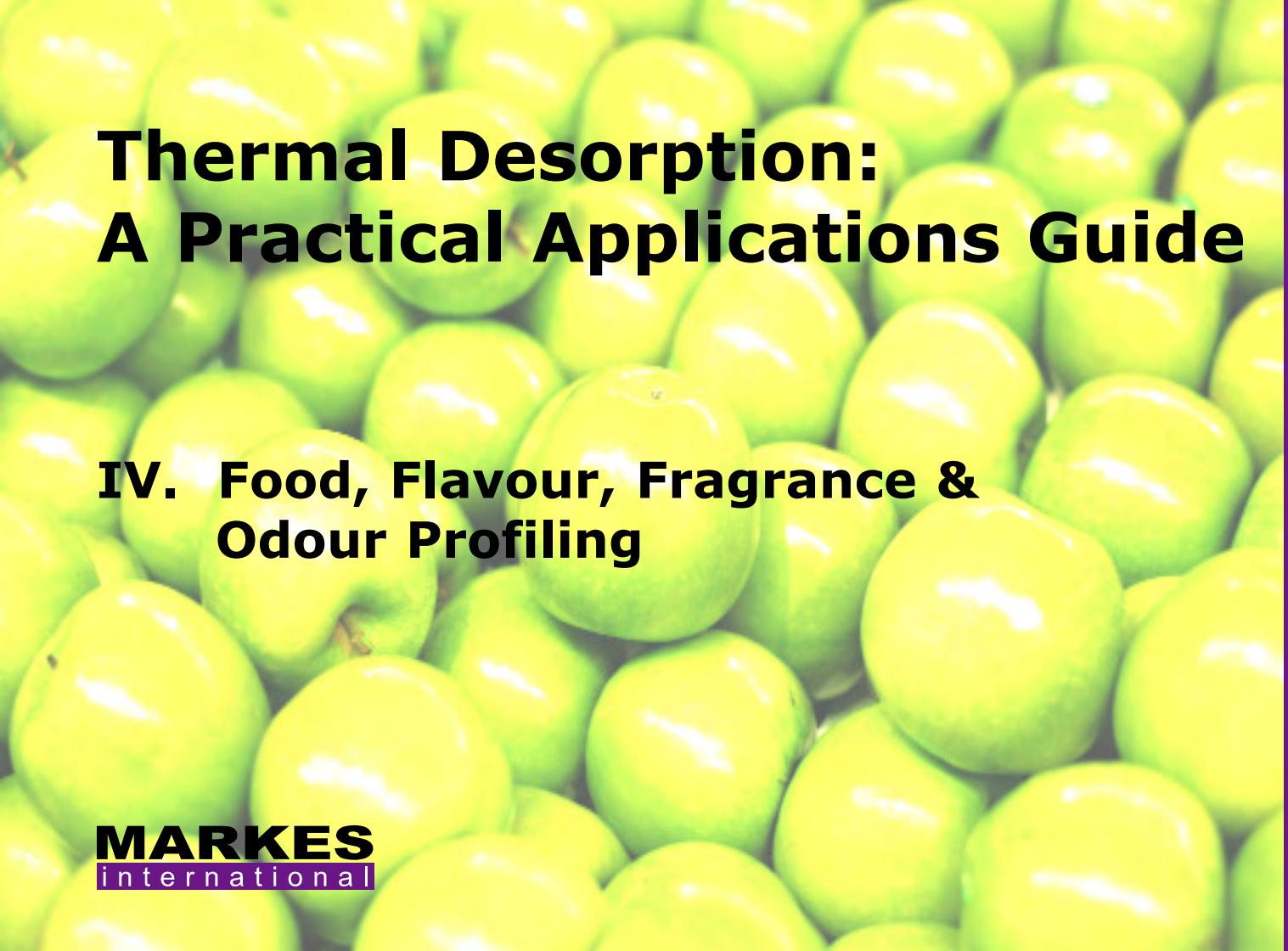
Thermal Desorption: A Practical Applications Guide

IV. Food, Flavour, Fragrance & Odour Profiling



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Introduction to Markes International Ltd.

Formed in 1997, Markes International Ltd. is one of the world's leading suppliers of thermal desorption (TD) equipment for monitoring trace toxic and odorous chemicals in air, gas and materials. Serving fast growing markets from environmental health and safety to materials testing and from food / flavour / fragrance to defence / forensic, Markes' global customer base includes major industry, government agencies, academia and the service laboratory sector.

Markes has introduced several highly successful brands of TD instruments to the market including: **UNITY™** – a universal TD platform for single tubes, the 100-tube **ULTRA™** TD autosampler, the **Air Server™** interface for canisters and on-line sampling, the **µ-CTE™** Micro-Chamber / Thermal Extractor for materials testing, the **TT24-7™** for continuous on-line monitoring and the **TC-20™** multi-tube conditioner.

Markes International also supplies a wide range of sampling accessories and consumables for all TD application areas.

What is TD?

Since the early 1980s, thermal desorption has provided the ultimate versatile sample introduction technology for GC / GC-MS. It combines selective concentration enhancement with direct extraction into the carrier gas and efficient transfer / injection all in one fully automated and labour-saving package.

Markes International Ltd., UK headquarters

Overview

Thermal desorption is now recognised as the technique of choice for environmental air monitoring and occupational health & safety. Relevant standard methods include: ISO/EN 16017, EN 14662 (parts 1 & 4), ASTM D6196, US EPA TO-17 and NIOSH 2549. Related applications include monitoring chemical warfare agents (CWA) in demilitarisation / destruction facilities & civilian locations (counter-terrorism).

TD is also routinely used for monitoring volatile and semi-volatile organic compounds (SVOC) in products and materials. Examples include residual solvents in packaging & pharmaceuticals, materials emissions testing and food / flavour / fragrance profiling.

This publication presents several of the real world applications of TD for measuring (semi) volatiles in food, flavour, fragrance and odours. Accompanying publications cover the applications areas of:

- Residual volatiles and materials emissions testing
- Defence & forensic
- Environmental monitoring and occupational health & safety

Applications

- Fragrance profiling of ingredients in toiletries and consumer products
- Identification of key olfactory components
- Characterisation / sourcing of natural products
- Odour profiling for potable spirits
- Quantitation of volatile components in dried foodstuffs
- Off-odour / taint analysis
- Biology / crop research
- Flavour profiling of GM foods

