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# Improving the Reliability of VOC Emissions Testing of Building Products

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## ABSTRACT

*Manufacturers of building materials and furnishings are increasingly using emissions chamber testing to demonstrate low volatile organic compound (VOC) emissions from their products and to qualify for “low-emitting” certification and sustainable labeling programs. Currently, however, there are limited standard test methods developed by voluntary consensus standard bodies for measuring VOC emission rates in chambers, which has led to concerns about the reliability of the test results. Round robin testing conducted to date has revealed significant inconsistencies in VOC emission profiles of the same product between testing laboratories with no means to establish the accuracy of the results. To address these inter-laboratory discrepancies and improve the reliability of product emissions testing, the National Institute of Standards and Technology (NIST) has identified the following technical strategies: 1) standardize emissions test methods; 2) develop proficiency standards for emissions testing, including standard reference materials; and 3) create a laboratory accreditation program. An initial plan for these strategies has been developed, and progress has been made on the development of a reference material to calibrate VOC emission rate facilities. To this end, NIST is working with Virginia Tech, who have synthesized a prototype reference material by loading or “charging” a polymer film with a specific VOC in the presence of supercritical carbon dioxide. The existence of a reference material to verify the accuracy of emission tests is expected to improve the reliability of the VOC emission rate data that are currently being used to make health-based purchasing decisions.*

## INTRODUCTION

In recent years, many building products and materials have been marketed for use in sustainable buildings. The U.S. Environmental Protection Agency (EPA) has defined green building products as those having less of an “impact on human health and the environment than other products that perform a similar function” (EPA 2002). One attribute commonly used to characterize these products is their emission rate of volatile organic compounds (VOCs). Exposure to VOCs in building air has been linked to numerous adverse health effects (Molhave 1991) as well as reduced productivity (Bako-Biro et al. 2004). As a result, designers and manufacturers are pursuing products and materials with lower VOC emissions. In general, there are two levels of product labels that take into account VOC emissions when rating products. At a more global level is “sustainable” or “green” building assessment programs that consider

many different attributes such as building energy, land and water use along with indoor air quality (e.g., Leadership in Energy and Environmental Design (LEED)), thereby reducing the relative importance of VOC emissions criteria on the overall rating. There are also single attribute assessment programs that certify products solely on VOC emissions (Malin 2006). Typically, these emissions assessment programs require an independent laboratory measurement of a material’s VOC emission rate in a test chamber. Product emission rates are then compared to specific pass/fail criteria (e.g., low emission limits, health effect limits, etc.) to determine a product’s eligibility for a low-emitting VOC label.

However, existing emissions assessment programs for building products and materials are not yet supported by reliable VOC emission rate measurements. To date, there are very few standard test methods developed by voluntary consensus

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standard bodies that are applicable to product emission rate testing, no reference materials for inter-laboratory comparison, and no formal laboratory accreditation programs. As a result, there is a lack of consistency in material emissions testing. For example, seven recent round robin studies using different indoor materials resulted in a coefficient of variation (COV) as high as 284% with an average COV of 56% (Howard-Reed and Nabinger 2006). Most COV values from these round robin studies were greater than 40%.

To improve the reliability of product emissions testing, the National Institute of Standards and Technology (NIST) and Virginia Tech are working together to address a number of emissions testing issues. This paper provides a discussion of those measurement needs as well as a description of efforts to develop a prototype reference material for emissions chamber testing and to standardize key aspects of the emissions testing process. Results from this work have the potential to improve the validity of the emissions assessment programs, as well as to increase market competitiveness for indoor materials/products manufacturers and product emissions testing laboratories.

## **PRODUCT EMISSIONS MEASUREMENT RELIABILITY ISSUES**

The most common method for measuring product emission rates is in a laboratory chamber intended to mimic the conditions of a real building, i.e., temperature, relative humidity and airflow. More recently, other measurement approaches have emerged, such as emission cells (ASTM 2005) and micro-chamber/thermoextraction test facilities (Schripp et al. 2007). Most emissions test protocols include the following six steps: 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. A more detailed description of the emissions process and most of these steps can be found in the ASTM standard guide for small chamber testing (ASTM 2006a), the ASTM standard practice for large chamber testing (ASTM 2001), and the ASTM standard practice for emission cell testing (ASTM 2005). Variation in each of these steps or protocols can have a significant effect on emission rate results (Howard-Reed and Nabinger 2006). Comprehensive standard test methods that address all six steps of the testing process are therefore still needed to ensure consistent product emissions results.

