









Multi-function TD autosampler with internal standard addition



## 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 $_{\!\!2}$ S. 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 Supplied by Agilent Technologies 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.
- **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.
- 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.
- 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. Internal standard addition to blank or sampled tubes as a further aid to analytical quality assurance.
- **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.
- Internal Standard addition included to fully comply with EPA method TO17
- Innovative, compact design minimises 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 innovations such as:

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

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

### ${\bf 1.\ Patent\ No.\ GB\ 2395785\ (Automated\ re-collection\ using\ a\ single\ TD\ autosampler)}.$

#### **Series 2 ULTRA**

ULTRA 2 is an innovative, robust and method-compliant thermal desorption (TD) autosampler for up to a hundred  $3\frac{1}{2}$ " 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.



<sup>2.</sup> Patent No. US 6,446,515 B2. 3. Patent No. GB 2337513; US 6,564656 B1.

<sup>4.</sup> 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). 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:

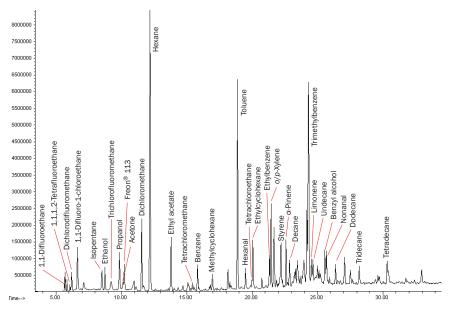
- Ambient-temperature/no-flow leak testing
- · Dry-purging and/or internal standard addition
- Pre-purge of air from the tube
- · Primary (tube) desorption.

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" tubes offering simultaneous analysis of volatiles & semi-volatiles, quantitative recovery of high boilers (including n-C<sub>40</sub>) 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



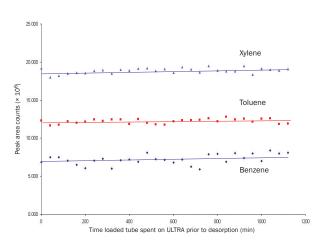
DiffLok™ caps seal tubes effectively and simplify TD automation

### **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.

Rapid tube cooling after desorption also ensures shortest possible thermal desorption cycle times, and fastest possible sample throughput,

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.



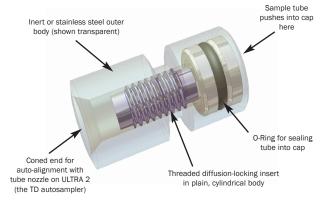
No analyte losses from Tenax tubes capped with DiffLok caps and analysed on ULTRA over a 24-hour period

### 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<sup>1</sup>.

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

#### Schematic of DiffLok cap





#### **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.

### **Industry-standard tube sizes**

Series 2 ULTRA autosamplers are available preconfigured for the industry-standard sized tubes:  $3\frac{1}{2}$ " (89 mm) long ×  $\frac{1}{4}$ " (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

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.

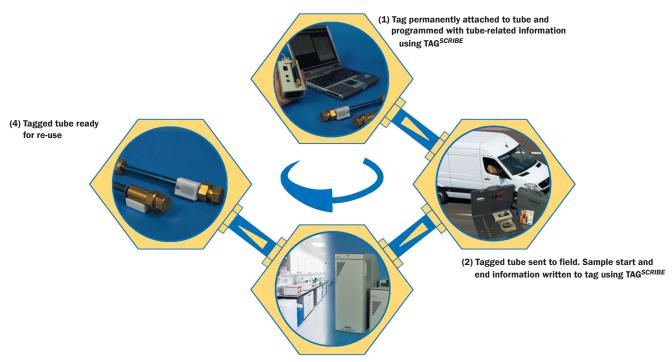
In combination with a Series 2 ULTRA-UNITY system, TubeTAG further enables:

- 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.

