

Series 2 **UNITY**

Thermal desorber

**A universal TD platform for tagged and
untagged tubes**



Series 2 UNITY

Thermal Desorption – GC(MS)

Thermal desorption (TD) is a highly versatile, sensitive and labour-saving sample preparation technique for the measurement of volatile and semi-volatile organic compounds (VOC and SVOC) in air and materials. It provides the ultimate sample introduction technology for GC/GCMS combining selective concentration enhancement with direct extraction into the carrier gas and efficient transfer/injection all in one fully automated and labour saving package.

TD is applicable to GC-compatible organics ranging in volatility from acetylene and freons to high boilers such as n-C₄₀, phthalate plasticisers and benzo- α -pyrene. It also offers quantitative concentration for some inorganic gases including; nitrous oxide, SF₆, CS₂ and H₂S. Key applications include:

- Environmental and workplace air monitoring
- Civil defence and forensic analysis
- Materials and materials emissions testing
- Food, flavour and fragrance profiling

Many material samples, such as polymers, paints, drugs, foods, textiles, etc. can be directly thermally desorbed. Weighed samples are heated in a stream of carrier gas allowing volatiles to be extracted into the gas flow, refocused and injected into the GC(MS) analyser as a discreet, concentrated band of vapour.

Alternatively, vapours in gas or air can be concentrated on- or off-line onto sorbent traps/tubes before TD-GCMS analysis. Several hundred litres of air or gas can be sampled and the vapours transferred/injected into the analyser in as little as 200 μ l of carrier gas. Concentration factors as high as 10⁶ can be obtained.

Thermal desorption is now recognised as the technique of choice for air monitoring (workplace and environmental) and is the subject of many international standard methods. Key examples include: EN ISO 16017, ISO 16000-6, EN 14662 (parts 1 & 4), ASTM D6196, US EPA TO-15 (canisters) & TO-17 (tubes), NIOSH 2549, UK Environment Agency guidance on landfill gas (LFGH 04) and US EPA guidance for on-line ozone precursor monitoring. Markes TD systems offer full compliance with all these standards.

Thermal desorption offers significant advantages versus solvent extraction (SE). Key benefits include: Typically 1000-fold enhancement in sensitivity, greatly enhanced recovery (>95% vs. 30-80 % with SE), re-usable sample tubes, no toxic solvent required and reduced analytical interference.

UNITY 2 Main Features

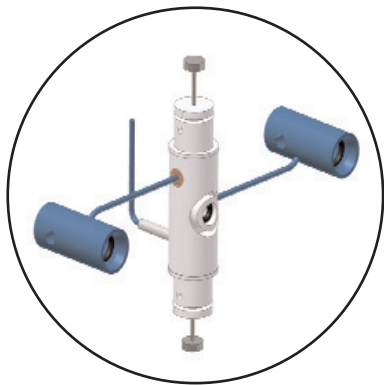
- Provides a **single platform for all TD applications** - labile species, semi-volatiles (up to n-C₄₀), ultra-volatiles (including C₂H₂ and freons), trace levels (ppt) and high concentrations (ppm/%).
- Repeat analysis *via* SecureTD-Q™: **Overcomes the one shot limitation** of conventional TD systems.
- **Cryogen-free** operation: Electrically-cooled, sorbent trapping to -30°C eliminates ice plug formation and reduces running costs.
- Compatible with 3.5-inch x ¼-inch OD sample tubes, with or without electronic tags. UNITY 2 allows an individual **TubeTAG™** to remain with a specific sample tube throughout its life, recording tube history and facilitating sample tracking between field and laboratory.
- **Fully method compliant** including stringent leak testing without heat or gas flow applied.
- **Fully upgradeable** to multi-tube, multi-canister and/or on-line automation. Can also be coupled with headspace for even greater sample flexibility.
- **Time saving** overlap mode allows desorption of a subsequent sample to begin while GC analysis of a previous sample continues.
- Plug-and-play TD: UNITY 2 adds to any commercial GC(MS) and is designed for **easy maintenance**.

Thermal Desorption (TD) Innovators

For over a decade Markes International has pioneered and commercialised enhancements to analytical thermal desorption instrumentation and associated sampling apparatus. The following examples of key technical advances have all been introduced by Markes since 1997 and now set the standard for TD instrumentation:

- SecureTD-Q (quantitative re-collection of split flow for repeat analysis), which overcomes the historical one-shot limitation of TD methods and simplifies method/data validation¹.
- Electronic tube tagging (TubeTAG)².
- Diffusion locking³ (DiffLok™) for enhanced sample integrity and robust (mechanically simple) automation.
- Patented inert valving⁴ for compatibility with every TD application on a single analytical platform – ultra-volatiles, semi-volatiles (up to n-C₄₀) plus reactive species - mercaptans, CS gas, etc. – all on one TD system.
- Automated internal standard introduction onto blank as well as sampled tubes.
- Electrically-cooled sorbent trapping with uniquely-fast trap heating rates for splitless capillary GC operation and optimum sensitivity without risk of ice formation.
- Off-line conditioning for multiple tubes without the need to blank-off unused tube connections.
- On-line desorbers with twin, electrically-cooled, reciprocally-operated focusing traps for truly continuous air monitoring (TT24-7™).
- Specialist sorbent tubes: Certified reference standards, SafeLok™ tubes³, Silcosteel™ tubes.
- A range of unique sampling tools, several of them patented⁵, for measuring volatile and semi-volatile organics in challenging matrices: liquids/solids/emulsions, breath, *in-situ* soil, polymers, natural products, construction products, etc.