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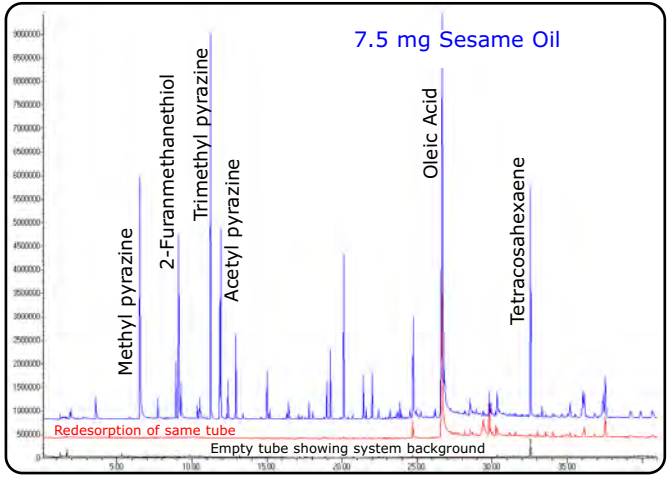
Profiling natural oils



Direct desorption of sesame oil sample, followed by a second desorption of the tube to illustrate efficient recovery of the components of interest in a single run

Typical analytes:
Methyl pyrazines, fatty acids and ethyl vanillin

Concentration: Low to high ppm

Background:

Detailed analysis of natural oils (*e.g.* sesame oil) may be required for several reasons; to identify key olfactory components, to characterise and source the material, and to identify oxidation products or other potential causes of taint. Traditionally this application has been carried out using multi-step liquid extraction or steam distillation with GC-MS analysis, but such procedures are lengthy, manual and inefficient. Direct thermal desorption/extraction of the oil using either the TD tube itself or a Markes Micro-Chamber / Thermal Extractor (µ-CTE) device, followed by TD-GC-MS analysis is a more efficient alternative.

TD conditions:

Sampling: Either incubate the oil sample in a µ-CTE chamber at 80 - 100ºC with vapour collection on Tenax TA™ tubes (gas flow ~100 ml/min) or load a few mg of oil onto a glass wool plug behind a 1 cm bed of Tenax in a standard TD tube.

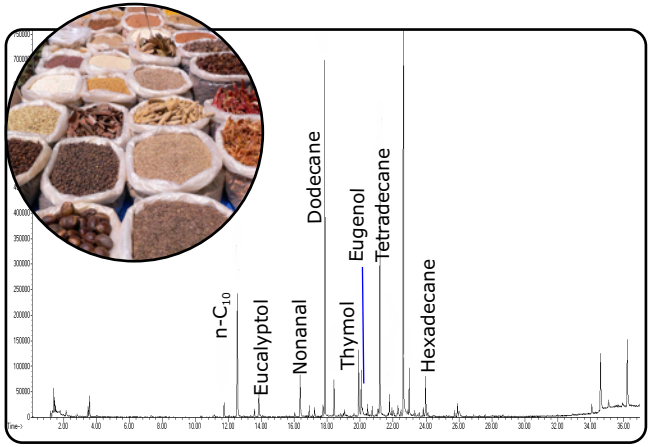
Desorption: 10 mins at 300ºC

Cold trap: Tenax

Split ratio: >100:1

Analysis: GC-MS (SCAN)

Direct desorption of volatiles from dried foodstuffs



Direct desorption of dried animal-feed pellets weighed into an empty glass tube

Typical analytes:
Carvacrol, cineole, thymol, eugenol and hydrocarbons

Concentration: Sub to low ppm

Background:

Direct desorption of homogeneous dried foods provides a high sensitivity and labour saving alternative to solvent extraction and allows analysis of a wider volatility range of components than equilibrium headspace. Foodstuffs compatible with this approach include:

- Ground spices
- Freeze-dried products such as ground or instant coffee
- Animal feed pellets

Typical TD-GC analytical conditions:

Sampling: 100–200 mg weighed into empty glass tube or PTFE liner

TD system: UltrA-UNITY

Desorption: 10 mins at 80°C

Trap: Quartz wool / Tenax

Split: ~25:1 split during trap desorption only

Analysis: GC-MS (SCAN)

Reference: TDTS23 Utilising the UNITY method development mode to analyse dried foodstuffs

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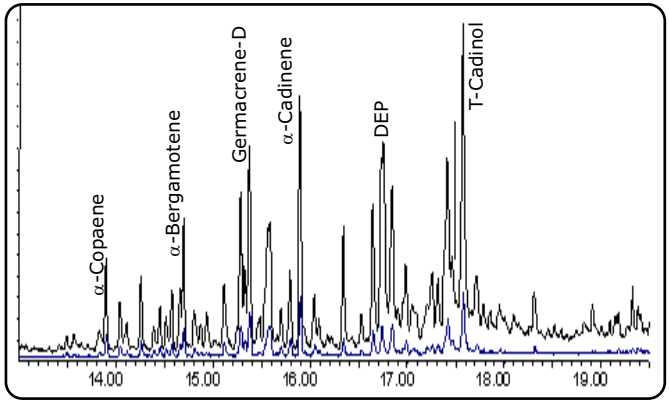
Natural products: Fragrance profiling



Vapours extracted from a leaf sample. Direct desorption (blue trace) followed by repeat analysis of re-collected sample (black trace) run with lower split ratio to enhance sensitivity

Typical analytes:

Range of terpenoid compounds, including:
α-Cedrene, α-Cadinene and T-Cadinol

Concentration: Sub to low ppm

Background:

Markes TD systems have an inert flow path that can be set at low temperatures, which makes them ideal for the direct desorption of labile volatiles such as terpenes and sulphur compounds. SecureTD-Q™ (*i.e.* quantitative re-collection of all split flow) facilitates repeat analysis of a sample under the same or different conditions (*e.g.* at a lower split setting, as shown) to demonstrate quantitative recovery through the system and to allow detailed analysis of minor components.

Typical TD-GC analytical conditions:

Sampling: ~100 mg of leaf sample weighed into an empty glass tube or PTFE liner secured with quartz wool

Re-collection on Tenax / UniCarb™ Silcosteel™ tubes

TD system: UltrA-UNITY

Desorption: 10 mins at 80°C

Trap: Sulphur trap

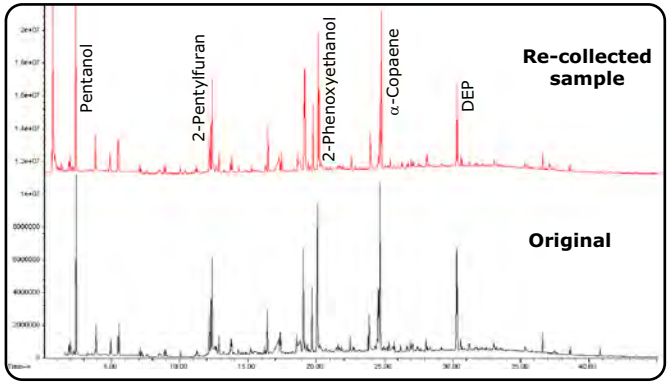
Flow path: 80°C to 150°C depending on target compounds

Split: ~25:1 & repeat analysis at 5:1 split.

