



Project Summary

Indoor Air Sources: Using Small Environmental Test Chambers to Characterize Organic Emissions from Indoor Materials and Products

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This report describes methods and procedures for determining organic emission rates from indoor materials/products using small environmental test chambers. The techniques presented are useful for both routine product testing by manufacturers and testing laboratories and for more rigorous evaluation by indoor air quality researchers.

This Project Summary was developed by EPA's Air and Energy Engineering Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The use of small environmental test chambers to develop emission characteristics of indoor materials and products is still evolving. Modifications and variations in equipment, testing procedures, and data analysis are made as the work in the area progresses. Until the interested parties agree upon standard testing protocols, differences in approach will occur. The purpose of this report is to provide assistance by describing equipment and techniques suitable for determining organic emissions from indoor materials. Specific examples are provided to illustrate existing approaches; these examples are not intended to inhibit alternative

approaches or techniques. The techniques described are useful for both routine product testing by manufacturers and testing laboratories and for more rigorous evaluation by Indoor Air Quality (IAQ) researchers.

The use of small chambers to evaluate organic emissions from indoor materials has several objectives:

- developing techniques for screening products for organic emissions;
- determining the effect of environmental variables (i.e., temperature, humidity, air exchange) on emission rates;
- ranking products and product types with respect to their emissions profiles (e.g., emission factors, specific organic compounds emitted);
- providing compound-specific data on various organic sources to guide field studies and assist in evaluating IAQ in buildings;
- providing emissions data for the development and verification of models used to predict indoor concentrations of organic compounds; and
- developing data useful to manufacturers and builders for assessing product emissions and developing control options or improved products.

It is emphasized that small chamber evaluations are used to determine source emission rates. These rates are then used in appropriate IAQ models to predict indoor concentration of the compounds emitted from the tested

material. The concentrations observed in the chambers should not be used as a substitute for concentrations expected in full-scale indoor environments.

Facilities and Equipment

A facility designed and operated to determine organic emission rates from building materials and consumer products found indoors should contain the following: test chambers, clean air generation system, monitoring and control systems, sample collection and analysis equipment, and standards generation and calibration systems.

Small environmental test chambers are designed to permit the testing of samples of various types of building materials and consumer products. They can range in size from a few liters to 5 m³. Generally, chambers of more than 5 m³ are considered "large". Large chambers permit the testing of complete assemblages (e.g., furniture); they may also be used to evaluate activities (e.g., spray painting). For the purpose of this guide, small chambers are assumed to be used to test samples of larger materials and products, as opposed to full scale materials or processes.

The test chambers should have non-adsorbent, chemically inert, smooth interior surfaces. Care must be taken in their construction to avoid the use of caulks and adhesives that emit or adsorb volatile organic compounds. Electro-polished stainless steel and glass are common interior surfaces. The chamber must have an access door with airtight, non-adsorbent seals. The chambers must be fitted with inlet and outlet ports for air flow. Ports for temperature and humidity probes may also be required. Ports for sample collection are needed only if the sampling is not conducted in the outlet air.

The chambers should be designed to ensure adequate mixing of the chamber air. Low speed mixing fans or multi-port inlet and outlet diffusers are two techniques that have been used successfully.

Clean air must be generated and delivered to the chambers. A typical clean air system might use an oilless compressor drawing in ambient air followed by removal of moisture (e.g., using a membrane dryer) and trace organics (e.g., by catalytic oxidation units). Other options include gas cylinders or charcoal filtered outdoor or laboratory air. The amount of air flow required should be calculated before a decision is reached on the supply system. The required purity of the air

must also be determined based on the type of samples to be evaluated.

Measurement and control are required for air flow, temperature, and humidity. Air flow can be automatically monitored and controlled by electronic mass flow controllers, or manual flow control (e.g., needle valve, orifice plate) and measurement (e.g., bubble meter, rotometer) can be used. Temperature can be measured automatically using thermocouples or thermistors; manual dial or stem thermometers can also be used. Control of humidity depends on the humidification system employed. If liquid injection is used, water flow is controlled by the pump setting. Control of humidity by saturated air requires temperature control of the water and flow control of the saturated air stream. Humidity can be measured using several types of sensors, including dew point detectors and thin-film capacitors. Temperature and humidity sensors should be located inside the chamber at least 5 cm from the inside wall and near the midpoint between the air inlet and outlet ports.

Sample Collection and Analysis

Indoor sources of organic emissions vary widely in both the strength of their emissions and the type and number of compounds emitted. To fully characterize organic emissions, the sample collection/analysis system must be capable of quantitative collection and analysis of volatile, semivolatile, polar, and non-polar compounds. Any small chamber sampling and analysis technique or strategy developed must consider the emission characteristics of the specific source being evaluated. The design and operation of sample collection and analysis systems must be appropriate for the organic compounds (and their concentrations) being sampled. Such systems generally include sampling devices (e.g., syringes, pumps), sample collectors (e.g., syringes, adsorbent media, evacuated canisters), and instruments to analyze organic emissions (e.g., gas chromatographs [GCs]).