Indoor product emissions measurements have been conducted for over 30 years (e.g., Berge and Mellegaard 1979; Kazakevics and Spedding 1979). It was not until the 1990s, however, that standards were first developed by voluntary consensus standard bodies, beginning with the publication in 1990 of the first ASTM emissions test standard titled "Standard Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions for Indoor Materials/Products (D5116)" (ASTM 2006a). Since then, additional standards have been developed, many of which are based on ASTM D5116, including several other ASTM standards (e.g.,

ASTM 2003; ASTM 2002a, etc.), European Union guides (e.g., ECA 1991), and purchasing specifications (e.g., CA DHS 2004). ASTM D5116 is easily considered the "most important guidance document on emissions testing" (Levin 2004), but it is only an ASTM guide, meaning it describes key factors influencing materials emissions and lists general options for conducting a test but does not specify detailed step-by-step test requirements (ASTM 2006b). As a result, ASTM D5116 does not lead to a specific test result, which leaves room for crucial differences in test procedures. For example, D5116 does not specify the chamber operating conditions (e.g., temperature, relative humidity, air change rate, etc.), which can have a significant impact on emission rate results. In fact, most of ASTM's emissions test standards are either guides or practices; an ASTM practice provides specific instructions for conducting a test but does not produce a specific test result (ASTM 2006b). The only related ASTM emissions standard test methods are the Test Method for Determining Formaldehyde Concentration in Air from Wood Products using a Small Scale Chamber (ASTM 2002b) and the Test Method for Determining Formaldehyde Concentrations in Air and Emission Rates from Wood Products Using a Large Chamber (ASTM 2002c), which are for a single material type, single chemical and specific test facility. Developing standard test methods that produce specific test results has been difficult, largely due to the complexity of the emissions process and the many points of variability in the testing procedures.

Several mass transfer processes affect the rate at which VOCs are emitted from a product to the air including diffusion within the material, desorption into the adjacent air, and convective mass transfer through the boundary layer. While every material is different, many can either be classified as a "dry" or "diffusion-controlled" source of chemical emissions or a "wet" or "evaporation-controlled" source of chemical emissions. A diffusion-controlled source is limited by the movement of contaminants within the material. The rate of migration depends primarily on the diffusivity of the VOC, temperature and structure of the material. An evaporation-controlled source is limited by the rate of contaminant transfer from the material surface through the adjacent boundary layer into the surrounding air. The rate of mass transfer from the surface is primarily dependent on VOC volatility, and air velocity and turbulence near the material surface.

Even with standard test methods, it is possible for different laboratories to obtain a different test result on the same sample and even for a single laboratory to not be able to reproduce its own test results. Therefore, in addition to standard test methods, there is also the need for proficiency checks to determine both within-laboratory and between-laboratory variability. To date, typical proficiency checks include characterizing the accuracy of the analytical system, the sampling method, and the ability to recover mass in the test equipment. However, these checks are generally unique to each laboratory and have not yet been standardized. To isolate issues with the analytical system and method, it is possible to directly inject compounds

at a known concentration or to use sorbent sampling tubes spiked with a known contaminant mass. Magee et al. (2003) reported within-laboratory analytical uncertainties of approximately 13% to 35% based on the relative response factor for 24 different compounds for thermal desorption-gas chromatography/mass spectrometry analysis of sample tubes collected from emission chamber tests. High Performance Liquid Chromatography (HPLC) analysis of carbonyl compounds from the same emission chambers showed reduced uncertainty (approximately 2.5% to 5%), indicating that both the nature of the specific analyte and the analytical method employed are important factors in determining measurement error. Assuming an accurate analytical system, it is also important to check the sample collection technique by sampling from an atmosphere of known concentration. A European inter-laboratory comparison study required that all participating laboratories collect samples from the same chamber with the coefficient of variation for replicate samples not to exceed 10% for each laboratory, and with deviations from the known reference value less than  $\pm 20\%$  for the mean and less than  $\pm 50\%$  for individual results (De Bortoli et al. 1999). Chamber mass recovery tests, in which one compares a known mass release to the mass measured in the chamber system, may be used to assess the chamber mixing and inertness as well as sampling and analysis. These tests involve comparing decay rates with known injections of contaminants (ASTM 2006a) or independent weight measurements of an evaporating solvent (ASTM 2001). The European guide for chamber emissions testing criteria for acceptable mass recovery is greater than 80% (ECA 1991).

