

UNITY 2™

ULTRA 50:50 Operators Manual

Version 1.0

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1. Loading tubes onto ULTRA 50:50

1.1. DiffLok™ analytical end caps for sample tubes

The DiffLok analytical end caps used with ULTRA 50:50 are fitted with patented DiffLok technology which reduces diffusive ingress effectively to zero. This preserves the integrity of both samples and clean desorbed tubes by preventing ingress or loss of volatiles while the tubes are on ULTRA 50:50. However when a tube is lifted into the ULTRA 50:50 desorption oven and sealed into the carrier gas flow path just prior to desorption, the application of pressure to the caps allows carrier gas to flow.

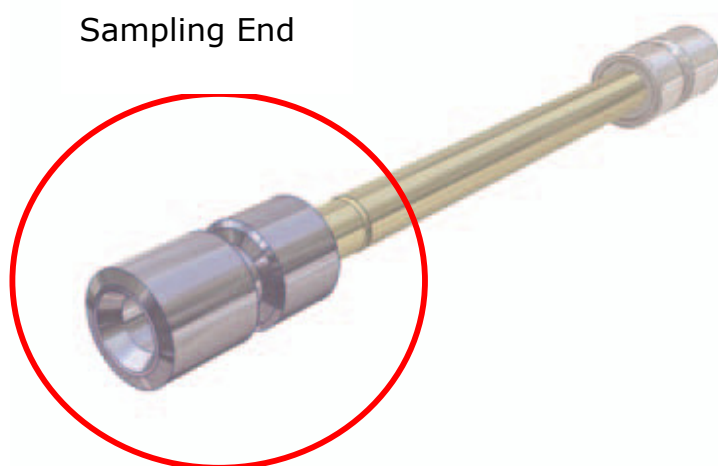
Each tube requires two DiffLok caps - a plain stainless steel cap which goes on the “cold end” or **non-sampling** end of the tube and an inert coated stainless steel cap (which is bluey/pink in colour) which goes on the “hot end” or **sampling end** of the tube.

Note: it is important to keep the hot end and cold end caps separate to ensure good quality analysis. Hot sample gas is desorbed from the tube and passes through the hot end cap into the thermal desorber - this is why the hot end cap is inert coated (as it is part of the sample path). Only carrier gas passes through the cold end cap.

1.2. Capping tubes with DiffLok caps

The caps should be pushed gently onto the correct end of the tube using a turning motion until they reach a natural stop. Do not force the cap further onto the tube once this stop has been reached. A tube correctly capped with a pair of DiffLok caps is 123 mm long, and tubes with correctly fitting caps will fit snugly inside the ULTRA 50:50 tray, but without any force being required to insert the tube or lift it from the tray.

The inert coated (bluey/pink coloured) cap should be placed on the sampling (desorption) end of the tube - this is normally the grooved end of stainless or coated steel tubes and, in the case of glass tubes, usually the end closest to the frit (if present) *i.e.* the end that is nearest the 'M' of 'Mi'



1.3. Loading tubes and trays into ULTRA 50:50

It is usually easier to withdraw the trays from ULTRA 50:50 and place them on the bench, before loading them with tubes. However, take care that the trays have been appropriately labeled before they are taken out of the instrument.

Each tube position in a tray is numbered (1 at the back to 10 at the front of the tray). There is a notch on the right hand side of each plastic partition between the tubes which will be on the right hand side when the tray is correctly orientated. Tubes should be loaded in the tray such that the sampling (grooved) end of the tube (capped with the blue/pink cap) is also on the right hand side.

With the tray orientated such that its label is towards you and tube position 1 is furthest from you, place the capped tubes in the desired positions in the tray with the sampling end on the right as described above. Loaded trays should then be pushed back into the appropriate tray locations in ULTRA 50:50.

Note: If the ULTRA 50:50 door is opened while a sequence is in progress, it is physically possible to access, remove or add any non-active tray to ULTRA 50:50. (The active tray will be recessed towards the back of ULTRA 50:50).

Caution: However, this must be done with great care. Any attempt to remove the active tray (i.e. the recessed tray) or to push a second tray into the same position in the stack, could result in serious mechanical failure.

It is normally advisable to load the complete sequence of tubes and trays into the system before closing the main door and initiating the complete sequence.

1.4. Microswitch on main chassis door

The main front chassis door on ULTRA 50:50 is configured with a microswitch which prevents up and down movement of the main tray stack while the door is opened.

Other movements, including further withdrawal of an already active tray, elevation of the active tube, etc., and all analytical desorption steps will continue as normal.

A sequence cannot be initiated with the ULTRA 50:50 door open. However, if the door is opened during a sequence it does not necessarily interrupt the series of analyses. The system will continue operating as described above until it becomes necessary to move the tray stack to access another tray. At this point the sequence will continue with desorption and analysis of the last tube in the current active tray, but will then pause and will be unable to resume until the door is shut. In general, if the ULTRA 50:50 door needs to be opened during a sequence, first examine system status via the Sequence Viewer to check that the system will not need to access a different tray within the period of time that the door needs to be open.

2. Software

The software main toolbar is identical to that of UNITY 2 except for a number of additional icons detailed below. Please refer to the UNITY 2 operators manual.

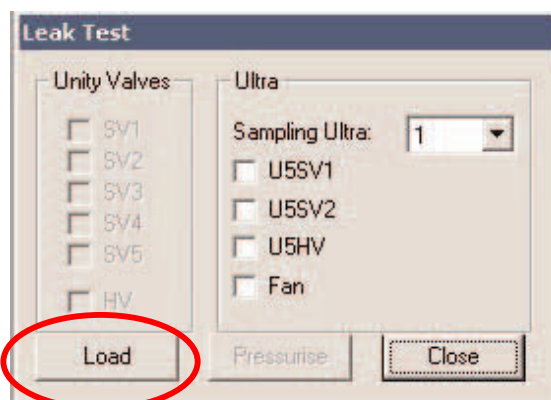


2.1. Leak Test Icon

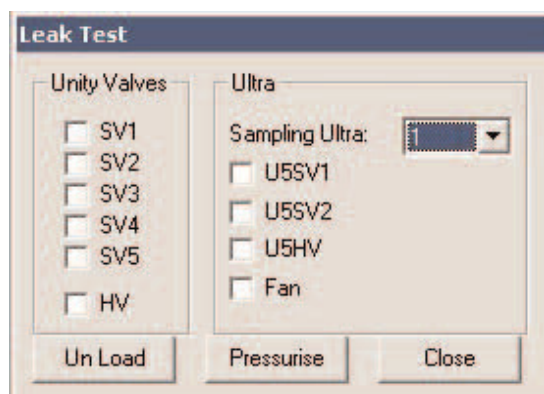


This function is only accessible from the active environment screen (see Section 2.4).

