EUROPEAN CONCERTED ACTION

INDOOR AIR QUALITY & ITS IMPACT ON MAN

COST Project 613

Environment and Quality of Life

Report No. 8

Guideline for the Characterization of Volatile Organic Compounds Emitted from Indoor Materials and Products Using Small Test Chambers

Commission of the European Communities
Directorate General for Science, Research and Development
Joint Research Centre - Environment Institute

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prepared by **Working Group 8**

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- Report No. 1: Radon in indoor air.
- Report No. 2: Formaldehyde emissions from wood based materials: guideline for the establishment of steady state concentrations in test chambers.
- Report No. 3: Indoor Pollution by NO_2 in European countries.
- Report No. 4: Sick building syndrome - a pratical guide.
- Report No. 5: Project inventory.
- Report No. 6: Strategy for sampling chemical substances in indoor air.
- Report No. 7: Indoor air pollution by formaldehyde in European Countries.

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SECTION 1

INTRODUCTION

A. SCOPE

The purpose of this report is to describe the methods and procedures for determining emission rates of volatile organic compounds (VOC) from indoor materials / products using small environmental test chambers. 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 importance for human health of VOC in indoor environments is explained in a report by WHO (WHO 1989), where also a definition of VOC, based on volatility, is given.

The report does not provide specific guidance for determining emissions of formaldehyde from pressed wood products, since this has been the subject of a specific guideline (COST Project 613, 1989). It is possible, however, that the guide could be used to support alternative testing methods.

While the ultimate purpose of any evaluation of indoor materials is to determine whether the emissions can contribute to health or comfort problems, this report is applicable only to the determination of the emissions themselves. The effect of the emissions (e.g., toxicity) is beyond the scope of the report.

Small environmental test chambers (see Section 2A for volume range) are the preferred tool for the qualitative and quantitative characterization of the vapours and gases emitted from various materials and products. They have the advantage of being limited in size, cheaper and easier to operate than large chambers. However they are inappropriate for testing combustion devices or large items (e.g. furniture) which require large chambers; also they could be regarded as too large and costly, in cases where microchambers (see Section 5C) are sufficient. 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.

A validation of this guideline through an interlaboratory comparison has been initiated: the results of this experiment will be reported.

B. TESTING OBJECTIVES

The use of small chambers to evaluate the emission of organic compounds from indoor materials has several objectives:

- develop techniques for screening of products to identify significant emitters of organic compounds; in particular a toxicological evaluation of the qualitative chamber measurements could be used to select for which compounds a quantitative measurement of emission rate is required;

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

C. MASS TRANSFER CONSIDERATIONS

Small chamber evaluation of emissions from indoor materials requires consideration of the relevant mass transfer processes. Three fundamental processes control the rate of emission of organic vapors from indoor materials: 1) diffusion within the material, 2) desorption of adsorbed compounds, 3) evaporative mass transfer from the surface of the material to the overlying air.

1) Diffusion within the material

The diffusion mass transfer within the material is a function of the diffusion coefficient (or diffusivity) of the specific compound. The diffusion coefficient of a given compound within a given material is a function of the compound's physical properties (e.g., molecular weight, size), temperature, and the structure of the material within which the diffusion is occurring. The diffusivity of an individual compound in a mixture is also affected by the composition of the mixture and by any inhomogeneity in the material itself.

2) Desorption

The desorption rate of compounds adsorbed on materials can be determined by the retention time (or average residence time) of an adsorbed molecule (Levine, 1978):

$$\tau = \tau_o e^{-Q/RT} \qquad (1)$$

Where, τ = Retention time, s
τ_o = Constant with a typical value from 10^{-12} to 10^{-15} s
Q = Molar enthalpy change for adsorption or adsorption energy, J mol^{-1}
R = Gas constant, 8,317 J mol^{-1}K^{-1}
T = Temperature, K

The larger the retention time, the slower the rate of desorption.

3) Evaporative mass transfer

The evaporative mass transfer of a given organic compound from the surface of the material to the overlying air can be expressed as:

$$E = k_m (VP_s - VP_a) \quad (2)$$

Where, E = Emission rate, mg h^{-1}
k_m = Mass transfer coefficient, mg $(hPa)^{-1}h^{-1}$
VP_s = Vapor pressure at the surface of the material, hPa
VP_a = Vapor pressure in the air above the surface, hPa

Thus, the emission rate is proportional to the difference in vapor pressure between the surface and the overlying air. Since the vapor pressure is directly related to the concentration, the emission rate is proportional to the difference in concentration between the surface and the overlying air. The mass transfer coefficient is a function of the diffusion coefficient (in air) for the specific compound of interest, the level of turbulence in the boundary layer above the surface of the material and the thickness of the boundary layer.

Variables affecting mass transfer

While a detailed discussion of mass transfer theory is beyond the scope of this guide, it is necessary to examine the critical variables affecting mass transfer within the context of small chamber testing:

Temperature affects the vapor pressure, desorption rate, and diffusion coefficients of the organic compounds. Thus temperature affects both the mass transfer from the surface (whether by evaporation or desorption) and the diffusion mass transfer within the material. Increases in temperature cause increases in the emissions due to all three mass transfer processes.

Air exchange rate (in h^{-1} units) is defined as the volume of outdoor air that enters the indoor environment in 1 hour divided by the volume of the indoor space. The air exchange rate indicates the amount of dilution and flushing that occurs in indoor environments. The higher the air exchange rate, the greater the dilution and the lower the concentration. If the concentration at the surface is unchanged, a lower concentration in the air increases the evaporative mass transfer by increasing the difference in concentration between the surface and the overlying air. However, this effect can be negligable if the actual air concentration is far from the vapour pressure equilibrium concentration.

Air velocity. The mass transfer coefficient (k_m) is affected by the air velocity in the boundary layer above the surface and the level of turbulence. Generally, the higher the velocity and the higher the level of turbulence, the greater the mass transfer coefficient. In a practical sense, above a certain velocity and level of turbulence, the resistance to mass transfer in the boundary layer is minimized (i.e., the mass transfer coefficient reaches its maximum value). In chamber testing, some investigators prefer to use velocities high enough to minimize the mass transfer resistance at the surface. For example, air velocities of 0.3 to 0.5 m s^{-1} have been used in evaluating formaldehyde emissions from wood products. Such velocities are higher than those observed in normal residential environments by

Matthews, et al. (1987), where in six houses they observed velocities with a mean of 0.07 m s^{-1} and a median of 0.05 m s^{-1}. Thus, other investigators prefer to keep the velocities in a range normally found indoors. However, if the chamber is not designed to have a laminar flow, it is difficult to realize reproducible low air velocities. In either case, an understanding of the effect of velocity on the emission rate is needed in interpreting small chamber emission data.

Humidity has an effect on water soluble gases and vapours so as to act as a transport medium. It is probable that humidity has an effect also on the transportation of non-polar gases and vapours.

D. USE OF THE RESULTS

Small chamber tests 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 themselves should not be used as a substitute for concentrations expected in full-scale indoor environments, because, even if product loading (see Section 4) and air exchange rate can be given realistic values, the (sorption) affect of walls, floor, ceiling and furniture is not given in the test chamber.

SECTION 2

FACILITIES AND EQUIPMENT

A facility designed and operated to determine organic emission rates from building materials and consumer products for indoor use should consist of the following: test chamber(s), a clean air generation system, monitoring and control systems, sample collection and analysis equipment, and standards generation and calibration systems. Figure 1 is a schematic diagram showing an example of such a facility.

A. ENVIRONMENTAL TEST CHAMBERS

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 roughly a few liters to 1 m^3. Generally, chambers of size between 1 and 10m^3 are of little advantage and chambers of more than 10 m^3 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 sub-samples of materials and products, as opposed to full scale materials or processes. A short description of "microchambers", i.e. chambers of the smallest size (milliliters), in which generally air velocity and air exchange rate cannot be regulated independently, is reported in Section 5. A review of a number of small chambers, their characteristics and use has been made by Gustafsson et al. (1991).

Chamber construction

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. Electropolished stainless steel and glass are common interior surfaces. The chamber must have an opening with airtight, non-adsorbent seals. The chambers must be fitted with inlet and outlet ports for air flow. Ports for temperature, humidity and anemometer probes may also be required. Ports for sample collection are needed only if the sampling is not conducted in the outlet air; sampling in the outlet is particularly beneficial in the case of tests carried out at low air velocity.

