Breathe Easier
Thermal desorption systems
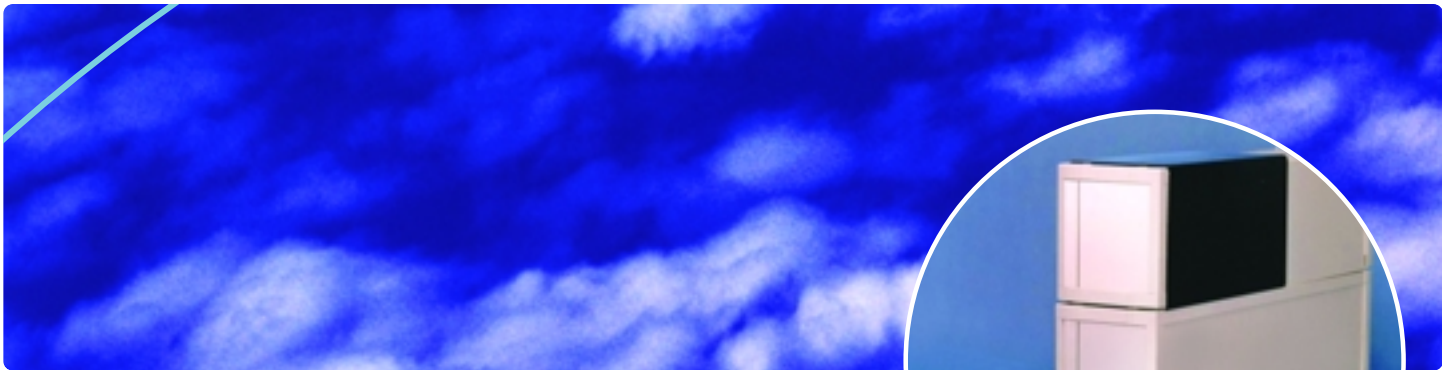
Thermal desorption systems for environmental health and safety monitoring

UNITY thermal desorber

Agilent has partnered with Markes International to bring you a complete system for the analysis of volatile and semivolatile compounds in air. In combination with Agilent's industry leading GC and GC/MS instruments, the Markes UNITY, ULTRA TD, and UNITY-Air Server products provide you with the most versatile, state-of-the-art systems for air analysis. The heart of the thermal desorption (TD) system is UNITY, a single-tube thermal desorber that offers long-term reliability plus the precision and analytical performance required by both routine and challenging applications. It incorporates all of the sample protection features required for compliance with key international standard methods plus SecureTD™ a unique repeat analysis capability. Markes International thermal desorption systems are compatible with industry standard sorbent tubes (.25-inch O.D. x 3.5-inch long) in glass, stainless and coated steel, as well as canisters, bags and on-line air sampling. And it is 100% sold and supported by Agilent's renown sales and service organization. Key features of UNITY and all associated automated systems include:

Electrically-cooled focusing trap — no liquid cryogen

Efficient retention

UNITY's electrically cooled focusing trap retains ultra-volatile components without the use of liquid cryogen. Two, three or even four sorbents can be packed in series for quantitative retention and efficient desorption of wide boiling range samples.

**Rapid trap heating for efficient desorption**

Trap heating rates in excess of 60°C/sec result in uncompromised high-resolution capillary chromatography — even under splitless conditions.
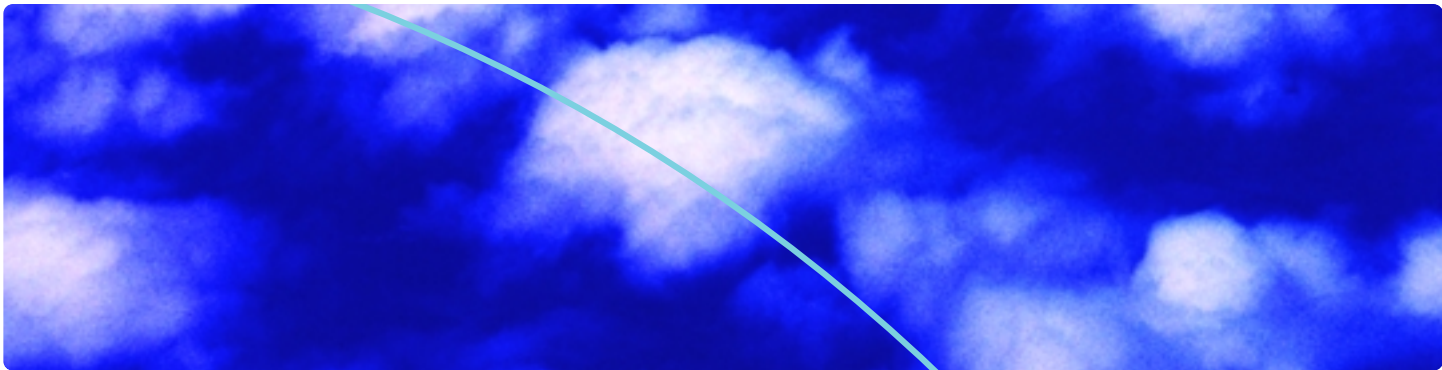
Easily changed

