REVIEW OF THE LITERATURE ON METHODS OF MEASUREMENT OF CHEMICAL EMISSIONS FROM CONSUMER PRODUCTS

AUTHORS

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Review of the literature on methods of measurement of chemical emissions from consumer products
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</table>
Executive Summary

A wide range of consumer products are used in the indoor environment and these can release volatile chemicals and particles into the air and thereby cause exposure of occupants to these substances by inhalation. As part of the European project EPHECT (Emission, Exposure Patterns and Health Effects of Consumer products in the EU), this report reviews the literature on emission tests and test protocols for 15 types of consumer products to provide a basis to formulate the most appropriate emission test protocols for laboratory testing experiments on product emissions, in order to generate representative data for health risk assessment.

The report categorises the laboratory studies into three main groups based on the type of test; i) Studies of composition of consumer products, ii) Chamber studies that measure emissions in a controlled environment but not involving simulating real use of product, and iii) Measurement of emissions during an in-use scenario either in a chamber/test room or in a building during actual use. A further group of studies was also reviewed which have investigated the chemical reactions that occur involving terpenes in particular, once released from consumer products into the indoor environment. Details of the methodology used in each study reviewed are contained in an Excel database that complements this review.

It is concluded that the three main approaches for studying consumer product emissions outlined in the review (i.e. composition, small chamber/large chamber and real room combined with modelling) provide useful information, but the most reliable data is provided by those tests that most realistically reproduce the exposure scenario. While field measurements may represent the true exposure the large variety of practices and environmental conditions that can occur means that this type of test does not enable efficient comparison of products or modes of product use. Therefore tests undertaken under controlled conditions in chambers where the product is used in a realistic manner and concentrations can be measured at locations relevant to the breathing zone of users and bystanders provide the most appropriate means of characterising product emissions.

There is a need for standardised tests appropriate for each product type to allow comparison of products and evaluation of their emissions. The processes of preparation of draft standard test methods, robustness testing and validation of international standards for testing
Measurement of chemical emissions from consumer products emissions from building and furnishing products provides a useful framework for the development of standards for consumer products
List of acronym

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>DNPH</td>
<td>2,4-dinitrophenylhydrazine</td>
</tr>
<tr>
<td>ESI-MS</td>
<td>direct infusion electrospray ionisation mass spectrometry</td>
</tr>
<tr>
<td>FID</td>
<td>Flame Ionization Detector</td>
</tr>
<tr>
<td>FLEC</td>
<td>Field and Laboratory Emission Cell</td>
</tr>
<tr>
<td>GC</td>
<td>Gas Chromatography</td>
</tr>
<tr>
<td>GCxGC</td>
<td>two-dimensional Gas Chromatography</td>
</tr>
<tr>
<td>HEPA</td>
<td>High Efficiency Particulate Air</td>
</tr>
<tr>
<td>HPLC</td>
<td>High-Pressure Liquid Chromatography</td>
</tr>
<tr>
<td>MS</td>
<td>Mass Spectrometry</td>
</tr>
<tr>
<td>MSD</td>
<td>Mass Selective Detection</td>
</tr>
<tr>
<td>NFP</td>
<td>Nanofilm spray product</td>
</tr>
<tr>
<td>PM$_{2.5}$</td>
<td>Fine particulate matter smaller than 2.5 µm</td>
</tr>
<tr>
<td>PM$_{10}$</td>
<td>Particulate matter smaller than 10 µm</td>
</tr>
<tr>
<td>PVC</td>
<td>Polyvinylchloride</td>
</tr>
<tr>
<td>RH</td>
<td>Relative Humidity</td>
</tr>
<tr>
<td>SIM</td>
<td>Selected Ion Monitoring</td>
</tr>
<tr>
<td>SOA</td>
<td>Secondary Organic Aerosol</td>
</tr>
<tr>
<td>SMPS</td>
<td>Scanning Mobility Particle Sizer</td>
</tr>
<tr>
<td>SPME</td>
<td>Solid Phase Microextraction</td>
</tr>
<tr>
<td>VOC</td>
<td>Volatile Organic Compound</td>
</tr>
<tr>
<td>TD</td>
<td>Thermal Desorption</td>
</tr>
<tr>
<td>TGA</td>
<td>Thermogravimetric Analysis</td>
</tr>
<tr>
<td>TOEM</td>
<td>Tapered Element Oscillating Microbalance</td>
</tr>
</tbody>
</table>
1. Introduction

1.1. Background

The present review has been conducted within the framework of the project EPHECT (Emission, Exposure Patterns and Health Effects of Consumer products in the EU). The project EPHECT is a European collaborative action which has received funding from the European Union, under the Health Programme. The project deals with consumer products known to be potential sources of hazardous and other health relevant air pollutants in dwellings. The prevalence of allergies, asthma and other respiratory diseases has increased in recent decades. Among other factors, this trend has been linked to air pollution. Short and long-term exposures to indoor air pollution are an important contribution to people’s total exposure to air pollutants.

Product emissions determine to a considerable extent the indoor air quality in dwellings. Certain consumer products, such as cleaner products, air fresheners, and personal care products, are known to emit hazardous and other health relevant air pollutants. These products are widespread in dwellings, and there are studies indicating health risks associated with their use. There is considerable scientific uncertainty about the underlying mechanisms of effect compared to our knowledge about the classical air pollutants such as nitrogen dioxide (NO₂), carbon monoxide (CO), and primary particulate matter. In general, emission rates, ventilation conditions, habits and use patterns of a product, determine both user’s and other occupant’s exposure, and thus the potential health effect. Therefore, there is an urgent need for more knowledge, and data compilation about European household use (patterns) of consumer products, on product emissions and the occurrence of secondary chemical reactions and, most importantly, on the interplay between these factors, in order to be able to conduct a health risk assessment. Against these current knowledge gaps, EPHECT aims to improve understanding of the multiple exposures to primary and secondary air pollutants arising from typical household uses of consumer products. Its overall purpose is to promote European citizen’s health, by proposing actions to control exposure to hazardous and other health relevant household consumer product emissions.

The project focuses on consumer products with the highest potential risk of causing adverse health effects – those (1) known to be potential sources of health relevant air pollutants in households, (2) that cause
Measurement of chemical emissions from consumer products exposure during use to key indoor air and emerging pollutants (a) prioritised by international concerted actions or organisations, and (b) reported as potentially hazardous and occurring in the products. The main aim of the work is to identify and quantify these key indoor air pollutants and emerging pollutants emitted by a selection of consumer product classes during simulated European use scenarios. Guidance on product emission testing will be generated, and exposure reduction guidelines and policy options will be formulated as recommendations for actions to decrease the risk of respiratory diseases.

As a partner in the EPHECT project the French Agency for Food, Environment and Occupational Health and Safety (ANSES) is responsible for carrying out a review of the literature on emission tests and test protocols for consumer products. This work will contribute to the formulation of the most appropriate emission test protocols for undertaking laboratory testing of product emissions, in order to generate suitable data for health risk assessment. IEH are assisting Anses with this task and this report presents a review of information published on emission tests and on emission test protocols for consumer products and pollutants that have been selected by the EPHECT project team.

This report highlights which test conditions and procedures are indispensable for consumer product emission testing, and which are not, according to the literature. Data gaps in consumer product emission testing are identified. The work will be based on a bibliographical reference list established by the EPHECT team. The review will include the identification of parameters to be considered in laboratory testing experiments, in order to conduct emission tests whilst simulating realistic use scenarios.

### 1.2. Selected consumer products and target pollutants

For the purposes of the REACH (Registration, Evaluation, Authorisation and restriction of Chemicals) regulations a consumer product or article is in general regarded as a product that can be purchased from retail outlets by members of the general public (ECHA, 2010). Exposure of consumers to chemicals from these products can occur as a result of their use or through being present in an environment where the products are used by others. Routes of exposure can be inhalation, dermal and oral. For non-volatile substances house dust can present an important pathway for exposure of young children by both oral and dermal routes.
The project EPHECT is concerned with inhalation exposure and addresses consumer products defined as any article chemically formulated, used in a non-permanent way in indoor environments, and intended for housekeeping, personal care activities or enjoyment. Because of the large number, the products have been classified according to their category of use (air freshener, all purpose cleaner, floor polish, etc.), their state (liquid, solid, etc.) and their packaging (flask, spray, etc.). The products within the same categories of use are likely to have the same or similar chemical composition. The state and the packaging format result in differences between products of the same categories of use, in terms of pollutant emissions. A list of 15 product classes shown in table 1 were selected as meeting the following criteria, defined in the EPHECT proposal:

- The product is used in households,
- The use of the product causes an exposure related to the use,
- The product emits key or emerging pollutants,
- The product has a considerable indicative household use frequency,
- The product mainly causes inhalation exposure,
- The use causes a health end point (allergy, asthma, etc.).

Table 1. Consumer product classes studied in the EPHECT project

<table>
<thead>
<tr>
<th>Class</th>
<th>Description and types</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. All purpose cleaners</td>
<td>Cleaning products that can be used on almost any hard surface i.e. stainless steel, ceramics, wood, glass, etc.; gel, liquid, cream, tissues.</td>
</tr>
<tr>
<td>2. Kitchen cleaning agents</td>
<td>Products used for cleaning different surfaces in the kitchen, i.e. oven cleaner, water softener, stainless steel cleaner etc.; liquid, spray, tissues.</td>
</tr>
<tr>
<td>3. Floor cleaner</td>
<td>Products recommended for cleaning different types of flooring such as hardwood, linoleum or tile. This is not to be confused with floor polish (see below); powder, spray, gel.</td>
</tr>
<tr>
<td>4. Glass and window cleaner</td>
<td>Cleaning products specially designed for surfaces such as glass, windows and mirrors; liquid, spray, tissues.</td>
</tr>
<tr>
<td>5. Bathroom cleaning agents</td>
<td>Products specially destined for cleaning different surfaces in bathrooms, i.e. lime scale remover, toilet cleaner etc.; liquid, spray, tissues.</td>
</tr>
<tr>
<td>----------------------------</td>
<td>----------------------------------------------------------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>6. Furniture polish</td>
<td>Products used on furniture in order to give it a smooth and shiny surface; liquid, spray, tissues.</td>
</tr>
<tr>
<td>7. Floor polish</td>
<td>Products which are used on non-carpeted floors such as hardwood and tile in order to give them a smooth and shiny surface. This is not to be confused with floor cleaners (see above); liquid, spray, tissues.</td>
</tr>
<tr>
<td>8. Combustible air fresheners</td>
<td>Products such as scented candles, incense or heated oils, which can be used inside homes and release fragrance when they are burned; candles, incense.</td>
</tr>
<tr>
<td>9. Air fresheners in sprays</td>
<td>Sprays which are used inside homes and release fragrance; spray.</td>
</tr>
<tr>
<td>10. Passive air fresheners (passive units)</td>
<td>Air fresheners which come as units/devices containing a gel, liquid or wipes/tissues which you leave in the room and which release fragrance; passive units.</td>
</tr>
<tr>
<td>11.1 Electric air fresheners</td>
<td>Air fresheners that you plug in to an electrical outlet; electric powered unit.</td>
</tr>
<tr>
<td>11.2. Insecticide</td>
<td>Products designed to kill ants and which can be used in homes (indoors); electric powered unit.</td>
</tr>
<tr>
<td>12. Coating products for leather and textiles</td>
<td>Products used on leather or textile items (i.e. furnishing, clothing or accessories), in order to protect them from water, scratch and wear and/or to improve their appearance; leather protector, shoe polish etc.</td>
</tr>
</tbody>
</table>
# Measurement of chemical emissions from consumer products

## Table 1: Consumer Product Classes

<table>
<thead>
<tr>
<th>Number</th>
<th>Product Class</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>13.</td>
<td>Hair styling products</td>
<td>Cosmetic products which are sprayed onto hair in order to keep it in place after styling; spray, gel.</td>
</tr>
<tr>
<td>14.</td>
<td>Spray deodorants</td>
<td>Cosmetic products for men or women which come as sprays and are applied on skin in order to prevent body odour; spray.</td>
</tr>
<tr>
<td>15.</td>
<td>Perfumes</td>
<td>Cosmetic products for men or women which are applied on skin in order to provide a pleasant fragrance; liquid, spray.</td>
</tr>
<tr>
<td>16.</td>
<td>Ethereal oils*</td>
<td></td>
</tr>
<tr>
<td>17.</td>
<td>Deodorizers*</td>
<td></td>
</tr>
<tr>
<td>18.</td>
<td>Toys (perfumed)*</td>
<td></td>
</tr>
<tr>
<td>19.</td>
<td>Magazines*</td>
<td></td>
</tr>
</tbody>
</table>

(*) non-compulsory consumer product classes

Regarding the target compounds, EPHECT focuses on air pollutants known to be risk factors of respiratory diseases, including (chemo-)sensory irritation of the airways, asthma and other pulmonary effects. Several types of compounds are known to be emitted by cleaning products, air fresheners or other household products, including aromatics, terpenes and derivatives (terpenoids), aldehydes, etc., and these may be introduced in the product composition as solvent, preservative, or fragrance. The list of target compounds for the EPHECT project is presented in Table 2 below. It includes a set of key pollutants prioritized by the WHO indoor air guidelines, the WHO air quality guidelines and the European INDEX project. In addition a selection of emerging pollutants that were identified from relevant literature were included in the list as well as some additional pollutants of interest that do not pose a risk of respiratory effects.
### Table 1. Target compounds for the EPHECT project

<table>
<thead>
<tr>
<th>Compound</th>
<th>INDEX 2005</th>
<th>SCHER</th>
<th>WHO 2010</th>
<th>WHO 2000</th>
<th>Compounds of special interest</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Terpinoids</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Limonene</td>
<td>√</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>α-pinene</td>
<td>√</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Geraniol</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>a-terpineol</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Linalool</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Aldehyde</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Formaldehyde</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acrolein</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Glutaraldehyde</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acetaldehyde</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Aromatics</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benzene</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Toluene</td>
<td>√</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Xylenes</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Styrene</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Naphthalene</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benz[a]pyrene</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Disinfectants</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chloramines</td>
<td>√</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hypochlorites</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hydrogen peroxide</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Carbon monoxide</strong></td>
<td>√</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Nitrogen dioxide</strong></td>
<td>√</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ammonia</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Silanes/siloxanes/polymers +/− fluorinated</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Quaternary ammonium chlorides</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Particles (spray and combustible products)</strong></td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Miscellaneous compounds</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCBs</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCDDs</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phthalates</td>
<td></td>
<td></td>
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</tr>
</tbody>
</table>

### 1.3. Purpose of emission testing

The primary purpose of testing of emissions of chemicals from consumer products is to assess the potential exposure of people to chemicals contained in the product that could cause an adverse effect on the health of either the product user or other persons (bystander) present in the micro environment where the product is used. The presence in the product of a substance with the potential to cause
Measurement of chemical emissions from consumer products

Harm is a hazard. If a hazard exists then there is a possibility, which could have a high or low likelihood, of somebody being harmed and this therefore represents a risk. For this risk to be significant there needs to be a pathway linking the hazard to the receptor, in this case the receptor being the consumer product user or bystanders. The process of assessing risks is widely used by employers, product manufacturers and regulators to understand and as far as possible minimise risks.

