In-Depth Survey Report

Evaluation of Engineering Controls in a Manufacturing Facility Producing Carbon Nanotube-Based Products

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Division of Applied Research and Technology
Engineering and Physical Hazards Branch
EPHB Report No. 356-13a

October 2012

DEPARTMENT OF HEALTH AND HUMAN SERVICES
Centers for Disease Control and Prevention
National Institute for Occupational Safety and Health

Site surveyed: nanomaterial manufacturing facility, Eastern USA

Survey dates: April 27–28, 2011

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Abstract

This report summarizes the study results of an evaluation of engineering controls used by a secondary manufacturer (user) of carbon nanotubes (CNTs) to synthesize composite materials. Direct-reading instruments (including Fast Mobility Particle Sizer, or FMPS, Aerodynamic Particle Sizer, or APS, and DustTrak Aerosol Monitor) with continuous real-time measurements were used to monitor manufacturing processes. An assessment of existing exposure controls was conducted using hood capture and exhaust airflow measurements and smoke visualization techniques. The primary processes conducted at the plant were (1) weighing out CNTs and placing them in a slurry, (2) mixing the CNT slurry with other materials in large vats, (3) depositing the slurry materials on a substrate, and (4) cutting the final substrate to meet final product specifications.

The task of weighing out the CNTs was performed in a Class II Biological Safety Cabinet (BSC), which was exhausted to the outside. The BSC was connected to a facility exhaust fan that ran continuously and had an integral supplemental fan that was used at the discretion of the worker. The face velocity of the BSC averaged 65 feet per minute (fpm) when the supplemental BSC fan was switched on and the external facility blower was running. When the supplemental blower was not switched on, the face velocity dropped to less than 10 feet per minute (fpm) across all external facility blower settings (low, medium, high). The results of this testing indicate that the supplemental BSC fan should always be turned on before working with any potentially hazardous materials inside the BSC.

Particle emissions were also found during the mixing process. This process involved weighing out raw materials and mixing them in large drums into a solution. Raw material weigh-out and preparation should be performed in ventilated enclosures (such as a chemical fume hood or powder transfer station) to prevent particle emissions during transfer and weighing, even though these bulk powders are not nanomaterials. Two control measures were used in the cutting process: (1) a downdraft table for cutting CNT-deposited substrates with a rotary cutter, and (2) a canopy hood for controlling exposures during the cutting substrate rolls with a powered saw. The performance of the downdraft table was difficult to quantify because emissions from hand rotary cutting release were minimal. The capture of emissions during rotary cutting tasks could be improved by changing the existing downdraft table to a backdraft slotted hood design commonly used for welding operations [ACGIH 2010]. This design would allow for a solid

work surface for cutting, while pulling the emissions away from the worker and into the exhaust system.

The canopy hood did not effectively collect particle emissions nor prevent worker exposure to airborne particles released from the powered saw cutting task. The monitoring data showed that operating the canopy hood (which was normally turned off) resulted in 15%–20% higher nanoparticle concentrations in the worker's breathing zone. The primary reason for this result is due to the positioning of the worker between the source of emission and the exhaust. This configuration is not recommended because it can cause saw emissions to be pulled through the worker's breathing zone. For this powered cutting process, the optimum control approach is to contain emissions at the source. The use of a ventilated shroud on the saw or the use of a ventilated enclosure around the process could effectively collect saw emissions and reduce the potential for worker exposure during this task.

Although there is currently no regulatory occupational exposure limit (OEL) for CNTs, the use of engineering controls is recommended to reduce the potential risks associated when working with these materials. This report presents the findings of our control assessment and provides recommendations on approaches to contain process emissions and reduce the potential for worker exposure.

Introduction

This study is part of the overall program to evaluate the effectiveness of control measures in companies that produce or use nanomaterials and is supported through the NIOSH Nanotechnology Research Center (NTRC). Workplace controls have been recommended to prevent or minimize exposure to engineered nanomaterials because potential risks associated with nanoparticle exposure have been reported based on toxicological research [Buzea et al. 2007; International Organization for Standardization 2008; Kaluza et al. 2009; Safe Work Australia 2009a]. Engineering controls, such as enclosures, fume hoods, glove boxes/bags, cleanrooms, laminar flow clean benches, and local exhaust ventilation, have been adopted in many nanomanufacturing workplaces [ICON 2006]. However, only limited data on the effectiveness of these engineering controls have been published to date.

The primary objective of this project is to conduct field evaluations to gain practical information on control approaches and to provide recommendations on measures for protecting workers from occupational exposure to nanoparticles. The study results should lead to developing better recommendations for the design and implementation of engineering controls in nanotechnology workplaces. This site survey was conducted by NIOSH, collaborating with researchers from the University of Massachusetts Lowell (UMass Lowell). This study focused on evaluating the performance of control measures used at the study site. Real-time measuring instruments were used to monitor nanoparticle emissions from tasks and processes, and to assess the control efficiency where control measures were used. Assessing control effectiveness is essential for verifying that the exposure goals of the facility have been successfully met.

Background

The company manufactures products containing carbon nanotubes (CNTs). Common processes include the weigh-out of CNTs, mixing of CNTs and other raw materials into solution, the deposition of these materials onto a substrate, and the cutting of the substrate materials and production of the final product assembly. During these processes, potential for worker exposure exists through the handling of CNTs, the mixing of the slurry, and the cutting of the final substrate material. Workers may be exposed to CNTs primarily through inhalation, dermal contact, and ingestion during handling of the nanomaterials.

From animal in vivo exposure studies and cell-culture-based in vitro experiments, CNTs have been shown to contribute to fibrotic lung response, inflammation, and granulomas, and they can induce oxidative stress and cellular toxicity. Some key factors determining the toxicological effects are CNT types [Jia et al. 2005; Murr et al. 2005; Tian et al. 2006; Inoue et al. 2008], purification [Carrero-Sánchez et al. 2006; Wick et al. 2007], surface area and surface chemistry [Tian et al. 2006], and structure [Poland et al. 2008]. Good summary reports of risk assessment studies for CNTs are available [Kobayashi et al. 2009; Safe Work Australia 2009a].

In 2008, the U.S. Environmental Protection Agency (EPA) formally issued a notice to manufacturers to show its intention to consider CNTs as new chemicals and therefore potentially subject to regulation under Toxic Substances Control Act (TSCA). NIOSH also released interim guidance about specific medical screening for workers exposed to engineered nanoparticles, including single walled carbon nanotubes [NIOSH 2009].

Occupational exposure limits (OELs) are useful in reducing work-related health risks by providing a quantitative guideline and basis to assess the performance of engineering controls and other risk management approaches. Currently, no regulatory standards for nanomaterials have been established in the United States. However, NIOSH has developed a draft recommended exposure limit (REL) of 7 micrograms of carbon nanotubes or carbon nanofibers per cubic meter of air as an 8-hour, time-weighted average, respirable mass concentration [NIOSH 2010]. This draft standard for carbon nanotubes is currently undergoing the public review process and may be revised as a result of this process. Other countries have established OELs for various nanomaterials. For example, the British Standards Institute [BSI 2007] recommends working exposure limits for nanomaterials based on various classifications such as solubility, shape, and potential health concerns as related to larger particles of the same substance. Germany's Institut für Arbeitsschutz der Deutschen Gesetzlichen Unfallversicherung, an institute for worker safety, has published similar guidelines [IFA 2009].

In the absence of governmental or consensus guidance on exposure limits, some manufacturers have developed suggested OELs for their products. For example, Bayer has established an OEL of 0.05 mg/m^3 for Baytubes® (multiwalled CNTs) [Bayer Material Science 2010]. For Nanocyl CNTs, the no-effect concentration in air was estimated to be 2.5 $\mu g/m^3$ for an 8 hr/day exposure [Nanocyl 2009].

Manufacturing Facility and Control Measures

Overview of Plant and Process

The facility is a downstream user of CNTs and incorporates them into its final products. A range of manufacturing procedures was performed in separate working areas as shown in **Figure 1**. The manufacturing processes can be described as weighing of CNTs, mixing of CNT solution, depositing the slurry onto substrate materials, the drying and cutting of the substrates, and assembling of the final products (**Figure 2**).

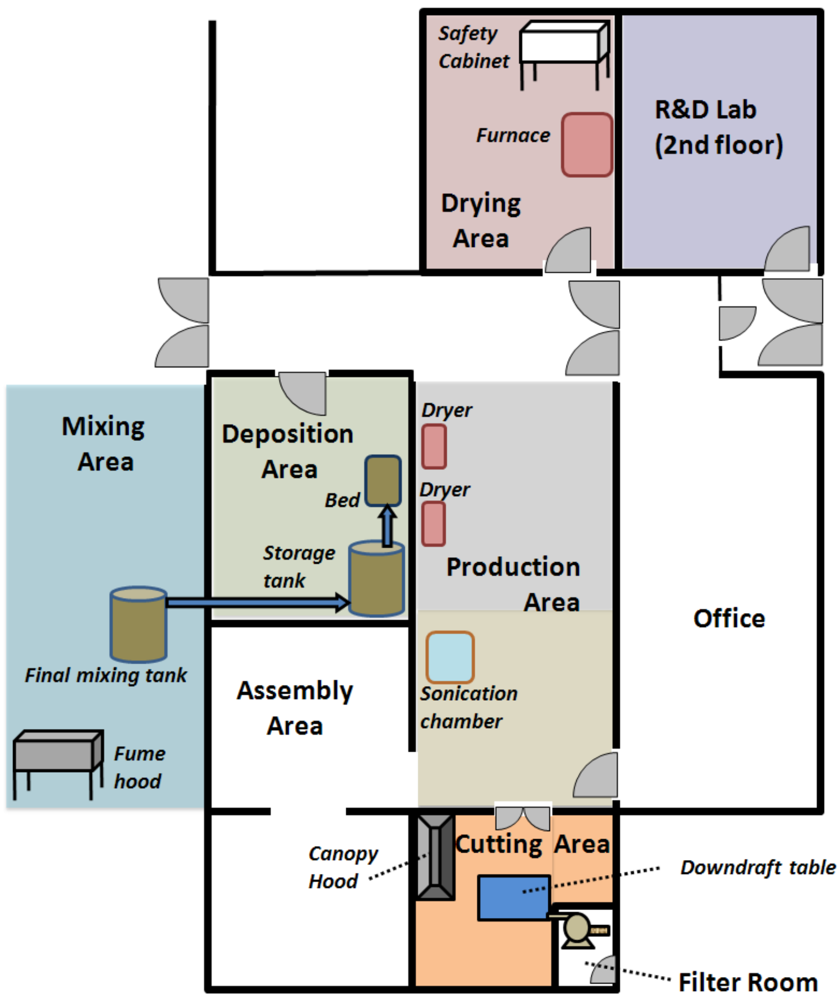


Figure 1: Facility layout.

