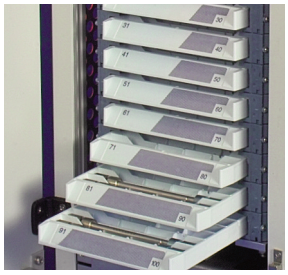
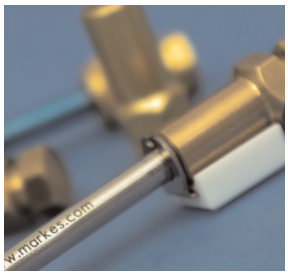
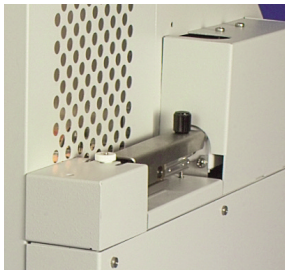






Series 2

ULTRA

Thermal Desorption Autosampler

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

MARKES international

Series 2
ULTRA

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 (VOCs and SVOCs) 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, SF_6, CS_2 and H_2S. 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 and paints 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 1 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 revolutionises tube and sample traceability.
- **Available for 3½″ long (standard) tubes or 4½″ long (DAAMS*) tubes,** with or without tags.
- **Stringent sample sealing**, before and after desorption. Patented DiffLok™ caps preserve sample and blank-tube integrity and ensure compliance with standard methods.
- **Rapid tube cooling** after desorption ensures shortest possible TD cycle times and fastest possible sample throughput, with and without automated re-collection.
- **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 minimises water interference and is recommended by standard methods.
- Innovative, compact design – **minimises bench space.**

* DAAMS: Depot Area Air Management System as used in some chemical agent test facilities

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 innovations such as:

- SecureTD-Q for repeat analysis and automated re-collection using a single TD autosampler[1]
- RFID tube tagging (TubeTAG)[2]
- Diffusion-locking for effective tube sealing and robust automation[3]
- Innovative low-volume valving specifically designed for TD[4]

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 3½″ or 4½″ 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.

1. Patent No. GB 2395785 (Automated re-collection using a single TD autosampler).
2. Patent No. US 6,446,515 B2. 3. Patent No. GB 2337513; US 6,564656 B1.
4. Patent No. GB 2336649.

Method-compliant automation

Series 2 ULTRA adds to any UNITY 1 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 (for TubeTAG sorbent tube tagging see separate brochure); either 3½″ long standard tubes or 4½″ long (DAAMS) tubes. Each tube is loaded into the analytical position in turn, and sealed into the carrier gas flow path. The following analytical steps then all take place on the ULTRA 2 autosampler:

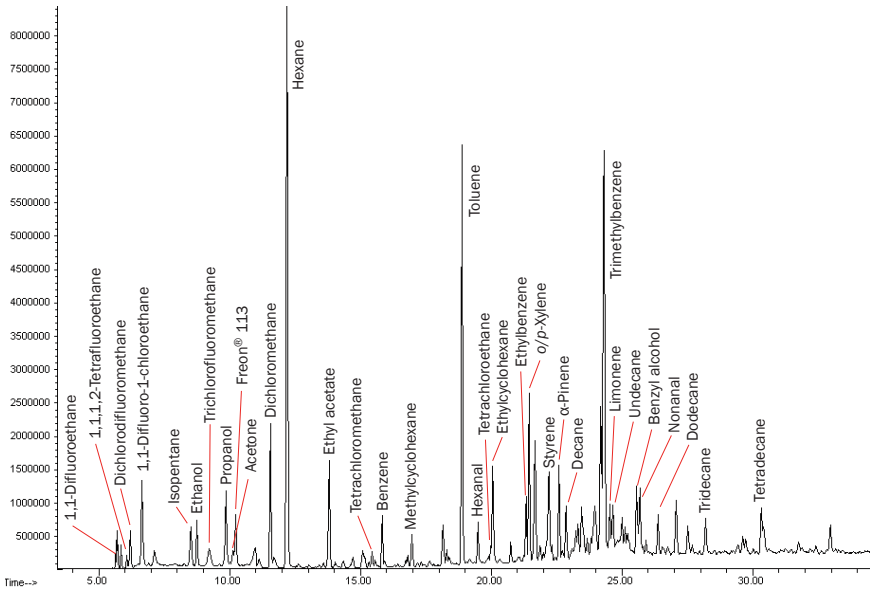
- Ambient-temperature/no-flow leak testing
- Optional dry-purging and/or internal standard addition
- Pre-purge of air from the tube
- Primary (tube) desorption.

