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Application

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Application Note 093

Rapid microchamber tests for screening chemical emissions from car trim in accordance with ISO 12219-3

Summary

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.

Introduction

'New car smell' results from the release of volatile and semi-volatile 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), and in extreme cases they may pose a risk to the health of occupants.

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

The ISO 12219 series for testing 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 VOC 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 VOC 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 (*i.e.* 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]

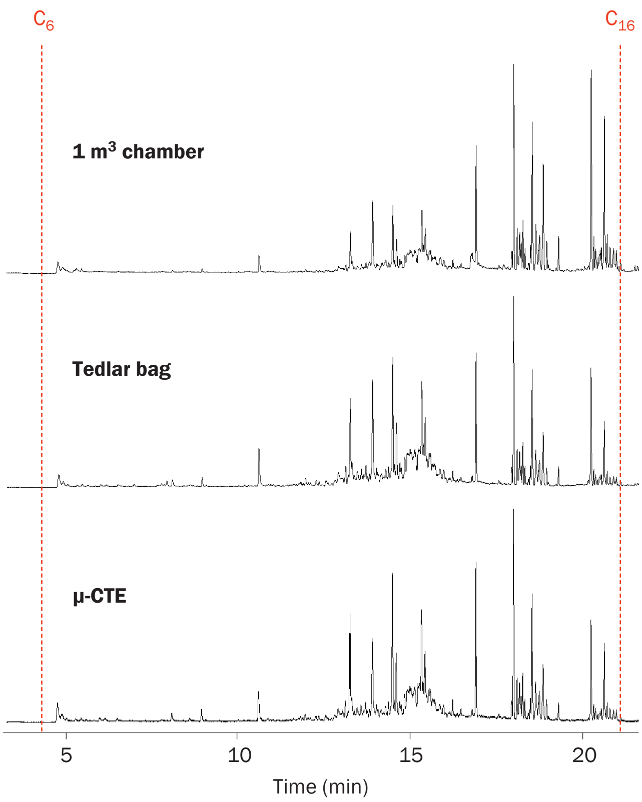

Figure 1: Correlation between chamber, Tedlar bag and µ-CTE emissions data.
Data reproduced with the kind permission of Professor Mangoo Kim, Kangwon National University, Korea.[1]

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 (*e.g.* different colours/patterns).
- Checking the quality of raw materials.
- Troubleshooting customer complaints.

Markes' **Micro-Chamber/Thermal Extractor™ (µ-CTE™)** is a leading example of commercial micro-scale chamber apparatus. In this Application Note, we demonstrate 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

Markes offers two versions of the µ-CTE, one with four 114-mL chambers and another with six 44-mL chambers (Figure 2). Both versions can operate at ambient or elevated temperatures to allow the extraction of vapour-phase organic emissions from various types of car interior trim components, such as textiles, plastic polymers and sealants. Multiple test specimens can be readily evaluated from the same sample if required. Schematics of the apparatus in operation for bulk and surface emissions testing are shown in Figure 3.

Figure 2: Markes' µ-CTE instruments: the six-chamber model (left) and four-chamber model (right).

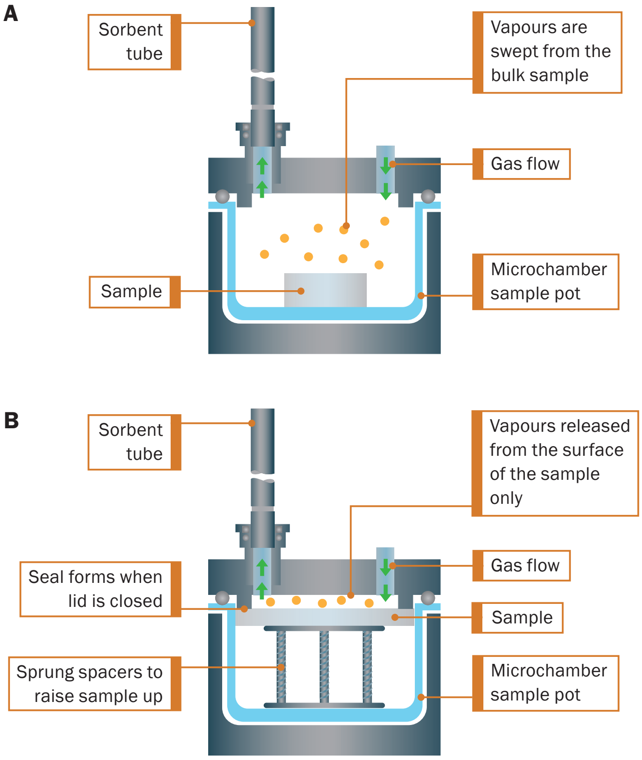

Figure 3: Operation of the µ-CTE for sampling emissions of volatile chemicals from (**A**) bulk samples, and (**B**) the surfaces of flat samples.