The trap is easy to change with a unique combination of o-ring seals and flexible connections.
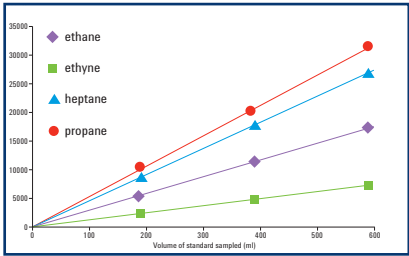
Retention of ultra-volatile compounds including acetylene (ethyne) from several hundred milliliters of air.
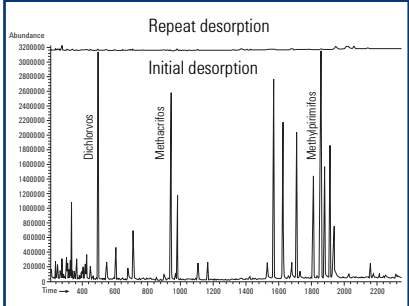
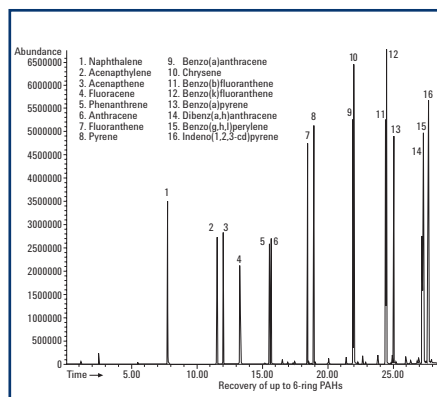No carryover confirms complete desorption of high boiling pesticides.
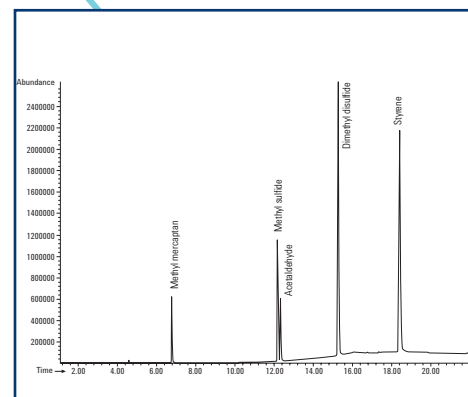
Short, inert flow path and valve[1]

Constructed entirely of inert materials, the short, narrow-bore flow path is efficiently and uniformly heated to prevent band dispersion and eliminate carryover.

This ensures compatibility with "sticky" compounds such as di-octyl phthalate, semi-volatiles and reactive compounds such as amines, mercaptans (thiols) and organophosphorous pesticides.

1 Patent Number GB 2336649

Abundance
6500000
6000000
5500000
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0

1. Naphthalene
2. Acenapthylene
3. Acenapthene
4. Fluoracene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benzo(a)anthracene
10. Chrysene
11. Benzo(b)fluoranthene
12. Benzo(k)fluoranthene
13. Benzo(a)pyrene
14. Dibenz(a,h)anthracene
15. Benzo(g,h,I)perylene
16. Indeno(1,2,3-cd)pyrene

Time → 5.00 10.00 15.00 20.00 25.00
Recovery of up to 6-ring PAHs

Recovery of up to 6-ring PAHs.

Abundance
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000

Methyl mercaptan
Methyl sulfide
Acetaldehyde
Dimethyl disulfide
Styrene

Time → 2.00 4.00 6.00 8.00 10.00 12.00 14.00 16.00 18.00 20.00

Analysis of compounds that illustrate the inertness of the system.

From splitless to double split

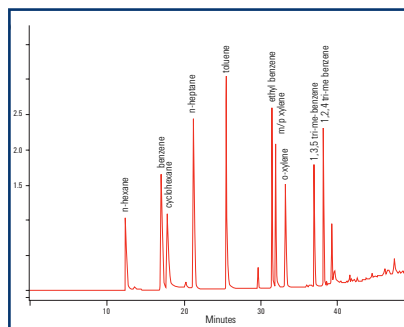### Splitless operation for optimum detection

The desorption of UNITY's cold trap is so efficient that it is capable of splitless operation with high-resolution capillary chromatography. This facilitates trace-level monitoring.

Single/double split operation

UNITY also supports single and double split capability for high concentration industrial monitoring and the analysis of % level volatiles in materials.

