MARKES international

Application

TDTS 100

Comprehensive analysis of aroma compounds released from incense sticks using TD–GC/MS

Summary

This Application Note describes the analysis of VOCs released from incense sticks (agarbatti) using three sampling methods – a Micro-Chamber/Thermal Extractor™ for comprehensive analysis, pumped in-room sampling, and an Easy-VOC™ grab-sampler for robust sampling in challenging environments.

Introduction

Incense sticks (agarbatti) are used worldwide, but are especially popular in Asia. They are employed primarily in religious ceremonies, both in religious buildings and in the home, but also for aesthetic pleasure and in aromatherapy. They consist of a fuel, usually charcoal or wood powder, an optional oxidiser, any of a wide variety of aromatic plant-derived materials (such as bark, seeds, resins and essential oils), and a binder to hold the constituents of the formulation together. Given this diversity of ingredients and the potential for harmful components to be released during the burning process, the chemical constituents of incense sticks are receiving increased attention.

In addition, manufacturers are understandably keen to ensure that all the fragrances added to the incense sticks are released during combustion. Such compounds have a clear effect on the consumer experience, and a full understanding of the VOC profile can help manufacturers to improve their products further.

This Application Note describes how Markes' thermal desorption equipment can be used to analyse incense sticks, and in particular to identify the changes to the chemical constituents that take place upon combustion.

Three sampling techniques are used, and in all of them the air is sampled directly into tubes, for subsequent analysis by thermal desorption (TD)–GC/MS:

(a) The Micro-Chamber/Thermal Extractor – to provide comprehensive analysis of the VOCs released, both before and during burning

(b) Pumped air sampling to assess the VOCs present in a room during burning of incense sticks

(c) A fixed-volume Easy-VOC pump – for manual grab-sampling of air under field conditions.

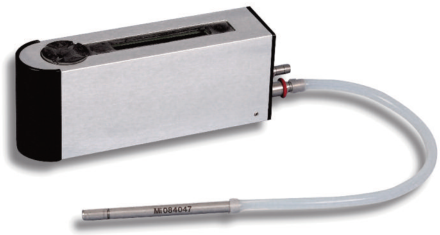
Background to sampling methodologies

Thermal desorption is a highly versatile sample preparation technique for the measurement of volatile and semi-volatile organic compounds in air and materials. Over the last 20 years, Markes has been at the forefront of developing cryogen-free TD technologies, with the UNITY 2 thermal desorber forming the cornerstone of the product range. In this Application Note we show how the UNITY 2 can be used in conjunction with the three sampling techniques listed above to characterise the aroma profile of incense sticks.

Markes' **Micro-Chamber/Thermal Extractor** is a stand-alone sampling device for screening VOCs and SVOCs from a wide variety of materials. Ideal for rapid sampling of both surface and bulk emissions, operation is simple, with short sampling times (typically less than 60 minutes). Vapours are released from the sample (at temperatures up to 250°C) into a sealed micro-chamber, and are then flushed directly on to a sorbent tube. As a result, the Micro-Chamber/Thermal Extractor is ideal for rapidly collecting everything released from a material,

and has been used to study a wide range of materials – from food and soild to plastic toys and flooring. Here, we show that its flexibility and ease-of-use makes it very well-suited for studying the VOC profile of a scented product[1].

For pumped sampling of air over time, the **FLEC constant-flow pump**[2] offers the benefit of maintaining constant sampling flow rates across tubes of varying impedance. A key advantage of this is that the system does not have to be recalibrated each time a different sorbent tube is used, saving time and improving productivity. In this case it was used to sample air from a room where an incense stick is being burnt – providing information about the odour profile that would actually be experienced by the occupants.



The third sampling methodology used here, Markes' **Easy-VOC**[3], is a hand-held device that grab-samples specified volumes of air (multiples of 50 or 100 mL) directly onto sorbent tubes. Its portability and the robust technology used makes it ideal for situations where setting up a pumped sample would be difficult. In this Application Note we show that the results obtained using Easy-VOC are similar to those using the pumped-tube method, making it a simple and easy-to-use alternative for field situations.

Experimental

For all three sampling methods, a target compound library based on the product formulation was constructed using NIST reference spectra. Chromatograms were searched using TargetView™[4] deconvolution and library-matching software from ALMSCO International (a division of Markes International Ltd).