There are a number of regulatory requirements for conducting risk assessments such as those concerned with occupational safety (e.g. COSHH – Control of Substances Hazardous to Health) and regulations on the use of chemicals (e.g. REACH) (HSE, 2011). The process of risk assessment for human health effects has evolved through national and international consensus and generally involves the following four steps (IGHRC, 2003):

- hazard identification,
- hazard characterisation,
- exposure assessment and
- risk characterisation.

Hazard identification and hazard characterisation are sometimes merged.

**Hazard identification** is the identification of the inherent properties of a substance (based on structure–activity relationships, in vitro, animal and human studies), that may lead to adverse health effects in exposed humans. Therefore information about the chemical composition of consumer products is important for this identification process.

**Hazard characterisation** is the evaluation of the effect of relevant factors on the qualitative and quantitative response to a chemical. Relevant factors include mechanism of action, species differences in response, route and level of exposure. Hazard characterisation is usually carried out through dose–response assessment of adverse effects (sometimes called assessment of dose–concentration–response relationships). Therefore this can be based on animal studies, chemical mechanistic evaluation and also studies of effects in exposed groups of people.

**Exposure assessment** is the measured, estimated or predicted intake of or exposure to a substance, in terms of its magnitude, duration and frequency for the general population, specific subgroups of the population or for individuals. Therefore this could apply data on the
Measurement of chemical emissions from consumer products

composition of a product or the rate of emission of a chemical from a product under defined conditions in a mathematical model to predict concentrations in a microenvironment and thereby calculate exposure of persons in that microenvironment. The rate of emission could be determined under controlled conditions in an experimental chamber and the experiment conducted to simulate a defined exposure scenario. It could also entail measurement in actual microenvironments to measure chemical concentrations during and following use of a product and thereby assess the exposure of persons in that microenvironment.

The approaches to exposure assessment can be considered as direct or indirect (IGHRC 2010);

- Direct methods involve measurements of exposure as they occur, for example personal monitoring.
- Indirect methods involve extrapolating exposure estimates from other measurements and existing data, and often involve the application of an appropriate exposure model.

Both approaches involve the definition and assessment of exposure scenarios; these are representations of an activity constructed from pattern of use information, together with assumptions and inferences about how exposure takes place for the defined population. The exposure scenario is used by the exposure assessor to evaluate, estimate or quantify exposures for the defined population.

When undertaking risk assessments the step generally considered to contribute significantly to the uncertainty is the exposure assessment (ECHA, 2008; IGHRC, 2010). The lack of good exposure data underlies this uncertainty. Poor availability of exposure data and the need for assessments to be carried out in an efficient and cost-effective way has steered the development of exposure modelling using computer based software and mathematical modelling. Exposure modelling can be defined as ‘a conceptual or mathematical representation of exposure’. Models can use either:

- the more traditional deterministic approach, in which single point estimates of input variables are used to produce single point exposure estimates, or
- probabilistic approaches, which allow input variables to be entered as distributions of possible values, and the outcome generated in the form of an exposure distribution.

IGHRC (2010) reviews a range of exposure models used by regulators with examples for the assessment of risks from contaminated land,
Measurement of chemical emissions from consumer products from ingestion of chemicals in food, exposure of workers and consumers to industrial chemicals (as required by REACH) and exposure to pesticides and biocides in particular. The report outlines the tiered approach to exposure modelling:

Tier 1; the assessment assumes that all the substance present in the product is released as a gas, vapour or airborne particulate into a standard room. An event duration of 24 hours is assumed and 100% of the substance is released at once into the room and there is no ventilation. An example is the ECETOC TRA consumer model.

Higher Tiers; these assessments use more realistic exposure scenarios and allow input of a range of parameters relating to the emission and room characteristics. An example is the ConsExpo model that addresses 3 types of emission process:

i) Constant Rate; this describes the release of a compound at a constant rate over a certain period of time. During this time, the compound is simultaneously removed from the air by ventilation of the room. This mode of release can be used if information on the evaporation properties of the compound is not available, but the time period during which the compound evaporates can be estimated.

ii) Evaporation; this describes the release of the compound from the surface of the product by evaporation, and can be used if information on the release area and the release rate of the compound from the product is available.

iii) Spray; this describes the indoor inhalation exposure to slowly evaporating or nonvolatile compounds in droplets that are released from a spray can.

Risk characterisation is the integration of hazard identification, hazard characterisation and human intake or exposure assessment in order to determine an acceptable level of intake or exposure, to predict whether or not effects in humans are likely to occur, or to predict the nature and severity of adverse effects which may occur in a given population exposed to a given concentration.

1.4. Scope of literature review

The main purpose of this review of emission measurement studies is to summarise the different experimental approaches and to identify the types of approaches necessary to assess the chemical releases from the range of products of interest listed in Table 1. It is primarily
Measurement of chemical emissions from consumer products concerned with experimental studies that have sought to provide data for the assessment of exposure to chemicals in consumer products. It is focussed on emission to indoor air and therefore does not directly address dermal exposure or ingestion of contaminants, although possibly both routes of exposure include a pathway involving airborne contamination produced by release from consumer products.

From the above summary of the risk assessment process it is apparent that studies of product composition are an important source of data for the risk assessment process and studies of this type are summarised. These studies can be informative about the likely emissions the products may produce when used in an indoor environment. However the information provided is limited because it does not provide data on actual release to indoor air. Studies that have studied product emissions to air under controlled or actual in use conditions are then described. Another aspect considered are studies of the possible physical and chemical changes that may occur after a chemical is released by a product but before it is inhaled by the receptor. The chemicals and particles formed by these changes are sometimes described as secondary emissions, distinguishing them from the primary emissions released directly from the product.
2. Method

A literature search was conducted by the EPHECT team and completed by IEH and this identified 93 papers relating to the measurement of chemicals in the consumer products of interest and their release upon use and consequent exposure of people by inhalation. To manage this information ANSES and IEH have set up an excel database that first categorises the papers into five main types with an emphasis of grouping together papers that use similar methodologies and those that are reviews rather than experimental studies. These types are;

A. Studies of composition of consumer products,

B. Chamber studies; measure emissions in controlled environment but not involving simulating real use of product,

C. Measurement of emissions during an in-use scenario either in a chamber / test room (C1) or in a building during actual use (C2),

D. Study of indoor air chemistry associated with chemicals released by some consumer products,

E. Other experimental studies and review papers.

As well as categorising as these types of study the database also records which product type is included in the study based on the classes in Table 1. The main target chemicals for each study are summarised in the database and for experimental studies details are given about the test conditions relating to the test environment, method of use of the product, type of sampling of air and methods of analysis. The parameters describing test conditions that are generally described in the published studies are presented in Table 3. These include most of the parameters reported in the international standards ISO 16000-9 (Indoor Air – Part 9: Determination of the emission of volatile organic compounds from building products and furnishing) and ISO 16000-6 (Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA sorbent, thermal desorption and gas chromatography using MS or MS-FID) which
Measurement of chemical emissions from consumer products
describe emission test procedures and sampling and analysis
techniques that are not specific to consumer products.

Table 2: List of information recorded in the database

<table>
<thead>
<tr>
<th>Reference of the publication</th>
<th>Class of the product</th>
<th>Target substances</th>
<th>Objective of the test</th>
<th>Type of exposure scenario</th>
<th>Condition of use</th>
<th>Description of the product</th>
<th>Application’s scenario</th>
<th>Amount</th>
<th>Rate of load</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Characteristics of the test</th>
<th>Emission test chamber versus experimental house</th>
<th>Total duration of the test</th>
<th>Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Characteristics of the test room</td>
<td>Description of the room</td>
<td>Volume</td>
<td>Furniture</td>
</tr>
<tr>
<td></td>
<td>Temperature</td>
<td>Humidity rate</td>
<td>Air inlet</td>
</tr>
<tr>
<td></td>
<td>Characteristics of the sampling</td>
<td>Numbers of sample</td>
<td>Times</td>
</tr>
<tr>
<td></td>
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</tr>
</tbody>
</table>

The results of the published studies are not given in the database. The full reference is provided to enable the reader to access further details if required. Its main use is a tool to identify studies concerning
Measurement of chemical emissions from consumer products particular product types with an emphasis on the methodology of the tests undertaken.

The present report reviews the main papers contained in the database and uses the same categories except that where appropriate review papers are included under one or more of the other categories depending upon their relevance.
3. Composition of consumer products

Direct analysis to determine the chemical composition of a product can provide useful information about the substances that may be emitted when it is used in an indoor environment. There are however a number of limitations including:

- Some products release their chemicals by combustion (e.g. candles) and therefore the content will not directly relate to the emission.
- Some products may release by evaporation and therefore involatile components may not be released.
- The composition does not provide data about rates of release from the product, although knowledge of mass released combined with composition data can be used to estimate emissions for some products e.g. when aerosol product of known composition is used.
- Other chemicals may be formed by chemical reaction when the product is released that are not present in the product.
- Headspace tests may be a better indicator of emissions than content but do not provide information on rates of emission applicable to real life scenarios.

In many cases, measurements by headspace can be considered as more indicative of emissions to indoor air than chemical content of a product. The methods can take a variety of forms, most broadly categorised as static and dynamic headspace tests. Some dynamic headspace tests have some similarity to small chamber tests of emissions, but for headspace studies conditions are not seeking to simulate an indoor use scenario and thereby determine an appropriate emission rate for the substance of interest during use of the product. In general compared with chamber tests the headspace test requires less sophisticated laboratory apparatus for the test (although not necessarily different apparatus for chemical analysis) and is quicker to undertake.

Studies of chemical content

Ezendam et al., (2009) collated information about the ingredients of scented consumer products marketed in Europe because of concerns
Measurement of chemical emissions from consumer products about the presence of 26 allergens. They used various published sources including manufacturer's data. One database contained 113 scented products; 48 of these were air fresheners and room perfumes and the other products were intended for steam baths or saunas. The data showed that the most frequently used fragrances in scented products (>40% of the products) were geraniol, linalool and citronellol.

Chiang et al., (2010) and Chiu et al., (2009) investigated five essential oils (lavender, lemon, rose, rosemary, and tea tree oils) that are used for medicinal purposes, food and cosmetics to determine their composition, thermal characteristics, and amounts of chemicals, in particular the carbonyl compounds, contained in vaporising gas during thermal processing. Composition was determined by direct analysis of aliquots injected into a GC/MS. Rates of emission were determined during thermogravimetric analysis (TGA) but it was commented that the shape of the vaporizing equipment, the operating parameters (i.e. liquid temperature, heating rate), the compositions of essential oils, the flow rate (ventilation of space), and the environmental conditions (i.e. ambient temperature, humidity, ventilation rate, etc.) could affect the emission factor. Therefore, the different conditions between essential oils emitted from a TGA system or used in the indoor environment should be carefully considered for the application of emission factors.

Chen et al., (2009) measured concentrations of brominated flame retardants in 69 children’s toys purchased in China. Two to three pieces of matrix taken from each toy was cut into fragments, and about 1 g subject to Soxhlet extraction with a mixture of acetone and hexane (1:1 v/v) for 24 h. The extracts were concentrated to 1-2 mL and further purified then analyzed by gas chromatography coupled with mass spectrometry (GC/MS) in the selected ion monitoring (SIM) mode. The content data was used to inform modelling of exposure by inhalation, dermal absorption and ingestion. Becker et al., (2010) reviewed risks from toxic chemicals in toys referring to cadmium, lead, brominated flame retardants, phthalates, azo dyes and bisphenol A as examples of chemicals of concern and identified the need for further action by government and industry in the USA to reduce risk of exposure.

Cohen et al., (2007) investigated the types and amounts of phthalates contained in 14 different air freshener products commercially available in the San Francisco Bay Area, USA, including eight aerosol sprays, five continuously emitting liquids, and one solid. Samples were analyzed by GC/MS according to a modified version of EPA method 8270 for semi-volatile organic compounds. One to two ml of liquid was obtained directly from a liquid product or by spraying an aerosol product into a 40 ml vial. For the gel sample a weighed amount of the
Measurement of chemical emissions from consumer products
gel was soaked in 1 ml of acetone in a sealed vial and the acetone extract was analysed. For the liquid products, a volumetric dilution was made directly into hexane and then analysed by GC/MS (using full scan and SIM).

Gockel et al., (1981) report water extraction of paper to determine the potential for formaldehyde release from carbonless paper; they further tested release to air (see next sub-section) because of concern that the extraction may enhance the release.

Hyuang et al. (2011) determined amounts of a number of ‘biogenic’ volatil organic compounds (VOC) (α-pinene, camphene, β-pinene, myrcene, 3-carene, p-cymene, D-limonene, eucalyptol, γ-terpinene, terpinolene, linalool and α-terpineol) in three categories of general purpose cleaning products (floor cleaners, kitchen cleaners and dishwashing detergents) and also air fresheners. The products were selected as being representative of products on the market in Hong Kong. A solid phase microextraction (SPME) coupled with GC/MS method was applied. The chemical composition and concentrations of individual biogenic VOCs varied broadly with household products due to their different functions and scents. It was estimated that the consumption of floor cleaners contributed mostly to total indoor biogenic VOC concentrations in Hong-Kong.

Norgaard et al., (2010) undertook analysis of 10 nanofilm spray products (NFP) by mixing sample with solvent before direct analysis by two mass spectrometric methods: (1) direct infusion electrospray ionisation mass spectrometry (ESI-MS) and ESI-MS/MS; (2) GC-MS and GC-MS/MS. The 10 products could be classified into three groups (NFPs 1–3). NFP 1 and NFP 2 contained hydrolysates and condensates of the organo-functionalized silanes 1H,1H,2H,2H-perfluorooctyl triisopropoxysilane and hexadecyl triethoxysilane, respectively. NFP 3 contained non-ionic detergents and abrasive as active ingredients.

Sarwar et al., (2004) as part of a study of the formation of particles by reactions of terpenes and ozone analysed the quantity of five terpenes (α-pinene, β-pinene, 3-carene, d-limonene, and α-terpinene) in each of five consumer products (a liquid air freshener, a solid air freshener, a general purpose cleaner, a wood floor cleaner, and perfume). One µL of liquid products (or a known mass for solid products) was placed in 1 mL of methanol for at least 24 hr. One µL of the resulting mixture was then injected directly onto a Tenax TA sorbent tube using a 10 µL gastight syringe. Samples were analyzed by thermal desorption (TD) and purge and trap control followed by GC separation and mass selective detection (MSD) (GC/MSD) using the procedures described earlier. d-Limonene was detected in all five products; the other four terpenes were detected in two or more products.

