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Markes International UNITY 2™ thermal desorber

Specification Sheet February 2008



SYSTEM SUMMARY

UNITY 2 is a cryogen-free two-stage manual thermal desorber for tagged or untagged tubes. It is used for sampling, concentrating, extracting and analyzing volatile and semi-volatile organic compounds from a range of real-world sample matrices.

UNITY 2 uniquely provides a **universal TD platform** – compatible with every TD application. It incorporates a patented, inert heated valve ensuring recovery of the broadest range of analytes: C_2 to n- C_{40} hydrocarbons **and** reactive species such as mercaptans.

UNITY 2 complies with all the recommendations of international standard methods for analytical TD (e.g. US EPA Methods TO-17 & TO-15, ASTM D6196-03, ISO 16017 Parts 1 & 2, ISO 16000 series, etc.).

The system is compatible with single, industry-standard ¼-inch (6.4 mm) O.D. x 3½ inch (89 mm) long sample tubes with or without TubeTAG™ RFID tags. The sample tubes are available, with or without sorbent packing, in stainless-steel, inert-coated stainless steel or class.

UNITY 2 uniquely offers quantitative recollection of both tube and trap desorption split flow to allow repeat analysis (SecureTD-Q™) This overcomes the one-shot limitation of older TD systems and simplifies TD method/data validation per standard methods.

UNITY 2 acts as an additional, stand-alone injector that may be connected to any make of GC(MS) and does not interfere with other GC accessories. It is typically interfaced to the GC via a direct coupling to the analytical column. For example, with Agilent GCs (6890 or 7890A), the back pressure regulated electronic pneumatic control (EPC) module of a S/S injector can be used to control the carrier gas flow through the entire TD-GC(MS) system. This 'locks' retention times, independent of split flow, desorption temperature and other analytical settings.

Flexible upgrade routes: UNITY 2 upgrade options include:

- one or two integrated MFCs for control of split and/or desorb flows
- ULTRA 2 for 100 tube automation.
 Unattended operation all week end
- accessories for manual or automated headspace-trap analysis
- options for automated, multi-channel canister / bag analysis or round-the-clock on-line air monitoring.

A range of sorbent tubes and accessories is also available including:

 TubeTAG kit- RFID tags for sorbent tubes and hand held tag read/write devices - Record sample information and track tube history

Additional UNITY 2 features include:

- An electrically-cooled focusing trap offering uniquely effective cryogen-free retention of ultra-volatiles (e.g. acetylene from >1.5 L of air), quantitative recovery of semi-volatiles (n-C₄₀, PAHs, etc), fast cooling, compatibility with reactive species and easy maintenance (easychange, low dry gas flow, transparent trap box cover, etc)
- A stringent, method-compliant (no flow / ambient temperature) leak test is carried out on every sample. Failed tubes are retained intact.
- Trap heating rates up to 100°C/sec and backflush desorption combine to facilitate splitless operation at flows below 2 ml/min thus optimizing sensitivity
- Single and double splitting options and less than 0.1% carryover ensure compatibility with samples over a wide concentration range (ppt to %)
- Pre-purge of air to vent and selective elimination of water and solvents minimize analytical interference.

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- PC control in most 32-bit Windows™ operating environments provides an intuitive user interface
- Allows the desorption of a subsequent sample to begin while a previous sample is still running (overlap mode) - optimizes productivity.
- Small footprint for operation in mobile labs or other confined environments

SYSTEM SPECIFICATION

Analytical Range:

UNITY 2 provides a 'universal' TD platform allowing analysis of compounds over a wide boiling range AND the ability to select low flow path temperatures for compatibility with labile compounds.....