(a) Micro-Chamber/Thermal Extractor experiment

A section of incense stick (40 mm) was cut and placed inside one of the micro-chambers, either unlit or burning. The sample was then left to equilibrate in a flow of pure, dry air for 10 min. A TD tube containing Tenax TA™ and SulfiCarb™ was then placed on the outlet port of the micro-chamber, with a diffusion-locking DiffLok™ cap on the end of the tube[5]. The tube was removed after 30 min and then analysed using the UNITY 2 thermal desorber.

Sampling using Micro-Chamber/Thermal Extractor:

TD tubes:	Inert-coated stainless steel, packed with Tenax TA–SulfiCarb
Purge gas:	Dry air
Flow rate:	50 mL/min
Temperature:	40°C
Equilibration time:	10 min
Sampling time:	30 min

TD:

Flow path:	200°C
Trap purge time:	1 min at 25 mL/min
Primary desorption:	300°C, 40 mL/min, 10 min
Focusing trap:	–10°C to 300°C, hold 5 min
Trap type:	U-T6SUL-2S (sulfur trap)
Split flow:	25 mL/min outlet only

GC/MS:

Column:	Ultra 2, 5% phenyl methyl siloxane, 50 m × 0.32 mm × 0.52 µm
Column flow:	1.8 mL/min, constant flow
Temp. programme:	50°C (2 min), 25°C/min to 160°C (0 min), 10°C/min to 280°C (3 min)
Total run time:	23.7 min
Carrier gas:	He
Mass scan range:	m/z 35–300
MS source temp.:	230°C
MS quad temp.:	150°C
MS transfer line:	280°C

(b) Pumped sampling experiment

Two incense sticks were lit and left to burn in a room with the door closed, while a constant-flow FLEC pump was used to sample the room air: (i) for 45 minutes during burning, and (ii) for 45 minutes after burning.

The room had a floor area of 2.22 m × 3.57 m, and a height of 2.66 m. The incense sticks and sample collection point were at opposite corners of the room, 4 m from each other. The incense sticks were at a height of 1.3 m, and the tube for sample collection was at 1.1 m, with the sampling end of the tube pointing vertically downwards.

Pumped-tube sampling:

TD tubes:	Inert-coated stainless steel, packed with Tenax TA–SulfiCarb
Pump rate:	40 mL/min
Pump time:	45 min
Total volume:	1800 mL

TD:

Pre-purge:	1 min at 20 mL/min to split
Sample desorption:	320°C, 40 mL/min, 10 min
Trap purge time:	1 min at 25 mL/min
Focusing trap:	5°C low, 320°C high, hold time 5 min
Trap type:	U-T6SUL-2S (sulfur trap)

Split flow: 25 mL/min outlet only (25:1) and re-collected
Flow path temp.: 180 °C

GC/MS:
Column: J&W DB-5, 60 m × 0.25 mm × 0.5 µm
Column flow: 1.1 mL/min constant flow
Temp. programme: 40 °C (1 min), 5 °C/min to 300 °C (4 min)
Total run time: 57 min
Carrier gas: He
Mass scan range: m/z 35–350
MS source temp.: 230 °C
MS quad temp.: 150 °C
MS transfer line: 280 °C

(c) Easy-VOC grab-sampler experiment

The setup conditions were the same as in (b). A 1000 mL sample (comprising ten successive 100 mL samples), was collected onto an inert-coated Tenax TA–SulfiCarb TD tube using an Easy-VOC sampler at the end of the burn period.

Easy-VOC grab-sampling:
TD tubes: Inert-coated stainless steel, packed with Tenax TA–SulfiCarb
Pump rate: 40 mL/min
Pump time: 45 min
Total volume: 1800 mL

TD:
Pre-purge: 1 min at 20 mL/min to split
Sample desorption: 320 °C, 40 mL/min, 10 min
Trap purge time: 1 min at 25 mL/min
Focusing trap: 5 °C low, 320 °C high, hold time 5 min
Trap type: U-T6SUL-2S (sulfur trap)
Split flow: 25 mL/min outlet only (10:1) and re-collected
Flow path temp.: 180 °C

GC/MS:
Column: J&W DB-5, 60 m × 0.25 mm × 0.5 µm
Column flow: 1.1 mL/min constant flow
Temp. programme: 40 °C (1 min), 5 °C/min to 300 °C (4 min)
Total run time: 57 min
Carrier gas: He
Mass scan range: m/z 35–350
MS source temp.: 230 °C
MS quad temp.: 150 °C
MS transfer line: 280 °C

Results and discussion

(a) Micro-Chamber/Thermal Extractor experiment

The Micro-Chamber/Thermal Extractor was employed to capture the complete VOC profile released from the sample. Both unburnt and burning incense sticks were tested, and the results shown in Figure 1. It is clear that many more compounds are released while the sample is burning – a result of the heat releasing the more volatile components from the incense stick, together with the chemical changes taking place upon combustion.