1. Patent# GB 2395785 (Automated re-collection using a single TD autosampler), 2. Patent# US 6,446,515 B2, 3. Patent# GB 2337513 US 6,564656 B1
4. Patent# GB 2336649, 5. UK patent application reference 0501928.6 (μ-CTE)



Introducing the UNITY 2 Thermal Desorber

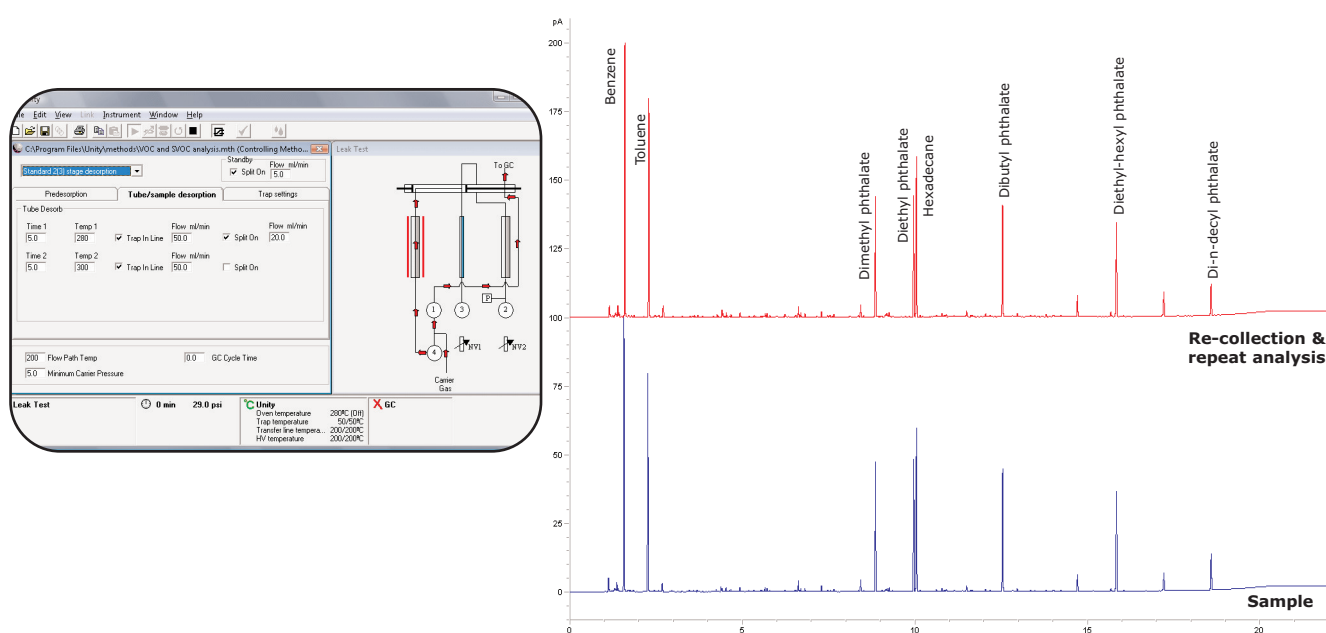
UNITY 2, Markes' next generation thermal desorption platform, harnesses this pedigree. It incorporates every feature required for TD method compliance, every system innovation of the last ten years and the field-proven reliability characteristic of all Markes TD instrumentation. Its introduction to the market place offers a state-of-the-art yet robust range of thermal desorbers with systems available at prices to suit every budget.

Building on the field-proven strengths of UNITY, UNITY 2 combines tube desorption with cryogen-free analyte re-focusing and efficient back-flush desorption of the focusing trap. Trap desorption injects analytes (split or splitless) into the GC capillary column in a narrow, focused band of vapour and triggers the GC(MS) run.

The patented, inert heated valve integrated into UNITY 2 facilitates the ambient temperature zero flow leak testing specified by standard methods and allows simultaneous analysis of volatile and semi-volatile compounds. The short, narrow-bore sample flow path is uniformly heated and optimizes analyte recovery.

SecureTD-Q™ (re-collection of tube and/or trap desorption split flow) offers quantitative repeat analysis and is available as standard on every UNITY 2. SecureTD-Q overcomes the one-shot limitation of older thermal desorbers.

UNITY 2 provides a method-compliant TD platform that is ideally suited to meet the ever more stringent demands of laboratory accreditation, data/method validation and good laboratory practice. It is operated using intuitive software loaded onto the same PC as ChemStation™.



