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™

M-CTE120

M-CTE250

Micro-Chamber/Thermal Extractor

The Markes Micro-Chamber/Thermal Extractor (μ-CTE™) units are 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.

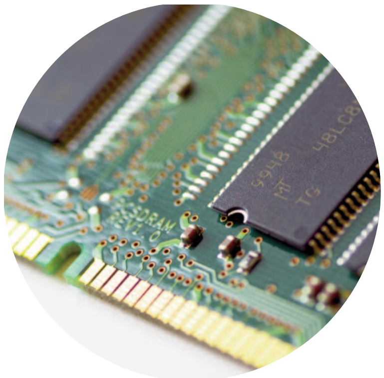
Operating anywhere between ambient temperature and a maximum of 250ºC, Micro-Chamber/Thermal Extractor units comprise either four or six sample chambers which are simultaneously swept with a controlled and constant flow of air or inert gas.

SVOC and VOC 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.

Accessories are also available to allow the μ-CTE to be used for studying vapour permeation through materials such as protective gloves/clothing or fuel system liners.

"Markes' Micro-Chamber/Thermal Extractor makes it easy to screen chemical emission levels from multiple samples within minutes of production" [1]



Micro-Chamber/Thermal Extractor options

The Micro-Chamber/Thermal Extractor is available in two versions:

- Six-chamber (44 mL capacity). Temperature range: ambient to 120ºC. Available with or without toggle valves. The toggle valve option allows gas flow to unused micro-chambers to be turned off, minimising gas consumption.
- Four-chamber (114 mL capacity) Temperature range: ambient to 250ºC.

Both versions of the µ-CTE comply with standard methods for emissions screening using micro-scale chambers *e.g.* draft ISO standard 12219-3. The higher temperature unit 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 µ-CTE units are designed to meet the growing demand for fast emissions screening as part of routine industrial quality control. They allow cost-effective and meaningful product emissions testing to be carried near the production line.

Relevant industry sectors include:

- Construction products *e.g.* plasterboard, insulation materials, adhesives, flooring and structural plastics
- Wood-based products
- Components & materials for constructing & 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 & natural materials
- Protective clothing & equipment

In addition to the primary quality control function, Markes' Micro-Chamber/Thermal Extractor provides 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.

1. "A microscale device for measuring emissions from materials for indoor use", T. Schripp *et al.* (2007), *Analytical and Bioanalytical Chemistry*, **387**(5), 1907–1919

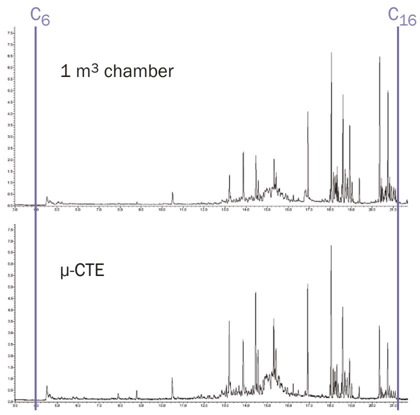
Test laboratory or manufacturer...

Correlation with conventional emission test chamber/cell data

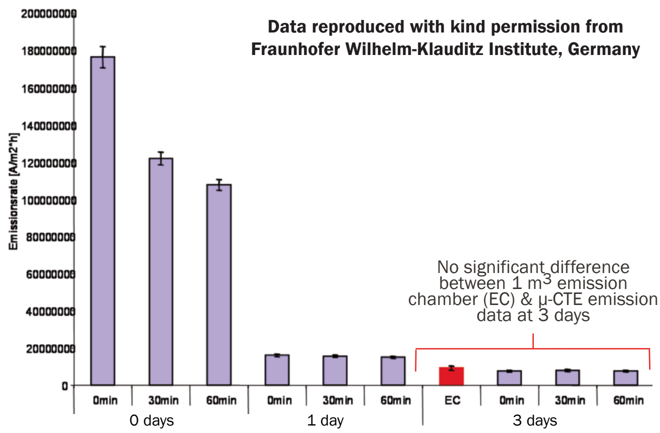
It is possible to use the μ-CTE for standard 3-day emissions testing of homogeneous samples *per* standard reference methods. However, the primary purpose of Markes' μ-CTE is to screen emissions of materials soon after production. Several independent comparative studies have shown that μ-CTE emissions 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), *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 with the 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 2 methods at 3 days

