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## Design of an exposure chamber to test samplers used in the evaluation of personal exposure to nanoparticles

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### Abstract.

The aim of this study was to design a laboratory size exposure chamber for the testing of samplers used to collect personal exposure samples for nanoparticles. A polyethylene cylindrical container with a diameter of 42 cm and height of 60 cm was used as the testing chamber. The chamber was divided into 2 parts by an aluminium honey comb. Particles generated using a 1 jet Collison nebulizer (BGI) operating at a flow rate of 4L/min were inserted into the chamber via a tube located near to the top of the chamber. A heater was inserted just after the nebulizer to avoid condensation of water in the tubing, and dilution air, running at 10L/min was inserted just after the heater. As particle charge can dramatically affect sampling a particle neutralizer was attached to the generation system so as to neutralize the particles before they enter the chamber. A diffusion dryer was used to remove any water from the air stream prior to enter the chamber. A fan was used to mix and distribute the generated particles. After generation and mixing, the particles passed through the aluminium honeycomb which is essential to eliminate any turbulent or unwanted air flow. Six sampling ports along with a pressure gauge were placed on the walls 15 cm from the bottom of the chamber. The pressure gauge was added to ensure the desired pressure is achieved during sampling. The sampling ports allowed for the connection of five samplers and sampling pumps as well as the connection of an ultrafine particle counter. The exposure chamber was developed to assess various samplers for carbon nanotubes and cellulose nanocrystals. Results showed that the chamber was working properly and that mixing was sufficiently uniform to test samplers.

### 1. Introduction

The Nanotechnology Consumer Products Inventory lists over 1,600 consumer products containing nanomaterials [1]. In 2008, there were about 400,000 workers worldwide exposed to nanomaterials. This number is estimated to grow up to 2 million workers in 2015 [2]. Carbon nanotubes, in particular, have become a concern since they are believed to have asbestos-like fiber toxicity leading to mesothelioma and pulmonary fibrosis [3]. Recent animal studies have shown that exposure to carbon nanotubes lead to changes in mesothelial tissue [3, 4, 5]. However, there are no standardized methods for the sampling and analysis of nanomaterials and only few recommendations for occupational

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exposure limits are available [6]. In an effort to protect their workforce in the lack of methodology and regulation, the National Institute for Occupational Safety and Health (NIOSH) wrote a comprehensive report in which they recommend the use of NIOSH methods 5040 and 7402 to assess exposure to carbon nanotubes. In addition, NIOSH recommends to use an exposure limit of  $1 \mu\text{g}/\text{m}^3$  for elemental carbon in the respirable fraction, providing that carbon nanotubes and nanofibers are predominant in the sample [7]. The Government of Alberta, Canada, is concerned about the exposure to carbon nanotubes and has asked the University of Alberta to verify if it is practical to routinely use these methods to assess workers exposure to carbon nanotubes.

Various samplers are available for the collection of respirable particles. However, in North America, the IOM sampler coupled with polyurethane foam (Institute of Occupational Medicine, Edinburg, Scotland), and the cyclone coupled with a total dust cassette are by far the samplers the most commonly used to collect respirable aerosols. It was decided to test both the IOM and the cyclone to evaluate the difference between these samplers, and their efficiency in collecting carbon nanotubes.

Unfortunately there was no known workplace using or producing carbon nanotubes. It was then decided to build an exposure chamber to test our samplers. This paper describes the design of the exposure chamber.

## **2. Materials and methods**

### *2.1. Exposure chamber system*

The exposure chamber system was composed of an aerosol generator system, an aerosol neutralizer, and the exposure chamber itself. The whole system was based on designs previously published [8, 9, 10].

