Innovative Thermal Desorption From Agilent Technologies

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What is thermal desorption (TD)?

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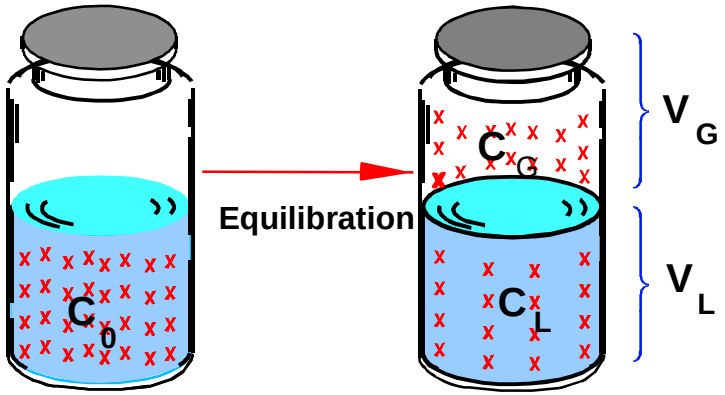
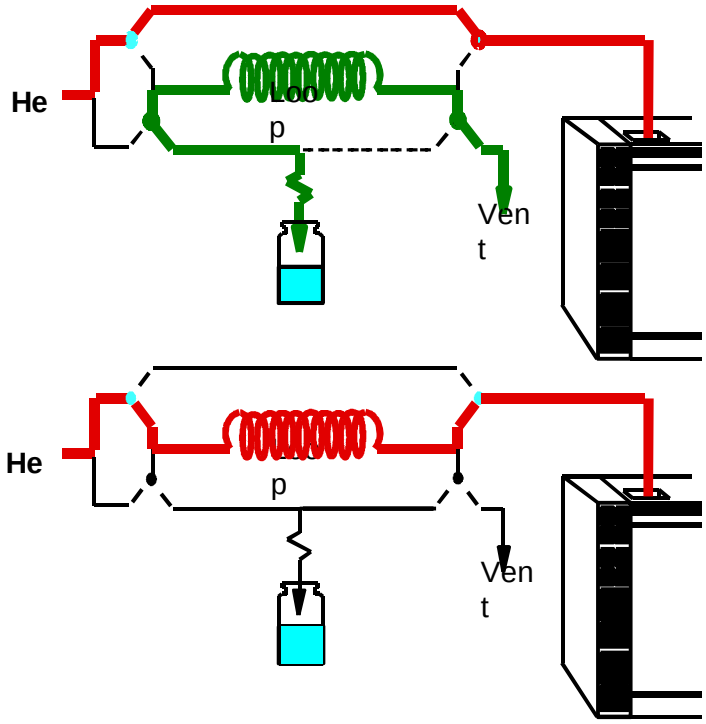
How does thermal desorption compare to Head Space?

- Thermal desorption (TD) is the process of extraction/desorption of analytes from the sample media using heat and a flow of inert gas.
- Head Space and TD system:





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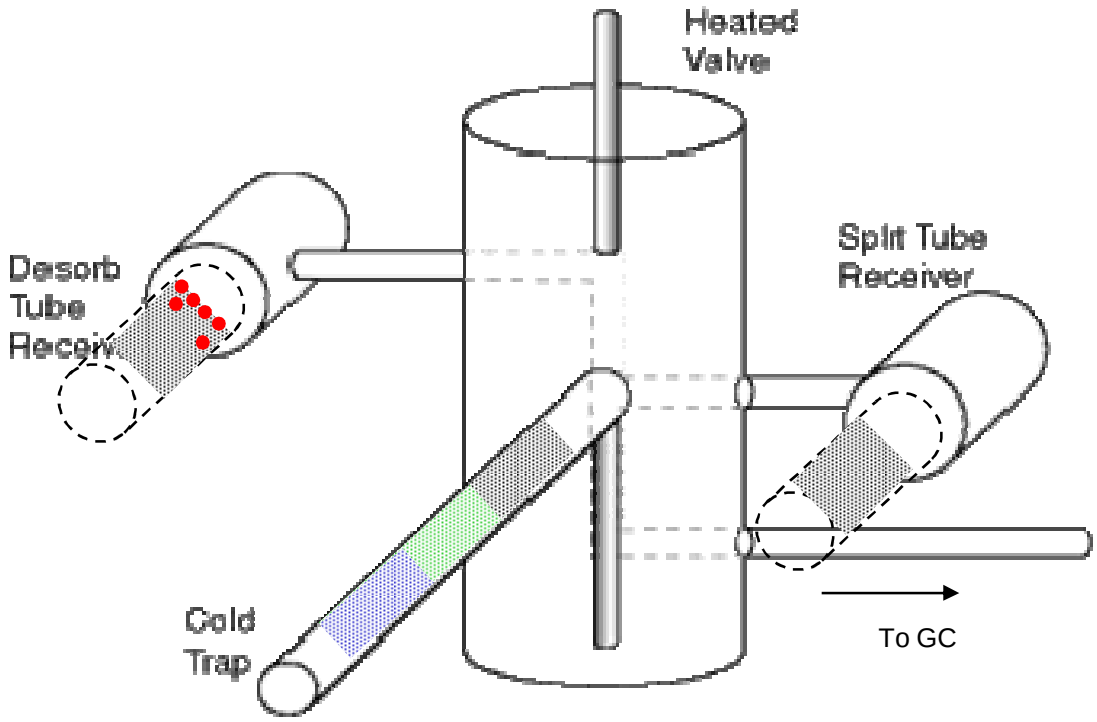
How do TD and SecureTD-Q work on Agilent systems?
Stage 1: primary (tube) desorption with optional (inlet) split