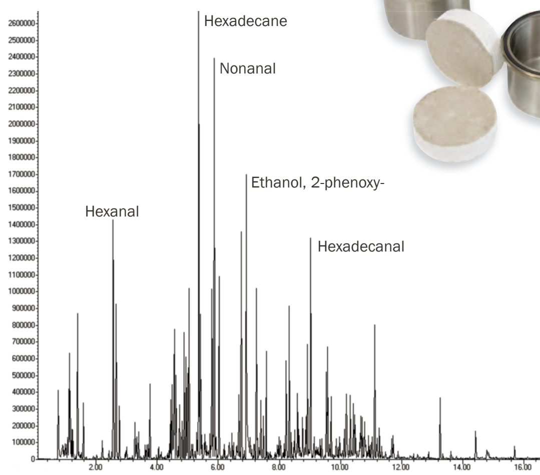
Three modes of operation

The μ-CTE facilitates rapid, low-cost assessment of both bulk and surface emissions, and can also be used for evaluating the permeability of barrier materials such as latex for rubber gloves or treated fabric for protective clothing.

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.



Screening of emissions from plasterboard (drywall)

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 snuggly in the micro-chamber with only the surface of interest exposed.

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 the four- and six-chamber μ-CTE units is 246 mm^2 and 128 mm^2, respectively. Samples of different thickness can be accommodated using appropriately sized spacers.

Permeation testing

Micro-chamber permeation accessories can be used with any version of μ-CTE. They allow a section of test material to be stretched and secured/sealed in place, leaving an area of about 6 cm^2 exposed to the permeation chamber. A small droplet of test compound is then introduced into the bottom of the permeation chamber via the septum and without coming into contact with the test material. Each complete assembly is then quickly placed into one of the 4 or 6 micro-chamber positions and the supply of air or nitrogen switched on to begin the test.

Vapour samples can be collected periodically during the incubation period using sorbent tubes, or can be monitored in real-time using one of Markes' near real-time thermal desorption air monitoring systems.