- Quantitative retention of acetylene from up to 1.5 L air without liquid cryogen
- Quantitative recovery of n-C₄₀
- Quantitative recovery of labile compounds mercaptans, CS gas, free-VX, amines, etc.
- Backflush desorption of the UNITY 2 focusing trap also allows simultaneous analysis of volatiles and semi-volatiles
- Recovery from sorbent tubes can be demonstrated on UNITY 2 using SecureTD-Q

Desorption Modes

- Tube Conditioning Mode desorption of the sample tube for cleaning purposes with all the effluent directed to vent i.e. away from the cold trap and other important components of the sample flow path
- 2(3) stage desorption mode normal two stage desorption of a sample with the additional option of an elevated temperature
- On-line monitoring, direct sampling, automation etc. are available when the system is configured with one of the appropriate accessories
- 'Direct sampling' mode for headspace-trap operation. Available when the system is configured with one of the appropriate accessories

Primary (Tube) Desorption Oven

Temperature range 35°C to 425°C settable in 1°C increments. [Note that the tube oven heats from ambient to the selected temperature at the start of tube desorption in order to minimize risk of flash vaporization & split discrimination when analyzing samples with unknown water/solvent content]

Desorption time 0 to 999.9 minutes settable in 0.1 min increments

Secondary Focusing (Cold) Trap

- Quartz cold trap: 2 mm I.D where packed and 0.9 mm I.D. at the sample input/output end. Collar at non-sampling end makes trap easy to change.
- Central 60 mm packed with between 1 and 4 sorbents. Backflush desorption ensures quantitative retention and release of wide boiling range samples
- Low temperature range -30°C to +50°C settable in 1°C increments. Uniform electrical cooling applied over full 60 mm length of sorbent bed. Will quantitatively retain acetylene from over 1500 ml of air (if packed with appropriate sorbents).
- High temperature range 35°C to 425°C settable in 1°C increments. Uniform heating applied over full 60 mm length of sorbent bed during trap desorption allowing quantitative release of semi-volatiles such has $n-C_{40}$, didecylphthalate and benzo-a-pyrene
- Heating rate: default setting is ballistic heating which reaches rates of 100°C/sec during the first critical stages of secondary (trap) desorption. Minimum trap desorption flow for high resolution capillary chromatography 2 ml/min without on-column focusing or 1 ml/min with on-column focusing
- Alternatively, programmed trap heating rates from 1°C/sec to 40°C/sec can be selected
- Time held at top temperature 0 to 999.9 minutes settable in 0.1 min increments
- Trap may be independently heated for conditioning purposes and for obtaining a system blank

Sample Flow Path

- Temperature range of sample flow path: valve 50°C to 210°C and transfer line 50°C to 225°C, both settable in 1°C increments. Uniform heating.
- Constructed entirely of inert materials: PTFE, quartz, inert-coated stainless steel and uncoated, deactivated fused silica.
- Allows quantitative recovery of both semivolatiles (including $n-C_{40}$) and reactive compounds.

Pneumatics

Requires pressure controlled 0-60 psig (0-415 kPa) supply of He, N₂ or H₂ carrier gas under manual or electronic control.

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Requires a pressure controlled supply of 40-80 ml/min dry (dew point -45°C or below) compressed air or nitrogen in the range 50 to 60 psig (340 to 415 kPa). The dry gas is used for both pneumatic actuation of the valve and for purging the cold trap box. Helium cannot be used as the dry gas supply

Pre-desorption checks and controls

- Leak test: each tube is pressurized and subjected to a stringent, ambient temperature leak test without carrier gas flow. Failed tubes are not desorbed, but are preserved intact for operator attention
- Pre-purge: Each tube is purged with carrier gas (in the desorption direction) at ambient temperature to remove oxygen before desorption. The air is purged to vent and non of it is allowed to reach the analyzer e.g. GCMS. Ambient purge time 0 to 99.9 minutes settable in 0.1 min increments
- An additional carrier gas pre-purge can be carried out at elevated temperature to remove water or other interfering solvent if required
- The cold trap may be selected to be in or out of line during either of the pre-purge stages
- The split may be selected to be open or closed during either of the pre-purge stages

Sample splitting and SecureTD-O (quantitative re-collection for repeat analysis)

The UNITY 2 splitter may be operated in the following modes:

- During primary (tube) desorption
- During secondary (trap) desorption
- During both desorption stages i.e. double splitter operation
- During neither desorption stage i.e. splitless

The splitter may be selected on or off during system standby and at any stage during pre- or dry purge.