Internal mixing and leaks

The chambers should be designed to ensure adequate mixing of the chamber air. Low speed mixing fans (radial ventilators, see below "Surface velocity") or multi-port inlet and outlet diffusers are two techniques that have been used successfully. One approach for determining if the chamber air is adequately mixed is to blend an inert tracer gas (SF_6 preferred) with the inlet air at constant concentration and flow and measure the concentration in the chamber outlet over time. The chamber concentration vs. time plot is then compared to the theoretical curve for a completely mixed chamber:

$$c = c_0 (1 - e^{-Nt}) \quad (3)$$

Where, c = Chamber concentration, mg m^{-3}
c_0 = Inlet concentration, mg m^{-3}
N = Air exchange rate, h^{-1} ; $N = Q/V$, where Q = Flow rate through chamber, $m^3 h^{-1}$, and V = Chamber volume, m^3
t = Time, h

If the measured data closely follow the theoretical curve, the chamber is well mixed. When the measured data lie above the theoretical curve, short circuiting of the flow is occurring and the chamber air is not well mixed. Short circuiting is probably caused by poor placement of the air inlet and/or outlet ports. If the measured data fall below the theoretical curve, some of the tracer gas may be adsorbed on chamber walls, or incomplete mixing may be occurring. Tests to determine the adequacy of mixing should be conducted not only in an empty chamber, but also with inert supports of the types of samples to be tested to ensure that placement of the samples in the chamber will not result in inadequate mixing.

Quantitative guidance on the mixing is unavailable. One method might be to derive the volume (V), considered as a variable, through a best fit of the measured data. One could then compare the actual chamber volume to the "apparent" chamber volume based on the curve fit. A difference of >10% between the actual and "apparent" volumes might be considered unacceptable.

Leaks should be checked e.g. by pressure drop measurements or by measuring occasionally the air flowrate simultaneously at the inlet and at the outlet port, in order to verify that they are equal.

Surface air velocity

If it is desired to maximize the mass transfer in the boundary layer, one should use a relatively high velocity (e.g., >0.3 m s^{-1}). This will require the use of a fan to direct the flow along the surface of the material. However, care should be taken that the flow be parallel to the material surface: radial ventilators fit this purpose. The test objectives may require low velocities, more representative of indoor environments: in this case one has to consider that a velocity of 0.1 m s^{-1} is the minimum measurable, lower velocities can only be calculated, e.g. dividing the air flowrate by the chamber section. The point where air velocity is measured should be as close as possible to the sample surface and roughly in the centre thereof.

Temperature control system

Temperature is preferably controlled by placing the test chambers in incubator cabinets or other controllable constant temperature environments. This way grants the best possible temperature homogeneity, independently of chamber mixing, and consequently cold spots (and condensation) on the walls are avoided. However, chambers achieving temperature control by conditioning of the incoming air and the use of highly insulated walls are commercially available.

Lights

Small chambers are normally operated without lights. If the affect of lighting on emissions is to be determined, appropriate interior illumination should be provided. If lighting is used, care should be taken to avoid heating of the chamber interior.

Clean air generation system

Clean air must be generated and delivered to the chambers. A typical clean air system might use an oil-less 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). However also conventional oil compressors can be used, provided that oil droplets are removed; also gas cylinders or charcoal filtered outdoor or laboratory air may be used. The amount of air flow required should be calculated before a decision is reached on the supply system. Typically a flow corresponding to at least 3 air changes per hour should be possible. The required purity of the air must be determined based on the type of samples to be evaluated, (see Section 7 - Quality Assurance/Quality Control).

Humidification control system

Humidity of the chamber air is controlled by adding deionized (or HPLC grade distilled) water to the air stream. Injection by syringe pumps followed by heating to vaporize the water can achieve desired humidity levels, although syringe pumps are prone to breakdown during prolonged, continuous use. Other types of pumps (e.g., HPLC) might also provide sufficient accuracy. Humidification can also be accomplished by other means, e.g. bubbling a portion of the airstream through deionized water at a controlled temperature (e.g., in a water bath), by spraying water through a nozzle, etc.. Coiled lines inside the constant temperature environment (e.g., incubator) can be used for inlet temperature equilibration before delivery to the test chambers. Inlet air can be dried through various systems (e.g. condensation by cooling, sorption on molecular sieves, permeation distillation drying tubes), the only constraint being to have a residual relative humidity compatible with the humidity level required for the experiment. The water used for humidification must not contain interfering organic material.

B. ENVIRONMENTAL MEASUREMENT AND CONTROL

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, rotameter) can be used. If the flow is controlled/measured before the humidification step, the possible correction for volume increase due to the water vapour should be considered. Chambers may be operated very slightly above atmospheric pressure (few hPa). Uncontrolled air exchange due to leaks must be $\leq 1\%$ of the controlled air exchange. Temperature control is discussed above (see Section 2.A). 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 by 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.

Microcomputer based measurement and control systems can be used to set air flow rates and monitor temperature, relative humidity, and air flow during the course of experiments. In this way, chamber environmental data can be continuously monitored, then compiled and reduced for archival storage or display with minimal operator effort. While automatic systems provide enhanced data collection and control, they are also expensive and complex. The simplicity and low cost of manual systems may be preferable under many circumstances.

SECTION 3

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 would have to 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, sorbent media, evacuated canisters), and instruments to analyze organic compounds (e.g., gas chromatographs [GCs]). The remainder of this section discusses the alternatives for small chamber sampling and analysis of organic emissions; technical details of specific systems are not included.

A. SAMPLING DEVICES

The exhaust flow (i.e., chamber outlet) is normally used as the sampling point, although separate sampling ports in the chamber can be used. A multiport sampling manifold can be used to provide flexibility for duplicate samples. A mixing chamber between the test chamber and the manifold could be included to permit addition and mixing of internal standard gases with the chamber air stream (see Fig. 1). Sampling ports with septums are needed if syringe sampling is to be conducted. The parts of the sampling system coming in contact with the vapours should be constructed of inert material (e.g., glass, stainless steel, Tedlar). Any ducting between the chamber and the sampling device should be as short as possible and maintained at least at the same temperature as the test chambers. The exhaust from the sampling system should be ducted into a fume hood, ensuring that any hazardous chemicals emitted by the test materials are isolated from the laboratory environment.

Samples can be drawn into gastight syringes, GC sampling loops, evacuated canisters, or through sorbent cartridges using sampling pumps. Gastight syringes and closed loops are frequently used when chamber concentrations are high and sample volumes must be small to prevent overloading of the analytical instrument. Larger volume samples can be pulled through sorbent cartridges using sampling pumps. Flow rate can be controlled by an electronic mass flow controller. The sampling flow rate should be a fraction of the chamber flow rate, low enough to prevent perturbing the chamber flow and infiltration of external air through leaks. Valves and a vacuum gauge may be incorporated into the system to permit verification of system integrity before samples are drawn. The entire system can be connected to a programmable electronic timer to permit unattended sample collection.

B. SAMPLE COLLECTION MEDIA

If the sample is collected via syringe or closed-loop sampling, it is injected directly into a GC or other instrument for analysis. Collection in a sampling bag (e.g., Tedlar) or vessel (e.g., glass, stainless steel) allows for larger samples. For many small chamber evaluations of indoor materials, low concentrations of the compounds of interest require large volume samples and collection on an appropriate sorbent medium is required. Several sorbent materials are available for use, individually or in combination, including Tenax, graphitized carbon, Porapak, XAD-2, activated carbon, glass beads and Ambersorb. The selection of the sorbent (or sorbent combination) depends on the compound(s) to be collected. For example, a sorbent combination of carbon molecular sieves and Tenax, in series, allows quantitative thermal desorption of compounds from C 2 to C 18. Desorption temperatures up to 250°C are commonly used for Tenax; however, Tenax artifact formation (decomposition with production of benzene, etc.) increases at increasing temperatures. Graphitized carbon sorbents can be desorbed at temperatures up to 400°C and thus in principle they are useful for sampling a much wider range of compounds than Tenax. However, while graphitized carbon shows promise, it is emphasized that limited data are available on its performance for the wide variety of compounds emitted indoors; some drawbacks (e.g. decomposition of aldehydes) have been observed (De Bortoli, et al. 1989). XAD-2 resin can be used to collect compounds considered to be semi-or non-volatile (i.e., boiling points above 240°C) which must be recovered with solvents. Thermal desorption however, whenever applicable, presents a much greater sensitivity because the whole sample is analyzed. Additional details on the selection and use of sorbents can be found in Adams et al. 1977; Brown and Purnell 1979; De Bortoli et al. 1989; Gallant et al. 1978; Harris et al. 1982; Krost et al. 1982; Pellizzari et al. 1976; Piecewicz et al. 1979; Rothweiler 1990; Schlitt et al. 1980.