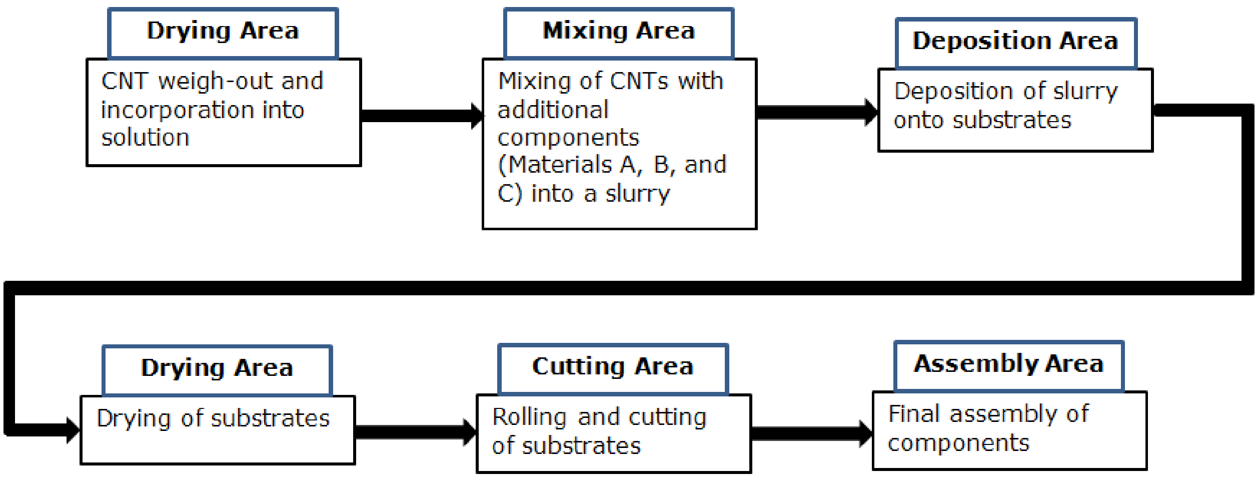
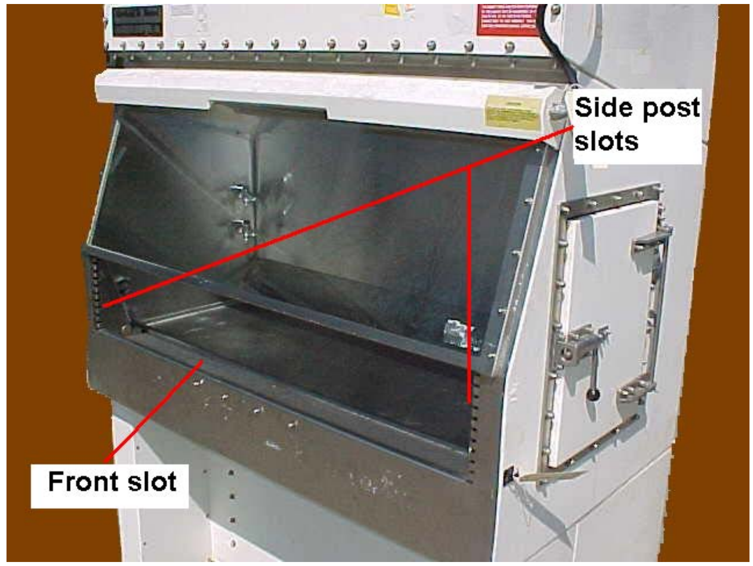


Figure 2. Flowchart of major production processes.

Weighing of CNTs (Drying Area)

The task of weighing CNTs and premixing them into solution was conducted in the drying area inside a Class II Biological Safety Cabinet (BSC). Once the CNTs were mixed into the solution, they were stored in small containers for further processing.

The BSC (Baker Company, Sanford, ME) was 50.5 inches (in.) wide, 23 in. deep, and had a face opening of 8 in. tall (**Figure 3**). The hood included two perforated plate exhaust grilles (front and rear) with side post slots. The front slot was 3.375 in. wide and the rear slot was 4.375 in. wide. A downward HEPA-filtered air shower over the work surface is used to provide a clean work area to minimize contamination of the product. This downward shower of air splits as it approaches the work surface; the front grille draws part of the air to the front grille, while the remainder is directed to the rear grille (**Figure 4**). These BSCs recirculate a portion (up to 70%) of the air after cleaning with a high efficiency particulate air (HEPA) filter. The BSC was equipped with 0.5 horsepower supplemental fan system to drive the exhaust and recirculation airflow.



(a) (b)

Figure 3: (a) Front view and (b) side view of Baker BioGARD Class II type B2 lab environmental hood. A similar lab hood was used at the study site for weighing CNTs. (photos taken from http://www.bid-on-equipment.com/detail~id~102174.htm#).

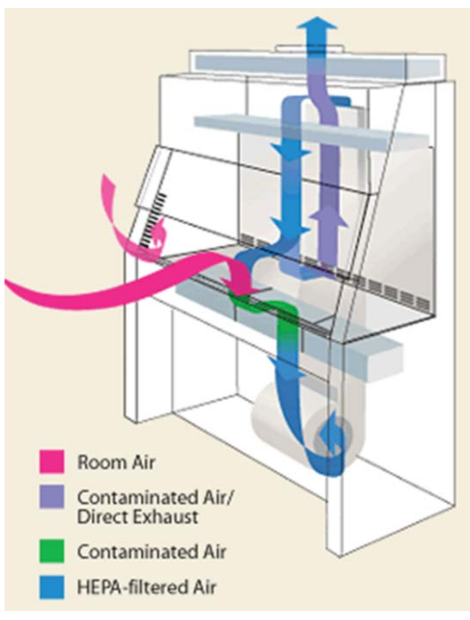


Figure 4. Airflow pattern for a Type II Biological Safety Cabinet. (Figure taken from http://www.bakerco.com/intro-to-biological-safety-cabinets.html)

A switch on the BSC frame allowed the supplemental fan to be turned on or off by the user. This unit was connected to a facility exhaust system that served several other hoods and a furnace exhaust. This ducting ran to a blower mounted on an external wall, and the fan speed/airflow rate was controlled by a variable frequency drive (VFD) located in an adjacent quality assurance/quality control (QA/QC) lab. When weighing small quantities of CNTs, workers would occasionally turn off the supplemental BSC fan and use the facility exhaust only to minimize disturbance to nanomaterials.

Mixing and Deposition of CNT Slurry (Mixing and Deposition Areas)

Following the weigh-out of CNTs into the solution, the containers were transported to the mixing area for further processing. Unlike other working areas, the mixing area was located in an open warehouse space. Three raw materials (hereinafter called materials A, B, and C) were stored in bulk packages in the mixing area prior to use. The general tasks of the mixing procedure are listed in **Table 1**. All the materials were mixed in a series of mixing tanks depending on the final product specifications before transfer to the final mixing tank. Although a fume hood was located in the mixing area, it was only used for storage of the CNT solution, and the exhaust fan was not operated. No other engineering controls were installed in the mixing area.

Table 1. General tasks of the mixing process.

Task ID	Task
M1	Weighing Material A
M2	Mixing Material A with water
M3	Weighing Material B
M4	Weighing Material C
M5	Transferring slurry of Material A to the final mixing tank
M6	Mixing material B into the final mixing tank
M7	Mixing CNTs
M8	Mixing Material C
M9	Final Mixing: Transferring slurry of CNTs and Material C into the final mixing tank

Following the completion of mixing, the slurry was pumped into a holding tank in the deposition area adjacent to the mixing area, and the materials were deposited on a substrate. The deposition area did not have any local exhaust ventilation systems installed. Following this process, the substrate materials were moved to the drying area, where they were placed in a furnace to drive off excess moisture. The furnace exhaust was connected to the facility exhaust system.

Cutting of Substrate Material (Cutting Area)

The dried substrate sheets were then tailored to required dimensions by cutting and rolling on a ventilated (downdraft) table. The cutting room had two independent local exhaust ventilation systems: (1) a downdraft table with exhaust slots used for controlling emissions from a rotary cutting tool, and (2) a canopy hood over a circular saw. A separate fan and high efficiency particulate air (HEPA) filtration system connected to the downdraft table was used to remove contaminants generated from the rotary cutting process.

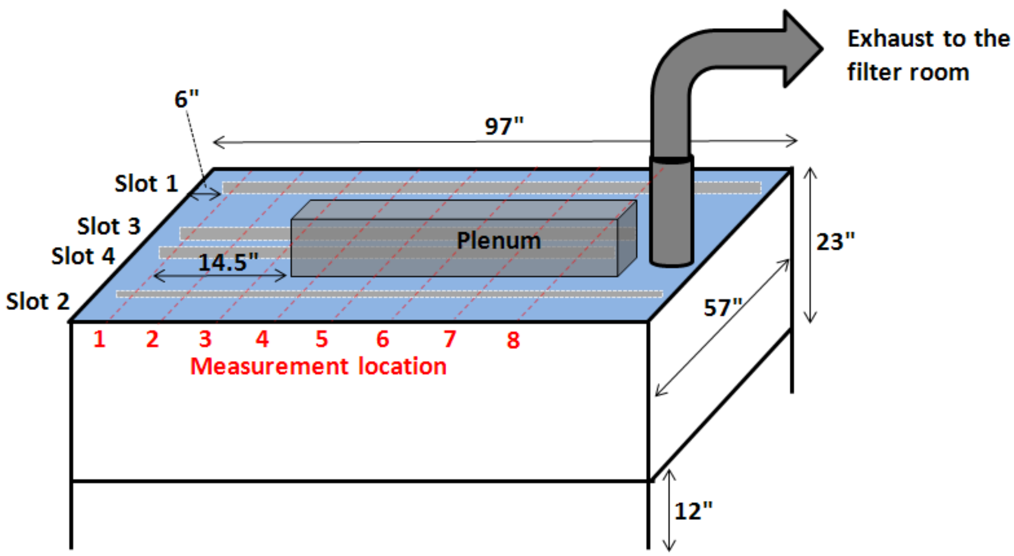


Figure 5: Top view of the downdraft table.

The downdraft table consisted of four slots, with the body of the table serving as an exhaust plenum. The slots were labeled 1–4 based on location **(Figure 5)**. Slots 1 and 2 run the length of the downdraft table along the edge, with slot 1 being 0.25 in. wide and slot 2 being 0.125 in. wide. Slots 3 and 4 are near the center of the table, and both are 0.25 in. wide and 14.5 in. long. The exhaust of the downdraft table was connected to a dust collector equipped with high efficiency particulate air (HEPA) filter cartridges in a small room next to the cutting area (**Figure 1**). The HEPA-filtered air was recirculated back into the facility.

The rolled substrates were cut by a powered circular saw under a canopy hood in the cutting area. The canopy hood recirculated air into the cutting room after filtration (**Figure 6**). This hood was reportedly not used during cutting but was evaluated during this survey. The hood measured 3.5 feet (ft) in width by 10 ft in length and was 7 ft above the floor. The cutting table was 2.5 ft. high, making the distance from the table to the hood approximately 4.5 ft. Air was exhausted from the canopy by a long perforated PVC pipe running along the rear of the hood with a series of 2.5 in diameter holes located approximately 12in. apart (on center).