- Clicking the icon opens the dialogue box shown below.



- Select the tube number you want to use for the leak test and press '**Load**'.
- ULTRA 50:50 proceeds to load the tube selected (during this time the buttons in the dialogue box becomes greyed out).
- Once the tube has been loaded the dialogue box changes to that shown below



- Click on the '**Pressurise**' button to pressurise the flow path - this button now changes to '**Test Leak**'.
- After allowing some time for the flow path to pressurise, click on the '**Test Leak**' button, which shuts off carrier to the flow path.

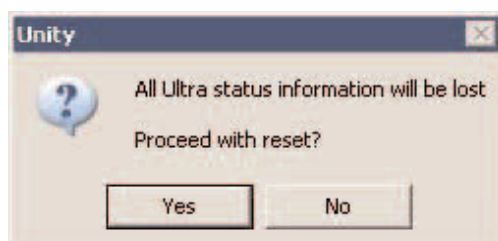
- Note the pressure reading on the status bar at the bottom of the main UNITY 2 screen. If the flow path is leak tight then the pressure reading will remain constant.
- The tube is returned to the tray on clicking on the '**Unload**' button.

2.2. The Reset ULTRA 50:50 icon



This icon is again only accessible from the active environment screen,

Once pressed it brings up the dialogue box shown below.



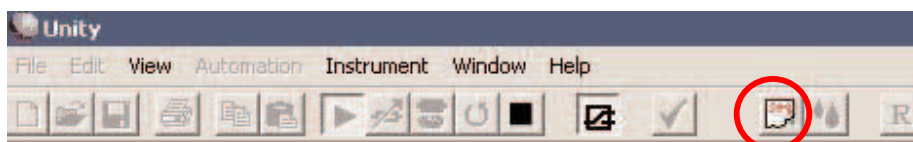
If the user decides to proceed, the ULTRA 50:50 carousel returns to the default position.

2.3. Operating environment

The software operates in two environments

Active - the active environment is available while the system is in Standby. It allows editing (and reviewing) of all method and sequence files.

Edit - the edit environment is available while the system is running by clicking on the 'Edit' icon as shown below or by selecting '**View**' and then '**Run/Edit View**'. It allows editing (and reviewing) of any non-controlling method or sequences. The edit environment is not available while the instrument is in standby mode.



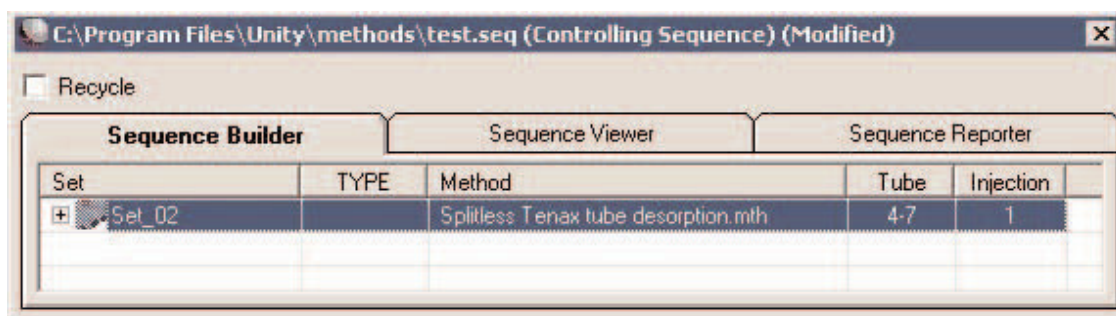
Once in the Edit Environment, select the normal '**Open File**' icon (or select '**File**' followed by '**Open**'). Then select the desorption method or sequence file you wish to edit.

Note: The system does not allow you to edit the current controlling method or sequence files. Methods and sequences may be modified, generated and stored using the edit environment without impacting the sequence that is currently in progress.

2.4. Active Sequence

This details the order or sequence of analyses that will be undertaken when the system is started. A sequence may be stored and recalled from memory but only one sequence can be designated as the active (controlling) sequence at any one time.

The Active Sequence section comprises three windows arranged in tabs



Sequence Builder	Builds an automated sequence
Sequence Viewer	Presents a visual representation of the sequence and its progress in real time
Sequence Reporter	Presents a complete history of a sequence of analyses including any deviations that occurred

2.5. Sequence Builder

The sequence builder is used to build / generate an automated sequence of analyses to be run by the ULTRA 50:50-UNITY 2. It is accessed by clicking on the relevant tab of the sequence window (in either active or edit environment).

A sequence is constructed of a number (one or more) of Sets. A Set is a series of tubes which initially are all assigned the same desorption method.

Sequences and Sets are created or amended using a drop down list which is displayed by right clicking anywhere on the Sequence Builder window. The options listed are as follows:

New Sequence	Opens a completely new Sequence window
Open Sequence	Opens the common dialogue box from which you can select the sequence file you wish to open
Add Sequence	Opens the common dialogue box from which you can select the sequence file you wish to append to the end of the current sequence
Add ULTRA Set	Adds another set to the sequence list
Delete item	Deletes the selected / highlighted line from the Sequence or Set

Sequences can be constructed either by opening and modifying an existing sequence (Open Sequence) or by creating a new sequence from scratch (New Sequence).

2.5.1. Adding an ULTRA set

A set is added to the opened sequence by selecting 'Add ULTRA Set' from the drop down menu list above. This opens a common dialogue box from which you should select the desorption method you wish to use for this set of samples. Once the desorption method has been selected then the Add Set dialogue box is opened.

