Micro-Chamber/ Thermal Extractor (µ-CTE)

Sampling technology for fast screening of toxic organic chemicals from products & raw materials

MARKES international

Micro-Chamber/ Thermal Extractor™

Micro-Chamber/Thermal Extractor

Markes' Micro-Chamber/Thermal Extractor™ (µ-CTE™) is designed to meet the growing demand for fast emissions screening in manufacturing industry and third-party test labs.

The µ-CTE offers a uniquely fast and cost-effective quality control (QC) tool for screening the levels of volatile and semi-volatile organic chemicals (VOCs and SVOCs) coming out of products and raw materials.

Demand for testing hazardous chemicals released (emitted) by construction products and consumer goods has seen rapid growth over recent years. This is driven by regulatory developments in Europe (Construction Product Directive/Regulation [CPD/R], Regulation, Evaluation, Authorisation and restriction of CHemicals [REACH], German and French national laws, *etc.*), the US (the Ca Air Resources Board [CARB] formaldehyde rule, the international Green Construction Code [IgCC] initiative, *etc.*) and in the Far East (*e.g.* Chinese REACH). Some of the latest regulations require both third-party certification and in-house product quality control of emissions for full compliance.

Typical sample types include wood-based products, plastic goods, insulation materials, electronics, foods, tobacco, decorative products, flooring materials, textiles and cleaning products. The µ-CTE is used for both characterising overall emission (odour) profiles and for analysing the type and amount of individual chemicals coming out of each sample material.

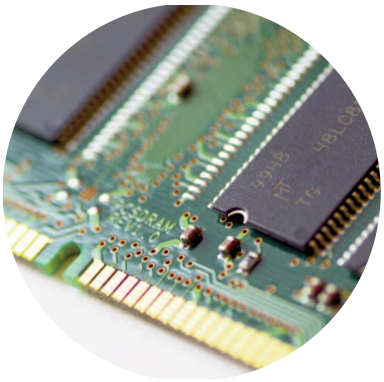
Operating anywhere between ambient temperature and a maximum of 250 °C, the Micro-Chamber/Thermal Extractor comprises four sample chambers which are simultaneously swept with a controlled and constant flow of air or inert gas.

SVOCs and VOCs emitted from samples (surface-only or bulk emissions) are collected on clean sorbent (*e.g.* Tenax®) tubes attached to the exhaust point of each micro-chamber. Pumps are not required. The sorbent tubes are subsequently analysed offline using thermal desorption–gas chromatography (TD–GC/MS).

Alternatively, formaldehyde is collected by connecting standard DNPH cartridges to each micro-chamber. The cartridges are subsequently analysed by liquid chromatography.

"Markes' Micro-Chamber/Thermal Extractor makes it easy to screen chemical emission levels from multiple samples within minutes of production"

T. Schripp *et al.* (2007), *Analytical and Bioanalytical Chemistry*, **387**(5), 1907–1919



Micro-Chamber/Thermal Extractor options

The Micro-Chamber/Thermal Extractor has four chambers (114 mL capacity) and a temperature range of ambient to 250 °C.

It complies with standard methods for emissions screening using micro-scale chambers, *e.g.* draft ISO standard 12219-3, and can also be used for analysis of semi-volatile emissions as described in draft ISO standard 16000-25. All chambers are constructed from inert-coated stainless steel and are compatible with reactive chemicals.

