TT24-7

Near Real Time Monitoring

Operators Manual

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VERSION 2.2

MARKES international

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TT24-7 User Manual - Table of Contents

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TT24-7 Operators Manual

Introduction

This user guide has been designed so that the operator of a TT24-7 system can start an analysis as quickly as possible after installation.

There are four principle sections to the manual.

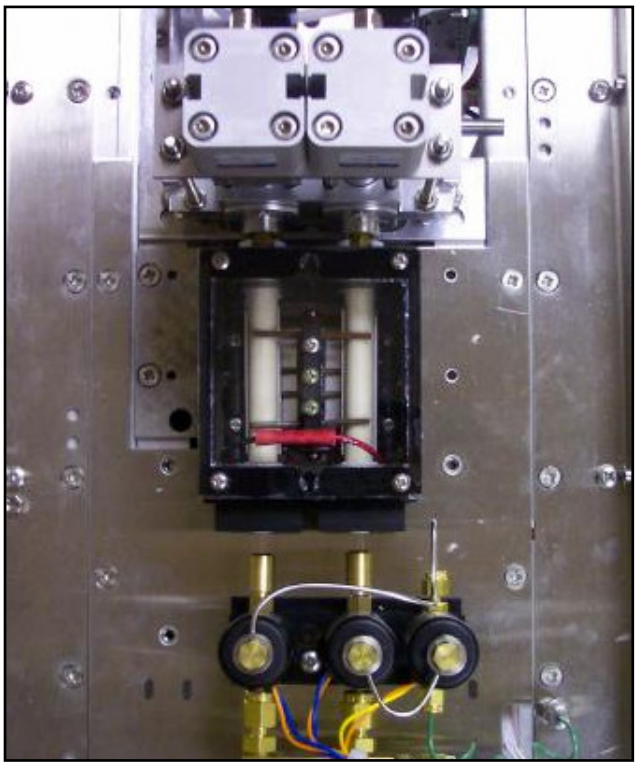
Section 1 Quickstart Guide - for those users who simply want to start analysing samples

Section 2 The TT24-7 Thermal Desorption System - where more detailed knowledge and understanding is required

Section 3 User Installed Components and Maintenance

Section 4 Troubleshooting

There are three TT24-7 hardware configurations available from Markes International. A visual inspection of the unit will ensure that you know which configuration you have prior to reading this manual.



TT24-7e1 *basic system configured only for continuous sampling*

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TT24-7e2

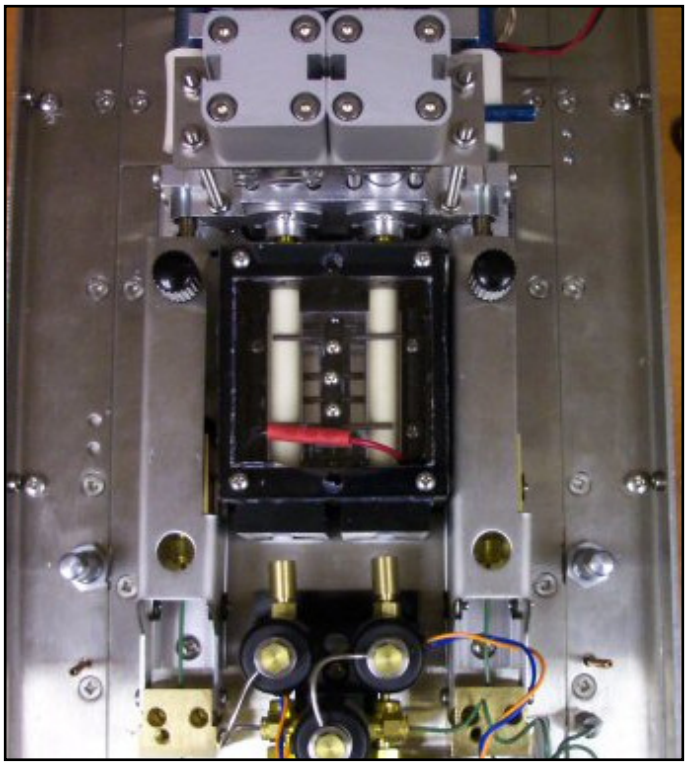
standard system configured with the following additional features:

- tube desorption

- pre-purge to vent / dry purge

- leak test

***Note:** the presence of the tube desorption oven on the left hand side of the trap box indicates that this is an e2 system*



TT24-7e3

standard system configured with the following additional features:

- tube desorption

- pre-purge to vent / dry purge

- split capability

***Note:** the presence of **both** the tube desorption oven on the left hand side **and** the split tube on the right hand side, indicates that this is an e3 system*

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1.0 Quickstart Guide using the TT24-7

This section is in support of the "Quickstart Guide to Operating the TT24-7 Software" (ITS015). This is a laminated card, which is supplied separately with the TT24-7. It is intended to help operators with a quick setup of the system where a more detailed knowledge is not required.

For the Quickstart approach to work successfully, it is assumed that the TT24-7 has been installed and tested by a qualified engineer. Quickstart uses a TT24-7 hardware configuration of e2/e3 to demonstrate the running of the system, however the instructions are also valid for TT24-7e1 systems.

More detailed information about the instrumentation and software is supplied in subsequent sections of this document.

Note: Never turn on the power to the TT24-7 system without the cold traps installed (see section 3.4)

1.1 Running the TT24-7 system

The TT24-7 (e2 and e3) can be operated in two sampling modes; continuous sampling, and single tube desorption. The TT24-7e1 can only be operated in continuous sampling mode.

As the principle operating mode for the TT24-7 is continuous sampling this will be included in the Quickstart setup. Both sampling modes are discussed in detail later in the manual (Section 2.2).