Patented TD heated valve is inert and low volume, ensuring quantitative recovery of high boilers

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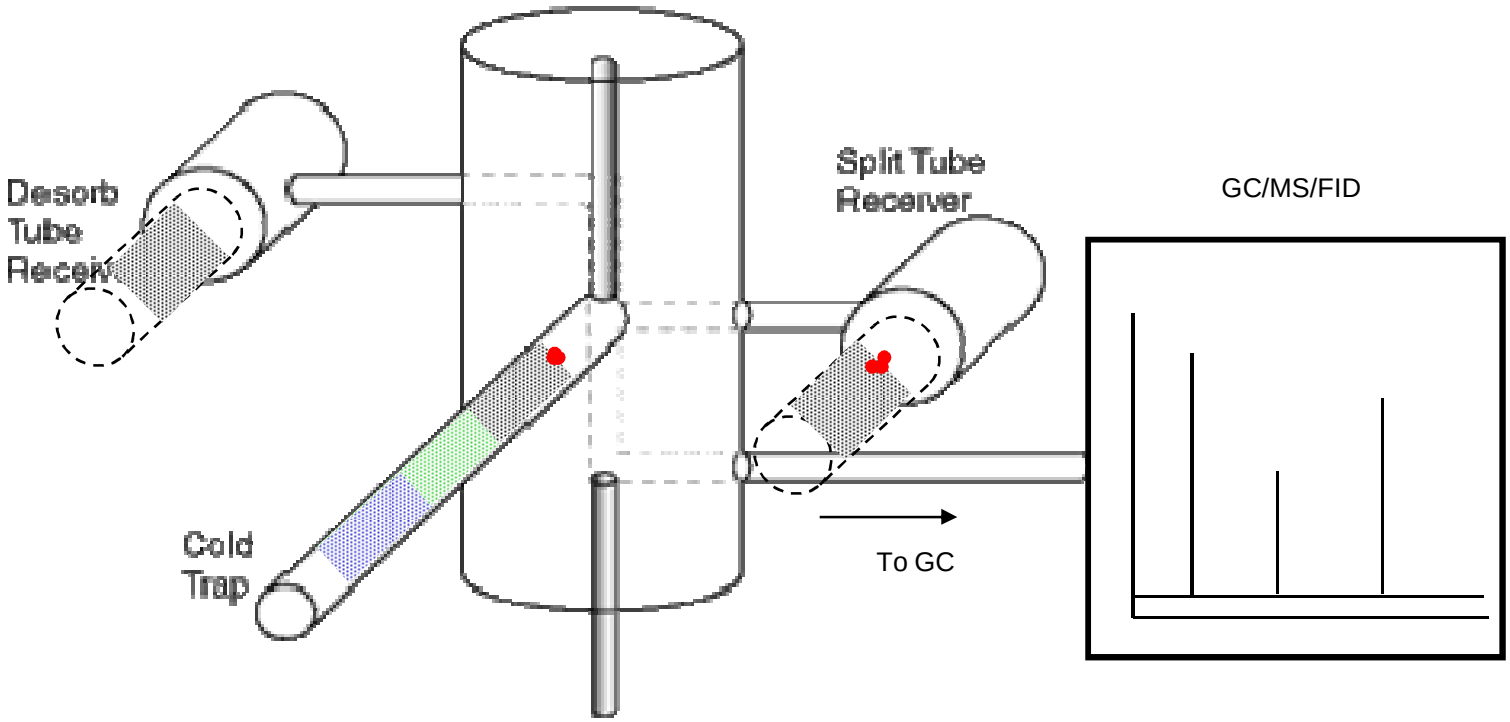
How do TD and SecureTD-Q work on Agilent systems?
Stage 2: secondary (trap) desorption with optional (outlet) split



Patented TD heated valve is inert and low volume, ensuring quantitative recovery of high boilers

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Overview of the thermal desorption process

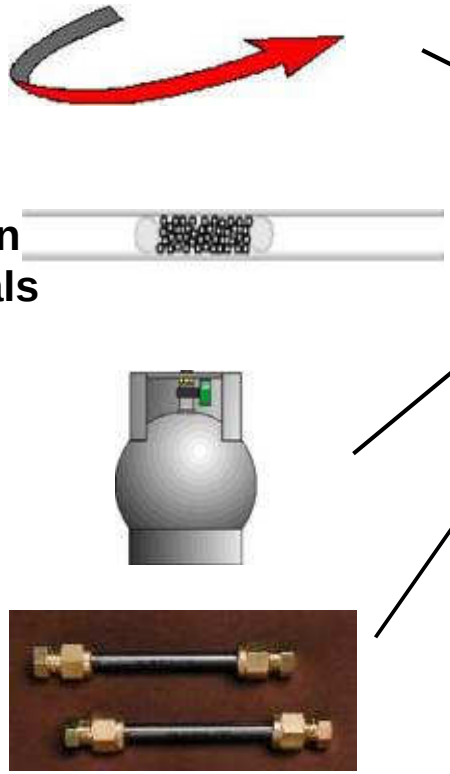
Sampling - Selective concentration – Desorption – Transfer - Measurement