Report by the Institute of Environment and Health and ANSES for EPHECT Project, WP5
Singer et al., (2006) measured the composition of five cleaning products and one air freshener by analysis of a dilute solution of each product in methanol. A small aliquot of the liquid product (1.5–10 µl) was combined in a conical vial with 5–10 ml high-pressure liquid chromatography (HPLC) grade methanol. The vial was sealed, then sonicated or shaken gently by hand. An aliquot of solution (2–35 µl) was withdrawn by syringe and injected into a Tenax tube under a 100 ml min⁻¹ helium purge maintained for 10–15 min to volatilize the methanol. The sample was then analysed by TD/GC/MS. The same products were used in large chamber tests of emissions (section 4.3).

Spruyt et al., (2006) measured the concentrations of VOCs, formaldehyde and phthalate in six types of air freshener sold in Belgium. Aliquots of products were dissolved/extracted in methanol (for VOCs or hexane (for phthalates) prior to analysis by GC/MS, or in the case of formaldehyde reaction with 2,4-dinitrophénylhydrazine (DNPH) and then HPLC analysis.

Tokarczyk et al., (2010) report a study to validate a method to determine the concentration of 2-butoxyethanol and other glycol ethers. Aliquots of liquid products were diluted in methanol and analysed by GC/MS.

In summary twelve experimental studies investigated the composition of consumer products and one publication reviewed information about ingredients in scented products. The experimental approaches involved either direct analysis of an aliquot of liquid product diluted in solvent or analysis of compounds released by solvent extraction of the solid product. The predominant analytical method applied was GC/MS. Compounds that have potential for release into air such as terpenes and formaldehyde are reported but the studies (with exception of one reporting TGA analysis of essential oils) do not demonstrate emission to air and do not provide information on the rate of any such emission.

**Headspace studies**

Colombo et al., (1991) measured VOCs in the headspace of five household products as well as undertaking small chamber tests of emissions as described in section 4.2. They found differences between the abundance ratio of individual VOCs in the headspace and chamber test results and comment that these cast serious doubts on the usefulness of headspace analysis to characterise emissions from a product applied as a thin liquid film, particularly in the case of water emissions.
Cooper et al., (1995) undertook headspace analysis of five products (two colognes, perfume, soap, and air freshener) to identify presence of fragrance VOCs. An aliquot of liquid or solid was placed in a conical flask and the headspace sampled with a sorbent tube subjected to TD followed by GC analysis, in combinaison with low resolution MS (GC/MS), high-resolution MS and matrix isolation Fourier transform infrared spectroscopy.

The European standard EN71-11 concerning safety of toys includes provision for analysis of volatile organic solvents in an informative annex of Part 11 of the standard. It describes a static headspace test whereby a sample taken from the toy is heated in a sealed vial to 90°C for 45 minutes and the headspace is injected directly into a GC/MS system. A second test which spans the categories of product composition and chamber testing of emissions is also described. This involves heating a sample (50 mg) at 40°C for 15 minutes in a ‘thermal extractor’. Nitrogen is passed through the ‘extractor’ (microchamber) and then a sorbent tube that is subsequently analysed by TD/GC/MS to determine amounts of VOCs released. Also described in the normative part of the standard are methods of analysis of substances recovered by aqueous extraction of samples of toys. The methods of analysis are substance dependent but include headspace analysis of the extract e.g. for methanol, benzene, toluene and xylene.

Kwon et al., (2007) used static headspace and GC/MS analysis to measure chemicals released from 59 household products in Korea. Between four (in the product class of nail colour removers) and 37 (in the product class of cleaning products) compounds were detected in the headspace gas phase of each product class. Several compounds were identified in more than one class. For example, acetone was determined in five of the eight classes (cleaning products, glues, nail colour removers, paints, and polishes). Kwon et al., (2008) evaluated the emission composition for 42 liquid household products sold in Korea, focusing on five product classes (deodorizers, household cleaners, colour removers, pesticides, and polishes). They used direct GC/MS analysis of aliquots of liquid and subsequently investigated amounts of 19 target compounds by purge and trap analysis whereby aliquots of product were added to methanol and then gas passed through the solution and volatiles released were collected on a sorbent prior to TD/GC/MS analysis.

Gockel et al., (1981) report a simple dynamic headspace approach whereby a sample of carbonless paper was cut up and inserted in a glass burette and air passed through the burette and thereafter analysed to determine the formaldehyde concentration using impinger.
Measurement of chemical emissions from consumer products
collection and colorimetric determination. Temperature and humidity
were not controlled. This simple approach demonstrated the paper to
be a source of formaldehyde and allowed comparison of different paper
types. Arguably the paper cut into small pieces with high edge to
surface exposure is not representative of full sheets.

Jo et al., (2008) undertook headspace analysis of VOCs from 26 air
freshener gel products on the market in Korea. Samples were placed
in 40 ml glass bottles in a water bath at 60°C for 90 minutes. One ml of
gaseous sample was drawn into a 10 ml pressure lock syringe and
transferred to a GC/MS system. Seven products, which had the highest
limonene levels as determined by a headspace test were chosen for
chamber testing of emissions (see section 4.2).

Knoppel and Schauenbug (1989) undertook dynamic headspace
testing of VOCs from 10 products (eight waxes or polishes and two
detergents). Aliquots of product were placed in a china cup which was
placed in a 600 ml flask and helium was passed through the flask and
then a Tenax sorbent tube which was analysed by TD/GC/MS.

Masuck et al., (2010) report that more than 5,000 different fragrance
substances are frequently used in cosmetics, household products,
textiles, shoes, and toys. They tested for presence of 24 fragrance
compounds by solvent extraction of samples of toys and analysis of
aliquots by GC/MS. Also the static headspace of samples (23 and
40°C) of scented toys was sampled using SPME fibres which were
analysed by TD/GC/MS. The authors consider the SPME method to be
more efficient and sensitive than solvent extraction.

Nazaroff et al., (2006) undertook a survey of consumer products
(cleaning products and air fresheners) on the market in California and
selected 21 for analysis of their composition. The broad objective was
to obtain information on vapour-phase compositions likely to result from
the use of these products with focus on ethylene-based glycol ethers,
other compounds classified in the US as “toxic air contaminants”
(TACs)potentially reactive with ozone. A gas sampling bag method was
developed and utilized for this purpose. In this method, small quantities
of the products were volatilized in a tedlar bag and then air drawn from
the bag with a gas tight syringe and then injected onto a sorbent tube
for GC/MS analysis. Some liquid products were tested by dilution in
methanol and GC/MS analysis. Among the 21 products whose
composition was tested, six contained ethylene-based glycol ethers,
primarily 2-butoxyethanol, with levels ranging from 0.8% to 9.6%. Only
one other toxic air contaminant, xylene, was detected, and in only one
Measurement of chemical emissions from consumer products

product. Twelve of the 21 products contained terpenes and other ozone reactive compounds at overall levels ranging from 0.2% to 26%.

Sack et al., (1992) used a purge and trap method with sorbent tube collection and TD/GC/MS analysis to determine 31 VOCs in 1159 common household products. Nine hundred and thirty five of the products contained one or more of the target compounds at a concentration exceeding 0.1%. Rastogi et al., (1998) analysed 71 deodorants using a GC/MS method and identified 226 compounds. Wallace et al., (1991) used headspace analysis to measure VOCs in 31 fragranced products (perfumes, colognes and soaps). One hundred and fifty different chemicals were determined in a semi-quantitative manner.

Steinemann (2009) used static headspace combined with GC/MS analysis to measure ingredients of three air fresheners and three laundry products. Ninety eight VOCs were identified and the most commonly identified were: ethanol, d-limonene (in all six products); α-pinene, β-pinene (in five); carene isomer, 2, 4-dimethyl-3-cyclohexene-1-carboxaldehyde (Triplal 1) (in four); and acetaldehyde, benzyl acetate, 3-hexen-1-ol, and linalool (in three). Five of the six products emitted one or more US Hazardous Air Pollutants (acetaldehyde, chloromethane, and 1, 4-dioxane). The authors identify several limitations of the study; the GC/MS analysis focused on compound identification and relative concentrations, rather than actual exposures, which would be important for understanding links between compounds and possible effect; the analysis examined only primary VOC emissions from each product, rather than the possible generation of secondary pollutants, which could be encountered in actual exposure situations; the analysis did not determine whether the VOCs were derived from the fragrance mix, the basic consumer product formulation, or both; this study did not investigate whether the chemicals identified in the products would be at levels that would trigger one or more of the laws, or would be associated with possible health effects.

Tran and Marriott (2007) investigated VOCs present in incense powder and smoke in a qualitative study of four different types of incense. Headspace extraction was performed by exposing a 65 mm polydimethylsiloxane/divinylbenzene (PDMS/DVB) coated fibre inside a 4 mL glass vial, 1 cm above the sample of incense for 60 minutes at room temperature. The fibre was then transferred to the GC, and thermally desorbed for 3.5 minutes into the glass liner of the GC injector at 250°C.
Measurement of chemical emissions from consumer products

Sampling of smoke volatiles emitted from burning incense was performed by exposure of SPME fibres in a stream of smoke constrained by a glass apparatus. Two approaches were applied:

i) SPME fibre is directly exposed to the smoke stream from the incense stick burning inside an inverted glass funnel. This experiment allows sorption of smoke volatiles and also potentially particulates from the smoke onto the fibre. For both side stream and direct smoke, the SPME fibre was exposed for 20 minutes.

ii) A T-piece was attached to the glass funnel, with the incense burnt in the bottom portion of the inverted funnel, and the SPME fibre was inserted into the side arm of the T-piece. The mainstream smoke was vented through the funnel neck, and volatile compounds allowed to diffuse into the side arm of the T-piece for SPME sampling. The side arm was sealed to avoid back flush from outside air, and to prevent free smoke passage into the side arm.

Three analytical approaches were applied to identify the VOCs collected; GC/FID, two-dimensional GC in tandem with FID (GCxGC/FID) (to improve resolution of chromatography) and GC/MS.

Verjup and Wolkoff (1994) measured VOCs in the headspace of 10 cleaning agents on the market in Denmark. 0.3 mL of the cleaner was applied to aluminium foil and placed in a nylon bag filled with 5 L of air. The next day 1 L of the air was sampled through a Tenax sorbent tube that was analysed by TD/GC/MS. The same products were tested using an emission cell (see section 3.2). Verjup and Wolkoff (1995) report a further method whereby an aliquot of cleaner is heated (110°C) in a teflon tube in a flow of nitrogen and the VOCs released are collected on a Tenax tube for TD/GC analysis.

Wallace et al., (1991) report the use of headspace sampling to determine polar VOCs from 31 fragrance products such as perfumes, colognes and soaps. A small amount of the product was placed in a headspace purge vessel and a stream of slightly humidifies nitrogen gas directed through the vessel and collected in a 1.8 L Summa canister. VOCs in air in the canister were determined by GC/MS. Poor recovery of some higher boiling chemicals resulted in supplementary tests being undertaken involving direct injection of headspace gas which gave higher recoveries.

Most of the 17 papers that have determined VOCs released from products using a headspace approach have considered air fresheners and cleaning products and particularly fragrance compounds.
Measurement of chemical emissions from consumer products

Predominantly static but also dynamic headspace methods have been applied. In addition to the published papers a European standard test method for toys provides a protocol for using headspace tests. The methods are useful for demonstrating the propensity of products to release chemicals to air and they provide information on the chemical composition of emissions. The relative amounts of chemicals in the static headspace may not be a good indicator of the composition of emissions in the dynamic situation which will occur in real environments. The dynamic headspace approach is itself limited with respect to its representation of the state of the product in use, for example by using small pieces of product with a much higher exposure of cut surfaces than occurs during normal product use.
4. Product Emissions to air

4.1 Introduction

The measurement of emissions of products to air can be carried out by experiments in controlled environments provided by chambers of various types and also in the less controlled (but measurable) conditions in a real room. The resulting concentration in the chamber may be used to calculate a rate of emission of chemicals from the product, using a mass balance approach of amount of product used, amount of chemical in the chamber air and amount of air diluting the product emissions over a defined period of time. The approach may be used to determine the types and concentration of chemicals occurring in the chamber under defined conditions. This could be the average concentration, often measured as the concentration in the air leaving the chamber.

Often of interest in large chamber and real room studies is the concentration of chemicals produced by the use of the product in a realistic manner. This could involve personal monitoring of an individual using a product as well as fixed site monitoring. As discussed in section 5, for some types of emissions such as terpenes the chamber/room tests may be used to investigate the chemical changes that may occur as a result of reactions involving the primary emissions.

There is currently no international or European standard that defines the test apparatus and appropriate conditions for testing of chemical emissions from consumer products. Standard methods for testing emissions of VOCs and formaldehyde from building and furnishing products using chambers and emission cells have been available since 2006 (2004 for formaldehyde from wood based materials) and these provide a basis for testing of consumer products (EN ISO 16000-9, EN ISO 16000-10, EN 717-1). Indeed the USA ASTM standard for large chamber testing of indoor products includes emissions from consumer products within its scope (ASTM D6670-01(2007)).

An important aspect of emission testing is preparation of the test specimen that is placed in the chamber. This is described by EN ISO 16000-11 for building and furnishing products and this includes guidance for testing of liquid products such as paints and adhesives, as well as solid products.

The focus of the international standards for emission testing is the long term emission with measurements being taken three and 28 days after
Measurement of chemical emissions from consumer products

test specimen preparation. While some studies have sought to measure the emission from freshly prepared wet products using this method, it is not ideal for this purpose; in general, the sample is prepared outside the chamber and then inserted and therefore some emissions are lost during the sample preparation process. Also the test is not intended for assessment of the personal exposure of those persons using the product, but it addresses the impact of emissions on indoor air. By calculation, using emission rates derived from the chamber test, the product emission rate determined can be used to predict the concentration in a defined real room scenario assuming the same concentration throughout the whole room i.e. fully mixed. Arguably this is appropriate for assessing the impact of long term emission, but is not applicable to assess the breathing zone concentrations of chemicals released during use of a product.

A number of factors relating to the design and operation of an emissions chamber impact its performance. The factors in Table 4 are described and defined within ISO 16000-9 to ensure an appropriate performance and to enable reproducible determinations of emissions from building and furnishing products.