1.1.1 Using an existing TT24-7 method

Running the TT24-7 system in its continuous sampling mode using an existing method is a simple four step process.

Step 1: Loading the TT24-7 method

To load the required TT24-7 method either click on the menu bar icon at the top of the software screen and select the desired method, or from the menu bar select File>Open, and then the required method. The file suffix is ".mth"

Step 2: Preparing the GC/GCMS to accept an external TT24-7 Start Signal

The TT24-7 initiates the GC, GCMS and associated software by sending a remote start signal at the point where the trap fires and releases its retained sample. Prepare the GC (or GCMS) method or sequence, including any data file name and storage location, so that on receipt of the start signal from the TT24-7 the analytical system commences.

Step 3: Check system ready status

Review the TT24-7 status bar at the base of the software to ensure all temperatures and flows are stable and at their set-point. The GC/GCMS system status should also be ready.

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Step 4: Start run

Press the "Start Run" icon on the menu bar, this will initiate the TT24-7 continuous sampling mode. Initially this will be on to trap A during which time trap B purges and trap fires in readiness for the first sample volume coming from trap A.

1.1.2 Creating a new TT24-7 method

Creating a new TT24-7 method follows a similar stepwise path which then leads into the process described above for an existing method (section 1.1.1).

Step 1: Choose the sampling mode

The first step to creating a new method is selection of the sampling mode as described in section 1.1 above. The user interface for both sampling modes is shown below, and varies slightly depending on the selection made (Figure 1.)

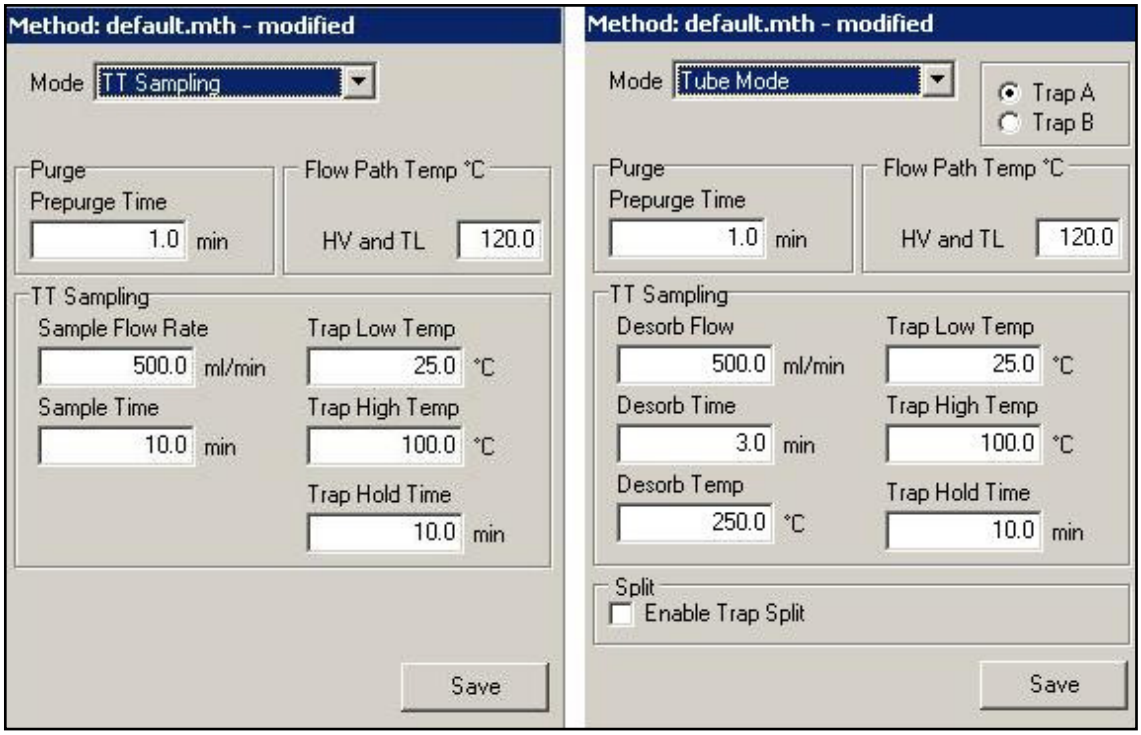

Figure 1. TT24-7 User Interface - showing both sampling modes

Step 2: Enter set point values

After selecting the sampling mode, the desired set point values for each field need to be specified so that the correct temperatures and flows are used for the sampling and desorption phases. The Quickstart guide supplied with the TT24-7 (ITS015) has a description of these fields on the reverse side (see section 2.9 for further details).

Note: the split functionality shown in the user interface above is only present if the split option is configured within the TT24-7. (Product Number TT24-7e3).

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Without this, the system samples in a splitless mode directly into the capillary column for maximum sensitivity.

Step 3: Save the method

After all the set-points are specified click the "Save" button shown on the interface above. This allows the operator to specify the correct method file name ending in a .mth suffix.

To operate the TT24-7 simply follow the stepwise process described above in section 1.1.1

2.0 The TT24-7 Thermal desorption System

This section covers the design philosophy of the TT24-7, the sampling protocols available, and the hardware options that can be configured to achieve these different sampling techniques.

2.1 An introduction to the TT24-7 system

Thermal desorption as an analytical technique can be broadly divided into two sampling categories namely off-line, or on-line. Off-line sampling incorporates both tube based (pumped or diffusive), and canister / bag (grab) samples, whereas for on-line analysis the sample is a flowing gas stream passing directly into the focusing trap for enrichment.

