

Rapid Microchamber Tests for Screening Chemical Emissions from Car Trim in Accordance with ISO 12219-3

Abstract

This Application Note demonstrates the efficiency and reliability of Markes' Micro-Chamber/Thermal Extractor to rapidly assess emissions of residual monomer from polymeric car trim components in accordance with ISO 12219-3.

MARKES
international

Introduction

'New car smell' results from the release of volatile and semivolatile organic compounds (VOCs and SVOCs) from car interior materials, such as plastic or wood trim, textiles, glues, and sealants. Many of these chemicals, such as benzene, formaldehyde, and phthalates are suspected to have a detrimental impact on vehicle interior air quality (VIAQ). In extreme cases, they may pose a risk to the health of occupants.

With increasing awareness of VIAQ issues, it is necessary for car manufacturers to monitor and control VOC and SVOC compounds released from interior trim components.

The ISO 12219 series for testing the indoor air of road vehicles describes several methods for testing cabin air quality and measuring emission rates of VOCs and SVOCs from materials used in vehicle cabins. These include:

- **ISO 12219-1: Indoor air of road vehicles—Part 1:** Whole vehicle test chamber—Specification and method for the determination of volatile organic compounds in cabin interiors.
- **ISO 12219-2: Indoor air of road vehicles—Part 2:** Screening method for the determination of the emissions of VOCs from car trim components—bag method.
- **ISO 12219-3: Indoor air of road vehicles—Part 3:** Screening method for the determination of the emissions of VOC from car trim components—micro-scale chamber method.
- **ISO 12219-4: Indoor air of road vehicles—Part 4:** Method for the determination of emissions of VOCs from car trim components—small chamber method.

While the larger-scale 12219-series tests take several hours, Part 3 provides manufacturers with a convenient tool for rapid emissions screening (that is, within minutes). It describes the use of micro-scale chambers that

can accommodate small articles or representative samples of trim. Previous investigations comparing results from micro-scale chambers with emissions data obtained from larger chambers show strong correlation (Figure 1)¹⁻⁴.