Table 4. Key chamber testing parameters prescribed in EN ISO 16000-9

<table>
<thead>
<tr>
<th>Factor</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Emission test chamber material</td>
<td>Surface treated (polished) stainless steel or glass; to minimise sorption of chemicals at surfaces and possible reaction</td>
</tr>
<tr>
<td>Air supply and air exchange rate (h⁻¹)</td>
<td>Controlled; affects dilution of emitted chemical and may influence rates of evaporation. Limits on background level of VOCs entering chamber in supply air (concentrations &lt;2 µg m⁻³ of target compounds)</td>
</tr>
<tr>
<td>Air mixing</td>
<td>Require effective mixing so that chamber volume is at equal concentration</td>
</tr>
<tr>
<td>Air tightness</td>
<td>Prevent uncontrolled exchange with external air</td>
</tr>
<tr>
<td>Air sampling</td>
<td>Commonly undertaken at chamber outlet but can be from within</td>
</tr>
</tbody>
</table>
Measurement of chemical emissions from consumer products

<table>
<thead>
<tr>
<th>Measurement of chemical emissions from consumer products</th>
<th>chamber provided it is representative of exhaust air</th>
</tr>
</thead>
<tbody>
<tr>
<td>Recovery and sink effects</td>
<td>Some sorption of chemicals to surfaces is inevitable, especially for higher boiling compounds. Maximum amounts of sorption are defined for two VOCs: toluene and n-dodecane (mean recovery have to be better than 80%).</td>
</tr>
<tr>
<td>Temperature (°C) and Relative humidity (RH) (%)</td>
<td>23°C and 50% RH defined for Europe; can affect rate of emission and sorption.</td>
</tr>
<tr>
<td>Air velocity in chamber (m s⁻¹)</td>
<td>Required to be measured and should be between 0.1-0.3 m s⁻¹; could affect rate of emission at surfaces.</td>
</tr>
<tr>
<td>Area specific air flow rate (i.e. ratio of air supply rate and area of test specimen) (m³ m⁻² h⁻¹)</td>
<td>Examples of appropriate values given in informative annex</td>
</tr>
<tr>
<td>Time of VOC measurement (hour or day)</td>
<td>Measure test chamber air concentration 3 and 28 days after placing test specimen in chamber (additional times may also be used); focus on long term emissions</td>
</tr>
<tr>
<td>Result calculation</td>
<td>Express as area specific emission rate (i.e. mass of compound emitted from a product) per unit time and area) (µg m⁻² h⁻¹) (after 3 and 28 days), or other depending upon the objective</td>
</tr>
</tbody>
</table>

This test therefore involves efficient internal mixing of the chamber and does not address gradients in concentration within a chamber that might occur over short time scales (relative to the mixing efficiency) or in circumstances of less effective internal mixing. Sample preparation is either external to the chamber environment or else the starting time of the test is when the product is formed.

An equally important aspect of testing of emissions is the determination of concentrations of chemicals in the chamber air. There are two main
Measurement of chemical emissions from consumer products international standards describing sampling and analytical methods for this purpose. These are ISO 16000-6 for determination of VOCs using sorbent sampling with analysis by thermal desorption and gas chromatography using mass spectrometry / flame ionisation detection and ISO 16000-3 for determination of formaldehyde and other carbonyl compounds. Some other national methods may also be used in some studies such as US EPA TO 17 which has a similar approach to ISO 16000-6.

The database produced from the studies of emissions from consumer products identified by the EPHECT team has sought to summarise the key testing parameters used in each study. Therefore issues such as chamber size, design, air supply, amount and application of product, duration of test and types of analytical methods supplied are recorded. These studies have been categorised into two broad types; those testing products in chambers without simulation of an in-use scenario and those using chambers or real rooms to measure concentration associated with a particular use scenario. These are summarised with an emphasis on describing the experimental approach used and the benefits and limitations of these approaches.

4.2 Small chambers tests

A range of types of small chamber have been employed in the study of consumer products. All but one of the studies involved air flow through the chamber to simulate ventilation in a room but one study had no air flow during the product test. A supply of air at a controlled humidity is used as a means of controlling the internal test chamber conditions. By ventilating the chamber the build up of high concentrations of substances of interest is prevented which might reduce the rate of emission from the surface; in a closed chamber this might reach a saturated vapour concentration. This is most likely to occur for liquid products with evaporating substances, i.e. external diffusion, and it is conceivable that evaporation of aerosols released by spray and combustion products would be influenced. Also if there is the possibility of reactions with air components there may be differences in the amount of products formed if the test chamber is closed without air exchange.

Generally the small size of the chamber, typically 1m³ or less, precludes preparation of the test specimen within the chamber and does not allow a person to be within the test environment in order to simulate their activities and provide the possibility of measurement of pollutants in their breathing zone.
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Bayer et al., (1988) compared sorbent tube sampling with analysis by TD/GC/MS and on line (gas sampling valve) GC/FID methods to measure VOCs from consumer products such as air freshener placed in a 240 L chamber with controlled temperature (25°C), humidity (RH 50%) and air change rate (1 h⁻¹). The product was placed in the chamber 24 hours before undertaking measurement of VOCs. The sorbent tube method had higher sensitivity, but the on line method was also considered informative, particularly for monitoring changing concentrations.

Colombo et al., (1991) measured emissions from five household products (liquid cleanser/disinfectant, liquid floor detergent, spray carpet cleanser, spray furniture polish, floor wax) using a small (0.45 m³) chamber with glass walls with an internal mixing fan. The air change rate was 0.5 h⁻¹. Samples were prepared on substrate materials (ceramic tile, carpet or wood) outside the chamber and the delay time before placement in the chamber was recorded. Amounts applied were measured by weight of substrate before and after application of product but fast evaporation made this approach not possible for the spray furniture polish. VOCs were measured by sorbent sampling periodically over a 24 hour period and analysis was by TD/GC/MS. An empirical model was used to describe the emission profile. The authors comment that concentrations may be higher in real use situations because product usage may be higher and the ventilation rate lower.

Gehin et al., (2008) investigated emissions of particles during a range of human activities including burning of candles and incense, use of spray products and household cleaning using a specialised chamber designed for the study of particulate emissions. The experiments were conducted in a 2.36 m³ hexagonal chamber with walls consisting of four panes of glass and two panes of polyvinylchloride (PVC) mounted in aluminium frames and treated with antistatic coating. The air supply was filtered (filter of class F9 and High Efficiency Particulate Air (HEPA) filter) and air entered the chamber near the floor and was removed via a funnelled steel effluent tube at the top. Air was mixed with a fan and the air change rate was 46.8 h⁻¹ and the average vertical air velocity was 0.1 m s⁻¹ so that no sedimentation can occur for particles with a diameter less than 20 mm with density 1000 kg m⁻³. The temperature and relative humidity were recorded.

In order to create realistic source emission conditions, all the activities were done from outside the chamber with airtight gloves. Therefore there was no user within the chamber to monitor concentrations in the breathing zone. The particle size spectrometers used were a DMS500 (CAMBUSTION) and a Portable Dust Monitor (PDM 1–108, GRIMM)
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AEROSOL Technik). The DMS500 measures particle concentration as a function of the equivalent electrical mobility diameter from 5 to 1000 nm. The tested sources included two types of candles and two types of incense. The gas lighter used to light the candles generated ultrafine particles while the candle extinction generated ultrafine and fine particles. The mean emission rate of each activity was calculated for the burning regime. Also tested were three spray products (two air fresheners, an insecticide, a cleaning product) using a 3 s spray duration. The authors comment that their results are comparable or higher than other results from the literature obtained with different experimental set ups suggesting that the sources are representative of the tested activities. The higher results are thought to be due to the measurement of a wider size range of particles than in the other studies.

Hagendorfer et al., (2010) investigated the release of engineered nanoparticles (ENPs) from consumer spray products. Spray simulation experiments were performed with a commercially available nano-silver spray product and a nanoparticle-free spray solution. The two most common spray types, a propellant gas spray and a pump spray, were investigated. A commercially available plexiglass glove box with a total volume of 300 L was modified. The glove box was equipped with an exhaust and a vacuum junction for fast particle evacuation. The air supply was directed through a double layer of HEPA filters. Time dependence of the particle size distribution in a size range of 10–500 nm and ENP release rates were studied using a scanning mobility particle sizer (SMPS). In parallel, the aerosol was transferred to a size-calibrated electrostatic TEM sampler. The deposited particles were investigated using electron microscopy techniques in combination with image processing software. Shortly before and during spraying the inlet and outlet of the box were closed to prevent air flow. A spray time of 1 s was used and subsequent evacuation after a spray experiment required around 20 minutes of flushing the glove box to reach background levels.

Jo et al., (2008) tested VOC emissions from seven gel air freshener products marketed in Korea. Seven products were tested in an electropolished stainless steel chamber (0.05 m³). The top of the chamber acted as a door, whereby sealing is done with a silicon gasket. Clean air for the chamber was supplied from a zero-grade air cylinder. The chamber temperature ranged between 19 and 25°C. Relative humidity was determined as being between 19 and 54% at the chamber inlet and outlet. The air in the chamber was mixed by a metal fan. Homogeneity within the chamber was tested by the simultaneous collection of samples at two different ports. Products were tested for
Measurement of chemical emissions from consumer products emissions under a typical indoor velocity (0.05–0.1 m s\(^{-1}\)). The ventilation rate was 1 ± 0.05 h\(^{-1}\). The air freshener was placed in the chamber and air was sampled periodically over a 5 hour period. Air samples were collected for 10 minutes by passing air through adsorbent tubes containing Tenax TA and target compounds were determined by TD/GC/FID.

Madany and Crump (1994) measured VOCs, including carbonyls, released from an incense commonly used in Arabian Gulf countries. Weighed amounts of incense were placed in a 1 m\(^3\) stainless steel chamber maintained at 23°C and 45% RH with an air change rate of 1 h\(^{-1}\). Air supply was filtered through particle and charcoal to provide low levels of VOCs and carbonyls in the in-coming air. Measurements were undertaken before burning of the incense and over a period of 29 hours after burning using an electrical heating device commonly used with the type of incense investigated. VOCs were determined by pumped sorbent (Tenax) sampling at the chamber outlet, with analysis by TD/GC/MS and carbonyls were by collection on DNPH cartridges and analysis by solvent desorption and HPLC. Diffusive samplers for VOCs and carbonyl were also placed in the chamber and the authors also report elemental analysis of the ash residue and of airborne particulates collected on a filter.

Manoukian et al., (2011) outline use of a 1 m\(^3\) chamber to determine VOC and particle emissions from one incense and one scented candle. The effect on emissions of temperature, air exchange rate and relative humidity conditions were investigated. Emitted compounds were monitored, during combustion and for 3 hours afterwards using cartridges (Tenax and DNPH) or quartz filters (for phthalates/Polycyclic aromatic hydrocarbon in particle phase) connected directly to the experimental chamber. The authors conclude that the study highlights that indoor conditions (e.g. temperature, relative humidity and air exchange rate) have different effects on the compounds emission. The effects and their interactions can be compound specific (e.g. more formaldehyde is emitted but less toluene as the air exchange rate increases).

Nicolas et al., (2011) present preliminary results of chamber testing of emissions from 53 cleaning products including liquids, wipes, powders, foam and aerosols. Details of the chamber are not provided but the test is described as being conducted in accordance with ISO 16000-9 and air sampling of VOCs and aldehydes was according to ISO 16000-6 and ISO 16000-3 respectively. Products were applied to glass plates for placement in the chamber.
Norgaard et al., (2009) investigated four types of nanofilm spray products that are applied to a wide range of consumer products such as bathroom tiles, flooring, textiles and windows to provide non-stick / self cleaning properties. These products were coatings for non-absorbing floor materials, for ceramic tiles, for glass and a multipurpose coating product. Three are delivered in hand pump spray bottles and one in a pressurized can. Spray tests were carried out in a closed aerosol chamber (0.66 m$^3$) made of stainless steel equipped with two fans to ensure mixing. The average temperature and relative humidity in all four experiments were 25°C and 23% RH, respectively. For spraying, the NFP containers were mounted in an automatic actuator to facilitate remote controlled spraying. An amount needed for coating 1 m$^2$ surface was sprayed horizontally toward a stainless steel target plate (46 cm $\times$ 28 cm) mounted at a distance of 35 cm from the spray nozzle. The NFP was released over a maximum period of 25 s. Air and particle sampling were performed at a position 20 cm behind the spray nozzle via Teflon tubes connected to Tenax sorbent tubes and a portable miniature mass spectrometer (MIMS) located outside the chamber. Sorbent tubes were analysed by TD/GC/MS. Two samples were taken within the first 5 minutes after the release of NFP and additional samples were taken with 10 minute intervals throughout the experiment. The MIMS was operated continuously for about 20 minutes to give continuous data on VOC concentrations. Particles were measured using a TSI FMPS model 3091, which measures the electrical mobility particle size in 32 channels with midpoints ranging from 6 to 523 nm. The authors report that a number of VOCs including cyclic siloxanes, limonene, chlorinated acetones and perfluorinated silane, and nanosize particles were emitted and several of the VOCs detected in the emissions, were not reported in the product safety sheets (MSDS) and their presence may not even be known to the manufacturer.

Person et al., (1991) selected 60 household products including glues, deodorisers, waxes and floor cleaning products based on a survey of the market in France. VOC emission rates were determined semi-quantitatively using a small chamber test. The chamber was cylindrical and constructed of glass with a volume of 4 L and experiments were conducted at laboratory temperature (22°C). Helium flowed from the base to the top of the chamber over a product sample placed in the centre of the chamber at a rate providing an air exchange of 3 h$^{-1}$. A mixing fan operated above the sample. Liquid products were applied to glass and then placed in the chamber. VOCs in gas leaving the chamber were collected using Tenax sorbent tubes and analysed by TD/GC. Carbonyls were collected on DNPH cartridges and analysed by HPLC following solvent desorption.
Salthammer (1991) undertook a literature review of VOC emissions from consumer products and also undertook chamber tests of emissions from a range of sprays, waxes, adhesives and liquid products available in Germany. The products were applied on a glass plate and placed in a 23.5 L glass chamber operated at 23°C and 45% RH with an air exchange rate of 1 h⁻¹. An internal mixing fan controlled air velocities. VOCs were measured by Tenax sorbent tubes followed by TD/GC/MS analysis and carbonyls by DNPH cartridge and HPLC analysis. A pumped charcoal tube was used specifically for terpenes and this was solvent desorbed before GC/MS analysis. Books and journals were tested after a conditioning period and then VOC monitored over a period of 24-48 h in the chamber.

See and Balasubramanian (2010) determined the emission factors of fine particulate matter smaller than 2.5 µm (PM₂.₅) and its chemical constituents emitted from six different brands of incense sticks widely used in Singapore. Controlled experiments using a 1 m³ chamber were conducted to measure the mass concentration of PM₂.₅ and to determine its chemical composition (elemental carbon (EC), organic carbon (OC), metals, and ions). Tests were conducted at a temperature of 25°C and 30% RH. No details are given about the chamber materials or the rate of air change used. One incense stick was burned at each of the four corners, and the resulting emissions were mixed by a small fan to ensure homogeneity of smoke particles. Before and after every run, the length and mass of the four incense sticks were measured. At the beginning of the combustion period, the incense sticks were lit with a propane lighter outside the chamber before they were brought into the chamber, and the door was immediately closed. The mass concentration of PM₂.₅ was monitored continuously with a Model 8520 DustTrak Aerosol Monitor during a pre-burning period, during burning and for 60 minutes afterwards. The particle emissions were collected with a MiniVol on a quartz filter (for carbonaceous analysis) or Teflon filter (for metals and ions analysis) until all the incense sticks burned out.