Optional electronic mass flow control (MFC) of split flows

UNITY can be configured with an electronic MFC module which enables split flows to be set and controlled through the Windows® based control software.

n-hexane
benzene
cyclohexane
n-heptane
toluene
ethyl benzene
m/p xylene
o-xylene
1,3,5 tri-me-benzene
1,2,4 tri-me-benzene

2.5
2.0
1.5

10 20 30 40
Minutes

Splitless analysis of volatile hydrocarbons in air for optimum sensitivity.
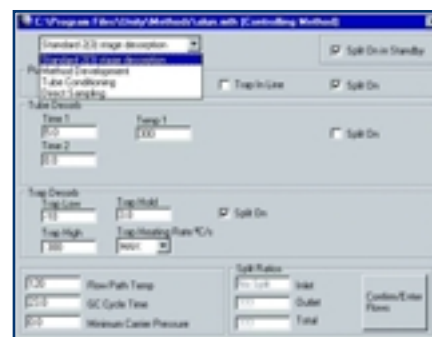
Compliance

Markes systems have been designed to satisfy the stringent performance and sample protection requirements of key thermal desorption standard methods such as US EPA TO-17, ASTM D-6196-97, ISO 16017, NIOSH 2549 and the UK HSE Methods for Determination of Hazardous Substances.
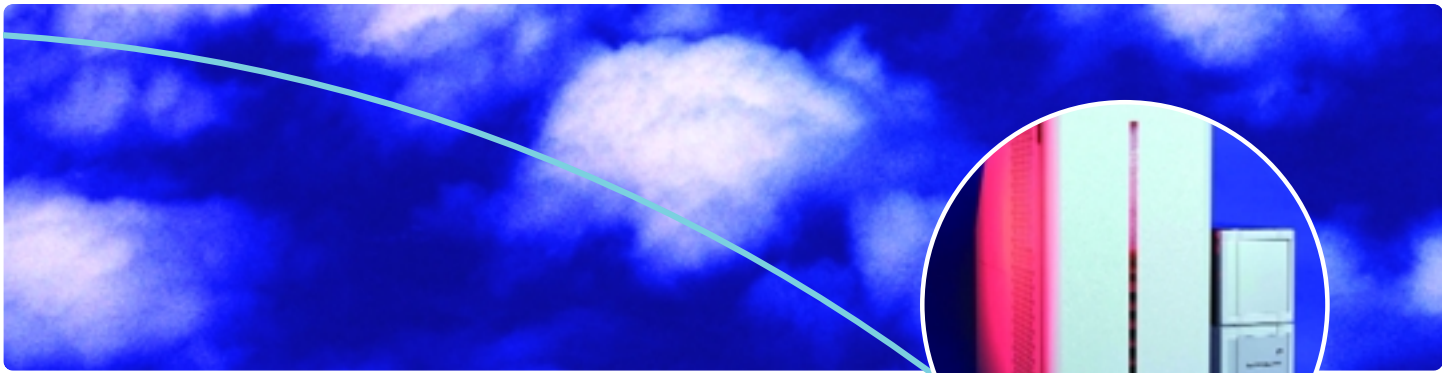
Versatility

With its inert flow path, multiple split options and wide temperature range, the inherent flexibility of UNITY is further enhanced by operational versatility.

Standard systems feature:

- repeat desorption of single samples at multiple temperatures
- a dedicated tube conditioning mode
- the option of purging at elevated temperatures before desorption

Software selection of the desorption mode using drop down menu.

UNITY & SecureTD™ – for complete confidence

SecureTD — complete confidence

All Mi systems feature SecureTD, a unique re-collection facility for the repeat analysis or archiving of critical samples. This provides ultimate confidence in analysis.

SecureTD allows a quantitative portion of each sample to be collected on a clean backup tube regardless of whether the original sample was in a tube or gas/air container.

The quantitative performance of SecureTD has been demonstrated for both routine and challenging applications. Chromatograms (a) to (c) show the analysis of a series of amines — data acquisition during the initial run ends prematurely, half way through the 4-Aminodiphenylamine (4A) peak. The subsequent blank chromatogram shows no system carryover. Analysis of the SecureTD tube allows the quantitation of 4A despite the failure of run 1.

Analysis and repeat analysis of high boiling amines using SecureTD.

Semi-volatile phosphorous pesticides. Repeat analysis confirms results and validates method.

SecureTD — TD method validation

Desorption of a re-collected sample should produce an exact match to the original chromatogram, but at a lower peak area — proportional to the split ratio used. By producing a replicate chromatogram or series of chromatograms from re-collected samples, SecureTD can be used to demonstrate lack of bias and quantitative performance. It is thus a convenient tool for method validation.