On-line

Direct desorption of materials

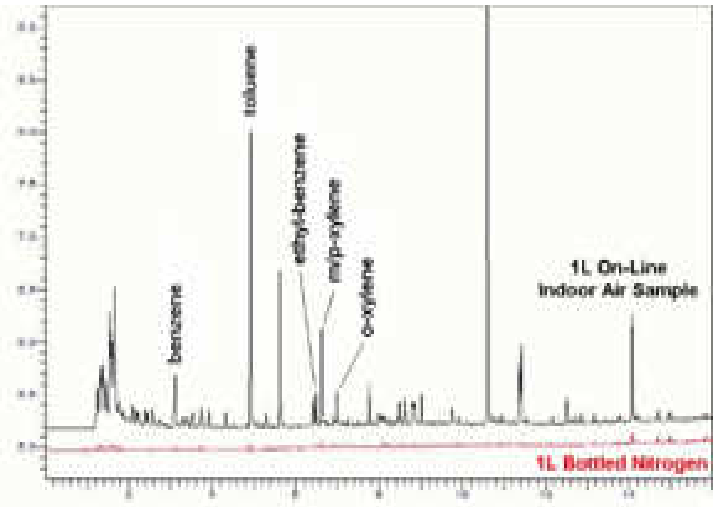
Canisters

Sorbent Tubes



Also purge and trap….

Electrically-cooled cryogen free focusing trap



~200 µL injection of vapour into GC(-MS)

Water and volatile Interferences may be purged to vent

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Summary of Applications for Food / Flavour / Fragrance

- Fragrance profiling of ingredients in consumer products / toiletries, plus identification of trace level toxics in product
- Characterisation of natural products
- Odour profiling
- Taint / off-odour analysis
- Volatile organics in dried foodstuffs

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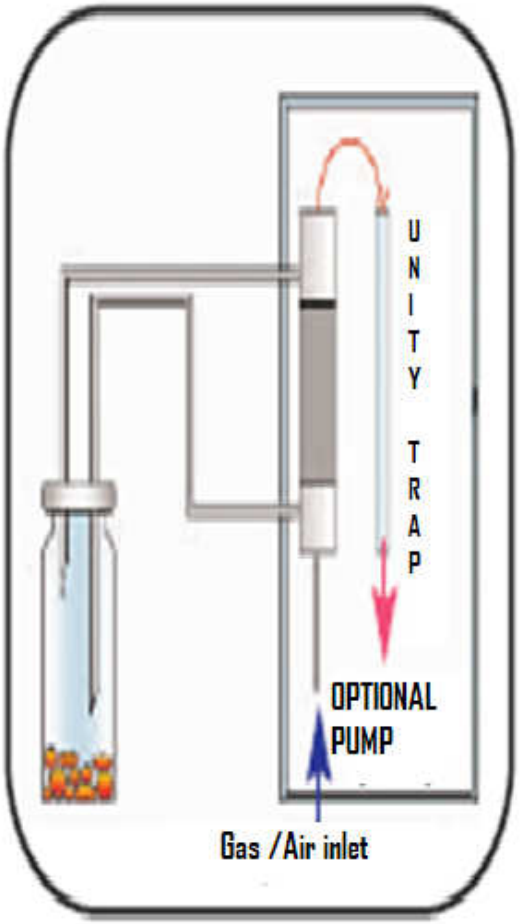
Direct Desorption

- Tube Sampling:

UNITY- Direct Heated Inlet for On-line Sampling of Headspace (HS) Vapours (Dynamic HS)



- Vapours from sealed vessels can be purged, with carrier gas, **directly into the focusing trap**
- HS vapours in air can also be pumped from the sample container through the focusing trap
- The sample inlet line is **heated and inert**
- The sampling cycle may be repeated before trap desorption
- **Converts equilibrium HS into pulsed 'purge and trap'**