Such proficiency checks are important for reducing and understanding the uncertainty in emissions test measurements, but are not currently used in a standard manner. For a credible product labeling program, emissions measurements should be validated with standardized proficiency checks. Also, the current proficiency checks do not address all six steps of the emissions testing process, specifically material selection, material preparation or chamber operation. A more complete check of the precision and variability of emissions measurements should include reference materials and inter-laboratory comparisons.

## **METHODS TO IMPROVE EMISSIONS MEASUREMENT RELIABILITY**

Three approaches are suggested to address the measurement reliability of emissions testing of indoor materials and products: 1) standard emissions test methods; 2) proficiency standards for emissions testing including standard reference materials; and 3) a laboratory accreditation program. These approaches are described below.

## **Product Emissions Standard Test Method Development**

Perhaps the most important measurement need is for standard test methods for emission measurements. However, it would be essentially impossible to develop a single test method that applies to the wide range of materials (e.g., both wet and dry), the variety of test facilities and conditions (e.g., microchambers, small chambers, large chambers, etc.), and the hundreds of compounds of interest that vary in concentration, composition, and toxicity (e.g. non-polar VOCs, polar VOCs, SVOCs, etc.). The few existing test methods usually focus on a single product, a single type of test apparatus, and specific chemical(s). For example, ASTM E1333 provides a test method for measuring formaldehyde emission rates from wood products using a large chamber (ASTM 2002b). Another approach is to develop a series of standard practices and test methods for each step of the emissions testing process. Table 1 shows a matrix of the steps in the emissions test process and the factors of material, test apparatus and chemical. The first step of the process includes selecting, packaging, transporting, and storing the material samples after manufacture, which will vary based on the type of material. The next step is preparing the material for testing, which is dependent on both the material and the test facility. For example, a dry flooring material sample is typically cut into smaller pieces with sealed edges, whereas a wet paint is generally applied to a small piece of substrate. Materials (wet or dry) also need to be prepared differently based on the size of the test facility. Each type of test facility is operated differently and may require different environmental settings (air change rate, temperature, relative humidity, etc.). The importance of certain operating conditions will also depend on the type of material being tested. For example, chamber air velocity is much more important for a wet, evaporation-controlled product than a dry, diffusion-controlled material. The method and frequency with which the test facility gas-phase samples are collected is dependent on all three factors. The type of material can dictate the frequency of sampling and duration of the emissions tests. The most appropriate sample collection medium (e.g., silica cartridges, sorbent tubes, etc.) depends on chemical type. The analysis of the gas-phase samples varies based on the structure and physical properties of the chemical sampled and its concentration in the test apparatus. Another important analytical issue is the capability to identify all of the VOCs present. And finally, analysis and interpretation of the data depends on the emission profile generated from sample time and frequency for each chemical and material.

Based on the relationships shown in Table 1, it is possible to formulate individual standard practices and test methods for each step and factor, or where appropriate, a combination of steps and factors. For example, test methods could be developed specifically for measuring the emission rate of non-polar VOCs from wet products tested in small chambers. Another approach is to develop standards for critical measurements used during emissions tests. For example, a test method could

**Table 1. Relationship Between Emissions Test Steps and Other Factors**

Emissions Test Step	Material	Test Apparatus	Chemical
Material Selection	X		
Material Preparation	X	X	
Test Equipment Operation	X	X	
Gas-phase Sampling	X	X	X
Sample Analysis		X	X
Data Analysis/Interpretation	X	X	X

be developed specifically for measuring air velocity in an emissions test apparatus.

**Measurement Validation Standards and Standard Reference Materials**

A second approach to improve measurement reliability is with measurement validation standards. Measurement proficiency checks could be defined in separate standards or included in emission test methods. In either case, the standard checks should cover all points of variability in the emissions testing process from material selection to chemical analysis. Figure 1 shows a flowchart of the emissions testing process and the corresponding proficiency checks at each step in the process. As discussed earlier, mass recovery tests, sampling checks, and analytical system checks are already in use, but these only address half of the emissions testing process. Possible ways to check the accuracy of earlier testing steps include reference materials and statistically robust inter-laboratory comparisons.