Tube based sampling has the benefit of two stage pre-concentration i.e. from the primary tube into the cold trap of the thermal desorption system (e.g. UNITY), and then from the cold trap into the capillary column. These two phases can typically enrich the sample by a factor of 10^6.

However for on-line analysis a much faster analysis time is required, so that analytical data can be produced in the shortest time interval. This is facilitated by sampling directly onto a cold trap for single stage enrichment. This ultimately leads to near real time analysis (NRT) where all the sampling parameters are running within the shortest time frame, and all the analytical conditions are optimized for high speed chromatography. Time frames as short as 5 minutes are possible with NRT.

Off-line analysis is therefore used for ultra low level sampling over extended time periods (i.e. 8 hr time weighted average TWA), whereas on-line analysis is used for detecting potentially harmful levels at extremely low concentrations in the shortest time frame.

The main rate limiting step in a single trap TD system for on-line analysis is the cool down time of the trap. During this phase which can last for ~ 5 minutes, no sample can be adsorbed onto the trap. For most applications this is quite adequate, and combined with high speed GC provides for excellent on-line analysis. However for certain compounds i.e. chemical warfare agents (CWA) and certain toxic industrial chemicals (TIC) this time frame is still too long, because of their extreme toxicity to the human population. Ultimately a sampling regime is

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required which is continuous, with no time blind spots. To achieve this, a system containing two traps (A and B) is required, working in tandem, so that while one is receiving and focusing sample, the other is desorbing into the analytical system. The process then alternates between each trap.

The TT24-7 has therefore been designed as a twin trap system to facilitate NRT analysis.

2.2 TT24-7 Hardware configurations

There are three TT24-7 hardware configurations available. Ensure that you know which configuration you have prior to reading this manual (see Introduction).

TT24-7e1 standard system configured only for continuous sampling

TT24-7e2 standard system configured with the following additional features:

- tube desorption
- pre-purge to vent / dry purge
- leak test

TT24-7e3 standard system configured with the following additional features:

- tube desorption
- pre-purge to vent / dry purge
- leak test
- split capability

2.3 Sampling modes

Depending on the hardware configuration of your TT24-7 there are two sampling modes available via the User Interface:

Continuous sampling - for continuous sampling from a stream of gas/air

Tube Desorb - allows the desorption of a sorbent tube

Note: TT24-7e1 only allows the continuous sampling mode of operation

TT24-7e2 and TT24-7e3 allow both sampling modes of operation

2.3.1 Continuous sampling

Available with hardware configurations e1, e2 and e3

For continuous sampling, the "TT Sampling" mode of operation must be selected in the TT24-7 software.

Gas samples which are above atmospheric pressure (Max 50 psi) can be analysed by the TT24-7 system. For samples where the pressure is at or below atmospheric pressure a vacuum pump is required to pull the sample through the instrument. Sample flow is regulated for all pressures using an internal mass flow controller (MFC) capable of controlling gas flows from 50 mL/min to 1 L/min. Sample gas is drawn into the TT24-7 through a ¼" Swagelok fitting on the right hand side of the instrument close to the valving assembly. This union could be extended with

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additional plumbing to the exterior walls of the TT24-7, to facilitate connection of a remote sample line or sampling bag (figure 2.)

Figure 2. Sample gas inlet on TT24-7

Continuous sampling starts by initiating the TT24-7 control software (see section 2.9), which in turn starts regulating the sample flow rate and time of the gas stream to be analysed. This results in a total volume of gas sampled onto each trap. After the sampling period the trap is purged for a defined period of time to remove residual air / moisture. After purging the trap, the system enters the trap fire phase in which sample is backflushed from the trap into the capillary column. Backflushing the trap with carrier gas provides an efficient mechanism to sweep compounds off the trap in the reverse direction to sampling at the point of trap fire. This allows the use of traps containing more than one sorbent bed in order of adsorption strength thereby covering a much broader range of analyte that can be analysed.

This process can be entirely splitless for maximum sensitivity, or if the split option is available (configuration TT24-7e3), the sample will be split at this point.

See sections 2.5.1 and 2.5.2.1 to review the flow schematics for continuous sampling.

To assist in understanding the sequence of events occurring during continuous

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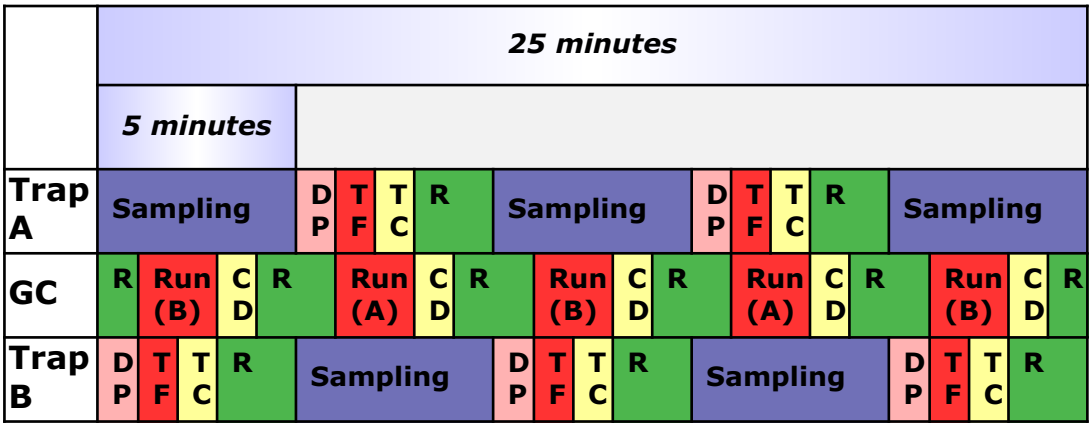
sampling, and how this relates to the GC oven cycle, figure 3 represents diagrammatically the operational status of both traps (A and B) relative to the GC cycle. An arbitrary sampling time of 5 minutes is shown.