Analysis: GC-MS (SCAN)

Reference: Markes brochure on TD validation featuring SecureTD-Q

Flavour profiling new crop varieties



TD-GC-MS analysis of volatiles from boiling potatoes using SecureTD-Q: Original sample and re-collected sample. Identical chromatographic profiles show recovery of labile analytes (*e.g.* terpenoids)

Analytes:

Diethyl phthalate, n-butyl butyrate, α-copaene, 2-(2-butoxyethoxy)-ethanol, 2-phenoxyethanol, decanal, octanoic acid, 2-ethylhexanoic acid, nonanal, 2-pentylfuran, pentanol, and hexanal

Concentration: Sub to low ppm

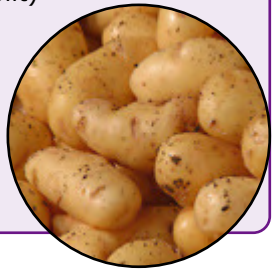
Background:

Development of new food crop species (*e.g.* genetic modification to aid pest resistance or to boost growth in arid areas) requires tests of the odour profile to make sure that the flavour is enhanced, or at least remains acceptable, in the new variety.

In the case of bulk, inhomogeneous materials like fresh fruit / vegetables, flavour profiles are best obtained by purging headspace volatiles from large (~ 1 kg) samples, cooked or raw, and collecting the vapours on tubes packed with Tenax sorbent. Tenax is completely hydrophobic so most water passes straight through during the vapour sampling process.

Typical TD-GC analytical conditions:

Sampling: 50 ml/min for 20 mins
Prepurge: 3 mins (to trap & split)
Desorption: 15 mins at 200°C
Trap: Tenax TA
Split flow: 20 ml/min
Analysis: GC-MS



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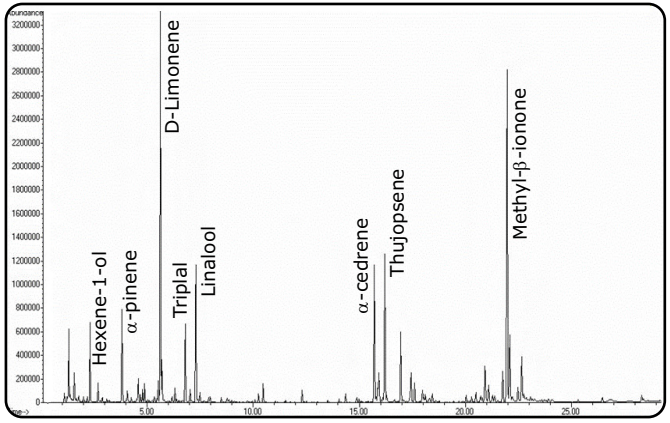
Fragrance profiling in consumer products



Headspace from sample of fabric conditioner

Analytes:

Isopropyl alcohol	α-Pinene	β-Pinene
α-Longipinene	Hexene-1-ol	Camphor
Methyl-β-ionone	γ-Terpinene	Myrcene
D-limonene	Triplal 1	Linalool
Hydroxy citronellal	α-Cedrene	γ-Muurolene
Thujopsene	β-Ionone	α-Chamigrene
Cyclamen aldehyde	α-Cedrol	α-Longipinene

Concentrations: ppm in headspace

Background:

Fragrance plays a major part in market acceptance and consumer satisfaction for products such as soaps & other toiletries, air fresheners and domestic cleaning materials.

TD provides a versatile, labour-saving and automated tool for GC-MS analysis of the fragrance profile of consumer products offering numerous sample handling options:

- Direct, in-tube desorption
- Dynamic purging of headspace vapours with on-line analysis
- Dynamic purging of headspace vapours onto sorbent tubes with off-line analysis

TD allows selective elimination of potential interferences such as water and some solvents thus simplifying fragrance analysis.

Typical TD-GC analytical conditions:

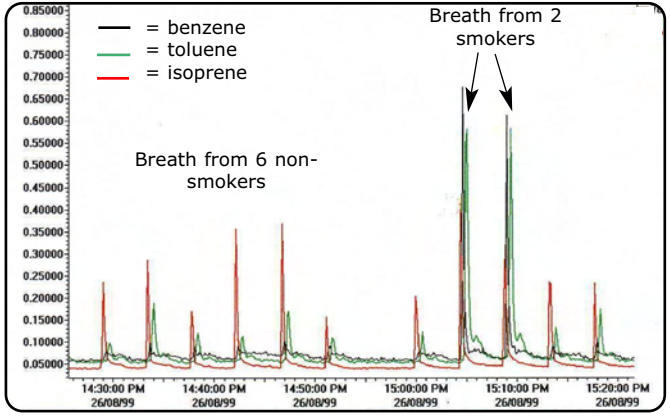
Sampling: ~200 ml headpace sampled onto Tenax tubes

Trap: U-T2GPH

Split flow: 30 ml/min during tube and trap desorption

Analysis: GC-MS

Halitosis – bad breath



Rapid TD-MS analysis of benzene and other hydrocarbons in the breath of smokers & non smokers

Typical analytes:
Hydrocarbons, oxygenates (esters, ketones alcohols, *etc.*) and other odorous VOCs

Concentrations: Low ppb

Background:

We are what we eat – and sometimes it comes back to haunt us in the shape of bad breath! Halitosis can also be caused by bacterial infections of the mouth / throat, some disease states and smoking.

The Bio-VOC™ Breath sampler from Markes International collects breath samples from the mouth & bronchial passages or from the alveoli (end-tidal air) and transfers them to sorbent tubes for subsequent analysis by TD-GC-MS. Applications of the Bio-VOC include biological monitoring of environmental / workplace exposure and disease diagnosis as well as breath odours.

Typical analytical conditions:

Sampling: Breath exhaled into Bio-VOC sampler and transferred to Tenax tube or Tenax / Carbograph 1TD focusing trap

Desorption: 280ºC for 10 mins

Trap: U-T2GPH

Trap conditions: +30ºC to 320ºC for 3 mins

Analysis: TD-GC-MS or TD with process-MS

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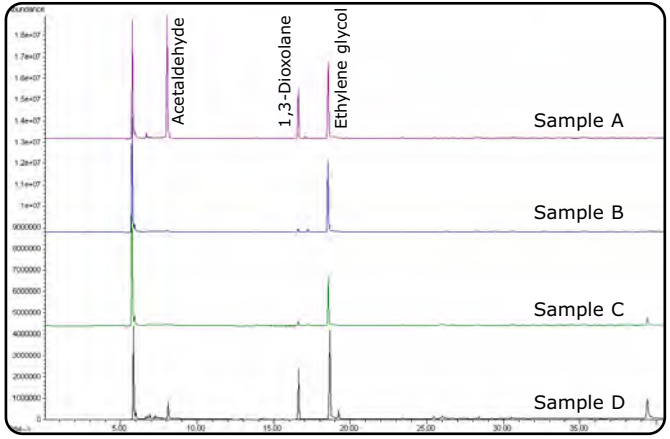
Taint and off-odour



Direct desorption of ground polyethylene terephthalate (PET) polymer to identify trace level VOCs contributing to taint. Comparison of PET polymers used in manfacture of soft drinks bottles

Typical analytes:

Residual monomers (*e.g.* acetaldehyde), pyrazoles and solvents

Concentrations: Sub to low ppm

Background:

Product taint can be introduced *via*:

- Odorous base materials
- Issues with the fragrance itself
- Packaging – everything from printed film to wood pallets
- Warehousing

Gentle direct desorption of the tainted product, *via* TD tubes, micro-chambers or bulk sample vessels and comparison with equivalent data from a control sample allows the taint components to be identified. The source can then be tracked *via* direct desorption of packaging, pallet fragments, base materials, additives *etc.* and analysis of vapour profiles and warehouse air collected on sorbent tubes.