Tube sampling

Diversified: Vapors Sampling

Diffusive Sampling

Axial and Radial

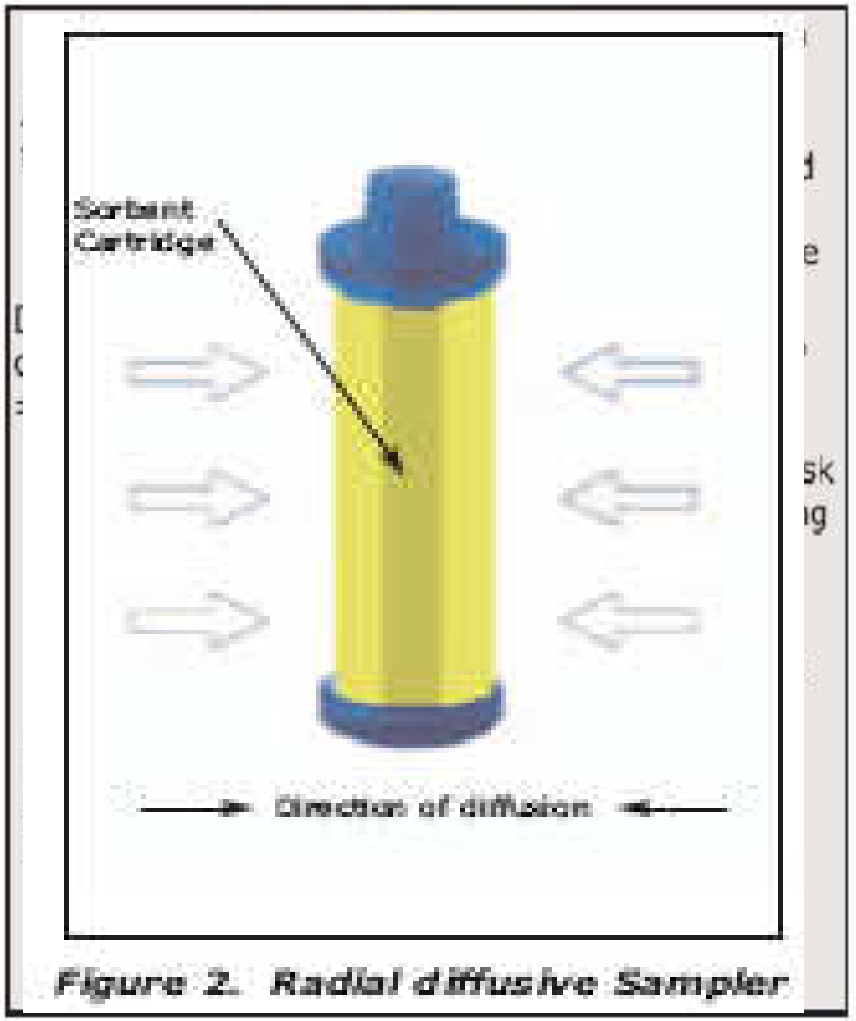


Figure 2. Radial diffusive Sampler

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Pump Tube Sampling Contd

- Flec Pump:
  - Constant Flow
  - Calibration
  - 4-1000ml/ min

SKC- Constant Pressure

Calibration Required

5ml – 4 L /min

Pumped Sampling- Extended

Multiple Tube Sampler-

Portable

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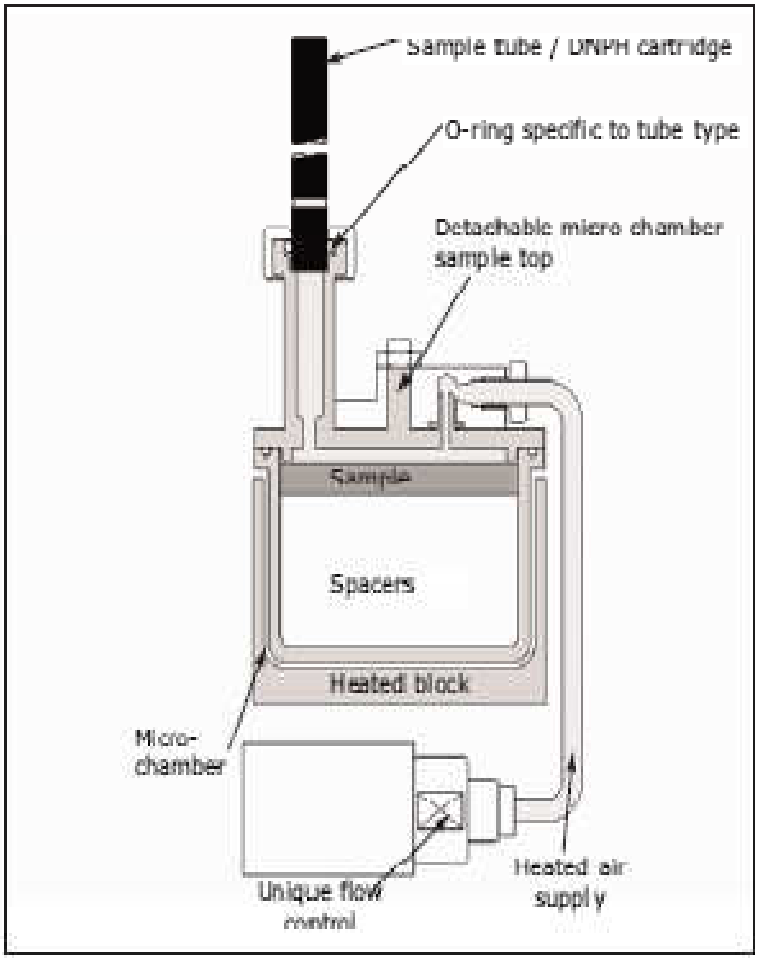
Tube Sampling Extended

Micro Chamber



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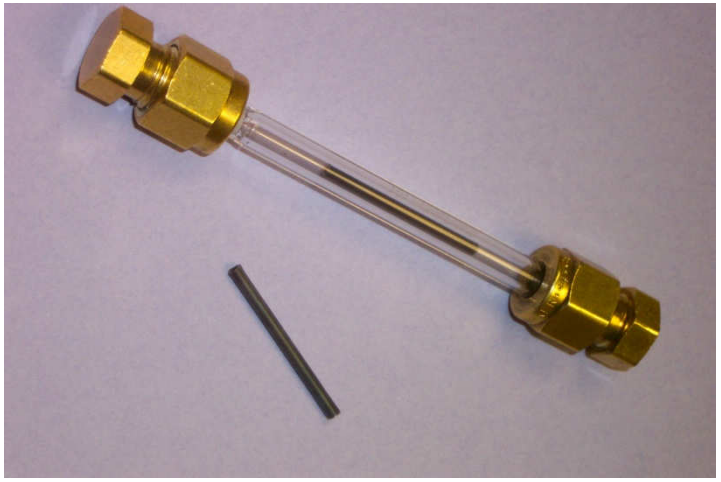
Micro Chamber-Extended

- Off-line sampling of bulk vapours onto tubes
- Applicability not limited to materials emissions testing
- Used for sampling VOCs from bulk foodstuffs, consumer products
  - e.g. Tobacco, shampoo, dairy products
- Possibility of foil liners to hold sample
- Permeation accessory – food packaging testing

SPE-tD Cartridges-Tube Sampling

Liquid Samples

- PDMS coated cartridge acts as adsorbent
- Primarily used for sampling dissolved volatiles in aqueous solutions (e.g. water, beverages)



Auto-Sampler

Auto sampler

Tray

Ultra 50 : 50

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Automated TD-GCMS system for sorbent tubes

