



Series 2 **ULTRA** Thermal Desorption Autosampler





Multi-function TD autosampler with optional reading/writing of electronic tube tags





Thermal desorption technology

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 is applicable to GC-compatible organics ranging in volatility from acetylene to $n-C_{40}$ and a few 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 drugs, foods, textiles, polymers, paints, *etc*. can be directly thermally desorbed by weighing them into empty TD sample tubes.

Alternatively, vapours in gas or air can be concentrated on- or off-line onto sorbent tubes/traps before TD-GC(MS) analysis.

ULTRA 2 Main Features

- Adds 100-tube automation to any UNITY or UNITY 2 TD platform to increase productivity.
- TubeTAG[™]: Reading/writing of **RFID tags on sorbent tubes is now fully integrated with automated TD operation**. This revolutionizes tube and sample traceability.
- **Stringent sample sealing**, before and after desorption. Patented DiffLok[™] caps preserve sample and blanktube integrity and ensure compliance with standard methods.
- Quantitative sample re-collection (SecureTD-Q) is offered as standard on every series 2 ULTRA-UNITY system for repeat analysis of critical samples and/or method validation.
- Automation of SecureTD-Q available using single or double ULTRA configurations; totally tag-compatible for fail-safe tracking of which sample was re-collected onto which tube.
- **State-of-the-art TD analytical performance.** ULTRA 2 simply adds automatic tube processing to the peerless thermal desorption analytical performance of UNITY 2.
- ULTRA-reliability field-proven, mechanically-simple automation ensures robust operation.
- Method compliance incorporates the ambient temperature/no-flow leak test of UNITY 2 as a guarantee of data integrity. Option of internal standard addition to blank or sampled tubes as a further aid to analytical quality assurance.
- Optional **dry-purging of sorbent tubes** (in the sampling direction) as part of the automated sequence. This minimizes water interference and is recommended by standard methods.
- Innovative, compact design minimizes bench space.

Innovation and excellence in thermal desorption

Since 1997 Markes International has re-engineered analytical thermal desorption for the 21st century. Harnessing unparalleled technical expertise, the company has developed a suite of 'universal' TD systems and unique sampling accessories incorporating key proprietary innovations such as:

- SecureTD-Q[™] for repeat analysis and automated re-collection using a single TD autosampler¹
- RFID tube tagging (TubeTAG[™])²
- Diffusion-locking for effective tube sealing and robust automation³
- Innovative low volume valving specifically designed for TD⁴

Many of these innovations now set the standard for TD instrumentation worldwide.

Series 2 ULTRA

ULTRA 2 is an innovative, robust and method-compliant thermal desorption (TD) autosampler for up to a hundred industry standard (3.5-inch) sample tubes, featuring the option of integrated reading/writing of RFID tube tags. It adds to any series 1 or series 2 UNITY TD platform to provide unmatched TD-GC(MS) analytical performance and extended unattended operation *e.g.* through an entire weekend.





Method-compliant automation

Series 2 ULTRA adds to any UNITY or UNITY 2 platform desorber to offer unattended thermal desorption of up to 100 capped tubes. The slimline design consists of ten horizontal trays each containing up to ten tagged or untagged tubes. Each tube in turn is loaded into the analytical position and sealed into the carrier gas flow path. The following analytical steps then all take place on the ULTRA 2 autosampler:

- Ambient temperature/no flow leak testing
- Optional dry purging and/or internal standard addition
- Pre-purge of air from the tube
- Primary (tube) desorption

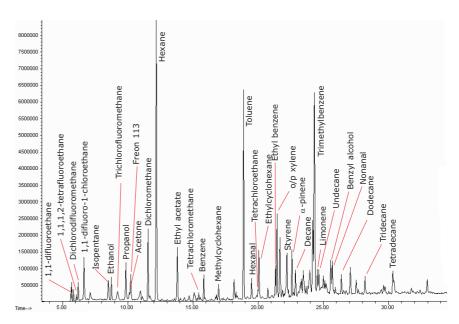
As vapours desorb from the primary sample tube in U_{LTRA} 2 they are swept through the short, inert, heated link line into the electrically-

cooled focusing trap of UNITY (2) in a stream of carrier gas. All subsequent stages of thermal desorption (trap purge, trap desorption, triggering of the GC(MS) run, *etc.*) take place on UNITY (2) in the normal way.