Typical analytical conditions for PET:

Sampling: 200 mg of ground polymer in an empty tube

Desorption: 160ºC for 10 mins

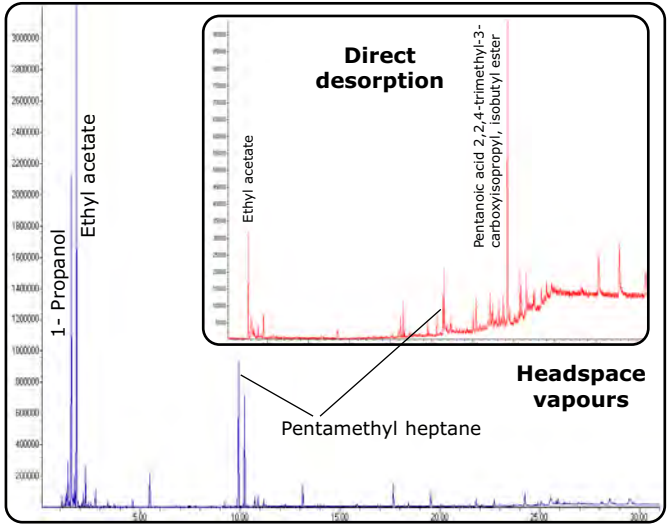
Trap: U-T6SUL

Trap conditions: -10ºC to 300ºC

Split: 40 ml/min during trap desorption

Analysis: GC-MS

Direct desorption of packaging



Direct desorption of residual solvents and semi-volatile additives from printed packaging film and sorbent tube sampling of headspace vapours from same

Typical analytes:

Alcohols, esters, ketones, alkanes and other odorous solvents

Background:

VOCs in food packaging can cause taint. In this example, TD was used to analyse printed biscuit wrappers in two ways:

- Direct desorption of the wrapper
- Desorption of Tenax TA tubes used to collect HS vapours from the sample

Note that headspace vapours from the packaging sample show high levels of volatile solvents which can migrate into fatty food stuffs, adversely affecting the taste.

Typical TD-GC conditions:

Sample: 10 x 5 cm area of film, rolled & inserted into an empty glass tube for direct desorption & 250 ml headspace sample drawn into a Tenax sorbent tube

TD system: UltrA-UNITY

Desorption: 10 mins at 60°C (direct TD) and 10 mins at 300°C (HS sample on Tenax tube)

Trap: Quartz wool, Tenax TA, Carbopack X™

Split: 30:1 during trap desorption

Analysis: GC-MS

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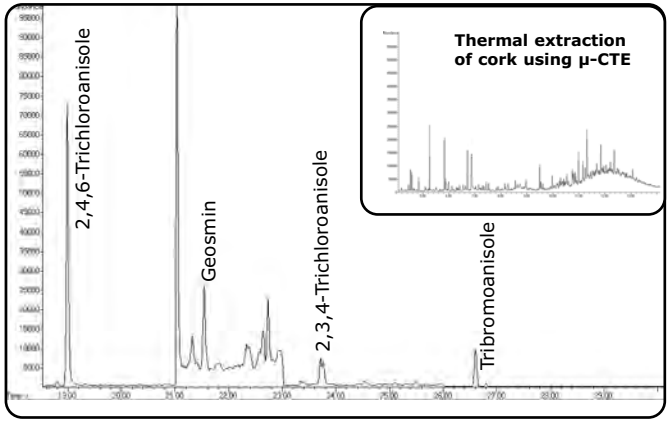
Trichloroanisoles in wine



TD analysis of 0.2 ng/L TCA and other odour compounds in the headspace of 1 L aqueous sample. Inset: Vapour profile from whole cork using µ-CTE

Analytes:

- 2,4,6-Trichloroanisole
- 2,3,4,6-Tetrachloroanisole
- 2,4,6-Tribromoanisole

Concentrations: Sub to low-ppb

Background:

A recent high-profile example of taint was linked to 2,4,6-trichloroanisole (TCA) in wine. TCA is produced from trichlorophenol by a microorganism that thrives in the production process of corks for wine bottles. This and other chemically similar analytes give the wine a mushroomy 'corked' aroma even at low concentrations (<5 ng/L).

The inert flow path of Markes TD systems facilitates TCA measurement in the headspace of aqueous samples at sub ng/L levels.

The Markes µ-CTE also facilitates direct thermal extraction of whole corks.

Typical TD-GC analytical conditions:

Sampling: On- or off-line sampling of headspace from 1 L aqueous samples at 60ºC onto Tenax trap. Whole cork incubated at 60ºC using µ-CTE with 70 ml/min flow of helium for 10 mins

Desorption: 280ºC for 10 mins

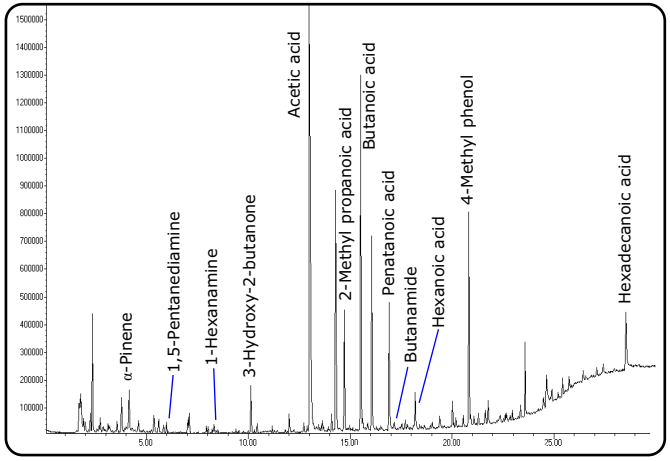
Trap: U-T2GPH

Trap conditions: -5ºC to 300ºC

Split flow: 30 ml/min

Analysis: GC-MS or GC-olfactometry

Odours from meat-processing



Chromatogram of odours from a swine facility

Typical analytes:

Carboxylic acids, monoterpenes, phenolic compounds, amines and amides

Concentrations: ppb

Background:

Thermal desorption is used extensively to monitor odours associated with meat processing. Applications include environmental / ambient-odour monitoring, product quality / flavour assessment, testing of animal odours (healthy and diseased) and at-line monitoring of production processes.