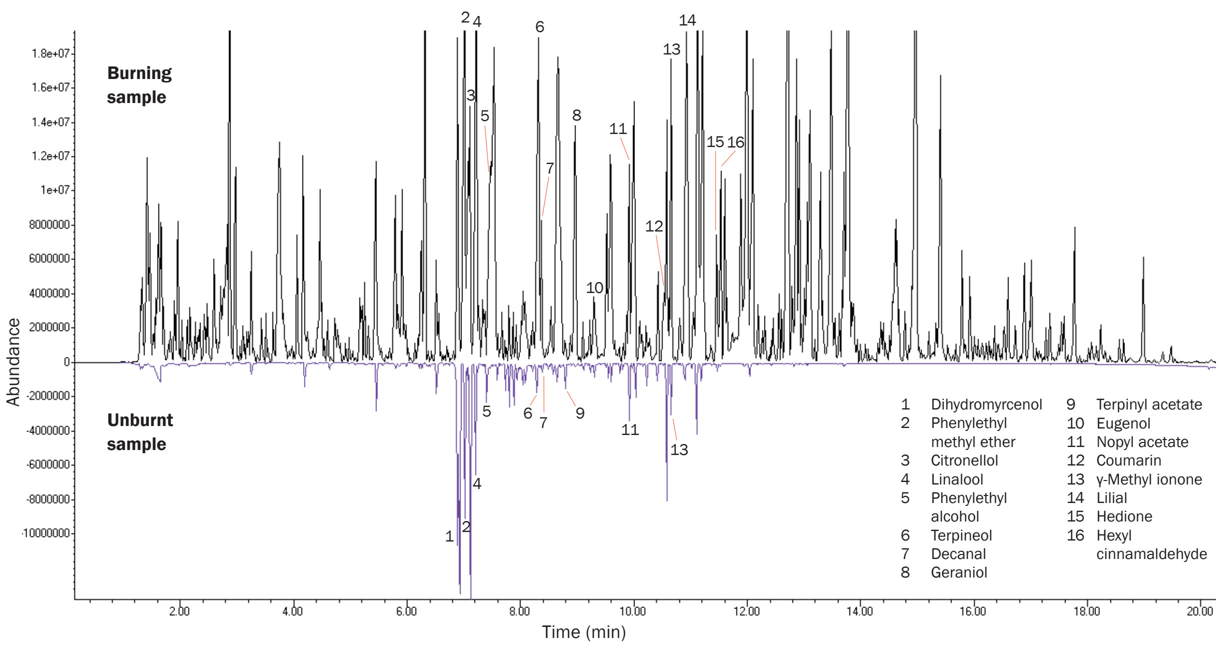


Figure 1: Analysis of the vapour profile from an unburnt incense stick (bottom), and of burning incense sticks (top), sampled onto sorbent tubes using the Micro-Chamber/Thermal Desorber and analysed by TD–GC/MS.

To maximise compound identification from these complex chromatograms, analysis was carried out using TargetView[4]. This software initially applies noise-suppression algorithms to minimise baseline anomalies, followed by spectral deconvolution to separate co-eluting peaks. The mass spectra of the deconvoluted components are then matched against a target library using principal components analysis to optimise the automated spectral matching process. A simple report is produced.

Use of TargetView allowed rapid and confident identification of a range of target analytes, even in the complex TIC obtained from the burnt sample.

(b) Pumped-tube experiment

Pumped-tube sampling of incense sticks was used to obtain information about the aroma that might be experienced by the occupant of a small room, both during and after burning.

Figure 2 compares the chromatograms, and shows that 12 of the 18 formulation compounds were present in the sample collected during the burn period. These are still present in the sample taken immediately after burning, although there is a clear reduction in component intensity, especially for the later-eluting components, as might be expected given their lower volatility.

As an extension to such an experiment, one could use equipment such as Markes' MTS-32™ multi-tube sampler[6] to take samples one after another for a specified time, allowing the decay of the fragrance profile to be monitored over time.