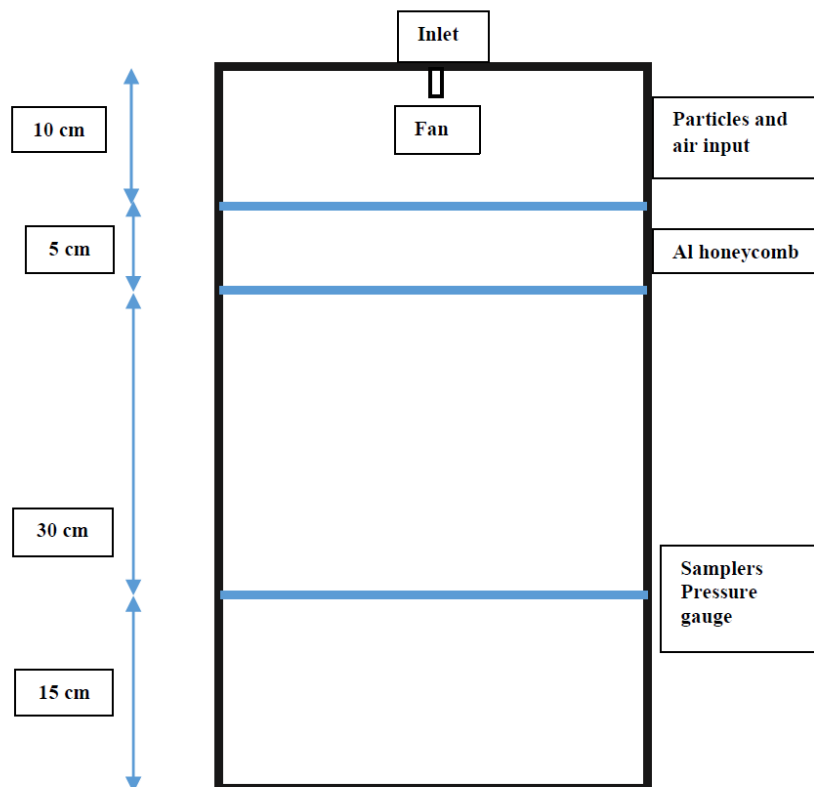
*2.1.1. Aerosol generation.* Aerosol generation was performed using a 1 jet Collison nebulizer (BGI, Waltham, Maryland)) followed by a heater to avoid condensation of water inside the tubing. A solution of carbon nanotubes in purified water (2.5 g/L) was prepared directly in the nebulizer jar and placed in an ultrasonic bath for 15 minutes. The gas pressure used was 50 psi, which produces a flow of approximately 4 L/min at the outlet of the nebulizer. To balance the inlet flow to the outlet flow inside the exposure chamber, purified compressed air (ultra-zero air, Praxair, Mississauga, Ontario), was added just after the heater at a flow of approximately 10 L/min.

*2.1.2. Diffusion dryer.* In order to remove water from the aerosol particles, a diffusion dryer filled with indicating silica gel was installed between the aerosol generating system and the aerosol neutralizer.

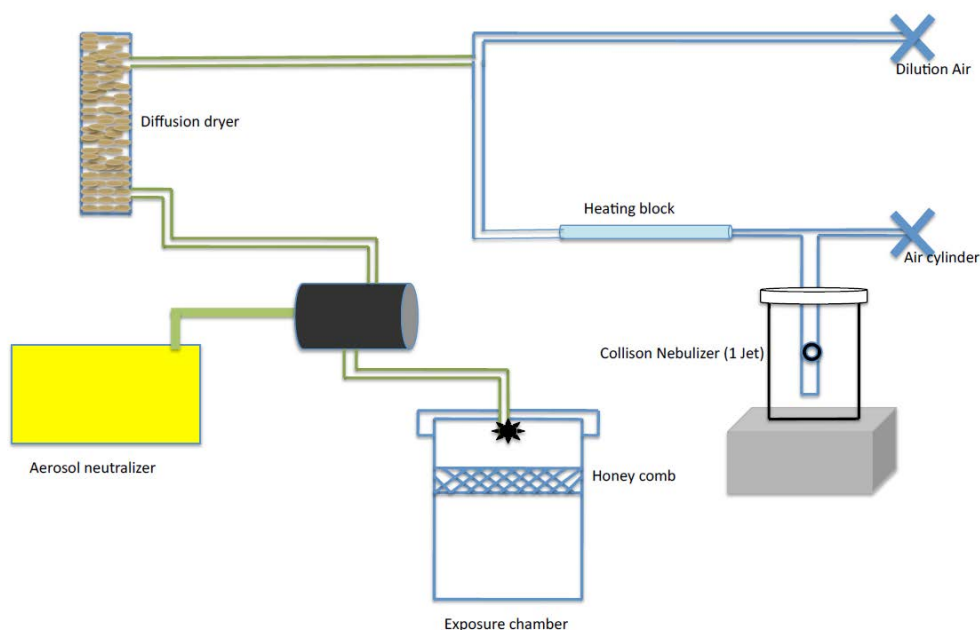
*2.1.3. Aerosol neutralizer.* Since the indoor air in the location where experiments are conducted (Edmonton, Canada) is extremely dry in winter (approximately 20% relative humidity), static charging is a significant problem. An EAN 581 electrostatic aerosol neutralizer (Topas, Dresden, Germany) was used to eliminate the effects of static charging and to balance the positive and negative charges inside the chamber.

