Reference Material to Improve Reliability of Building Product VOC Emissions Testing

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Building materials and products are a significant source of volatile organic compounds (VOC) in the indoor environment. Reducing VOC emissions can improve occupant health and productivity, and perhaps support lower building ventilation requirements and associated energy usage. As a result, use of low VOC emission products is a key part of sustainable or "green" building labeling programs. To demonstrate that products have low emission rates, manufacturers rely on several different emissions assessment programs. Typically, these programs require a third-party independent laboratory measurement of a product's VOC emission rate in a test chamber. Test chamber emission rates are then compared to pass/fail criteria to determine a product's eligibility for a program label. However, existing green product labeling programs are not yet supported by consistent estimates of VOC emission rates. For example, it is common to see large coefficients of variation (> 40 %) in material emission rates measured between different laboratories for the same test material (Howard-Reed and Nabinger, 2006).

Much of the within and between laboratory variation is due to the complexities associated with product emissions testing. First, there are multiple steps in the testing process, including: 1) selection, packaging, transport and storage of material/product samples; 2) preparation and conditioning of sample specimens; 3) operation of emission test equipment; 4) gas-phase sampling; 5) sample analysis; and 6) data analysis and interpretation. In addition, product emission rates change with time and chamber air concentrations of many chemicals are relatively low and challenging to measure. Also, test chambers and analytical equipment are not standardized and thus vary between laboratories. Finally, there are many different test methods and product labeling programs in use today.

It is not necessary that every laboratory conducting product emissions testing use the same type of chamber or analytical equipment. However, every laboratory should be able to demonstrate that its testing equipment and methods can measure product emission rates with an acceptable uncertainty. The most common performance-based validation approach for product emissions testing has been the use of inter-laboratory studies (ILS). Recently, Europe conducted several large-scale inter-laboratory studies to qualify labs for various labeling schemes, such as the German AgBB (German Committee for Health-related Evaluation of Building Products) product label. However, inter-laboratory studies require the use of a homogeneous emissions source and the coordination of many different laboratories over a period of several months. And, in the end, it is only possible to characterize laboratory performance based on a measured mean value and standard deviation, not a "true" reference value.

One way to address several of these ILS issues, and perhaps eliminate the need for expensive inter-laboratory studies, is to use a reference material with a known emission rate. The International Organization for Standardization (ISO) defines a reference material as a substance "sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process," e.g., calibration of an apparatus, assessment of an apparatus, or the assessment of a measurement method (ISO 2008). Today, there are no homogeneous building materials that have an independently known emission rate. As a result, the National Institute of Standards and Technology (NIST) is working with Virginia Tech (VT) to develop a series of reference materials that mimic the emissions characteristics of real building materials and chemicals in a chamber test.

The development of this reference material for product emissions testing has involved several steps. The first step was to select a suitable substrate and representative VOC for the prototype. The polymer polymethyl pentene was determined to be a pure, uniform and stable material choice. Toluene was chosen as the first VOC since it is a chemical commonly found in building products, is relatively easy to measure, and is compatible with the chosen polymer film. The films were loaded through a diffusion process by exposing them to a toluene gas stream until equilibrium is reached between the material-phase and the gas-phase. Up to 42 films have been loaded in a single batch, with tests showing no significant differences between films produced within one batch. A microbalance holding an extra single film was also exposed to the toluene gas stream and was used to confirm the batch of films had reached equilibrium, typically after 10 to 14 days of loading. Once the films were saturated with toluene, they were wrapped in aluminum foil, sealed in a plastic bag, and placed in a cooler with dry ice for shipment to test laboratories. It has been determined that cold temperatures (approximately -20 °C) are required to preserve the integrity of the films over time. As a result, test laboratories are instructed to store the films in a laboratory freezer until ready for use.

The next step in the reference material's development was to characterize its performance in a test chamber. Chamber tests were conducted at NIST using a test method based on ASTM's Standard Guide for Small-Scale Environmental Chamber Determination of Organic Emissions from Indoor Materials/Products (ASTM D5116-10). The reference material test method specifies the following chamber set points: temperature (23 °C), relative humidity (50 %), and airflow rate (in the range of 0.050 m^3/h to 0.065 m^3/h). The sampling and analytical methods are left to the discretion of each laboratory. Again, the reference material is designed to be a tool for laboratories to demonstrate their ability to achieve relatively accurate results. NIST chamber results showed the prototype film to behave as expected with an emission profile similar to a typical "dry" material (e.g., flooring) (Cox et al., 2010).

Once the performance of the film was well-established in the NIST chamber, the reference films were distributed to13 different test laboratories from several countries through a series of multi-laboratory comparisons (Howard-Reed et al., 2011a; Howard-Reed et al., 2011b). Relative standard deviations between laboratories were often less than 10 %, indicating the material is a sufficiently homogeneous and consistent emissions source in different chambers. Next steps in the development process include improving the material's packaging for shipment and storage and verifying its loading and performance under different environmental conditions. There are also plans to expand the reference material development to include other chemicals, such as formaldehyde.