Key:

DP	-	Pre / Dry - Purge
TF	-	Trap fire
TC	-	Trap cooling
CD	-	GC oven cool down
R	-	Ready (awaiting sampling (TD), awaiting start run (GC))

Figure 3. Schematic representation of TT24-7 sequence in continuous operation

When the TT24-7 starts, trap A immediately goes into sampling mode, whereas trap B goes through a purge, trap fire, and trap cool sequence of events, this also instigates the first GC oven cycle. This maybe considered as a first run effect for both traps (trap A firing eventually), and the GC oven. After trap A fires both traps will be conditioned and the GC column cleaned of any residual material.

It is important to note that the GC cycle time must be less than the TT24-7 sampling time, or this will add to the overall cycle time of the method. If the GC cycle time increases during the continuous sampling mode, e.g. because the oven cool down time increases, then the system will go into an extended sampling mode for the trap which is currently sampling. This continues until the GC does eventually become ready, at which point the trap fires, and the sequence continues. See section 2.5 for further information on the TT24-7 flow schematics. Additionally the sampling time must be greater than the sum of purge, trap fire and trap cooling times (typically 2 minutes).

2.3.2 Single tube desorption

Available with hardware configurations e2 and e3.

The TT24-7 may be configured with the capacity to desorb a single tube. For this type of analysis "Tube Mode" must be selected as the sampling mode in the TT24-7 software (figure 1).

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The tube to be analysed is inserted into a dedicated tube oven located on the left hand side of the (front facing) instrument. The tube is retained and sealed by two o-rings (P/N U-COV10), one at either end of the oven housing. Figures 4 and 5 below show the TT24-7 with the tube accessory fitted.

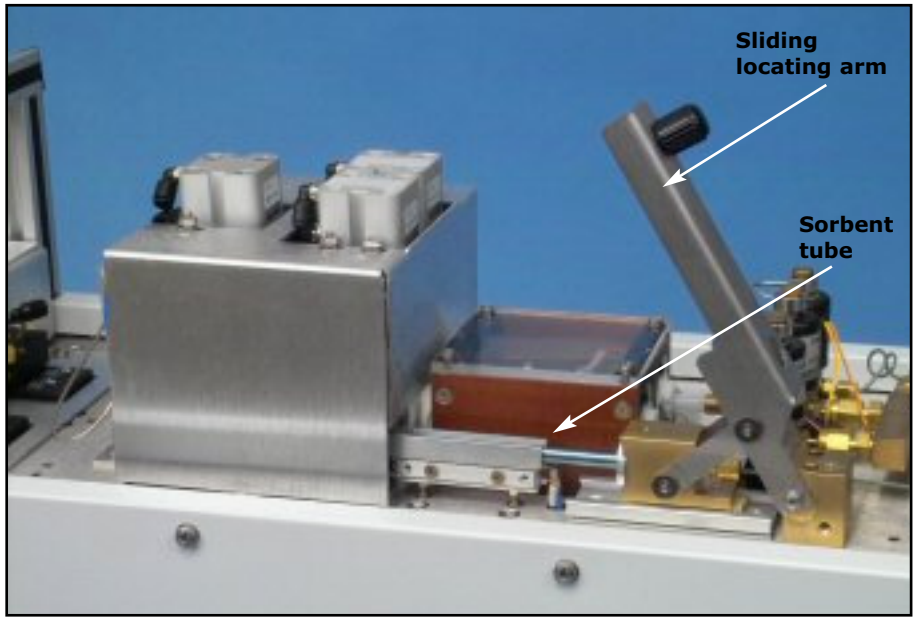

Figure 4. TT24-7e2 showing tube accessory fitted and sorbent tube ready to be sealed into position

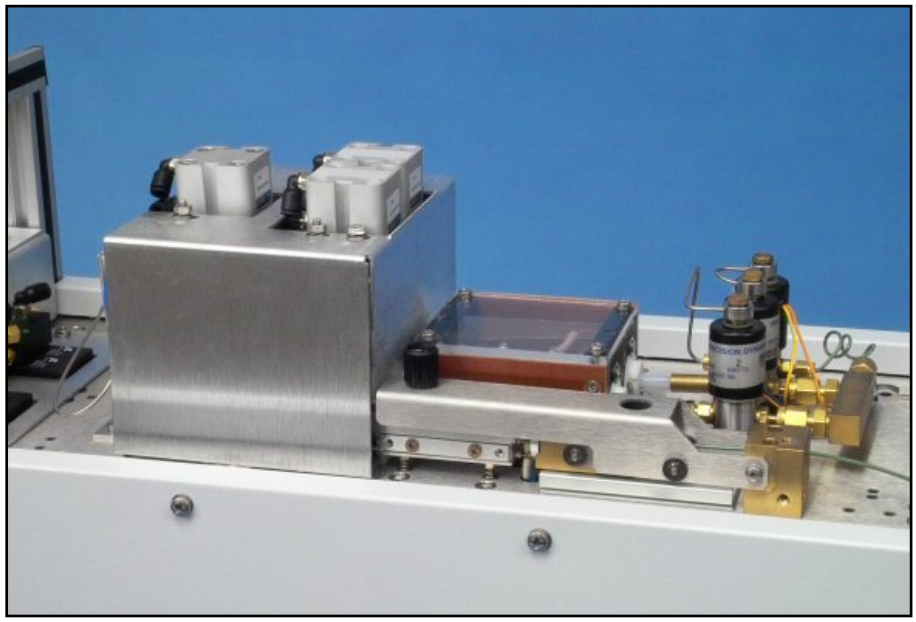

Figure 5. TT24-7e2 showing tube accessory fitted and sorbent tube sealed into flow path

The flow schematics for the tube desorption process can be seen in section 2.5.2

The ability to desorb a single tube using the TT24-7 considerably extends the functionality of the system, beyond that of a single mode continuous sampler.