Silva et al., (2011) measured VOC and particle emissions from candles using a 47.9 L cylindrical glass chamber. The physical parameters used were: 23°C (±2°C); RH 35% (±5%) and air exchange rate 9.5 h⁻¹. The candle was weighed, placed in the test chamber and lit. The duration of the test was the time of the candle burning. Sampling of particulate matter smaller than 10 µm (PM₁₀) was undertaken for the duration of the experiment and VOCs were collected by active sampling on Tenax. Analysis of VOCs was carried out using TD/GC/MSD and PM₁₀ was collected on filters using CIS (conical
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Spruyt et al., (2006) tested VOC and formaldehyde emissions from 20 different air fresheners of various types (candles, scented candles, incense sticks and cones, gels, liquid and liquid based, spray and spray based) sold in Belgium. Three types of tests were conducted; emission tests with small scale environmental test chamber (0.72 m³), composition determination of six selected products and tests in dwellings. The emission test consists of exposing/using the product in a small scale environmental test chamber (0.72 m³). The test was based on requirements of ISO 16000-9 but with some variation such as test duration (24 - 48 hours). The air velocity above the surface of the products was kept at approximately 0.2 m s⁻¹ and the air exchange rate was 0.5 h⁻¹. The use of every air freshener was maximized to simulate worst case exposure scenarios, but limited to either the physical limitations (incense), manufacturer’s recommendations or practical considerations (maximum test duration of 48 hours).

Sampling of chamber air for VOCs was by use of sorbents (multi-
sorbent Carboxieve SIII – Carbotrap) followed by analysis with TD/GC/MS. The ten compounds with the highest concentrations (areas in GC chromatogram) were identified and reported. The presence of a limited set of other target VOC’s was also verified. Formaldehyde, acetaldehyde and the total carbonyl (sum of all aldehydes and ketones except for formaldehyde and acetaldehyde) compounds were determined by pumped sampling with adsorbent tubes impregnated with DNPH and subsequent solvent desorption and HPLC analysis.

Tichenor and Mason (1988) report use of small chambers (166 L) to determine rates of emissions from a wide range of building and consumer products in the USA. The consumer products included furniture polish, floor wax, air freshener and moth crystals. Screening dynamic headspace tests preceded the chamber tests involving placement of a sample in a 1 L teflon lined container followed by purging of the headspace through a Tenax sorbent tube for analysis of VOCs by TD/GC/MS. The work pre-dates the development of standardised protocols for testing emissions from building and furnishing products and discusses the importance of control of environmental factors such as temperature, humidity, air exchange rate and sorption to chamber walls.
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Uhde et al., (2011) undertook chamber tests of three types of diffusive air fresheners: glass with reed sticks, glass with wooden stopper, open glass with wooden balls. Diffusers were filled/sprayed with the included fragrance liquid according to the manufacturer's instructions. The diffusers were then tested in environmental test chambers under defined climatic conditions: 23 °C, 50% RH, air exchange rate 0.5 h⁻¹. No details of chamber design are provided. The concentration of volatile substances was determined according to ISO 16000-6 (sampling on Tenax and TD/GC/MS).

Verjup and Wolkoff (1994) used a field and laboratory emission cell (FLEC) to determine VOC emissions from 10 cleaning products over a one week period. FLEC is described in EN ISO 16000-10 for the determination of VOC emissions from building products. A 70 µm film of liquid was applied to an aluminium plate and then placed under the emission cell for one week. The air passing through the cell was at 22°C and 50%RH and the surface air velocity was approximately 1cm s⁻¹. Air leaving the emission cell passed through Tenax TA tubes and these were analysed for VOCs by TD/GC/MS. Emissions of non-polar VOCs declined immediately after the start of test whereas peak emissions of polar compounds such as 2(2-butoxyethoxy)ethanol occurred after 3-7 hours. In a further study VOCs in the air leaving the emission cell were determined by a photoacoustic detector that gave a continuous reading; this gave an informative characterisation of the emission profile but was semi-quantitative and could not speciate individual VOCs present in a mixture (Verjup and Wolkoff, 1995). In a companion paper Verjup and Wolkoff (1995a) discuss the extrapolation of emission data obtained by the emission cell method to a standard room scenario.

Zhu et al., (2001) measured the emission of 2-butoxyethanol from five cleaning products using an emission cell, FLEC. An aliquot of the liquid was pipetted into a petri dish and that was placed within the FLEC cell (contained in a specialised base unit). Clean air at 23°C passed through the cell at a rate of approximately 100 ml min⁻¹ and air sampling of the air leaving the chamber was sampled at intervals with a charcoal tube over a period of 3 days. Relative humidity on the incoming air was set at 50% unless a water based product was under test in which case dry air was used. The sampling tubes were solvent extracted and the eluent analysed by GC/MS. The authors comment that the emissions from liquid products are impacted by air velocity and consider that use of the sub-unit resulted in lower air velocities more appropriate to indoor environments than if the FLEC was placed directly over the petri dish.
In summary, 20 papers described the application of small chambers to the study of emissions from consumer products. Noteworthy is the diversity in size and type of chamber employed as well as the test conditions, e.g., temperature, humidity, and air exchange. Chambers include emission cells (where the cell is placed on the surface of the test specimen) and chambers with openings that allow spray into the test area and more commonly closed chambers ranging in size from 4 L to 1 m³ (and one specialist 2.36 m³ chamber used to study particulate emissions). A few studies refer to the use of chambers meeting the requirements of the international standard ISO 16000-9 developed for testing of emissions from construction products. While most studies focus on VOC measurement, one is specific to formaldehyde and six include measurement of particulates and three of these are specific to particulates. One study (Manoukian et al., 2011) demonstrated that the test conditions (temperature, humidity, air exchange rate) can influence the amount and composition of emissions from scented candles and incense.
4.3. In-use scenario product testing using large chambers, test rooms and real rooms

Simulation of product use is possible in well controlled conditions in large chambers where parameters such as temperature, humidity, air exchange rate, quality of in-coming air and internal mixing of the air are maintained during the test. Other tests may be conducted in rooms within buildings where environmental conditions are only partially controlled. A third approach is to not seek any control of conditions but to monitor concentrations in real environments where consumer products are used.

The following sub-section describe tests in fully controlled (chambers) and partially controlled (test rooms) involving simulated use of consumer products to determine emissions. A subsequent sub-section describes investigations in real rooms where no control of conditions was applied.

Large chambers and Test rooms

Bello et al., (2010) simulated sink, mirror, and toilet bowl cleaning tasks in a large ventilated bathroom (exhaust fan on) and a small unventilated bathroom (exhaust fan off) using a general purpose, a glass, and a bathroom cleaner. All tasks were performed for 10 minutes. The simulation plan was developed after observing actual cleaning practices in hospitals. Products were sprayed and then wiped using paper towels for mirror and sink cleaning; and a brush for toilet bowl cleaning, as commonly done at the worksite. The main reason for performing simulations was to control task frequency, duration, and environmental conditions such as ventilation and possible interferences from other sources of volatile compounds. Airborne total volatile organic compounds (TVOC) and ammonia generated during the tasks were measured with direct reading instruments. Volatile organic ingredients of the cleaning mixtures were assessed utilizing the sorbent tube method EPA TO-17. Measurements were undertaken in the breathing zone of the person undertaking the cleaning task. The authors recognize that cleaning tasks performed at actual worksites are likely to differ from the simulated tasks in several ways:
1) the duration of tasks is more variable;
2) tasks are performed consecutively in one room (e.g. mirror, sink, and toilet all in one bathroom);
and 3) the cleaning task cycle is repeated multiple times in institutions such as hospitals and schools where numerous bathrooms are cleaned in a single day.
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Due to these differences, workplace exposure concentrations are likely to be different from those obtained by the simulation.

Berger-Prieb et al., (2009) investigated the release of biocides contained in five aerosol insect sprays and two electro-vaporizers that were used according to manufacturer’s instructions. Experiments were conducted in equally sized model rooms (volume about 40 m$^3$) which were furnished like normal living rooms. Walls and ceilings were covered with woodchip wall paper, and floors with textile carpets. In each room there was a cupboard, shelves, a sofa, a coffee table, a chair, a dining table as well as a window and radiator. During the experiments, a ventilator was constantly operated in the rooms in order to simulate air circulation. The air temperature, relative humidity, and pressure were monitored continuously in the rooms but these are not reported and the air exchange rate was not determined.

Each spray product was applied in the middle of the room between the door and window. During spray application personal measurements were done during the spraying operation and for up to 2–3 minutes thereafter. During the spraying procedure, the inhalation exposure of the spray user was recorded with a personal aerosol exposure monitor (Respicon) that enabled on-line concentration monitoring of three particle size fractions and collection of airborne biocides using glass fibre filters followed by polyurethane foam (PUF) plugs (for sampling of chlorpyrifos). Filters and plugs were solvent extracted for GC/MS analysis. The electrovaporizers were plugged into a power outlet in the wall and used for 6 hours per day. A stationary Respicon TM was positioned in the middle of the room, and sampling was done over a period of 60 minutes. The study also involved measurements of dermal exposure, active ingredients of urine (person spraying and bystander in room for 5 minutes) and use of modelling to predict exposure during spray use.

Gibson et al., (1991) measured concentrations in the centre of a room and in the breathing zone of a person using two types of hard surface cleaner to determine concentrations of diethylene glycol mono butyl ether (DGMBE) in air. Two types of room were used; unfurnished rooms (32.6 m$^3$) isolated from the ventilation system, but not airtight and a bathroom (10.4 m$^3$) made as airtight as possible with sealing tape etc. The product was applied with a sponge to walls in the unfurnished room and a range of surfaces were cleaned in the bathroom. Cleaning occurred for 20 minutes and monitoring of air was for up to 24 hours. The aim was therefore to measure a worst case exposure. Air exchange rates were not measured. DGMBE in air was determined by collection on a charcoal tube, solvent desorption and
Isola et al., (2004) measured air concentrations of nine fragrance chemicals produced by simulated use of three surrogate products. For testing a pressurised aerosol air freshener a 14.5 m$^3$ sized chamber was applied. The chamber was operated at 23°C, 50% RH with an air exchange of 0.6 h$^{-1}$. This poster paper did not describe the materials used for the chamber construction but in a photograph the walls appear to be made of glass. Pumped Tenax sorbent tubes were used to collect the chemicals of interest at adult and child breathing heights and these were analysed by TD/GC/MS. Also particles were investigated using a TSI 3320 Aerodynamic Particle Sizer (APS). For the pressurised aerosol product, the test material was shaken then released in an upwards direction with a slight sweeping motion for approximately five seconds. The experiment was repeated in triplicate using the same container with monitoring up to two hours after product use. Rogers et al., (2005) report the study in further detail and comment that the experiments were conducted in a non-disturbed atmosphere and the test substance was applied towards the adult breathing zone. The authors consider that these conditions do not occur in typical residential applications and in an actual air freshener application, the product release would not occur directly in the subject’s breathing zone and the atmosphere would be mixed by the residents causing enhanced dilution. Thus, it is argued that the simulated exposure conditions represent a worst-case exposure scenario.

Isola et al., (2004) report that the same 14.5 m$^3$ chamber was also used to measure the fragrance chemicals in air when a fine fragrance was pump sprayed onto a manikin (two actuations at three locations). Sampling locations focus on the individual (adult) user (zero distance), a person near the user (1.5 ft), and a person in the workplace (5.0 ft) as well as a child’s breathing height. Measurements were carried out over a period of five hours.

The authors used two smaller stainless steel chambers (approximately 6 m$^3$) maintained at 23°C, 50% RH with an air exchange rate of 1 h$^{-1}$ to investigate the fragrance chemicals released from a plug-in vapouriser. The vapouriser was located at an adult breathing zone height, plugged into a wall socket, and the VOCs were measured over a 29 day period.

Ji et al., (2010) undertook an investigation of the impact on indoor particle concentrations of burning an incense (pinewood based) manufactured in France for 15 minute periods in the living room of an experimental test house. The house was on three levels with the
basement being the lowest level which was used as a garage. The living room was on the ground floor with four bedrooms and a bathroom on the first floor. The total volume of the house was 319 m$^3$ plus the basement. It was mechanically ventilated and using carbon dioxide (CO$_2$) decay, the air exchange rate was determined as 0.89 h$^{-1}$ in winter and 0.69 h$^{-1}$ in summer. All internal doors were kept closed except for one toilet. Number concentration (using condensation particle counters), particle size distribution (four types of instrument applied), mass concentration (using a tapered element oscillating microbalance (TEOM)) particle specific surface area (nanoparticle aerosol monitor), and particle mass were measured. Instruments were located in several rooms at breathing height and outdoors and experiments were conducted both in winter and summer. At one location a Time of Flight Aerosol Mass Spectrometer (AMS) provided on line analysis of volatile and semi-volatile organic compounds in air.

Lee and Wang (2004) investigated emissions of particulates (PM$_{10}$, PM$_{2.5}$), 11 individual VOCs, carbonyls, carbon monoxide (CO), carbon dioxide (CO$_2$), nitrogen oxides (NOx), methane (CH$_4$) and non-methane hydrocarbons (NMHC) from ten types of incense commonly used in Hong Kong. The controlled experiments were conducted in an 18.26 m$^3$ stainless steel environmental test chamber maintained at 23°C and 50% RH. The air exchange rate was 0.5 h$^{-1}$ and the air supplied was passed through a clean air system with activated charcoal particle filters and HEPA filters. Fans installed on the ceiling of the chamber ensured air mixing. Air samples were drawn from the chamber via non-absorbent tubing into a pre-evacuated canister and VOCs were determined by GC/MS. A pump located outside the chamber drew chamber air through sampling cartridges containing DNPH to determine carbonyl compounds. Teflon tubing connected the chamber with a CO analyzer, a NO–NO$_2$–NOx analyser and a CH$_4$–NMHC analyzer. Two Dust-Trak air monitors were used to measure PM$_{10}$ and PM$_{2.5}$ concentrations simultaneously in the chamber. Measurements were taken during and for 30 minutes after incense burning and results expressed as emission rates (in milligrams per hour or in milligrams per gram of incense). While the authors describe taking background measurements before each test there is no discussion of chamber cleaning procedures or possible sink effects. All VOCs measured were very volatile or volatile, the least volatile being styrene and semi-VOCs were not investigated.