Uncompromised TD analytical performance

Series 2 ULTRA automates UNITY 2 without compromising its peerless analytical performance. The combined system provides a universal TD platform for up to 100 3.5-inch tubes offering **simultaneous analysis of volatiles & semi-volatiles**, quantitative recovery of high boilers (including $n-C_{40}$) and the option of low flow path temperatures for compatibility with labile analytes such as mercaptans. Systems offer splitless capillary operation for trace level work and single or double split methods for high concentration samples such as stack emissions and residual solvent in materials.

Series 2 ULTRA-UNITY systems maintain all the analytical advantages of UNITY 2 (as described in the associated brochure) and offer the following additional benefits relating to the automatic processing of TD sample tubes.



Clean indoor air pumped onto multi-sorbent tube and analysed by TD-GCMS

DiffLok[™] caps seal tubes effectively & simplify TD automation



100-Tube automation optimizes productivity

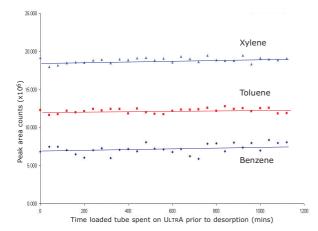
ULTRA 2 maximises the throughput and revenue generation potential of your TD-GC(MS) system. It offers capacity for up to 100 sample tubes together with overlap mode. Sample overlap means that desorption of a subsequent tube can begin while GC(MS) analysis of a previous sample continues – thus minimizing analytical cycle times.

With typical GC(MS) cycle times of 40 minutes, your series 2 ULTRA-UNITY TD-GC(MS) system offers unattended processing of 100 tubes over a standard 60-hour weekend; this represents significant revenue potential with minimal labour costs.

Patented tube sealing mechanism

Tubes on series 2 ULTRA autosamplers are sealed with Markes' unique patented DiffLok caps. DiffLok caps simply push on to both ends of every tube and preserve sample integrity by preventing both analyte loss and artifact ingress. Even volatile analytes are confidently preserved on sorbent tubes giving identical recovery for standards at the beginning and end of a 100-tube sequence. DiffLok caps have been field proven to seal sampled and blank tubes much more effectively than older push on cap designs (Ref 1).

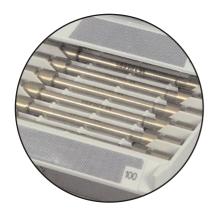
Ref 1: Diffusive Monitor Issue 9 (Nov 1997), Losses from ATD-400 by P. P. Ballesta







Highly efficient tube sealing



ULTRA-reliable automation

DiffLok caps remain on the sample tubes throughout automated analysis, thus simplifying the mechanical function of the instrument (no uncapping and recapping is required) and ensuring reliable operation. ULTRA's unsurpassed mechanical reliability has been extensively field proven.

DiffLok caps are available in either stainless steel or inert coated steel for compatibility with reactive components.

Method compliance

Series 2 ULTRA-UNITY systems are fully compliant with all TD standard methods and feature the mandated ambient temperature/no-flow leak test of UNITY (2) integrated seamlessly with the TD-GC(MS) sequence. Data processing remains synchronized with the analytical process at all times. Any tubes which fail the leak test are returned intact to the sample tray awaiting operator intervention. After a tube leak test failure ULTRA 2 records this in the sequence reporter and proceeds to load and leak test the next sample in the sequence.

TubeTAG reading/writing for enhanced tube traceability

Series 2 ULTRA autosamplers herald a revolution in automated thermal desorption. Every ULTRA 2 offers the option to read information from RFID tags attached to sorbent tubes and to automatically input this information into the sequence table. At the end of each analysis, ULTRA 2 can also write to the tube tag *e.g.* to increment the number of thermal cycles, input any high back-pressure anomolies, change the tube status and clear sample-specific data from the tag ready for the next field monitoring operation.