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An example of this flexibility is the capacity of the system to desorb the analytes retained within the tube onto either trap A or trap B. This is a user selectable function within the TT24-7 "Tube Mode" sampling configuration in the software (See section 2.9). This enables equivalency of trap performance to be assessed by desorbing a test sample initially into trap A and then into trap B, and comparing the data. This could also be used as a method validation tool to check for system bias for each trap.

Single tube desorption also enables calibration of the detector within the associated GC or GCMS system. If a tube is spiked with a standard of known concentration and analysed then the detector can be easily calibrated. This will enable quantitative analysis of subsequent samples either from the tube or using the continuous sampling mode.

The tube desorption configuration can also be used for real tube sampling. In certain applications, a confirmatory tube is positioned within an environment to back up a continuous monitoring station. The sampling process could be pumped or diffusive. If the continuous system alarms, then tubes could be analysed on the TT24-7 to confirm this response.

2.3.3 Split sampling

Available with hardware configuration e3 only.

The TT24-7 may be configured with the capacity to split the sample at the point of trap fire. This functionality can be turned on and off in the TT24-7 software (see section 2.9).

When split sampling is not configured the TT24-7 will desorb sample from the trap in a splitless manner directly into the analytical capillary column. For those applications where maximum sensitivity is required this is the preferred mode of sampling. However there are instances where a more flexible mechanism for loading sample into the column is required. In this instance the ability to split the sample at the point of trap fire is advantageous.

The split flow is controlled by a small needle valve on the right hand side of the TT24-7. Figure 6 shows the TT24-7 with the split accessory fitted (TT24-7e3). Measurement of the split flow is achieved using a digital flow measuring device connected to this vent line.

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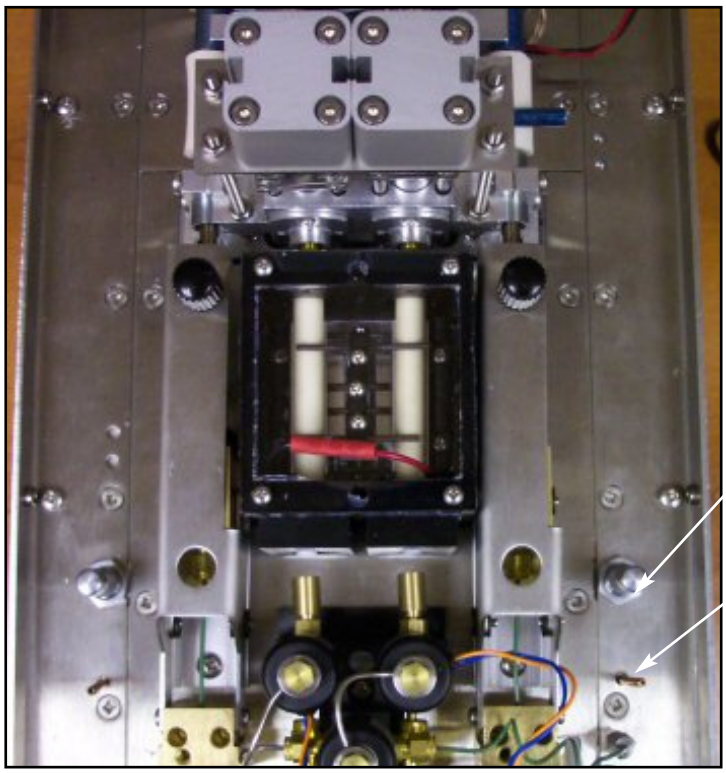

Figure 6. TT24-7e3 showing the tube desorb and split accessories fitted

The split ratio is then a function of the column flow relative to the total flow entering the trap.

$$\text{Split Ratio} = \frac{\text{Column Flow}}{\text{Column Flow + Split Flow}}$$

An additional benefit of split mode sampling is the increase in carrier gas flow rate passing through the trap during the trap fire phase. This will be considerably higher than in splitless mode, where the flow through the trap is equal to column flow. Depending on the column / detector this ranges typically from approximately 10 mL/min down to 3 mL/min. The increase in flow is directly proportional to the split flow, and results in a faster movement of retained sample from the sorbent bed into the column. This allows for higher initial operating temperatures in the capillary column as the band width of sample entering the column will be much sharper. In splitless mode the column needs to be cooler to promote an efficient focusing effect on the stationary phase. This is a standard procedure for splitless injection.

The flow schematics for the split configuration (e3) are shown in section 2.5.3

2.4 Principle system components

The main functional components of the TT24-7 (excluding electronics), consist of:

2.4.1 Valving assembly

2.4.2 Pneumatics module

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2.4.3 Peltier cooled cold traps (A and B)

2.4.4 Heated sample transfer line

2.4.5 Optional tube desorption and / or sample split pneumatics

2.4.1 Valving assembly

The valving assembly within the TT24-7 consists of up to three heated valves. Each valve is manufactured from a solid block of PTFE, and machined to provide the required flow paths for sampling and desorption of a sample gas stream.

For hardware configuration e1 which only offers the continuous sampling configuration, two valves are required, one for each trap. Each valve has three points of connection i.e. where the trap locates into the valve, where sample gas enters the valve, and where the desorbed analytes leave the valve. Figure 7 is a schematic which shows these connections, and the two opposing pins at the top and bottom of the valve which control the active flow path within the valve i.e. sampling, dry-purging, (backflush) desorption etc. These pins are either up or down, and are actuated by an external gas supply.