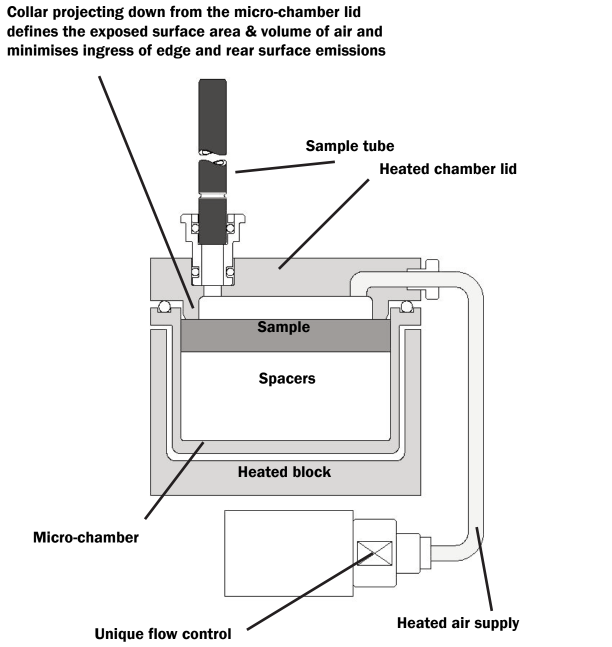
VOC, SVOC & formaldehyde analysis

Micro-Chamber/Thermal Extractor operation

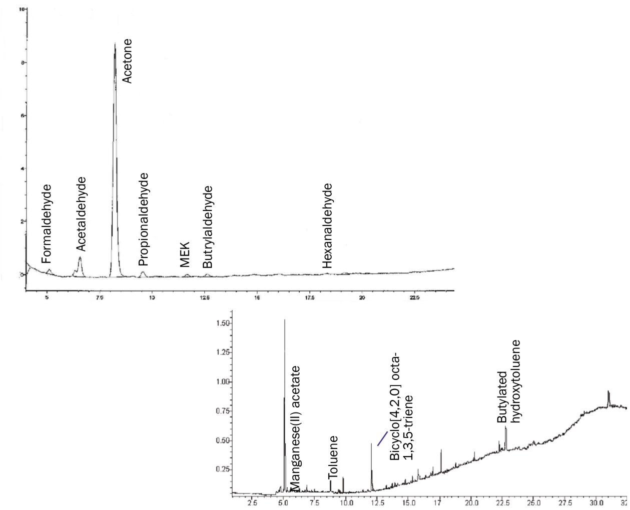
When the µ-CTE unit has reached its set temperature, the individual micro-chambers containing correctly-positioned samples are placed into the unit and the lids are sealed. A controlled flow of air or inert gas is passed through all chambers simultaneously. After an equilibration period (typically 20–30 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

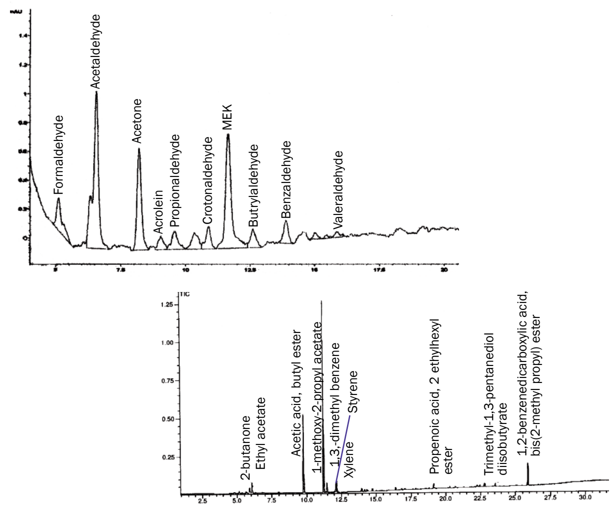
Unique technology (UK patent application 0501928.6) maintains a constant flow of air or gas through each micro-chamber regardless of sampling tube impedance or whether a sampling tube is attached. No pump or mass flow controller is required. This makes the system fundamentally easy to use and ideal for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



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



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



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

	No tube	Single sorbent (Tenax)	3-bed multi-sorbent
Flow rate (mL/min) Low Flow	42.4	42.3	42.1
Flow rate (mL/min) High Flow	346	344	337

Increasing tube resistance

Stability of micro-chamber flow – independent of sorbent tube resistance

Multi-tube format compatible

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

- **(S)VOC:** Industry standard (89 mm long x 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.

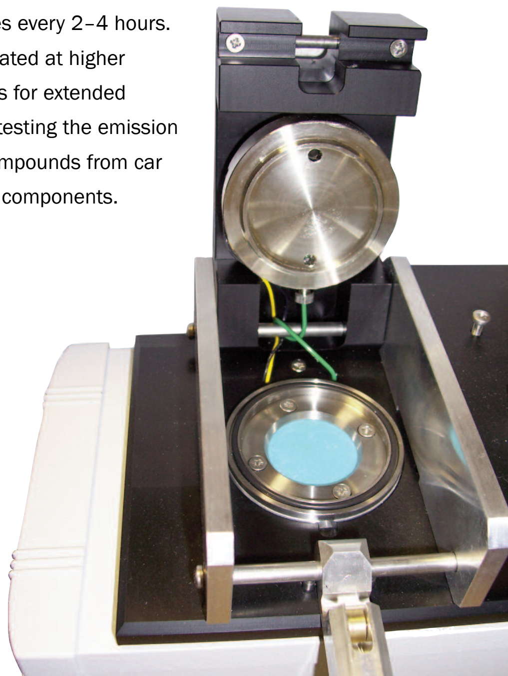
Orientation of the air/gas inlet at right angles to the emitting sample surface maximises turbulence and eliminates areas of still or low-flow air/gas. Surface air velocities are roughly uniform across the sample surface and range from approximately 0.5 cm/s at 50 mL/min inlet gas flow to approximately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