Maupetit and Squinazi (2009) undertook an investigation of VOCs and formaldehyde emissions from 43 incense and scented candle products available in France. The measurements were conducted in a test room within an experimental test house. The room was unfurnished and had
Measurement of chemical emissions from consumer products
a volume of 32.3 m³, with a concrete floor, painted walls and ceiling. Outside air was blown into the room via a single window and the air extracted by a mechanical extract attached to the base of the door corresponding to air exchange rate of 0.6 h⁻¹. During the experiments, the temperature was 20 ± 2°C. The relative humidity inside the room depended mainly on the relative humidity of fresh air. VOCs were measured in the extract from the room using pumped sampling with sorbent tubes and TD/GC/MS analysis in accordance with ISO 16000-6. Carbonyls were measured by DNPH cartridge collection and analysis by HPLC according to ISO 16000-3. During the first series of measurements on eight products, six VOCs were determined (benzene, toluene, xylene, styrene, naphthalene, limonene) but thereafter only benzene was measured. For each series of measurements, a couple of air samples were collected four times: i) during combustion (sampling time adjusted to the duration of combustion for incense and one hour to burn candles) ii) for one hour after combustion), iii) one to two hours after combustion), iv) two to three hours after combustion). Concentrations in air and the mass of product burnt were reported. The measured data was used to inform an assessment of risks to population health through exposure to VOCs from incense and scented candles. The same experimental house and chamber was used by Manoukian et al. (2011a) to investigate concentration time profiles and emission rates of four scented candles and four incenses. The monitoring of emitted substances was undertaken as previously (sampling on Tenax tubes and DNPH cartridges) and by online monitoring using a PTRMS (Proton Transfer Reaction Mass Spectrometer) for VOCs and a condensation particle counter.

Rowley and Crump (2005) investigated the dispersion of aerosol sprays in a 22 m³ chamber with controlled temperature and air exchange and compared measured and modelled propellant and solvent concentrations. The sprays were generated using remotely activated aerosols fitted with valve and actuator combinations to produce a distribution of either fine or coarse particles to represent four types of product (hair spray, surface cleaner, insect spray and polish). Continuous infra red and pumped Tenax sorption tubes (with subsequent TD/GC/FID analysis) were used to measure concentrations of volatile hydrocarbons and ethanol released with spray times of 4 to 20 s. Measurements were taken over a period of 24 hours for each test at up to 9 sampling points and tests were conducted at two air exchange rates (0.5 and 1 h⁻¹). The study found that a simple exponential decay model could adequately predict the concentration of aerosol components in a room after a suitable time interval for mixing following spraying.
Singer et al., (2006) quantified emissions and concentrations of glycol ethers and terpenoids from cleaning product and air freshener use in a 50 m³ room ventilated at approximately 0.5 h⁻¹. Five cleaning products were applied full-strength (FS); three were additionally used in dilute solution. These products include one glass/surface cleaner, four general purpose cleaners, and one scented-oil air freshener available for sale in California. 18 experiments were undertaken simulating product use.

The chamber was designed to simulate a typical residential environment. Construction materials include wood framing with plywood underlying the floor, two walls and ceiling. The chamber walls and ceiling were finished with gypsum wallboard coated with low VOC paint. The plywood subfloor was covered with aluminium sheeting. A portion of the floor was covered with vinyl tiles to provide a surface for mopping. A laminate table with top was placed in the middle of the room. Chamber air was mixed with four small axial fans. Ventilation was mechanically provided, with outdoor air passed through a bed of activated carbon to remove organic gases and ozone. The air-exchange rate was fixed and checked during each experiment by measuring the concentration decay of injected SF6 tracer gas. Temperature in the chamber was controlled by thermostatic regulation of the air temperature of the building in which the chamber is contained. Relative humidity was not controlled, but experiments were limited to days in which it was in the range of 40–70% at the start.

Surface cleaning protocols were applied to simulate varied applications including cleaning, degreasing, or disinfecting of hard surface counters, tabletops, stovetops, or glass. Products were applied to a section of the laminate tabletop. Four products were dispensed by spraying and one was dispensed by pipette. Amounts represented high but realistic use rates, based on the results of product use surveys. After application, the product was left undisturbed for approximately one minute. In two experiments, the wetted surface was wiped clean with paper towels. In other experiments, the surface was scrubbed using a wetted and loosely wrung cellulose sponge backed with abrasive material. After scrubbing, the tabletop was dried with paper towels and the sponge was rinsed in a bucket of clean, warm water. Next, water was applied to the tabletop with the sponge to simulate rinsing. Finally, the tabletop was dried with a second set of paper towels. In some experiments, the paper towels were removed from the chamber upon completion of the cleaning activity and in others they were deposited in a wastebasket with plastic liner and remained in the chamber for 24 h. The full application procedure, including scrub, wipe, rinse, and wipe steps was
Measurement of chemical emissions from consumer products completed in approximately 2.5 min to 3.5 min. Materials were weighed before and after each use.

A second protocol represented general use of dilute cleaning solutions. This included solution preparation, wet mopping, and dry mopping of floor tiles. The procedure lasted approximately 7–7.5 min and ended with the removal of all cleaning materials from the chamber. In a further experiment a scented oil air freshener was plugged into an extension cord mounted on one of the table legs. The product container was weighed at intervals over a 73 h period.

VOCs were collected onto sorbent tubes packed with Tenax TA and Carbosieve SIII or with Tenax TA only. Air was drawn directly onto the sorbent tubes, which were fixed horizontally at least 30 cm from the chamber wall. Experimental samples were collected at 1.8–5.5 cm$^3$ min$^{-1}$ using peristaltic pumps periodically over a 24 h period. Samples were collected in duplicate, and pairs were analyzed for at least one to two periods of each experiment. For the air-freshener experiment, samples of 2–5 min duration were collected at intervals over three days beginning with the start of use. Results were expressed as concentrations in chamber air and emission rates for each product and target chemical.

Solal et al., (2008) outline tests of VOC and formaldehyde emission from 32 consumer products available in France: air fresheners, glass cleaners, furniture polishes, toilet products, carpet and floor cleaning products. Experiments were conducted in an emission test chamber according to ISO 16000-9 or in an experimental house. The chamber (called CLIMPAQ) was 0.0509 m$^3$ and operated at 23°C, 50% RH and a specific ventilation rate of 1.25 m$^3$ m$^{-2}$ h$^{-1}$. Tests were also conducted in the bedroom and the toilets of an experimental house, without any furniture, except for the emission test of the furniture polishes when a laminated table was placed in the centre of the room. VOCs and formaldehyde were sampled directly from the air extraction system. Measured concentrations were therefore representative of the pollutant level in the test room, even if in some cases, the distribution of indoor concentrations can be heterogeneous (e.g. in case of combustion processes, use of sprays etc.). VOCs were sampled on Tenax TA and analysed using TD/GC/MSD/FID according to ISO 16000-6. Formaldehyde was sampled on DNPH cartridges and analysed using HPLC according to ISO 16000-3.

Vernez et al., (2004) report measurements and simulations to assess workers' exposure to solvent vapours and aerosols during the waterproofing of a tiled surface. To investigate aerosols formed during
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spraying of a fluororesin based waterproofing product an experiment was conducted in a fume hood whereby the product was sprayed against an aluminium coated surface over which an upward air of flow was directed. Smaller particles, constituting the overspray mist, escaped in the air stream and were measured using a light dispersion-based device, a Grimm Dust Monitor. Solvent emissions were determined in a 13 m^3 stainless steel experimental room with controlled ventilation conditions. The resin mixture to be measured was coated onto a 1 m^2 metallic plate placed on a table in the centre of the room. The solvent concentration was measured in the down stream air for six hours with a direct-reading photoionization detector giving a total VOC concentration, without speciation of individual VOCs. Details of air exchange rate, room construction and temperature and humidity were not provided in the paper.

Wallace et al., (1987) reported chamber tests of VOC emissions from building materials and consumer products in the USA, including cleaners and a spray pesticide. They employed a 34 m^3 chamber with aluminium walls. The air flow was directed from floor to ceiling. Cleaning agents (steel wool pads, liquid detergent, scouring powder) were used to clean table surfaces in the chamber and the insecticide spray was applied to the table surface in simulated use experiments. The air change rate was 0.6 h^{-1} and the temperature and humidity were monitored and reported. VOCs were collected by pumped sampling onto Tenax sorbent over the 4 h test period and analysis was by TD/GC/MS. The authors comment that the average concentrations determined by the study would have been lower than the peak values that would have occurred during or shortly after the product was used. Also they consider that the ratio of surface area cleaned to volume of the building would be less in a real scenario than used in the chamber test.

Wang et al., (2006) investigated the chemical composition of particulates (PM_{2.5}) released from incense burning. Experiments were conducted in an 18.26 m^3 stainless steel environmental test chamber maintained at 23°C, 50% RH and with an air exchange rate of 0.5 h^{-1}. Inlet air was passed through activated charcoal particle filters and HEPA filters. Mixing fans were installed at the ceiling of the chamber to ensure adequate air mixing. Prior to the incense burning, the chamber was conditioned for about 4 h at the standard test temperature, RH and air exchange rate. PM_{2.5} samples were collected on 47 mm teflon membrane and Whatman quartz microfibre filters with the sampling position at head height (1.5 m). Ten different incenses were tested with a burn time of 25 to 50 minutes. Air sampling continued for one hour after burning was complete.

Report by the Institute of Environment and Health and ANSES for EPHECT Project, WP5
Wooley et al., (1990) determined release of ethanol during use of liquid laundry and hand dish washing detergents. The motivation was to quantify emission to ambient air and the impact on photochemical air pollution rather than indoor exposure and possible health risks. A 20 m$^3$ stainless steel chamber with controlled air flow (1-2.5 h$^{-1}$) and internal mixing fans was used to simulate use of the cleaning products. Dish washing involved hand washing of a selection of plates and cups and the laundry test involved use of a washing machine installed in the chamber. Two scenarios of typical and high product use were carried out. Ethanol concentrations were determined with an infra red spectrometer.

The 16 papers outlined above that describe product tests using large chambers and test rooms cover a wide variety of test conditions. These range from purpose built chambers with close control of environmental conditions, to rooms in an experimental house where the whole house climate is closely controlled but not the air change rate of specific rooms, to other test rooms where control is more limited e.g. window and door closing to minimise ventilation, control of temperature but not humidity. The studies involved simulated use of the product and some determined concentrations in air at locations considered relevant for the breathing zone of people whereas others only measured the general atmosphere such as the air leaving the chamber. A few studies focussed on particles but most concerned VOCs (including formaldehyde).

**Real and simulated exposure scenarios in uncontrolled environments**

Ho and Yu (2002) measured concentrations of carbonyl compounds in a home and temple in Hong Kong during incense burning under normal room conditions. Fixed site pumped samplers were used that were analysed by GC/MS. No measurements of ventilation rate or other environmental conditions were reported.

Jo et al., (2008) measured concentrations of target VOCs inside 10 vehicles with and without a gel type air freshener. Two groups of five cars (gasoline-fuelled and diesel-fuelled passenger cars) were used in the study. Each car was tested twice by a passenger in the car using pumped sorbent tubes during the evening commute period: the first one was with an air freshener and the second one was without. After the first test, which was conducted at least two days after the
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placement of the air freshener, the windows of the automobile were left open for a minimum of two hours. The authors report that possible confounding parameters include adsorption, condensation, and inhalation by the technicians present in the car during sampling as well as other VOCs from materials and traffic exhaust.

Lamas et al., (2010) developed a method for measuring fragrance chemicals in indoor air and applied it to their determination in nine indoor environments in Spain where aerosols, electrical diffusion units, as well as different common cleaning products were used. The method entailed pumped sampling through florasil sorbent, ultrasonic assisted solvent extraction and analysis by GC/MS. Lamas et al., (2010a) report development of a modified procedure involving concentration with an SPME fibre and report measurements in some indoor environments including 10 where air fresheners were used according to manufacturer’s instructions. Details of the sampling strategy applied are not given in the papers.

Long et al (2000) addressed the temporal variability of indoor particles characteristics and sources through a comprehensive follow-up study in nine homes. Continuous indoor and outdoor size distribution and PM$_{2.5}$ mass concentration data were collected using a TEOM. These data were supplemented with time-integrated fine particle speciation measurements using Harvard Impactors, including elemental/organic carbon (EC/OC) concentrations. Real-time size distribution measurements were made using two particle-sizing instruments: the SPMS and the aerodynamic particle sizer. In addition, semi-continuous air exchange rates and time-activity information were collected for each home throughout each sampling period. Scripted indoor activities -- including cooking, cleaning activities, resuspension events, ventilation changes, and household appliance usage -- as well were performed to assess the individual contributions of specific indoor activities to indoor particle concentrations.

Madany and Crump (1994) report measurements of VOCs and carbonyls at a fixed location within a normally furnished 36 m$^3$ office during burning of an incense product that was also investigated in a small chamber (see above). Windows and doors were kept closed but no measurements of air exchange rate were available.

Ramirez et al., (2010) report the development of a pumped sampling with sorbent tube method for determination of synthetic musk fragrances, preservatives (parabens) and an insecticide (N,N-diethyl-m-toluamide or DEET) concentrations in air. These compounds are contained in a wide variety of consumer products. The preferred
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method was trapping on pumped Tenax sorbent tubes followed by analysis by TD/GC/MS. The method was applied to measure concentrations in a range of outdoor and indoor environments including hairdressing salons. HHCB (galaxolide) was the most abundant compound and appeared in all real samples and at the highest concentrations, ranging from 1256 ng m\(^{-3}\) in the hairdresser's to 5.9 ng m\(^{-3}\) in suburban air. Musk xylene and AHTN (tonalide) were also present in all the samples with a maximal concentration in the hairdresser's air of 766 and 138 ng m\(^{-3}\), respectively. The most abundant paraben was methyl paraben, which was also detected in all the samples with a maximal concentration of 313 ng m\(^{-3}\) in the hairdresser's. Methyl paraben is commonly used in combination with propyl paraben, but was only found in the pharmacy and in the hairdressers. It should also be noted that DEET was only found at a low concentration (2.5 ng m\(^{-3}\)) in the pharmacy air probably because it is present in the formulations of commercial insect repellents. The sampling strategy and details about the indoor environment such as activities during sampling are not described.

As well as undertaking small chamber tests (see 4.2) Salthammer (1991) reported tests of VOC emissions from two air freshener sprays in a model room. The furnished model office had a volume of 64 m\(^3\), a floor covered with textile carpet and walls painted with gypsum board. The air exchange rate was 0.3 h\(^{-1}\) (all windows and doors were closed). 8.1 g of product was sprayed and VOCs measured between 0.5 and 4 h after spraying. No details are provided about sampling location.

In addition to small chamber tests Spruyt et al., (2006) undertook testing of six different air freshener product types in two rooms each, under real life circumstances. The rooms chosen were located in dwellings of laboratory staff and are considered as a random selection. The products were selected based on the TVOC concentration profiles, and considered to be representative of their types. The aim of using a set of air fresheners indoors was to examine what concentrations are actually reached in an average living room while using the product.