Typical TD-GC analytical conditions:

Sampling: 0.5-2 L vapour sampled onto Tenax / Carbograph 1TD tubes

Desorption: 300ºC for 5 mins then 320ºC for 5 mins

Trap: U-T2GPH

Trap conditions: +20ºC to 300ºC

Split: Low split during trap desorption only

Analysis: GC-MS

Chromatogram reproduced with the kind permission of APS Adamsen, of LugTek, Denmark - experts in odours from livestock production

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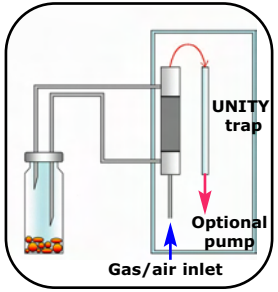
Multi-purpose Direct Inlet Accessory – For direct sampling / concentration of headspace vapours



The multi-purpose Direct Inlet Accessory (U-INLET) may be added to any manual UNITY thermal desorption platform to provide a simple and convenient mechanism for concentration of headspace vapours from a wide range of bulk sample containers.

This on-line approach allows vapours to be either pumped or swept through an inert, heated sampling line directly into the electrically-cooled focusing trap of UNITY without first being collected on a sorbent tube.

The UNITY-Direct Inlet system significantly improves the sensitivity of conventional headspace methods by allowing multi-stage extraction and concentration before analysis. The dynamic headspace approach also eliminates the need for equilibrium to be reached, thus reducing the time required for analysis.

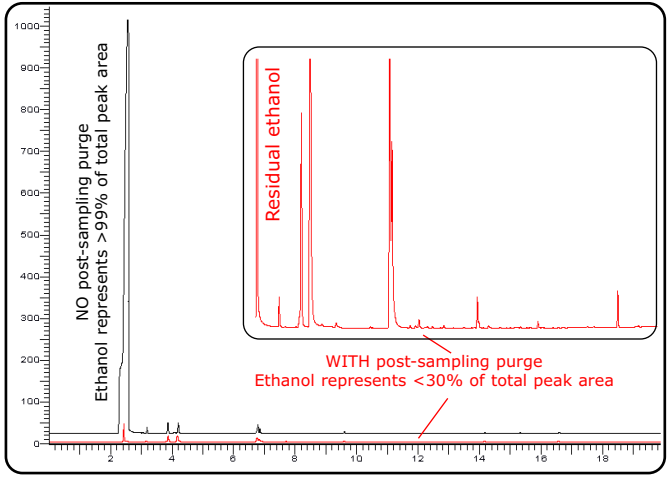
Sample vessel

The UNITY-Direct Inlet system is compatible with a wide range of sample vessels. It may be used for purging headspace vapours from smaller, sealed containers (such as reaction vessels or headspace vials) or for pumping air from open or compressible containers such as bell jars or Tedlar™ bags.

Key application areas include:

- Characterisation of VOC profiles from natural products and manufactured goods - food, flavour, fragrance analysis
- Monitoring emissions from living organisms - plants, microbes, fungi, insects, *etc.* as they change with time
- Monitoring malodours generated from food packaging (*e.g.* drink bottles)
- Off-odour / shelf-life testing
- Sampling from drinks / spirits, with the option of selectively purging the ethanol

Whisky: Aroma profiling



Purging of water and ethanol from whisky HS vapours allows selective concentration of key olfactory compounds – ketones, esters, *etc.*

Typical Analytes:

Ketones, aldehydes, esters
essential oils (*e.g.* juniper and coriander in gin)

Concentrations: Sub to low ppm

Background:

Thermal desorption facilitates detailed analysis of the flavour profile of potable spirit by allowing selective elimination of water and ethanol while key olfactory components – ketones, esters, essential oils, *etc.* – are quantitatively retained.

Headspace vapours are pumped / purged onto Tenax tubes under conditions which concentrate the target analytes while allowing most of the water, ethanol and other very volatile polar components to breakthrough. An example of whisky analysis, with and without selective elimination of water and ethanol, is shown opposite. Selective concentration of key olfactory components simplifies meaningful odour profiling.

Typical TD-GC analytical conditions:

Sampling: Sample placed in headspace vial at ~40°C. Connected to UNITY *via* Direct Heated Inlet Accessory

Sampling mode: Pulsed mode, 6 extractions of headspace

Trap: Tenax TA

Analysis: GC(-MS)

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Micro Chamber / Thermal Extractor (µ-CTE)

The µ-CTE offers a convenient approach for sampling volatiles from bulk samples at low to moderate temperatures.

The µ-CTE contains six (6) 28 mm deep x 45 mm diameter chambers into which samples are placed. A controlled flow of air or carrier gas is purged through all of the chambers simultaneously, sweeping the volatiles onto sorbent tubes attached to each chamber lid.

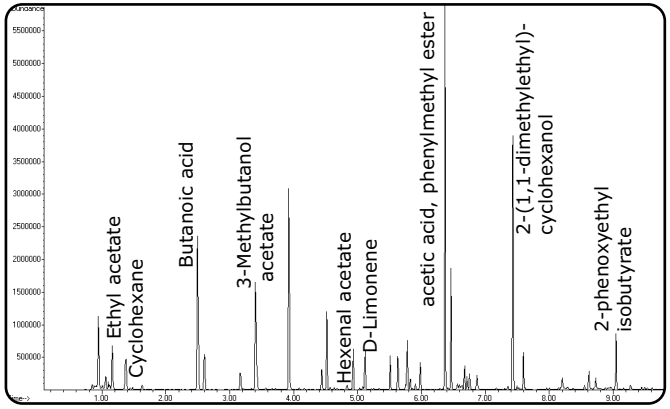
The µ-CTE can be heated from ambient to 120°C and is available with stainless steel or Silcosteel chambers. It is convenient for samples which are too inhomogeneous for direct desorption in empty tubes.

µ-CTE accessories are available to facilitate surface emissions testing and permeation studies (*e.g.* of packaging) as well as volatile analysis in bulk samples.

Key Applications include:

- Bulk sampling of volatiles from fruits, vegetables and other inhomogeneous foodstuffs
- Fragrance profiling of tobacco blends / substitutes
- Permeation testing of packaging
- Fragrance profiles from consumer products

Fragrance profiling of toiletries using the µ-CTE



Fragrance profile from shampoo obtained using the µ-CTE

Typical Analytes:

Esters, fatty acids, terpenes and solvents

Concentrations: ppm

Background:

The Markes µ-CTE is ideal for fragrance profiling of aqueous solutions and emulsions such as shampoo. Six replicate or different shampoo samples can be measured into individual micro-chambers, incubated at low temperatures (*e.g.* 30-40ºC) and the fragrance components purged onto attached sorbent tubes. Use of hydrophobic sorbents in the tubes allows quantitative retention of organic compounds of interest while water is purged to vent.