Experimental Design

The first step in designing an experiment for chamber tests of indoor materials/products is to determine the test objectives. For example, a builder or architect would be interested in emissions from a variety of materials to be used under a given set of conditions for a specific building. In this case, the experiment would be designed to handle many materials with one set of

environmental conditions. A manufacturer might want to know the emissions, characteristics of a single product under both normal and extreme conditions and would design a test to cover the appropriate range of environmental variables. IAQ researchers interested in the interactions among variables would use a more complex design involving ranges of several variables.

A basic experimental design for small chamber tests should include consideration of the effects of various parameters on the emission characteristics of the materials to be tested. Five variables are generally considered to be critical parameters: temperature, humidity, air exchange rate, product loading, and time (or product age).

For each material tested, a test matrix is developed to allow the variables of interest to be investigated. As is normal in experimental programs of this type, the desire to collect data over an extensive parameter range is limited by cost and time constraints. To maximize the information production within available resources, a statistical consultant can be used to provide guidance on appropriate experimental designs.

Experimental Procedures

A preliminary evaluation of the product/material is performed to guide selection of appropriate test strategies and analytical techniques. This evaluation is conducted to obtain information on the specific compounds to be quantified. Only a single compound is to be quantified, selection of the appropriate sampling and analysis strategy is straightforward, and no further screening is needed. When a more complete characterization is desired, more information is required. The composition of the emissions expected from a source can be evaluated initially by surveying available information, including: a) reports or papers on previous studies of the source, b) ingredients listed on the product label, c) Material Safety Data Sheets, and d) information obtained from the manufacturer or appropriate tracing organizations. Such information is usually insufficient to identify the compounds of interest, but it does provide some guidance in what compounds to look for. Another problem is that the compounds emitted from the source may be formed during the use of the product or material and will not be listed as ingredients. Therefore, further analyses are required and testing must be conducted to determine the actual compounds being

emitted. One technique involves headspace analysis of the source emissions.

Headspace Analysis

The process of identifying the organic compounds present in the "headspace" or air above the material is termed "headspace analysis." Both static (i.e., closed container) and flow-through headspace analyses are used. Headspace components are usually identified by gas chromatography coupled with a mass selective detector (GC/MS) operated in the scan mode, although other detectors can be used if sufficient information is available on the retention times for all compounds of interest for a given GC column, gas flow, and temperature program.

While the headspace analysis provides useful information on the direct emissions from the material or product of interest, it does not ensure that all emissions will be identified. Compounds not found in the headspace may be emitted later due to being formed in the drying process or by interactions with the substrate.

Chamber Testing

Chamber testing requires a preparation phase as well as a testing phase. The preparation stage begins with development of the test plan that specifies environmental conditions for each test, method of application of the material, conditioning period and methods of sample collection and analysis. Development of the test plan is followed by calibration of environmental control and measurement systems, sample collection and concentration devices, and analytical systems as specified in the Quality Assurance (QA) Plan. At this stage the information from the GC/MS headspace analysis is evaluated to provide guidance in selection of analytical columns and detectors, sample collection media and an appropriate internal standard.

Prior to actual testing, chambers are cleaned and placed in position in the temperature controlled environment and purged at test conditions. Chamber background is monitored to ensure that background contamination is within QA limits. At this point, the chamber conditions are at test setpoints of flow and relative humidity, all analytical systems have been calibrated, the quality control system has been developed, and an internal standard has been selected. A chamber background sample is then taken to quantify any contribution of

organic compounds from the clean air system and/or the empty chamber. In addition, any substrate materials, such as wood, that will be used during the tests must be included to account for actual background. Once all the preparatory steps have been completed, testing of the selected material/product can commence.

The types of test specimens used in the chambers vary according to the material or product being tested. Solid materials are tested "as is". If emissions from edges may differ from the normally exposed surface, the edges should be sealed. For example, particleboard specimens can have their edges sealed with sodium silicate to eliminate the excessively high edge emissions previously reported. "Wet" materials are applied to a solid substrate. For example, a wood stain would be applied to a board; a vinyl floor wax to floor tile. As noted above, the uncoated substrate should be placed in the chamber during background tests to determine the magnitude of its organic emissions. Also edge effects should be eliminated by edge sealing. Wet materials are applied to the substrate outside the chamber and placed in the chamber shortly thereafter. The start of the test (time = 0) is set when the door to the chamber is closed. Small chambers are not suitable for evaluating the application phase of wet material use. Thus, emissions from the earliest portion of the drying cycle (i.e., from application until placement in the chamber) will not be measured. The time between application and the start of the test should be less than ten minutes; the time of application and the test start time should both be recorded.