The split and desorb flows are controlled by needle and solenoid valves downstream of the sample flow path. Alternatively the split flow may be controlled by electronic mass flow control (MFC) using the U-MFC100-2S, U-MFC200-2S or series 2 Air Server / CIA 8 accessories. With any of these accessories fitted, different split flows can be selected for each stage of operation (standby, pre-purge, tube desorb and trap desorb) and split & desorb flow settings may be stored and recalled as part of the desorption method.

The split vent line contains a charcoal filter in front of the control valves (and MFC) to prevent contamination of the valves/MFC and laboratory atmosphere. The charcoal filter has the same external dimensions as a standard sorbent tube. The flow path between the main UNITY 2 heated valve and the charcoal filter is a mirror-image of the short, inert heated flow path connecting the sample tube to the heated valve. When required, the charcoal filter may be replaced with a conditioned sorbent tube to quantitatively re-collect the split effluent from tube & trap desorption (inlet and outlet split). This is called SecureTD-Q. SecureTD-Q allows repeat analysis, method / data validation and archiving of critical samples.

Dimensions and Weight

Height: 40 cm (15.7 in)

Width: 16 cm (6.3 in)

Depth: 51 cm (20 in)

Weight: 16 Kg (24 lb)

Environmental Conditions

- Ambient operating temperature 15°C to 30°C
- Ambient operating humidity 5 to 95%, noncondensing

Power Requirements

- 90 to 253V, 47/63 Hz, 650W (UNITY 2 selfadjusts to local voltage input e.g. UK 220-250V, US 110-120V)
- Power supply unit rated to 650W.
- Input inrush current of <40 amps

UNITY 2 Safety and Regulatory Approvals

- EN 60950-1
- EN 61010-1
- CE marked and compliant with the Low Voltage Directive (73/23/EEC) EN60950

EMC Performance

- IEC 61326:2002
- EN 61326:1997 + A1:1998 + A2:2001 + A3:2003

Designed and manufactured under a quality system registered to ISO 9001.

UNITY 2 power supply unit (PSU) conforms to the following approvals

- UL60950-1 & CSA22.2 No. 60950-1 UL Recognised. C-UL for Canada
- IEC/EN60950-1 BSI Kitemark and CE mark.
- IEC/EN61010-1 and IEC/EN60601-1. CB Report and BSI Kitemark

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 UL60601-1 & UL61010-1-UL Recognized, C-UL for Canada

Data System - Minimum PC Specification

UNITY 2 software will run on most 32-bit versions of Windows™, however use of currently supported versions of Windows is strongly recommended. In general a PC with sufficient resources to run 32-bit Windows should have enough performance to control UNITY 2. The following defines the recommended minimum PC specification:

- 400 MHz processor
- 256 MB RAM
- Minimum of 10 MB of free disc space (for the UNITY SERIES 2 software installation)
- Minimum XGA (1024x768 pixel) screen resolution, 256 color

The PC should have a free spare serial COMs port for communication with UNITY 2. Alternatively, PCI or USB options are available

GC remote cable connections

UNITY 2 includes a GC interface cable which connects to the ready output and start input of your GC(MS) and data handling systems. The cable supports automatic start of the entire analytical system when the UNITY 2 cold trap desorbs and allows UNITY 2 to check the ready status of the analyzer and associated data handling. The UNITY 2 cold trap will not desorb unless and until it receives a ready signal from the GC(MS) system.

UNITY 2 accessories and upgrade options include

- ULTRA 2, 100 tube autosampler
- Manual or automated headspace-trap operation
- Integrated electronic mass flow control of split and / or desorb flow. MFCs available in 2-100 ml/min or 5-200 ml/min flow ranges.
- Series 2 Air Server[™] options (3- or 8-channel) for continuous on-line air/gas monitoring or method-compliant analysis of multiple canisters/bags
- Kits to interchange between 3.5- and 4.5-inch tube versions of UNITY 2(available from Markes)

For further information

For more information about our thermal desorption products and services please visit our web site at www.markes.com.