The chambers of both units 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 onto 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 16000-6.[5]

Experimental

The polymer acrylonitrile–butadiene–styrene (ABS) is a thermoplastic resin widely used in the automobile industry, *e.g.* for interior trim, headlight housings and grilles.

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 cm^2 with a clean scalpel, with gloves being worn to reduce the risk of contamination. Sample B was supplied in the form of five moulded 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 and they were placed in separate chambers (Figure 4).

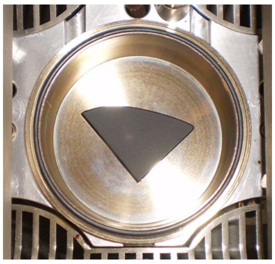

Figure 4: Samples were centred in the bottom of each chamber (with the same surface facing upwards) to ensure equivalent exposures and a uniform flow of gas across the surface.

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 min 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 min. All tubes were analysed by TD–GC–MS using a similar procedure to 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.

TD:

Instrument:	TD100™ (Markes International)
Flow path:	140 °C
Trap purge time:	1 min
Primary desorption:	8 min at 250 °C
Focusing trap:	–10 °C to 300 °C, 4 min hold, 20:1 split
Trap type:	U-T12ME (Markes International)

GC–MS:

Column:	DB-624™, 30 m × 0.32 mm × 1.8 µm
Pressure:	4 psi, constant-pressure mode
Initial flow:	2 mL/min
Temp programme:	35 °C (5 min) then 30 °C/min to 230 °C, hold for 2 min
Total run time:	13.50 min
Carrier gas:	Helium
Mass scan range:	m/z 25–300
MS source temp.:	230 °C
MS quad temp.:	150 °C
Transfer line temp.:	280 °C

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

Results

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

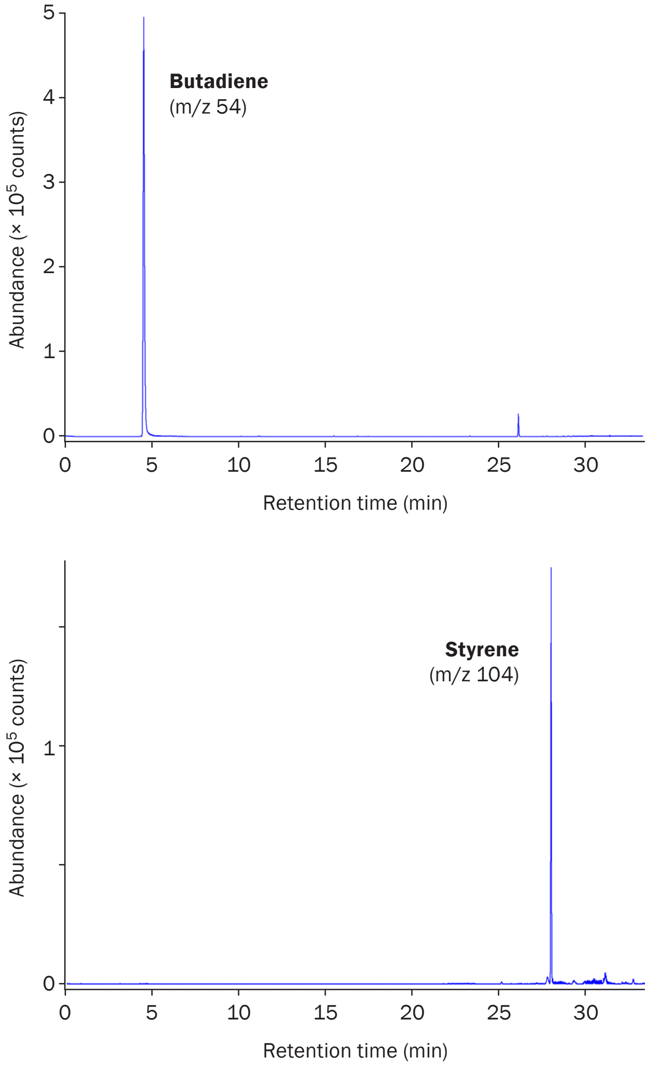

Figure 5: Extracted-ion chromatograms for butadiene and styrene, obtained from Sample A.