If sorbent collection is used, the laboratory must be equipped with appropriate storage capabilities. Airtight glass tubes or chemically inert bags have been shown to be suitable for storing sorbent tubes. Flushing the storage containers with high purity nitrogen prior to use will help ensure their cleanliness. Storage of samples in a freezer at about -20°C will keep them cleaner than at room temperature. Sorbent samples should be desorbed and analyzed within a few days of collection. The storing procedure should anyhow be checked by blank and spiked samples.

Sample cartridges with sorbents which are best eluted by thermal desorption are introduced into a small oven and connected on line with the GC column; on heating, the vapours are fed to a cryotrapping device from which the organic compounds are thermally desorbed to the GC column: devices to perform these functions are commercially available. Solvent extraction and liquid injection to the GC can also be employed.

C. ORGANIC ANALYSIS INSTRUMENTATION

A variety of analytical instruments are available for determining the concentration of the organics sampled from the chamber, with GCs being the most commonly used. GCs have a wide variety of columns available for separating

organic compounds. Capillary columns are generally preferred. Several detectors can be used depending on the purpose of the test and the compounds of interest.

Several detectors can be used depending on the scope of the measurements : mass selective detector (MSD), flame ionization detector (FID), electron capture detector (ECD), etc. Sufficient material has to be collected to satisfy sensitivity and detection limit requirements of the instrument employed. Some compounds are not easily measured with GCs; for example, low molecular weight aldehydes require other instrumentation (e.g., HPLC).

D. STANDARDS GENERATION AND SYSTEM CALIBRATION

The correct operation of the whole procedure (including mixing, leaks, sink effects, etc.) can be checked by addition of calibrated vapours or gases to the test chamber or to the sampling manifold from permeation devices or gas cylinders. Internal standards for quality control are added at the head of the sampling system (Health and Safety Executive 1981, Sanchez et al. 1987). The internal standard should not be added to the chamber due to the potential for adsorption on the material being tested. Calibration of the analytical system can also be achieved by spiking sampling tubes with the compounds of interest. The number of control samples and analyses (internal standards, spiked and duplicate samples, etc.) should be sufficient to assure good quality control.

SECTION 4

EXPERIMENTAL DESIGN

A. DEFINITION OF TEST OBJECTIVE(S)

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. The same may happen if data for a source emission inventory is to be collected. A manufacturer might want to know the emission 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.

B. EXPERIMENTAL PARAMETERS

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. Six variables are generally considered to be critical parameters: temperature (T), humidity (H), air exchange rate (N), surface air velocity (v), product loading (L), and time (t):

Temperature

Temperature affects the vapour pressure, diffusion coefficients (diffusivity), and desorption rates of the organic compounds in the materials/products and can have a major impact on emission rates.

Humidity

Humidity has been shown to affect the emission rate of formaldehyde from particleboard and may have similar effects for other water soluble gases or materials subject to hydrolysis. Humidity can be expressed in relative (% of saturation) or absolute (g water / g air) terms.

Air exchange rate

Air exchange rate (h^{-1}) is determined by the mass flow rate of clean air to the chamber divided by the chamber volume. The air exchange rate reflects the amount of dilution and flushing that occurs in indoor environments and has a major impact on chamber concentrations.

Air velocity

As discussed in Section 1.C., the velocity near the surface of the material being tested can affect the mass transfer coefficient; for instance it has been observed that a tenfold variation of air velocity from 0.2 to 2 m s^{-1} caused the emission rate from a carpet to increase by 30% (Ullrich 1990).

Product loading

Product loading is the ratio of the test specimen area to the chamber volume. This variable allows product usage in the test chambers to correspond to normal use patterns for the same product in "full scale" environments. However, the loading factor should be adjusted in such a way that concentrations are far from the saturated vapour pressure and meet analytical needs. The ratio of air exchange rate (N) to product loading (L) is often selected as a parameter in designing chamber experiments (see equations 4 and 5 in Section 6). In some cases however, the configuration of the source makes product loading an inappropriate parameter. For example, studies of sealants often employ elongated beads. In this case, the configuration and length of the bead are appropriate experimental design parameters.

Product age

Age is a critical parameter, since most materials have emission rates that vary with time. Fresh, wet solvent containing products can have emission rates that vary several orders of magnitude in a few hours; other materials such as pressed wood products may have emission rates that take several years to decay.

C. PRODUCT HISTORY

Information on the history of the material/product to be tested is useful in designing the testing programme. Details of manufacture, production, or assembly may be useful in determining compounds to be emitted. Information on product age, treatment (e.g., coatings, cleaning), storage conditions (i.e., time, temperature, humidity, ventilation), and handling/transportation may provide additional insight. For example, older materials may emit at a lower rate than new materials; materials stored at high temperatures may also have lower emission rates when tested; and storage or transportation with other materials may cause adsorption of organics which will be re-emitted during the chamber tests.

D. TEST MATRIX

For each material tested, a test matrix is developed to allow the variables of interest to be investigated. As is normal in experimental programmes of this type, the desire to collect data over an extensive parameter range is limited by cost and time constraints. Table 1 is an example of a test matrix developed to evaluate the effect of several variables on emission rates. This test matrix covers five

experimental conditions, each with two replicates (A and B). The test matrix was designed to evaluate the effect of specific parameters as follows:

a) Effect of Temperature (T) -Tests 1 and 5;

b) Effect of Air Exchange Rate (N) -Tests 1, 2 and 3;

c) Effect of Product Loading (L) -Tests 2 and 4;

d) Evaluation of Constant N/L-Tests 1and 4.

TABLE 1. EXAMPLE TEST MATRIX

Test No.	Temp. (°C)	Airflow ($l\ min^{-1}$)	N (h^{-1})	Surface Area (m^2)	L ($m^2\ m^{-3}$)	N/L ($m\ h^{-1}$)
1A	23	1.4	0.5	0.035	0.2	2.5
1B	23	1.4	0.5	0.035	0.2	2.5
2A	23	2.8	1.0	0.035	0.2	5.0
2B	23	2.8	1.0	0.035	0.2	5.0
3A	23	5.5	2.0	0.035	0.2	10.0
3B	23	5.5	2.0	0.035	0.2	10.0
4A	23	2.8	1.0	0.070	0.4	2.5
4B	23	2.8	1.0	0.070	0.4	2.5
5A	35	1.4	0.5	0.035	0.2	2.5
5B	35	1.4	0.5	0.035	0.2	2.5

E. RECOMMENDED TEST CONDITIONS

For routine testing of indoor materials, the following test conditions are recommended:

Temperature = 23° C
Relative humidity = 45 %
Air exchange rate = 0.5 and/or 1.0 h^{-1} *

Concerning product loading, realistic values for some building materials are given in Appendix 2.

*If the surface air velocity remains constant or is sufficient to avoid limiting evaporative transfer, the effect of varying the air exchange rate will only be on concentrations.

SECTION 5

EXPERIMENTAL PROCEDURES

A. COMPOSITION OF EMISSIONS

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. If 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. An initial evaluation of the composition of the emissions expected from a source can be conducted by surveying available information, including: a) reports or papers on previous studies of the source, b) ingredients listed on the product label, c) information obtained from the manufacturer or appropriate trade organizations. In Europe we lack (at least in a form available to the public), except few exceptions (e.g., "Fiches toxicologiques" issued by the Institut National de Recherche et de Securité in France), what in the USA is designed as Material Safety Data Sheets: it would be helpful if such information was more widely available. Such information is usually insufficient to indentify the compounds of interest, but it does provide some guidance. 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.

Initial tests should be simple and semi-quantitative data may be sufficient in order to determine whether the types and amount of emissions are likely to be of concern for indoor air quality. These tests can be carried out using either headspace analysis or microchambers.

B. SCREENING BULK PRODUCTS BY HEADSPACE ANALYSIS/MICROCHAMBER

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 or dynaic headspace analyses are used (Merril et al. 1987). For materials with high emission rates of organic compounds, a sample quantity of 0.1 to 0.25 g may be more than enough to meet the detection limit requirements of an MSD operated in the scan mode or other detectors. Low emission materials, such as carpet, may require a different approach. A purge gas (e.g., nitrogen) can be pulled over the material (i.e., flow-through) and collected on a sorbent trap. Sufficient material and sampling time must be used to accumulate components to a level adequate for detection by the MSD or other detector. While headspace analyses are normally conducted at ambient temperature (e.g., 23° C) and atmospheric pressure, it may be necessary to increase the temperature (and thus the emissions) or collect a larger sample if insufficient material is collected for the detector being used. If temperature is increased, it must be noted that the emission pattern might change: if this is the case the new pattern must be documented.