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ULTRASpec

Markes International ULTRA 2™ thermal desorption autosampler

Specification Sheet February 2008

To be read in conjunction with USpec, the UNITY 2™ specification sheet



SYSTEM SUMMARY

The UNITY 2 Thermal Desorber is designed to analyze single, 3.5-inch sample tubes with or without RFID tags. The ULTRA 2 autosampler connects to series 1 or 2 UNITY and allows up to 100 tubes to be desorbed and analyzed sequentially.

Tubes on ULTRA 2 are contained in 10 trays each holding up to 10 tubes. While on ULTRA 2, all tubes are capped and sealed at both ends with patented* DiffLok™ caps. These eliminate ingress of atmospheric vapors *via* diffusion and prevent loss of retained volatiles while samples are waiting for analysis.

DiffLok caps provide a much more effective seal than push-on PTFE caps used on some automated TD systems¹.

DiffLok caps do not need to be removed by the instrument prior to analysis as they allow a flow of gas through the tube when pressure is applied. This simplifies mechanical operation making ULTRA 2 very reliable. The sample flow path through the cap at the sample outlet end is heated and inert coated to prevent condensation and make it completely inert.

Primary desorption of each tube takes place on the ULTRA 2 autosampler under control of the Windows™ compatible sequencing software. Tubes are moved into position inside the static desorption oven and sealed into the carrier gas stream with a minimum of simple robotic operations.

Desorbed vapors are transferred in the gas stream, through a short, inert, heated transfer line to the focusing trap of series 1 or 2 UNITY for subsequent secondary desorption and analysis. UNITY controls the carrier gas through ULTRA 2 and takes care of any sample splitting during both primary (tube) and/or secondary (trap) desorption steps. For details of this part of the desorption process see the UNITY 2 specification sheet.

Standard series 2 ULTRA-UNITYTM systems offer manual SecureTD- Q^{TM} for re-collection and repeat analysis of critical samples or for use during method development/validation.

The SecureTD-Q process may be automated by either the addition of an ULTRA 50:50 module for re-collection onto the same autosampler or by the addition of a second ULTRA 2 autosampler and a connection kit to the split side of UNITY 2 to give a fully automated series 2 AutoSecure TD^{TM} system.

ULTRA 2 features:

- 100 tube capacity offers extended unattended operation over an entire week end for optimum productivity
- Option of integrated tubeTAG read/write capability for enhanced tracking of tube history
- ULTRA 2 simply adds automation to the peerless analytical performance of UNITY
- Tubes are sealed using patented DiffLok caps to prevent ingress of contamination and loss of volatiles both before and after analysis
- Minimal linear robotic movements required for operation, thus increasing reliability
- Versatility and throughput: software allows multiple sets of tubes, requiring different TD methods, to be linked together in a single automatic sequence.

SPECIFICATIONS:

Primary (Tube) Desorption Oven

- Temperature range 50°C to 425°C (Max: 395°C for units shipped before July 08)
- Settable in 1°C increments
- Desorption time 0 to 999.9 mins
- · Settable in 0.1 min increments



Pre-desorption checks and controls

The pre-desorption checks and controls are as for UNITY 2. However the leak test is automated such that if a tube fails, it is replaced in its position in the tray. The DiffLok caps maintain the integrity of the failed tube. A 'blank' GC run is then triggered (this keeps the GC(MS) sequence in synchronization with the desorber) and the system goes on to the next tube in the sequence.

The position of the tube that failed the leak test is stored in system memory and included in the run log file. At the end of a sequence, the system presents a log file for the sequence, including leak test failures. Log files may be stored indefinitely.

If three tubes fail the leak test in succession, a system error is reported and the instrument stops and waits for operator intervention.

Automatic sequencing of tubes

A sequence of tubes comprising several 'sets' may be entered into the sequence table via the user interface on the PC. Tubes may be included in more than one set in a sequence.

A set normally comprises a series of tubes which are to be analyzed by the same desorption method. Multiple (up to 100) desorptions may be carried out on each individual tube. An entire sequence can be recycled any number of times as required.