Sample protection

Markes International TD systems feature stringent and automatic leak testing of every tube before desorption. Failed tubes are retained intact for operator attention. Tubes are also purged with carrier gas to eliminate air, prior to desorption. This prevents sorbent and/or sample oxidation.

Sequence of re-collection and repeat analyses of phthalate plasticisers (including dinonylphthalate), illustrates use of SecureTD for validating analyte recovery.

ULTRA™ multi tube autosampler

UNITY can be simply upgraded by the addition of ULTRA — a multi-use autosampler for the automatic desorption of up to 100 industry standard tubes. The slimline design consists of 10 trays each containing up to 10 tubes. The combined ULTRA-UNITY™ system occupies only 41 cm of bench space. All key UNITY features — analyte range, sample protection, split flexibility, etc. — are maintained on systems upgraded with an ULTRA autosampler.

Unique sealing caps

Patented DiffLok™ caps[2]
Tubes on the ULTRA autosampler are sealed with DiffLok caps to preserve sample integrity. DiffLok caps provide an effective tube seal until pressure is applied causing gas to flow.

2 Patent Number GB 2337513

No loss of sample or ingress of contaminants
DiffLok caps maintain the integrity of both sampled and cleaned tubes via a unique diffusion limiting design. Tubes sealed with DiffLok caps show negligible ingress of contaminants or loss of sampled components over time. The cap on the sampling end of the tube has an inert coating to prevent breakdown of labile compounds.

Simple, reliable operation

No cap removal
As DiffLok caps do not need removing or replacing during analysis, the system offers unrivaled reliability.

Minimal mechanical movement
With a total of four linear movements, each capped tube in turn is lifted into the single desorption oven and sealed into the carrier gas flow path.

Low maintenance
The self-aligning, cone-shaped tube receivers provide a robust gas-tight seal while minimizing o-ring wear. O-ring seals last for well over 1000 operations and are readily accessible for user exchange.
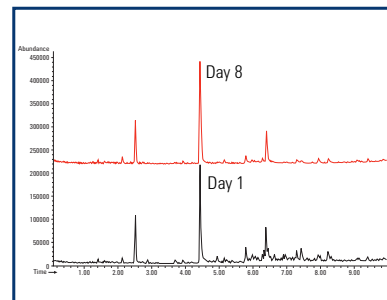
Analysis of conditioned Tenax tubes which had been capped with DiffLok caps for up to 8 days — showing no ingress of contaminants.
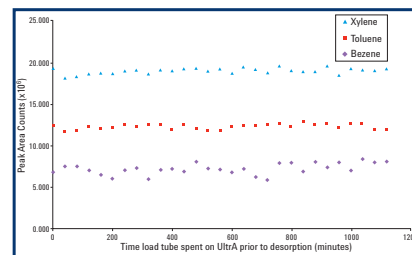
Analysis of 32 Tenax tubes simultaneously loaded with 88 ng of benzene, toluene and xylene. Tubes were capped with Difflok Caps and analyzed over a period of 24 hours.
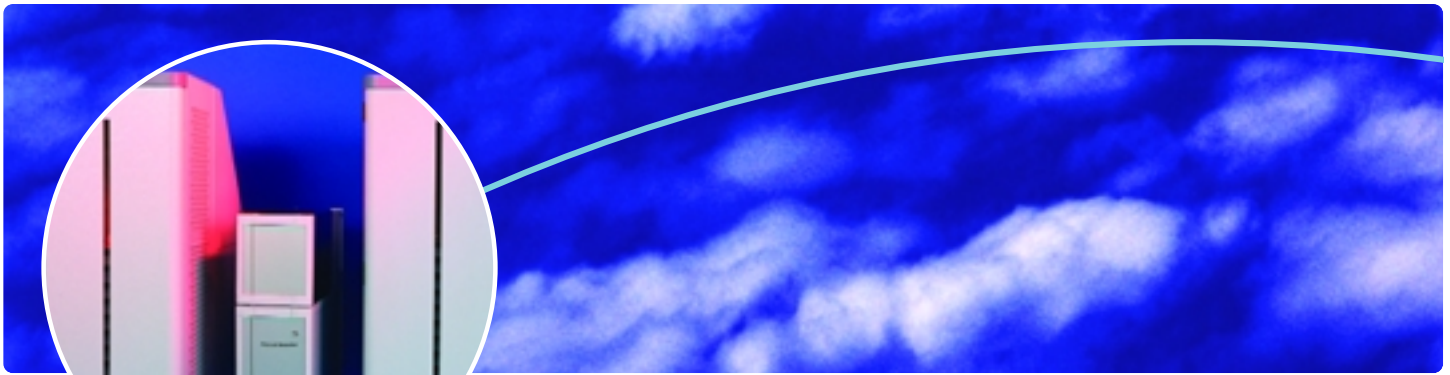
Windows® based control software

Intuitive control
Evolved from the UNITY user-interface, UltrA control software is intuitive and Windows based.