Based on the study objectives, some (or all) of the compounds identified in the headspace analysis are selected for measurement and quantification in subsequent chamber tests. Criteria for selection of compounds may include: major peaks in the gas chromatograph; known carcinogen, toxicant, or irritant; and low odour threshold (see also criteria mentioned in section 1.B.).

While the headspace analysis provides useful information on the direct emissions from solid material or product of interest, it does not ensure that all emissions will be identified particularly in the case of liquid materials. Sampling and analysis techniques may be insufficient, or compounds not found in the headspace may be emitted in the chamber due to them being released in the drying process (Colombo et al. 1990) or formed by interactions with the support. Moreover the possibility that compounds are formed by degradation and/or reactions due to aging of the material must not be overlooked.

The concept of dynamic headspace analysis can be applied more effectively by the use of a simple, very small chamber. This microchamber can be an economic, fast and efficient tool for screening a broad range of materials and products. The superiority of such a microchamber compared with headspace devices is due to the fact that liquid or paste products are tested spread on a support, like in the chambers, and not in the bulk form.

As an example a simple arrangement for a microchamber is given in Appendix 1.

Use of the microchamber

The materials to be screened in a microchamber have to be handled in different ways according to their nature. Bulky solids have to be cut down to small pieces. Granular materials can be introduced directly into the microchamber. To get the emission profile of "wet" products like glues, waxes, paints, adhesives, etc., an aluminium or glass (microscope slide) support has to be coated with the product of interest. After a defined time to evaporate the solvent, this support with the testing material has to be placed into the microchamber. Liquids of low volatility can be introduced in a small open vial, which is placed upright in the microchamber.

The microchamber itself is placed in an environment with controlled temperature. A gas cylinder is the most convenient source of clean gas, which can be controlled by means of a mass flow controller. The whole gas flow can be sampled by sorption tubes (see Section 3.B.) without any pumps and gas flow measuring devices.

C. 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 (see Section 4), 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 Plan (see Section 7). At this

stage the information from the screening test is evaluated to provide guidance in selecting analytical columns, detectors, sample collection media, and an appropriate internal standard.

Internal standard

The internal standard, an organic compound added at known rate to the chamber exhaust, must meet several criteria:

a) it must be readily available (i.e., suitable for use in a permeation or diffusion device or available in a gas cylinder);

b) it must have a retention time on the analytical column that does not overlap with other compounds emitted by the material;

c) its adsorption onto and recovery from the sample collection media used during the testing must be quantitative.

Also it is desirable that the internal standard be inexpensive and have sufficiently low, if any, toxicity.

Chamber preparation

Prior to actual testing, chambers are cleaned by scrubbing the inner surfaces with an alkaline detergent followed by thorough rinsing with tap water. Deionized water is used as a final rinse. Chambers are then dried, placed in position in the temperature controlled environment, and purged at test conditions. Chamber background is monitored to ensure that background contamination is sufficiently low. 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 the internal standard(s) has (have) 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 support materials, that will be used during the test must be included to account for actual background. Once all the preparatory steps have been completed, testing of the selected material / product can commence.

Specimen preparation

The types of test specimens used in the chambers vary according to material or product being tested. Solid materials are tested "as is". If emissions from edges may differ considerably from the normally exposed surface, the edges should be sealed to avoid errors in estimating emission rate. For example, particleboard specimens can have their edges sealed with a low emitting self-adhesive aluminium tape to eliminate excessively high edge emissions. "Wet" materials are applied to a solid support. For example, a wood stain would be applied to a board, or a vinyl floor wax to a floor tile. As noted above, the uncoated support should be placed in the chamber during background tests to determine the magnitude of its organic emissions. Also, where the support edges affect the

emission of the material to be tested, the effect should be eliminated by edge sealing. The possible effect of the support on the emission of vapours should be evaluated (e.g. wood will reduce the emission of solvents compared with a ceramic tile, due to sorption). Wet materials are applied to the support outside the chamber and placed in the chamber as soon as possible. The start of the test (time = 0) is set when the door to the chamber is closed. As discussed in Section 1.A., 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 as short as possible (typically less than 10 minutes) particularly in the presence of very volatile compounds. The time of application and the test start time should both be recorded.

Specimen conditioning

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 be carried out using the same environmental parameters 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., 1 to 2 weeks).

Specimen contamination

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 organic compounds from the other materials. Subsequent testing could provide data which represent the re-emission of the adsorbed compounds rather than emissions from the original material. A good way of preserving samples for later tests is wrapping them with aluminium foil.

D. SAMPLING

Collection of a representative sample of air leaving the chamber requires the use of a sampling strategy that is appropriate to the range of volatility of the compounds present. The information obtained from the GC/MS headspace or microchamber 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 onto various media.

Sampling techniques and sampling frequency and duration must also be appropriate to describe the variation of the concentrations of compounds in the chamber air stream over time.

For quasi-constant emission rate sources, the sampling times are not critical since the chamber concentration will reach a constant equilibrium value or will

decrease very slowly. A criterion for stopping measurements is the following: four consecutive measurements (performed on different days) must give results within 0.95-1.05 of their mean and show no progressive increase or decline. If they show a progressive increase the test should be continued. If they show a progressive decline, the test can be stopped if the least square line through the four points has a slope $\leq 3\%$ of the mean concentration per day (COST Project 613, 1989).

When testing wet materials such as glues, waxes, and wood finishes, chamber concentrations may change by orders of magnitude over a period of minutes, as shown in Figure 2. Accurate description of the variation 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 cryofocussing device. Sample volumes less than 1 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.,1cm^3) can be injected directly into the GC. Larger volume samples are taken by pulling the 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.

Extreme care must be employed in handling the sample cartridges to avoid contamination (see Section 3B).

SECTION 6

DATA ANALYSIS

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

A. ENVIRONMENTAL CONDITIONS

Environmental conditions (i.e., temperature, relative humidity, flow rate) can be recorded manually or automatically stored (e.g., on floppy disks) by a computer based system. Summary statistics that describe the environmental condition "setpoints" and the actual values achieved (including variability) can be computed, and a data summary sheet prepared (see Table 2).

TABLE 2. EXAMPLE OF A SUMMARY OF THE ENVIRONMENTAL DATA

Test ID Number: PWF10

Material: Polyurethane Wood Finish

Sample Size Weight = 2.39 g Area = 347 cm^2

Chamber No.: 1 Chamber Volume: 0.166 m^3

Material Loading (L): 0.21m^2 m^{-3}

Start Date	Start Time	End Date	End Time
16.6.87	11.05	19.6.87	13.00

Environmental Conditions of the Chamber

Parameter	Setpoint	Average	Standard Deviation	Maximum/ Minimum
Temp (°C)	35.0	34.91	0.18	35.4 / 34.5
RH (%)	50.0	54.25	1.57	60.4 / 45.2
Flow (l min^{-1})	2.8	2.72	0.01	2.86 / 2.67

B. GAS CHROMATOGRAPHY DATA

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 for future review or reprocessing.

The conditions of the test chamber and the results of the GC analysis are combined to give chamber concentrations for individual compounds and total organics. In calculating concentrations, the following factors are considered:

* gas chromatographic system background (including sorbent blank for sampling cartridge);

* chamber background (determined by sampling an air volume of chamber background, including support, not smaller than test samples);

* elapsed time (period of time in minutes from start of test to midpoint of sampling period);

* flow rate of the airstream carrying the internal standard;

* mass of internal standard added and mass observed, providing percent recovery;

* mass observed for individual selected organic compounds;

* an estimate of the total organics reported as a given compound (e.g. toluene) or following the proposal by Seifert (1990).

* sampling duration and flow rate; and

* test chamber flow rate.

Chamber concentrations for total organics and individual compounds for each sample are calculated using a multi-step process:

* data should be normalized to the recovery of the internal standard.

* normalized mass is adjusted for system background and chamber background.

* the adjusted mass is divided by sample volume to generate sampling manifold concentration data.

* finally, chamber concentration is calculated by multiplying the sampling manifold concentration data by the ratio of flow out of the chamber plus standard addition flow divided by flow out. This compensates for dilution of the chamber effluent with the internal standard flow.

Chamber concentration data coupled with sample size and chamber air exchange rate are then used to estimate emission factors, as discussed below.