A reference material would be a sample with a known emission rate (within a specified uncertainty) that could be used to calibrate test facilities and measurement methods (ISO 1993). Although true performance characteristics cannot be established until the reference material is developed and thoroughly tested, the following list includes material characteristics and performance criteria that will be considered:

- Known reference emission rate and associated uncertainty. The reported emission rate of a reference source will be based on multiple measurement methods, including VOC weight loss and material properties. The reported emission rate for the reference material will be provided with an uncertainty and a level of confidence of approximately 95%. The uncertainty of the emission rate will be based on all measurements used to determine the emission rate. A successful outcome would exist if all of the methods agree within their respective uncertainties.
- Preparation of reference material with reproducible emission rate. To determine variability between reference samples, statistically-based sampling is needed. Material variability results are reflected in the emission rate uncertainty estimate.

- Factors affecting emission rate. The emission rate should be affected by test equipment conditions, and the dependence on chamber conditions should be well understood through statistically robust comparative experimental designs. These results will also help define test method conditions for using the reference material.
- Mass transfer processes. The reference material should have similar mass transfer processes to those of common indoor materials and products. In order to meet this criterion, a range of reference materials with different properties will be needed to represent both “wet” and “dry” materials.
- Material size. The reference material should be available in multiple sizes to fit in different test facilities and still emit a measurable quantity of VOCs.
- Sampling and analytical capabilities. The reference material should emit a range of VOCs to test both sampling and analytical capabilities.
- Stability and storage. This criterion will require extensive testing to determine the duration and environmental conditions (temperature, relative humidity, etc.) of storage to keep material within known emissions rate and uncertainty.

In addition to reference materials, inter-laboratory comparisons are also needed. A reference material alone can only assess laboratory equivalency for a specific condition. To obtain an overall picture of a laboratory’s ability to measure emission rates of any product, any day, in any chamber requires a comparative experimental design. To understand such variability, a Taguchi parameter approach (Taguchi 1996) that includes a primary factor for comparison and other robustness factors is useful. Table 2 shows an example inter-laboratory comparative design where the primary factor is test laboratory and the robustness factors are chamber, operator, material, and loading ratio. The response factor would be the product emission rate of ten different analytes in this example. In the case of six test laboratories and four ‘two-level’ robustness factors, the total number of tests required,  $6 \times 2^4$  or 96, would generate 960 results for ten analytes. The number of tests for each lab would be  $2^4$  or 16. It is possible to reduce the number of required tests by using a fractional factorial design.