Sequence building
Automated sequences are easily constructed via the sequence builder. Tubes may be assigned individual desorption methods and are classified as sample, calibrant or blank. All sequences may be stored and recalled for future use.

Graphical sequence viewer
The sequence viewer gives a convenient graphical display of the position, classification and operating status of each tube. It also serves as a template for the operator when loading tubes.

Sequence reporter
Events associated with every analysis, such as the time and date of each tube desorption, and deviations such as tube not found or leak test failure, are all recorded in the sequence reporter. Any tube sequence failure triggers the GC start to keep the analyzer in step with the desorber.

Sequence builder showing sample set, type, desorption method to be used and tube number.

Sequence viewer showing the position and status of each tube in the sequence. Tubes are colour coded according to their type.

Sequence reporter showing the log of all events, including any deviations, associated with each tube desorption.

Mass flow control (MFC) of gas flow (optional)
This accessory provides closed-loop electronic control of the split flow throughout system operation. It allows independent user selection of different split flows for all 4 stages of operation — standby, prepurge, tube desorption and trap desorption — and facilitates the automatic sequencing of methods with different split ratios.

This accessory is also available for UNITY-only systems.

Internal standard/dry purge (ISDP) modules (optional)
As a QA check on every analysis, the ISDP accessory allows fixed volumes of gas-phase internal standard to be introduced from a gas sampling valve loop to the sampling end of each sorbent tube before desorption. Dry purging of tubes, in the sampling direction, before analytical desorption, is also possible using ISDP or using a separate Dry Purge only module.
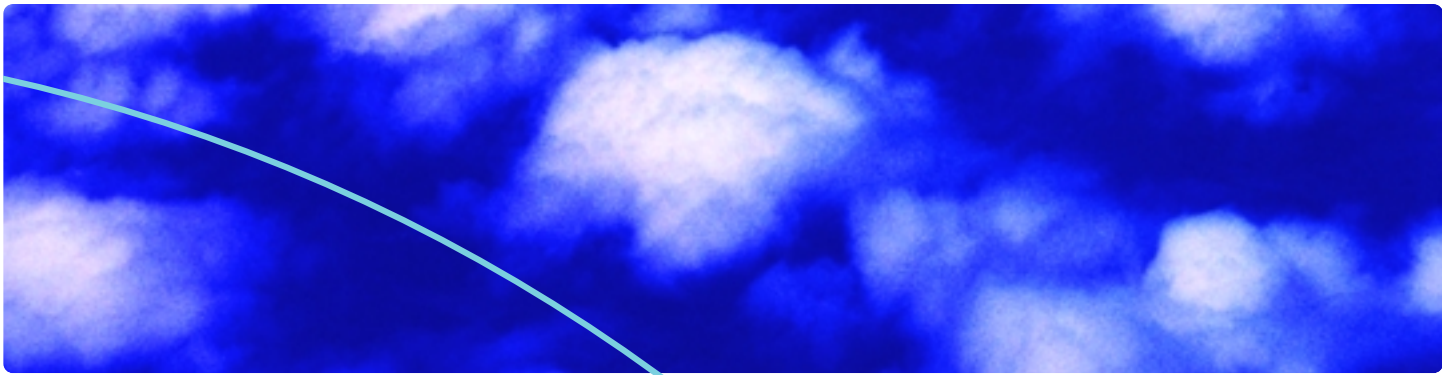
Additional water management features

Careful selection of trap sorbents and focusing temperatures allows selective elimination of water and other unwanted solvents in many cases, while still providing quantitative retention of volatile and semi-volatile target analytes.

Permeable membrane dryers are also available for on-line concentration using Markes desorption systems — see Air Server™ overleaf. These simplify the continuous monitoring of trace-level apolar volatiles using conventional detectors.

Analysis of volatile compounds from a 100 % relative humidity, 50 °C air sample with and without dry purge.
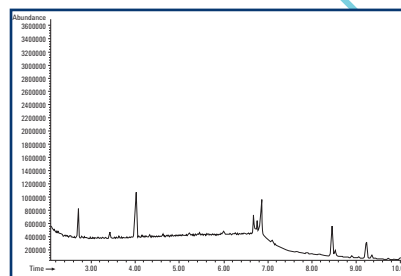
Without dry purge — the large amount of water present has given a high background and poor chromatography.
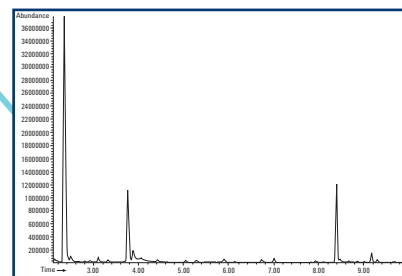
With dry purge — good chromatography and reduced background levels.