Temperature range

Micro-Chamber/Thermal Extractor units can be operated at ambient temperature or elevated temperatures. Each unit can be temperature controlled within 1°C to a maximum of 120°C or 250°C for the six- and four-chamber units respectively.

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 or six samples to be processed every hour.

Formaldehyde monitoring, *e.g.* per 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 or six 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.

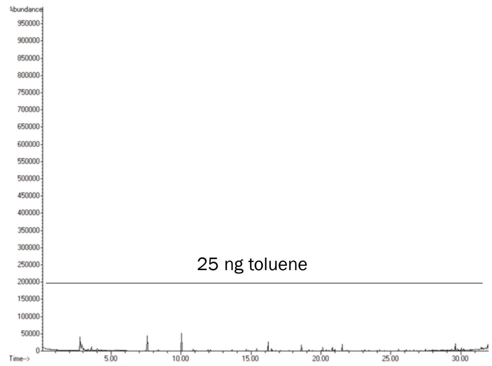


Permeation testing

Construction products – from plasterboard to grout

Offline analysis of trapped vapours

After vapour sampling, trapped organic vapours undergo analysis by thermal desorption (TD)–GC(MS), *per* standard methods ISO 16000-6, ISO/EN 16017-1, ASTM D6196, *etc.* Alternative analysers, for example systems combining TD with process MS or enose 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 on-going. Offline operation also allows chemical analysis by third party laboratories if preferred.

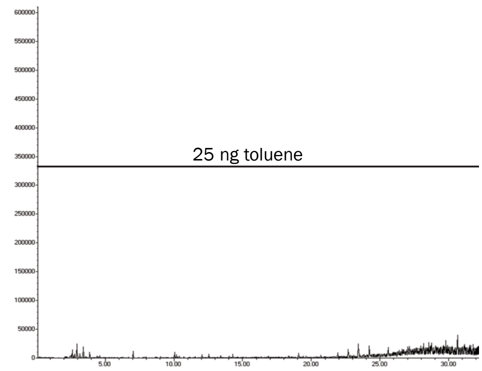


Blank profile from 6-chamber unit at 120ºC. TVOC <30 ng

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 4-chamber unit at 200ºC. TVOC <12 ng