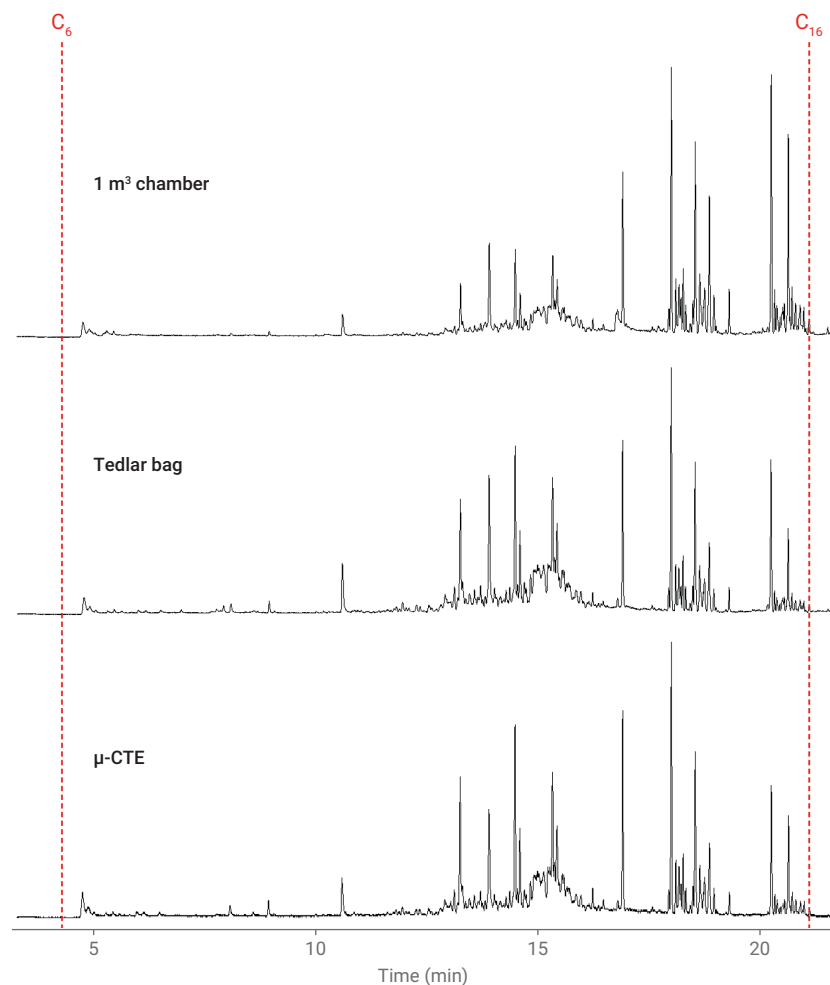


Figure 1. Correlation between chamber, Tedlar bag, and μ -CTE emissions data. Data reproduced with the kind permission of Professor Mangoo Kim, Kangwon National University, Korea¹.

Microchambers are ideal for in-house industrial applications such as:

- (S)VOC emission screening for routine quality control.
- Evaluating prototype low-emission materials/products during development.
- Monitoring product uniformity/conformity between formal certification tests.
- Comparing emissions from products within a range (for example, different colors/patterns).
- Checking the quality of raw materials.
- Troubleshooting customer complaints.



Figure 2. Markes μ -CTE instrument: four-chamber model.

Markes' Micro-Chamber/Thermal Extractor (μ -CTE) is a leading example of a commercial micro-scale chamber apparatus. This Application Note demonstrates the efficiency and reliability of the μ -CTE in qualifying and quantifying the emission of residual monomer from polymer according to ISO 12219-3.

Background to the μ -CTE

Agilent offers the version of the Markes μ -CTE with four 114-mL chambers (Figure 2). It can operate at ambient or elevated temperatures to allow the extraction of vapor-phase organic emissions from various types of car interior trim components, such as textiles, plastic polymers, and sealants. Multiple test specimens can readily be evaluated from the same sample if required. Figure 3 shows schematics of the apparatus in operation for bulk and surface emissions testing.

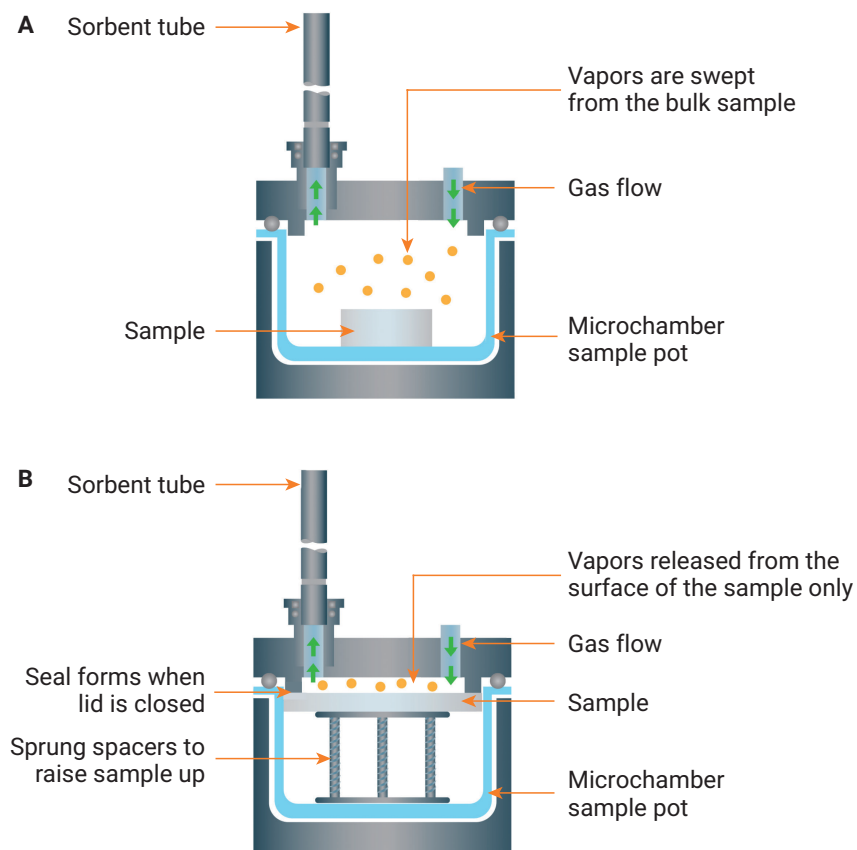


Figure 3. Operation of the μ -CTE for sampling emissions of volatile chemicals from (A) bulk samples, and (B) the surfaces of flat samples.