The following parameters may now be entered:

Set name	Enter name used to distinguish this particular set of samples.
Method	The stored desorption method to be used to analyse this set of samples (this was selected from the common dialogue box (see above) earlier, however double clicking on the method name here, will allow you to change this method choice if required.
Start tube	The first tube to be analysed in this set.
Last tube	The last tube to be analysed in this set
No of Injections	The number of desoprtions required per tube
Re-collection	None - do not re-collect any samples in this set Same - re-collect the sample back onto the same (sample) tube Next tube - if this option is selected the system will re-collect the sample onto a particular tube number. The tube number is selected by using the drop down menu '1st' to indicate the tube number from which the re-collection should start. The re-collection will then proceed numerically. i.e. if tube 63 is selected, a five tube re-collection set will be made onto tubes 63, 64, 65, 66, 67.
Sample (base) Name	An identifier for this set of samples (for example if the base name is "Room 1_" and there are seven tubes in this set, they will be named "Room 1_1", "Room 1_2", "Room 1_3" etc.

Any number of sets of tubes can be added to a single sequence by repeating the 'Add ULTRA Set' operation. The same tubes can be included in multiple different sets for example if the user wishes to desorb a set of tubes at several different temperatures or re-run the tubes in conditioning mode after the series of analytical desorptions has completed.

2.5.2. Viewing & changing parameters for tubes within a set

A tube by tube listing of any set can be viewed by clicking on the '+' alongside the set name. This expands the set and shows a drop down list of all the tubes in the set. Clicking on the '-' re-compresses the set again.

Set	TYPE	Method	Tube	Injection	Re-collection
Set_02		Splitless Tenax tube desorption.mth	6-14	1	Yes
Set_04		Double split Tenax tube desorption.mth	1-5	1	Yes
Set_05		Single split Tenax tube desorption.mth	20-25	1	No
Set_07		Tenax tube.mth	1-20	1	Yes
Set_03		Tenax tube.mth	21-23	1	Yes
Set_09		Tenax tube.mth	1-20	1	Yes
Sample04_1	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	1	1	21
Sample04_2	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	2	1	22
Sample04_3	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	3	1	23
Sample04_4	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	4	1	24
Sample04_5	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	5	1	25
Sample04_6	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	6	1	26
Sample04_7	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	7	1	27
Sample04_8	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	8	1	28
Sample04_9	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	9	1	29
Sample04_10	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	10	1	30
Sample04_11	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	11	1	31
Sample04_12	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	12	1	32
Sample04_13	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	13	1	33
Sample04_14	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	14	1	34
Sample04_15	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	15	1	35
Sample04_16	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	16	1	36
Sample04_17	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	17	1	37
Sample04_18	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	18	1	38
Sample04_19	Sample	C:\Program Files\Unity\methods\Tenax tube.mth	19	1	39

Each individual tube has the following parameters associated with it:

Set	A unique name (e.g. the ID No.) can be entered for each tube
Type	Each tube may be designated as sample, blank or calibrant
Method	Specifies the desorption method applied to that tube
Tube	The position of the tube in the ULTRA 50:50 tray stack
Injection	The number of replicate desorptions required for that tube
Re-collection	The position of the re-collection tube in the ULTRA 50:50 tray stack. If no re-collection has been selected then 'No' is returned in this column.

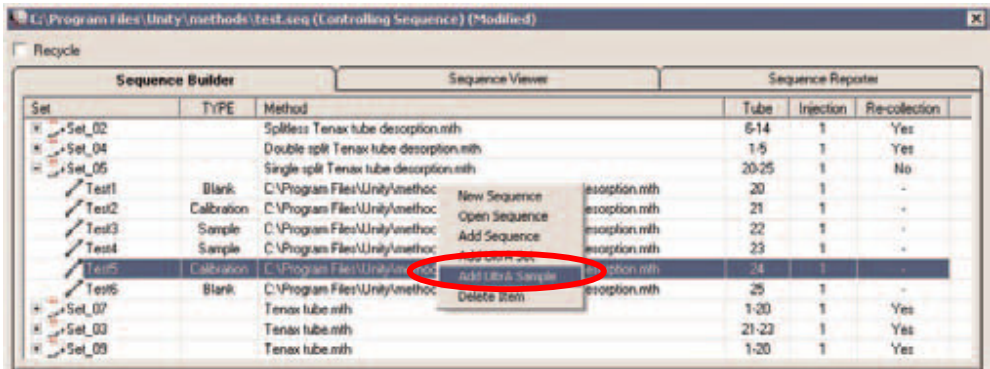
Each of the individual tube parameters (except the Set column) may be edited by highlighting the field of interest and modifying it as required.

The desorption method may be modified for one or all of the tubes in a set. If more than one desorption method is selected within a set, the method column header will display 'various'.

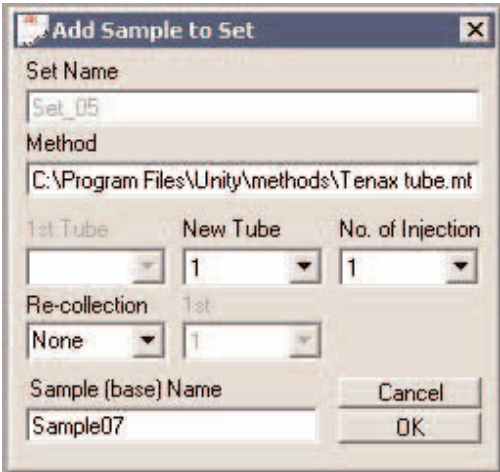
If the method parameter is highlighted, double clicking on it takes you to the stored method files. This flexibility means that although the Set function provides a quick and easy way of generating a long sequence of tubes, this does not restrict selection of separate desorption methods for individual tubes, or groups of tubes, within a set if required.

2.5.3. Adding new samples to an existing set

To add new samples to an existing Set, first expand the Set (by clicking on the '+' sign) to display all the samples already listed in that Set. Highlight a single sample and right click. A drop down list will be displayed, similar to that in Section 2.5 above but with the additional function 'Add ULTRA Sample'.



Select 'Add ULTRA Sample' and a common dialogue box appears asking you to 'Select Method to be used with Set or Sample'. Once the method is selected, press 'OK' and the 'Add Sample to Set' window appears.



This allows you to define other parameters for the new sample such as the tube number, number of injections and Sample Name. Select 'OK' to close the window after the appropriate parameters have been entered.