The µ-CTE is compatible with air or inert carrier gas to allow analysis of product fragrance under inert or oxygenating conditions.

Typical TD-GC analytical conditions:

Sampling: 5 ml shampoo incubated in the µ-CTE at 30ºC. Vapours swept onto Tenax tubes in a 70 ml/min flow of helium for 5 mins

Desorption: 280ºC for 10 mins

Trap: U-T2GPH

Trap conditions: 30ºC to 300ºC for 3 mins

Split flow: 30 ml/min during trap desorption

Analysis: GC-MS

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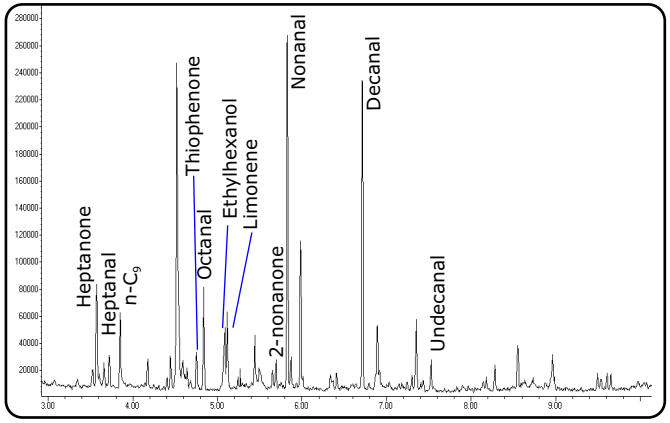
Flavour profiling of dairy products



Flavour profile obtained from natural greek yoghurt

Typical analytes:

Lactones from octalactone to tetradecalactone, fatty acids from C_6 to C_{16}, C_5 to C_{15} ketones, C_6 to C_{14} aldehydes, esters from ethyl butanoate to ethyl decanoate and C_{12} to C_{20} hydrocarbons

Concentrations: Sub to low-ppm in headspace

Background:

Milk and related dairy products have a complex aroma profile comprising fatty acids, lactones, ketones, aldehydes, esters, and hydrocarbons.

Several millilitres of milk or yoghurt can be conveniently measured into stainless or Silcosteeled micro-chambers and incubated at temperatures between ambient and 80ºC under a flow of pure air or inert carrier gas. Emitted vapours are collected on Tenax tubes connected to the exhaust of each micro-chamber. Water is selectively eliminated.

Typical TD-GC analytical conditions:

Sampling: 10 ml (g) yoghurt incubated at 70ºC in the µ-CTE, swept onto Tenax tubes in a 70 ml/min flow of helium for 10 mins

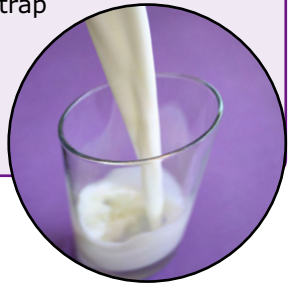
Desorption: 280ºC for 10 mins

Trap: U-T2GPH

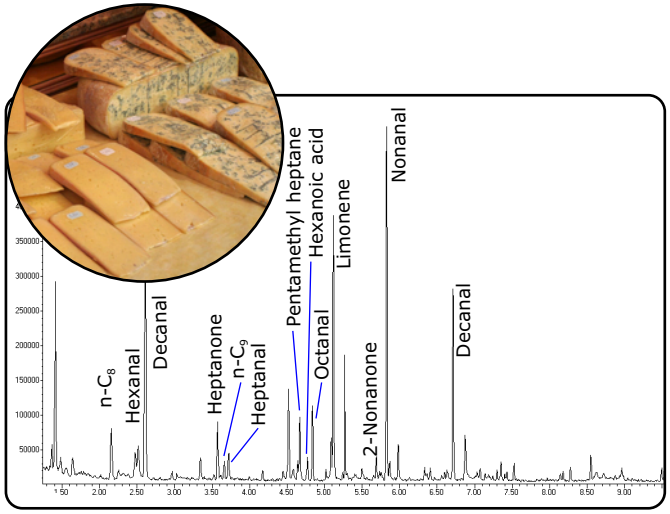
Trap conditions: 30ºC to 300ºC for 3 mins

Split flow: 30 ml/min during trap desorption

Analysis: GC-MS



Flavour profiling of cheese



Aroma / flavour profiling of cheese using the µ-CTE

Typical analytes:
Ketones, lactones, pyrazines, sulphurous compounds, esters, aldehydes, free fatty acids and alcohols

Concentrations: Sub to low-ppm in headspace

Background:

There is extensive research into the complex aroma profiles of different types of cheese. For example, over fifty aroma-active compounds have been detected in cheddar cheeses.

Thermal desorption / dynamic headspace offers an automated and versatile alternative to multi-step liquid extraction and vacuum distillation. Small cubes of cheese or grated cheese slurries mixed with distilled water can be incubated (*e.g.* using the Markes µ-CTE) purged with inert gas or pure air and the vapours collected using on- or off-line sorbent traps. Subsequent analysis is by TD-GC-MS or TD-GC with olfactometry.

Typical TD-GC analytical conditions:

Sampling: 2 g grated cheese mixed with 5 ml warm water and incubated in the µ-CTE. Vapours swept onto Tenax tubes in a 70 ml/min flow of helium for 10 mins

Desorption: 280ºC for 10 mins

Trap: U-T2GPH

Trap conditions: 30ºC to 300ºC for 3 mins

Split flow: 30 ml/min during trap desorption

Analysis: GC-MS

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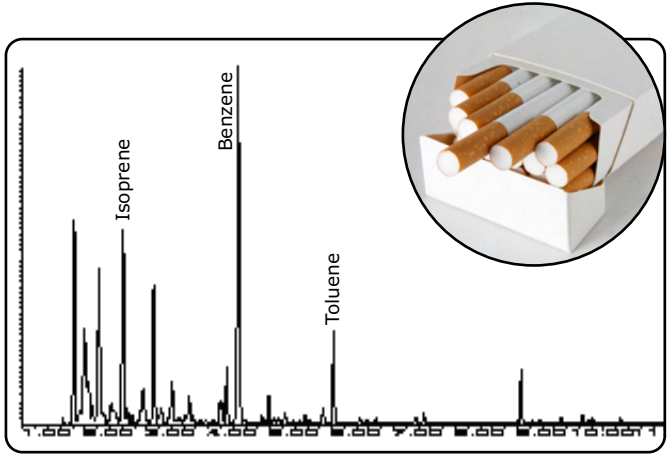
Applications for thermal desorption in the tobacco industry



VOC profile of tobacco smoke drawn through a cigarette filter, using a smoking machine

Analytes:
Benzene, toluene and isoprene

Concentrations: Sub- to low ppb levels in air. Higher levels in headspace vapours

Background:

In addition to sampling / analysis of environmental tobacco smoke (ETS), thermal desorption has extensive uses in the tobacco industry. Key applications include:

- Aroma profiling of tobacco / tobacco substitutes
- Monitoring filter efficiency by collecting vapours from smoking machines (see opposite)
- Tracking the cause of taint in batches of tobacco products

These applications are carried out using sorbent tube sampling, direct thermal desorption and sampling accessories such as the µ-CTE.