As vapours desorb from the primary sample tube in ULTRA 2, they are swept through the short, inert, heated link line into the electrically-cooled focusing trap of UNITY 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 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 one hundred 3½″ or 4½″ 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 a multi-sorbent tube and analysed by TD–GC–MS

DiffLok™ caps seal tubes effectively and simplify TD automation

100-Tube automation optimises productivity

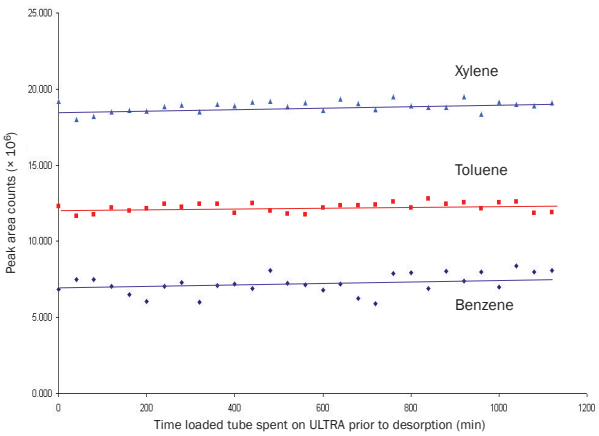
ULTRA 2 maximises the throughput and revenue generation potential of your TD–GC(–MS) system. It offers capacity for up to 100 sample tubes together with overlap mode. Sample overlap means that desorption of a subsequent tube can begin while GC(–MS) analysis of a previous sample continues – thus minimising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

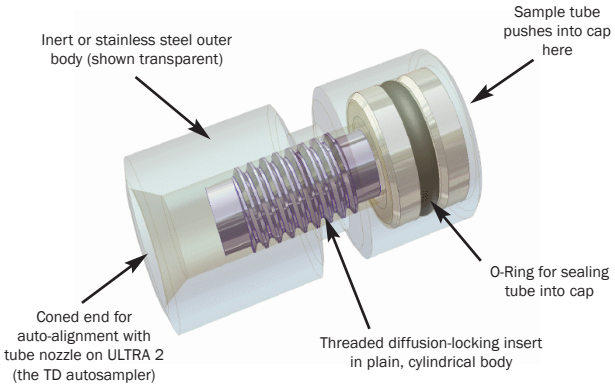
Tubes on Series 2 ULTRA autosamplers are sealed with Markes' unique patented DiffLok caps. DiffLok caps simply push on to both ends of every tube and preserve sample integrity by preventing both analyte loss and artefact ingress. Even volatile analytes are confidently preserved on sorbent tubes, giving 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[1].

1. P.P. Ballesta, Losses from ATD-400, *The Diffusive Monitor*, November 1997.



No analyte losses from Tenax tubes capped with DiffLok caps and analysed on ULTRA over a 24-hour period

Schematic of DiffLok cap



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.

Multiple tube sizes

Series 2 ULTRA autosamplers are available preconfigured for the following tube sizes:

1. 3½″ (89 mm) long × ¼″ (6.4 mm) o.d. tubes as specified in international standard methods for atmospheric monitoring (environmental or workplace air). *Note that these tubes are available from Markes in stainless steel, glass or inert-coated steel*
2. Two versions of 4½″ (115 mm) long DAAMS tubes:
   A. 6 mm o.d., glass (standard-flow) tubes
   B. 10 mm o.d., glass (high-flow) tubes with 6 mm ends

Note that ULTRA 2 systems configured for 4½″ tubes can accomodate both standard and high-flow tubes, i.e. options 2A and 2B.

In all cases, Series 2 ULTRA is compatible with electronically-tagged or untagged tubes.