Chromatogram showing simultaneous analysis of VOC and SVOC with Secure TD-Q repeat analysis validating recovery across the range

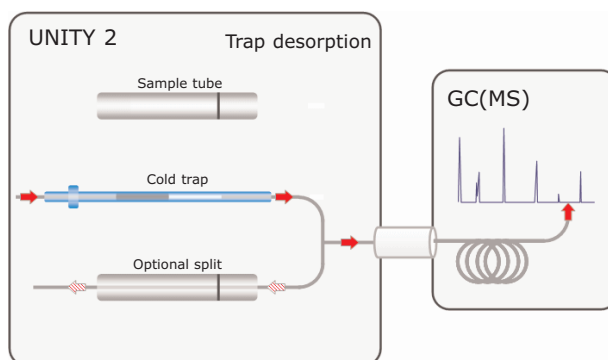
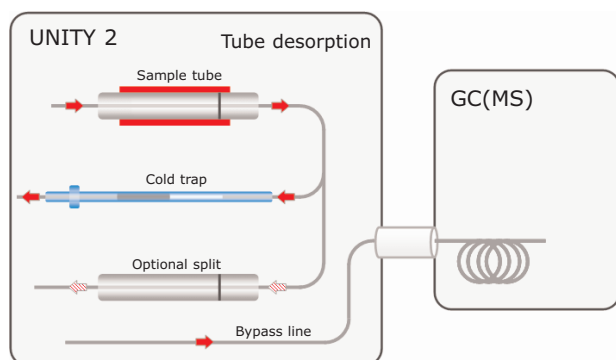
One TD platform...

Universal TD platform for all applications

UNITY is renowned for its application versatility and UNITY 2 takes this flexibility to another level. With extended electrical cooling of the entire 60 mm sorbent bed, the UNITY 2 focusing trap offers quantitative, cryogen-free retention of ultra volatiles such as freons and acetylene from larger volumes of air/gas. Markes offers a range of off-the-shelf pre-packed UNITY 2 traps, each containing up to 4 sorbents. Some of these are almost universally applicable, others are optimized for the retention of ultra-volatiles/volatiles (*e.g.* C₂ hydrocarbons/freons to n-C₁₂) and still others offer quantitative retention of target analytes with selective purging of water or solvents prior to analysis.

Tube desorption is now possible at temperatures above 400°C. Critical sections of the internal flow path have also been optimized, both for enhanced recovery of higher boiling 'sticky' compounds and quantitative analysis of reactive components. Example analytes include: 5/6-ring PAHs, phthalates, PCBs, hydrocarbons to n-C₄₀, mercaptans, explosives and chemical warfare agents.

The patented heated valve built into UNITY 2 is specifically designed for analytical TD. It can be operated at the low temperatures that are required for enhanced recovery of labile components and also allows quantitative recovery of high boilers such as n-C₄₀.

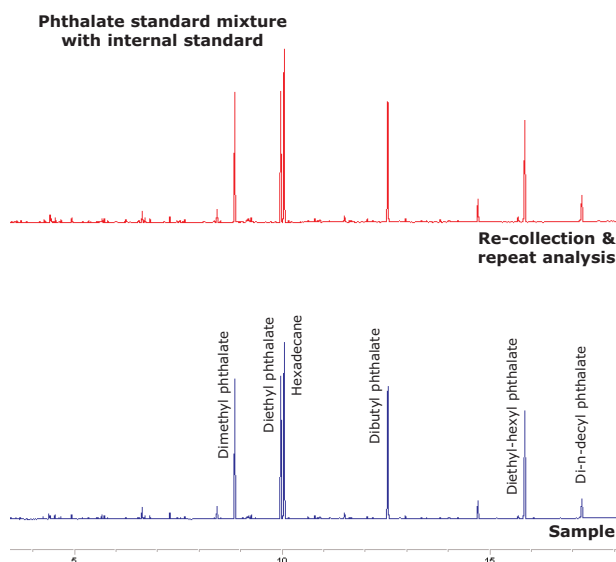
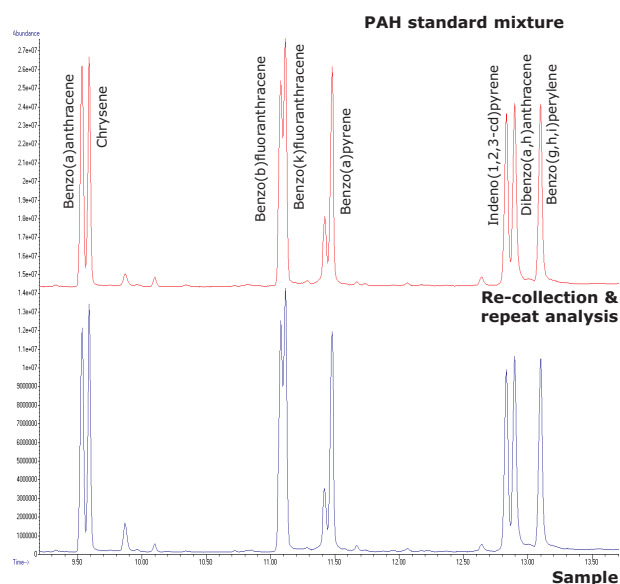
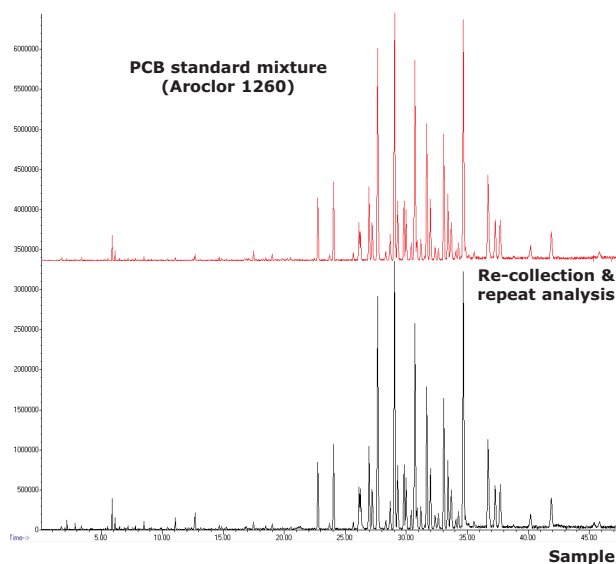