*2.1.4. Exposure chamber.* A 60 cm height and 46 cm outside diameter polyethylene tank was used as the testing chamber. The aerosol inlet was inserted on top of the chamber along with a fan to promote mixing of the particles. An aluminium honeycomb (5 cm height) was inserted into the chamber 10 cm from the top to eliminate unwanted turbulence inside the chamber. Six ports and a pressure gauge were placed at 15 cm from the bottom of the chamber. The ports allowed for the connection of five samplers and a P-Trak (TSI, Shoreview, Minnesota). The pressure gauge was used

to verify the pressure inside the chamber since the chamber had to be run at atmospheric pressure. Layout of the chamber is shown in Figure 1 and scheme of the whole system is shown in Figure 2.



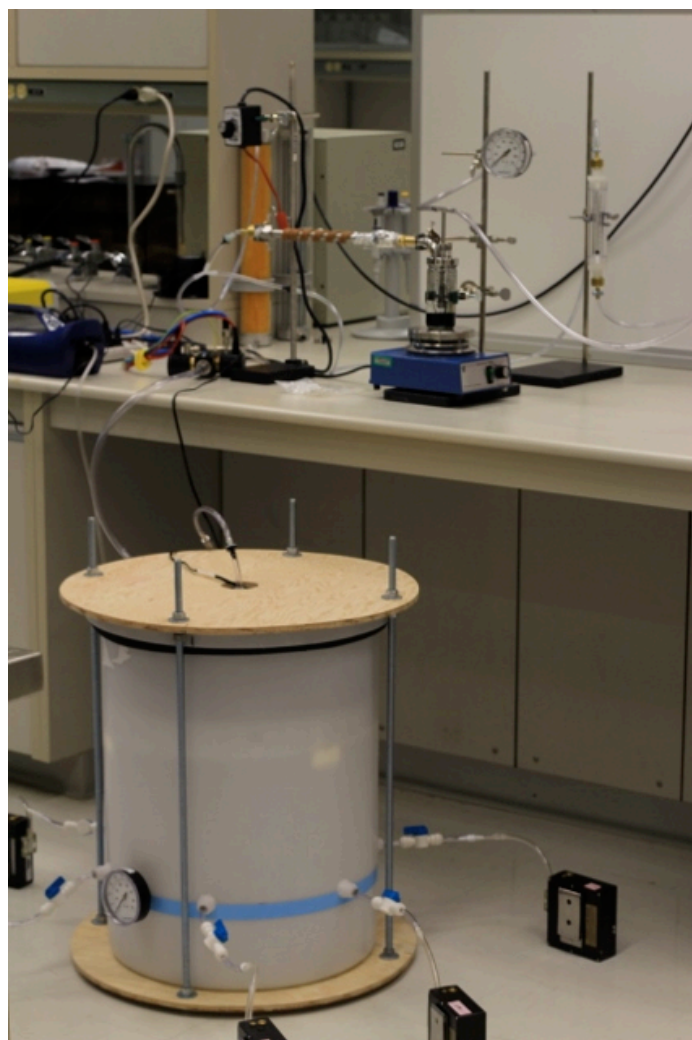
**Figure 1.** Layout of the exposure chamber



**Figure 2.** Design of the overall system

## 2.2. Testing of samplers

Testing was performed using two types of samplers: 1) the IOM aerosol sampler (conductive plastic SKC, Eighty Four, Pennsylvania), and 2) a total dust cassette connected to a cyclone (GS3, SKC, Eighty Four, Pennsylvania). The IOM sampler was equipped with polyurethane foam to collect only the respirable fraction. The IOM sampler runs at 2 L/min while the cassette/cyclone assembly runs at 2.75 L/min. Preliminary testing was performed using GLA-5000 PVC filters 25 mm and 37 mm diameter, 5  $\mu$ m porosity (SKC, Eighty Four, Pennsylvania). The chamber was operated 4 times at a carbon nanotube concentration of 2.5 g/L. In each run, 2 samplers of one type and 3 samplers of the other type were connected to the sampling ports and the P-Trak was attached to the 6<sup>th</sup> port just to control the particle count. In total ten filters were collected with the IOM sampler and ten filters were collected with the cassette/cyclone assembly. Pumps were calibrated prior to and after sampling and average flow was used. PVC filters were weighed before and after sampling in a temperature and humidity-controlled room. Filters were allowed to equilibrate in the room for 24 hours before being weighed. The sampling set-up is shown in Figure 3.