The combination of ULTRA 2 and TubeTAG offers a major step forward in analytical quality control for air monitoring and all TD applications.



TD automation with electronic tube tracking

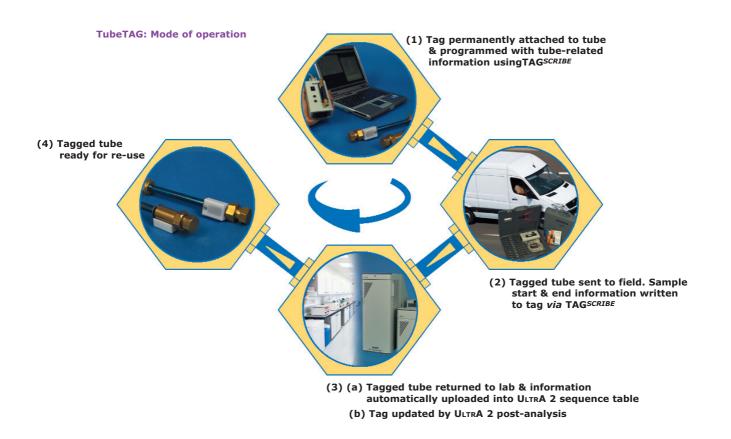


TubeTAG technology allows error-free tracking of samples from field to laboratory & within a laboratory (transit tagging) and logging of key tube-specific data such as type & date of sorbent packing.

In combination with a series 2 ULTRA-UNITY system TubeTAG further enables:

- Automatic logging of the history of a tube throughout its life: Number of thermal cycles, back-pressure anomolies, number of leak test failures, *etc.*
- Automatic input of sample-specific information to the ULTRA TD automation sequence table: Tube number, sampled volume, diffusive sampling time, date of sampling, *etc.*

ULTRA 2 complete with the TubeTAG read/write option is a genuine breakthrough in automated TD technology. Imagine a future in which you can instantly identify the sorbent(s) in each tube, when it needs to be repacked and whether or not that tube has had a history of leaktest failures or back-pressure issues. Imagine the benefit of error-free automatic input of key sample data into the automation sequence. This is what ULTRA 2 offers today.





SecureTD-Q: Quantitative sample re-collection

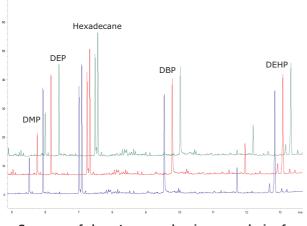
Every series 2 ULTRA-UNITY system features manual re-collection of the total split flow (*i.e.* the split during both tube and trap desorption) as standard. This allows repeat analysis of critical samples and simplifies method validation as per standard methods such as ASTM D6196.

Automated re-collection

ULTRA 2 also offers automated sample re-collection options using either a single ULTRA autosampler (see ULTRA *50:50* system) or double ULTRA configuration (see AutoSecure TD system).

Series 2 ULTRA *50:50* systems offer automated and quantitative re-collection of the trap desorption split flow for 50 or 100 tubes.

Series 2 AutoSecure TD systems, incorporating two ULTRA 2 autosamplers, offer quantitative re-collection of the total split flow (*i.e.* the split flow during both tube and trap desorption) for all 100 tubes.

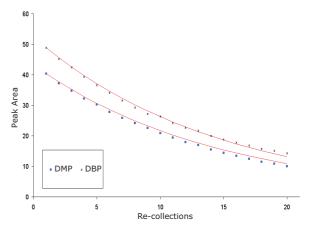


Sequence of chromtagrams showing re-analysis of re-collected phthalate mixture with internal standard

Automated re-collection now enhanced with TubeTAG

Use of series 2 ULTRA TubeTAG read/write capability during automated desorption/re-collection, greatly enhances the automation and traceability of SecureTD-Q. Automated desorption/re-collection systems incorporating TubeTAG log the unique ID numbers of original sample tubes and of the corresponding re-collection tubes, both on the respective individual tube tags and in the automation sequence. Subsequent interrogation of an individual tube tag can be used to confirm if that tube is either:

- A sample tube, which has now been desorbed, with the sample re-collected on tube ID# Mi 0XXXXX, or
- A tube that now contains the re-collected sample originally desorbed from tube ID# Mi 0YYYYY



Plot showing the theoretical and measured peak areas for repeated sample re-collection and re-desorption

Flexible upgrade options

ULTRA 2 options and accessories

The following options and accessories are available for $\ensuremath{\mathsf{ULTRA}}$ 2:

Integrated TubeTAG read/write capability

TubeTAG read/write capability is available for any series 2 ULTRA (see above) and can be factory configured or field installed. It is compatible with all other ULTRA 2 options and accessories.

50:50 Option

The ULTRA 2 can be factory configured with a "50:50" option for automated sample re-collection using a single TD autosampler. The resulting series 2 ULTRA 50:50 package is a patented, cost effective, space saving solution for automatic thermal desorption and **re-collection** of up to 100 tubes.

A standard series 2 ULTRA-UNITY offers manual re-collection of individual samples (see above). Addition of a *50:50* option to the ULTRA 2 complements this facility by offering automatic re-collection of trap desorption (outlet) split flow. This means that the outlet split flow from up to 50 samples can be recollected onto 50 fresh (conditioned) sorbent tubes. Alternatively, the outlet split flow from up to 100 samples can be re-collected back onto the original sorbent tubes.

The *50:50* option also facilitates **automatic dry purging** of sorbent tubes in the sampling direction prior to thermal desorption, as recommended in standard methods such as US EPA TO-17. All other features are the same as on standard series 2 ULTRA-UNITY systems.

For high throughput laboratories with a focus on data quality, series 2 ULTRA *50:50* offers cost-effective automatic re-collection for any single (outlet) split method. Sample and re-collection tubes are arranged in **colour-coded trays** for operator convenience and a clear user interface displays sequence status and the post run sequence report.

Error-free operation of series 2 ULTRA *50:50* is further enhanced by using the TubeTAG read/write option to link the identity of each desorbed tube to that used for sample re-collection (see above)



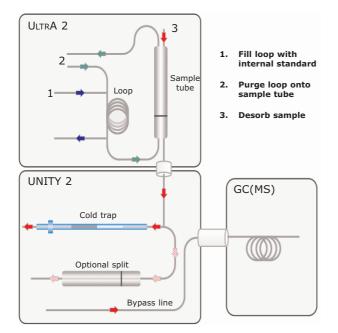
Parallel connection of automated canister analysis/on-line air monitoring also faciliated (see Air Server brochure)

Automating re-collection for repeat analysis

Manual or automated flow control

Series 2 ULTRA systems usually harness the manual or electronic mass flow control (MFC) incorporated into the attached series 2 UNITY TD for control of both desorption and split gas flows (see UNITY 2 brochure for further details). For added versatility series 2 ULTRA *50:50* units also offer the option of 1 or 2 integrated electronic mass flow controllers for special applications. These provide versatile electronic control of both split and desorption flows during an automated sequence with multiple TD methods.

Series 1 UNITY desorbers can also be upgraded with a series 2 ULTRA *50:50* configured with one or two integrated MFCs. Configuring a series 1 UNITY with a series 2 ULTRA *50:50* incorporating MFCs, brings both tube automation and enhanced electronic mass flow control of split and/or desorption flows to the original UNITY TD platform.



Schematic depicting a series 2 ULTRA (ISDP) - UNITY system

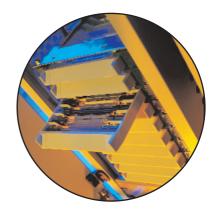
Internal Standard/Dry Purge (ISDP) option

An ULTRA 2 factory configured with an ISDP option, incorporates all the features of the ULTRA 2 autosampler but with the added benefit of an inbuilt internal standard (IS) addition and automatic tube dry purging capability. When using an ULTRA 2/ISDP system, a precise aliquot of gaseous internal standard is transferred from a gas valve loop to the sampling end of a sorbent tube immediately after the leak test and before tube desorption. Addition of IS aids analytical quality assurance and is recommended in standard TD methods. Typical compounds used as internal standards include toluene-d8 and bromofluorobenzene (BFB).