The chambers are constructed of inert-coated stainless steel to avoid interference and eliminate sink effects. The chambers are supplied with a constant flow (up to 500 mL/min) of dry air or inert gas and, after equilibration, emissions are collected into a connected sample tube. Thermal desorption (TD) sorbent tubes are used for retention of VOCs and SVOCs, and DNPH cartridges are used for formaldehyde.

Tenax TA is the most commonly used sorbent for VOCs ranging in volatility from *n*-hexane to *n*-hexadecane, and tubes packed with two or three sorbents are available to extend this volatility range if needed. Analysis of the sorbent tube is then performed by TD with gas chromatography and mass spectrometry (GC/MS) according to ISO 16000-6⁵.

Experimental

The polymer acrylonitrile-butadiene-styrene (ABS) is a thermoplastic resin widely used in the automobile industry, for example, for interior trim, headlight housings, and grills.

Two irregularly shaped samples of ABS (A and B) were obtained for bulk emissions testing. Sample A was cut into six pieces of roughly 2 cm² using a clean scalpel, with gloves being worn to reduce the risk of contamination. Sample B was supplied in the form of five molded pieces that were small enough to fit directly into the chambers of the μ -CTE. The mass and exact area of each piece were recorded, then the pieces were placed in separate chambers (Figure 4).

A temperature of 65 °C and a flow of helium at 50 mL/min were set, and the system was left to equilibrate for 20 minutes, as specified in ISO 12219-3. A two-bed sorbent tube containing Tenax TA and Carbograph 5TD was then attached and emissions collected for 15 minutes.

Instrument conditions

Parameter	Value
TD	
Instrument	TD100 (Markes International)
Flow path	140 °C
Trap purge time	1 minute
Primary desorption	8 minutes at 250 °C
Focusing trap	-10 °C to 300 °C, 4 minutes hold, 20:1 split
Trap type	U-T12ME (Markes International)
GC/MS	
Column	DB-624, 30 m × 0.32 mm, 1.8 μ m
Pressure	4 psi, constant-pressure mode
Initial flow	2 mL/min
Temperature program	35 °C (5 minutes) then 30 °C/min to 230 °C, hold for 2 minutes
Total run time	13.50 minutes
Carrier gas	Helium
Mass scan range	<i>m/z</i> 25–300
MS source temperature	230 °C
MS quad temperature	150 °C
Transfer line temperature	280 °C