Schematics depicting the flow path of gas during tube desorption (left) and focusing trap desorption (right)

...every application

"60 mm sorbent trap length, combined with -30°C cooling, uniquely fast trap heating and patented TD valving ensure UNITY 2 offers optimum performance for the widest possible TD application range"

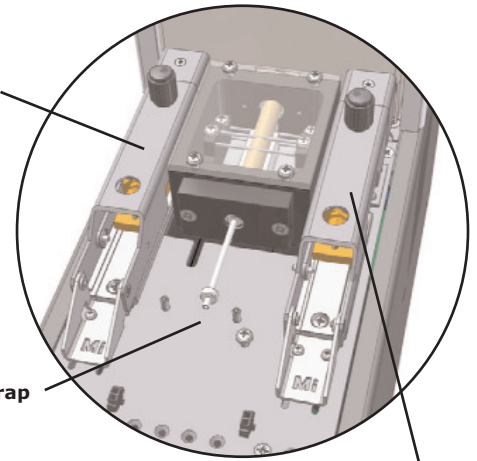
Quantitative recovery of high boilers (e.g. PCBs, phthalates and PAHs)



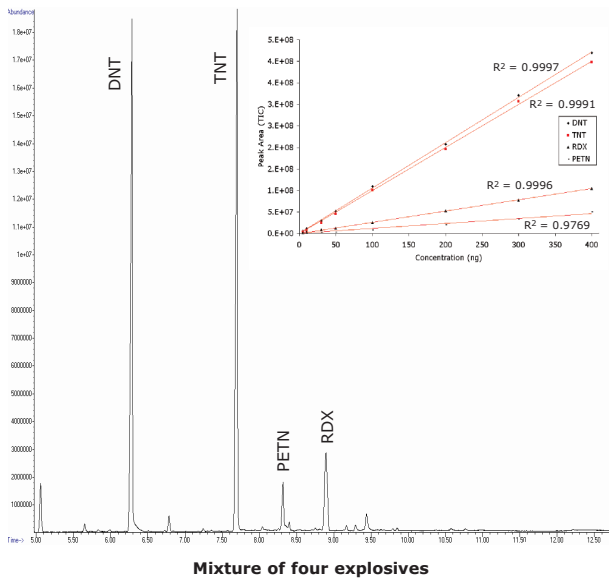
Sample tube desorption

Easy-change focusing trap

Split filter/re-collection tube



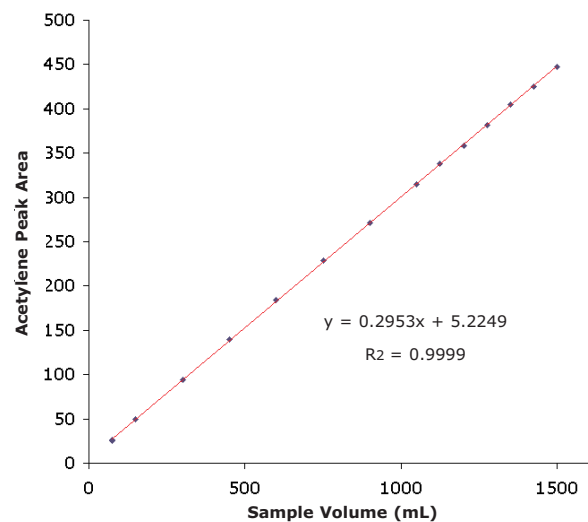
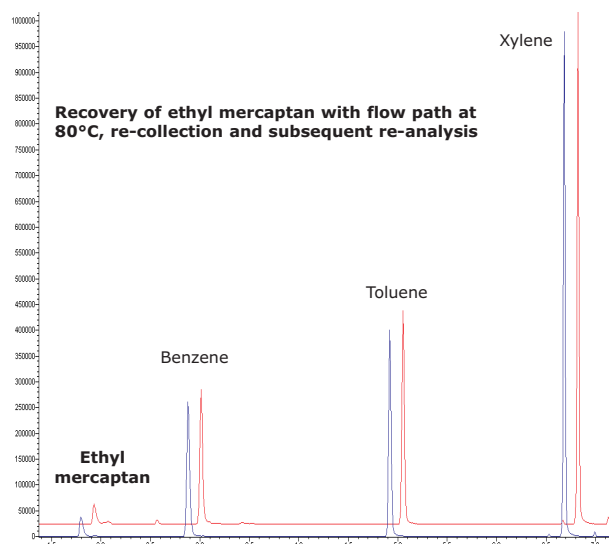
Compatible with labile analytes (e.g. explosives, mercaptans)



Enhanced user accessibility

Simplifying routine maintenance operations has been a key focus during UNITY 2 development. Changing the focusing trap, for example, is a simple user-enabled process. No tightening or loosening of fittings is required and a new easy-grip collar at the non-sampling end of each trap makes it very easy to withdraw and replace. Other relevant features include simple user access to split filters and transfer line connections. Sealing o-rings have also been selected both for extended life (typically at least 12 months) and negligible artifact formation.