2.5.4. Deleting an item

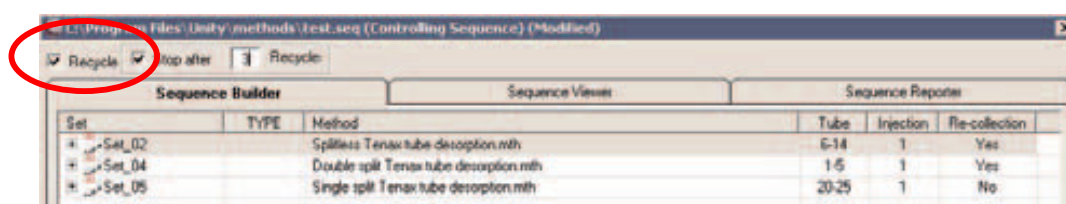
To delete either a whole set or a single sample from an expanded set listing, open the drop down item list. Select '**Delete item**' and the highlighted item will be deleted.

2.5.5. Storing and re-calling sequences

Having completed the sequence table using the Sequence Builder, use the '**Save**' or '**Save As**' functions. Stored sequences can be recalled using the '**Open File**' icon, by selecting '**File**' and then '**Open**' or by right clicking on the Sequence Builder window and selecting '**Open Sequence**' from the list.

2.5.6. Sequence Recycling

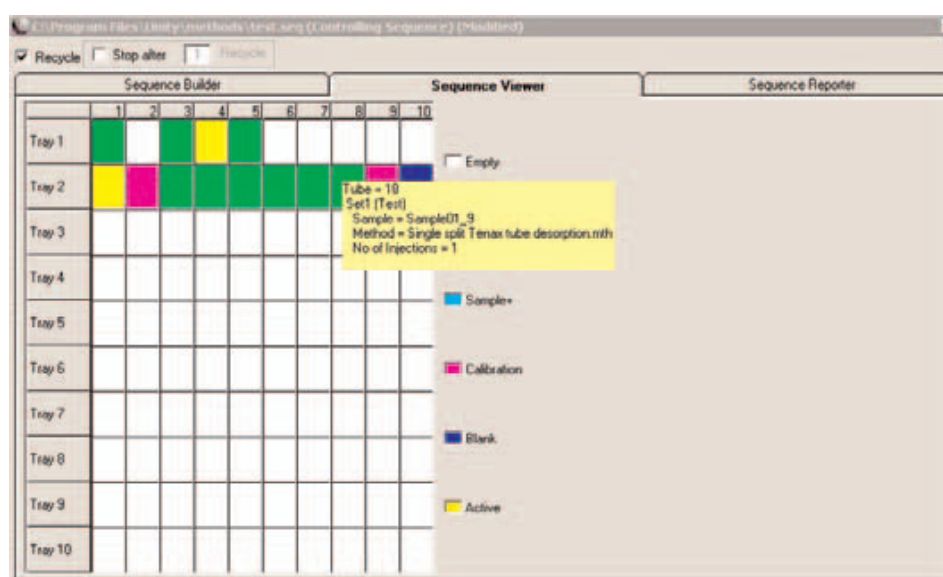
A sequence may be recycled one or more times by placing a check in the recycle box at the top of the Sequence Builder window



This opens two additional check boxes, '**Stop after**' and '**Recycle**'. If the '**Stop after**' box is ticked then this will activate the '**Recycle**' box - enter the number of cycles that the sequence should go through before stopping. If the '**Stop after**' check box is not ticked, the sequence will continue recycling indefinitely until the '**Stop**' icon is pressed or '**Instrument**' '**Stop**' is selected from the drop down menu bar.

2.5.7. Sequence Viewer

The sequence viewer gives a graphical overview of the sequence that has been built. It is accessed by clicking on the relevant tab of the sequence window. A grid is displayed which graphically indicates the position of all the tubes in the sequence.



Tubes are designated as follows:

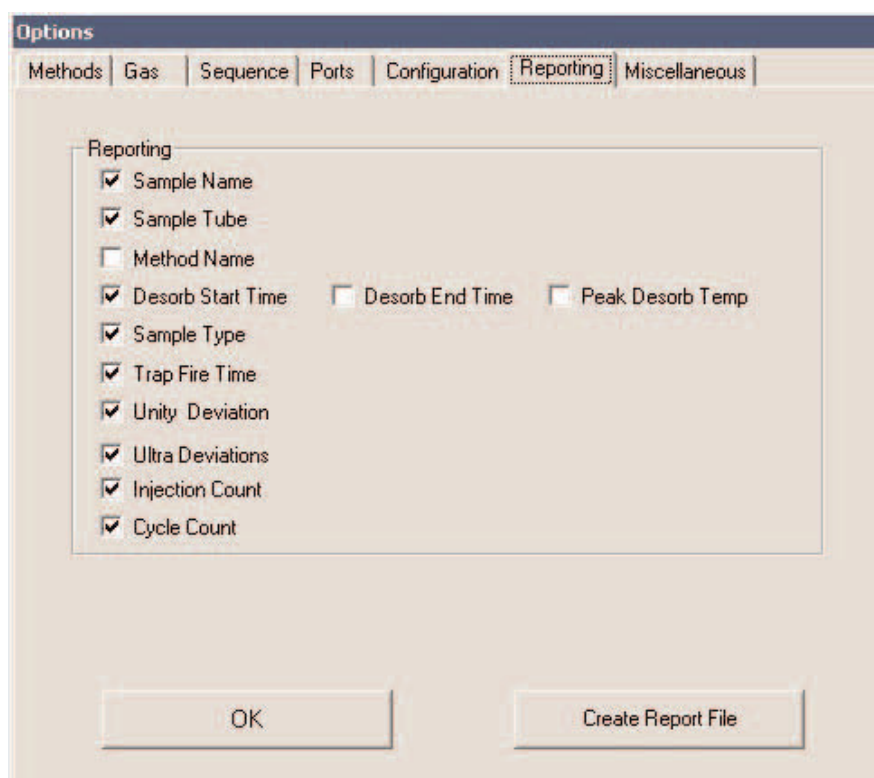
Sample	A sample to be analysed
Sample+	A sample tube which is called up by more than one set in the sequence
Blank	A tube designated as a blank
Calibration	A calibration tube
Active	The tube currently being analysed by ULTRA 50:50
Empty	No tube in this position

The tubes designations are distinguished by colour and the choice of colour (for all options except Empty) may be selected using assignment boxes. (Simply right click on the assignment box and select the colour from the options displayed). Information on any tube can be viewed by clicking on the respective box in the grid. As a sequence progresses, the Sequence Viewer is continually updated to display current system status.