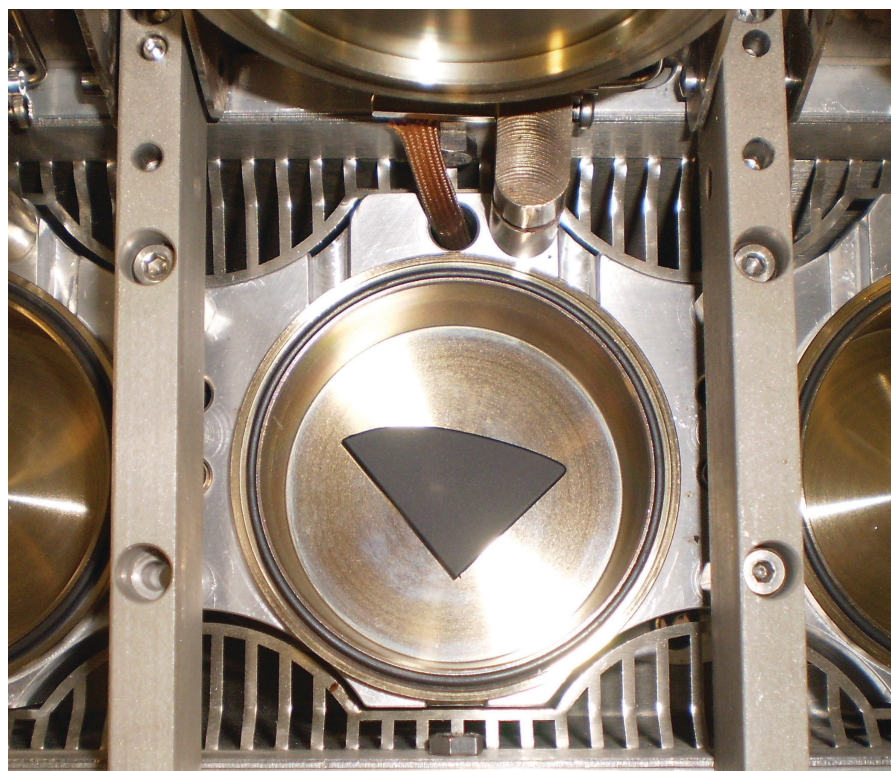


Figure 4. Samples were centered in the bottom of each chamber (with the same surface facing upwards) to ensure equivalent exposures and a uniform flow of gas across the surface.

All tubes were analyzed by TD-GC/MS using a similar procedure as that described in ISO 16000-6. A thick-film capillary GC column, designed for volatiles, was used in this case because of the specific interest in the very volatile component butadiene.

The system was calibrated by introducing known volumes of standard gas into clean sorbent tubes.

Results and discussion

Responses for butadiene and styrene were calculated by integrating the extracted-ion chromatograms (Figure 5).

The extracted-ion data were then used to derive area-specific emissions for Sample A and mass-specific emissions for Sample B (Figure 6), which illustrates the reproducibility that can be achieved when using the μ -CTE for sampling.

Relative standard deviations (RSDs) between 4.6 % and 16.3 % represent exceptional reproducibility within the context of material emissions testing. Moreover, the figure of 16.3 % includes

one obvious outlier; excluding this figure gives an RSD of 4.3 %. Previous interlab and intralab studies using chamber and equipment have shown RSDs in the order of 20–30 % to be more common.⁶

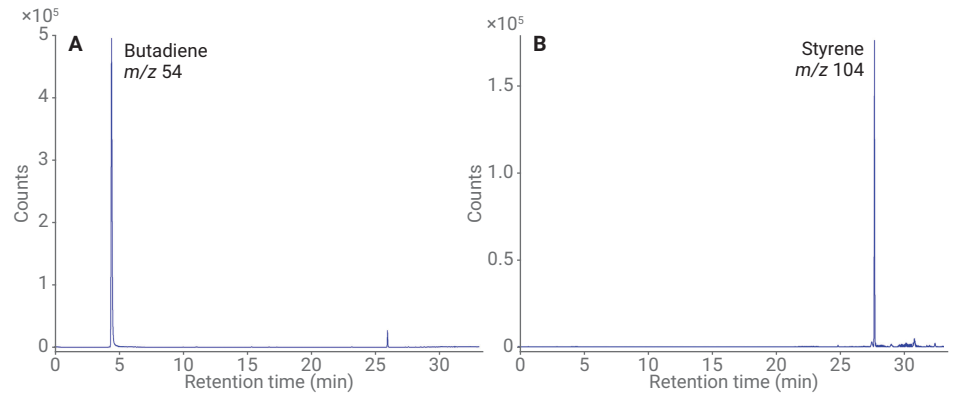


Figure 5. Extracted-ion chromatograms for butadiene and styrene, obtained from Sample A.