Cryogen-free retention of ultra-volatile analytes from large volumes of air/gas





Sample tracking with TubeTAG

Error-free tube and sample tracking

Electronic tube tagging and tracking allows individual TD sample tubes to be tracked from laboratory to field and within a laboratory. RFID tags can be programmed with tube- and sample-specific data such as tube ID number, type and date of packing, number of thermal cycles, sampling times/flows and customer reference number. (More information is given in the TubeTAG brochure.)

TubeTAG is being heralded as a future standard requirement for large scale environmental monitoring projects, industrial hygiene applications, civil defence, *etc.* and is particularly important whenever monitoring data is being linked to human health/comfort or public safety. UNITY 2 can be used to analyse tagged tubes (tags do not have to be removed) thus simplifying the process of tracking tube history and performance. With TubeTAG compatibility as standard, UNITY 2 ensures a future-proof analytical platform.



UNITY 2 configured for both tube and canister/on-line automation



Unattended operation for extended periods

Featuring cryogen-free operation and lower consumption of supply gases than other brands of TD, UNITY 2 offers long-term unattended operation. It needs only 40 ml/min dry gas flow, which equates to >4 months continuous operation on a standard cylinder, making it ideal for continuous field monitoring, for example at remote environmental stations.

Fully upgradeable: Tubes, canisters & on-line gas streams

The UNITY 2 series TD range now includes options for automated sequencing of up to 100 tubes and up to 8 on-line air/gas streams or canister/bags. A single UNITY 2 platform desorber can be simultaneously configured for both on-line/canister sequencing and ULTRA 2 automation for up to 100 sample tubes.

UNITY 2 upgrade options include:

- The Universal Heated Inlet for interfacing **headspace vapours from custom sample vessels** directly to the UNITY 2 focusing trap.
- **On-line/canister sequencing.** Including 3- or 8-channel options for compliance with key methods such as continuous monitoring of trace-level ozone precursors (C₂-C₁₀ hydrocarbons) and US EPA Method TO-14/15 "Air Toxics".
- **Series 2 ULTRA 100-tube automation.** The ULTRA is available with built in options for everything from internal standard addition to automated sample re-collection.
- **Headspace (HS)-TD:** connection kits are now available for connecting UNITY 2 to the Agilent G1888 automated HS sampler. Combined systems offer **automated HS-trap**, full functionality TD and conventional equilibrium headspace all on one analytical system.

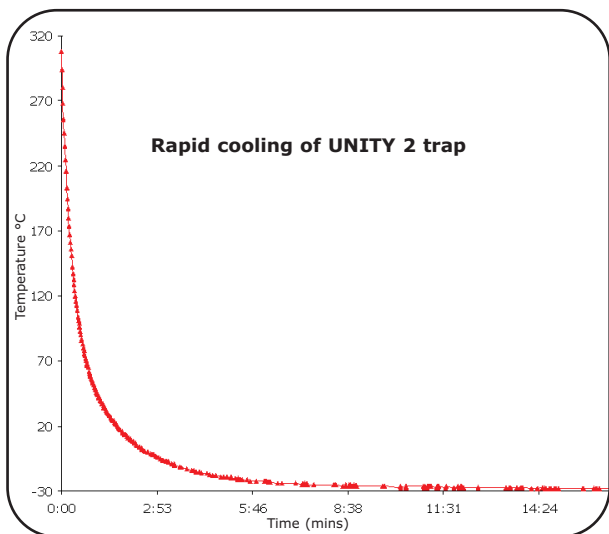
Simple connectivity

100-tube ULTRA and multi-channel canister/on-line options are now even easier to connect and disconnect from UNITY 2 as requirements change; UNITY 2 provides plug-and-play TD.

Please see relevant brochures for further information on all of these upgrade options.

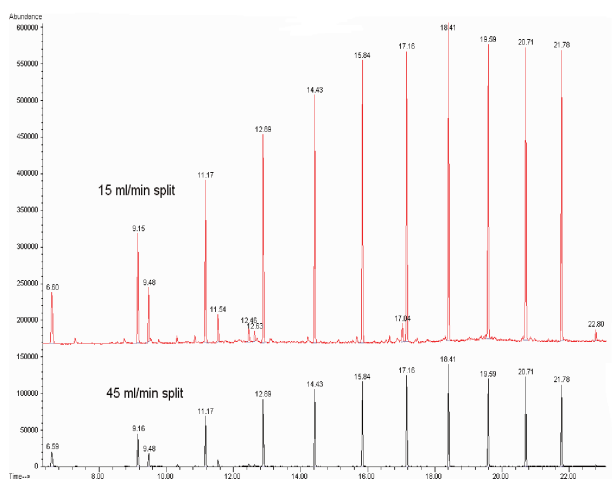
Fully upgradeable...fully flexible

Productivity & flexibility



Optimised productivity

UNITY 2 trap cooling times have been reduced to minimize cycle times and complement the system's expanded automation capabilities. This feature coupled with overlap mode (whereby a subsequent sample tube is desorbed while GC analysis of the previous sample is still ongoing) make UNITY 2 highly time efficient thereby optimising productivity.