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 increase the number of thermal cycles, input any high-back-pressure anomalies, 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.

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

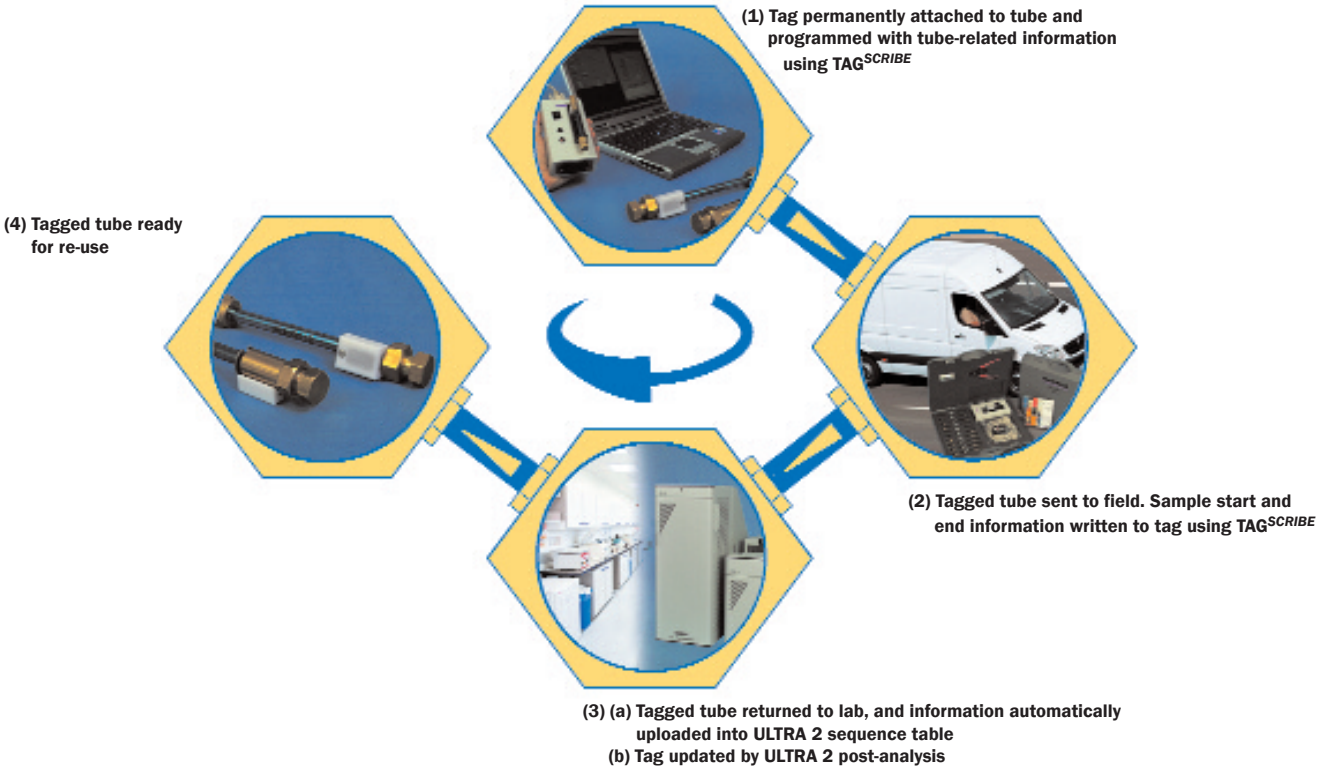
In combination with a Series 2 ULTRA-UNITY system, TubeTAG further enables:

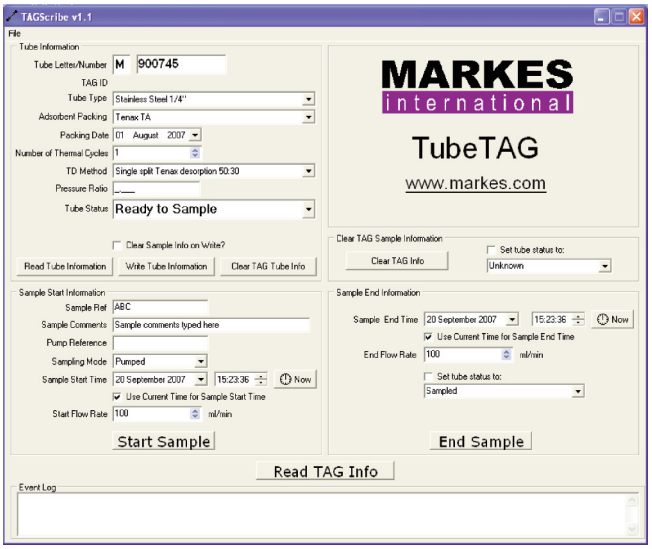
- Automatic logging of the history of a tube throughout its life: Number of thermal cycles, back-pressure anomalies, number of leak test failures, *etc.*
- Automatic input of sample-specific information to the ULTRA TD automation sequence table: Tube number, sampled volume, diffusive sampling time, date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

TD automation with electronic tube tracking

TubeTAG: Mode of operation





TubeTAG software user interface

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 integrated seamlessly with the TD–GC(–MS) sequence. Data processing remains synchronised with the analytical process at all times. Any tubes that 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.

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 stipulated in 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 (outlet) split flow for 50 or 100 tubes. Active tube cooling post-desorption allows automated re-collection without extending cycle times.

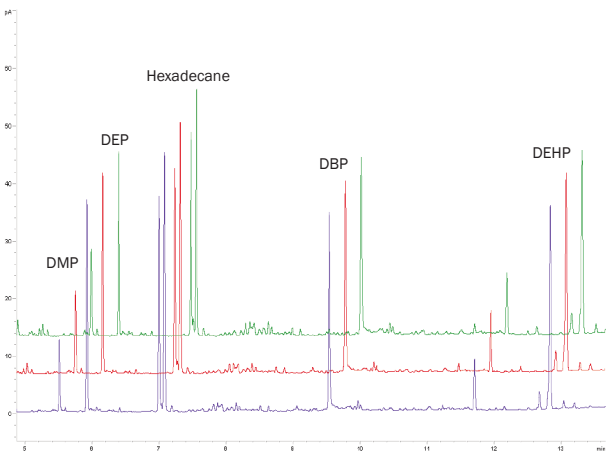
Series 2 AutoSecure TD™ systems, incorporating two ULTRA 2 autosamplers, offer quantitative re-collection of the total split flow (*i.e.* the split flow during both tube and trap desorption) for all 100 tubes.

Automated re-collection now enhanced with TubeTAG

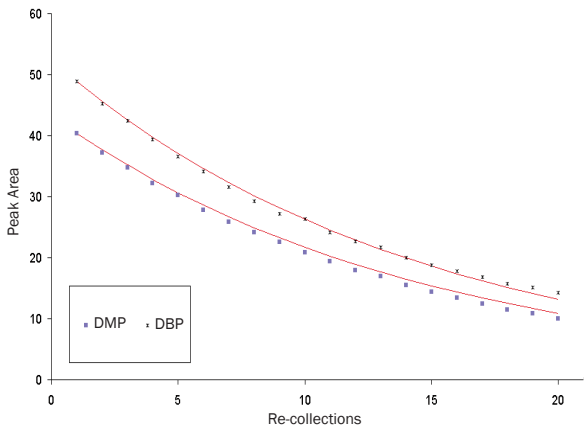
Use of Series 2 ULTRA TubeTAG read/write capability during automated desorption/re-collection greatly enhances the automation and traceability of SecureTD-Q. Automated desorption/re-collection systems incorporating TubeTAG log the unique ID numbers of original sample tubes and of the corresponding re-collection 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.

Note that Series 2 ULTRA 50:50 or AutoSecure systems are configured to accommodate re-collection tubes that are the same dimension as the sample tubes, i.e. an ULTRA 2 configured for desorption of 3½″ tubes will accommodate 3½″ re-collection tubes and an ULTRA 2 configured for desorption of 4½″ tubes will accommodate 4½″ re-collection tubes.