Typical analytical conditions:

Sampling: Multiple "puff" volumes taken into bag, then transferred onto sorbent tube or vapours sampled directly into tube

Desorption: 280ºC for 10 mins

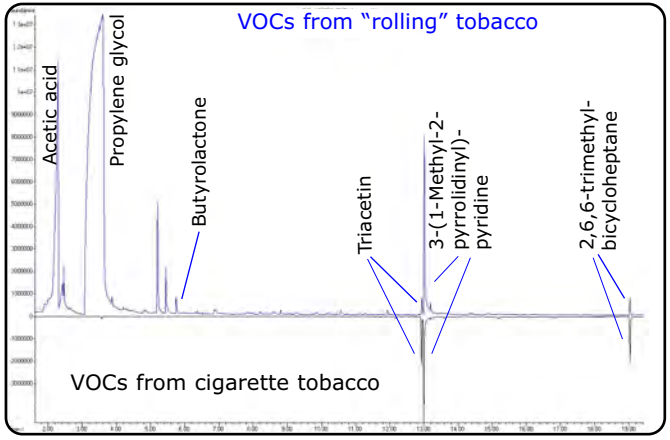
Trap: U-T2GPH, 30ºC to 300ºC for 3 mins

Split flow: 30 ml/min during trap desorption

Analysis: GC-MS

Reference: TDTS76 Applications of thermal desorption in the tobacco industry

Monitoring the aroma / flavour profile of tobacco using the µ-CTE



Vapour profiles from "rolling" tobacco (top) and cigarette tobacco (bottom) collected using a stainless steel micro-chamber at 50ºC

Typical analytes:

Glycols, triacetin, acetic acid, pyridines, hydrocarbons

Concentrations: Various

Background:

The Markes µ-CTE provides an ideal sampling accessory for inhomogeneous materials such as tobacco. Crumbled tobacco samples can be placed in Silcosteel micro-chambers, incubated at temperatures up to 120ºC and purged with air or inert carrier gas to sweep volatiles onto inert sorbent tubes. Subsequent analysis is *via* TD-GC-MS. The chromatograms opposite show comparative odour profiles from two types of tobacco.

The µ-CTE is similarly convenient for sampling whole cigarette filters (before or after smoking), cigarette paper and cigarette packaging materials.

Typical analytical conditions:

Sampling: 1 g of tobacco incubated in the µ-CTE at 50ºC. Vapours swept onto Silcosteel Tenax tubes in a 100 ml/min flow of helium for 10 mins

Desorption: 300ºC for 10 minutes

Trap: U-T2GPH

Trap conditions: -10ºC to 300ºC

Split: Double split, 300:1

Analysis: GC-MS (SCAN)

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On- and off-line monitoring of product shelf life

Twin trap TT24-7 for continuous monitoring

Air Server for semi-continuous monitoring

Markes' continuous or semi-continuous TD systems for on-line monitoring of changing odour profiles

Typical Analytes:
Aldehydes, ketones, esters, sulphur and nitrogen containing compounds, fatty acids, *etc.*

Concentrations: ppb to ppm

Background:

Versatile TD sampling accessories like the Markes Micro-Chamber / Thermal Extractor allow up to 6 product samples to be simultaneously incubated in a stream of air or inert gas for accelerated shelf-life tests and odour studies. Vapours are collected on attached sorbent tubes and analysed off-line using ULTRA-UNITY.

Custom made sample containers for bulk fresh or prepared foods – pizza, canned meat, *etc.* – can also be linked to off-line sorbent tubes or **monitored continuously** using either the TT24-7 or UNITY-Air Server / Direct Inlet. These on-line TD systems allow near real-time assessment of odour profiles as they change with time.

Typical on-line monitoring parameters:

TD system: TT24-7, UNITY-Direct Inlet or UNITY-Air Server

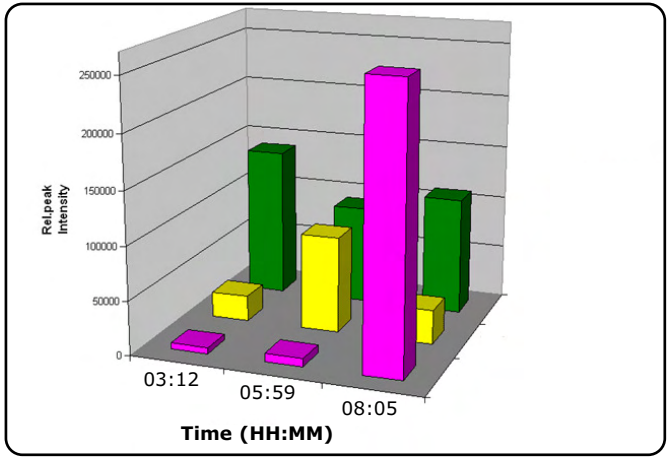
Sampling: 10-50 ml/min for 20 mins

Trap: Tenax or U-T2GPH at +30ºC

Trap desorption: 320ºC for 5 mins

Analysis: GC-MS or GC-olfactometry

On-line monitoring of fragrance profiles as they change with time



Concentrations of vapour phase organic compounds changing with time

Typical Analytes:

Aldehydes, ketones, esters, sulphur and nitrogen containing compounds, fatty acids, *etc.*

Concentrations: ppb to ppm

Background:

Most natural (*e.g.* floral) fragrances and many of the fragrance profiles of consumer products, such as air fresheners, change with time and ambient conditions. Markes continuous and semi-continuous on-line monitoring vapour systems allow round-the-clock profiling of fragrance allowing changes to be tracked as a function of time and ambient conditions – temperature, humidity, sunlight intensity, *etc.*

Typical on-line monitoring parameters:

TD system: TT24-7, UNITY-Air Server, UNITY-Direct Inlet

Sampling: 10-50 ml/min for 20 mins

Trap: U-T2GPH

Trap conditions: -10ºC to 320ºC for 3 mins

Analysis: GC-MS

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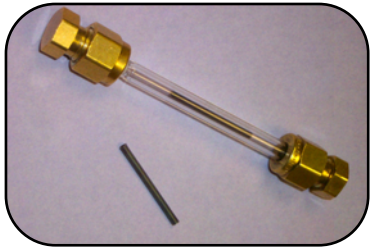
SPE-tD™ cartridges

Markes' SPE-tD cartridges offer a simple, convenient method for sampling less volatile impurities in aqueous samples – applications which would otherwise require manually-intensive extraction or distillation techniques before GC(-MS) analysis.



