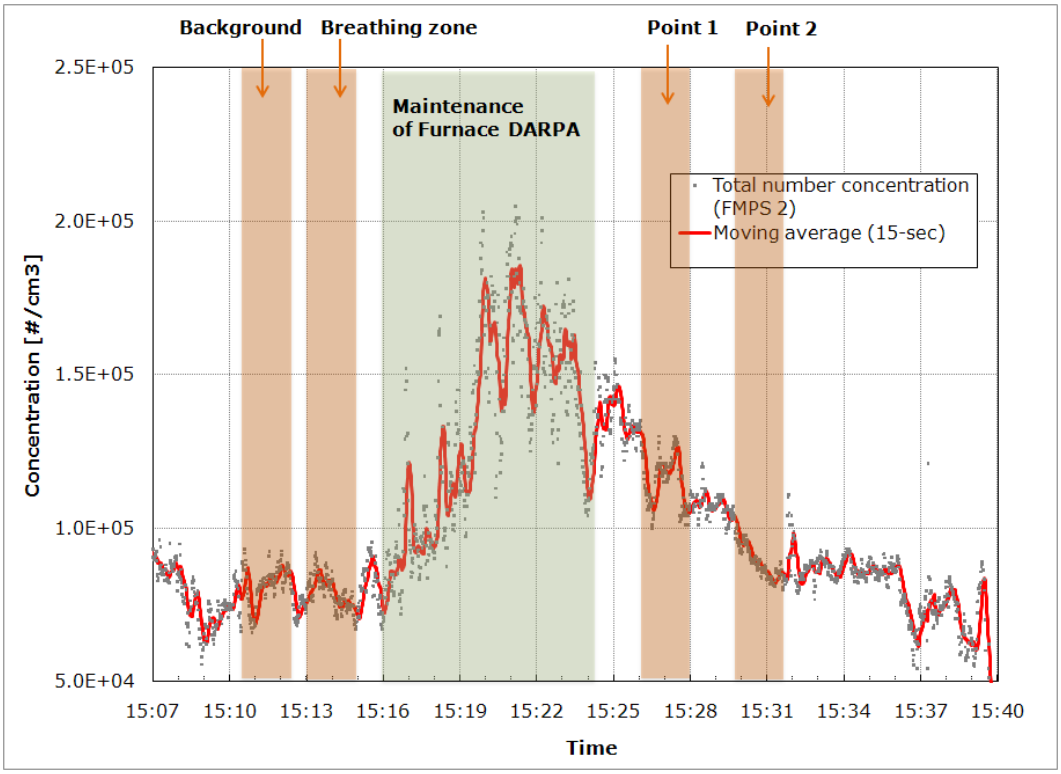

Figure A-1: Enclosure efficiency check by FMPS 2 for Furnace 1 on 9/23/2010. The sampling procedures were marked by colored columns to highlight sampling durations and locations. The sampling was halted by moving the sampling ports into the DARPA Furnace to check particle emissions from its 6-minute maintenance. The enclosure test was resumed by finishing two measurements inside Furnace 1.

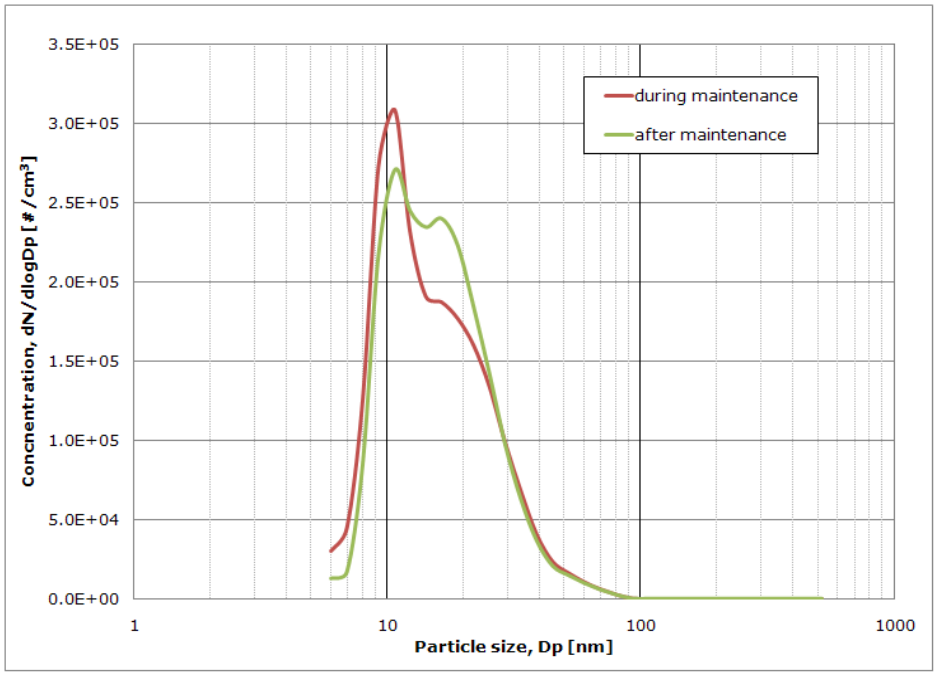

Figure A-2: Average size distribution of nanoparticles from the maintenance task performed at the DARPA Furnace monitored by FMPS 2 during process monitoring for Furnace F1 on 09/23/2010. The size distributions after maintenance were calculated by the data between 15:24 (stopping maintenance monitoring of the DARPA Furnace) and 15:26 (before sampling at point 1 inside the F1 enclosure) as referred to in **Figure A-1**.