AutoSecure TD™ automated re-collection

An UltrA-UNITY system can be further enhanced by the addition of a second UltrA configured for SecureTD re-collection of every sample in a sequence. The second UltrA may be easily reconfigured to sampling mode for maximum operational flexibility.

SecureTD automation

Minimal bench space
Even with two autosamplers, a complete AutoSecure TD system only occupies 75 cm (2'6") of bench space.

Sample integrity
DiffLok caps are fitted to both desorption and re-collection tubes ensuring the integrity of primary and re-collected samples.

Sequence building
Automated sequences comprising desorption and re-collection are easily constructed and reviewed via the sequence builder.

Graphical sequence viewer
The sequence viewer gives a clear graphical display of the position and status of both desorption and re-collection tubes.

Interchangeable sample trays
Trays on both autosamplers are interchangeable, so re-collected tubes can be analyzed by simply transferring trays from one autosampler to the other.

Sequence builder showing a set of tubes for re-collection.

Sequence viewer showing the position and status of each sample tube and each SecureTD re-collected tube.

Air Server™ for on-line/canister automation

UNITY can also be upgraded by the addition of an Air Server autosampler for the introduction of whole-air/gas samples (canisters, bags or gas streams) directly into the cold trap, without the requirement for a sorbent tube. The process can be automated either for a sequence of multiple containers or for unattended, round-the-clock monitoring of gas/air streams.
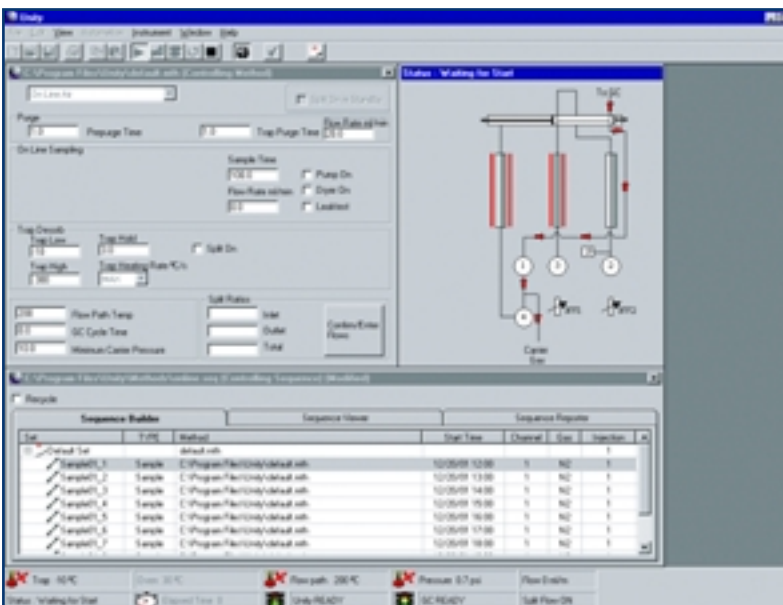
UNITY-Air Server™ operation

In on-line mode, users select sampling flow time parameters as part of the desorption method.

In operation, user-determined volumes of gas are pumped or drawn into the focusing trap at electronically controlled flow rates. Only inert, non-emitting components are allowed to come into contact with the sample and the flow path is purged with carrier gas between samples to eliminate carryover.

The standard system features automatic interchange between sample, zero and calibration gas streams at a user defined frequency. Sequences may be recycled indefinitely to minimize system programming. Custom solutions for up to 8 canisters or on-line air streams are available.
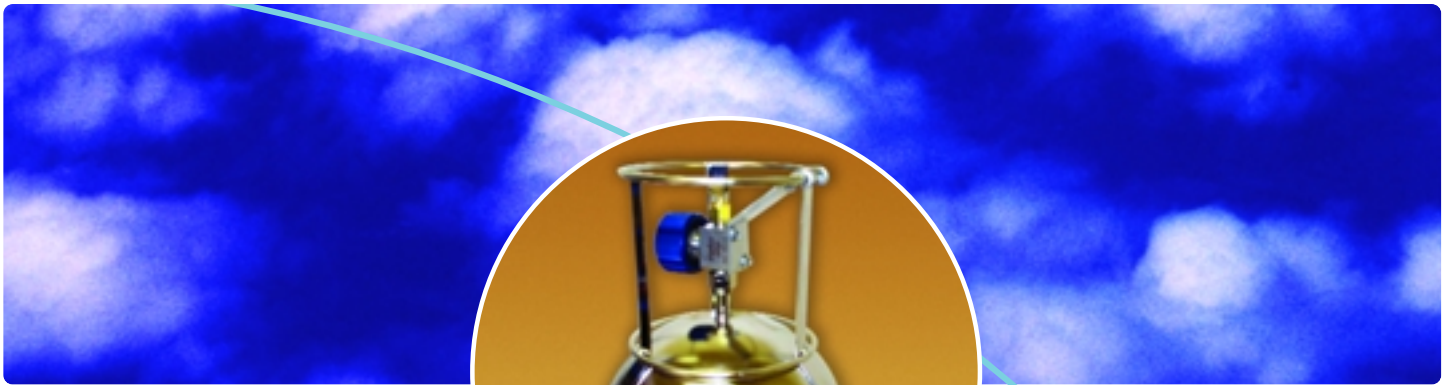
Timed start