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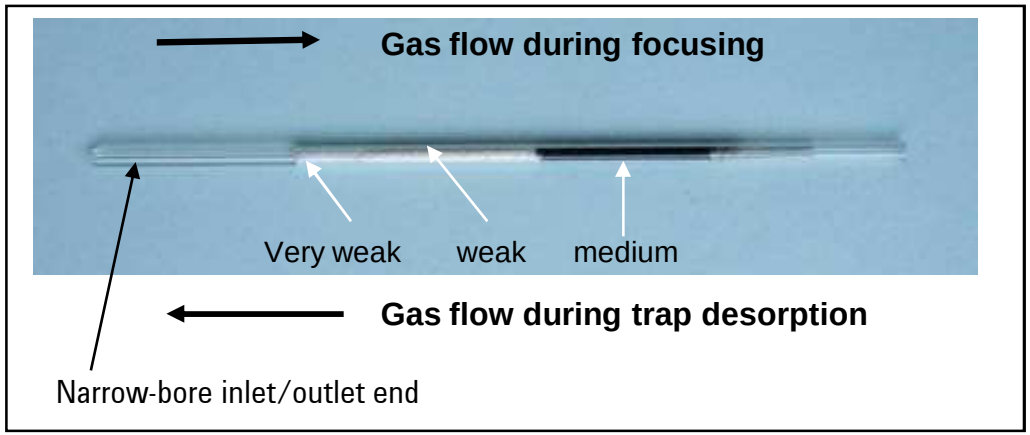
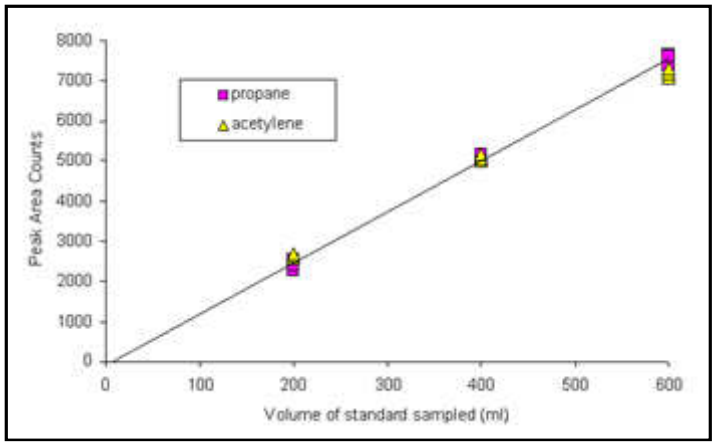
Electrically-cooled, focusing trap (cryogen-free) for operation over *wide boiling range*



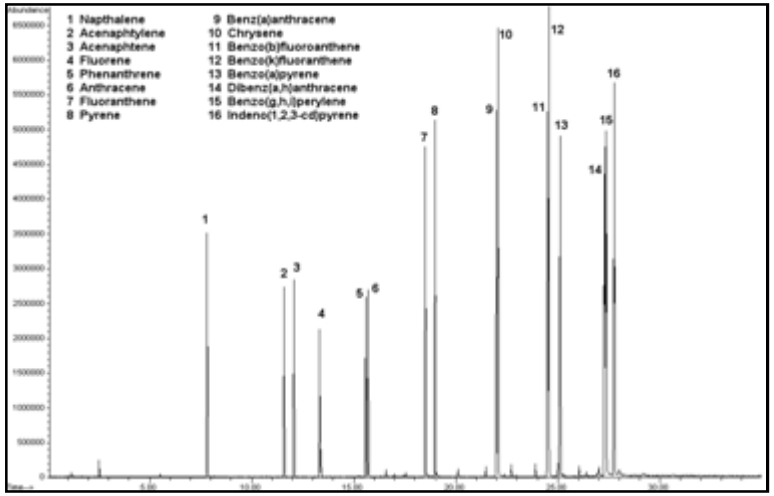


Quantitative retention of acetylene – No liq. N_2

Quartz cold traps: easy to change without breakage. The 60 mm x 2 mm bore sorbent bed and 100°C/sec heating rate give optimum retention and efficient desorption, **without risk of ice blockage**.

Other key features include:

- **Backflush** desorption
- Compatible with **splitless** desorption at 2 ml/min
- **No liquid cryogen** required.
- Compatible with both whole air and sorbent tubes.
- **Simple purging of solvent / water**



Quantitative recovery of 5/6-ring PAHs

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Guarantee sample and data integrity with a method-compliant leak test

- The TD flow path is ‘broken into’ every time another TD sample tube is introduced. International standard methods for thermal desorption require:
  - **A stringent leak test before each tube is desorbed**
  - **That the leak test occurs at ambient temperature and without gas flow**
- Every tube on an Agilent UNITY or ULTRA-UNITY TD is automatically leak tested
- The leak test is carried out by isolating the tube, trap and other TD flow path components at elevated pressure. If there is a leak, the pressure in the flow path falls and this is detected by a pressure transducer
- Carrier gas continues to be supplied to the GC(-MS) throughout the leak test
- Tubes which fail the leak test are preserved intact for the operator to check at the end of a sequence & the numbers are logged in the sequence reporter. The instrument proceeds to the next sample.
- Leak test failures trigger the start of a blank GC run to keep the TD sequence synchronised with the GC(-MS) sequence

Seamless interfacing of the TD with Agilent 6890 EPC carrier gas control facilitates Retention Time Locking