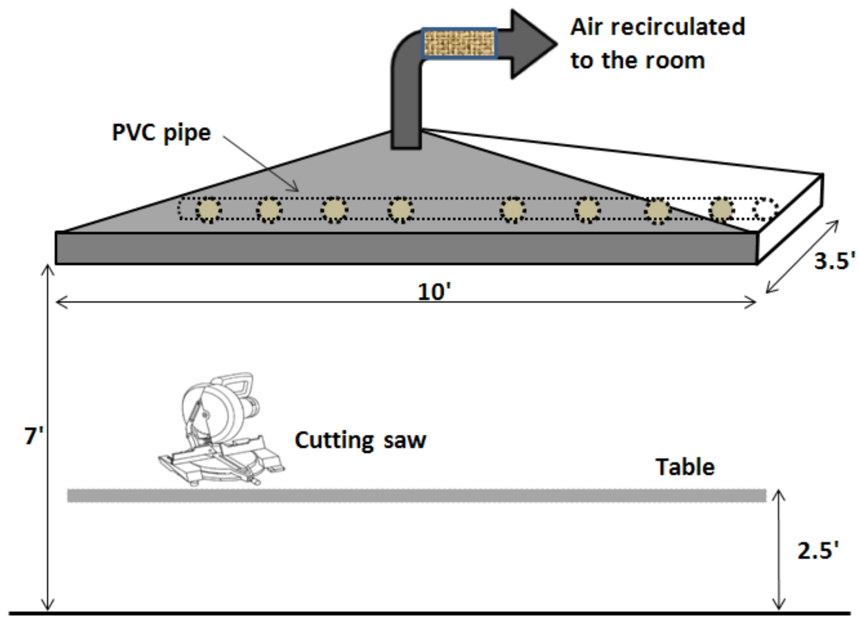


Figure 6: Canopy exhaust hood.

Research and Development (R&D) Activities (Production Area and R&D Lab)

In addition to these processing areas, CNTs were handled in the R&D Lab for research activities, including the weighing and mixing of small quantities of CNTs. In the production area, an insulated box was used to house a sonicator used primarily for R&D activities (**Figure 1**). These activities were monitored to evaluate potential for exposure during the conduct of these tasks.

Methodology

A variety of methods were used to evaluate the existing engineering controls, including measuring exhaust flow rates, face (or capture) velocity, and slot velocity for each hood. In addition to the face and slot velocity measurements, a smoke tracer was used to confirm that the direction of the airflow is correct and to assess the effect of secondary airflows on hood performance. Direct-reading instruments were used to measure background concentrations in the work area and in the worker's breathing zone and to evaluate particle emissions from processes and tasks.

Instrumentation

Direct–reading instruments were used to provide continuous measurements of concentrations to correlate with the specific production equipment and work processes. 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 (Table 2).

Nanoparticles, released from any nanomaterial production processes, tend to quickly agglomerate into larger-sized particle clusters. The APS and FMPS help provide a full spectrum of airborne particle size and number distributions to cover both nano-sized primary particles up to larger agglomerate sizes typically seen in production plants. In this study, researchers used two FMPSs and two APSs to measure the source and the worker's breathing zone. A DustTrak laser photometer was used to measure the particle mass concentration, which is traditionally used as a metric for exposure assessment. Exhaust air velocities and duct flow rates were measured using a Model 8386 VelociCalc Plus hot wire anemometer (TSI Inc., Shoreview, MN) outfitted with an electronic manometer.

Table 2. Direct-reading instruments used in this study

Instrument Name	Metrics	Specifications
FMPS (Model 3091, TSI Inc.)	Number	(1) Determining number size distributions with an array of electrometers. (2) Size range from 5.6 to 560 nanometers (nm)
APS (Model 3022, TSI Inc.)	Number	(1) Measuring number size distributions with light-scattering technique. (2) Size range from 0.5 to 20 micrometers (µm).
DustTrak (Model 8533, TSI, Inc.)	Mass	(1) Single channel basic photometric instrument. (2) Size range from 0.1 to ~15 µm (size segregated mass fractions for PM_1, $PM_{2.5}$, respirable, PM_{10} and total) for concentration range from 0.001 to 150 mg/m^3.
Note: Both research teams utilized FMPS and APS instruments during the survey: FMPS 1 and APS 1 refer to the NIOSH instruments, while FMPS 2 and APS 2 refer to the UMass Lowell instruments.		

Sampling/Control Evaluation Plan

Particle concentrations were monitored by direct-reading instruments during typical worker tasks throughout the manufacturing process and R&D activities. Special attention was paid to the tasks related to dry powder handling and cutting processes that potentially released nanomaterials into the workplace environment. Where local exhaust systems were in place, control effectiveness was evaluated, including the BSC (drying area), the downdraft table (cutting area), and the canopy hood (cutting area).

CNT Weigh-out (Drying Area)

The BSC was used for CNT weigh-out and dispersion of CNTs into solution. The BSC was tested in the as-used condition with equipment/supplies located inside the hood, sometimes blocking areas of the face and exhaust grilles. The BSC exhaust flow was driven by a facility exhaust fan during normal operation. In addition, a supplemental integral fan mounted on the BSC (with a local on/off switch) was used at the discretion of the operator. To fully evaluate the BSC exhaust, face velocities were measured with the supplemental BSC fan turned off and the facility exhaust blower operated at low, medium, and high fan speeds (VFD at 36, 68 and 90 Hz). Exhaust duct velocities were measured at the nominal (medium) and high facility exhaust fan speeds with the supplemental BSC fan turned off. The BSC exhaust duct flow rate was also measured at the reported standard operating conditions—supplemental BSC fan on and facility VFD at 68 Hz. The VFD was set at 68 Hz during the work shift with a night time setback of 36 Hz.

Face velocity measurements were made across the BSC opening using a thermal anemometer (VelociCalc Plus, Model 8386, TSI Inc.). For this study, the open hood face was divided into seven grid points extending across the face of the BSC. Measurements were taken at the center of each grid and perpendicular to the plane of the opening. A Pitot tube was used to measure velocity pressure in the hood exhaust duct. Two 10-point orthogonal traverses were performed in the BSC exhaust duct to determine average duct air velocity [ACGIH 2007]. The volumetric airflow rate through each duct was determined by multiplying the average velocity and the cross-sectional area of the duct.

During CNT weighing and solution preparation, direct reading particulate measurements were taken inside and outside the hood and in the worker's breathing zone. These measurements were made with the BSC supplemental fan on and off to assess the effect of operating with only the facility exhaust on, and with the facility exhaust on plus the BSC supplemental fan on.

Mixing and Deposition of CNT Slurry (Mixing and Deposition Areas)

Direct-reading instruments were also used to identify particle emissions from potential sources and assess worker exposure. For this assessment, workers were requested to perform the manufacturing procedures listed in **Table 1** step by step. During the deposition process, the instruments were used to monitor the particle concentrations above the deposition bed during slurry draining. In addition, background concentrations were monitored prior to the start of mixing processes (using FMPS 1).

Cutting of Substrate Material (Cutting Area)

CNT-deposited substrates were manually cut and rolled on the downdraft table. Slot air velocities of the downdraft table were measured at multiple points using a hot-wire anemometer (**Figure 5**). A qualitative smoke test was conducted for the downdraft table to visualize its capture efficiency. The task of cutting rolled substrates was done by a powered saw under a canopy hood. The hood consisted of a PVC duct located within the canopy with holes drilled across the length of the canopy. The exhaust velocities were measured at each exhaust hole along the canopy hood to estimate the overall exhaust flow rate. The canopy exhaust hood was reportedly not used during normal cutting operations, but an evaluation of its performance was conducted. The canopy hood exhaust fan was turned on and off to evaluate the potential impact of utilizing this exhaust system during cutting of substrate rolls.

Emissions and worker exposure were characterized by measuring particle concentrations near the worker and the source. Particle concentrations were measured during both rotary and powered saw cutting using direct-reading instruments in the following locations: FMPS 1, APS 1, and DustTrak around the worker's breathing zone, and the FMPS 2 near the cutting tool (i.e., emission source).

R&D Activities (Production Area and R & D Lab)

Two R&D activities/tasks were monitored during the survey: (1) the sonication of materials in the production area, and (2) the handling and cutting of small amounts of CNTs in the R&D lab. The FMPS instruments were used to monitor aerosol concentrations inside and outside the sonication chamber simultaneously during nanomaterial mixing. Dust mass concentrations were also monitored (using the DustTrak) during the handling of small amounts of CNTs, the cutting of substrates using a rotary cutter, and the cleaning of work surfaces in the R&D laboratory.

Results

Engineering Control Evaluation

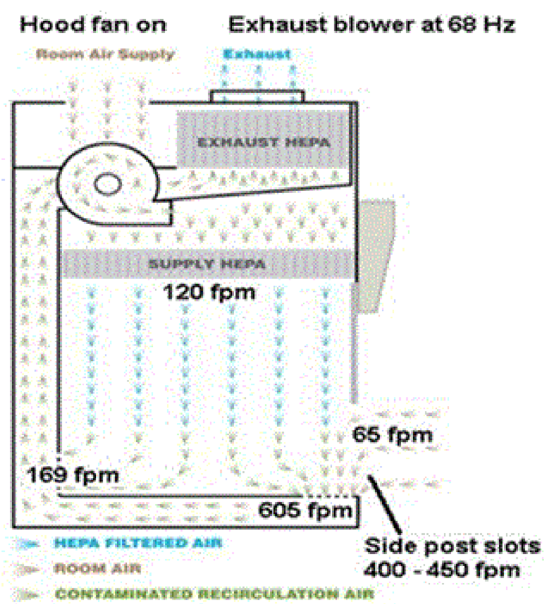
Results of evaluations of engineering controls implemented within the plant are detailed, including the biological safety cabinet (CNT weigh-out), the downdraft table (substrate cutting), and the canopy hood (substrate roll cutting).

(1) CNT Weigh-out (Drying Area)

An evaluation of the task of weighing out CNT materials within the BSC was conducted. This evaluation included measuring particulate concentrations during the weigh-out process and measuring airflow rates under a variety of operational conditions. When the supplemental BSC fan was turned off (**Figure 7-a**), the facility exhaust fan did not provide adequate exhaust flow to contain materials inside the hood at either the medium or high exhaust flow rates (VFD at 68 Hz or 90Hz). Airflow measurements in the exhaust duct indicated exhaust flow rates of 7 cubic feet per minute (cfm) at a VFD setting of 68 Hz and 96 cfm at 90 Hz. Extremely low hood face velocities were also measured when the supplemental BSC fan was turned off, with average velocities of 0 fpm at 68 Hz and 7 fpm at 90 Hz. When the supplemental fan was turned on, the face velocity and exhaust airflow rates increased markedly. An average face velocity of 65 fpm was measured when the supplemental BSC fan was turned on, based on velocity measurements taken across the face of the hood. In addition, the overall exhaust airflow increased to 183 cfm with both the supplemental BSC fan and facility exhaust on—approximately double that with only the facility exhaust on. The BSC downflow air velocity was 120 fpm based on a three-point measurement taken across the supply HEPA filter (see Figure 7-b). Air velocity measurements collected at the front and rear exhaust grilles, side post exhaust slots and hood face are shown in **Figure 7-b** for normal operations—facility exhaust fan VFD at 68 Hz and BSC supplemental fan powered on.

Hood fan	Off		On
Facility exhaust blower (Hz)	68	90	68
Exhaust flow rate (cfm)	7	96	183
Hood face velocity (fpm)	~ 0	7	65

(a)



(b)

Figure 7. BSC hood airflow rates and velocity measurements show (a) summary of hood volumetric airflow data, and (b) airflow velocity at key points within the hood when the facility exhaust was operated at 68 Hz and

the supplemental hood fan was on (figure shown from Class II BSL Cabinet [NuAire Inc. 2011]).