In-house quality control of product/material emissions

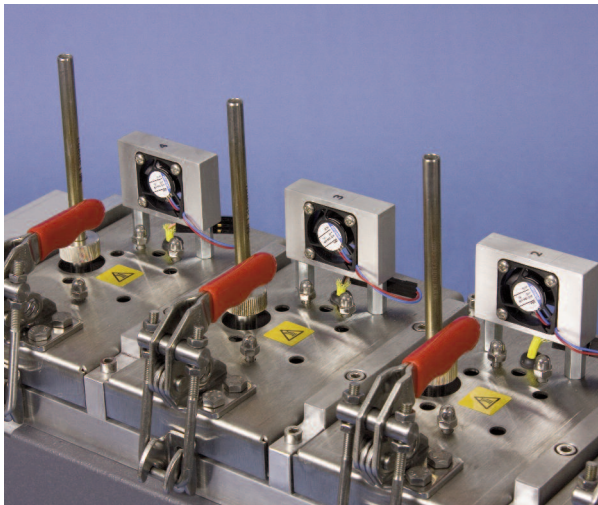
Historically, product certification with respect to VOC/SVOC emissions has required samples to be submitted to accredited independent testing laboratories employing 3 to 28-day reference test methods. While this remains a necessary part of product labeling, *e.g.* for CE marking, these long-term tests are not practical for routine in-house checks of product emissions by manufacturers.

Markes' compact Micro-Chamber/Thermal Extractor is designed to meet the growing demand for fast emissions screening as part of routine industrial quality control. Importantly, it allows cost-effective and meaningful product emissions testing to be carried out near the production line.

Relevant industry sectors include:

- Construction products *e.g.* plasterboard, insulation materials, adhesives, flooring and structural plastics
- Wood-based products
- Components and materials for constructing and equipping cleanroom fabrication facilities
- Car trim components *e.g.* wood veneers, moulded PVC, adhesives
- Electronics (semi-conductor industry/PC components)
- Carpets and textiles
- Cleaning and personal products
- Paints and decorative materials
- Plastics *e.g.* toys, vehicle trim components, packaging
- Foods, tobacco and natural materials
- Protective clothing and equipment

In addition to the primary quality control function, Markes' Micro-Chamber/Thermal Extractor provides the manufacturing industry with the ideal tool for other important in-house tests. Examples include evaluating emission variations across a product range, testing new (*e.g.* low emission) materials under development, comparing product emissions levels with best-in-class competitors, checking raw materials, profiling fragranced products and verifying emission performance before sending samples for third-party testing.



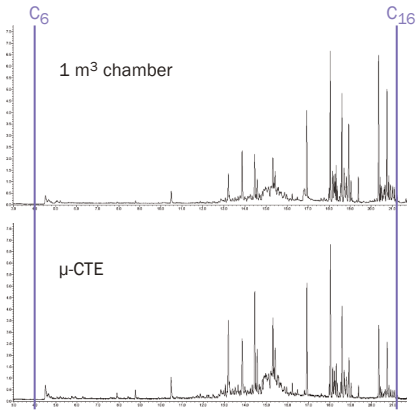
Test laboratory or manufacturer...

Correlation with conventional emission test chamber/cell data

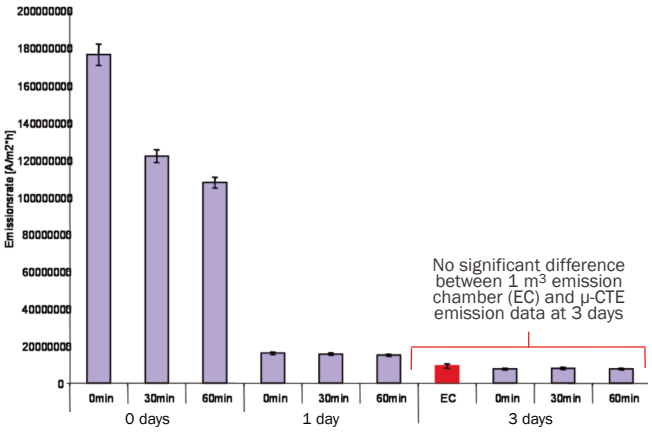
It is possible to use the µ-CTE for standard 3-day emissions testing of homogeneous samples as per standard reference methods. However, the primary purpose of Markes' µ-CTE is to screen emissions of materials soon after production. Several independent comparative studies have shown that µ-CTE emissions data, obtained within a few minutes of sample preparation, correlate with those from longer-term small-chamber reference methods[1–4]. This means micro-chambers can be used to monitor chemical emissions as part of routine QC.