Resettable methods

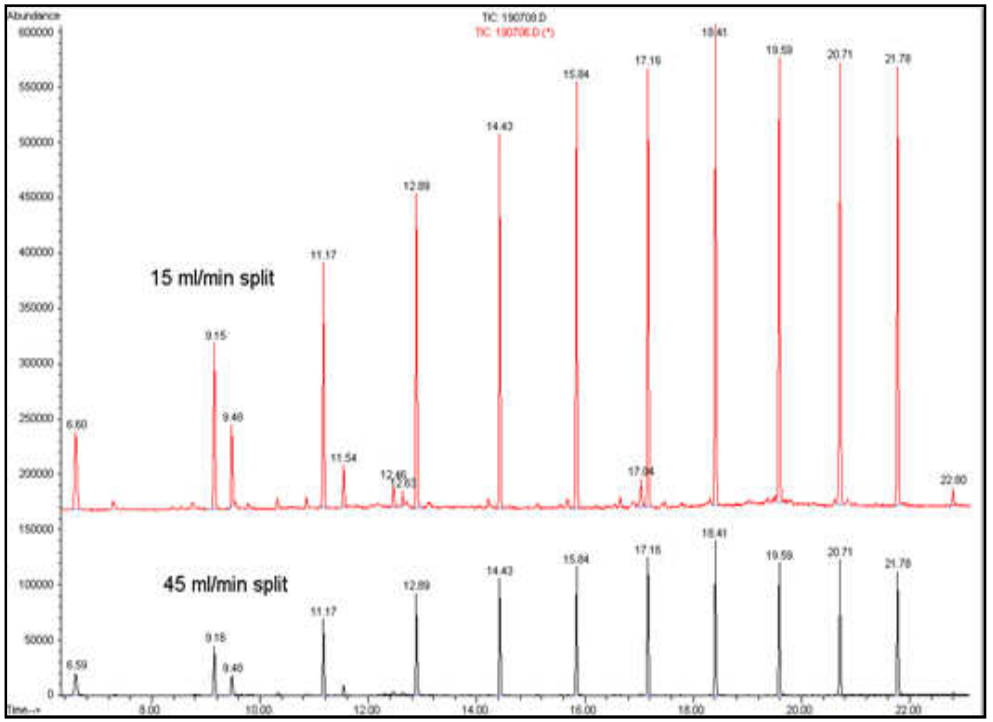
- EPC through the entire TD-GC(MS) system offers total flow read-out, enhanced leak diagnostics and pneumatic programming that is directly linked to the GC oven programme.

Constant pressure

- Using the EPC module of a 6890 / 6850 S/S injector with column head-pressure-regulation enables complete stabilisation of TD-GC(-MS) retention times even under different split flow conditions

More reliable compound identification.

- Retention time stability allows the creation of Rt-locked databases for TD applications and the use of **spectral Deconvolution Reporting Software (DRS) – see next slides**



UNITYe – GCMS with and column head pressure regulated EPC offers retention time stability independent of split flow, trap temperature, sorbent choice, etc

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Other enhancements offered by Agilent TD systems

- **Horizontal tubes prevent material samples shifting or falling out of the tube** during desorption. (Material samples – drug powders, plastic pellets, adhesives, etc – can move, fall out or even drip out of tubes which are manoeuvred and desorbed vertically. This can lead to blockage of the TD flow path and catastrophic system failure.)

- **Multiple tubes can be desorbed** into the focusing trap before secondary (trap) desorption and GC(MS) analysis. This offers one analysis for a ‘chain’ of multiple sorbent tubes used at a single monitoring point. It can also be used to boost sensitivity for ultra-trace level monitoring applications.

TC-20 - Multi-tube conditioning and dry-purge unit**

- The TC-20 offers cost-effective, **simultaneous conditioning of up to 20 tubes**
- Conditioning temperatures from 50 to **400°C**
- The TC-20 also allows ambient temperature **dry purging** of up to 20 tubes simultaneously - **in the sampling direction**
- Delivers same flow through all tubes, however many or few are attached
- Allows use of low cost N_2
- Frees up TD-GCMS analytical capacity

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Application areas for thermal desorption
(www.markes.com)

Environmental and occupational health & safety

Residual volatiles & materials emissions testing

Food, flavour, fragrance & odour profiling

Military, forensic and counter-terrorism

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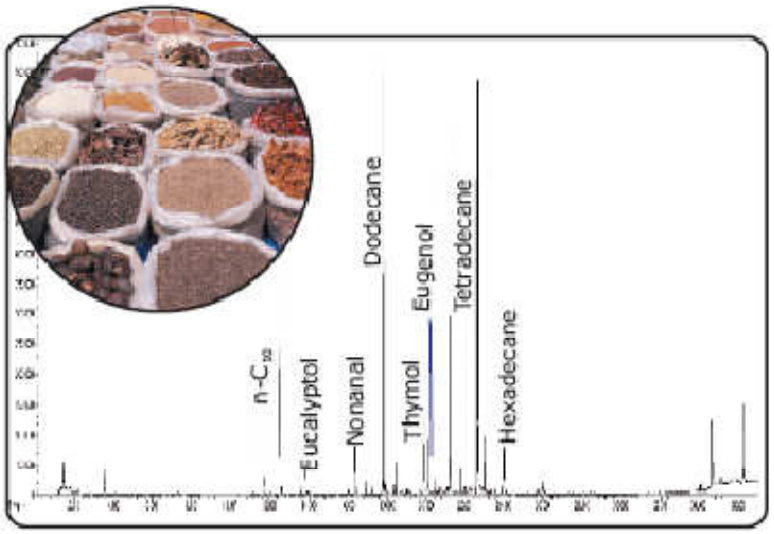
Direct Desorption