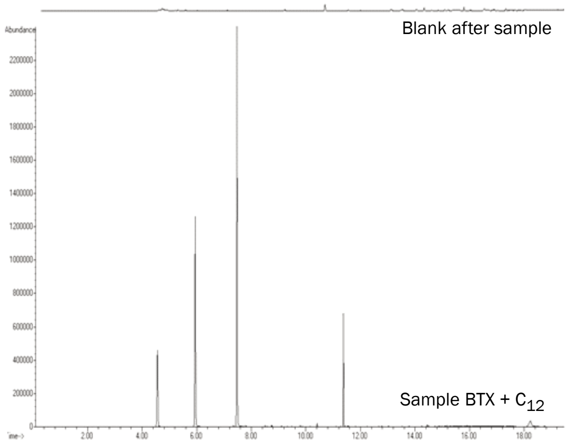
Consumer goods – from toys to textiles

Real-world applications

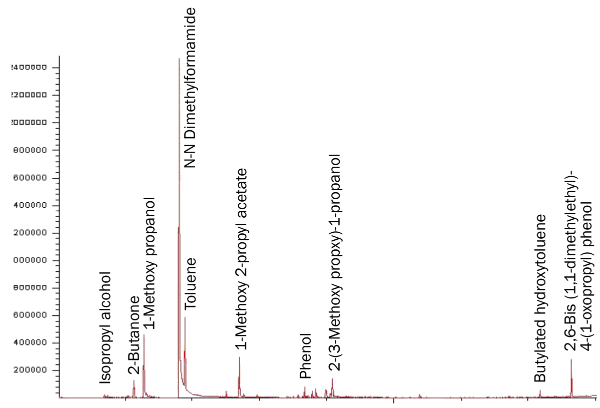
Markes Micro-Chamber/Thermal Extractor units are used extensively for testing VOC and SVOC emissions from construction products and car trim components.

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

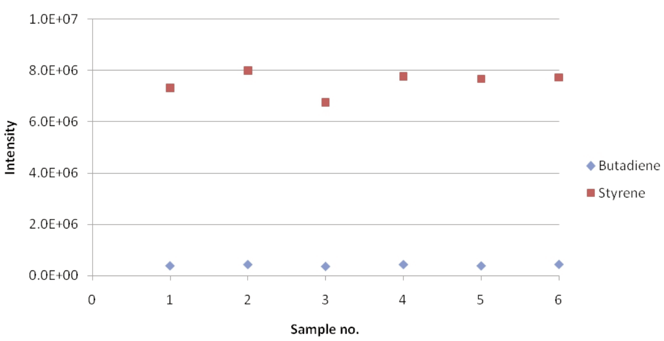
They include adhesives, wood-based panels, laminate and resilient flooring materials, polyurethane foam, pvc, textiles, plasterboard, timber and carpeting. Semi-volatile emissions can also be evaluated in two steps as described in ISO 16000-25.



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)

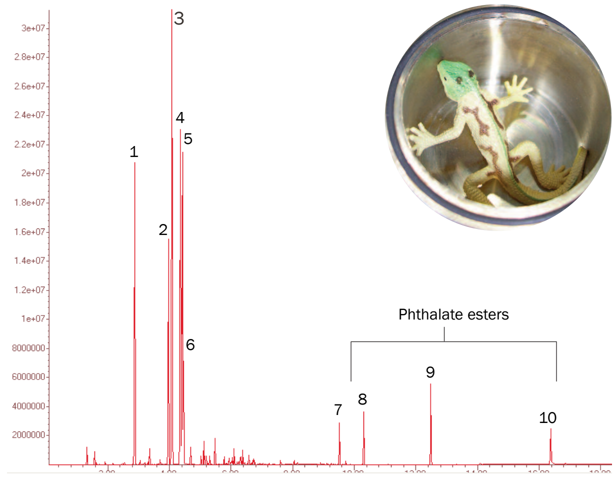


Precision of μ-CTE chambers for ABS terpolymer samples showing responses for 1,3-butadiene and styrene

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:

- 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



1. Toluene
2. Ethyl benzene
3. p-xylene
4. o-xylene
5. Cyclohexanon
6. 2-butoxy ethanol
7. Tricyclodecane
8. Diethyl phthalate
9. Dibutyl phthalate
10. Dioctyl phthalate