It is possible to generate a series of analyses, configured to start at a pre-determined date and time with a pre set interval between each sample.

On-line sampling mode software screen.
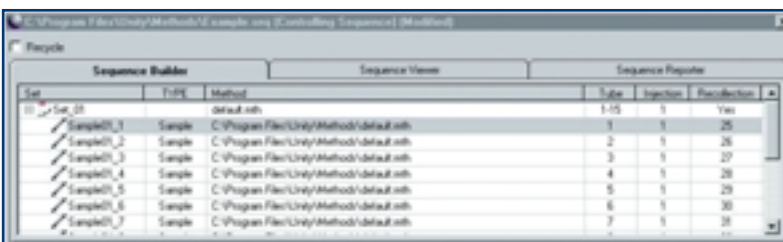
Sequence builder for Air Server operation, showing timed start for each analysis.

Air analysis solutions

The state-of-the-art electrically cooled focusing trap at the heart of the UNITY-Air Server meets and exceeds the specifications given in modern ambient air standards such as US EPA Guidance for Photochemical Assessment Monitoring (C2 to C10 ozone precursors), US EPA Method TO-15 and ASTM Standard D-5466.

Ozone precursors

A key application is the on-line monitoring of volatile hydrocarbons ranging from acetylene to trimethylbenzene — so called ozone precursors.

Quantitative retention of acetylene from 600 mL air and splitless desorption allows detection limits of 30 ppt (C4 hydrocarbons) and 50 ppt (C2 -C4 hydrocarbons) to be readily achieved with simple FID detection.

T014 / T015 canister sampling methods / Air Toxics

These methods are used to test ambient air for toxic compounds and current methodology (US EPA TO15) lists nearly 100 target VOCs — 'Air Toxics'.
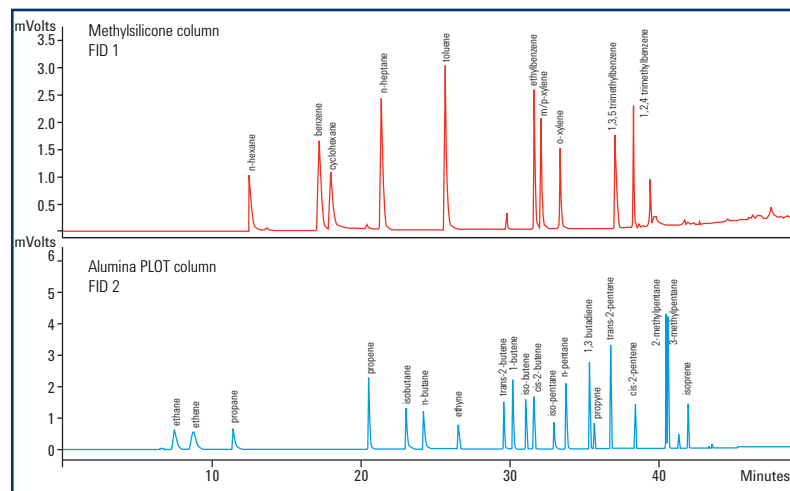
Samples may be taken on-line from ambient air directly into UNITY-Air Server, or may be sampled into specially prepared canisters to be transported to the laboratory for subsequent analysis via the UNITY-Air Server system.
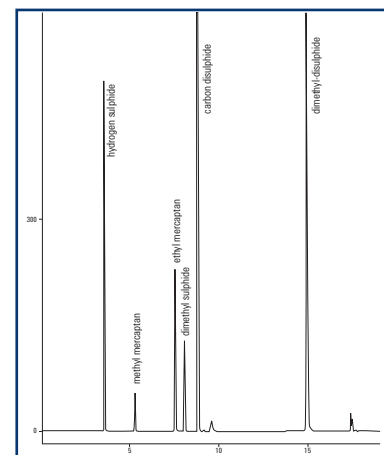
Odorous compounds