C. EMISSION DESCRIPTION

Emission factors for organic compounds released from indoor materials are usually expressed in terms of mass area^{-1} time^{-1}. In some cases, emission factors are reported as mass mass^{-1} time^{-1} or, in the case of caulk beads, mass length^{-1} time^{-1}, when a standard bead diameter is used. (For convenience, the remainder of this section shall use emission factor units of mg m^{-2} h^{-1}). 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.

Constant emission rate

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:

$$EF = Q\,c / A \qquad (4)$$

Where, EF = Emission factor, mg m^{-2} h^{-1}
c = Chamber concentration, mg m^{-3}
Q = Flow through chamber, m^3 h^{-1}
A = Sample area, m^2

An equivalent expression is also used:

$$EF = N\,c / L \qquad (5)$$

Where, N = Chamber air exchange rate, h^{-1};
L = Chamber loading, m^2 m^{-3}

Note that N = Q / V and L = A / V, where V = Chamber volume, m^3

Decreasing emission rate

For sources that have emission rates that decrease over the test period, a different procedure is required. The following method applies to sources with initially high emission rates that decrease with time. Most "wet" sources exhibit such behaviour.

Models have been developed to analyze the results of the chamber tests to provide emission rates (Tichenor, et al., 1988). The simplest model (i.e., neglecting sink and vapor pressure effects) assumes: a) the chambers are ideal continuous-stirred tank reactors, and b) the change in emission rate can be approximated by a first order decay, as shown in Equation 6:

$$EF = (EF)_o\, e^{-k\,t} \qquad (6)$$

Where, $(EF)_o$ = Initial emission factor, mg m^{-2} h^{-1}
k = First order rate constant, h^{-1}
t = Time, h

The mass balance for the chamber over a small time increment dt is:

Change in mass = Mass emitted - Mass leaving chamber

This can be expressed as:

$$V\,dc = A\,(EF)_o\,e^{-k\,t}dt - Q\,c\,dt \qquad (7)$$

Equation 7 can be rearranged:

$$(dc/dt) + Q\,c/V = (A/V)\,(EF)_o\,e^{-k\,t} \qquad (8)$$

Equation 8 is a linear, non-homogeneous differential equation. Given that c = 0 when t = 0, the solution to Equation 8 is:

$$c = L\,(EF)_o\,(e^{-k\,t} - e^{-N\,t})\,/\,(N - k) \qquad (9)$$

Using a non-linear regression curve fit routine, implemented on a personal computer, values of $(EF)_o$ and k can be obtained by fitting the concentration vs. time data from the chamber to Equation 9. In order to conduct such analyses, initial guesses of $(EF)_o$ and k are required. A good initial guess of k is:

$$k = N\,e^{(k\,-\,N)\,t_{max}} \qquad (10)$$

Where, t_{max} is the time of maximum concentration (c_{max}). Equation 10 is obtained by substituting c (Equation 9) into Equation 8 and setting (dc / dt) = 0 at $t = t_{max}$. Note that Equation 10 has two roots, one of which is k = N. The other root should be selected. Graphical, trial and error, or root trapping techniques can be used to guess k from Equation 10. Once a guess of k is achieved from Equation 10, an initial guess of $(EF)_o$ can be obtained from Equation 9. Figure 3 illustrates the curve fitting process for total organics data from a wood stain chamber test; the solid line is the "best fit" of Equation 9, and the data points are shown as diamonds. (Figure 3 shows only the first 10 hours of data, but the fit was made over the total test period). Once $(EF)_o$ and k are determined, the value of EF at any time t can be calculated from Equation 6.

It is emphasized that the methods for determining emission factors presented above are not applicable to sources that do not exhibit constant or simple exponential decay emissions over time and other emission models, such as e.g. those described below, may be required.

General empirical model

The following empirical model has been fitted successfully to concentration data obtained from a variety of emission processes and emitting materials. Depending on the value of its parameters, the model may equally well fit to data which, starting from zero, increase with time eventually reaching an asymptotic steady equilibrium value or to data which pass through a maximum and then decline

towards zero or towards some intermediate steady equilibrium value (Colombo, et al., 1990, the first of the two quoted references contains a full description of the model).

The time dependence of VOC concentrations in the chamber is described by the double-exponential equation:

$$c = a\,(1 - e^{-k_1 t}) - b\,(1 - e^{-k_2 t}) \qquad (11)$$

where c (mg m^{-3}) and t (h) still denote concentration and time, a and b (mg m^{-3}) are the linear parameters and k_1 and k_2 (h^{-1}) the rate parameters of the equation.

Again, the four constants a, b, k_1 and k_2 can be obtained through a non-linear least squares best fit routine. Initial guesses for a and b are usually efficient at about the concentration maximum and at the difference between the maximum and the steady value, respectively. Moreover, if c_1 is the first concentration value measured at a time t_1 and lying in the rising part of the emission curve, then a reasonable guess for k_1 is $2c_1 / (t_1 c_{max})$. For rapidly emitted compounds, e.g. those from "wet" sources, an efficient guess for k_2 lies in general at N, the chamber air exchange rate. However, for more slowly emitted compounds, e.g. those from diffusion- limited sources such as boards, plywoods, etc., k_2 is more suitably set at 0.1N or even at 0.01N. Although not strictly necessary, the visual inspection of data plots can be of assistance for guessing purposes. Figure 4 illustrates the curve fit to longifolene concentration data from a particle board test.

Without significant loss of accuracy, for concentration data increasing asymptotically to a steady value, Equation 11 may be simplified in most instances to:

$$c = a\,(1 - e^{-k_1 t}) \qquad (12)$$

For data passing through a maximum and subsequently approaching zero, Equation 11 reduces to:

$$c = a\,(e^{-k_2 t} - e^{-k_1 t}) \qquad (13)$$

the initial guesses being still established as described above.
These simplifications make possible the use of personal computer non-linear regression routines, which are sometimes restricted to estimating a maximum of three parameters.

Neglecting sinks, the mass balance for the chamber over a small time increment dt is now:

$$V\,dc = A\,EF\,dt - Q\,c\,dt \qquad (14)$$

where the Equation 14 symbols have meaning and units as described in this section, sub-section C.

By rearrangement, Equation 14 allows one to compute emission factors at any time as:

$$EF = [V\,(dc/dt) + Q\,c] / A = [(dc/dt) + N\,c] / L \qquad (15)$$

It has to be pointed out that all the above calculation methods do not explicitly account for several factors that may impact emission rates, including:

-the effect of chamber concentration on evaporative mass transfer, as described by Equation 2;

-the effect of adsorption and re-emission from sinks; and

-the effect of diffusion within the sample support.

Models have been developed (Dunn and Tichenor, 1988), based on fundamental mass transfer processes (see Section 1.C.), to include consideration of these factors, but their use requires sophisticated statistical tools and analysis experience that are not generally available. As advances in source models are made, these "tools" will become more available.

SECTION 7

QUALITY ASSURANCE / QUALITY CONTROL

Small chamber testing of organic compound emission from indoor materials/products should be conducted within the framework of a Quality Assurance Project Plan. This plan should contain a project description, data quality objectives / acceptance criteria, QA / QC approaches / activities, and QA / QC audits. Before formulating such a plan and, more generally, before starting chamber experiments, it may prove useful to consider the OECD "Principles of good laboratory practice"(OECD 1981).

Project description

A brief description should include what materials are to be tested; how the testing is to be conducted; and who is responsible for various project activities. The project experimental design (see Section 4) should contain the necessary information for this part of the Quality Assurance Project Plan.

Data quality objectives / acceptance criteria

This section of the Plan defines the precision and accuracy desired for each parameter being measured. Table 3 provides an example. The values reported in the Table must be regarded as indicative and should be validated through an interlaboratory comparison.

TABLE 3. EXAMPLE OF DATA QUALITY OBJECTIVES / ACCEPTANCE CRITERIA

Parameter	Precision	Accuracy
Temperature	± 0.5°C	± 0.5°C
Relative Humidity	± 5.0%	± 10%
Air Flow Rate	± 1.0%	± 2.0%
Support Area	± 1.0%	± 1.0%
Sample Weight(wet samples)	± 10%	± 10%
Organic Concentration	± 20%	
Emission Rate	± 25%	

Note : precision and accuracy are normally reported as ± 1 standard deviation unless otherwise noted. The error on emission rate derives from the composition of the other errors; however a further source of error (normally impossible to estimate) is due to the mathematical model used. Accuracy estimates for concentration and emission rates will be available after the interlaboratory comparison mentioned in the introduction.