Sequence of chromatograms showing re-analysis of re-collected phthalate mixture using ULTRA 50:50



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 ULTRA 2:

50:50 Option

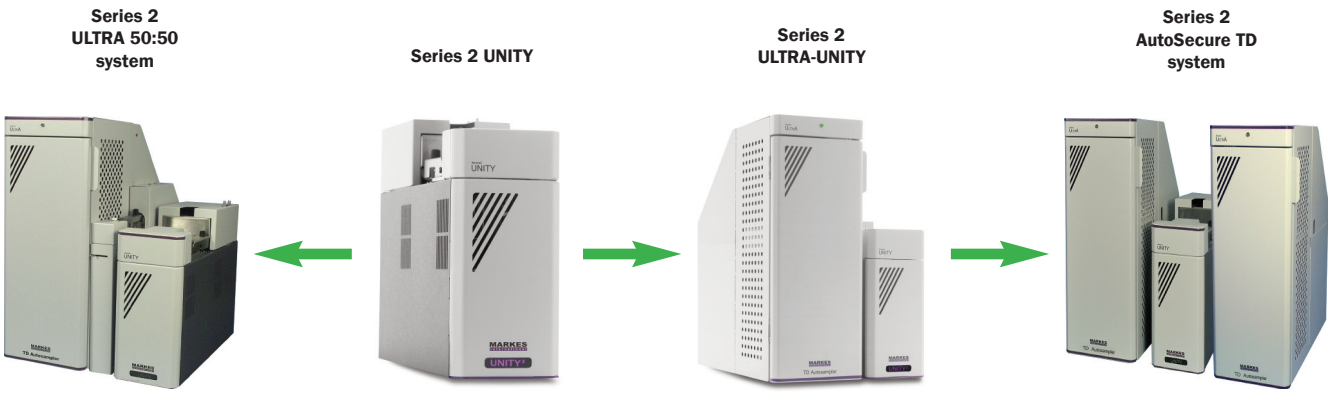
The ULTRA 2 can be factory-configured with a '50:50' option for automated sample re-collection of up to 99.8% of each sample using a single TD autosampler. The resulting Series 2 ULTRA 50:50 package is a patented, cost-effective, space-saving solution for automatic thermal desorption and **re-collection** 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 re-collected 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 Method 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).

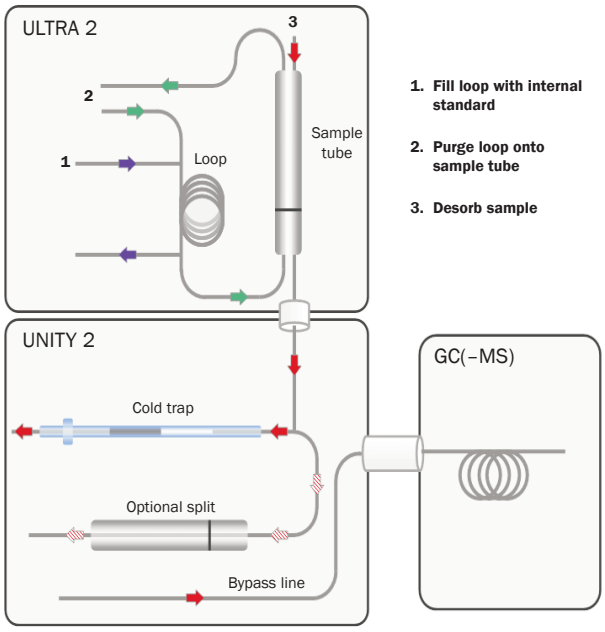


Parallel connection of automated canister analysis/on-line air monitoring is also faciliated (see Air Server brochure)

Manual or automated flow control

Series 2 ULTRA systems 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 full details). These provide versatile electronic control of both split and desorption flows during an automated sequence with multiple TD methods.

They also allow the selection of split ratios from zero to 125,000 : 1, accommodating a uniquely wide range of applications and analyte concentrations.



1. **Fill loop with internal standard**
2. **Purge loop onto sample tube**
3. **Desorb sample**

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-d_8 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 also offer a range of stand-alone off-line accessories for dry-purging sorbent tubes and introducing gas- or liquid-phase standards.