(2) Downdraft Table (Cutting Area)

Slot velocities were measured for each slot at several points along the downdraft table (**Figure 5**). Overall, airflow to the slots was not balanced, with average slot velocities ranging from 500–3,000 fpm. Specifically, slot 1 was 1,250 fpm; slot 2 was 500 fpm; slot 3 was 3,000 fpm, and slot 4 was 2,800 fpm. A qualitative smoke test was conducted to study the airflow profiles on the table surface: slot 1 showed good capture up to 2–3 in. from the slot; slot 2 up to 1 in.; and slots 3 and 4 up to 3–4 in. As expected, slot 2 had the lowest effective capture, because it had the lowest overall slot velocity and was 0.125 in. wide versus the other three slots, which measured 0.25 in. wide. Based on these measurements, the cutting work would need to be carried out very close to the slots (within about 2 in.) for the table control to be effective.

(3) Canopy Exhaust Hood (Cutting Area)

Air was exhausted from the hood by a long PVC pipe running along the rear of the canopy hood with a series of 2.5 in. holes about 12 inches apart (on center). The centerline velocity of each hole from left to right was 560, 630, 750, 975, 1,020, 765, 545, 475 fpm (**Figure 6**). The overall exhaust flow rate was estimated to be 195 cfm. The overall low exhaust flow rate and the distance of the exhaust pipe from the worktable dramatically reduce the canopy hood effectiveness. More importantly, however, is that the design of the hood placed the worker between the source of emissions and the exhaust. This design means that the particulates generated during sawing would likely be carried through the worker’s breathing zone.

Process/Task Exposure and Emission Evaluation

This section summarizes the results of emissions and exposure measurements made with the direct-reading aerosol instruments throughout the plant. Efforts were made to characterize the potential for exposure during primary tasks through the production process. These evaluations help identify the tasks associated with high potential for exposure which would benefit from the implementation of engineering controls.

CNT Weigh-out (Drying Area)

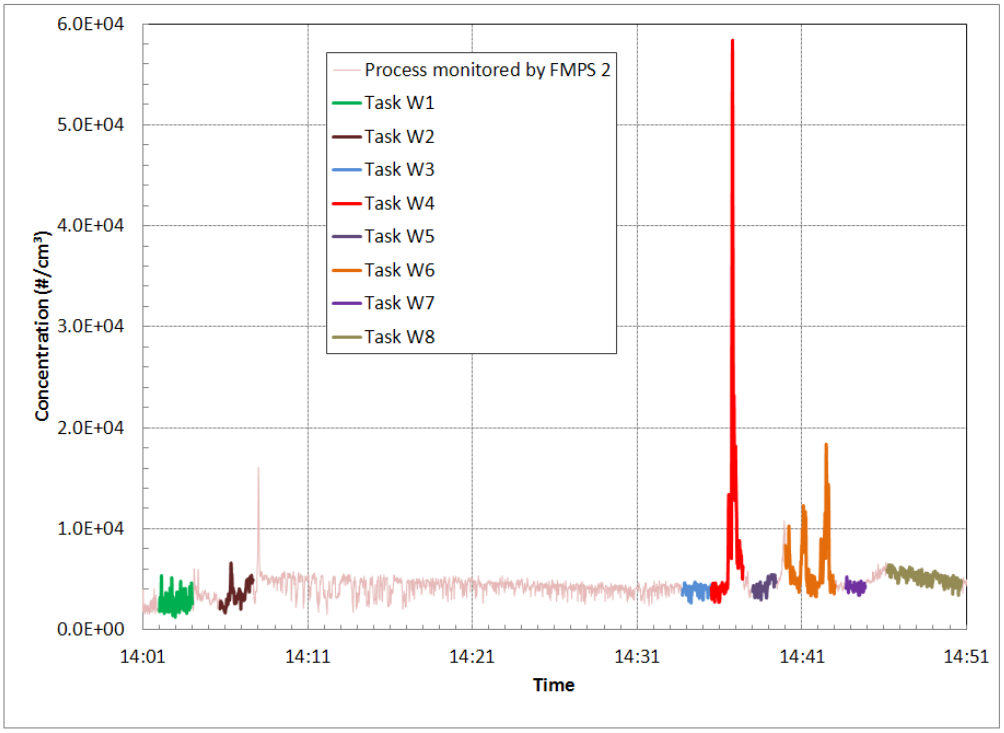
A series of detailed tests were conducted to evaluate the performance of the BSC used for the CNT weighing process (**Table 3** and **Figure 8**).

When the supplemental BSC fan was turned off, the monitoring data from Tasks W2 and W4 showed 2.5 times higher particle concentrations inside the BSC than background. In addition, the average concentration around the worker's breathing zone (W5 and W7) reached 4,291 particles/cm^3 during weighing of CNTs. However, when the supplemental hood fan was turned on, the task average worker breathing zone concentration was reduced to 2,749 particles/cm^3 (36% reduction) during CNT weigh-out. The data from Task W8 also showed that the operation of the supplemental BSC fan reduced background concentrations following the weighing and handling tasks. The use of the supplemental hood fan in addition to the facility exhaust ventilation effectively reduced particle concentrations inside the hood and in the worker's breathing zone during weigh-out activities.

Table 3. Summary of FMPS measurement data during the CNT weighing process.

Task ID			**Average total concentration** (particles/cm^3)	
	Hood fan (Facility exhaust blower at 68Hz)		***OFF***	***ON***
W1	Background check before weighing	Outside hood	**2,800**	**3,209**
W2	Background check before weighing	Inside hood	**3,515**	X
W3	Background check during weighing	Outside Hood	**4,027**	X
W4	Weighing CNTs	Inside hood	**8,564**	**1,207**
W5	Weighing CNTs	Worker's breathing zone	**4,381**	**2,749**
W6	Weighing CNTs	Inside hood	**6,292**	**1,170**
W7	Weighing CNTs	Worker's breathing zone	**4,200**	X
W8	Background check after weighing	Outside hood	**5,234**	**3,059**

(a)



(b)

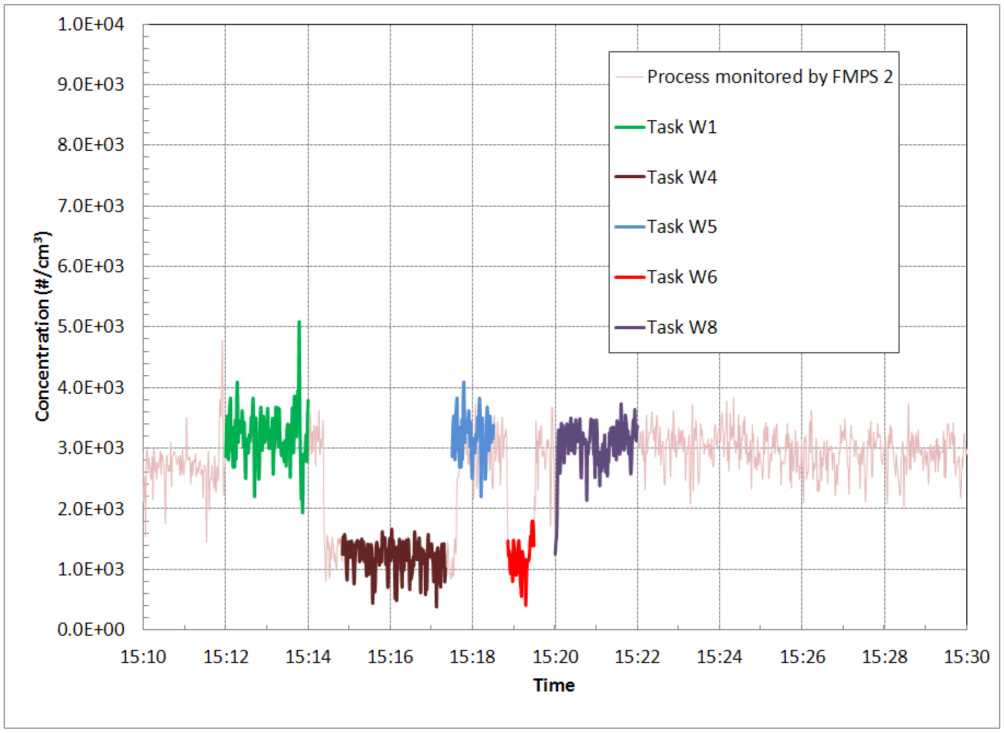


Figure 8. Evaluation of the laboratory hood for the CNT weighing process, when the hood fan was turned **(a)** off, and **(b)** on.

Mixing and Deposition of CNT Slurry (Mixing and Deposition Areas)

(1) Mixing Process

The mixing process was evaluated with the real-time particle monitoring instrumentation using both general area monitoring and worker breathing zone measurement. Area monitoring in the mixing area showed an average total number concentration for particles <560 nm of approximately 4,467 particles/cm^3. The measured size distribution was polydispersed with maxima at 20, 40, and 90 nm. The instruments did not show a significant increase in airborne concentrations during process monitoring except during the weighing of raw material C in powder form. The sampling ports of the FMPS 1, APS 1, and DustTrak were co-located during the monitoring session and showed transient increases in particle concentration at the same time during this process (**Figure 9**).

When handling raw material C, the average concentration of fine particles <560 nm did not significantly increase above background. However, the concentrations of large particles >0.5 µm increased four-fold in particle number and eight-fold in mass **(Table 4)**. Size distribution analysis presented in **Figure 10** showed that released particles were concentrated around 1 µm.

The mixing of the final slurry occurred in an open mixing tank using a power mixer with no lid in place. During this process, there was some fluctuation of particle concentration for fine particles <560 nm, however, concentrations of larger particles (0.5–20 µm) were steady throughout the final mixing process.

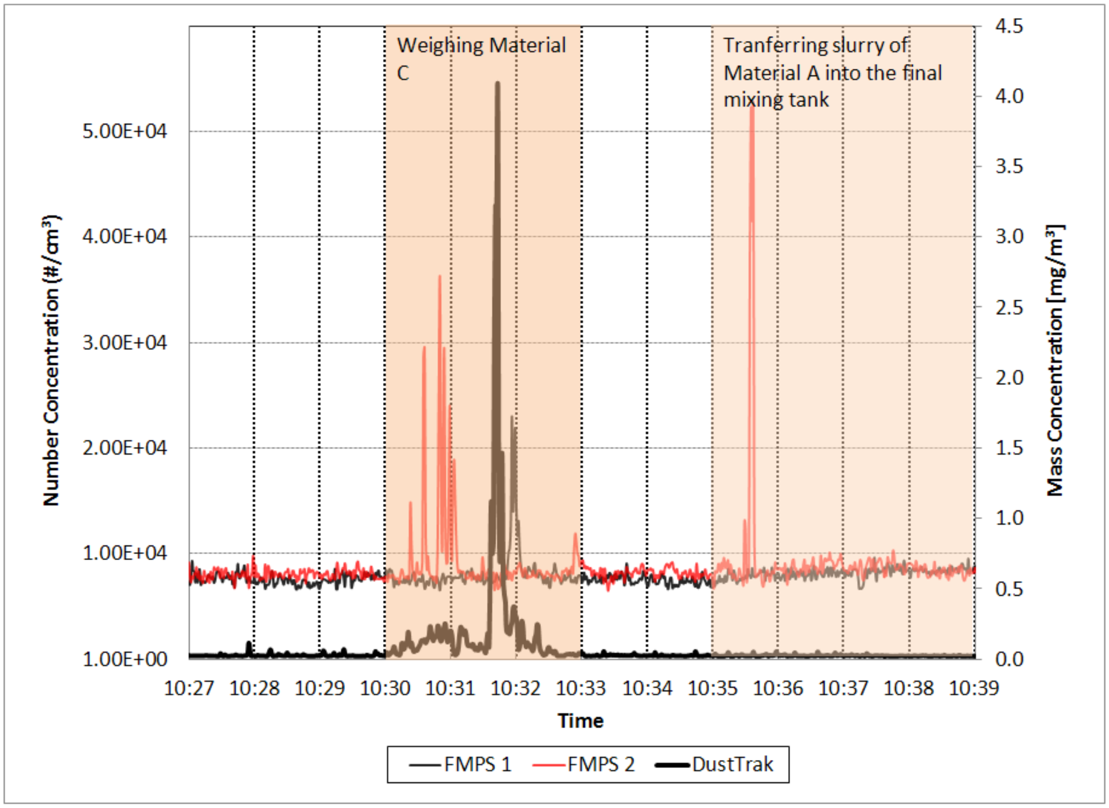


Figure 9: Monitoring the mixing process with FMPSs and DustTrak on 04/27.