SPE-tD cartridges comprise a hollow tube, coated inside and out with polydimethyl siloxane (PDMS) for optimum capacity. The cartridge is placed into the aqueous sample and agitated. Volatile and semi-volatile organics in the sample, partition between the aqueous matrix and PDMS, reaching an equilibrium state over time. This allows semi-quantitative analysis of less volatile organics and direct comparison of organic impurity levels in two similar samples.

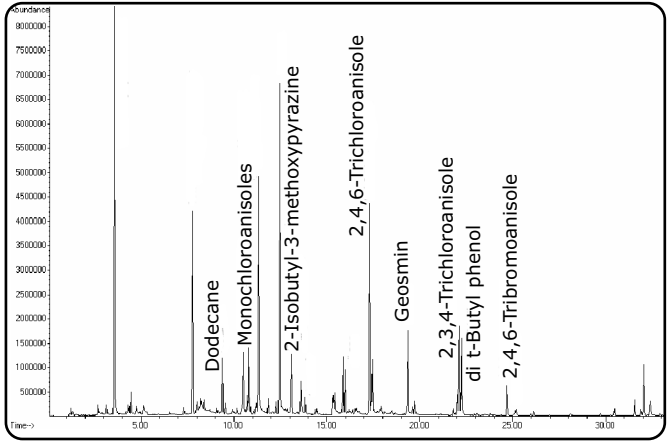
After equilibration, the SPE-tD cartridge is removed from the sample, rinsed in pure water to remove solid residues (if necessary) and placed into an empty TD tube. The cartridge is then dry purged with pure carrier gas, on- or off-line, prior to analysis by direct TD-GC-MS.

Solid phase extraction / TD methods provide a complementary sample preparation tool to automated headspace (HS) and purge-and-trap (P&T) techniques, which favour volatiles. Use of SPE-tD cartridges in combination with HS or P&T allows full characterisation of aqueous samples.

Key Applications include:

- Off-odours / taints in drinking water
- Semi-volatiles in processed fruit juices
- Profiling of hydrosols (aqueous fraction from steam distillation of natural oils)

SPE-tD extraction of organics from drinking water



Profile of sub-ppb level organics extracted from drinking water using the SPE-tD cartridge

Typical analytes:

Geosmin, methyl isoborneol, phenols and trichloroanisoles

Concentrations: Sub to low ppb

Background:

High capacity solid phase extraction using Markes SPE-tD cartridges provides a convenient approach to monitoring semi-volatile off-odour components in drinking water. SPE-tD cartridges used in combination with subsequent high sensitivity TD-GC-MS analysis offer trace detection limits (sub-ppb) and complement purge-and-trap / equilibrium headspace methods for volatiles.

The example opposite shows sub-ppb impurities absorbed by a SPE-tD cartridge from a 1 L sample of drinking water.

Typical TD-GC analytical conditions:

Sampling: SPE-tD cartridge placed into 1 L water sample and agitated for 2 hours

Desorption: 60ºC for 10 mins

Trap: U-T2GPH

Trap Conditions: 30ºC to 300ºC

Split flow: 10 ml/min during trap desorption

Analysis: GC-MS

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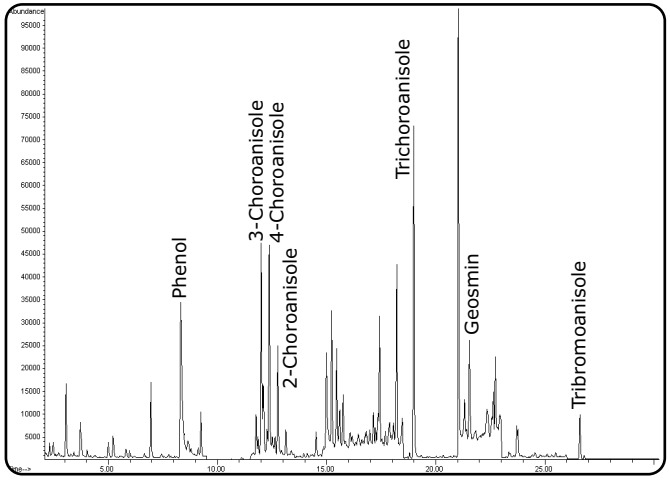
Drinking water: Pre-empting odour complaints



On-line monitoring of key odour components in the headspace of river water

Typical analytes:
Methyl i-borneol, chloroanisoles and geosmin

Concentration: 200 ppt in example shown

Background:

Much drinking water is sourced from natural rivers and streams. Continuous on-line monitoring of the headspace of river water, for unusually high levels of key odour components such as geosmin, can be used to prevent tainted water entering the drinking water supply and causing public concern.

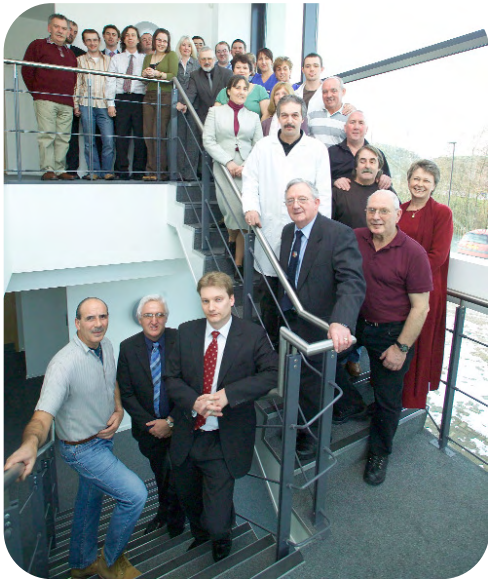
Analytical options include either UNITY-Air Server or the TT24-7 on-line TD systems operating continuously with GC-MS in unattended monitoring stations at strategically important points along the river system. Hourly data from multiple remote field monitoring stations can be sent to a central network hub by telemetry.

TD conditions:

Trap: Tenax TA / Carbograph 1TD
Heated inlet temp: ~70°C
Sampling flow rate / time: 50 ml/min; 15-20 mins
Post sampling purge time: 15 mins at 50 ml/min
Trapping temperature: 40°C
Desorption: 300°C for 5 mins
Flow path temperature: 200°C
Analysis: GC-MS

The Markes International advantage

- Markes is the market leader in TD
- Unparalleled reputation for product quality and reliability
- Excellence in technical and applications support
- For further information on Markes comprehensive range of instruments, sampling accessories and consumables please use one of the contact numbers / email address below or browse the web site



The Markes International team

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