To support this, micro-chamber technology is now the subject of extensive method development for rapid emissions screening within key standards agencies[5–8].

1. T. Schripp, B. Nachtwey, J. Toelke, T. Salthammer, E. Uhde, M. Wensing and M. Bahadir (2007), A microscale device for measuring emissions from materials for indoor use, *Analytical and Bioanalytical Chemistry*, **387**(5), 1907–1919.
2. PARD Report: Correlation between the VDA 276 test and micro-chamber testing. Issued by WMG, University of Warwick, UK.
3. M. Lore, E. Goelen, *et al.* (2010), HEMICPD Report, published by the Belgian Science Policy, Reference D/2010/1191/12.
4. M. Kim (Kangwon National University, Korea) (2010), Presentation to ISO TC146 SC6 WG13 (Document # N0087).
5. ASTM D7706, Standard practice for micro-scale test chambers for rapid assessment of vapour-phase organic compounds (VOC) emitted by materials.
6. ISO DIS 12219-3, Draft standard for screening car trim component emissions using micro-chambers.
7. VDI 2083-17 (proposed ISO), Clean room technology – Compatibility with required clean lines class and surface clean lines.
8. Screening VOC emissions from textile floor coverings (GUT carpet label).



Relative Micro-Chamber/Thermal Extractor correlation for fast emission screening. Data reproduced by kind permission of Prof. Man-Goo Kim, Kangwon National University, Korea



Area-specific emission rates determined for the µ-CTE (blue) and an emission chamber (red) up to 3 days for a building product. Agreement is observed between the two methods at 3 days. Data reproduced by kind permission of the Fraunhofer Wilhelm-Klauditz Institute, Germany

Two modes of operation

The µ-CTE facilitates rapid, low-cost assessment of both bulk and surface emissions.

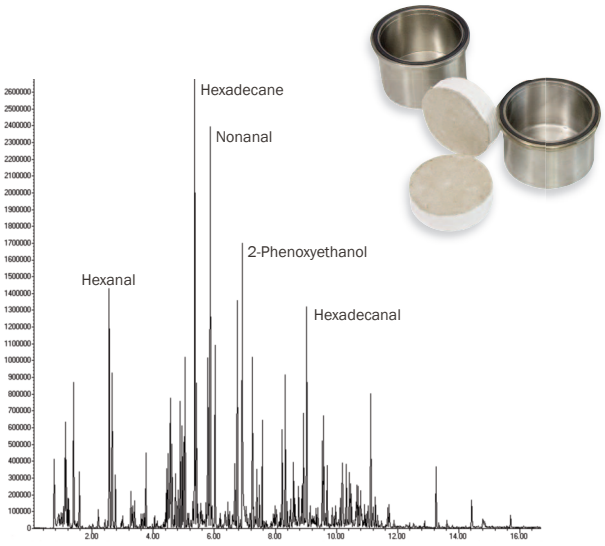
Bulk emissions testing

Samples for bulk emissions testing are placed directly into micro-chamber sample pots. Example materials include polymer beads, foams, liquid test samples, powders, complete small modules (e.g. printed circuit boards, plastic toys and other small molded components) or natural materials such as fresh foods or tobacco. Sample holders are available for resinous or viscous samples.

During bulk emissions testing, clean air passes over and around the entire sample, with organic chemicals being swept from the chamber and collected onto attached sorbent tubes.

Surface emissions testing

In real-world use, some products and materials only have one surface exposed to the indoor (or in-vehicle) environment. In these cases samples may be cut or punched out of the test material so that they fit snugly in the micro-chamber with only the surface of interest exposed.