In some cases, emissions data are desired on later stages of a material/product life-cycle (e.g., several months after a coating has been applied). In these cases, the specimen must be conditioned prior to testing. Conditioning should occur under the same environmental parameters (temperature, humidity, air exchange rate, and product loading) as those used for chamber tests. If this is not possible, the conditioning environmental parameters should be well documented. Ideally, the sample should be conditioned over its complete life cycle up to the time of testing. If this is not possible, conditioning should be conducted for a period of time sufficient to allow the emissions to equilibrate to the test conditions (e.g., one to two weeks).

Care should be taken in testing materials which have been used or stored

with other materials. In such cases, the material of interest could have acted as a "sink" and adsorbed organics from the other materials. Subsequent testing could provide emissions data which represent the re-emission of the adsorbed compounds rather than emissions from the original material.

Collection of a representative sample of chamber effluent requires the use of a sampling strategy that is appropriate to the ranges of volatilities of the compounds present. The information obtained from the GC/MS headspace analysis can be used to select appropriate sample collection and concentration media. As discussed above, the sampling method can range from syringe/pump sampling to adsorption on various sorbent media.

Sampling techniques must also be appropriate to the concentrations of compounds in the chamber air stream. When testing wet materials such as glues, waxes, and wood finishes, chamber concentrations may change by orders of magnitude over a period of minutes. Accurate description of chamber concentration with time may require sampling very frequently or use of a continuous or semi-continuous monitor. A combination of both techniques is the most effective way to characterize rapidly changing emissions. The concentration of individual compounds varies as the material ages. In some cases, compounds not detected in the headspace or in the first few hours of testing may become the major emission component. Therefore, a total hydrocarbon monitor can be effective in tracking rapidly changing concentrations but may provide an incomplete qualitative picture.

It is important, therefore, to monitor changes in the emission profile as the material dries. The sampling strategy should provide a means to collect approximately the same mass in each sample. Thus, the sample volume is an important consideration. When chamber concentrations are high, sample volume must be kept low to avoid breakthrough in the collection trap or overloading of the concentrator column of a purge and trap device. Sample volumes of less than one L can be drawn directly by gastight syringes, then injected through a heated port to a clean air stream flowing through sampling cartridges. Much smaller samples (e.g., 1 cc) can be injected directly into the GC. Larger volume samples are taken by pulling chamber air stream through sample cartridges as described above. Since the flow through

the cartridges is constant, increasing the sampling time will increase the sample volume. It may be necessary to conduct trial runs to develop a sampling strategy.

The analysis technique depends on the sampling strategy and adsorbent media employed. Methods of introducing the sample to the GC include direct injection, thermal desorption followed by purge and trap concentration, and solvent extraction followed by liquid injection.

Data Analysis

Data reduction and analysis is a multistep process. Electronic spreadsheets can be used to reduce and compile the environmental and chemical analysis data with minimal data entry steps. Chamber concentration data are used in various models to produce estimates of material/product emission rates.

Environmental and GC Data

Environmental data (i.e., temperature, relative humidity, flow rate) can be recorded manually or automatically stored (e.g., on floppy disks) by a PC-based system. GCs (including GC/MS) are interfaced to computing integrators (or PC-based chromatographic data analysis systems) for plotting of the chromatograms and computation of the

areas of peaks obtained. The data output is printed on paper as an analog chromatogram plus a summary report. The data can also be stored on magnetic media for future review or reprocessing.

The environmental information and the GC analysis results are combined to give chamber concentrations for individual compounds and total organics. Chamber concentration data coupled with sample size and chamber air exchange rate are then used to estimate emission factors.

Emission Factors

Emission factors for organics from indoor materials are usually expressed in terms of mass/area-time. In some cases, emission factors are reported as mass/mass-time, or, in the case of caulk beads, mass/length-time. They are calculated for individual organic compounds, as well as for total measured organics. The method for calculating the emission factor depends on the type of source being tested.

For materials with a relatively constant emission rate over the test period, the chamber concentration will reach and maintain a constant equilibrium value. For such materials the calculation of the emission factor, when sinks are ignored, is straightforward:

$$EF = C(Q/A) \quad (1)$$

where, EF = Emission factor, mg/m²-hr

C = Equilibrium chamber concentration, mg/m³

Q = Flow through chamber, m³/hr

A = Sample area, m²

For sources that have decreasing emission rates over the test period, a different procedure is required. This method (described in detail in the full report) applies to sources with initially high emission rates that decrease with time. Most "wet" sources exhibit such behavior. Equation 2 describes the rate of change in emission factor as a first order reaction:

$$R = R_0 e^{-kt} \quad (2)$$

where, R₀ = Initial emission factor, mg/m²-hr

k = First order rate constant, hr⁻¹

t = Time, hr

It is emphasized that these methods for determining emission factors are not applicable to sources that do not exhibit either constant or simple exponential decay emissions over time, and other emission models may be required.

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The complete report, entitled "Indoor Air Sources: Using Small Environmental Test Chambers to Characterize Organic Emissions from Indoor Materials and Products," (Order No. PB 90-110 131/AS; Cost: \$15.00, subject to change) will be available only from:

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