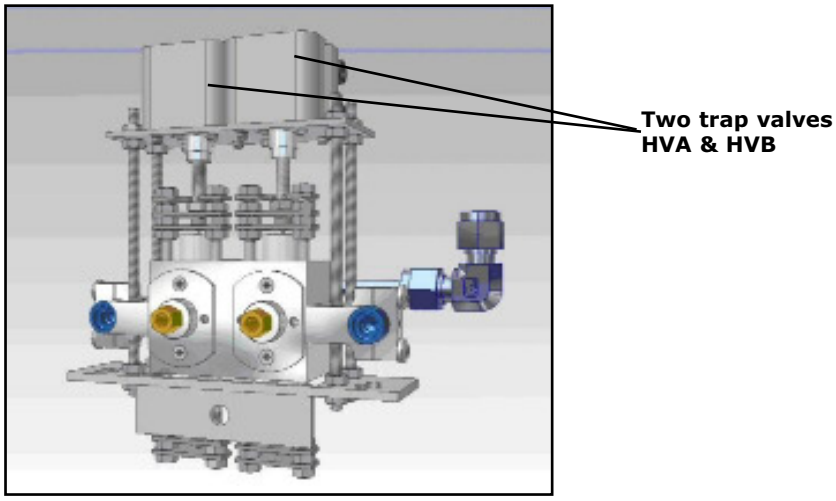

Figure 7. Schematic showing the two TT24-7 trap valves

When the additional configurations of tube desorption and split sampling are built into the TT24-7 (configurations e2/e3), then a third heated valve is required, which connects via a T connection to the two valves described above.

If the split function is configured (configuration e3) this (rear) valve has four points of connection, i.e. to the T piece connecting the two front valves, to the tube desorption line, to the split effluent line and finally to a transfer line leading to the capillary column.

See section 2.5 for an overview flow schematic of this 3 heated valve configuration.

Figure 8 shows the schematic of this rear valve.

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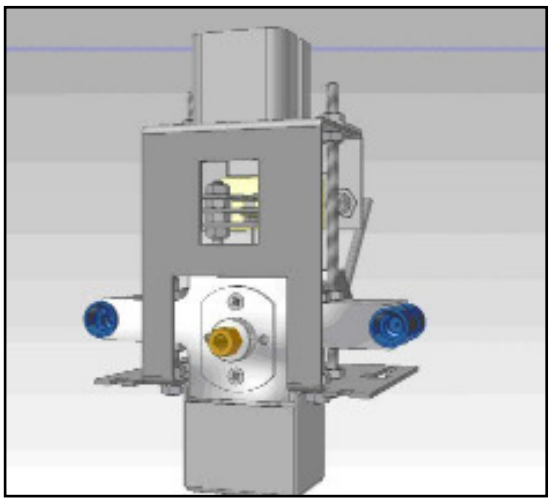

Figure 8. Schematic of third (rear) valve

Figure 9 shows the relative position of these three valves valve within the TT24-7. It also shows the location of the heated transfer line connection port into the TT24-7, the twin trap box assembly, the pneumatics module, and the tube desorption assembly.

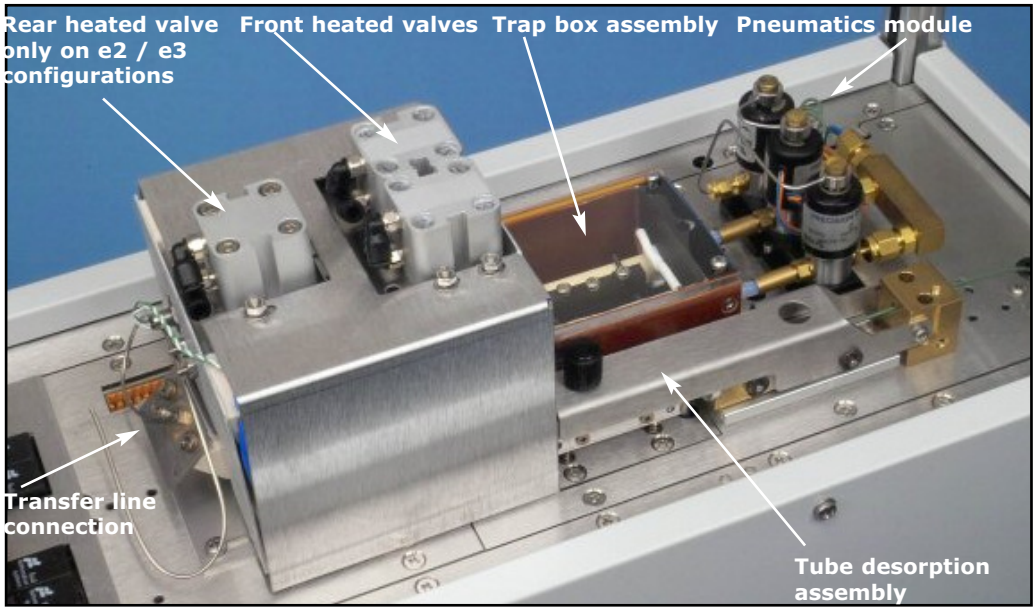

Figure 9. Position of the principle system components of the TT24-7

2.4.2 Pneumatics module

The pneumatics module is a single sub-assembly located at the front of the TT24-7 as seen in Figure 9. It is locked in position by a single M4 retaining screw. Removal of a trap from its location in the valve box requires the pneumatics assembly to be removed. See section 3.4 for complete details of how to remove / replace the traps.

The module consists of three solenoid valves SV1, SV2, and SV3. See section 2.5 for full details of flow schematics.