A unique advantage of this reference emission source is that its emission profile can also be predicted by a fundamental mass transfer model (Cox et al., 2010; Liu et al., 2011). The emission of a VOC from a homogeneous dry material to a well-mixed chamber is dependent on its internal diffusion within the material, characterized by diffusion coefficient D, and its partitioning between the material-phase and air-phase at the material/air interface, characterized by its partition coefficient K. By measuring D, K, and the initial VOC concentration in the material, the film's emission rate of toluene can be predicted for a given film thickness and surface area. Using a specific chamber's volume and airflow rate allows the determination of the chamber air VOC concentration as a function of time. The model's predicted results have been compared to the chamber measurements to date. In general, the model has compared well with the chamber test results when no measurement issues occurred. However, the model has also been responsible for identifying measurement issues when there are discrepancies. For example, the model identified an issue with an early sample holder design. The sample holder was designed to press the reference film flat against an aluminum platform such that the toluene would only emit from one side of the material. However, the model assuming that one side of the material was exposed underestimated the measured results indicating a larger than expected toluene source. It was later confirmed that the samples were not held perfectly flat on the platform and that air gaps existed beneath the samples. A new sample holder was designed to expose both sides of the film leading to results that match a two-sided emissions model (see picture in Figure 1 below), thus illustrating the value of the predictive model.

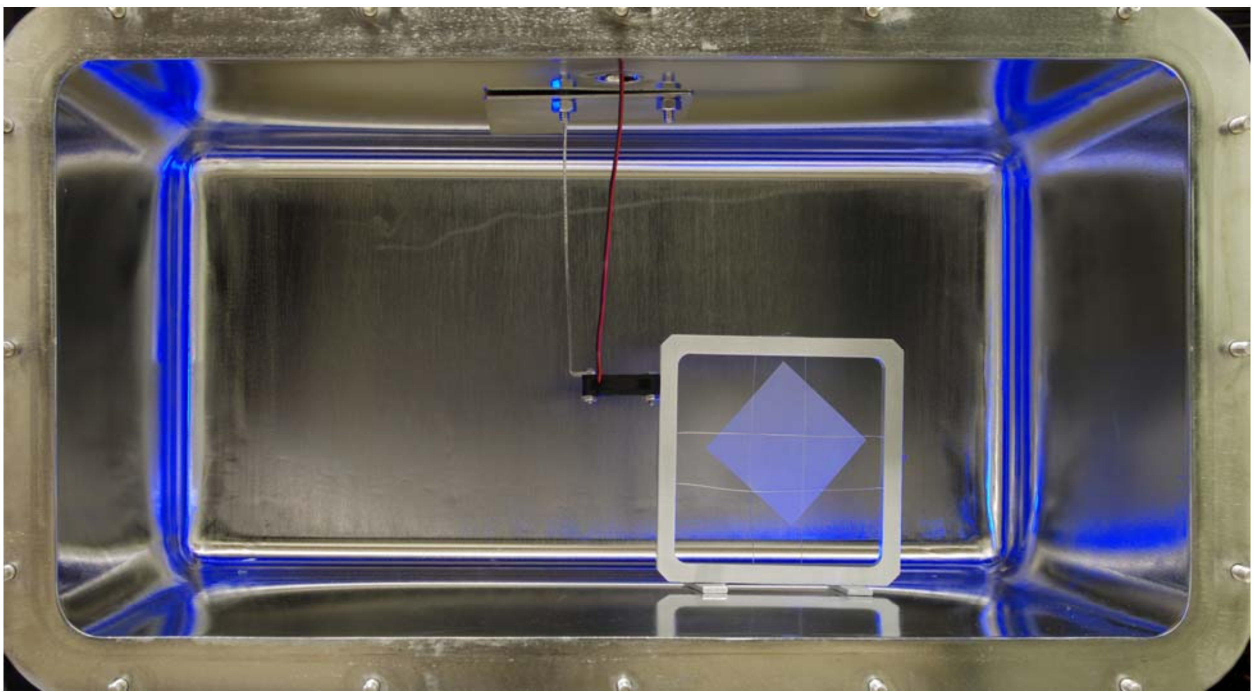

Figure 1. Reference film in NIST 50 L stainless steel chamber.

Once commercially available, the reference emissions source will have several potential industrial applications. First, it will provide laboratories valuable feedback regarding their chamber performance and analytical capabilities. Since the material will have an independently known reference emission rate, time-consuming and expensive inter-laboratory studies would no longer be required to assess a laboratory's performance and test laboratories would have the option to assess their performance at any time. If a laboratory's measured results do not match the reference value, the reference material could be coupled with an analytical check standard to identify the root cause of the disagreement. Inconsistencies between test laboratory results have been a source of frustration for several stakeholders, including 1) manufacturers who pay a lot of money for these tests but sometimes have limited confidence in the results; 2) emissions test laboratories who question the results from other laboratories, since there is no way to check the accuracy of the emissions results; and 3) consumers who have limited confidence they are actually purchasing a low emitting product. A reference material with an independently known emission rate has the potential to build consensus and confidence in emissions testing as well as "level the playing field" for product testing laboratories and manufacturers. And, ultimately, the consumer should be getting a better product for a healthier indoor environment.

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