Two compound classes were quantitatively determined, (selection of) VOC's on active charcoal, and (selection of) aldehydes on DNPH impregnated sorbents. Before every measurement, a sample was taken of the room air to determine the background concentrations present. The sample concentrations with the background concentrations subtracted represents the increase in concentrations due to the use of the air fresheners. The sampling time was two hours, and the air freshener was used in the first hour. The concentrations are intrinsically related to the inhabitants’ usage patterns. Uncontrolled
Measurement of chemical emissions from consumer products factors like the ventilation rate and sink effect can also influence the concentrations reached in dwellings. The authors comment that concentrations measured are thus snapshots, and that a much larger sample size would be needed to draw statistically founded conclusions that can be extrapolated to a population.

Su et al., (2007) selected the three best selling essential oils in Taiwan and conducted tests on their impact on indoor air quality when used in a bedroom (21.6 m³; air exchange rate 1.8 h⁻¹) and small office (28.2 m³; air exchange rate 1.3 h⁻¹). 300 ml of each essential oil were diluted with 50 ml water for use in an incense evaporator with a burning candle. Before evaporating, 30 minute background sampling was performed to measure background levels of various indoor air pollutants, including CO, CO₂, total VOCs, and PM₁₀, using a continuous monitor. CO₂ and CO were measured by using a Q-track monitor. PM₁₀ was measured by a Dust-track monitor with the detection range within 0.06–5000 mg m⁻³. Total VOC was measured by using a ppb photoionisation detector with the detection range within 0–200 ppm. Airborne microbes were also collected before study. VOCs were measured by pumped Tenax TA and Carboxen sorbent tubes with analysis by TD/GC/MS. Monitoring during evaporating essential oils began after background profiles had been established, and were continuously recorded for at least 3 h for each round of test with triplicate tests completed for each essential oil in each testing space. There are few details about the rooms used for monitoring such as furnishing, temperature and humidity or the location of the sampling instruments in the room relative to the source.

Vincent et al., (1993) measured occupational exposure of 29 cleaning staff to 2-butoxyethanol during use of window cleaning products in offices and for cleaning cars. The liquid product is sprayed manually by the cleaner and then wiped with a cloth. Personal samplers consisting of pumped charcoal tubes were operated for half day shifts. The exposed samplers were solvent desorbed and analysed by GC/FID. Biomonitoring in the form of analysis of urine of the workers was also undertaken.

Vincent et al., (2007) report the development of a method to determine quaternary ammonium compounds in air, including didecyldimethylammonium chloride (DDAC) and benzalkonium chloride (BAC) that are widely used in disinfectants. The method involved trapping of aerosols on XAD-2 resin with subsequent solvent elution and analysis by liquid chromatography coupled with MS (LC/MS) or ion chromatography. The method was applied to measure DDAC in the air.
Measurement of chemical emissions from consumer products of a hospital in France during disinfection activities. No details are provided about location of samplers with respect to the activity and the environmental conditions occurring at the time of sampling.

Wang et al., (2007) report investigations of air pollutants from burning of incense at two temples in Hong Kong during peak and non-peak periods for numbers of pilgrim visitors. The target air pollutants included particulate matter (PM$_{10}$, PM$_{2.5}$), volatile organic compounds (VOCs), carbonyl compounds, carbon monoxide (CO), nitrogen oxides (NO$_x$), methane (CH$_4$), non-methane hydrocarbons (NMHC), organic carbon (OC), elemental carbon (EC), and inorganic ions. Methods included collection of carbonyls by pumped sampling with DNPH cartridges and analysis by solvent desorption and HPLC and collection of VOCs over an 8 h period by Summa canister and analysis by GC/MS. Sampling probes and inlets were placed at head height (1.6 m) and at a distance of 1.5 m from the incense source as this represented the typical separation of the pilgrims and the incense burner.

The 12 papers reporting studies in real scenarios in largely uncontrolled environments cover a range of indoor environments such as vehicles, dwellings and occupational environments. These studies include area monitoring and some also undertook personal monitoring. Most concern air fresheners and incense and report concentrations of VOCs and carbonyls and some report other analytes such as particles and inorganic gases. Some report the background concentration prior to the activity of interest. Given the broad range of products available worldwide and the range of possible exposure situations arising from use of these products the available studies are limited in both number and scope.
5. Indoor air chemistry and product emissions

As well as the primary emissions that are released to air as a result of chemicals present within a product other chemicals, sometimes termed secondary emissions, may be formed by chemical reaction. For some indoor products such as paints the reactive chemistry may be an essential step in the formation of the final product through curing reactions. Other reactions involving emitted chemicals may occur at surfaces or in the atmosphere that could be considered as inadvertent with respect to the product source.

Morrison (2009) describes the various gas phase reactions that can occur in indoor environments. These reactions are of potential importance because the types and amounts of chemicals in air may be changed with respect to the primary emissions from sources. If the resulting chemicals have different properties then they could impact the health and well being of occupants and it is possible that these effects would not be envisaged by a risk assessment of the primary emissions. Therefore improved understanding of indoor chemistry would inform our understanding of occupant exposure but currently the ability to predict occurrence and products of such chemistry is quite limited. Two broad types of reaction are identified; i) homogeneous chemistry that occurs in a phase of uniform composition such as the atmosphere and ii) heterogeneous chemistry that takes place at surfaces. These involve a number of reaction types as follows;

Homogeneous chemistry;

- Gas phase organic oxidation chemistry; ozone
- Gas phase organic oxidation chemistry; hydroxyl radical
- Gas phase organic oxidation chemistry; nitrate radical
- Condensed phase chemistry; oxidation
- Condensed phase chemistry; hydrolysis

Heterogeneous chemistry;

- Ozone and fresh indoor surfaces
- Ozone and soiled surfaces
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- Acid-base chemistry that affects the absorption properties of surfaces.

Nazaroff et al., (2006) reviewed the literature concerning occurrence of indoor chemistry involving chemicals released from cleaning products and air fresheners. They concluded that constituents, especially terpenes and related compounds, can react rapidly not only with ozone, but also with the hydroxyl radical (OH) and with the nitrate radical (NO₃), all of which may be present in indoor air. The chemistry can generate a host of secondary pollutants, some of which are stable and can be measured (such as formaldehyde), and some of which are short-lived or otherwise pose analytical challenges. This latter group, known as “stealth chemicals,” might conceivably contribute to adverse health consequences. These reactions can occur both in the gas phase and also on indoor surfaces. Nazaroff and Weschler (2004) also reported a review of the literature on formation of secondary air pollutants by chemical reaction of chemicals released from cleaning products and air fresheners.

Studies that have considered the role of indoor air chemistry in transforming the primary emissions from consumer products have taken several approaches; i) measurements in chambers with chemicals of interest and reactive gas such as ozone, ii) modelling of reactions to predict products of indoor air chemistry and rates of product formation, iii) measurements in chambers / test rooms using consumer product as source of chemicals with addition of reactive gas.

Bothe and Donahue (2010) investigated the formation of secondary organic aerosol (SOA) when citronella oil candles were burnt in a chamber in the presence of ozone. Their goal was to quantify the SOA in terms of the amount and the timescale for the formation. A large smog chamber consisting of a 10 m³ teflon bag within a temperature controlled room was used within which candles were burnt for a few seconds thereby filling the chamber with ∼10 μg m⁻³ of aerosol mass. The accompanying vapours were oxidised with ozone to observe any subsequent SOA formation that was determined using a SMPS.

Coleman et al., (2008) also studied SOA formation using a small (198 L) teflon lined chamber and several household products that release terpene vapours. Ozone was introduced and particles monitored by SMPS (10-400 nm) and an optical particle counter for particles 0.1-2.0 μm. Three cleaning products were tested: a pine oil cleaner (POC); an orange-oil degreaser (OOD); and a heated, scented-oil air freshener (AFR). Different ozone levels and air exchange rates were applied. The authors comment that in any real environment there would likely be
much greater spatial heterogeneity than in these small-chamber experiments but consider that SOA formation and growth has been measured in realistic settings and has exhibited similar characteristics (size distribution and growth dynamics). This chamber was also used in a study reported by Desaillats et al., (2006) using the same products where the focus was on determination of chemical species and elucidating the chemical reaction mechanisms.

Forester et al., (2009) also used a teflon chamber to quantify formation yields of oxygenated organic reaction products for gas-phase reactions of the hydroxyl radical and ozone with the common cleaning product terpene compounds limonene, R-terpineol, and geraniol. Lamorena and Lee (2008) conducted experiments using teflon bag chambers to identify the emissions from a car air freshener and to identify the formation of ultra-fine particles and secondary gaseous compounds during ozone-initiated oxidations with emitted VOCs. Formation of ultra-fine particles (4.4–160 nm) was observed when ozone was injected into the chamber containing emitted monoterpenes from the air freshener. Particle number concentrations, particle mass concentrations, and surface concentrations were measured in time dependent experiments to describe the particle formation and growth within the chamber. The irritating secondary gaseous products formed during the ozone-initiated reactions include formaldehyde, acetaldehyde, acrolein, acetone, and propionaldehyde. Ozone concentration (50 and 100 ppb) and temperature (30 and 40°C) significantly affected the formation of particles and gaseous products during the ozone-initiated reactions.

Ham and Wells (2011) investigated the products from the reaction of ozone and a liquid pine-oil based cleaner (POC) and other terpene/terpene alcohol mixtures on a urethane-coated vinyl flooring tile. For all mixtures and the POC solution, experiments were performed using the FACS (FLEC Automation and Control System), described in (Flemmer, Ham et al. 2007), with the following initial parameters: ozone was set to 100 ppb, the relative humidity, at 5% and 50%, the flow rate through FLEC, at 300 mL min⁻¹, and the room temperature, at 22 ± 2°C. The ozone/air mix was then regulated to 100 ppb for the air flow through the FLEC. All samples were analysed by GC/MS system. Results showed that the α-terpineol + ozone reaction products were the prominent species that were observed including in the POC/ozone surfaces experiments. Furthermore, α-terpineol + ozone reactions generate the largest fraction of oxygenated products even in equal mixtures of other terpene alcohols.
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Liu et al., (2004) undertook a study of emissions of VOCs from a plug-in air freshener in a room size chamber and studied reactions with ozone. The chamber was 29.8 m$^3$, lined with polished stainless steel and supplied with air cleaned by HEPA filtration (for particles) and VOC sorbent. Fluorescent lighting was outside the chamber and lit the inside of the chamber through a glass screen. Three experiments were conducted with pine-scented air fresheners; two involved addition of ozone to the chamber. VOC emissions and some of the reaction products were identified and quantified using TD/GC/MS or FID analysis of pumped sorbent tubes, on line cryogenic trapping and GC/FID, aldehydes by DNPH derivatisation, denuder and filter collection of particles and semi VOC and particle concentration using spectrometers. Source emission models were developed to predict the time/concentration profiles of the major VOCs (limonene, α-pinene, β-pinene, 3-carene, camphene, benzyl propionate, benzyl alcohol, bornyl acetate, isobornyl acetate, and benzaldehyde) emitted. Gas phase reactions of VOCs from the air freshener with ozone were simulated by a photochemical kinetics simulation system using VOC reaction mechanisms and rate constants adopted from the literature. The concentration time predictions were considered to be in good agreement with the data for ozone and VOCs emitted from the air freshener and with some of the primary reaction products. Systematic differences between the predictions and the experimental results were found for some species. Poor understanding of secondary reactions and heterogeneous chemistry in the chamber was identified as the likely cause of these differences.

Nazaroff et al., (2006) reported investigations of secondary pollutant formation when emissions from three products (air freshener and cleaning products) available on the market in California react with ozone. Fifteen experiments were carried out in a bench-scale chamber (198 L and teflon lined) under well-controlled conditions. Each experiment produced a nucleation event in which substantial amounts of new ultrafine particles were generated as a consequence of the reactive chemistry. Using the same products, eleven experiments were carried out employing simulated use in a room-sized chamber (50 m$^3$, finished with painted gypsum wallboard and with floor of aluminium sheet and areas of vinyl flooring) with a constant air exchange rate of ~1 h$^{-1}$, with (~120 ppb) and without the presence of ozone. These experiments examined the effect of ozone on the primary VOC constituents of the cleaning products and air freshener, as well as the resultant production of secondary pollutants, including formaldehyde, the hydroxyl radical, and fine particulate matter. When terpenes and related compounds in cleaning products and air fresheners were exposed to ozone reactive chemistry occurred resulting in reduced
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concentrations of primary constituents of the products, reduced ozone concentrations, enhanced concentrations of formaldehyde, measurable levels of the hydroxyl radical, and substantial secondary production of particulate matter. These 50 m³ chamber tests were also reported by Singer et al., (2006).

Sarwar (2004) investigated the formation of fine particles through the reaction of terpenes released from five consumer products (air freshener, cleaners, perfume) and ozone. Experiments were conducted in an 11 m³ stainless-steel chamber at two ozone concentrations (background and 100-200 ppb). Inlet air was provided from within the general occupied space of the building and it was not conditioned and thus contained varying amounts of common indoor pollutants, including terpenes, ozone, and particles, but at background concentrations that were very small relative to concentrations in the chamber during experiments. VOCs were measured by sorbent tube followed by TD/GC/MS analysis and particle number and mass were measured by two spectrometers. Results demonstrated that fine particle formation/growth can occur following the application of such products in indoor environments during periods of elevated indoor ozone concentrations and, for some products, even during periods of relatively low indoor ozone concentrations. The results suggest that ozone reactions with terpenes emitted from a range of common consumer products can lead to elevated exposures to particles with diameters <1 µm.

Shu and Morrison (2011, 2012) measured the impact of ozonation of dihydromyrcenol adsorbed onto three surfaces relevant to the indoor environment: PVC, glass and latex paint. Dihydromyrcenol is a monoterpenic alcohol extensively used in fragranced consumer products. An area-specific second-order reaction rate coefficient, associated with the ozone/dihydromyrcenol reaction on surfaces was quantified. Stainless steel sorption tubes packed with Tenax-TA and SPME (65 µm PDMS-DVB Coating StableFlexTM; Supelco) were both used to sample air and collect dihydromyrcenol. Both Tenax-TA tubes and SPME fibres were analyzed by TD coupled with GC/FID system. The carrier gas flow rate was 20 cm³ min⁻¹. The initial oven temperature of 100°C was increased to 250°C at a rate of 35°C min⁻¹. Dihydromyrcenol adsorption isotherms were measured to determine surface coverage. Adsorption experiments were carried out in a temperature controlled chamber (headspace) at 25°C, and at three relative humidity conditions (20%, 50%, and 80%) using a series of 5 L Pyrex bottles with customer modified caps. A plug-flow reactor (PFR) system was used to measure ozone/dihydromyrcenol reaction rates on surfaces. The total volume and (surface area) were 100 cm³ glass
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beads (4000 cm$^2$), 120 cm$^3$ PVC beads (2840 cm$^2$), 120 cm$^3$ painted beads (1880 cm$^2$). The surface area of the Teflon reactor was 283 cm$^2$.