Automating re-collection for repeat analysis

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.

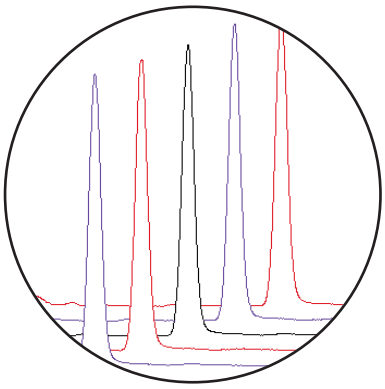
AutoSecure TD System: The ultimate in TD automation

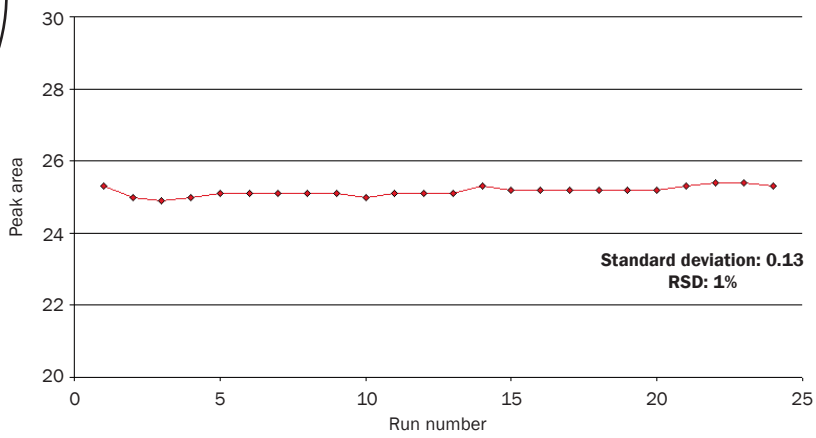
Series 2 ULTRA-UNITY systems, with or without ISDP, can be further upgraded by the **addition of a second ULTRA 2** for automated and quantitative re-collection of total split flow, *i.e.* the split flow during primary (tube) desorption (inlet split flow) as well as split flow during secondary (trap) 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″) 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 Series 2 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 for bromofluorobenzene: 24 repeats over a 24-hour 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 in parallel with Air Server and CIA technology for automated on-line air monitoring and canister analysis (see associated brochures). 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.

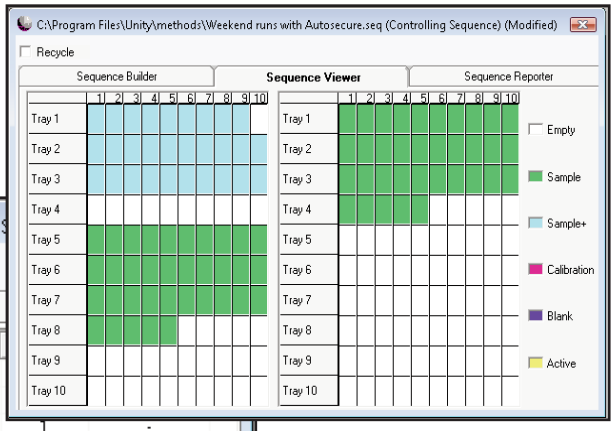
Intuitive control software

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.