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

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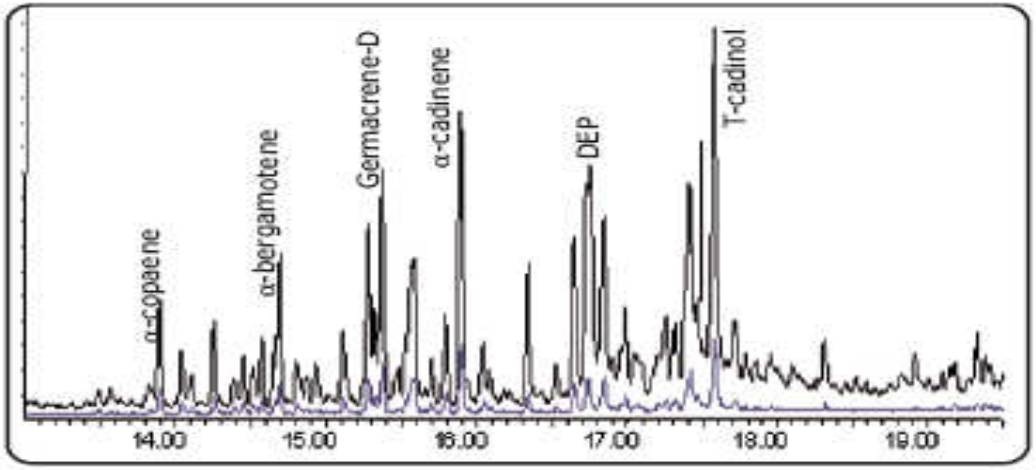
Direct Desorption

Natural products: Fragrance profiling

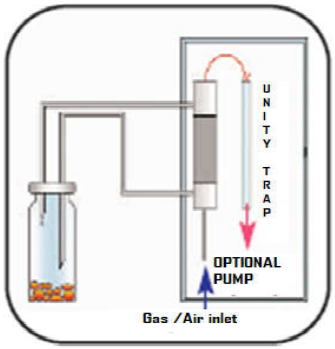


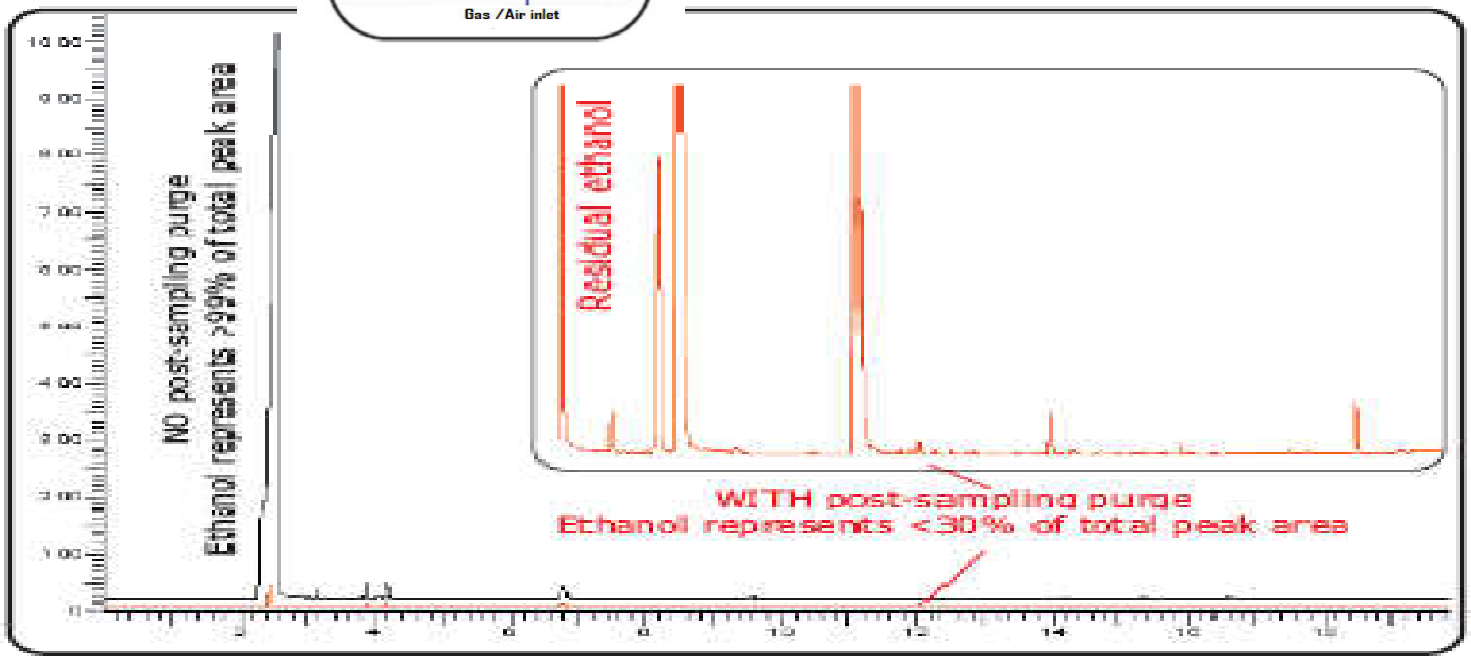
Vapours extracted from a leaf sample. Direct desorption (blue trace) followed by repeat analysis of re-collected sample (black trace) run with lower split ratio to enhance sensitivity

Typical analytes:
Range of terpenoid compounds, including:
α-cedrene, α-cadinene and T-cadinol