An 8.2 m$^3$ chamber was used to measure ozone-dihydromyrcenol surface conversion rates. It was equipped with painted drywall on all surfaces including the floor and door. The chamber was ventilated with laboratory room air that was filtered through activated carbon and a HEPA filter. The air exchange rate was maintained at 0.6 +/- 0.05 h$^{-1}$. A small fan was placed in the chamber to ensure a well mixed air. The ozone analyser collected samples from either the gas inlet duct or the centre of the chamber. The ozone and dihydromyrcenol were injected, at respectively concentrations of (3.2 +/- 0.1).10$^{12}$ molecule cm$^{-3}$ and (0.06-5.4).10$^{12}$ molecule cm$^{-3}$, at about 1.5 m upstream from the supply register for the chamber, resulting in a mixing residence time of about 40 s. The temperature and relative humidity were those of the laboratory and not independently controlled. The reaction probability range is (0.06-8.97).10$^{-5}$ and this was sensitive to humidity, substrate and mass adsorbed. The second-order surface-specific rate coefficient range was (0.32-7.05).10$^{-15}$ cm$^4$ s$^{-1}$ molecule$^{-1}$ and is much less sensitive to humidity, substrate or mass adsorbed. The authors predict that more than 95% of dihydromyrcenol oxidation takes place on indoor surfaces, rather than in building air.

In their chamber study of emissions of VOCs from diffusive air fresheners Uhde et al., (2011) undertook one test involving addition of ozone into the chamber three days after the diffuser was placed in the chamber. The added ozone amount was low to reflect realistic concentrations (<120 μg m$^{-3}$) and any aerosol generated was analyzed in a size range of 5.6 to 560 nm with a particle counter/spectrometer. Limited aerosol formation was observed with vanilla scented products, whereas a flower scented diffuser produced substantial particle concentrations in the chamber when ozone was added.

Vu et al., (2011) tested an air freshener in a 1 m$^3$ chamber. The chamber had 32 blacklights providing a source of ultra violet light radiation. Temperature and relative humidity were controlled at 20°C and less than 20%, respectively. Each test lasted for about 4 h with all blacklights turned on for UV experiments. The particle number size distribution was measured using a SMPS in order to detect particles ranging from 14 to 723 nm in diameter. Total particle number concentration was also monitored by using an ultrafine condensation particle counter in order to detect particles > 3 nm. The air freshener contained various VOCs including terpenes. There was no particle formation in the absence of UV irradiation whereas photolysis of VOCs occurred forming secondary VOCs and formation of ultrafine particles.
Waring et al., (2008) investigated the formation of fine and ultrafine particles resulting from reaction between terpenes released from air fresheners and ozone produced by portable air cleaners (ion generators without fans (IG)). A set of screening experiments were performed to determine the impact of SOA formation on steady-state particles concentrations. Five IG were operated separately in presence of either a plug-in liquid or a solid air freshener. All tests were performed in a 14.75 m$^3$ stainless steel chamber. Nylon sampling lines were installed approximately 1.5 m from the floor in the centre of the chamber to measure particles and ozone. The 6 mm OD tube lengths were approximately 3.5 m for the particle measurements and 3 m for the ozone measurements. Three oscillating fans were operated in the chamber to ensure that the air was well-mixed. The chamber air exchange rate was measured during all the tests by releasing approximately 3 L of CO$_2$ into the chamber and then monitoring its continuous decay with a TSI model 8551 Q-Trak. Ozone initiates reactions with certain unsaturated organic compounds that produce ultrafine and fine particles (in a range of 4.61-157 nm diameter), carbonyls, other oxidized products, and free radicals. Terpenes and aldehyde concentrations were sampled for one IG; a decrease in the concentration of terpenes and an increase in formaldehyde was observed.

In summary, in addition to the review papers, 13 experimental studies were identified that investigated the reactions occurring between chemicals released from consumer products and reactive gases. These were conducted in a range of chambers including those designed to have inert teflon surfaces, the more standard metal as well as materials used for normal construction of buildings such as dwellings. Their focus is the reactions between terpenes and ozone and the particles and other chemicals such as formaldehyde that may be formed. Mostly the reactions are in the dark and a source of ozone is introduced but one study used UV lamps within the chamber. The potential for production of secondary products is clearly demonstrated and some understanding has been gained about the influence of concentration and test conditions on the reactive chemistry.
6. Discussion

Consumer products are a diverse range of items and the EPHECT project has sought to identify particular types of products with potential to cause inhalation exposure to substances in indoor environments that present a possible risk to human health. To present such a risk there needs to be a significant pathway linking the source of a particular substance to the receptor. In this case the pathway under consideration is the release to indoor air and the presence of the substance in the breathing zone of building occupants. Therefore key to understanding the risks posed by this pathway is knowledge of which substances are present in the source and the inhalation exposure this may cause to occupants. This exposure is a function of the concentration of the substance in the inhaled air of occupants and of the time period for which that concentration is inhaled. Therefore knowledge of the exposure of people is key to undertaking an assessment of the risks to health that may be associated with particular products.

The prime data for assessing inhalation exposure to a substance is continuous data for the concentration of the substance in the breathing zone of persons of interest. Such measurements do not necessarily link the exposure to a particular source as the substance may be present in the air due to a range of sources, including those not related to consumer products. Also such studies are generally practical for only small groups in particular environments and therefore information is limited and not applicable to other population groups and circumstances causing exposure. It is also not applicable to the study of potential exposure arising from a new product / substance as at that stage there may be no relevant groups to monitor.

The risk assessment process therefore necessitates use of other information to assess exposure. This entails prediction of exposure based on information about the source. The more relevant this information is to the actual release of substances in the micro-environment of interest the less the uncertainty about the predicted exposure. As well as information about the source there is a need to understand the behaviour of the substance released to air, such as its distribution, sorption to surfaces and chemical changes. Also critical is the activity pattern of the receptor to understand their proximity to the released substance and the time they are present in a particular microenvironment. These factors can be defined in terms of an exposure scenario and one approach is to define a worst case exposure scenario for the purpose of initial risk assessment and
Measurement of chemical emissions from consumer products depending on that outcome to consider further refinement if appropriate.

A range of modelling tools are available to inform the process of assessing exposure and the uncertainty of the assessment will depend upon the adequacy of the assumptions made about the exposure scenario. Therefore use of measured data relevant to a particular exposure scenario such as the rate of emission of a substance to indoor air under particular environmental conditions will reduce that uncertainty.

Experimental studies that have provided data relevant to understanding exposure to substances arising from consumer products have been summarised in this literature review. There is a gradation in the quality of information provided by the different types of studies reviewed and hence they have been categorised in the following order;

1. Studies of source composition
2. Small chamber studies of product emissions
3. Large chamber / test room / real room studies of concentrations of substances in air arising from use of the product in a defined scenario.

As shown by the review some published papers report undertaking two or all three types of study. All provide useful information but with limitations and they can often be regarded as adequate or fully appropriate for particular purposes.

Studies of *source composition* are used commonly to understand which products have the potential for release of particular substances. This is most appropriate for those products that emit the substance passively and least appropriate where the use of the product entails major change to its properties, the most extreme case being combustion. Different approaches to the study of source composition provide different information as well. Analyses can provide information on total content, extractable content and also concentrations in a static or even a dynamic headspace. The headspace method provides information that has the closest link with small chamber studies, but is most informative for identifying substances likely to be emitted rather than providing quantitative data on the amounts emitted and the time profile of such emissions.

The small chamber approach seeks to provide quantitative data on the rate of release of substances over a defined time period under controlled conditions. If properly controlled with clear protocols and in
Measurement of chemical emissions from consumer products combination with quality control and quality assurance procedures it can provide repeatable data that can be applied to compare and categorise some types of indoor products. This has been demonstrated in the case of VOC and formaldehyde emission testing of building and furnishing products where international standards describe appropriate test procedures and these underlay national and industry based schemes for the assessment of emissions to indoor air and the labelling of products based on their emissions under the controlled test conditions. It should be noted however that these tests and associated labelling address the longer term emissions from these types of products. The sources themselves are not intermittent and not as directly related to human activity as in the case for consumer products such as use of sprays and lighting of candles. Therefore this type of approach is most suited to long term, fairly constant sources such as air freshener gels where both the rate of release and the concentration produced in the indoor air are not rapidly changing.

A further limitation of small chambers for some products that also applies to some building products is that it can be necessary to prepare a sample for testing outside of the chamber for practical purposes e.g. application of liquid product to a solid substrate. Some volatile substances will be lost during this preparation process. This may not be of concern for assessing longer term emissions but could be relevant if using the small chamber approach to assess emissions immediately following product use. A further characteristic of small (and some large chambers) is that they are designed to achieve full internal mixing and air velocities in the chamber may be higher than in some real indoor environments. This can have a particular impact on the rate of emission from liquid products whereas for solid products where the rate of emission is limited by diffusion within the material this is not an issue. Unclear from the literature reviewed is the possible impact of air velocity on combustion sources and the rate and nature of substances thereby released. One study of burning incense and a scented candle indicates that effects of changes in air exchange rate, temperature and humidity do impact the rate of emission and can be compound specific. Some specialised chambers have been used to investigate particle concentrations having regard to the role of air velocity for determining particle deposition.

Large chambers provide the opportunity to use the product within the chamber and thereby capture all emissions during realistic activities. The chamber can be used to achieve full mixing or else it can be specialised to simulate air flow in particular rooms. It provides well controlled environmental conditions that can be repeated for other tests. Within a large chamber it is possible to sample at different
Measurement of chemical emissions from consumer products locations and thereby measure concentrations more relevant to those in the breathing zone (of product user, bystander or child). Depending on ethical issues it is possible for people to use the product in the chamber and undertake personal sampling. Otherwise various devices for product release and manikins can be used to simulate this use quite closely.

The conditions of test need to be defined in order that the test represents the emission scenario/s most appropriate for providing data for conducting a product risk assessment. This includes selection of products for testing that are representative of those used in different countries and defining protocols for use of the product during test that are relevant to use in practice by the consumer e.g. frequency and duration of use. Information of this nature is being collected as part of the EPHECT project through a comprehensive market survey of consumer product use in 10 European countries.

Most chambers are designed to minimise possible sorption of substances to surfaces. Most commonly this is by careful selection of wall materials, such as use of polished stainless steel and it is empty of furnishings. Such chambers can be furnished/ lined specifically to investigate such ‘sink’ effects although emissions from these materials must be taken into account when evaluating the data. Usually the chambers have an air supply that is filtered to remove particulates (removal depending on type of system) and is treated by passing through a charcoal bed or similar to remove VOCs. Generally consideration is not given to other air components such as nitrogen dioxide and ozone. Also most chamber tests are undertaken in darkness or without any purposeful simulation of daylight and lighting in real rooms.

The combination of ‘clean’ input air, inert surfaces and poor simulation of lighting means that standard chamber tests provide an environment within which chemical and physical changes in the primary emissions that may occur in real indoor environments may not be well represented. As shown in the literature review reactive chemicals such as terpenes can undergo chemical reaction and photochemistry can play a role. Specialised chambers to understand these reaction mechanisms and the substances produced have been applied in some studies and these often use teflon or teflon lined enclosures. The implication of these reactions for assessing exposure to substances in indoor air remains an area of active research and implications for health relevant risk assessment remain unclear.

A further approach is use of real rooms for product testing that have partially controlled conditions e.g. temperature, and other conditions
Measurement of chemical emissions from consumer products may be measured and reported. From the literature important factors such as air exchange rate are in practice not always determined, probably because of resource issues. Use of such real rooms provides the opportunity for assessment under real conditions of use but the lack of control of all factors means that it may not easily be repeated or reproduced in other rooms and the role of particular factors such as air change rate in determining the exposure may not be elucidated. Tests can also be conducted in real rooms with no control of environmental conditions; this could be to undertake measurements during normal use of a product with no interference from researchers about how the product is used. This can be valuable information and provides a basis for comparison with the tests under controlled conditions to check that the scenarios being used in such tests reflect those occurring in practice.
7. Conclusion

Chamber tests involving measurement of emission of substances under controlled environmental conditions offer the most appropriate means of obtaining measured data for the rate of emission of substances from consumer products into indoor air and the concentrations arising from product use.

For those products where exposure arises from consumer interaction, large chambers that provide the possibility of release from the product according to a defined protocol with measurements at locations appropriate to the breathing zone of receptors are the most appropriate approach.

Testing should be undertaken according to clear protocols to enable comparison and repeatability of measurements. Standards for testing emissions from building products provide a basis for such tests but would need adaption for application to consumer products. In particular protocols for simulation of product use and strategies for measurement to determine short term peak exposure of users as well as longer term mean exposures of other building users should be addressed. The definition of the test parameters and the selection of products for testing should be informed by a detailed understanding of the market in different European countries and the patterns of use of the products by consumers. Information of this nature is being collected as part of the EPHECT project. Consideration would also need to be given to appropriate target analytes and for example particles and their associated chemicals are important for some consumer product types which are not addressed in current international standards for building and furnishing products.

Limitations of testing should be recognised and consideration given to possible implications for exposure under particular conditions and for particular population groups. Examples are the substances that may be formed by chemical reaction in the indoor environment.

Determination of chemical composition can be informative for prioritising selection of products for chamber testing given the vast range of products on the market. It can also provide data for carrying out tier 1 exposure modelling which is a recognised tool for risk assessment to consider ‘worst case’ scenarios, although there are limitations in its application such as when the chemical release is not correlated with content (e.g. combustion process or formation by chemical reaction).
An objective of this review has been to identify which test conditions and procedures are indispensable for consumer product emission testing. All approaches outlined (i.e. content, small chamber / large chamber and real room combined with modelling) provide useful information but the lowest uncertainty is achieved by those tests that most realistically reproduce the exposure scenario. While field measurements may represent the true exposure the large variety of practices and environmental conditions that can occur means that this type of test does not enable efficient comparison of products or modes of product use. Therefore tests undertaken under controlled conditions in full-scale chambers where the product is used in a realistic manner and concentrations can be measured at locations relevant to the breathing zone of users and bystanders provide the most appropriate means of assessing products. The results of testing under such controlled conditions and according to meaningful protocols defining product use should be compared with measurements in actual room conditions. This would enable the derivation of appropriate assessment factors that could be applied to results of smaller chamber tests as part of the product assessment process.

Some of the studies reviewed have developed their own protocols for product use and defined chamber test conditions. A few have discussed repeatability of testing although none have addressed inter-laboratory reproducibility or compared in-chamber and in-field exposures. There is a need for standardised tests appropriate for each product type to allow comparison of products and evaluation of the emissions. Draft protocols for such tests would need to be subjected to robustness testing to evaluate the effect of factors such as air velocity on emissions e.g. for combustion sources, and air mixing e.g. influence on localised concentrations during spray product use. The processes of preparation of draft standard test methods, robustness testing and validation of international standards for testing emissions from building and furnishing products provides a useful framework for the development of standards for consumer products.
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