Characterization of Airborne Nanoparticle Released from Consumer Products

Letter Report to U.S. Consumer Product Safety Commission
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Andrew K. Persily
Michael Magee
Donghyun Rim

Indoor Air Quality and Ventilation Group
Energy and Environment Division
Engineering Laboratory
National Institute of Standards and Technology
Gaithersburg, Maryland

This letter report provides a project update reflecting the activities to date under the FY2012 interagency agreement between CPSC and NIST. The objective of that agreement is to develop testing and measurement protocols for determining the quantities and properties of nanoparticles released from flooring finishes and interior paints, including their subsequent airborne concentrations. This document focuses specifically on the project task titled Chamber Measurements of Airborne Nanoparticles, including the design of a chamber for measuring airborne concentrations of nanoparticles generated by abrasion flooring and paint samplers.

Background
Beginning in the first year of this project, the initial focus was to design and conduct benchtop experiments to determine the chemical composition, morphology, and size of the particles that are released as a result of abrasion. These experiments have involved the use of a Taber abrader per ASTM Standard Test Method D 4060-95 (2007) to simulate typical mechanical abrasion of a number of UV-cured polyurethane floor and acrylic latex paint samples. These samples were prepared to include silica (SiO$_2$), alumina (Al$_2$O$_3$), and titanium (TiO$_2$) nanoparticles that are increasingly being used to improve the scratch resistance of polymeric coatings. Those benchtop measurements were intended primarily to determine the number, as well as the chemical and morphological properties, of nanoparticles that accumulate on the sample surfaces after abrading.

Another key objective of this work is to characterize the airborne release of these nanoparticles in a chamber configured to represent a ventilated space as a means of quantifying the exposures that might exist in a real building. The original plan was to conduct these measurements in an existing 30 m$^3$ environmental chamber at NIST by subjecting the materials to an abrasion protocol (adapted from the benchtop tests) within the chamber and then measuring the resulting airborne
particle concentrations over time. Airborne nanoparticle concentrations would be measured over a size range of 3 nm to 100 nm using a scanning mobility particle sizer (SMPS) consisting of an electrostatic classifier, a nanodifferential mobility analyzer and a water-based condensation particle counter. NIST has been using such an SMPS system for other airborne nanoparticle measurements in residential settings and expects it will also work well for these tests.

During FY2012, the use of the 30 m$^3$ chamber for these measurements was considered more closely and a number of design and implementation issues were identified. The primary issues concerned safety and transferability of the test procedure. In the area of safety, the need to contain the released nanoparticles to control the potential exposure of staff in and near the laboratory housing the chamber was one such issue. Approaches considered included a filtered “anteroom” at the entrance of the chamber, depressurization of the chamber with a HEPA filtered exhaust, and effective chamber cleaning protocols. The complexity of these measures became a matter of potential concern as to their manageability for the tests planned for this project. In addition, one of CPSC’s key goals for this project is to develop test protocols that can be standardized and used by others to make similar measurements on other materials in other settings. Using such a large and unique chamber does not support such transferability. Therefore, another approach to the airborne measurements was considered, specifically the use of a smaller chamber that would surround the abrader device and reside within the nanohood being used to contain the benchtop experiments. The next section of this letter report describes the conceptual design of this small chamber.

**Conceptual Design of Abrader Enclosure for Airborne Nanoparticle Measurements**

Figure 1 contains a schematic of the abrader design being proposed for the airborne nanoparticle measurements under this effort. As depicted in this schematic, the chamber will be located within the XPert Nanohood already installed at NIST, which is being used for the benchtop experiments, and will surround the Taber abrader in which the samples are housed during testing. The relevant dimensions for the abrader, hood and chamber are as follows:

- Taber Abrader: L 50 cm x D 48 cm x H 43 cm
- XPert Nanohood: L 178 cm x D 58 cm x H 76 cm
- More typical lab exhaust hood: L 122 cm x D 66 cm x H 66 cm
- Nominal chamber dimensions for initial design: L 63 cm x D 55 cm x H 56 cm

The chamber will be sized to fix in a more typical exhaust hood rather than the larger XPert Nanohood being used by NIST. This smaller size will make the chamber more amenable to use in a wider range of laboratory facilities. Based on dimensions provided above, the volume of the chamber is about 200 L.
A number of design issues have been considered and are considered to be largely resolved as summarized below.

**General:** The chamber needs to be sufficiently airtight such that airflow into and out of the chamber can be controlled, but it need not be “hermetically sealed.” In order to limit the entry of nanoparticles from the volume of the nanohood into the chamber, the design is based on maintaining a slight positive pressure in the chamber relative to its surroundings. This will be done by supplying the chamber with compressed air from the laboratory supply, which will be controlled with an electronic mass flow controller and pass through a HEPA filter. The chamber will be exhausted using a vacuum pump contained in a TSI-3089 nanoparticle collector, but at a rate lower than the supply airflow into the chamber to maintain a slight positive pressure.

The chamber will have a front viewing window and access panel so that the operation of the abrader can be observed during a test and so that tests can be set up without having to move the hood. All chamber materials will be made of conductive material, such as stainless steel, to reduce electrostatic charge on the surface that will attract particles. Static-dissipative polycarbonate and acrylic material are currently being considered for the window/access panel. The current plan is to use a NEMA Type 1 or Type 3 enclosure for the chamber per the specifications above. The manufacturing of the enclosure, including the door and the static-dissipative window in the door, will be outsourced. Circular holes for the hose fittings and abrader power wire will be installed at NIST after the enclosure is delivered.