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These three-way (on / off) solenoid valves control the source and flow direction of gas passing through the traps. These flows consist of:

The sample gas for analysis (continuous or tube)

The carrier gas to backflush the traps

The purge gas to flush the traps of air and moisture prior to trap fire

SV1 and SV3 are connected to the traps A and B, and SV2 connects sequentially to both valves.

When either SV1 or SV3 are ON they are connecting the associated trap (SV1=trap A, SV3=trap B) to the mass flow controller for any of the trap sampling modes discussed in section 2.3.

When either SV1 or SV3 are OFF they are either receiving carrier gas from SV2 to backflush the trap at trap fire into the capillary column, or they are receiving dry–purge gas from the traps to remove air and moisture in the trap.

2.4.3 Peltier cooled traps A and B

The TT24-7 contains two identical quartz cold traps which are located within the trap housing (figure 10). The two traps lie parallel to each other and connect at one end into a heated valve and at the other end into the pneumatics assembly. The location of the trap box is shown in Figure 9.

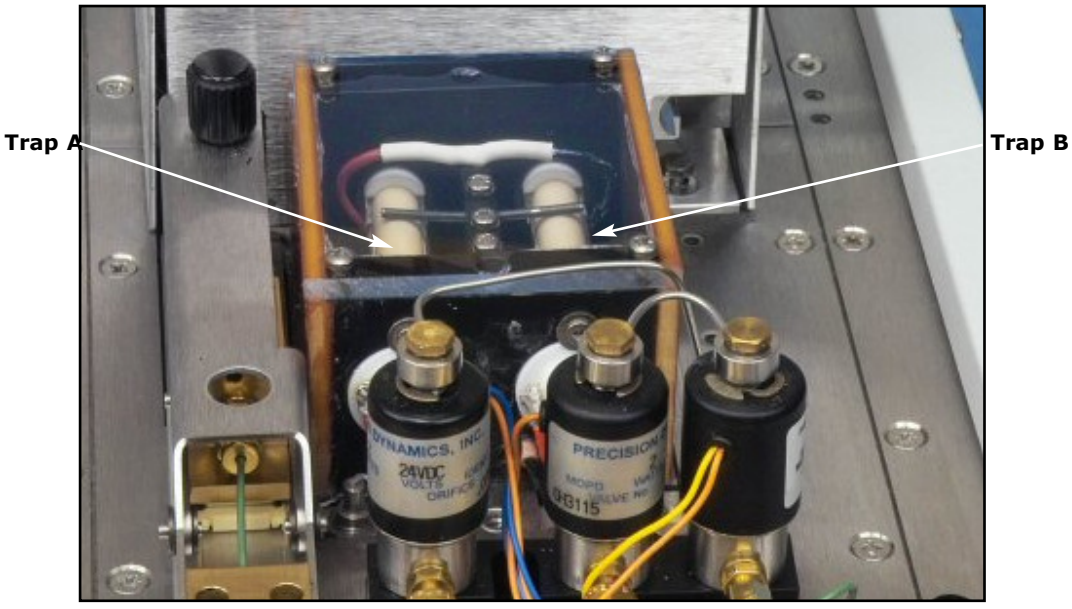

Figure 10. Position of Trap A and Trap B within the trap housing

Each trap is inserted through a ceramic heater sleeve and at the point of trap fire heats at rates approaching 100°C/second. This extremely fast heating rate removes the retained analytes from the sorbent bed(s) very efficiently which allows the traps to operate in splitless mode for maximum sensitivity.

Each trap is positioned directly on to its own Peltier cooling element. This electro-thermo cooling enables each trap to cool to sub-ambient temperatures during the

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sampling phase which greatly enhances the trapping efficiency of the sorbent material. The lower temperature is a function of the sample temperature, valve temperature and sampling flow rate.

Dry air, nitrogen or helium is used to purge the trap box to prevent the build up of moisture and the potential for icing which would effect the temperature measurement of the traps.

The narrow bore traps are made of quartz, are restricted at the inlet / outlet end and have a quartz collar on the unrestricted end (figure 11). They may be packed with up to 60 mm of sorbent(s) which are separated and retained by quartz wool. Sample enters the trap through the restricted inlet end and at the point of trap fire the trap is backflushed (carrier gas flow reversed) and the analytes are desorbed back out of the restricted end.

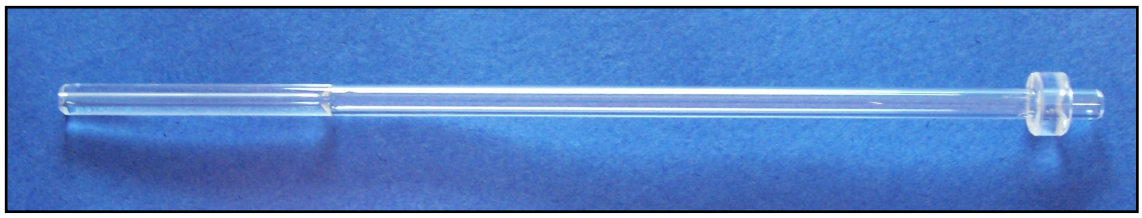

Figure 11. TT24-7 cold trap - note the restricted inlet / outlet end and the quartz collar on the unrestricted end.

Backflushing the trap allows the use of multiple sorbent beds in the trap, each bed having a different sorbent strength, with the weaker sorbent at the start of the trap. An example would be Tenax TA™ followed by HayeSep D™, or Chromosorb 106™ etc. This sequence of sorbent materials allows for a much broader range of analytes to be retained on the trap over that for a single bed. However greater consideration is then required in terms of maximum operating temperature, as many sorbents have quite different maximum upper temperature values. (See Appendix 3 for further information regarding sorbent selection and sorbent maximum temperatures).