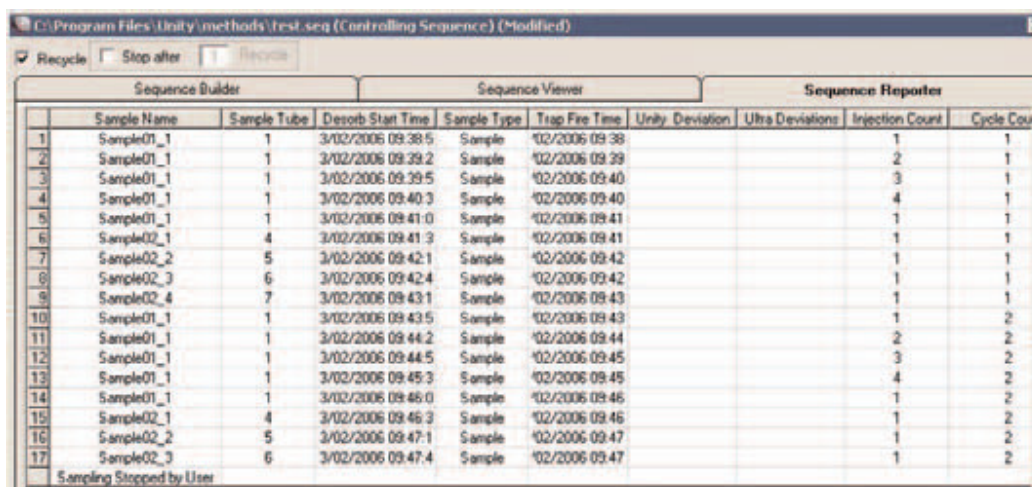
2.5.8. Sequence Reporter (Run Log)

The Sequence Reporter presents a complete history of a sequence of analyses including any deviations that occurred up to that point. Examples of deviations that may occur include leak test failure, missing tube or ULTRA 50:50 door open. Any tubes present on the system, but unassigned in the sequence, will be ignored by the system and will not be reported. To access the Sequence Reporter, select the appropriate tab in the Active Sequence section.

The number and assignment of columns in the Sequence Reporter table can be modified to display additional information such as the time and date of each specific tube and trap desorption. To re-assign existing table columns or create and assign parameters to new columns, select the '**Options**' function under '**View**' on the main header bar of the desorber control software, click to select '**Reporting**' from the list and then check the desired parameters from the list presented.



At the end of every sequence a Sequence Report (run log) is created.



The screenshot shows a window titled "C:\Program Files\Unity\methods\test.seq (Controlling Sequence) (Modified)". It contains a "Sequence Builder" section with checkboxes for "Recycle", "Stop after", and "Recycle". Below this is a "Sequence Viewer" and a "Sequence Reporter" table. The table has columns: Sample Name, Sample Tube, Desorb Start Time, Sample Type, Trap Fire Time, Unity Deviation, Ultra Deviations, Injection Count, and Cycle Count. The data shows 17 samples, with some having multiple injections and cycles. The status at the bottom is "Sampling Stopped by User".

	Sample Name	Sample Tube	Desorb Start Time	Sample Type	Trap Fire Time	Unity Deviation	Ultra Deviations	Injection Count	Cycle Count
1	Sample01_1	1	3/02/2006 09:38:5	Sample	02/2006 09:38			1	1
2	Sample01_1	1	3/02/2006 09:39:2	Sample	02/2006 09:39			2	1
3	Sample01_1	1	3/02/2006 09:39:5	Sample	02/2006 09:40			3	1
4	Sample01_1	1	3/02/2006 09:40:3	Sample	02/2006 09:40			4	1
5	Sample01_1	1	3/02/2006 09:41:0	Sample	02/2006 09:41			1	1
6	Sample02_1	4	3/02/2006 09:41:3	Sample	02/2006 09:41			1	1
7	Sample02_2	5	3/02/2006 09:42:1	Sample	02/2006 09:42			1	1
8	Sample02_3	6	3/02/2006 09:42:4	Sample	02/2006 09:42			1	1
9	Sample02_4	7	3/02/2006 09:43:1	Sample	02/2006 09:43			1	1
10	Sample01_1	1	3/02/2006 09:43:5	Sample	02/2006 09:43			1	2
11	Sample01_1	1	3/02/2006 09:44:2	Sample	02/2006 09:44			2	2
12	Sample01_1	1	3/02/2006 09:44:5	Sample	02/2006 09:45			3	2
13	Sample01_1	1	3/02/2006 09:45:3	Sample	02/2006 09:45			4	2
14	Sample01_1	1	3/02/2006 09:46:0	Sample	02/2006 09:46			1	2
15	Sample02_1	4	3/02/2006 09:46:3	Sample	02/2006 09:46			1	2
16	Sample02_2	5	3/02/2006 09:47:1	Sample	02/2006 09:47			1	2
17	Sample02_3	6	3/02/2006 09:47:4	Sample	02/2006 09:47			1	2

Sampling Stopped by User

This report file can be exported and viewed using standard Excel™ spreadsheet software.

To export the Sequence Report go to: **C:\Program Files\Unity\Reports** where you will find a **'csv'** file stored under the name of the sequence and the date - for example: test_13Feb2006_093833.csv. Open the file into Excel as normal.

3. System Operation

3.1. Loading a sequence

Open the stored sequence file by selecting **'File'** and **'Open'** from the drop down menu or by clicking the usual open file windows icon. Designate the file as the controlling sequence by selecting **'File'** and **'Load'** from the drop down menu or by clicking on the **'Load'** icon in the windows tool bar. Only one sequence can be designated as the controlling sequence at any one time.