Table 4. Summary of average total concentrations of the mixing process monitored by direct-reading instruments on 04/27.

	Average total concentration		
Task	FMPS 1 (particles/cm^3)	APS 1 (particles/cm^3)	DustTrak (mg/m^3)
Weighing Material B (10:27–10:30)	7,579	40.6	0.028
Weighing Material C (10:30–10:33)	8,120	**173.9**	**0.220**
Background (no activity, 10:33–10:35)	7,487	41.4	0.027
Transferring slurry of Material A to the final mixing tank (10:35–10:39)	8,171	38.5	0.026

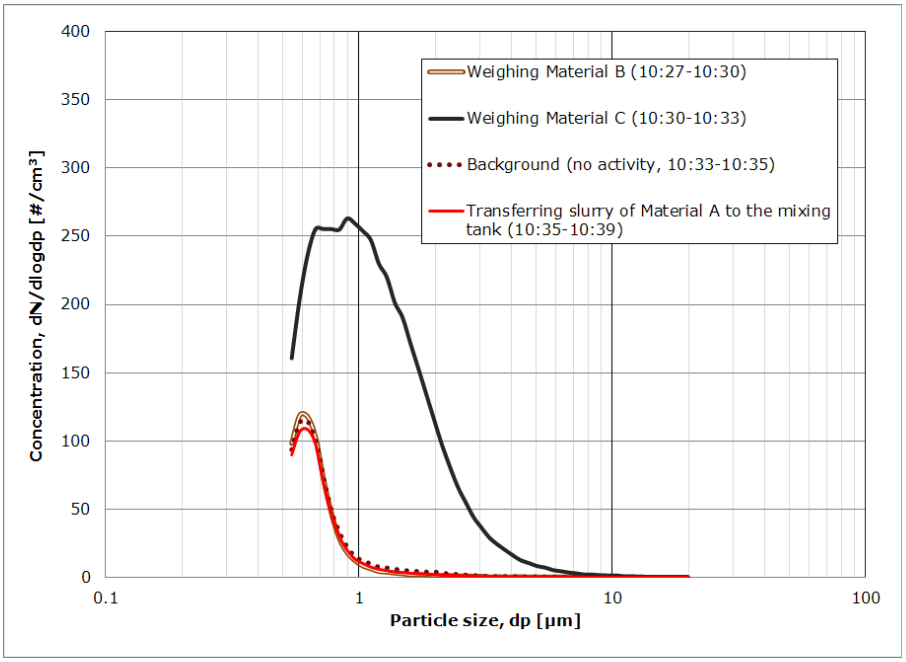


Figure 10: Size distribution analysis for the mixing process from 10:27 to 10:39 a.m. on 04/27.

(2) Deposition Process

The deposition process was monitored and the results are summarized in **Figure 11** and **Table 5**. Background concentrations increased during the monitoring of the deposition area. The increase in particle concentration was not likely due to the pumping of the slurry from the mixing area to the deposition tank, because the slurry was contained within a closed piping system.

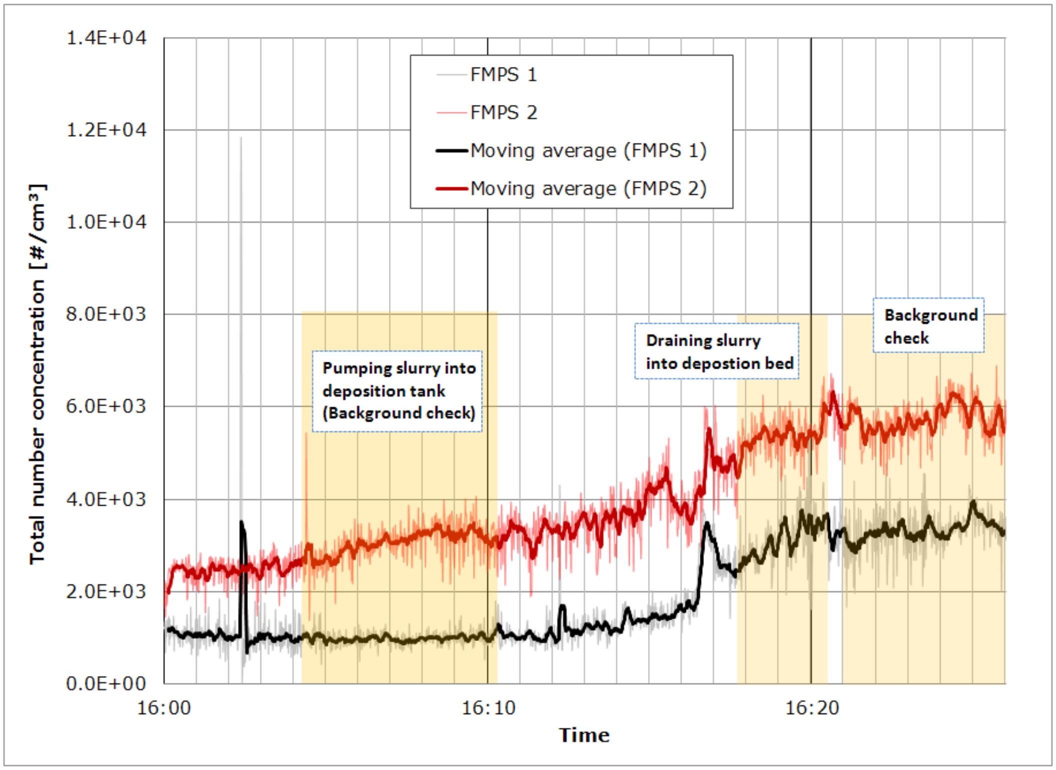


Figure 11: Monitoring data for the deposition process with FMPSs on 04/27.

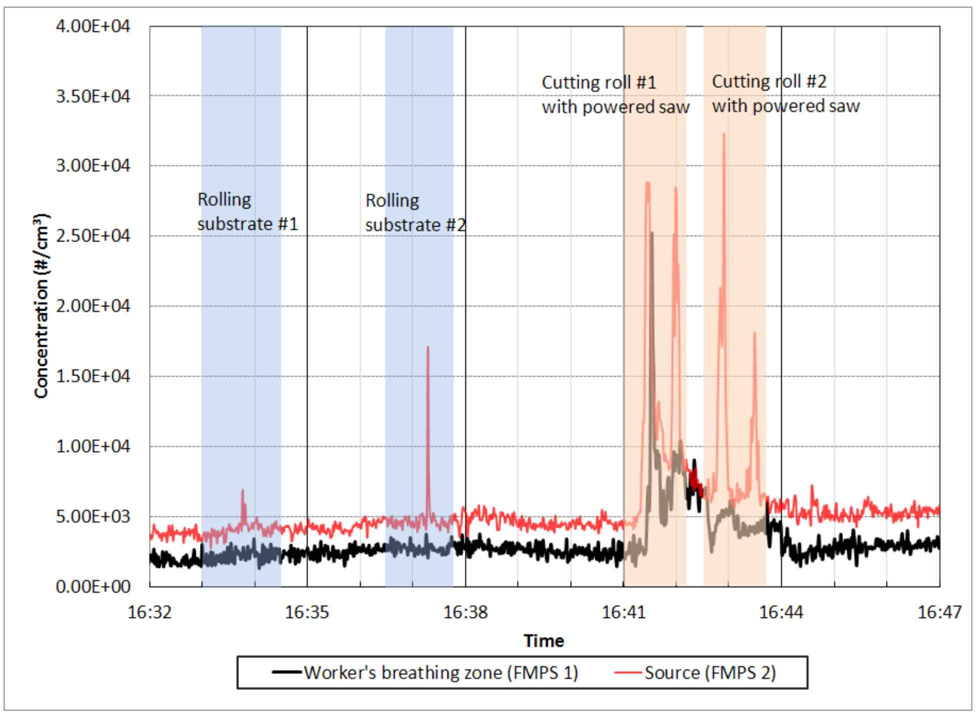
Table 5. Measurement data from the deposition process with FMPSs on 04/27.

Time Period	Measurement	Average total number concentration (particle/cm^3)	
		FMPS 1	FMPS 2
16:04–16:12	Background check	997	3,067
16:17–16:20	Draining slurry into deposition bed (sampling ports on top of the deposition bed)	3,111	5,355
16:21–16:26	Background check	3,329	5,726

Cutting Process

When monitoring processes in the cutting area, FMPS 2/APS 2 were used to monitor emission sources while FMPS 1/APS 1/DustTrak were located near the worker's breathing zone. The tasks of manually rolling CNT-deposited substrates and cutting the substrate rolls with the powered saw were monitored (**Figure 12**). During the evaluation of the manual cutting task using the rotary cutter (on the downdraft table), no particle emissions were detected. During the rolling of substrate sheets, only two transient area peaks were captured (**Figure 12-a**), but no noticeable change was detected in the worker's breathing zone. However, during the cutting of substrate rolls with the powered circular saw, fine particle concentration increased to approximately 30,000 particles/cm^3 (six times higher than background). Particle size analysis showed that the primary modes of airborne particles were 10 and 110 nm, with 10 nm particles detected in the worker's breathing zone (**Figure 13-a**). In addition, larger particles (>500 nm) were also detected in the worker's breathing zone (**Figure 12-b and 13-b**).