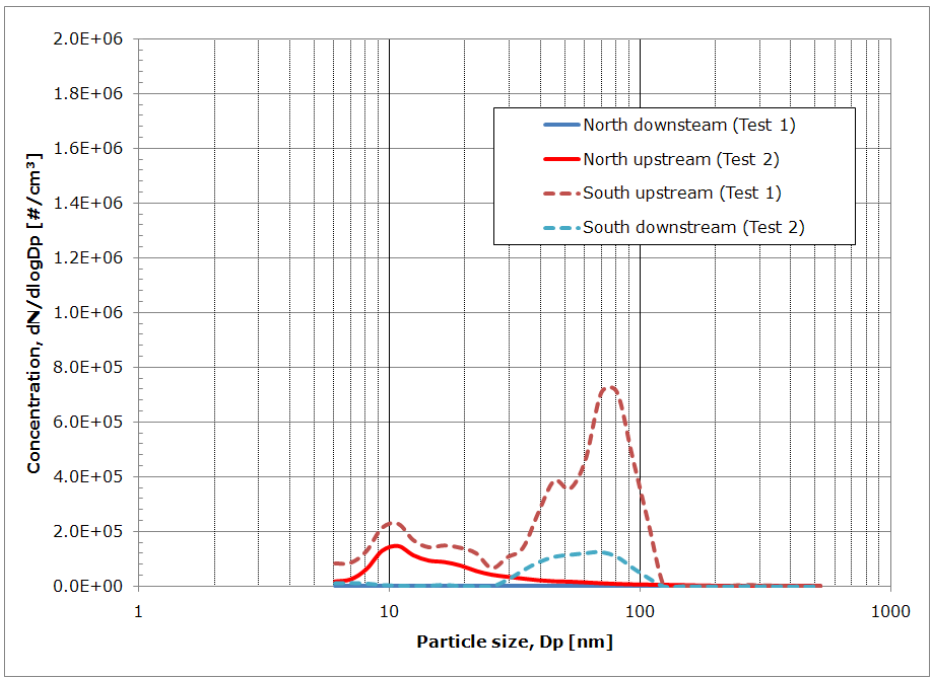

Figure A-3: Average size distributions from EVS upstream and downstream measurements by FMPS 1 on 09/24/2010.

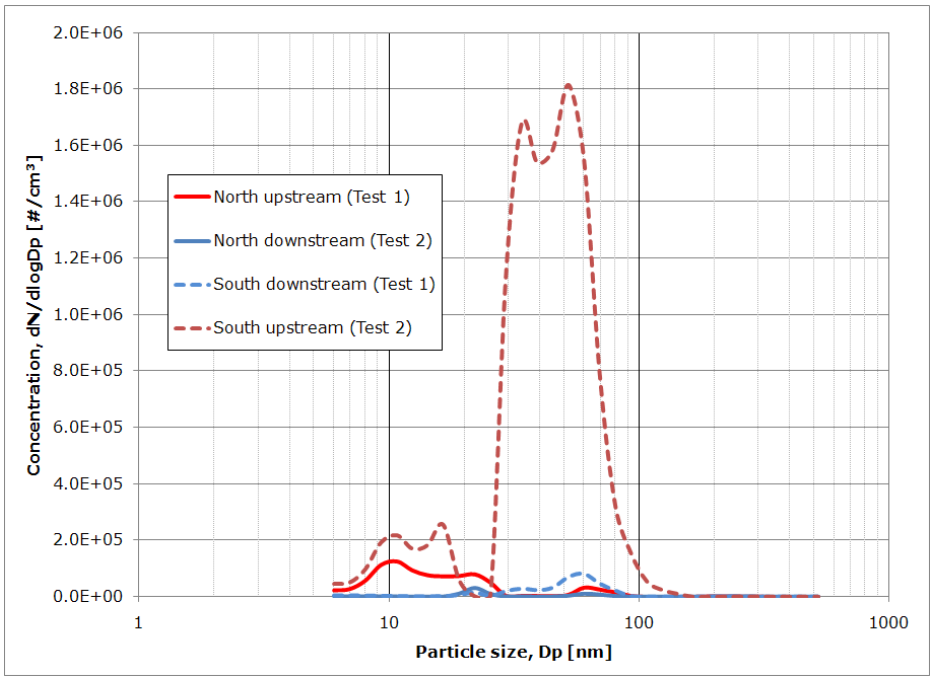

Figure A-4: Average size distributions from EVS upstream and downstream measurement by FMPS 2 on 09/24/2010.

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