QA / QC Approaches / Activities

Record should be kept of the following data:

*receipt, storage, and disposition of materials;

*GC standards preparation;

*weight loss data for all permeation tubes used in the preparation of calibration mixtures;

*environmental systems calibration data;

*chamber background measurements;

*maintenance and repairs of all equipment;

*all pertinent information for each test, including sample details, sample ID number, and GC run ID number;

*details of thermal clean-up and QC validation of sorbent cartridges;

*location and content of electronically stored data ; and

*standard operation procedures.

QC activities are carried out by project staff in a routine, consistent manner to provide necessary feedback in operation of all measurement systems. Such activities might include:

*routine maintenance and calibration of systems;

*daily recording of GC calibration accuracy and precision (i.e., control charting);

*periodic monitoring of percent recovery of the internal standard that was added to all samples;

*collection and analysis of duplicate samples;

*QC checking of organic collection sorbent tubes including sample storage; and

*periodic analysis of calibration gases supplied by an independent source, if available.

QA / QC Audits

Finally, the QA / QC programme might include periodic audits by personnel from an independent body to evaluate compliance with protocols of the Quality Assurance Project Plan.

Finally interlaboratory comparisons are recommended as an additional tool for the valdation of the whole procedure. They can identify errors in procedures for compounds for which no calibration gas exists, i.e. the majority.

SECTION 8

REPORTING TEST RESULTS

The report of the test results should contain test objectives, facilities and equipment, experimental design, sample descriptions, experimental procedures, data analysis, results, discussion and conclusions, and information on QA / QC.

Test objectives

Describe the purpose of the test programme.

Facilities and equipment

Describe the test chambers, clean air system, environmental measurement and control, sample collection (including sorbents if used), analytical instrumentation (e.g., GC / MS), and standards generation and calibration.

Experimental design

Describe the test conditions, including temperature, humidity, air exchange rate, and material loading; include a test matrix if appropriate.

Sample descriptions

Describe the sample(s) tested, including type of material/product, size or amount of material tested, product history, brand name (if appropriate), and sample selection process (e.g., random). For wet samples, describe the sample support. Also, provide information on sample conditioning, including duration and environmental conditions.

Experimental procedures

Describe the experimental procedures used during the testing, including details of the sampling and analysis techniques and references to published methods. For wet samples, provide information on the application method.

Data analysis

Show the methods, including appropriate models or equations, used to analyze the chamber data to produce emission factors.

Results

Provide emission factors for each type of sample tested and for each environmental condition evaluated. Emission factors can be provided for individual organic compounds and/or total organics. For sources with variable emission rates, provide appropriate rate constants.

Discussion and conclusions

Discuss the relevance of the findings and provide conclusions. For example, describe the effect of temperature and/or exchange rate on the emission factor.

Quality assurance / quality control

Describe the Data Quality Objectives and discuss adherence to the Acceptance Criteria. This should be done for both the environmental variables and the results of chemical analysis. Provide the results of duplicate and replicate sampling, and discuss the outcome of any audits.

An example format for reporting test results is given in Appendix 3.

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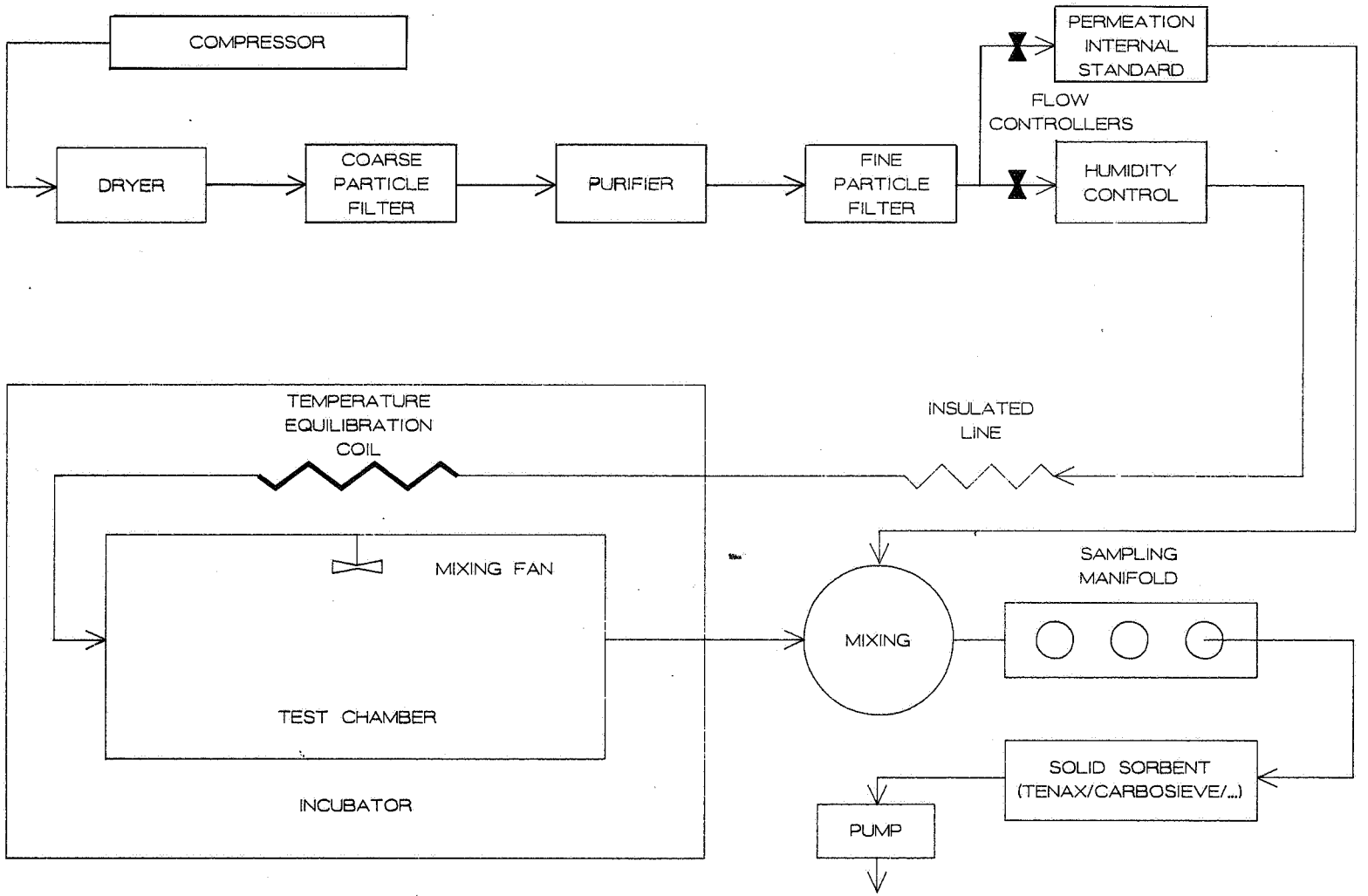


Figure 1. Schematic diagram of example small chamber test facility.