Screening of emissions from plasterboard (drywall)

Planar materials can be lifted up within the micro-chambers using spacers until they reach the collar projecting down from each micro-chamber lid. This collar is a unique and effective innovation from Markes International, which precisely defines the air volume above the sample and the surface area under test. In the case of rigid materials, it also eliminates interference from cut edges and rear surfaces; only the exposed surface of the test material is accessible to the air/gas flow. The area of exposed sample surface in each of the four chambers is 246 mm^2. Samples of different thickness can be accommodated using appropriately sized spacers.

VOC, SVOC & formaldehyde analysis

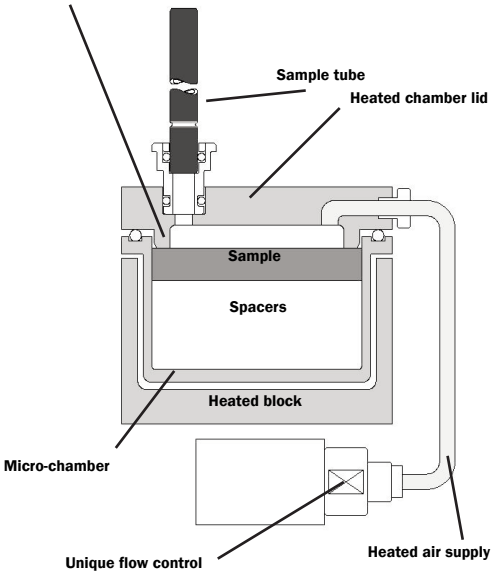
Micro-Chamber/Thermal Extractor operation

When the µ-CTE unit has reached its set temperature, the individual micro-chambers containing correctly-positioned samples are placed into the unit and the lids are sealed. A controlled flow of air or inert gas is passed through all chambers simultaneously. After an equilibration period (typically 20–30 minutes), conditioned sorbent tubes or DNPH cartridges (formaldehyde analysis) are attached to each micro-chamber to begin the vapour sampling process. As the pure air or gas passes over the surface or around the bulk sample, vapours are swept from the material, out of the micro-chamber and onto the attached sorbent tube.

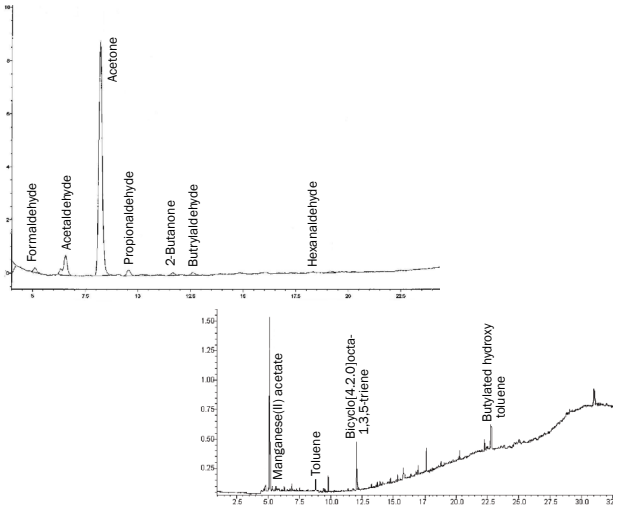
No pump required

Unique technology (UK patent application 0501928.6) maintains a constant flow of air or gas through each micro-chamber regardless of sampling tube impedance or whether a sampling tube is attached. No pump or mass flow controller is required. This makes the system fundamentally easy to use and ideal for routine operation by the manufacturing industry.