3.2. Initiating a sequence

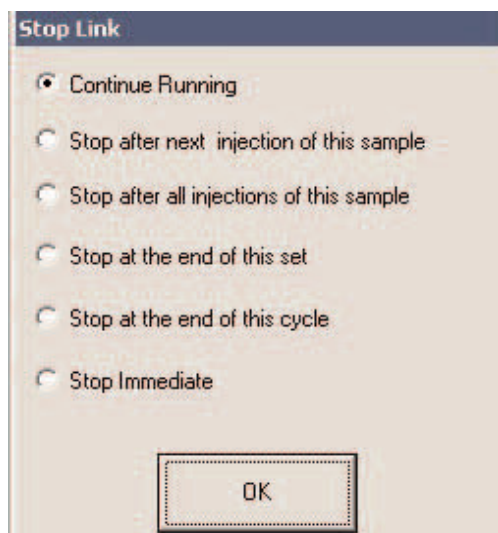
To start a sequence, press the **'Run'** icon on the main toolbar. The UNITY 2 method associated with the first tube of the sequence is recalled from memory.

Note: If a method has been modified but not stored, then when the run is started the original non-modified method will be re-called from memory and used. Ensure you save any changes to your operating method.

ULTRA 50:50 then selects and loads the first tube designated in the sequence and begins the series of runs. Steps in the desorption and analysis sequence of each tube are described in Section 3.5 below.

3.3. Stopping a sequence

A Sequence may be stopped at any time using the **'Stop'** icon on the main toolbar. Clicking the icon brings up the **'Stop Link'** dialogue box.



This instructs UNITY 2 and ULTRA 50:50 to stop the Sequence and return to Standby. A number of options are available according to whether you wish to stop immediately or at the end of a designated cycle, injection, tube etc.

3.4. Extending a sequence while it is in progress

To add additional samples to a sequence that is already in progress. First stop the sequence (Section 3.3), then generate a new or modified sequence (Section 2.5). This sequence should contain those samples from the old sequence that were not yet analysed plus the new set(s) of tubes you wish to add. The sequence must be saved (Section 2.5.5) and loaded (Section 3.1) to make it the controlling sequence. The new tubes should be added to empty or unfilled trays in the stack, (this is best completed while the sequence is stopped). Restart the system with the new/modified sequence (Section 3.2).

Note: Remember that if the thermal desorption sequence is altered and restarted as described, the GC(-MS)/data processing sequence will almost certainly need changing to reflect this.

Caution: It is not advisable to add tubes or trays to ULTRA 50:50 mid-sequence. However, if this is necessary, under no circumstance must the active tray be moved even if it has not yet begun to be withdrawn into the machine.

Caution: Take care if opening the ULTRA 50:50 door mid-sequence (Section 1.4)

3.5. Sequence of Operation

Once a sequence has been loaded and initiated, the system moves to load the first tube in the selected sequence. After the tube has been elevated into the ULTRA 50:50 oven and sealed into the carrier gas flow path, it is subjected to a series of operating steps depending on the mode selected in the active method.

Two modes are available:

- 2(3) stage desorption mode
- Tube conditioning mode

The operating steps are analogous to those described in the UNITY 2 operators manual. The main differences from the standalone UNITY 2 operations during 2(3) stage desorption are as follows:

3.5.1. Load tube

A tube cannot be loaded into the ULTRA 50:50 oven until the oven is at or below the Load Temperature.

The Load Temperature set under '**Options**' and is recommended to be set at 60°C. Click on '**View**' and '**Options**' and open the '**Configurations**' tab. Once the oven has reached the load temperature the tube lift mechanism raises the tube into the oven cradle and the tube is sealed into the carrier flow path.

3.5.2. Pressurise

The main flow path components are all pressurised to whichever carrier gas head pressure has been selected for the column and chromatographic system in use.

3.5.3. Leak test

The carrier gas supply is shut off after pressurisation to leak test the system. The leak test includes all system seals around the sample tube and cold trap, those into and out of the main heated valve, all the solenoid valves and the connection of the capillary transfer line into UNITY 2. Seals connecting the charcoal trap or re-collection tube in the split flow path are also included.

Any leak will cause the pressure transducer reading to fall. A pressure fall equivalent to or greater than 5% of the original carrier gas pressure over the 30 second leak test will result in a leak test failure. If a tube fails the leak test, the test is repeated, if the tube fails a second time it is removed from the oven and ULTRA 50:50 moves on to the next tube in the sequence. The leak test failure is noted in the sequence reporter. Three consecutive leak test failures as part of a sequence will cause the sequence to stop.

3.5.4. Dry Purge

The desorption method contains an additional parameter to those in the UNITY 2 desorption method: **Dry Purge Time** - If dry purge is required the time should be entered here. The default is 1.0 minute but the time can be set between 0.0 and 99.9 minutes in 0.1 minute increments.

Dry purging is the process of passing dry, clean gas through a sample tube ***in the sampling direction*** to purge the tube of high concentrations of water. It is often required where samples are taken in humid conditions onto sorbent tubes which contain hydrophilic sorbents such as Carbonised Molecular Sieves. During dry purge carrier gas is passed through the UNITY 2 heated valve into the ULTRA 50:50, through the ULTRA 50:50 heated valve and onto the front (grooved) end of the sample tube.

3.5.5. Unload ULTRA 50:50 sample tube and Load ULTRA 50:50 re-collection tube

After completion of the primary desorption the tube oven starts to cool. Once it has cooled to the UnLoad Temperature the desorbed tube is unsealed. If the sample is to be re-collected onto the **same** sorbent tube, the tube is then re-sealed and undergoes a Leak Test as described in section 3.5.6.

If however the sample is to be re-collected onto a **different** sorbent tube, the tube is unloaded and returned to the sample tray. The ULTRA 50:50 then moves the tray containing the re-collection tube to the active position and, assuming the desorption oven is not above its Load temperature will load the re-collection tube into position.

The UnLoad and Load Temperatures are set under '**Options**' and are recommended to be set at 110°C and 60°C respectively. Click on '**View**' and '**Options**' and open the '**Configurations**' tab.

3.5.6. Leak test re-collection tube

The re-collection tube (whether the same or different to the sample tube) is then leak tested as described in section 3.5.3). If the tube passes the leak test then the sequence of operation continues. If the tube fails the leak test twice, then the sequence of operation is as follows:

Re-collection on a different sorbent tube

If the re-collection tube fails the leak test twice then the ULTRA 50:50 will unload the tube to the tray. The original sample tube will then be re-loaded and leak tested. If this sample tube passes the leak test then the sequence of operation will continue and the split portion of the sample will be re-collected back onto the original sample tube.