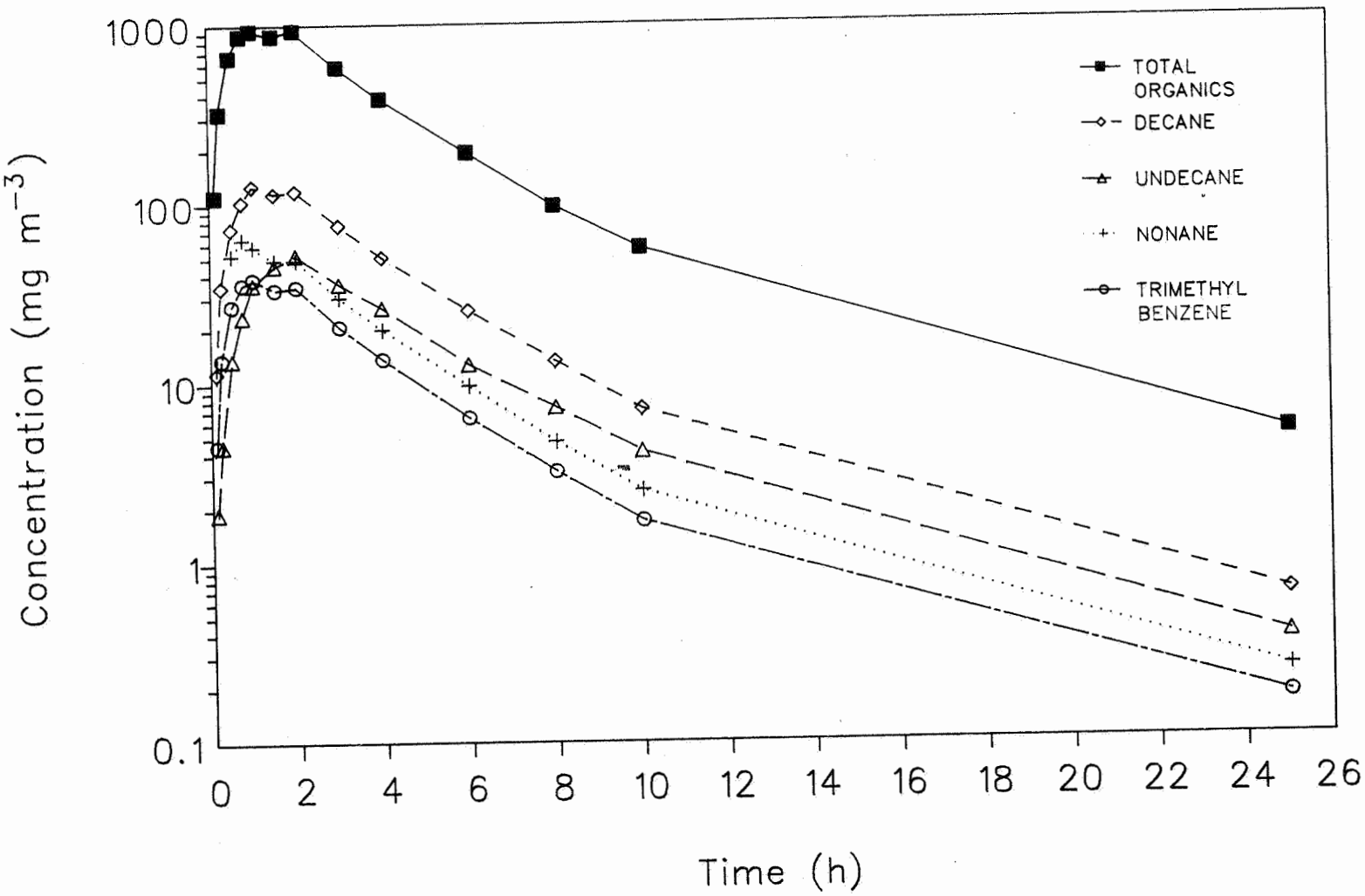


Figure 2. Chamber concentration vs. time for a wet source. (wood stain)

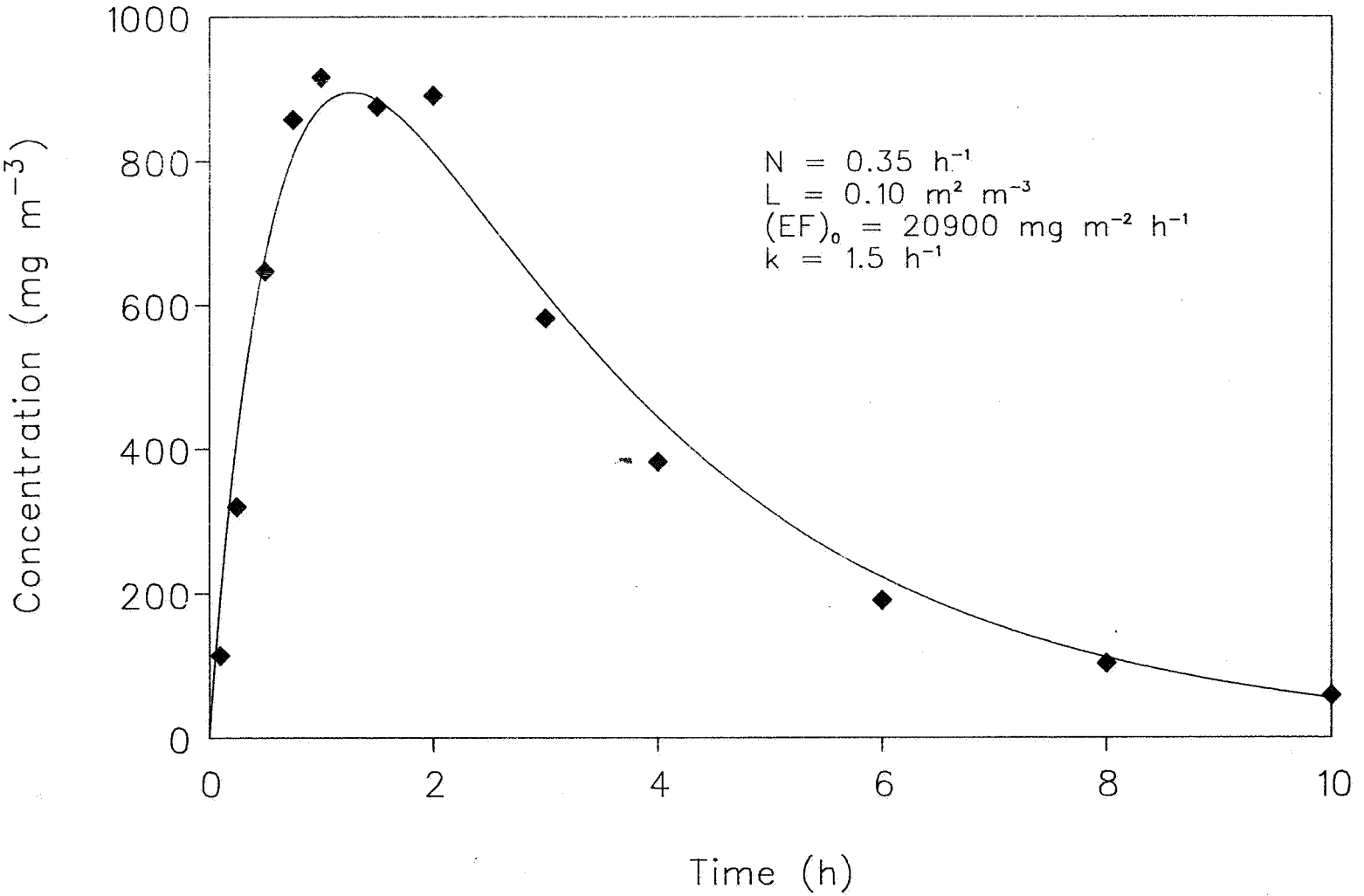


Figure 3. Example chamber concentration curve for a wet source. (total organics from wood stain)