(a)



(b)

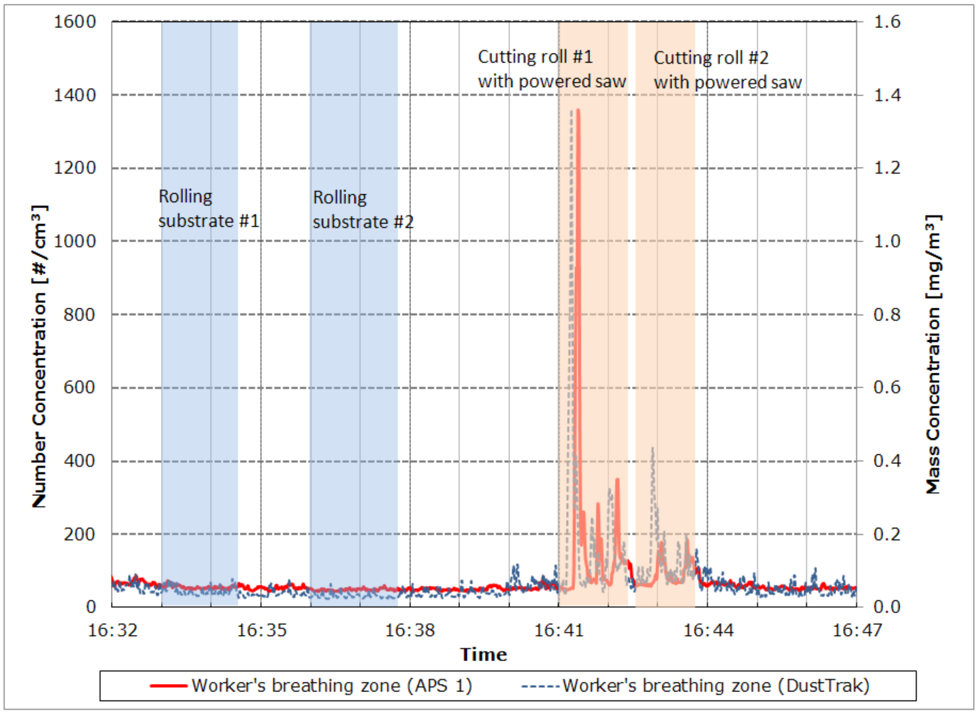
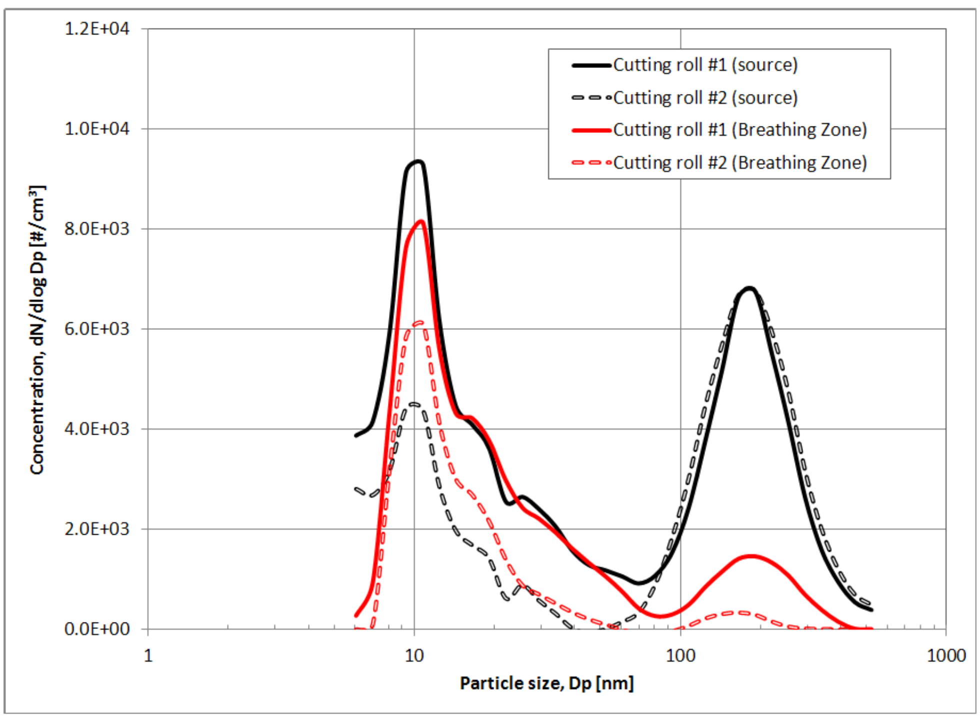


Figure 12. Process monitoring by **(a)** FMPSs, and **(b)** APS 1 and DustTrak, for rolling substrates on the downdraft table and cutting rolls by the powered circular saw with the canopy hood off (normal operation) on 04/27.

(a)



(b)

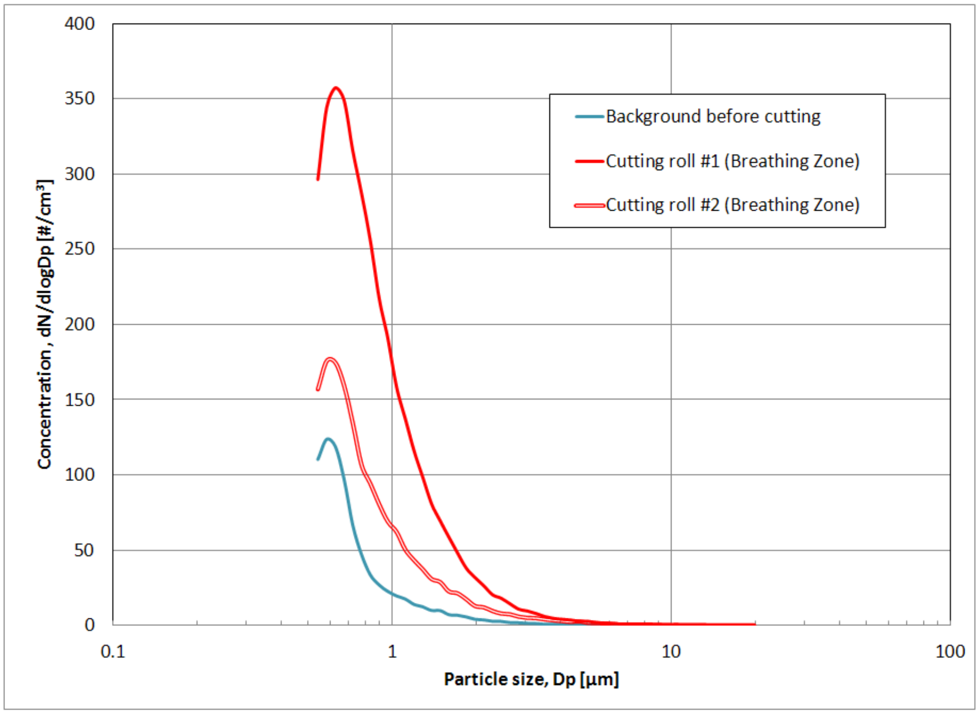


Figure 13. Particle size distribution analysis from **(a)** FMPSs and **(b)** APS 1 during the cutting of substrate rolls using the powered circular saw.

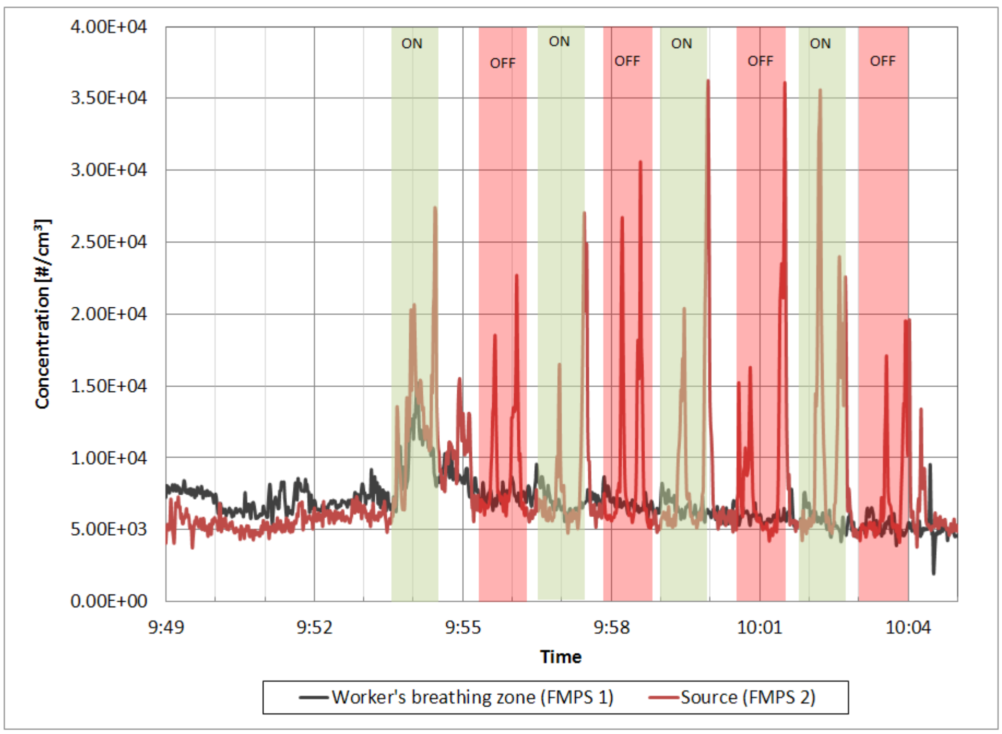
Because particle concentrations were detected in the worker's breathing zone, the powered cutting process was studied further. A canopy exhaust hood was located above the circular saw cutting table. During the initial testing of the cutting of substrate rolls using the circular saw, the exhaust of the canopy hood ventilation system was not on (normal operation). To evaluate the potential effectiveness of the existing canopy hood, the hood exhaust was turned on and off alternatively while a worker cut eight substrate rolls with the circular saw (one minute cut per roll). The monitoring data are presented in **Figure 14** and summarized in **Table 6**.

The canopy hood did not mitigate particle emissions or reduce workers' exposure to airborne particles released from the cutting task (**Table 6**). The FMPS data showed that operating the canopy hood increased particle concentrations approximately 15%–20% at the source and in the worker's breathing zone compared to the hood off condition. The concentration of large size particles >0.5 µm increased by approximately 23% at the source when using the hood. The concentration levels of large particles in the worker's breathing zone, however, were similar under both test conditions.

Table 6. Summary of particle concentrations during powered circular saw cutting of substrate rolls with the canopy hood turned on and off.