**Figure 3.** Sampling set-up.

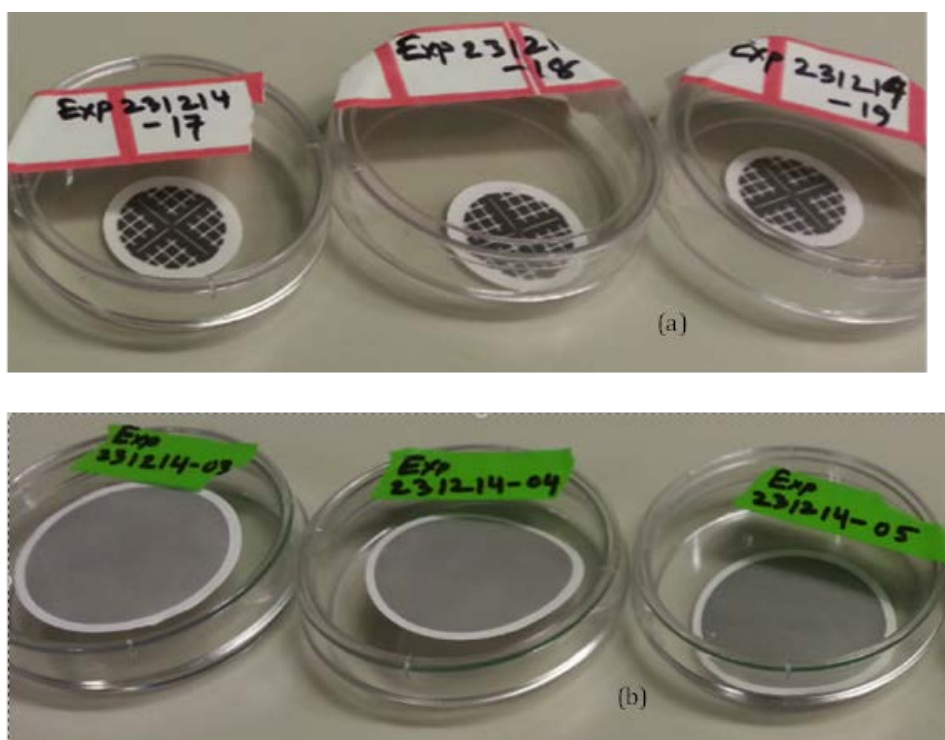
### 3. Results and discussion

Preliminary testing showed that the mixing inside the chamber was not working properly as a visual difference was noted between filters (Figure 4).



**Figure 4.** Preliminary testing performed using IOM samplers. Sample on the left shows fewer particles on the filter than the other four samples.

It was found that there were leaks at this particular port. All the ports were sealed to avoid potential leaks using silicone paste. In the following test, filters did not show any visual difference in particulate loading (Figure 5) therefore both samplers were tested and particulate concentration was measured.



**Figure 5.** Loading on filters for a) IOM sampler, and b) cassette/cyclone assembly



For the cassette/cyclone assembly, the mean mass concentration was  $0.81 \text{ mg/m}^3$  (standard deviation:  $0.14 \text{ mg/m}^3$ ,  $n = 10$ ). The IOM sampler gave a mean mass concentration of  $0.83 \text{ mg/m}^3$  (standard deviation:  $0.19 \text{ mg/m}^3$ ,  $n = 10$ ). Average concentrations were similar for both samplers but the IOM sampler showed a higher variation than the cassette/cyclone assembly with respective coefficients of variation of 22.5% and 17%. Number concentration of particles obtained from the P-Trak showed a fast increase from approximately 800 part/mL for the baseline up to 80,000 part/mL during the experiment. The particles concentration stayed stable during the experiments and went back to baseline rapidly at the end of the experiment.

The results show some variation in the concentration of particles within the exposure chamber probably due to the presence of turbulence inside the chamber. However, the variation was considered to be acceptable to test methods and samplers for the collection of nanoparticles. It is interesting to note that, for carbon nanotubes, both types of samplers collect the same amount of particles, therefore they are both suitable to use for the collection and analysis of carbon nanotubes. Further work will include the testing of samplers for the analysis of elemental carbon in carbon nanotubes, testing of the chamber and method development for cellulose nanocrystals and titanium dioxide nanoparticles.

### Acknowledgments

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