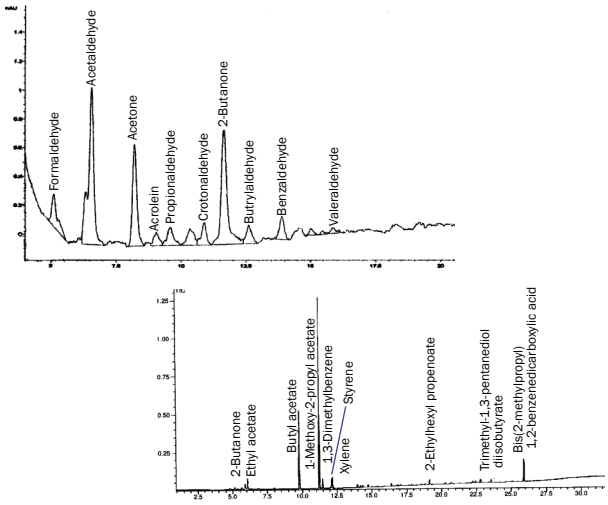
Collar projecting down from the micro-chamber lid defines the exposed surface area and volume of air, and minimises ingress of edge and rear surface emissions



Schematic of single micro-chamber as used for surface emissions testing



Emissions from polyurethane foam car trim component. Formaldehyde (HPLC) chromatogram (top) and VOC (TD–GC/MS) chromatogram (bottom)



Emissions from wood veneer. Formaldehyde (HPLC) chromatogram (top) and VOC (TD-GC/MS) chromatogram (bottom)

Multi-tube format compatible

The Micro-Chamber/Thermal Extractor is compatible with the following:

- **(S)VOC:** Industry standard (89 mm long × 6.4 mm O.D.) sorbent tubes and 6 mm O.D. sorbent tubes.
- **Formaldehyde:** DNPH cartridges with a 4 mm 'luer' outlet.

Enhanced recovery of SVOCs

Efficient heating of all micro-chamber components (sample pans, chamber lids, air/gas supply tubing, *etc.*) prevents surface adsorption/condensation and sample-to-sample carryover. Internal surfaces coming into contact with sample vapours comprise inert coated stainless steel to minimise sink effects and accommodate thermally labile species. Micro-chambers are readily removed from the µ-CTE for easy cleaning.

Orientation of the air/gas inlet at right angles to the emitting sample surface maximises turbulence and eliminates areas of still or low-flow air/gas. Surface air velocities are roughly uniform across the sample surface, and range from approximately 0.5 cm/s at 50 mL/min inlet gas flow to approximately 5 cm/s at 350 mL/min.

Temperature range

Micro-Chamber/Thermal Extractor units can be operated at ambient temperature or elevated temperatures. Each of the four chambers can be temperature-controlled within 1°C to a maximum of 250°C.

In the case of testing emissions from building materials/products, moderate temperatures (*i.e.* 30–60°C) are used to boost sensitivity and compensate for the relatively small sample size without affecting the correlation with data from conventional chambers/cells at ambient temperature. Typical equilibration times range from 20–30 minutes for VOCs, with subsequent vapour sampling (15–20 minutes) at 50 mL/min. These conditions allow four samples to be processed every hour.

Formaldehyde monitoring, *e.g.* according to ISO 16000-3 or ASTM D5197, typically requires much larger volumes of vapour to be sampled (*e.g.* 250 mL/min for 2–4 hours) and may require humidification of the inlet gas supply. Throughput in this case is therefore four samples every 2–4 hours. The µ-CTE can also be operated at higher temperatures and flow rates for extended periods, for example when testing the emission of semi-volatile 'fogging' compounds from car trim materials or electronic components.

Offline analysis of trapped vapours

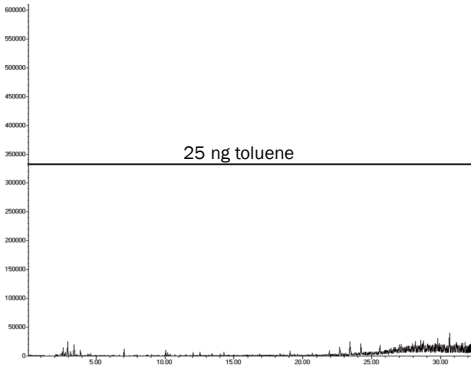
After vapour sampling, trapped organic vapours undergo analysis by thermal desorption (TD)–GC(MS), as per standard methods ISO 16000-6, ISO/EN 16017-1, ASTM D6196, *etc.* Alternative analysers, for example systems combining TD with process MS or e-nose detectors, may also be applicable in some cases. The analytical process is carried out offline, allowing a fresh set of samples to be introduced to the µ-CTE while analysis of the previous set is ongoing. Offline operation also allows chemical analysis by third-party laboratories if preferred.