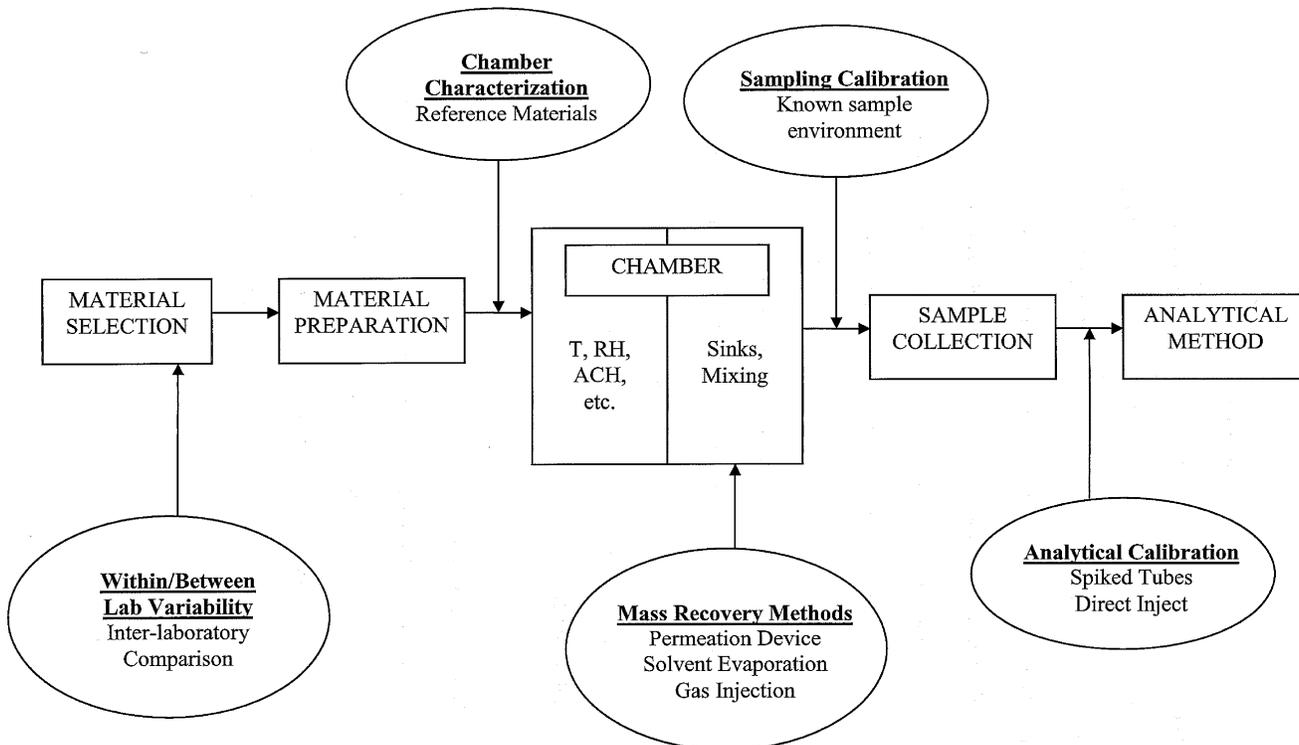


Figure 1 Emissions testing proficiency checks.

Table 2. Example Comparative Experimental Design for Laboratory Round Robin

Factor	Levels
X1: Laboratory (primary factor)	Lab 1, 2, 3, 4, 5, 6
X2: Chamber	Chamber A, Chamber B
X3: Operator	Operator A, Operator B
X4: Material	Sample A, Sample B
X5: Loading Ratio	High, Low

A fractional design consists of a subset of tests that still provides information regarding main effects but not multi-level interactions (Box et al. 1978). A properly designed factorial analysis could answer a number of key questions including 1) Are the emission measurements taken across all labs equivalent? 2) Which labs are different? 3) What is the within-laboratory performance?

### Laboratory Accreditation

Another way to improve the reliability of emissions test results is with a formal laboratory accreditation program. Currently, there are several laboratory accreditation bodies, e.g., the National Voluntary Laboratory Accreditation Program (NVLAP)(NVLAP 2006). In general, these accreditation bodies follow ISO standards (ISO/IEC 2005; ISO 2003) for evaluating the technical competence of a laboratory. Technical assessors conduct an evaluation of all factors in a laboratory that affect the generation of test data, including:

technical competency of staff; validity and appropriateness of test methods; traceability of measurements and calibrations to national standards; suitability, calibration and maintenance of test equipment; testing environment; sampling, handling and transportation of test items; and quality assurance of test and calibration data (ILAC 2007). Often, there is an initial cost to a testing laboratory to join the accreditation program, followed by a periodic renewal fee. Despite these costs, test laboratories choose to be accredited for the marketing advantage as well as international recognition. Laboratory accreditation also provides manufacturers more confidence that their products are being tested correctly and accurately.

### REFERENCE MATERIAL DEVELOPMENT AND INITIAL RESULTS

In the context of the three approaches discussed above for improving the reliability of emissions testing, Virginia Tech and NIST are working on the development of reference mate-

rials for calibration of emissions test equipment. As mentioned earlier, it is anticipated that a range of reference materials will be required to adequately check the many types of materials, test equipment, and chemicals. However, this paper introduces the concept developed by Virginia Tech to manufacture a reference material that could be used to calibrate the emissions process for a dry, diffusion-controlled source.