Gas-phase IS can be added either to sampled or **blank tubes**. When standard is added to the sampling end of blank tubes, they are not desorbed but replaced in the ULTRA 2 tube tray ready for field monitoring. In this case the internal standard provides a quality check on every aspect of the monitoring process – tube storage, transport, sampling, and analysis.

Dry purging of tubes, before analytical desorption, is also facilitated using ISDP. Tube dry purging is carried out in the sampling direction, with or without internal standard addition.

Note that Markes International also offers a range of stand alone off-line accessories for dry purging sorbent tubes and introducing gas or liquid phase standards.



ULTRA 2 with 50:50 and ISDP

Series 2 ULTRAS may be factory configured with internal standard addition, *50:50* automated re-collection plus dry purge capability, all on the same system.

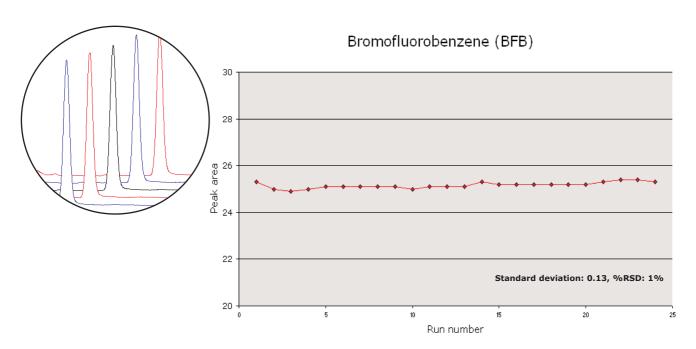
This comprehensive ULTRA 2 configuration is further enhanced by the addition of **TubeTAG** read/write capability for automatic logging and tracking of sample tubes, re-collection tubes and the history of each tube.

AutoSecure TD System: The ultimate in TD automation

Series 2 ULTRA-UNITY systems, with or without ISDP, can be further upgraded by the **addition of a second ULTRA 2** for automated and quantitative re-collection of total split flow, *i.e.* the split flow during primary (tube) desorption (inlet split flow) as well as split flow during secondary (trap) desorption (outlet split flow), for all 100 tubes. The slimline design of ULTRA 2 lends itself to this configuration and the total series 2 ULTRA-UNITY-ULTRA system **(AutoSecure TD)** only occupies 75 cm (30-inches) of benchspace. Tubes are sealed with DiffLok caps, both on the desorption and re-collection ULTRA, to preserve the integrity of sampled tubes, desorbed tubes and re-collection tubes.

The user interface is very straightforward, simplifying the sequencing process and may be further enhanced by the addition of TubeTAG read/write capability on both ULTRAs to automatically log sample tubes and link them with their associated re-collection tubes.

Trays on both autosamplers are interchangeable, so re-collected tubes can be analysed by simply transferring trays from one series 2 ULTRA to the other.



ISDP reproducibility data: 24 repeats over a 24 hr period

Unrestricted upgrade path: Plug-and-play TD

All series 2 ULTRA configurations may be readily connected and disconnected to any series 1 or 2 UNITY TD platform. ULTRA 2 can also be connected to UNITY (2) in parallel with Air Server technology for automated on-line air monitoring/canister analysis (see associated brochure). In addition to this, series 2 AutoSecure TD systems offer a unique and cost-effective route to increasing productivity: With the addition of a second UNITY 2, an AutoSecure TD system can be readily transformed into two standard series 2 ULTRA-UNITY systems. This can be used, for example, to increase sample throughput when demand for TD-GC(MS) analysis is especially high.