Volatile sulphur compounds (e.g. hydrogen sulphide, methyl mercaptan etc.) have a very low odor threshold and may need to be monitored round the clock in some locations.
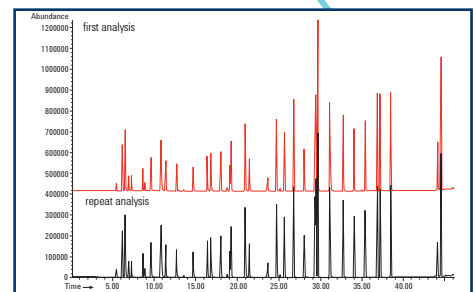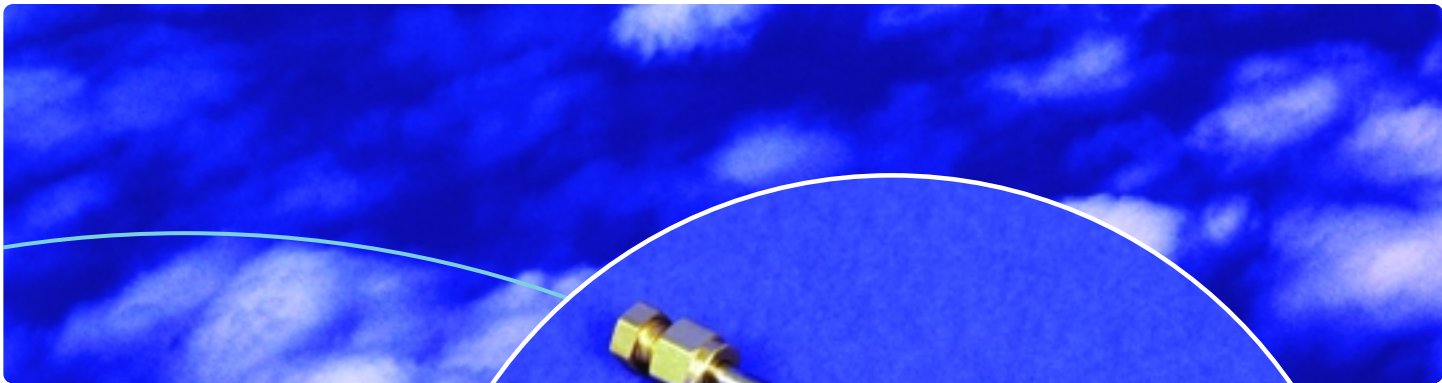

Repeat analyses of a TO14 'Air Toxics' mixture from a canister.

On-line sampling of ambient air for volatile sulphur compounds.

Splitless TD-GC-FID analysis of acetylene to trimethylbenzene ozone precursors using a 2-column Dean's switch system for optimum resolution.

Mi — Experts in specialized sampling

Markes International is a specialist provider of state-of-the-art thermal desorption instrumentation and ancillary sampling equipment. The company offers a comprehensive range of routine consumables (sorbent tubes, certified standards, diffusive samplers, etc.) and specialist sampling accessories. Whatever the application, highly trained staff are pleased to provide the technical expertise and detailed applications support required.

Industry standard sorbent tubes

These 3.5 x .25 inch tubes are available in glass, stainless steel or coated stainless steel and are all compatible with pumped (active) sampling. Both the stainless steel and coated stainless steel tubes are also compatible with passive (diffusive) sampling.

Agilent – the source for total solutions

To ensure the best performance and maximum efficiency from your Agilent analytical instrumentation for trace level environmental analysis, count on Agilent PerfectFit consumables and supplies. These include high-efficiency columns, precise syringes, perfectly-sized vials, high-quality septa, clean, deactivated liners and more.

High sensitivity. Fast separations.
For trace levels of analysis, Agilent J&W Scientific GC/MS columns offer unsurpassed sensitivity by supplying the best column inertness and low column bleed. For fast, high resolution HPLC analyses, look to our line of rugged and reliable StableBond and Eclipse ZORBAX Rapid Resolution, 3.5 µm columns.

Meticulously designed. Rigorously tested.
We test each and every GC and HPLC column that we manufacture. Our tight release specifications and manufacturing processes ensure high quality, reproducible chromatography from column to column.

Increase performance. Maximize efficiency.
Because productivity and system performance are critical to your everyday operations, Agilent provides a complete range of support and repair services to ensure that your Agilent instrumentation is operating at peak efficiency at all times. With an Agilent system, you'll have one of the most technically advanced, knowledge-able and dedicated phone support and service operations just a phone call away.

An unchanging commitment in a changing world.
With over three decades of experience in providing analytical instrumentation and productivity tools for the environmental industry, Agilent has made a strong com-mitment to the industry. Coupled with quality columns and supplies, unmatched technical support and service, Agilent is a single-source environmental testing partner, offering broad-based knowledge, experi-ence and technology.

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