A dry material consists of a matrix interspersed with low concentrations of VOCs that result from the material manufacturing process. To mimic such a material, Virginia Tech has developed a method using supercritical carbon dioxide (CO<sub>2</sub>) to dissolve a specific VOC in a simple polymer matrix. The initial polymer being used is poly(ethyl methacrylate) (PEMA), which is inexpensive and has a low glass transition temperature that facilitates swelling in the presence of supercritical CO<sub>2</sub> at low temperatures. The base polymer is created with a solution of 10% PEMA (by weight) in chloroform. Once completely dissolved, the PEMA solution is cast onto a glass plate and covered to ensure a slow evaporation rate of the solvent, which in turn prevents bubble formation in the film. After the polymer has hardened, it is placed in a vacuum oven for several hours at 60 °C to ensure complete removal of any remaining solvent.

To “charge” the polymer with a VOC, the polymer film is sealed in a pressure-vessel containing liquid VOC at the bottom. Once the vessel is sealed, some of the liquid vaporizes and comes into contact with the polymer film. The pressure of the system is raised to 3400 kPa using a high pressure CO<sub>2</sub> cylinder and a pressure screw as shown in Figure 2.

Once the system reaches the desired pressure, it is held constant for several minutes to allow the CO<sub>2</sub> and VOC vapor to enter the swollen polymer. After equilibrium with the polymer has been reached, the pressure is slowly lowered over several minutes and the sample is removed for analysis.

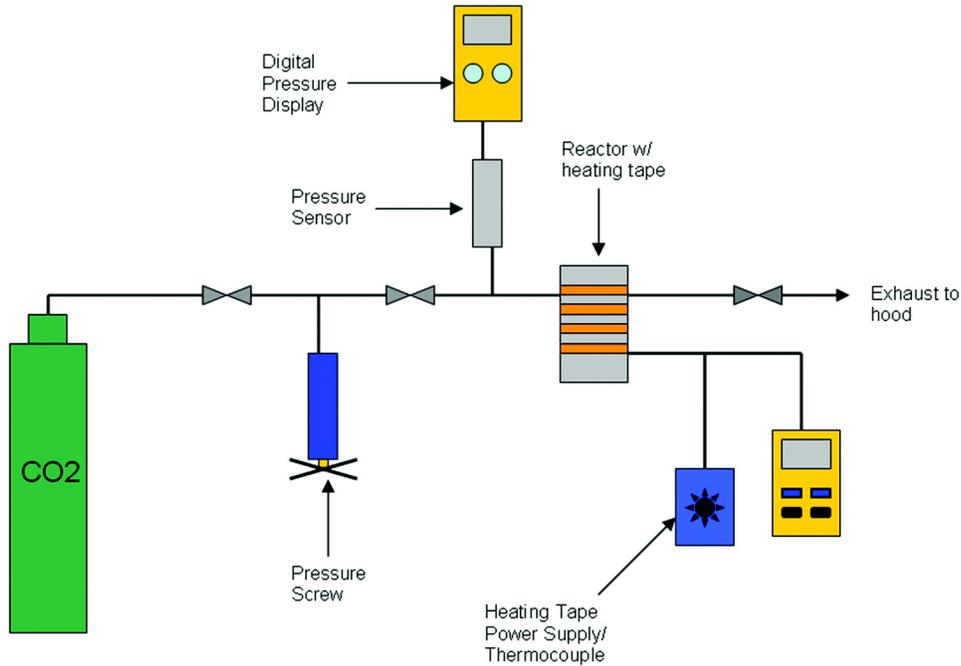
Initial polymer samples have been created and tests are underway to characterize the mass desorption profile of the sample. To determine the mass loss over time, the polymer sample is placed in a microbalance and weighed. Weight measurements are taken every few minutes over a period of several days or until the change in mass has leveled out. During the course of measurement, a constant flow of dry nitrogen gas is passed over the surface of the polymer. This flow reduces the concentration gradient close to the surface that might be caused by stagnant air and also prevents humidity or other contamination from interfering with the desorption process. Figure 3 shows the normalized mass (defined as Measured Mass - Final Mass) / (Initial Mass - Final Mass) as a function of time for a PEMA sample that has been pressurized with CO<sub>2</sub> and charged with toluene. As shown in Figure 3, the loss of mass from the toluene-charged PEMA occurs over a period of about two days. Initial results also suggest that the loaded polymer is behaving as a diffusion-controlled source. As shown in Figure 3, a simple Fickian desorption model (Cox et al. 2001b) provides a good fit to the experimentally measured normalized mass curve.