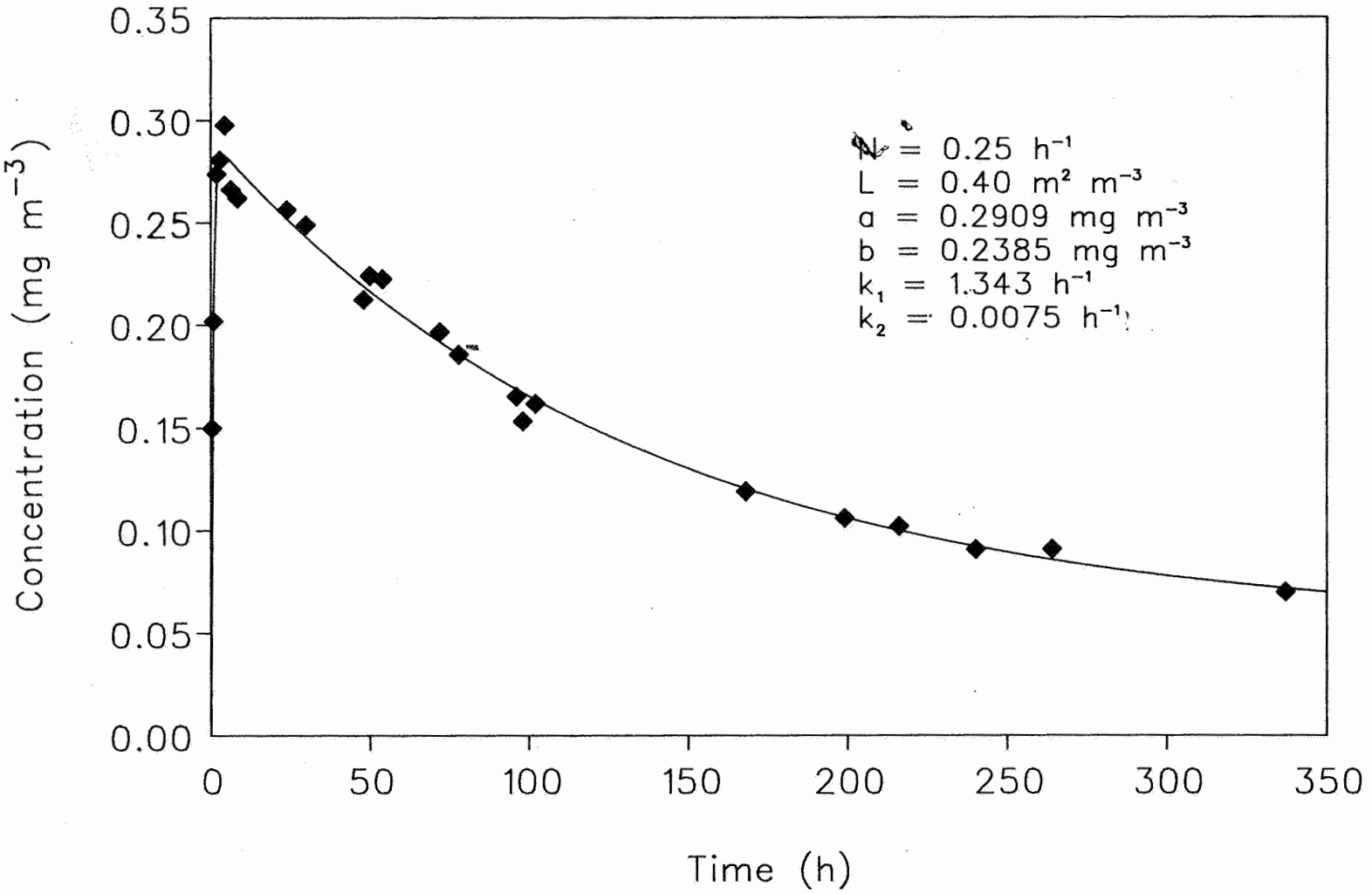
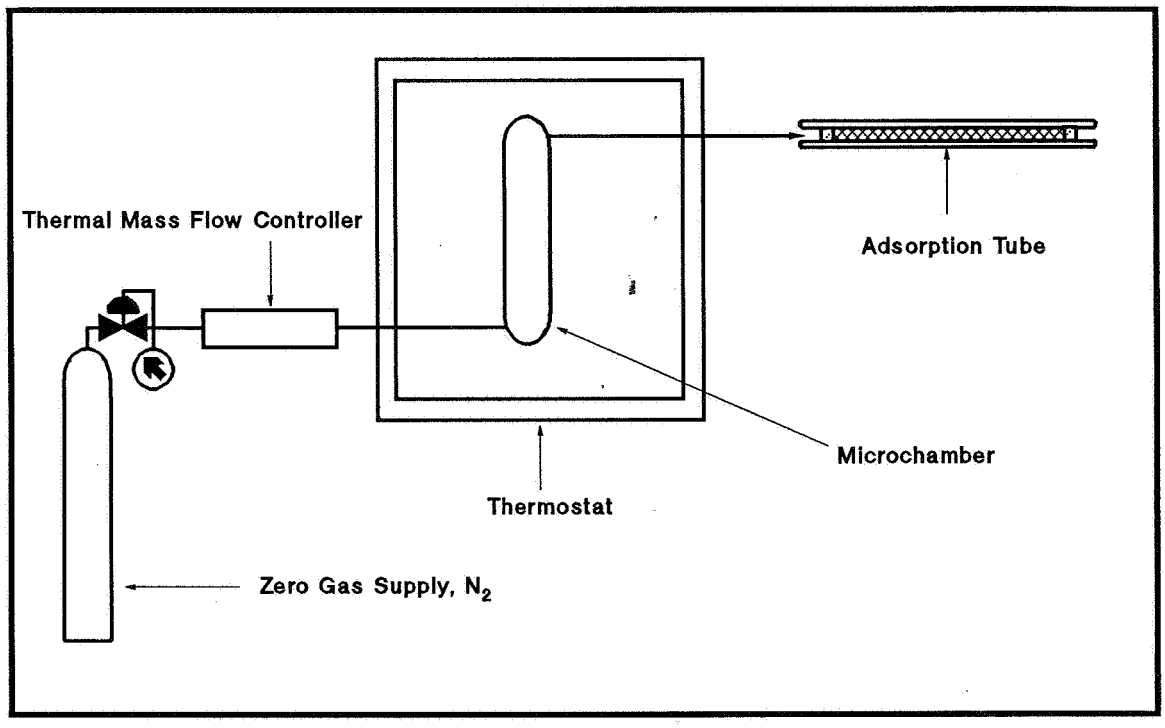


Figure 4. Example chamber concentration curve for a diffusion-limited source. (longifolene from particle board)

APPENDIX 1

Schematic Diagram of Microchamber Test Facility



APPENDIX 2

Values of Product Loading in the Chamber *

Flooring materials	0.41	$m^2.m^{-3}$
Ceiling materials	0.41	$m^2.m^{-3}$
Wall claddings, etc.	0.4	$m^2.m^{-3}$
Door surface	0.11	$m^2.m^{-3}$
Window frames	0.012	$m^2.m^{-3}$
Sealants	0.012	$m^2.m^{-3}$

*These values are taken from Nordtest Method NT-BUILD 358, Approved June 1990 "Building materials : emission of volatile compounds, chamber method", Nordtest, Post-box 111, 02101 ESBO (Finland).

APPENDIX 3

Example Format for Reporting Test Results

A. Testing Laboratory

Name

Phone and fax

Address

Certification(s) / literature references

B. TEST OBJECTIVES

C. EQUIPMENT

1. Test chamber(s)

   (a) Capacity
   (b) Wall material
   (c) Clean air system

2. Sample collection

   (a) Location of sampling port
   (b) Sampling equipment

3. Analytical instrumentation

   (a) Desorption
   (b) GC (column, detector)
   (c) GC/MS
   (d) HPLC

4. Calibration

   (a) Standard generation

D. TEST SPECIMEN

1. Product Identification

   (a) Manufacturer
   (b) Product type (including application)
   (c) Numerical designation (batch number)
   (d) Other identifying information (incl. composition of the product as given by the manufacturer).
   (e) Used or not (e.g. material sample from a building)

2. Acquisition and handling

   (a) Date received
   (b) Test specimen age or date of manufacture
   (c) Description of packaging and transportation
   (d) Storage conditions in the laboratory
   (e) Selection process (e.g. random)

3. Conditioning

4. Preparation of test specimen (describe support and application method for wet samples)

E. CHAMBER TEST CONDITIONS

1. Temperature

2. Humidity

3. Air velocity

4. Air exchange rate

5. Chamber loading ratio

6. Dates of testing

F. SAMPLE COLLECTION AND ANALYSIS

1. Timing and duration

2. Collection media

3. Sampling rate

4. Chemical identification/quantification methods and procedures

G. DATA ANALYSIS

1. Equation(s) or model and parameter values

2. Statistical procedure(s)

H. RESULTS

1. For predominant compounds, additional compounds and Total Volatile Organic Compounds (TVOC)[1] report:

   (a) emission factor [2] at t = 0;

   (b) time needed to attain 1/2 of (a);

   (c) time needed to attain 1/100 of (a);

   (d) time of last sampling;

   (e) emission factor at last sampling.

   For compounds give CAS and/or ECDIN number.

2. Report fraction identified and quantified of TVOC.

3. If absence of any substance is claimed report relevant detection limit(s).

I. QUALITY ASSURANCE AND QUALITY CONTROL

N.B. Make reference to published method whenever available.

(1) TVOC may be expressed as toluene equivalents (give detection limit for toluene) or following Seifert (1990).

(2) Give emission factors as follows:

surface materials and coatings : milligrams $hour^{-1}$ m^{-2}; sealants and adhesives : milligrams $hour^{-1}$ m^{-1} or kg^{-1}.

European Communities - Commission

EUR 13593 – European concerted action
"Indoor Air Quality and Its Impact on Man"
COST Project 613:
Guideline for the Characterization of Volatile Organic Compounds Emitted from Indoor Materials and Products Using Small Test Chambers

The Community-COST Concertation Committee

Luxembourg. Office for Official Publications of the European Communities

1991 – I-VIII, 52 pp. – 21.0x29.7 cm

Series: Environment and quality of life

EN

The report describes procedures for determining emissions of volatile organic compounds from indoor materials and products using small environmental test chambers. Consideration is given to facilities and equipment, sample collection and analysis, experimental design and procedures and to data analysis. The techniques presented are useful for both routine product testing and in depth investigations by indoor air quality researchers.