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

Relative standard deviations (RSDs) of 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 inter- and intra-lab studies using chamber equipment have shown RSDs in the order of 20–30% to be more common.[6]

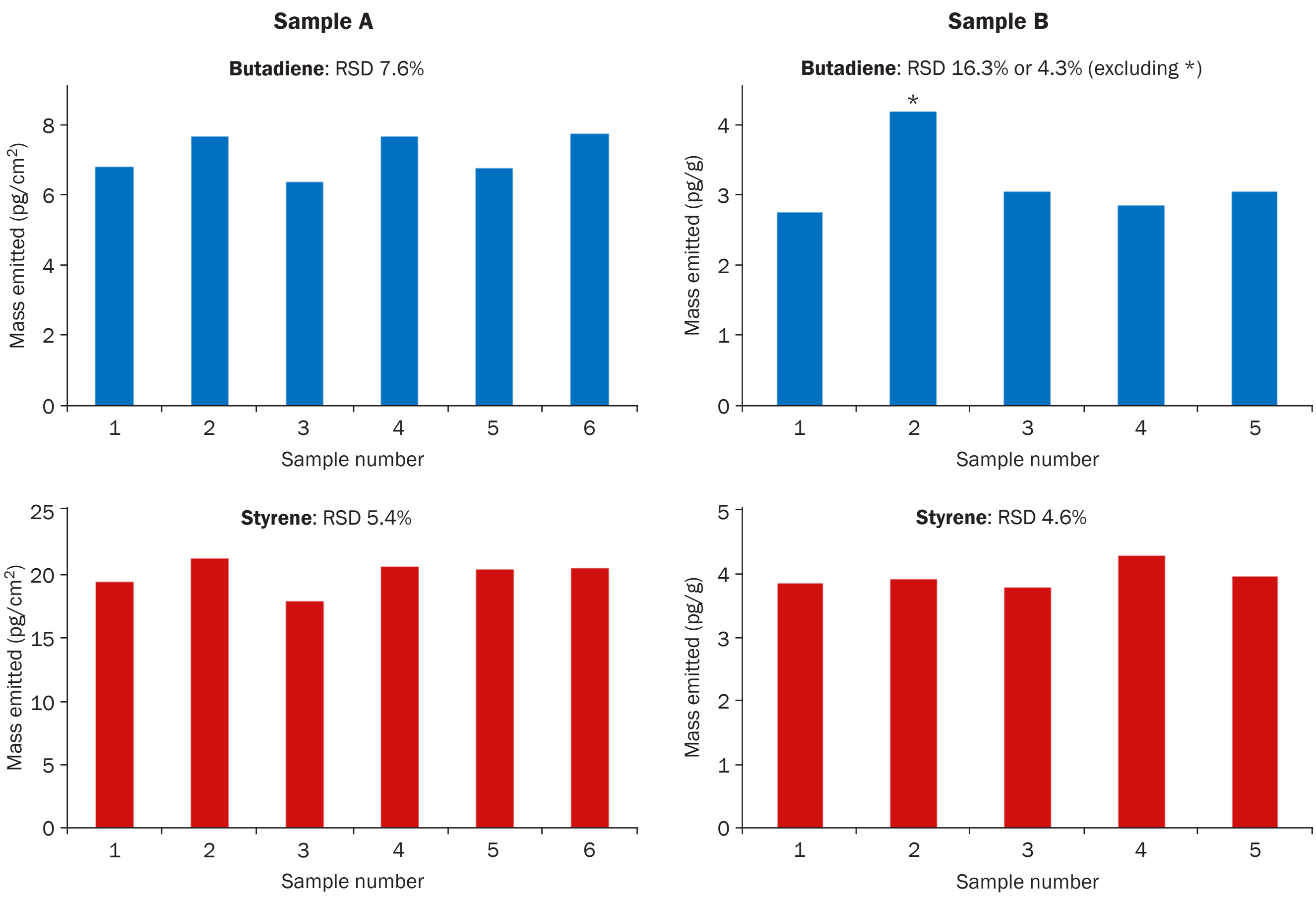

Figure 6: Emissions of butadiene (blue) and styrene (red) emitted from Sample A (mass per cm^2 surface area) and Sample B (mass per gram of sample). The outlying run for Sample B is indicated by *.

Conclusions

Markes' μ-CTE provides industry with 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

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