Sampling location			Source		Worker's breathing zone		
Canopy hood	Roll No.	Starting time	FMPS2 (particles /cm^3)	APS2 (particles /cm^3)	FMPS 1 (particles /cm^3)	APS1 (particles /cm^3)	DustTrak (mg/m^3)
On	1	9:53:29	12,384	209	10,027	93	0.092
	3	9:56:31	8,675	245	6,873	71	0.053
	5	9:59:01	10,036	310	6,543	70	0.046
	7	10:01:40	10,013	360	5,678	61	0.057
		Average	**10,277**	**281**	**7,288**	**74**	**0.062**
Off	2	9:55:20	9,000	220	7,377	85	0.075
	4	9:57:51	8,890	237	6,818	75	0.065
	6	10:00:29	8,774	277	5,927	82	0.111
	8	10:03:01	7,497	182	5,279	66	0.054
		Average	**8,540**	**229**	**6,350**	**77**	**0.076**

(a)



(b)

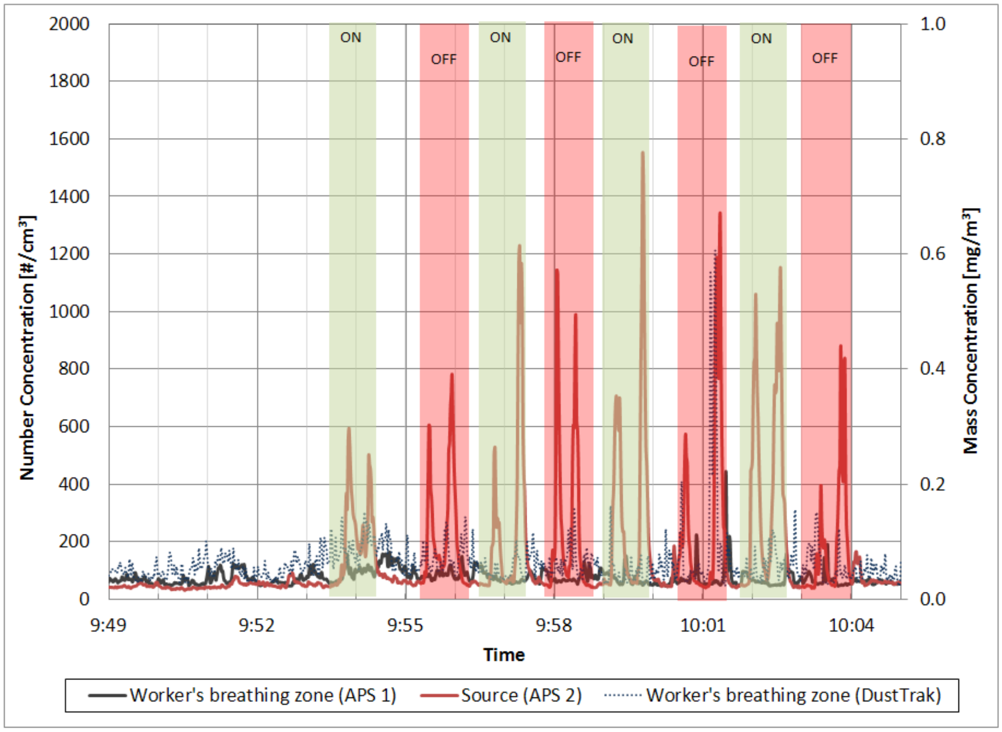


Figure 14. Particle concentration monitoring data of **(a)** FMPSs, and **(b)** APSs and DustTrak during the evaluation of the canopy hood used for the powered saw cutting.

R&D Activities

(1) Handling Nanomaterials in the Laboratory

Small quantities of CNTs were handled in the laboratory for product research and development. There were no engineering controls used in this laboratory area. Activities including manual cutting, weighing, and work surface cleaning were monitored using the DustTrak. As shown in **Figure 15**, the average background concentration in the laboratory was approximately 0.009 mg/m^3. The only task that showed an increase in particle concentration above background was the cutting of substrate material with a manual rotary cutter. A transient peak concentration of greater than 3.5 mg/m^3 was measured at the source, but the average concentration for the whole process was 0.1 mg/m^3. The average mass concentration measured during cleaning was 0.03 mg/m^3 and during weighing was 0.02 mg/m^3.

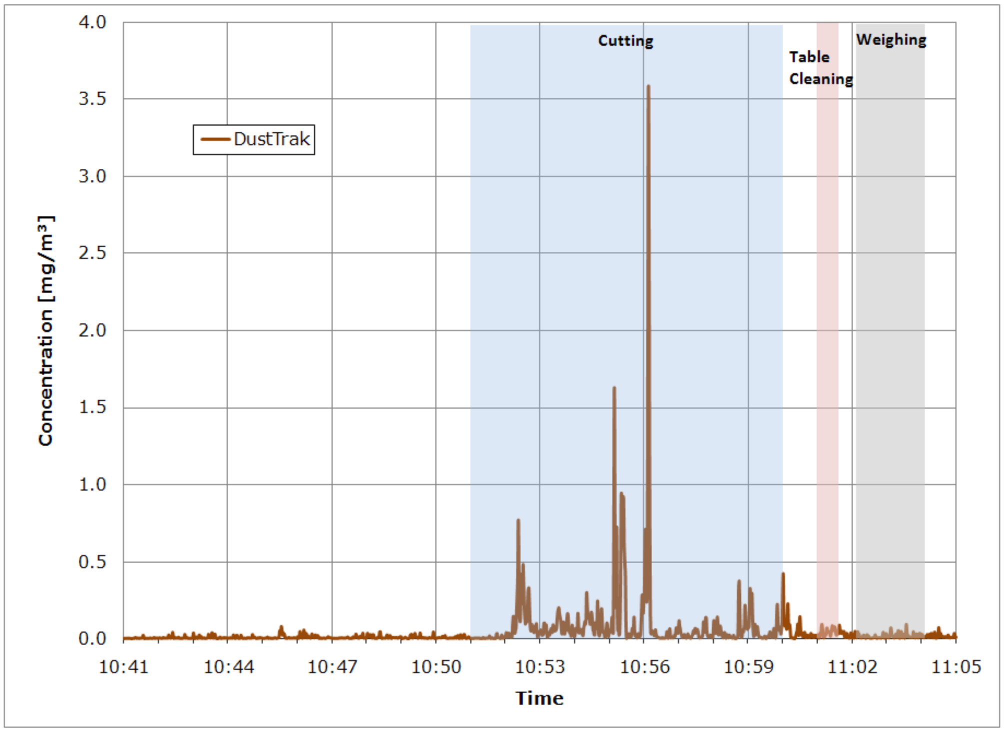


Figure 15. Mass concentration variations of the R&D activities monitored by DustTrak on 04/28.

(2) Sonication Chamber

The sonication process was monitored by two FMPS instruments: FMPS 1 used for background check and FMPS 2 for monitoring inside the chamber. The monitoring data showed no significant increase in particle concentration (**Figure 16**) during the monitoring period.

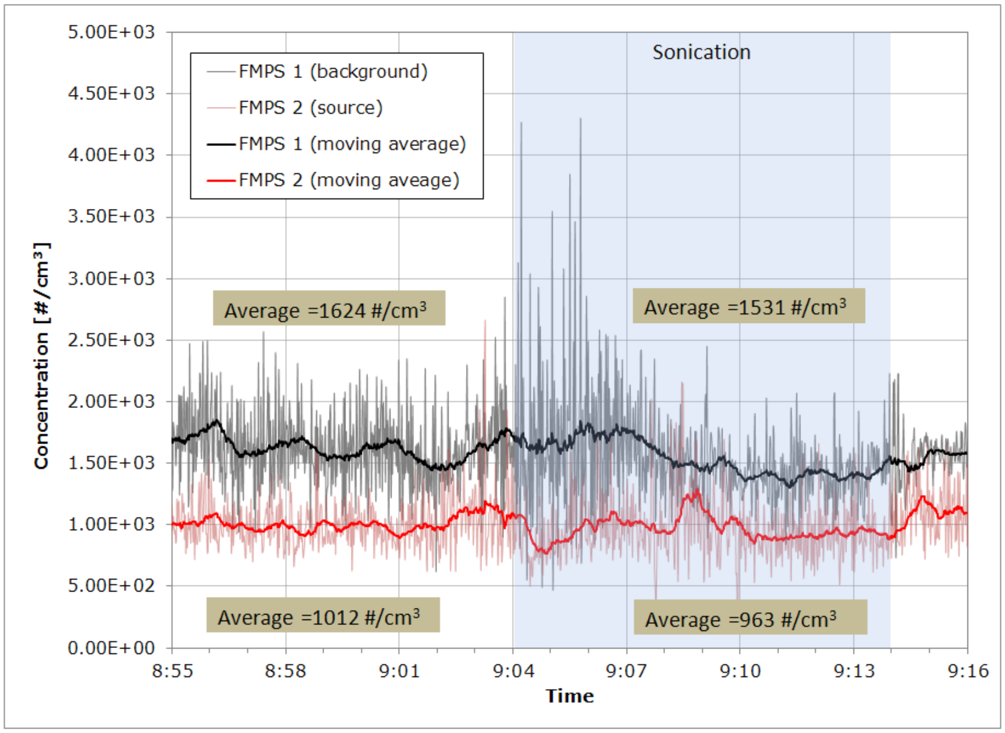


Figure 16. Sonication chamber monitored with FMPSs on 04/28.

Conclusions and Recommendations

A properly designed supply air ventilation system can provide plant ventilation, building pressurization, and exhaust air replacement. It is important to confirm that the local exhaust ventilation (LEV) system is operating as designed by measuring exhaust airflows. System checks should be completed periodically to ensure adequate system performance, including smoke tube testing, hood slot/face velocity measurements, and duct velocity measurements using an anemometer. These system evaluation tasks must become part of a routine preventative maintenance schedule to verify system performance. It is important to note that the collection and release of air contaminants may be regulated; companies should contact agencies responsible for local air pollution control to ensure compliance with emissions requirements when implementing new or revised engineering controls.

To minimize exposure and reduce the risk of exposure to nanomaterials, a few standard precautions should be followed in areas where exposures may occur:

- Use walls, doors, or other barriers to isolate rooms where nanomaterials are handled from the rest of the plant.
- Maintain production areas where nanomaterials are being produced or handled under negative air pressure relative to the rest of the plant.
- Place hoods, when possible, away from doors, windows, air supply registers, and aisles to reduce the impact of cross drafts.
- Provide supply air to production rooms to replace most of the exhausted air.
- Direct exhaust air discharge stacks away from air intakes, doors, and windows.
- Keep work areas inside of fume hoods and biological safety cabinets free of unnecessary clutter to improve their performance.

The following sections summarize the results of the process monitoring and engineering control evaluations. Where appropriate, recommendations have been included to improve performance of existing systems and to provide suggestions for alternate methods of containing and reducing the emission of nanomaterials to the workplace.

(1) CNT Weighout

Weighing nanomaterials (CNTs in this case) and other dry powder materials is a common process for nanomanufacturing. The task of weighing out nanomaterials can lead to worker exposure primarily through the scooping, pouring, and dumping of these materials. Many different types of commercially available laboratory fume hoods can be employed to minimize exposure during the handling of nanopowders. Other controls have also been used in the pharmaceutical and nanotechnology industries for containment of powders during handling and manipulation, including gloveboxes, biological safety cabinets, and newer nanomaterial handling enclosures.

The use of the BSC reduced the particle concentration by 36% in the worker's breathing zone during CNT weigh-out (**Table 3**) and 40% outside the hood following weigh-out when the supplemental fan was turned on. The average face velocity for the BSC was approximately 65 fpm when the supplemental hood fan was on and only 7 fpm when the only facility exhaust fan was operating (BSC fan not turned on). OSHA specifies that the average face velocity for chemical hood should be between 60–100 linear feet per minute [CFR 1910.1450 App A*]. The ACGIH Industrial Ventilation manual recommends 80–100 fpm for laboratory fume hood face velocity [ACGIH 2010]. The use of the hood with the supplemental fan yielded reasonable face velocities and reduced nanoparticle concentrations measured in the breathing zone. The BSC supplemental fan should be used whenever CNTs are being handled inside the BSC. In addition, the BSC hood work area was cluttered with equipment blocking the hood face area and some of the exhaust grilles. The BSC work space should be kept as clean as possible with those items not needed immediately stored outside of the cabinet to avoid blocking the airflow into the BSC.