If however the original sample tube fails the leak test twice then the sequence of operation will continue **however** the split portion of the sample will be passed through the ULTRA 50:50 split tube and vent and not re-collected. The system will then report an error and stop awaiting operator intervention.

Re-collection on the same sorbent tube

If the original sorbent tube now fails the leak test twice prior to re-collection then the sequence of operation will continue **however** the split portion of the sample will be passed through the ULTRA 50:50 split tube and vent and not re-collected. The system will then report an error and stop awaiting operator intervention.

3.5.7. Unloading the tube

After completion of the primary desorption the tube oven starts to cool. Once it has cooled to the UnLoad Temperature the desorbed tube is unsealed and unloaded back into the tray. The UnLoad Temperature is set under '**Options**' and is recommended to be set at 110°C. Click on '**View**' and '**Options**' and open the '**Configurations**' tab

3.5.8. Loading the next tube

The next tube in the sequence will be lifted and sealed into position as soon as the previous tube is replaced in the tray provided that the ULTRA 50:50 desorption oven is at or below its Load temperature (Section 3.5.5). This ensures that the tube does not get warm prematurely causing loss of volatiles during leak test and pre-purge.

Under default conditions, with a GC cycle time of 0, the system will proceed with leak test and purge of the next tube, but will not begin primary (tube) desorption until the GC reaches 'Ready' status. However, if a GC cycle time has been entered (to optimise sample throughput), UNITY 2 will calculate when to start the primary (tube) desorption so that the GC will come 'Ready' just prior to the end of the desorption time. See the UNITY 2 Operator's Manual for further information. This

means that after the first tube, cycle times for subsequent samples are determined just by the GC(-MS) analysis cycle time.

3.5.9. ULTRA 50:50-UNITY 2 systems with manual SecureTD-Q facility

The manual SecureTD-Q facility is available on all ULTRA 50:50-UNITY 2 systems in the same way as it is on stand-alone UNITY 2 systems. Further information is given in the UNITY 2 Operators Manual and on the Markes International website (www.markes.com).

3.5.10. Automated SecureTD-Q operation

The 50:50 module allows quantitative and automated re-collection of the outlet split flow from up to 50 sorbent tubes into 50 re-collection tubes.

Complete automation of the SecureTD-Q process for both inlet & outlet split is easily achieved by the addition of a second ULTRA autosampler to an ULTRA-UNITY 2 system. The second ULTRA automates the re-collection process. A complete Ultra-UNITY 2-Ultra system for automated desorption and re-collection is called AutoSecureTD™. Note that adding a second ULTRA to an ULTRA 50:50 setup would make the 50:50 module redundant.

3.6. Leak test failures

If a tube fails the leak test, the test will be repeated a second time. If the tube fails again, it is automatically unsealed and replaced intact in its original tray position. A record of the failure is stored in the Sequence Reporter (Section 2.5.8). The desorber automatically triggers '**GC system start**' to keep the GC(-MS) sequence in step with the desorber. ULTRA 50:50 then locates and seals the next tube in the sequence into the desorber flow path. This tube is leak tested and if fails twice, the same functions are carried out by the instrument as described above.

If three tubes fail twice, in succession, the instrument stops in standby mode, awaiting user intervention. The GC start is not triggered for a third time as the sequence has now been paused. See the UNITY 2 Technical Support Manual for the most likely causes of leak test failures.

3.7. Tube not found

If ULTRA 50:50 moves to a tube location included in the sequence, but finds no tube present, a record of the '**tube not found**' error is stored in the Sequence Reporter (Section 2.5.8). The desorber automatically triggers the GC system start to keep the GC-(MS) sequence in step with the desorber. ULTRA 50:50 then looks for the next tube in the sequence and if it cannot find that tube either, the same functions are carried out by the instrument as described above. ULTRA 50:50 will continue to look for tubes in the order determined by the sequence and will continue to log an error message and trigger the GC start for all tubes that aren't found until a tube from the sequence is successfully found.

3.8. Tray not found

If ULTRA 50:50 moves to a tray location specified in the sequence, but finds no tray present, a record of the '**tray not found**' error is stored in the Sequence Reporter (Section 2.5.8). In this case, the desorber does not trigger GC start, but awaits operator intervention in standby mode while displaying the error '**Tray not found**' on the main system status bar.

4. Setting and measuring gas flows

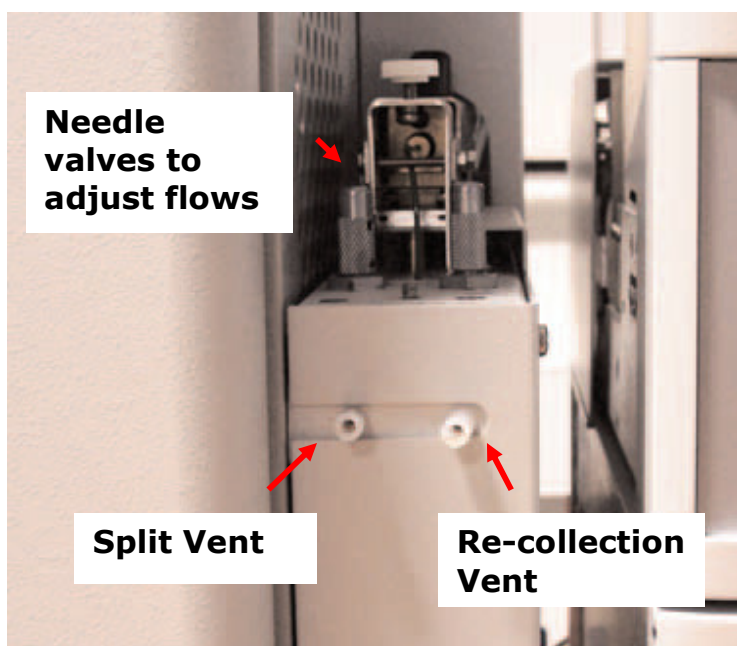
4.1. Setting the flow through the cold trap during primary desorption

The flow through the cold trap during primary desorption (desorb flow) is set and measured in the same way as described in the UNITY 2 Operators Manual.