Retention time locking for GC(MS)

Enhanced electronic control of gas flows

UNITY 2 is compatible with the back pressure regulated **electronic pneumatic control (EPC™)** of Agilent GCs. EPC offers precise control of carrier gas flow/pressure through the entire TD-GC(MS) system as part of the GC(MS) programme. This stabilizes retention times independent of TD analytical conditions (split flows, sorbents, desorption temperatures, etc.) ultimately aiding analyte identification. EPC also allows accurate monitoring and closed-loop control of the column head pressure, whatever the stage of thermal desorption operation and whatever the required flow.

Multiple splitting options

Building on UNITY's compatibility with analyte levels from sub-ppt to percent, every UNITY 2 offers unsurpassed split versatility. Users can select from splitless operation, single split (split open during either tube or trap desorption) or double split (split open during both tube and trap desorption.)

Every UNITY 2 features **SecureTD-Q** quantitative re-collection of all split flow, for repeat analysis and method/data validation (see overleaf and separate brochure.)

Accurate automatic control over split and desorb flows with MFC options

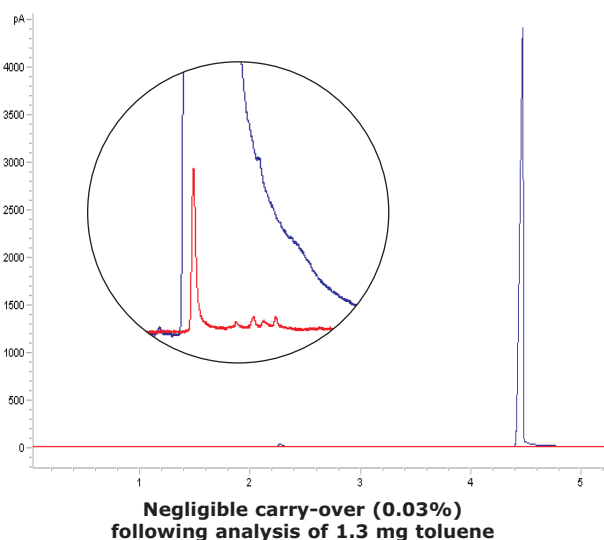


Automatic control over split flows

Standard UNITY 2 systems feature the full range of split options (splitless, single split and double split), but with manually controlled flow rates. Electronic control of split and desorb flows is offered by 1 or 2 optional **mass flow controllers (MFC) integrated into UNITY 2**. These provide versatile electronic control of both split and desorption flows, featuring flexible split flow rate selection combined with precise control at low flows. The UNITY 2 MFC modules also facilitate flow rate recall during an automated sequence with multiple TD methods.

Negligible carryover

With less than 0.01% carryover, UNITY 2 facilitates automated multi-method analysis. High level samples, such as stack gases or residual solvents, can be included in the same automated sequence as sub-ppb environmental air monitoring samples if required.

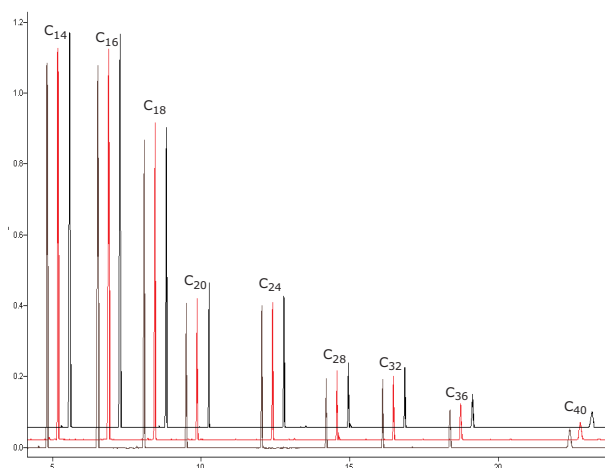


Building on the strengths of UNITY

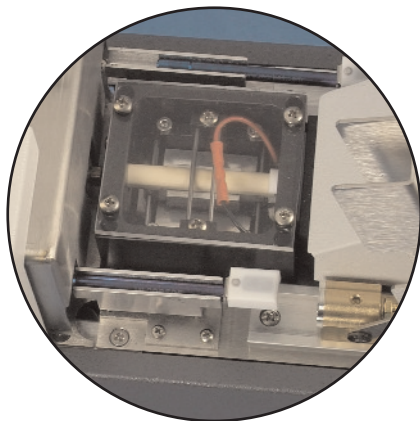
In addition to the enhancements described above, UNITY 2 has drawn key strengths from its eminent predecessor. Key features pioneered on UNITY and now available on UNITY 2 include:

SecureTD-Q

- **Repeat analysis:** SecureTD-Q (*i.e.* quantitative re-collection of any and all split flow during both tube and trap desorption) was pioneered by Markes International in 1998. Each sample is re-collected on a conditioned tube (tagged or untagged) and may be used for **repeat analysis, method/data validation or archiving of critical samples**. SecureTD-Q is available as standard on every UNITY 2 and **overcomes the one-shot limitation** of traditional TD systems and methods. (For more information see separate brochure.) No other single tube desorber offers quantitative recovery of all split flow for repeat analysis.