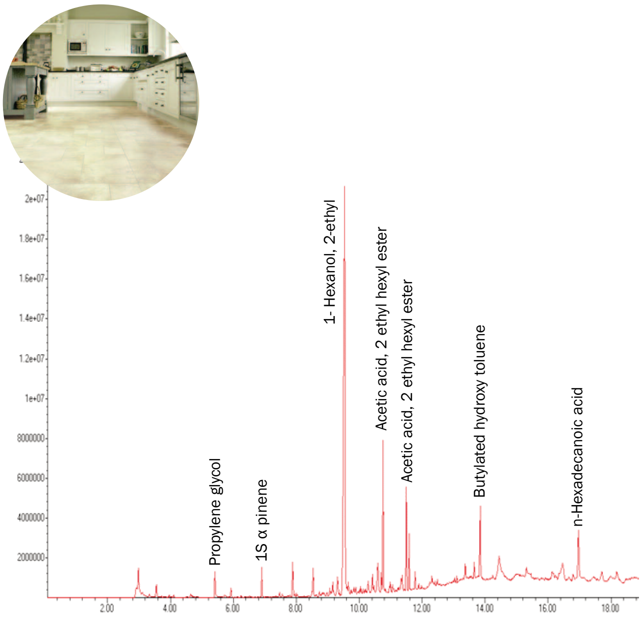
A typical chromatogram for plastic toy animals; compounds ranging in volatility from C_7–C_{24}

Trademarks

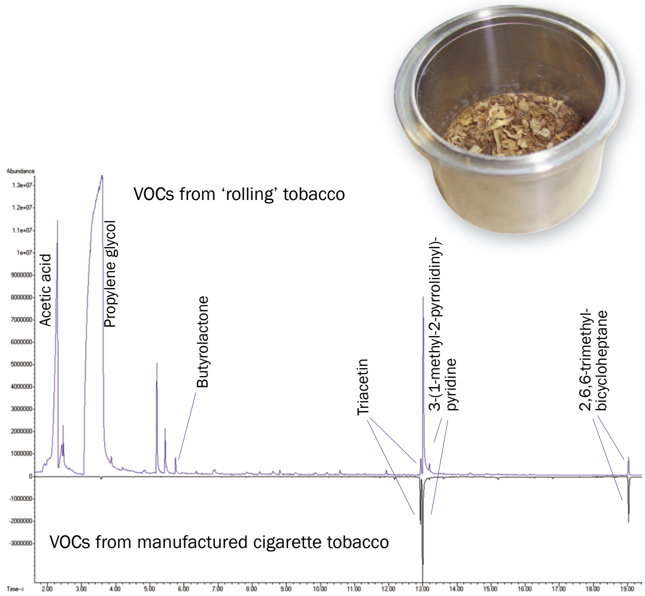
Micro-Chamber/Thermal Extractor™ and μ-CTE™ are trademarks of Markes International Ltd, UK

Tenax® is a registered trademark of Buchem B.V., Netherlands

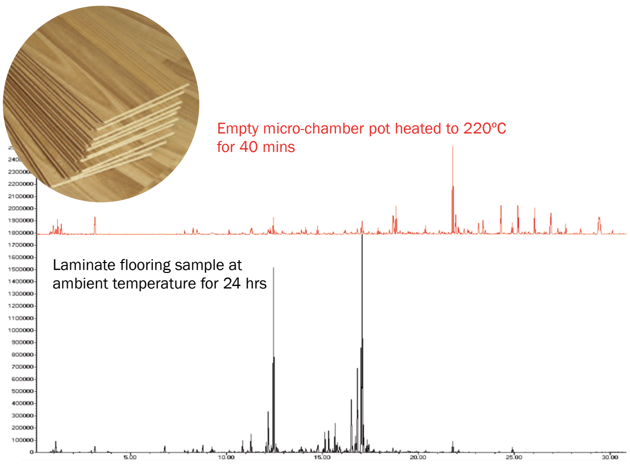
Excellent tool for emissions testing – R&D through to routine QC



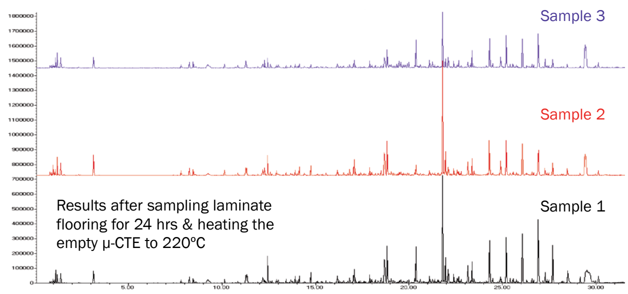
Surface emissions from a sample of vinyl flooring and adhesive tested using the µ-CTE at 40°C with 50 mL/min air flow for 20 mins