Individual tubes may be identified as calibrant, blank or sample.

The sequence may be viewed in the convenient 'Sequence Viewer' screen both before initializing the run and while a sequence is in progress. A 'comma separated values' log file is produced and appended-to as the sequence progresses.

Any sequence deviations are recorded to the log file. If any deviations occur in a sequence, for example leak test failure or missing tube, the GC run is initiated to keep the analytical system in synchronization with the desorber. Sequences may be stored and recalled for re-use.

Tube conditioning mode is available on series 2 ULTRA-UNITY configurations allowing automated, sequential tube conditioning without risk of trap contamination.

SYSTEM SPECIFICATION

Dimensions and Weight

- Height: 62 cm (24 in)
- Width: 24 cm (9.5 in)
- Depth: 53 cm (20.9 in)
- Weight: 23 Kg (51 lb) unloaded, 28 Kg (60 lb) fully loaded with 100 capped tubes

Environmental Conditions

- Ambient operating temperature 15°C to 30°C
- Ambient operating humidity 5 to 95% RH non-condensing

Number of Tubes

100-tubes can be housed in 10 trays

Power Requirements

 90 to 253V, 47/63 Hz (ULTRA 2 self-adjusts to local voltage input e.g. UK 220-250V, US 110-120V)

Heat Output During Operation

- Power supply unit rated to 650W.
- Input inrush current of <40 amps

ULTRA 2 Safety and Regulatory Approvals

- EN 60950-1
- EN 61010-1
- CE marked and compliant with the Low Voltage Directive (73/23/EEC) EN60950.

EMC Performance

- Emissions EN61326
 - Conducted Class A
 - Radiated Class A

ULTRA 2 is designed and manufactured under a quality system registered to ISO 9001.

ULTRA 2 power supply (PSU.) conforms to the following safety approvals:

- UL60950-1 & CSA22.2 No. 60950-1 UL Recognised. C-UL for Canada
- IEC/EN60950-1 BSI Kitemark and CE mark.
- IEC/EN61010-1 and IEC/EN60601-1. CB Report and BSI Kitemark
- UL60601-1 & UL61010-1-UL Recognised, C-UL for Canada



Data System - Minimum PC Specification

As for UNITY 2, except that two serial ports are required. Note that if insufficient serial ports are available on the control PC additional ports can most simply added using either a USB hub and USB to serial cables or a PCI card.

ULTRA 2 Software

If the ULTRA 2 is to be added to an existing series 1 or 2 UNITY installation, it will have been shipped with current Markes International TD Control Software. Use this software to replace any older versions of TD software.

Control software for series 2 ULTRA-UNITY TD systems includes configuration options, desorption methods, sequence generation, sequence logging and system status viewing functions.

Electrical connections

As well as the standard connections included with UNITY 2, ULTRA 2 is shipped with its own power lead and RSC-232 PC cable

ULTRA 2 Options:

- TubeTAG read/write module for ULTRA 2. User installable
- ULTRA 50:50*: The addition of a "50:50" module to ULTRA 2 allows the automated recollection of trap desorption (outlet) split flow for up to 50 or 100 tubes using a single ULTRA 2 autosampler. Series 2 ULTRA 50:50 systems also facilitate automated dry purging of tubes.
- Series 2 ULTRA 50:50 systems can be configured with one or two integrated mass flow controllers if required
- AutoSecure TD System: The addition of a second ULTRA 2 autosampler and connection kit will fully automate the SecureTD-Q function (quantitative re-collection of both inlet and outlet split for all 100 samples onto clean conditioned tubes.)
- Series 2 ULTRA or ULTRA 50:50 autosampler pre-configured with Internal Standard Addition/Dry Purge accessory: Allows the introduction of 1 ml of gas phase standard onto the inlet end of a blank or sampled tube. Also facilitates automated dry purging of tubes.

For further information

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- 1 Losses from the ATD 400, PP. Ballesta, The Diffusive Monitor, Issue #9, Nov 1997
- * Patented # GB 2395785.
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