The emission rate of a VOC from a polymer film can be measured three different ways. First, it is possible to determine a single-VOC film’s emission rate by measuring its change in mass as a function of time using a microbalance as described above. It is also possible to use a physically-based diffusion model to predict the emission of a single VOC or multiple VOCs from dry materials (e.g., Little et al. (1994)). The three principal model parameters are the initial material-phase concentration of the VOC (C<sub>0</sub>), the material/air partition coefficient (K), and the material-phase diffusion coefficient (D). A more detailed description of these parameters and how they are measured is provided elsewhere (Cox et al. 2001a; Cox et al. 2001b). Using these independently measured parameters and the model, which has been validated for three different VOCs emitted from vinyl flooring (Cox et al., 2002), it is possible to predict the emission rate of a dry reference material, assuming that the VOC and material properties are consistent with the model assumptions. These two independently determined emission rates could then be compared to the third way to estimate a material’s emission rate, which is with a test chamber or other type of test apparatus. The emission rate determined by each method should agree within the uncertainty range of the various methods.

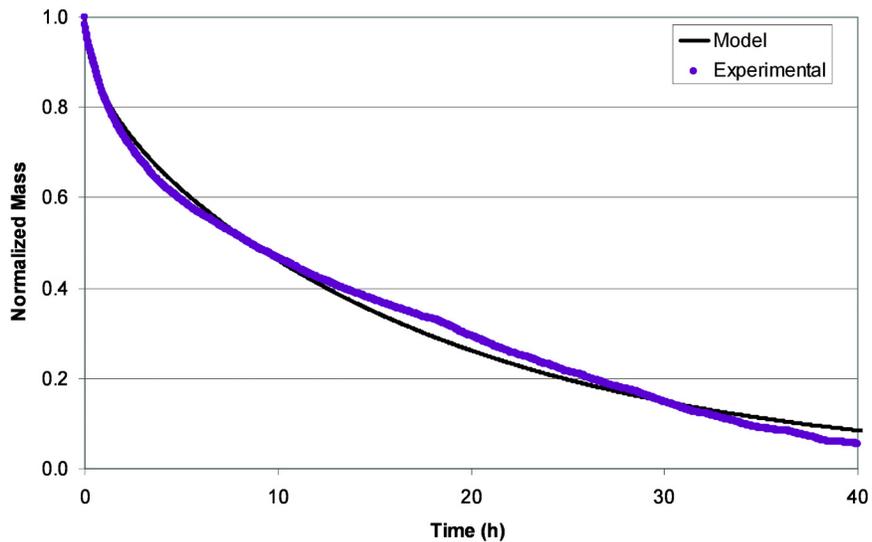
The next steps in the process of developing a reference material for emissions testing include: assessing the repeatability of the polymer manufacturing process, measuring the reference polymer’s VOC emission rate in NIST’s 0.053 m<sup>3</sup> stainless steel chambers, and conducting a statistically robust inter-laboratory comparison with several testing laboratories. If this dry reference material is successful, the next steps will include expanding to more material types, test equipment, and a greater range of chemicals.

## CONCLUSIONS

For emissions assessment programs to achieve maximum impact, the associated measurements of product emissions characteristics have to be deemed reliable. Currently, that is not the case for product and material emissions assessment. There are several steps, however, that the emissions testing community can take to significantly improve the reliability of their measurements. First, and perhaps most importantly, a consensus needs to be reached on standard test methods to generate consistent and comparable emission rate results. Since a single comprehensive test method will be difficult to establish, a series of standards with a smaller scope may be preferable. In addition to test methods, standard proficiency checks with reference materials and statistically robust inter-laboratory comparisons should be developed to validate the entire emissions testing process. Reference materials have the potential to build consensus and confidence in emissions testing as well as “level the playing field” for product testing laboratories and manufacturers. Based on preliminary results, it appears that it will be possible to manufacture a dry diffusion-controlled material that can be used to calibrate small chambers. Finally, standard methods and measurement validation



**Figure 2** Diagram of polymer charging system.



**Figure 3** A simple Fickian diffusion model is used to fit the normalized mass loss from the polymer sample charged with toluene.

are key parts of a formal laboratory accreditation program that could be used to maintain the reliability of the measurements. Improving measurement reliability will require significant investment from manufacturers and testing laboratories. But with a scientifically sound testing process in place, there is the potential for substantial growth in the sustainable building products market.

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