(2) Mixing and Deposition of CNT Slurry

Worker exposure was measured during the handling of raw material (**Figure 9** and **Table 4**) during preparation of the slurry. To reduce potential for worker exposure and emissions to the workplace, raw material preparation and handling should be performed with engineering controls to prevent particle releases to the work environment during material handling and accidental spills. The use of a ventilated workstation could be employed to prevent the release of dust into the workplace during material handling. **Figure 17** shows a ventilated enclosure that could be used for raw material preparation to prevent particle emissions during transfer and weighing even though these bulk powders are not nanomaterials. This containment device allows for the manual weighing and transfer of dusts from a bulk container (such as a large bag to smaller containers) to the slurry. The exhaust air from the enclosure should be exhausted outside of the facility and away from outdoor air intakes.

*Code of Federal Regulations. See CFR in references.

Figure 17. Containment for handling bulk powders (modified and reprinted from Flow Sciences Inc. 2011).

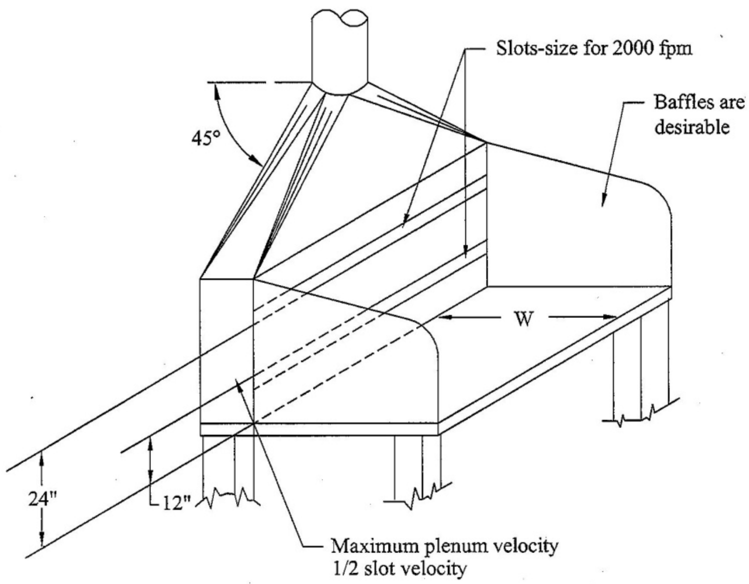
(3) Cutting of Substrate Materials

Two control measures were used for the cutting process: (1) the downdraft table for cutting CNT-deposited substrates with a rotary cutter, and (2) the canopy hood for cutting substrate rolls with a powered saw. Low emissions were measured during manual rotary cutting of substrate with concentrations not significantly different from the background. However, DustTrak data showed that similar rotary cutting in the R&D area did increase particle concentrations near the source. In general, this process is a lower energy task not expected to be associated with the release of large quantities of nanomaterials.

The evaluation of the performance and design of the downdraft table indicate that it is not likely to be highly effective for capturing contaminants generated during cutting or other tasks conducted on the table. However, improvements in the design and effectiveness of this table could be made. Because the manual cutting tasks are performed by workers along a bench-top station, the addition of slotted backdraft ventilation could be considered. There are commercially available sources for backdraft workstations, or they can be fabricated using appropriate design guidance **(Figure 18)**. This type of hood could be fairly easily retrofitted to the cutting area using the existing exhaust ventilation system. The implementation of a bench top slotted hood or other ventilated workstation design should be considered if production increases or if more energetic processes are used on this table.

Figure VS-90-01, from the book *Industrial Ventilation—A Manual of Recommended Practice, 25th ed.* contains recommendations for a welding ventilation bench hood (Figure 18). This type of design would be appropriate for the manual cutting workbench. The key design parameters are the overall flow rate of 350 cfm/ft of bench length, a slot velocity of 2,000 fpm and a maximum plenum velocity of ½ of the slot velocity. These design characteristics should provide adequate airflow to capture minimally energetic emissions for a work bench no greater than 2 feet wide. If implemented, baffles should also be placed along the length of the bench at appropriate work intervals to enhance hood performance. The addition of horizontal baffles attached at the top of the non-tapered portion of the hood and extending 6 inches or more will further enhance the slot hood performance.

FIGURE VS-90-01



Q = 350 acfm/ft of hood length
Hood length = required working space
W = 24" maximum, if W greater than 24" see Chapter 3
Minimum duct velocity = 2000 fpm
$h_e = 1.78\ VP_s + 0.25\ VP_d$

General ventilation, where local exhaust cannot be used:

Rod, diam.	acfm/welder
5/32	1000
3/16	1500
1/4	3500
3/8	4500

or

A. For open areas, where welding fume can rise away from the breathing zone: acfm required = 800 x lb/hour rod used

B. For enclosed areas or positions where fume does not readily escape breathing zone: acfm required = 1600 x lb/hour rod used

For toxic materials higher airflows are necessary and operator may require respiratory protection equipment.

Other types of hoods
Local exhaust: See VS-90-02
Booth: For design see VS-90-30
Q = 100 acfm/ft^2 of face opening
MIG welding may require precise airflow control

ACGIH®	TITLE WELDING VENTILATION BENCH HOOD	FIGURE VS-90-01 DATE 1-10

CHECK CODES, REGULATIONS, AND LAWS (LOCAL, STATE, AND NATIONAL) TO ENSURE THAT DESIGN IS COMPLIANT.

Figure 18. Ventilated bench hood design to control emissions from manual cutting of substrate materials using the rotary cutter [ACGIH 2010].

Cutting with powered tools typically results in the release of much higher concentrations of particulates due to the higher level of energy imparted to the work piece. Overall, the use of a powered saw generated increased nanoparticle generation well above background concentrations. In this study, the use of a canopy-type hood above the process did not effectively control the emissions and resulted in a measureable increase in the worker breathing zone during cutting. Canopy hoods are generally used for capturing contaminants from hot processes. This allows the canopy hood to benefit from the buoyancy from the thermal updraft caused by the process to collect and contain process generated contaminants.

The canopy hood was ineffective for controlling emissions from cutting of substrate rolls using the powered saw (**Table 6**). The canopy hood drew particulates from the saw through the worker's breathing zone. When using exhaust ventilation, it is important that the worker not be placed in between the source of exposure and the ventilation exhaust (**Figure 6**). When this occurs, contaminants generated by the process can be pulled into the worker breathing zone prior to being exhausted. Circular and radial saws can be retrofitted with local exhaust ventilation to effectively capture dust generated during the cutting process at the source. The ACGIH industrial ventilation manual provides guidance for the design and use of local exhaust ventilation for powered tools [ACGIH 2010]. These designs typically include a ventilated shroud around the saw blade and a ventilation takeoff point at the cabinet for floor-mounted table saws (**Figure 19**). For circular saws, many manufacturers have developed dust controls that are integrated into the design of the saw. As production increases, the use of dust control is recommended for reducing worker exposure, controlling emissions to the work environment, and maintaining cleanliness in the production areas.

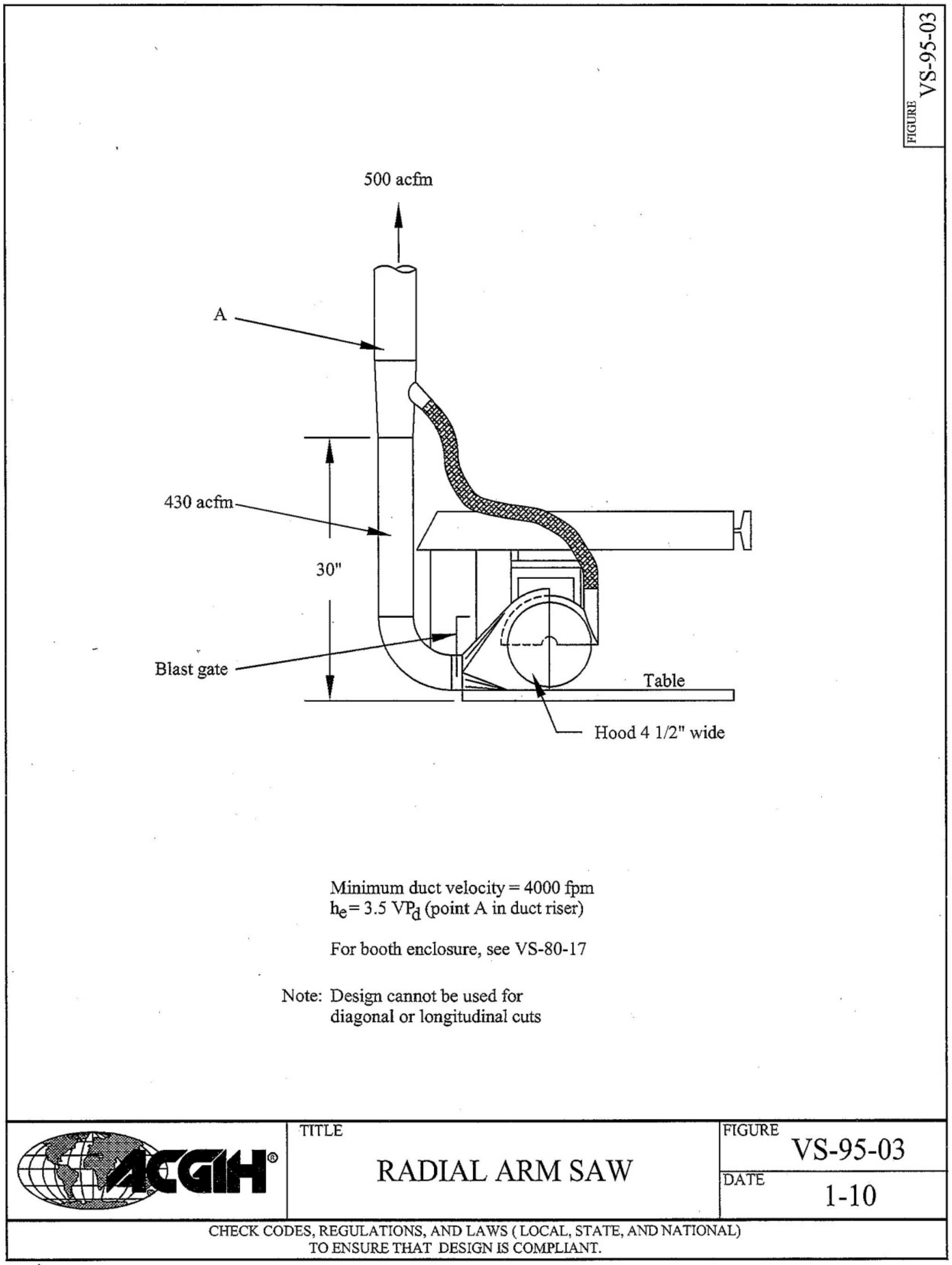


Figure 19. LEV designs to control dust emissions from the powered radial saw [ACGIH 2010].

(4) R&D Activities

Though most R&D activities involve handling a small quantity of nanomaterials, particle emissions were measured during rotary cutting of the test substrate deposited with CNTs. The same caution should be taken for the tasks of weighing and cutting in the laboratory by using similar engineering controls in the production areas. Performing these tasks inside a ventilated enclosure such as a chemical fume hood, BSC, or nanomaterial handling cabinet is recommended. During the sonication process, no particle emissions were detected from the sonication chamber (**Figure 16**). However, other researchers have found that sonicating materials can be a source of particulate emissions during nanomaterial production and use [Johnson et al. 2010].

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Acknowledgements

The authors would like to acknowledge the support and cooperation from the management and staff of the study site.

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