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 x 3 x 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)

MARKES international

Humidifier Accessory

Supplies the Micro-Chamber/Thermal Extractor™ with humidified air or gas

The Micro-Chamber/Thermal Extractor is a dynamic headspace unit that is widely used for the screening of chemical emissions from products and materials, typically those used in buildings and vehicles. It is also increasingly being used as a research tool, particularly for odour-profiling of foods, fragranced products and biological samples.

While many of these Micro-Chamber/Thermal Extractor applications employ dry air/gas, there has been rising interest in the use of humidified air/gas. This allows the closer simulation of conditions used in some reference tests, and in real-life scenarios more generally. It also improves the recoveries of some less volatile polar compounds.

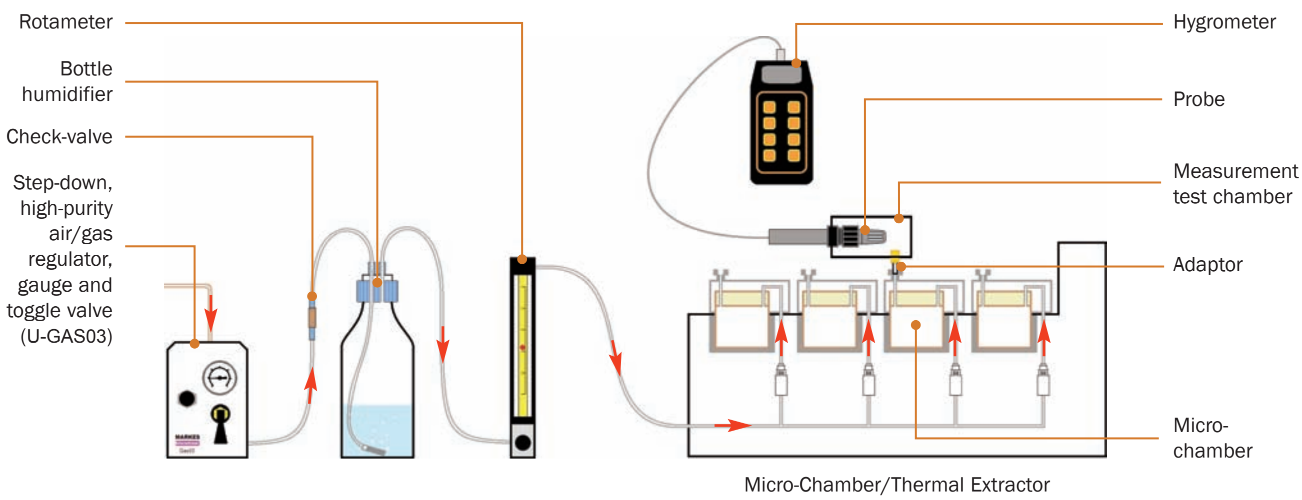
The Humidifier Accessory meets this demand by supplying the Micro-Chamber/Thermal Extractor with air/gas at up to 50% relative humidity at room temperature, improving its value as a tool for emissions testing, routine quality assurance and advanced research.

Product highlights

- Supplies air or gas to the Micro-Chamber/Thermal Extractor at up to 50% relative humidity.
- Allows closer simulation of conditions used in reference tests.
- Enhances the recovery of some less-volatile polar compounds.