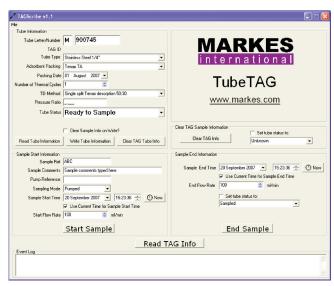
ULTRA 2 complete with the TubeTAG read/write option is a genuine breakthrough in automated TD technology. Imagine a future in which you can instantly identify the sorbent(s) in each tube, when it needs to be repacked and whether or not that tube has had a history of leak-test failures or back-pressure 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**



(3) (a) Tagged tube returned to lab, and information automatically uploaded into ULTRA 2 sequence table
(b) Tag updated by ULTRA 2 post-analysis



TubeTAG software user interface

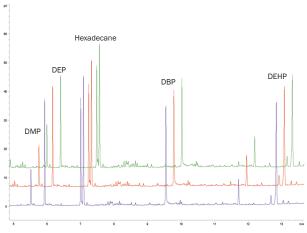
### **Method-compliance**

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.

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



Sequence of chromatograms showing re-analysis of re-collected phthalate mixture

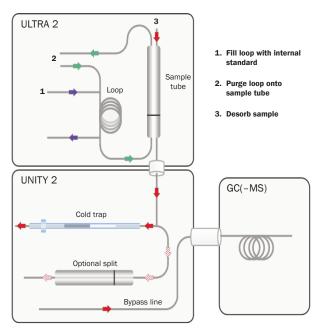
### Internal Standard/Dry-Purge (ISDP)

The ULTRA 2 with ISDP incorporates all the features of the ULTRA 2 autosampler and with the added benefit of an inbuilt internal standard (IS) addition and automatic tube dry-purging capability. When using the 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- $\mathbf{d_8}$  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.



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

### Flexible upgrade options

### 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).

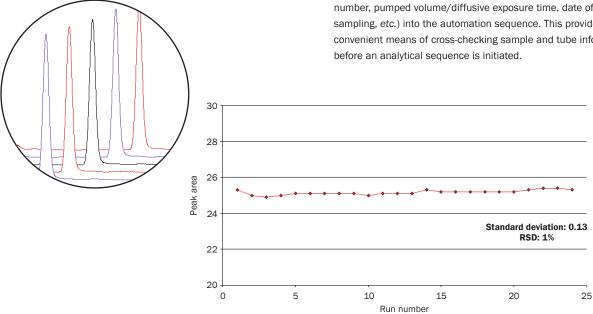
### **Intuitive control software**

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.



ISDP reproducibility data for bromofluorobenzene: 24 repeats over a 24-hour period



#### **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.

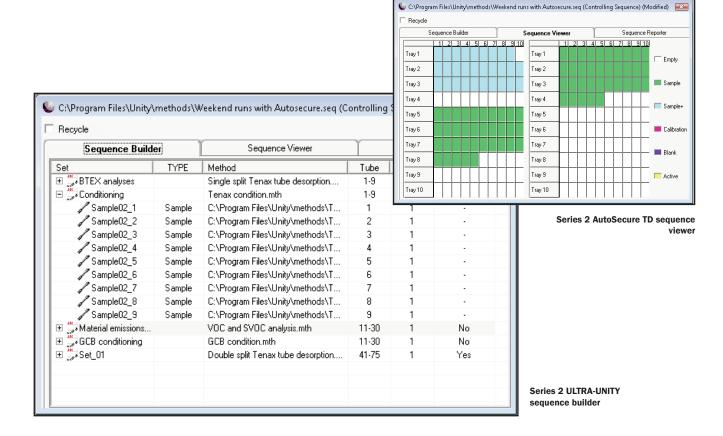
Series 2 ULTRA systems automatically read the information stored on the relevant tag as each tube is loaded ready for desorption. The sequence reporter is then automatically populated with the relevant information for that sample tube.

#### **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.



# Automating re-collection for repeat analysis

### 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



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

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