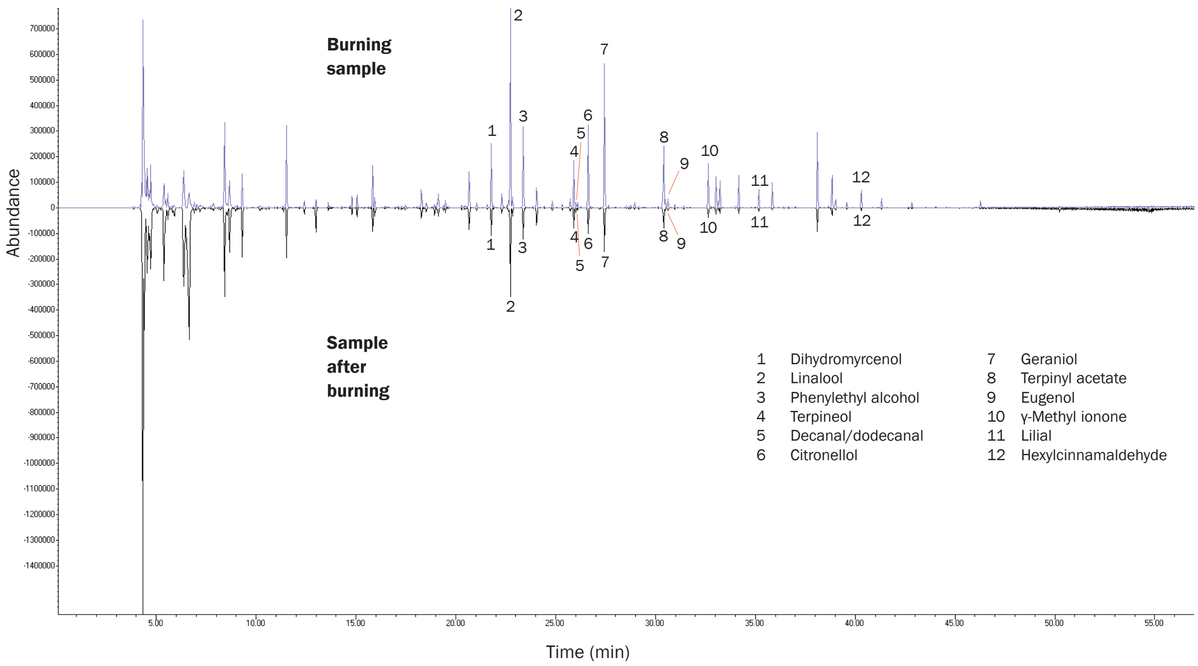


Figure 2: Comparison of the vapour profiles of incense sticks, during burning (top) and immediately after burning (bottom), sampled onto sorbent tubes using the constant-flow FLEC pump and analysed by TD–GC/MS.

(c) Easy-VOC grab-sampler experiment

Although conventional pumped sampling over time is normally the method of choice for monitoring VOCs present in air, there are situations where it might be inconvenient to set up a pumped sampling experiment, for example because of time constraints. In such cases, the Easy-VOC is an ideal solution, providing rapid and robust grab-sampling of air without the need for powered pumps.

Figure 3 demonstrates the similiarity of air profile data collected using the Easy-VOC and the FLEC constant-flow pump for the case of the burning incense sticks. The same 12 compounds are identified, with the only difference being a slight bias towards early-eluting components for the Easy-VOC. This reflects the difference between grab-sampling (which allows measurement of the VOC profile at one point in time) and constant-flow pumped sampling (which allows a time-weighted average profile to be obtained).

Conclusions

This study demonstrates the capabilities of Markes' instrumentation for monitoring VOCs released from incense, before, during and after burning, in three complementary ways.

The Micro-Chamber/Thermal Extractor, by completely enclosing the sample, provides the best possible levels of detection for all combustion products. Its ease of operation would make it ideal for carrying out quality control of products – for example, to check the consistency of different batches.

Pumped sampling, in contrast, provides a better representation of the VOCs actually present in room air (such as might be experienced by consumers).

Finally, the Easy-VOC provides qualitatively similar results to the pumped-tube method, making it more convenient for some field monitoring applications, *e.g.* large-scale studies of homes.

In all three cases, the UNITY 2 thermal desorber has been demonstrated to be compatible with the key components of the odour profiles of incense formulations.

Further benefits are also available, by automation of thermal desorption and sample re-collection using the TD-100™ [7]. This would facilitate larger-scale studies and repeat analysis of samples using the same or different TD–GC/MS conditions.