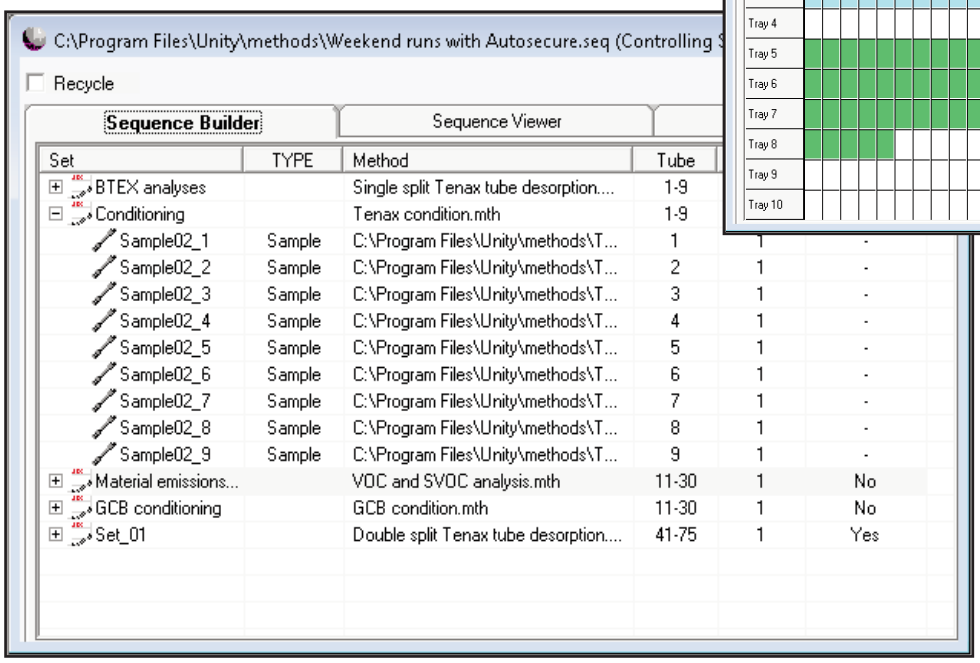
Sequence building

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.



Series 2 AutoSecure TD sequence viewer



Series 2 ULTRA-UNITY sequence builder

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 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 TubeTAGs

Series 2 ULTRA autosamplers can also be used to automatically add or modify relevant information on each individual TubeTAG post-run. Options for this are fully user-selectable and include:

- Increasing 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 anomalies 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

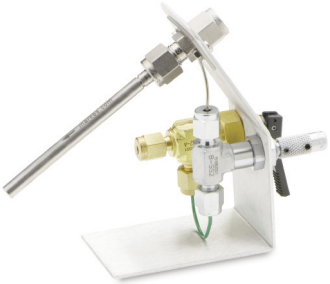
Series 2 (ULTRA)-UNITY is complemented by Markes' comprehensive portfolio of thermal desorption instrumentation and associated sampling equipment. Many of the innovative and labour-saving accessories available are unique to Markes, including specialist low-flow sample tubes, multi-sample test equipment for material emissions screening, calibration accessories, breath samplers and soil probes. Full details are given in Markes' Thermal Desorption & Accessories Catalogue.

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 and transferring them to sorbent tubes

Micro-Chamber/Thermal Extractor for measuring emissions from materials and consumer products

MTS-32 for 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	✓	✓	✓	✓ Manual of both splits	✓ *via* UNITY 2	✓	✗	✗
Series 2 ULTRA 50:50	✓	✓	✓	✓ As above plus automatic (outlet split only)	✓ *via* UNITY 2	✓	✗	✓
Series 2 ULTRA with ISDP	✓	✓	✓	✓ Manual of both splits	✓ *via* UNITY 2	✓	✓	✓
Series 2 ULTRA 50:50 with ISDP	✓	✓	✓	✓ As above plus automatic (outlet split only)	✓ *via* UNITY 2	✓	✓	✓
Series 2 AutoSecure TD	✓	✓	✓	✓ Automatic (all split flows)	✓ *via* UNITY 2	✓	✗	✗
AutoSecure TD with ISDP on desorb ULTRA	✓	✓	✓	✓ Automatic (all split flows)	✓ *via* UNITY 2	✓	✓	✓

Trademarks

Markes International Ltd

Gwaun Elai Medi-Science Campus
Llantrisant
RCT, CF72 8XL
UK

T: +44 (0)1443 230935
F: +44 (0)1443 231531
E: enquiries@markes.com
W: www.markes.com

Markes International, Inc.

11126-D Kenwood Road
Cincinnati
Ohio 45242
USA

T: 866-483-5684 (toll-free)
F: 513-745-0741
E: enquiries@markes.com
W: www.markes.com

Markes International GmbH

Schleussnerstrasse 42
D-63263 Neu-Isenburg
Frankfurt
Germany

T: +49 (0)6102 8825569
F: +49 (0)6102 8825583
E: enquiries@markes.com
W: www.markes.com