99% recovery of C₄₀ validated using SecureTD-Q

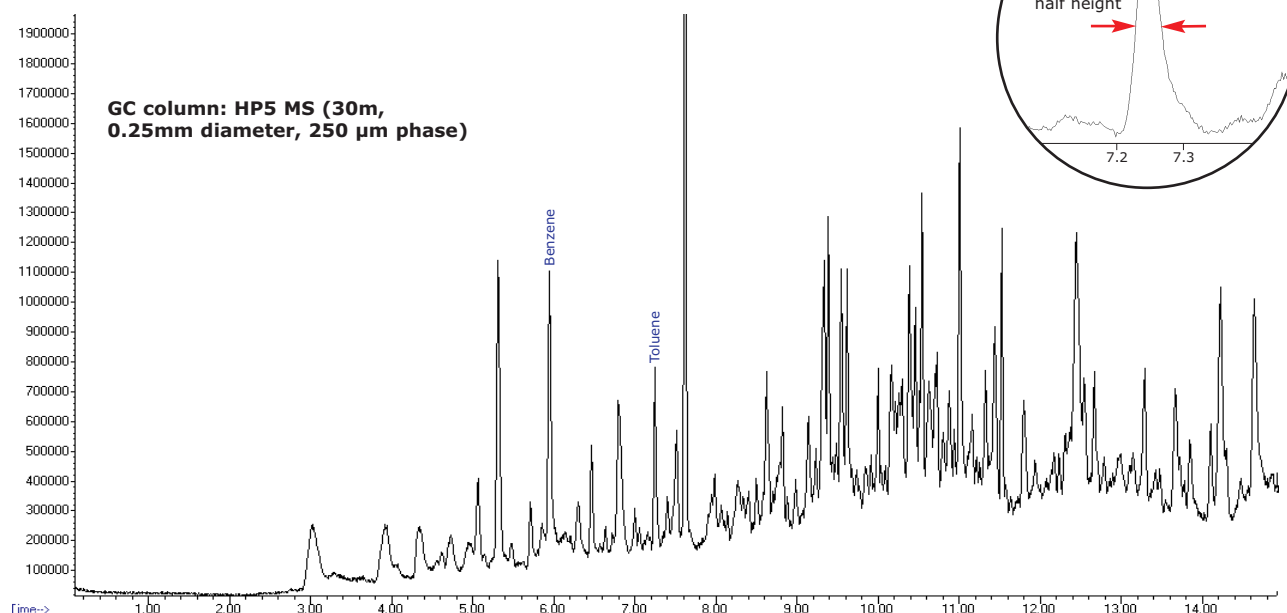


Peerless analytical performance

- UNITY 2 provides a universal TD platform for very volatile, high boiling and reactive compounds at high and low concentrations.
- Unsurpassed trap heating rates (100°C/sec) and unique trap heater design offer optimum desorption efficiency even under low flow (<2 ml/min) splitless conditions. This ensures **best possible detection limits for trace level compounds**.
- Pre-purge of air to vent and flexible water management options prevent risk of sorbent/analyte oxidation and **minimise analytical interference**.

Ease of use

- **Cryogen-free operation** for minimal running costs, optimum up-time and no risk of ice formation blocking the flow path during desorption of humid samples.
- **Small footprint.** At only 16 cm (6-inches) wide, UNITY 2 occupies minimal bench space.
- **Intuitive user interface.** Every TD method parameter is displayed on one easy-to-understand screen minimizing the operator learning curve.



Example of splitless desorption of indoor air contaminated with low levels of toluene and benzene
Insert showing good shape for toluene peak

Repeat analysis as standard

Method compliance and system validation

- **Full compliance with key international standard methods** such as EN ISO 16017, ISO 16000-6, ASTM D6196-03, US EPA Method TO-17, NIOSH 2549 and other similar standards.
- Stringent no-flow, ambient temperature **leak test to ensure data integrity**. Failed tubes are preserved intact for operator intervention. Fully interlinked ready/start signals ensure that trap desorption only occurs when the rest of the analytical system is ready for analysis.
- **Validation of desorption efficiency/analyte recovery** is simplified using SecureTD-Q (see above). Use of repeat analysis for TD method/data validation is described in standard TD methods such as ASTM D6196.
- Continuous monitoring of carrier gas pressure, downstream of the sorbent tube and focusing trap. This pressure reading may be logged for every sample and for all stages of TD operation **allowing tubes with anomalously high back pressures to be identified**. Moreover, if a tube is completely blocked this is detected by the

UNITY 2 leak test and logged as a sample failure. In the case of tagged tubes, back-pressure can be manually recorded on the tag and stored as part of tube history if required.

Productivity and versatility

- UNITY 2 can be **upgraded** to automatic tube desorption and/or automated canister/on-line analysis and/or headspace-trap.
- **Time saving overlap mode**: When one sample is being analysed *via* GC(MS) another can be leak tested and desorbed.
- **System versatility**. UNITY 2 is compatible with any commercial GC(MS). The selective concentration offered by UNITY 2 allows target analytes to be quantitatively retained while unwanted interferents (water, solvents, *etc.*) are purged to vent.