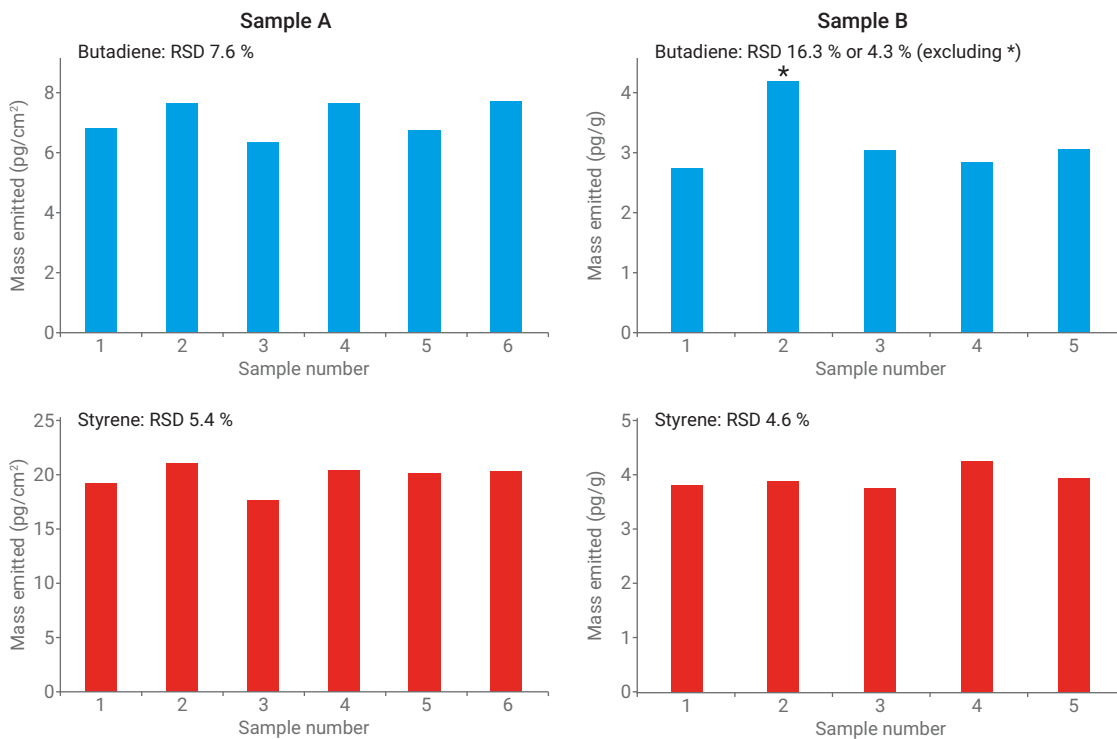


Figure 6. Emissions of butadiene (blue) and styrene (red) emitted from Sample A (mass per cm^2 surface area) and Sample B (mass per gram of sample). The outlying run for Sample B is indicated by *.

Conclusions

The Markes μ -CTE is a fast, straightforward, and efficient tool for material emissions sampling in compliance with ISO 12219-3. The excellent reproducibility shown in this study further confirms the suitability of the μ -CTE for evaluating emissions of monomers and other volatiles from plastics used in car manufacture, such as ABS.

References

1. Kim, M. Kangwon National University, Korea, Presentation to ISO TC 146 SC6 WG13, **2010** (Document No. N0087).
2. Williams, G. J.; Pharoah, M. Correlation between the VDA 276 test and microchamber testing, PARD Report (issued by WMG, University of Warwick, UK), **2009**.
3. Schripp, T.; *et al.* A microscale device for measuring emissions from materials for indoor use, *Analytical and Bioanalytical Chemistry*, **2007**, 387: 1907–1919, <http://dx.doi.org/10.1007/s00216-006-1057-2>.
4. Lor, M.; *et al.* Horizontal evaluation method for the implementation of the construction products directive HEMICPD, Programme to stimulate knowledge transfer in areas of strategic importance (TAP2), Belgian Science Policy, **2010**.
5. ISO 16000-6: Indoor Air – Part 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/FID.
6. Wilke, O.; *et al.* Investigations for the improvement of the measurement of volatile organic compounds from floor coverings within the health-related evaluation of construction products, BAM Federal Institute for Materials Research and Testing, **2009**.

Trademarks

- Micro-Chamber/Thermal Extractor™, μ -CTE™ and TD100™ are trademarks of Markes International.
- Carbograph™ is a trademark of LARA s.r.l.
- Tenax® is a registered trademark of Buchem B.V., Netherlands.

www.agilent.com/chem

This information is subject to change without notice.

© Agilent Technologies, Inc. 2018
Printed in the USA, August 21, 2018
5994-0197EN