Inter-laboratory study approach to validate the performance of a prototype reference material for product emissions testing

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1 Introduction

Product emissions chamber testing involves a complex protocol with several sources of measurement uncertainty (ASTM, 2010; ISO, 2006). Despite the potential for significant variability in measurement results within and among laboratories, there is no standard method available to assess the accuracy of emissions chamber test results. Recently, Germany's Federal Institute for Materials Research and Testing (BAM) started a program to evaluate the performance of laboratories testing for the AgBB (German Committee for Health-related Evaluation of Building Products) labelling scheme (Wilke et al., 2009). Test method validation was conducted through inter-laboratory studies (ILS) with over 30 participants. Simultaneously, the National Institute of Standards and Technology (NIST) and Virginia Tech (VT) started a program to develop a series of reference materials that mimic real building products, can be tested in typical emissions chambers, and have independently known emission rates (Cox et al., 2010). BAM, NIST and VT subsequently joined forces to conduct a series of inter-laboratory studies using the prototype reference material. A pilot ILS was conducted with BAM and NIST followed by an expanded ILS with several participating countries. Results from these inter-laboratory studies have the potential to define an internationally-accepted standard approach to validate results from product emissions tests.

2 Materials/Methods

The development of a standard emissions test method validation protocol is being pursued through a series of inter-laboratory studies using a prototype reference material. The prototype material consists of a polymethyl pentene (PMP) film loaded to equilibrium with toluene (Cox et al., 2010). The film's material and chemical properties have been well-characterized such that it is possible to predict its emission rate with a fundamental mass transfer model (Cox et al., 2010). The film's emission rate has also been measured several times in NIST environmental chambers and in a recent pilot ILS (Howard-Reed et al., 2011).

Each ILS has been designed to send laboratory participants multiple reference films made in the same batch, with the participants measuring the toluene emission rate in small environmental chambers with set points of 23 °C, 50 % RH, and 0.065 m^3/h airflow rate. Sample collection times at 1 h, 2 h, 4 h, 7 h, 24 h, 27 h, 30 h, 48 h, 51 h, and 54 h were specified. However, the sample collection method and analysis was not prescribed. In general, laboratories tended to use similar sample collection and analytical techniques including sorbent sample tubes, thermal desorption and gas chromatography-mass spectrometry.

3 Results

To date, results for the pilot ILS with BAM and NIST are available (see Figure 1). The uncertainty for each point was determined based on propagation of errors associated with the air sample volume and analytical calibration parameters required to determine the chamber concentration of toluene. NIST ran all three samples in a single chamber, whereas BAM used two chambers to run the samples. BAM also used two thermal desorption systems to

analyze chamber air samples and an internal standard. An effort was made to run some samples at the same starting age (7 days and 36 days after sample manufacture).

In general, there was good agreement between the toluene concentrations measured for each reference sample. A summary of the relative standard deviations calculated for selected sampling time points within each laboratory and between laboratories is provided in Table 1.

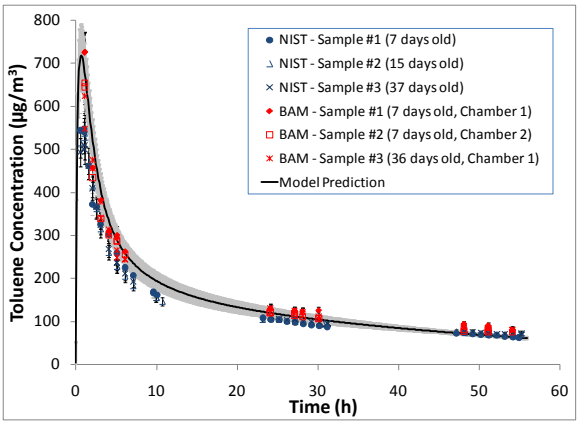

Figure 1. Toluene concentrations for BAM and NIST chambers.

Table 1. Relative Standard Deviations (%)

Sample Time	Within Labs BAM	Within Labs NIST	Between Labs*
2 h	3.5	5.8	8.0
6 h	3.5	4.8	10
24 h	3.2	3.7	8.7
48 h	7.5	2.0	9.0

*Based on emission rate to correct for slightly different airflow rates between laboratories.

Figure 1 also shows a comparison of the measured toluene concentrations to those predicted by the mass transfer model. The measured results were within the uncertainty range of the model for both labs except the initial 4 h for NIST and the final 4 h for BAM. In fact, the average toluene concentration measured at a sampling time of 24 h across both laboratories was within 1 % of the toluene concentration predicted by the model.

4 Conclusions

It is not necessary that every laboratory use the same type of chamber or analytical equipment to measure the emissions of building materials and products. However, every laboratory must demonstrate that its chosen testing equipment and methods can measure product emission rates within an acceptable uncertainty (Zimmerman Jr., 2010). Results from this study show that method validation can be achieved by multiple laboratories using a homogenous emissions source with established pre-determined emission rate. Based on the pilot ILS results presented here and elsewhere, it is possible for test laboratories to measure concentrations within 10 % of a reference concentration value, providing confidence in their ability to accurately measure product emission rates. This level of accuracy will be further evaluated in the next ILS involving more than 10 laboratories. Future efforts will also include reference films loaded with other chemicals beyond toluene and reference materials that represent wet sources.

5 References

ASTM. 2010. *ASTM Standard D5116-2010*, Standard Guide for Small-scale Environmental Chamber Determination of Organic Emissions from Indoor Materials/Products. West Conshohocken, PA: ASTM International.

Cox, S.S, Liu, Z, Little, J.C, Howard-Reed, C, Nabinger, S.J, and Persily, A. 2010. Diffusion-controlled reference material for VOC emissions testing: proof of concept. *Indoor Air*, 20, 424-433.

Howard-Reed, C, Liu, Z, Benning, J, Cox, S, Samarov, D, Leber, D, Hodgson, A.T, Mason, S, Won, D, and Little, J.C. 2011. Diffusion-controlled reference material for volatile organic compound emissions testing: pilot inter-laboratory study. Submitted to *Building and Environment*.

ISO. 2006. *ISO Standard 16000-9:2006*, Determination of the emission of volatile organic compounds from building products and furnishing – Emission test chamber method. Switzerland: International Organization for Standardization.

Wilke, O, Horn, W, Wiegner, K, Jann, O, Bremser, W, Brödner, D, Kalus, S, Juritsch, R, and Till, C. 2009. Investigations for the improvement of the measurement of volatile organic compounds from floor coverings within the health-related evaluation of construction products. Final Report for BAM: Federal Institute for Materials Research and Testing, Berlin, Germany.

Zimmerman Jr., R.L. 2010. Ten best practices of good laboratories. *ASTM Standardization News*, November/December, 25 – 31.