Select '**Instruments**' '**Set Gas Flows**' and select '**YES**'. The desorb flow (flow through the trap) can now be adjusted via the left hand needle valve on UNITY 2 and measured through the corresponding outlet above the needle valve, using a suitable flow measuring device.

4.2. No sample re-collection - setting and measuring the "split vent" flow

When ULTRA 50:50 is operated without sample re-collection, any split flow which is selected (inlet or outlet split) is directed through the charcoal split filter tube and out of the left hand split vent at the front of the 50:50 module



To set and measure the split flow, ensure the controlling method has "**Split on in Standby**" selected. Unscrew the white thumbscrew and remove the small front cover to expose the needle valves. Adjust the left hand valve to change the flow. The subsequent split flow is measured out of the left hand vent.

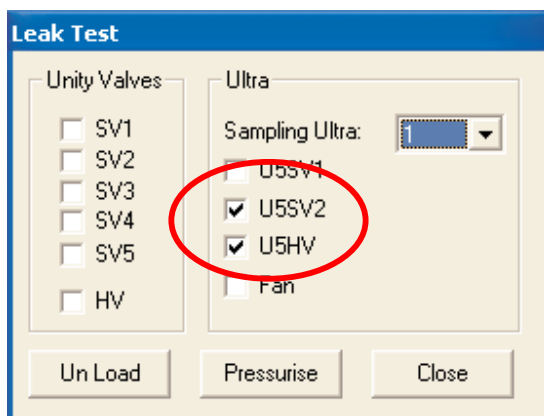
Note: Once the actual flows have been set they will be applied to all methods / sets in a sequence. However, this does not mean that all methods and analyses in a sequence must be subjected to the same split ratio. By selecting whether or not the split is open during different stages of the desorption process (notably tube and trap desorption), it is possible to change from zero to single to double split automatically - i.e. without manual adjustment of the needle valves.

More information is given in the UNITY 2 Operators Manual and in Markes TD Technical Support Note TDTS 22.

4.3. With Sample re-collection - setting and measuring the re-collection (outlet) split flow

When ULTRA 50:50 is operated with sample re-collection, the outlet split (i.e. split during trap desorption) will be directed through the nominated re-collection tube and out of the right hand split vent at the front of the 50:50 module. The re-collection split flow can only be measured with a tube loaded into the sampling position in ULTRA 50:50.

Click the **"Leak Test"** icon select the tube number that you will load for the flow measurement, and load the tube. Ensure all the tick boxes in the dialogue box are unticked except for U5HV2 and U5HV.



The re-collection (outlet) split flow will now be switched on and can be set and measured through the right hand needle valve and measurement point on the front of the 50:50 module.

4.4. Dry purge flow

Dry purge is an optional part of an ULTRA 50:50 2(3) stage desorption method. Dry purge can be incorporated into any 50:50 method either **with** or **without** sample re-collection. The dry purge flow is vented through the right hand vent, i.e. the same vent as is used for the re-collection split flow above.

4.4.1. Dry Purge with sample re-collection

When using dry purge in a method **with** sample re-collection, the dry purge flow will be **the same flow** as set for the re-collection (outlet) split flow.

4.4.2. Dry Purge without sample re-collection

When using dry purge in a method **without** sample re-collection, the dry purge flow must be set **in the same way** as the re-collection (outlet) split flow above even though the re-collection split will not be used in the method.

4.5. Mass Flow Controller (with Conventional or Electronic Carrier gas Control)

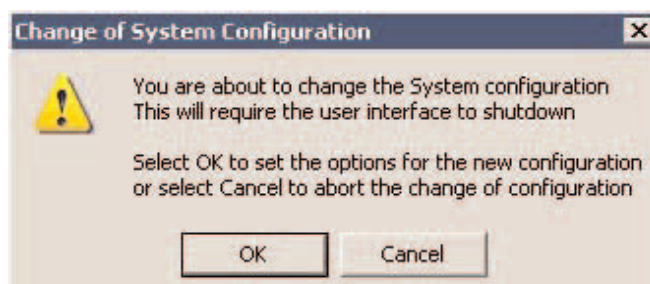
ULTRA 50:50-UNITY 2 systems can be configured with a 1,2 or 3 mass flow controllers.

Use of an MFC controller means that methods which require completely different gas flows may be put together in the same sequence and run automatically. Further information about the set up of an MFC accessory may be found in the ULTRA 50:50 Installation Manual.

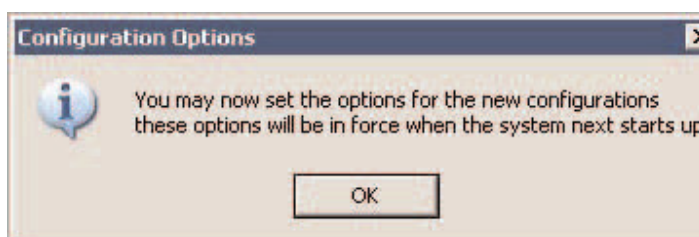
5. Deconfiguring ULTRA 50:50 from UNITY 2

Caution: The interface tube at the end of the ULTRA 50:50-UNITY 2 heated interface is **HOT**. Both UNITY 2 and ULTRA 50:50 must be switched off and the systems allowed to cool before ULTRA 50:50 is deconfigured from UNITY 2.

- Open the go to '**View – Options - Configuration tab**' and select '**Manual**' from the list of sampling options. A '**Change of Configuration**' warning box will appear.



- Press '**OK**' to accept the option and the '**Configurations Options**' box appears



- This reminds the user that the change in configuration will take place at the next system start up. Click '**OK**' on the '**Options**' '**Configurations**' tab. This will close the user interface.
- Once the instruments have been allowed to cool, carefully lift the levered tube oven cover on the left-hand side of UNITY 2. This releases the interface tube from one of the tube seals on UNITY 2. Repeat for the split / re-collection tube oven.
- Keeping the interface tubes in line (as much as possible) with the tube desorption and split / re-collection oven, pull them gently free from whichever ends remain sealed.
- Switch UNITY 2 back on using the main power switch and restart the software.
- Single tube UNITY 2 operation can then continue as described in the UNITY 2 Operators manual.