Example international standard methods for thermal desorption

US EPA Method TO-17: Pumped monitoring of ambient air

EN ISO 16017: Pumped or diffusive sampling of ambient, indoor and workplace air, plus materials emissions

ASTM D6196: Pumped or diffusive sampling of ambient, indoor and workplace air plus materials emissions

ISO 16000-6: TD-GCMS/FID analysis of materials emissions collected using emission test cells or chambers

ASTM F1982: TD-GCMS analysis of compounds of interest for semi-conductor applications

EN 14662: Benzene in ambient air

Various occupational hygiene-related national standards, including **NIOSH 2549 (US)** and **MDHS 72/80 (UK)**

Automated TD requirements for compliance with standard methods

No flow, ambient temperature **leak test**

Dry purge/internal standard addition in sampling direction

Pre-purge of air to vent to prevent interference

Inert flow path for compatibility with reactive compounds like mercaptans

Cryogen-free focusing trap desorbed rapidly in backflush mode for simultaneous compatibility with very volatile and semi-volatile analytes

Tubes sealed throughout autosampler operation

Quantitative repeat analysis for method and data validation (ASTM D6196-03)

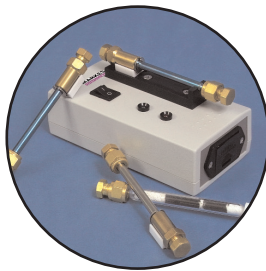
Markes International...

Markes International: Everything for thermal desorption

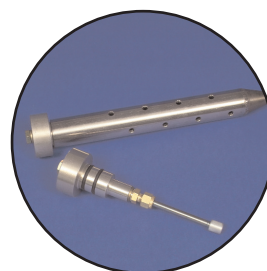
UNITY 2 is complemented by the comprehensive portfolio of thermal desorption instrumentation and associated sampling equipment provided by Markes International. Many of the innovative and labour-saving accessories available are unique to Markes including specialist low flow sample tubes, multi-sample test equipment for materials emissions screening, calibration accessories, breath samplers, soil probes, *etc.*



Wide range of empty and prepacked TD sample tubes



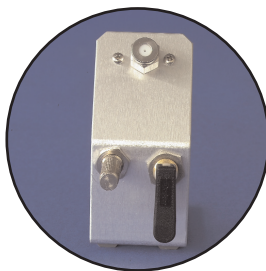
TubeTAG RFID tag system for sorbent tube informatics



VOC-Mole soil probes for *in situ* monitoring of contaminated land



TC-20 multi-tube conditioning/dry purge unit for up to 20 tubes



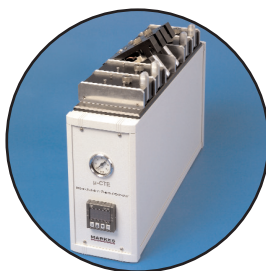
Calibration accessory for TD tubes



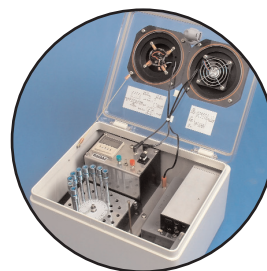
Canisters and related accessories



Bio-VOC sampler for collecting alveolar breath samples & transferring them to sorbent tubes



Micro-Chamber/Thermal Extractor (μ-CTE) for measuring emissions from materials & consumer products



MTS-32 sequential pumped sampling onto multiple tubes

TD application guides

A series of TD application guides is available from Markes International. Titles include:

- Environmental Air Monitoring and Occupational Health & Safety
- Residual Volatiles & Materials Emissions Testing
- Defence and Forensic
- Food, Flavour, Fragrance & Odour Profiling

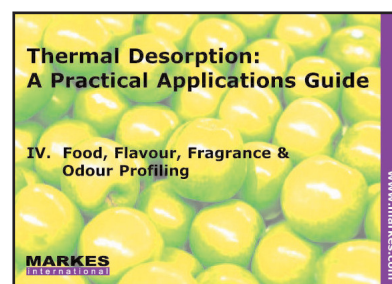
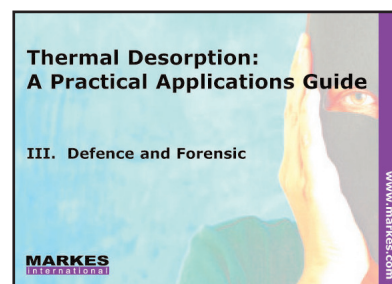
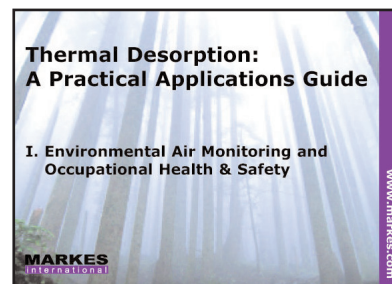
Each 16-32 page guide features example TD applications with key analytical conditions.

Trademarks

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EPC and ChemStation are all trademarks of Agilent Technologies.

Silcosteel is a trademark of Restek Inc., USA



...experts in thermal desorption

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