Construction products – from plasterboard to grout

Key performance criteria

Blank profile and sink effects

Blank profiles from µ-CTE units show low/sub-ng quantities of individual VOCs, and low levels of total VOC (TVOC) background, even at elevated temperatures. This satisfies the most stringent requirements of relevant standard methods.



Blank profile from the µ-CTE at 200 °C. TVOC <12 ng

Real-world applications

Markes' Micro-Chamber/Thermal Extractor units are used extensively for testing VOC and SVOC emissions from construction products and car trim components.

A wide range of construction materials and car trim components have been successfully analysed using the µ-CTE.

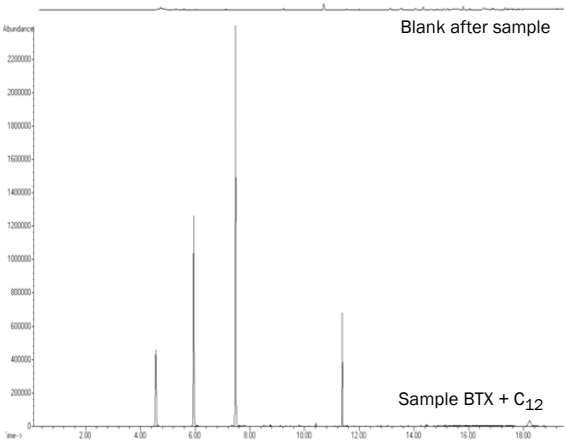
These include adhesives, wood-based panels, laminate and resilient flooring materials, polyurethane foam, PVC, textiles, plasterboard, timber and carpeting. Semi-volatile emissions can also be evaluated in two steps as described in ISO 16000-25.

In addition to these mainstream applications, the Micro-Chamber/Thermal Extractor has also proved popular for testing emissions and hazardous chemicals in many consumer goods. Key examples include phthalates in toys and solvents in printed circuit boards.

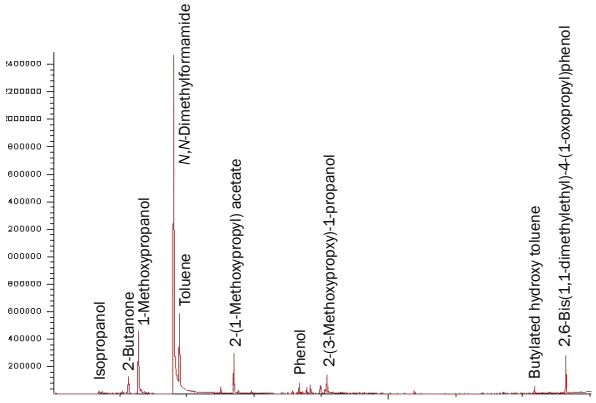
The µ-CTE also provides an adaptable and robust general-purpose sample preparation device, allowing aroma profiling, emissions testing and VOC/SVOC content analysis for a wide range of samples and materials:

Consumer goods – from toys to textiles

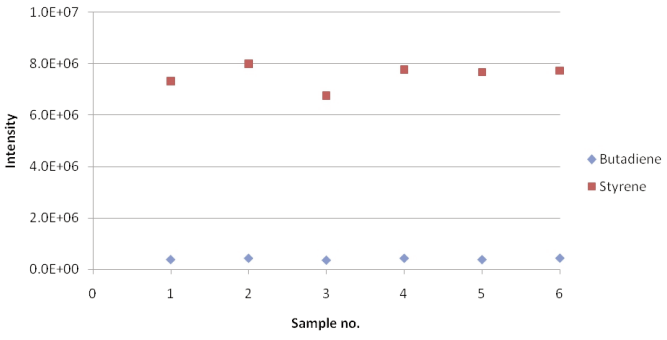
- Tobacco profiling
- Aroma profiling of fresh and prepared foods – cheese, potato crisps, *etc.*
- Aroma profiling of consumer goods (*e.g.* shampoo)
- Characterising the vapour profile of biological samples; animal waste products, plant material, GM foods, *etc.*
- Residual solvents in packaging materials
- Effectiveness of coatings to protect buildings and surfaces against chemical attack



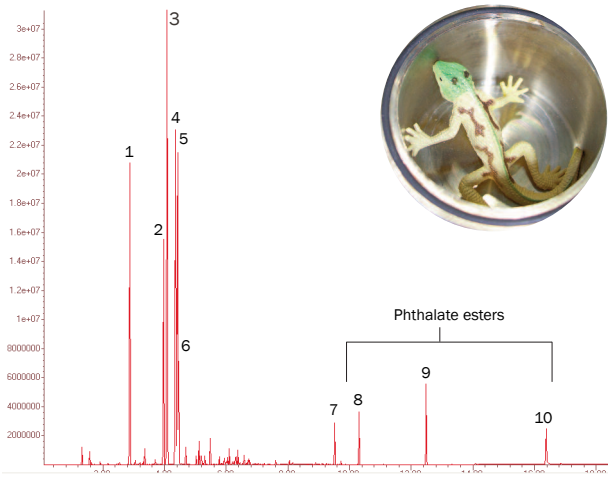
Carryover/sink effects (µ-CTE conditions: 40 °C, 50 mL/min air flow, sampled for 20 min)



Analysis of volatile and semi-volatile emissions from car trim materials. In this case, unusually stringent conditions were used (90 °C, 250 mL/min air flow and 30 min sampling time)



Precision of µ-CTE chambers for ABS terpolymer samples showing responses for 1,3-butadiene and styrene

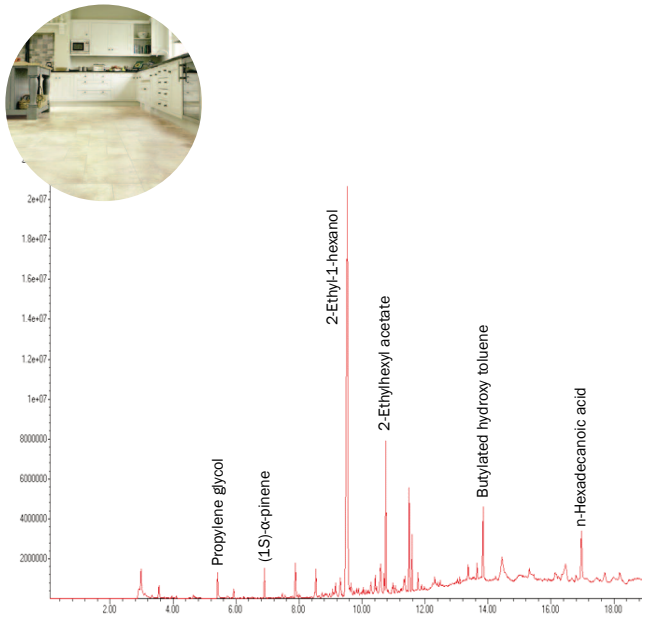


1 Toluene
2 Ethylbenzene
3 *p*-Xylene
4 *o*-Xylene
5 Cyclohexanone
6 2-Butoxyethanol
7 Tricyclodecane
8 Diethyl phthalate
9 Dibutyl phthalate
10 Dioctyl phthalate