Concentration: Sub to low ppm

Aroma Analysis in Whisky

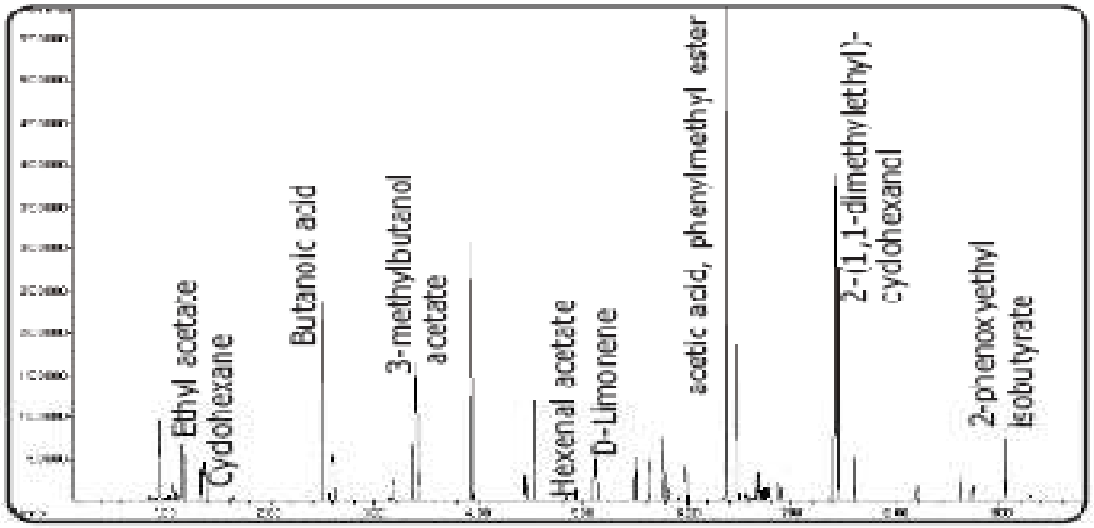




Purging of water and ethanol from whisky HS vapours allows selective concentration of key olfactory compounds – ketones, esters, *etc.*

Micro Chamber

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

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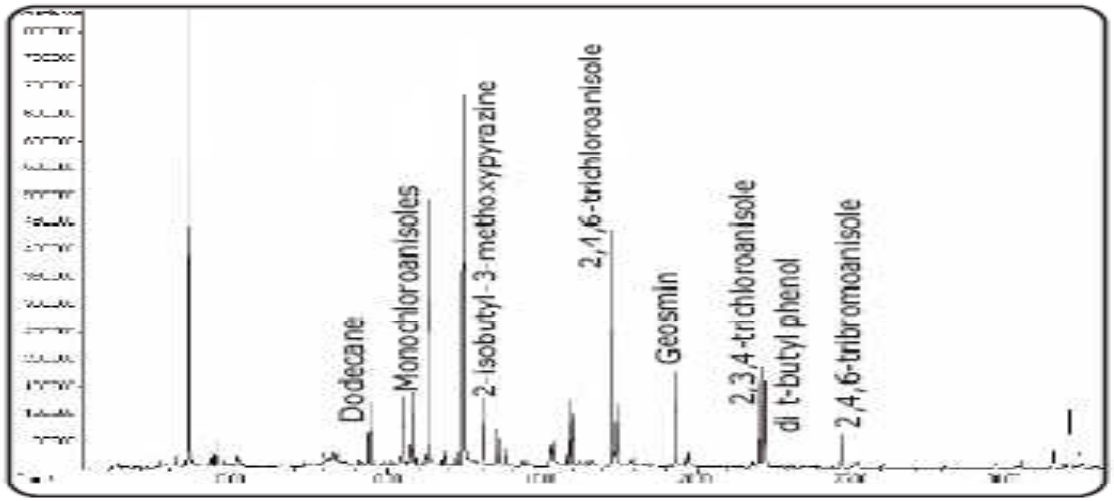
SPE-td

SPE-tD extraction of organics from drinking water



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

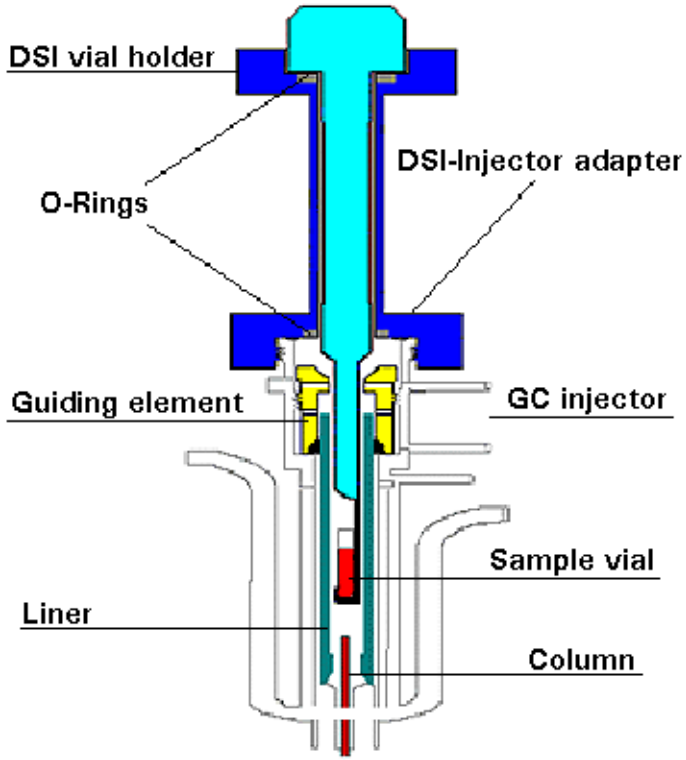
Typical analytes:
Geosmin, methyl isoborneol, phenols and trichloroanisoles

Concentrations: Sub to low ppb

Chromatoprobe

Direct sample Insertion:

Sample Amount
Resolution
Targeted Analysis



(Adapted from Proff Amirov website for understanding and discussion).

Conclusion

Thank You

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