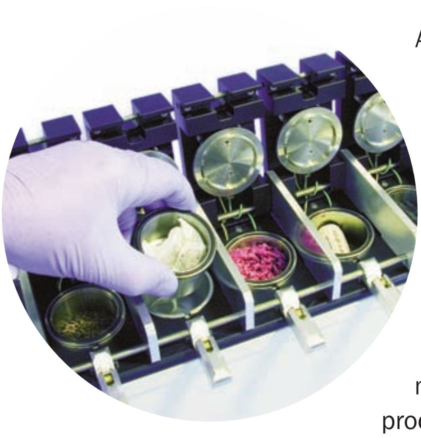
The Humidifier Accessory includes:

- Humidifier assembly, comprising:
  - Bottle humidifier.
  - Check-valve.
  - Rotameter (for control of flow rate).
- Single regulator pneumatics accessory (U-GAS03) to control air/gas supply pressure.
- Hygrometer (for humidity measurement).
- Humidity measurement test chamber.



Schematic of the Humidifier Accessory being used with a Micro-Chamber/Thermal Extractor

Markes' Micro-Chamber/Thermal Extractor



As modern buildings are made more airtight to minimise energy loss, indoor air quality has become an important public health concern. A consequence of this is that construction materials and other products used indoors now often need to be certified with respect to their chemical emission levels. Reference tests for this usually involve placing representative product samples in a test chamber (typically 100 L to 1 m^3 volume) at 23 °C under a flow of humidified air. Released vapours are then trapped on sorbent tubes and analysed using standard thermal desorption (TD)–GC–MS analytical procedures. These reference tests work well, but can take up to a month per sample.

Markes' Micro-Chamber/Thermal Extractor – low-temperature six-chamber model (left) and high-temperature four-chamber model (right)

Complementing these reference tests, Markes' Micro-Chamber/Thermal Extractor allows the quick and reliable screening of chemical emissions from products and materials. It accommodates multiple samples, and in less than an hour can generate results that correlate with those from long-term reference tests. This makes it perfect for routine quality assurance and factory production control in industrial laboratories.

To order the Humidifier Accessory, or to discuss how the Micro-Chamber/Thermal Extractor could benefit your application, talk to our technical specialists by calling +44 (0)1443 230935, or email enquiries@markes.com.

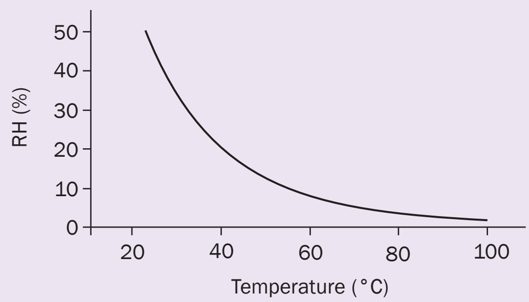
Technical specification

Part number

- M-HUMID-MCTE.

Humidity output

- At 23 °C the relative humidity (RH) output is dependent on the pressure of air/gas supplied:
  – At 14 psig, RH = 50% (±5%)
  – At 20 psig, RH = 42% (±5%)
- A minimum of 30 minutes' equilibration is required to achieve a stable humidity level.
- Relative humidity falls as the air/gas temperature increases. The graph below shows the predicted effect of increasing temperature upon the relative humidity of an air/gas stream at 50% RH and 23 °C.



Inlet gas pressures and sampling flow rates

- All Micro-Chamber/Thermal Extractor units have a low- and high-flow range setting, and can be readily connected to and disconnected from the Humidifier Accessory.
- Without the Humidifier Accessory connected:
  – Inlet air/gas pressure: Up to 60 psig
  ⇨ Uniform flow into microchamber: 10–500 mL/min.
- With the Humidifier Accessory connected:
  – Inlet air/gas pressure: Up to 20 psig
  ⇨ Uniform flow into microchamber: 10–90 mL/min (10–60 mL/min at 50% RH).

Dimensions

- A minimum of 325 mm × 200 mm bench space is required in addition to the Micro-Chamber/Thermal Extractor itself.

Requirements

- Air/gas: High-purity air or nitrogen supply regulated to 14–20 psig using the U-GAS03.
- Water: 350–400 mL high-purity water for the bottle humidifer. Distilled/deionised or HPLC-grade water is recommended.

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