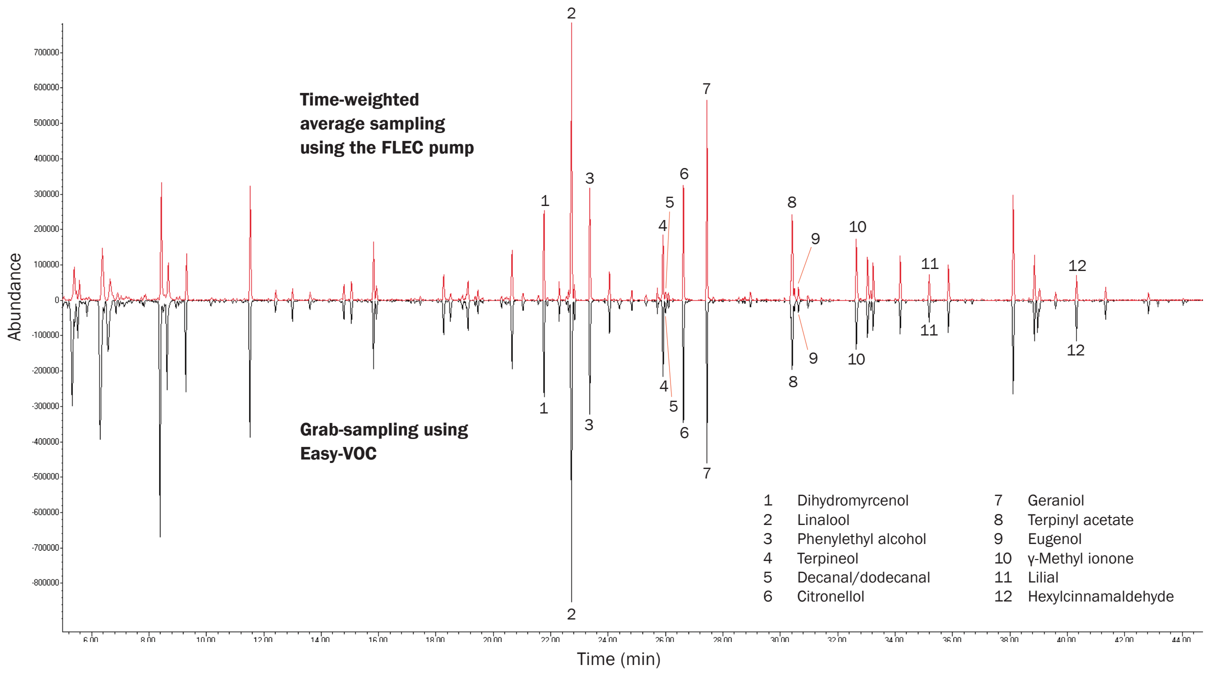


Figure 3: Comparison of the VOC profiles of burning incense sticks, sampled onto sorbent tubes using the constant-flow FLEC pump (top) and the manual Easy-VOC grab-sampler (bottom), and analysed by TD–GC/MS.

www.markes.com

Trademarks

DiffLok™, Easy-VOC™, Micro-Chamber/Thermal Extractor™, MTS-32™, SulfiCarb™, TD-100™ and UNITY 2™ are trademarks of Markes International Ltd, UK.

TargetView™ is a trademark of ALMSCO International (a division of Markes International Ltd).

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

FLEC® is a registered trademark of Chematec, Denmark.

References and notes

1. The Micro-Chamber/Thermal Extractor is also becoming increasingly popular for testing of construction materials, to help manufacturers conform to emissions regulations. See Application Notes TDTS 67, 69 and 95 for examples, and for further information visit http://www.markes.com/Instrumentation/Micro-ChamberThermal-Extractor-CTE.aspx.
2. For further details of the FLEC pump, please visit http://www.markes.com/Sampling-Accessories/Air-Sampling-Pumps.aspx.
3. To find out more about the Easy-VOC, please visit http://www.markes.com/Instrumentation/Easy-VOC.aspx.
4. For more examples of the use of TargetView, visit http://www.almsco.com/Products/Applications.aspx.
5. Markes' patented DiffLok caps allow sorbent tubes to be handled without risk of contamination, while at the same time eliminating the need for capping/recapping. Please see Application Note TDTS 61 for more details.
6. For further details of the MTS-32, visit http://www.markes.com/Instrumentation/MTS-32-Sequential-Tube-Sampler.aspx.
7. For further details of the TD-100, visit http://www.markes.com/Instrumentation/TD100.aspx.

Applications were performed under the stated analytical conditions. Operation under different conditions, or with incompatible sample matrices, may impact the performance shown.

Version 1
February 2012