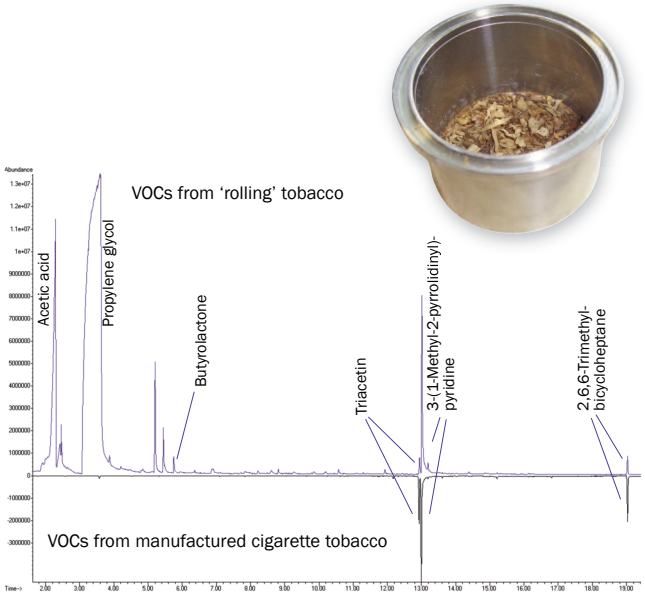
A typical chromatogram for plastic toy animals; the compounds range in volatility from C_7–C_{24}

Trademarks

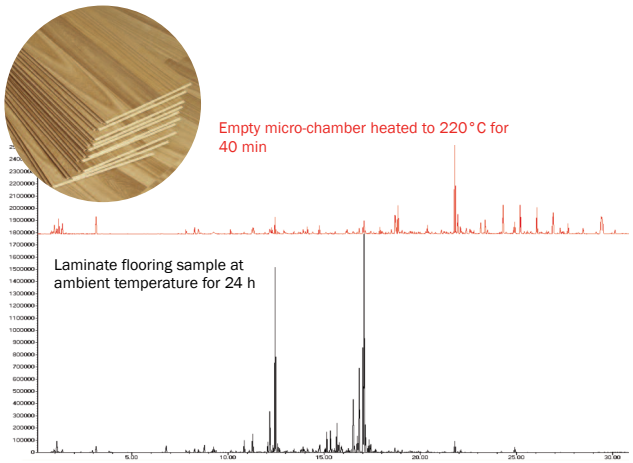
Excellent tool for emissions testing – R&D through to routine QC



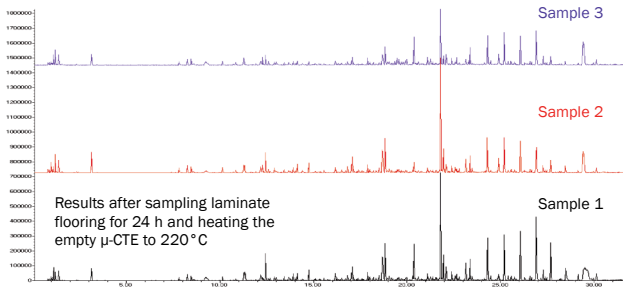
Surface emissions from a sample of vinyl flooring and adhesive tested using the µ-CTE at 40 °C with 50 mL/min air flow for 20 mins



Vapour profiles from 'rolling' tobacco (top) and manufactured cigarette tobacco (bottom), collected on Tenax tubes using a µ-CTE at 50 °C



Step 1: Sample of laminate flooring analysed in 2 steps according to ISO 16000-25



Step 2: ISO 16000-25 test carried out on 3 replicate samples of the same laminate flooring (area 4 × 3 × 0.8 cm)

"An industrial QC laboratory could use the µ-CTE to establish an acceptable (control) level of emissions for products/materials straight from the production line, which subsequently go on to pass formal 3- and 28-day certification tests."

T. Schripp *et al.* (2007), *Analytical and Bioanalytical Chemistry*, **387**(5), 1907–1919

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