**Chamber sampling for particle measurement:** The chamber air will be sampled through a sampling port, as shown in Figure 1, which will be connected to either a handheld particle counter (TSI model CPC-3007 as referred to in the figure) or the SMPS described earlier. In addition to
the exhaust outlet a sampling tube located near the center of the chamber will be installed to
to sample particles within the chamber with either the CPC-3007 or the SMPS. Note that the TSI-
3089, CPC-3007 and the SMPS all have HEPA filters on their outlets, so the air exhausted to the
room from these devices will be filtered for nanoparticles.

**Chamber flow rate:** The design flow rate through the chamber will be in the range of 0.25 air
changes per hour to 1.0 air changes per hour or h⁻¹. These values correspond to typical ventilation
rates in buildings and building rooms. Given the chamber volume of about 200 L, those flow rates
correspond to a range of roughly 1.0 L/min to 3.5 L/min (0.05 cfm to 0.15 cfm). The filtered
supply air will be delivered to the chamber in a manner that does not disrupt the airflow in the
vicinity of the sample being abraded and that is consistent with the air speeds in ventilated spaces.
The current design includes a supply vent located in the bottom side corner of the chamber while
the exhaust outlet is in the upper corner opposite the supply to allow for effective air distribution
and to reduce short-circuiting of airflow within the chamber.

**Background concentration:** Interpreting the airborne nanoparticle concentrations measured
during the abrasion tests requires an understanding of, and potentially an adjustment for,
background nanoparticle concentrations in the lab housing the nanohood. These adjustments could
be quite complicated given the unpredictable variations in background levels over time, which are
driven largely by variations in outdoor concentrations. Given that the nanohood re-circulates room
air through its high-efficiency filters, it was considered possible that perhaps this filtration would
reduce the background particle level in the room sufficiently such that the background
concentrations would not be an issue. However, as described below, the extent of reduction is not
sufficient to justify ignoring the background level in the room.

The supply airflow to the room from the building HVAC system is constantly introducing
nanoparticles into the room due to the nanoparticles contained in the outdoor air. The flow rate of
the air through the nanohood and its subsequent filtration reduces the particle concentration in the
room. The following calculation provides an estimate of the reduction in the room particle
concentration due to filtration of recirculation air through the nanohood based on a single-zone,
steady-state mass balance of particles in the room. This mass balance equates the rate of entry into
the room to the rate of removal by the nanohood as follows:

\[ Q_{lab} C_{sup} = Q_{nh} C_{lab} \]

where \( Q_{lab} \) is the airflow rate into the lab, \( Q_{nh} \) is the airflow rate through the hood, \( C_{sup} \) is the
airborne concentration in the air supplied to the lab, and \( C_{lab} \) is the concentration in the lab air. The
nanohood removal efficiency is assumed to be 100%. \( Q_{lab} \) can be estimated by multiplying the lab
volume of 135 m³ by a typical laboratory ventilation rate of 5 air changes per hour, which is about
188 L/s. The flow rate through the nanohood is roughly 225 L/s based on its rating information.
Solving for the concentration in the lab air yields:
\[ C_{\text{lab}} = C_{\text{sup}} \frac{Q_{\text{lab}}}{Q_{\text{nh}}} \]

Based on the estimated airflows for \( Q_{\text{lab}} \) and \( Q_{\text{nh}} \) above, the concentration in the lab air is about 84% of the concentration in the supply air. Therefore the nanohood reduces the background concentration in the room by only about 15%, which is not adequate to neglect the background concentration.

To provide further information on the background nanoparticle concentrations in the laboratory housing the nanohood, a series of measurements were made using a TSI model CPC-3077 laminar-flow handheld particle counter. The Model 3007 can detect particles between 10 nm and 1 \( \mu \)m in diameter, reported as the total number in this size range, with an accuracy for concentrations up to 100,000 particles/cm\(^3\) within ±20%, as stated by the manufacturer. The CPC operates by drawing an aerosol sample continuously through a heated saturator, in which alcohol is vaporized and diffuses into the sample stream. The aerosol sample and alcohol vapor pass into a cooled condenser where the alcohol vapor becomes supersaturated and ready to condense. Particles present in the sample stream serve as condensation sites for the alcohol vapor. Once condensation begins, particles grow quickly into larger alcohol droplets and pass through an optical detector where they are counted.

Figures 2 through 4 present three time profiles of the background particle concentration in the laboratory housing the nanohood without the abrader running. These data show the variability in these particle concentrations over time and between the different measurement periods. This variability supports the inability to predict these background levels and the need to filter the air coming into the chamber that will be used to monitor the airborne nanoparticle levels due abrasion of the samples.
Figure 2  Background nanoparticle concentrations in laboratory on 6/5/12

Figure 3  Background nanoparticle concentrations in laboratory on 6/6/12 (Test #1)
Next Steps in Research Effort
At the beginning of FY2013, NIST will procure the materials and instrumentation needed to construct the chamber described in this report. It will be assembled and tested to verify that it is performing as intended, including the evaluation of chamber pressures, flow rates and uniformity of particle concentration within the chamber. In addition, chamber cleaning protocols will be developed along with procedures to confirm that the chamber is effectively cleaned between tests. Once those items have been completed, testing will commence to evaluate the airborne nanoparticle concentrations associated with sample abrasion.

Disclaimer
Certain trade names or company products are mentioned in the text to specify adequately the experimental procedure and equipment used. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment is the best available for the purpose.