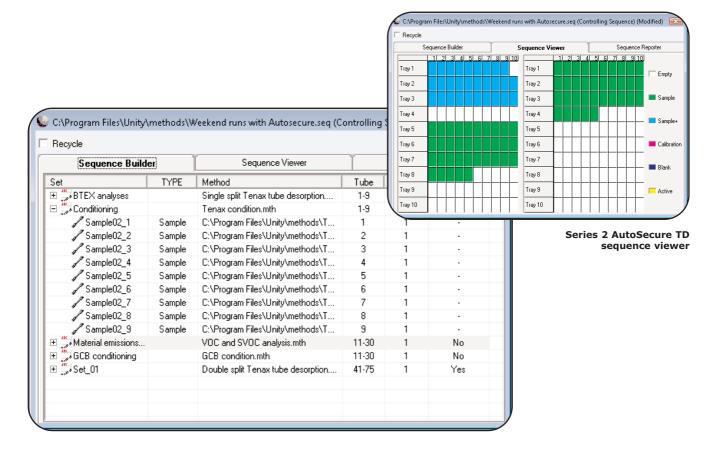
Intuitive control software

Series 2 ULTRA control software is intuitive and integrated with the UNITY (2) TD control software to provide one comprehensive and easy-to-understand user interface.

Sequence building

Automated sequences are easily constructed *via* the sequence builder. Samples may be assigned individual desorption methods and can be analysed either sequentially or with random access. Each tube is classified as sample, calibrant or blank and all sequences may be stored and recalled for future reference or repeat use.

Series 2 ULTRA systems incorporating TubeTAG read/write capability can be used to pre-screen tubes loaded into the system and automatically upload any relevant sample information (tube ID number, pumped volume/diffusive exposure time, date of sampling, *etc.*) into the automation sequence. This provides a convenient means of cross-checking sample and tube information before an analytical sequence is initiated.



Graphical sequence viewer

The sequence viewer presents a clear graphical display of the position, classification and operating status of each tube. It can also serve as a template for the operator when loading tubes.

Sequence reporting

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(MS) system to start a blank run to keep the analyser in step with the desorber.

Series 2 ULTRA systems incorporating optional TubeTAG read/write capability automatically read the information stored on the relevant tag as each tube is loaded ready for desorption. The sequence reporter is then automatically populated with the relevant information for that sample tube.

Optional automatic update of tube tags

Tag read/write-configured series 2 ULTRA autosamplers can also be used to automatically add or modify relevant information on each individual tube RFID tag post run. Options for this are fully user-selectable and include:

- Incrementing the number of thermal cycles
- Changing the tube status from sampled to analysed
- Inputting the ID of the tube used for re-collection of that sample
- Clearing the sample-related information from that tag
- Logging if that tube exhibited any back-pressure anomolies or failed the leak test

This capability represents a revolution in quality control and tracking of sorbent tube performance and history.



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



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



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



TubeTAG RFID tag system for sorbent tube informatics



Calibration accessory for TD tubes



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



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



Canisters and related accessories



MTS-32 sequential pumped sampling onto multiple tubes

Summary

	100- tube	Choice of tube sizes	Effective tube seal	Sample re-collection	Optional electronic control of split/desorb flows	Optional automatic reading/ writing of RFID tags	Internal Standard	Dry purge
ULTRA 2	1	1		Manual of both splits	via UNITY 2	1	×	×
Series 2 ULTRA <i>50:50</i>	1	1	*	As above plus automatic (outlet split only)	via UNITY 2 and/or integrated MFC options	V	×	~
Series 2 ULTRA with ISDP	1	1	~	Manual of both splits	via UNITY 2	1	1	1
Series 2 ULTRA <i>50:50</i> with IS	1	1	V	As above plus automatic (outlet split only)	via UNITY 2 and/or integrated MFC options	1	1	~
Series 2 AutoSecure TD	\checkmark	1	1	Automatic (all split flows)	via UNITY 2	\checkmark	×	×
AutoSecure TD with ISDP on desorb ULTRA	\checkmark	1	V	Automatic (all split flows)	via UNITY 2	1	V	~

Trademarks

SecureTD-Q[™], TubeTAG[™] and DiffLok[™] are all trademarks of Markes International Ltd.

...experts in thermal desorption

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