Backflushing the traps allows each analyte to come off the retaining bed alone, without contact with a stronger sorbent material which could be irreversible or detrimental to peak shape.

Note: Installation and removal of the cold traps is a user operation. However, it is necessary to follow the instructions carefully to avoid damaging the traps. Full instructions are given in section 3.4. Collared traps are compatible with the TT24-7 Trap Extraction Tool (TTD-5032)

Note: You may have TT24-7 traps in stock which are of an earlier design with NO collar on them - these traps may be used in your TT24-7 system but are not compatible with the Trap Extraction Tool - extra care must be taken when removing these traps from the system.

2.4.4 Heated transfer line

The analysis and detection of desorbed analytes from the cold traps is achieved using conventional analytical instrumentation. This includes GC, GCMS and in

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certain instances direct MS technology. For speciation of components, gas chromatography is the preferred choice using capillary columns for maximum separation efficiency. Typically columns with an internal diameter of 0.32 mm or 0.25 mm are used, with lengths ranging from 10 m to 60 m. All standard stationary phases are suitable. For splitless injection, carrier flow rates equal to or greater than 3 mL/min are recommended for efficient transportation of analytes from the sorbent bed into the column. In split mode sampling the flow rate is by definition considerably greater than this anyway.

Interfacing the TT24-7 to the GC capillary column can be achieved by a number of mechanisms. The most common approach uses a length of uncoated, deactivated fused silica transfer line, which connects and seals into the TT24-7 using a $^{1}/_{16}^{th}$ Swagelok nut and a 0.4 mm id graphitised vespel ferrule. Installation of the transfer line is discussed in more detail in section 3.3.

The transfer line is surrounded by a length of PTFE sleeving of slightly greater internal diameter than the outer diameter of the fused silica. The PTFE is used to assist in the insertion and feeding of this length of tubing through the outer heated jacket of the transfer line. It also maintains a constant temperature profile down the length of the fused silica preventing hot spots. The outer layer of the transfer line is insulation material which is used to maintain a consistent and controllable temperature along its 1.2 m length (figure 12).

The transfer line is maintained at the same temperature as the heated valves, i.e. the valve temperature controls the transfer line temperature setting. This is typically up to 200°C.

Connection to the capillary column within the GC oven requires a suitable hole in the GC covers to allow the outer insulation of the transfer line to pass as far as possible to the surface of the oven without assimilating heat from the oven. For certain GC systems the split / splitless injection port is used as a conduit after the internal liner etc. is removed.

The capillary column can then be connected to the transfer line using deactivated glass unions, or deactivated steel connectors and ferrules.

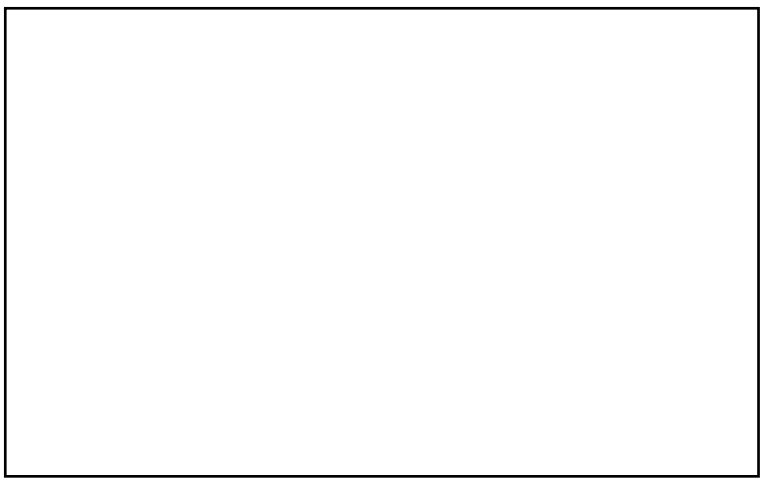

Figure 12. Transfer line connection between TT24-7 and GC system

As an alternative to the fused silica transfer line approach, certain capillary

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columns are available which have a built in length of uncoated tubing at the end of the column. This length can be inserted through the PTFE sleeve described above and connected directly into the TT24-7. The advantage of this method is the exclusion of a connector within the transfer line, which may be the source of problems with leaks, or activity (see Section 4 - Troubleshooting for further details). The disadvantage of this technique is that changing columns needs disconnection at the TT24-7 fitting and removal from the transfer line.

Where a single column is likely to be used consistently the latter approach is recommended, where multiple columns are to be used in an interchangeable manner the former is advised.

2.4.5 Tube desorption and sample split pneumatics

These sampling options have been discussed in general in section 2.3.2 and 2.3.3. Additional information can be found in section 3.0 - User Installed Components and Maintenance and the sampling schematics can be found in section 2.5.

2.4.6 Electronic pneumatic control (EPC)

Two gases pass into the TT24-7 system during its operation (excluding the trap box purge flow), namely carrier gas and sample gas.

The sample gas is typically air however other gas types can be accommodated i.e. nitrogen, hydrogen, helium and carbon dioxide. Control of the sample gas flow rate is achieved using an internal mass flow controller with an operating range from 50 mL/min to 1 L/min. The gas type must be specified for correct calibrated control which is based on its density. See section 2.9.2.5.1 for further information.

The carrier gas should be chosen for optimum performance of the associated chromatographic system and this is typically helium, although other choices are available e.g. nitrogen and hydrogen. The carrier gas flowing through the system performs several functions including dry purging the traps prior to trap fire, back flushing the traps at trap fire, transporting sample from the tube accessory or capillary inlet into the selected trap etc.

If split sampling is configured then this also places demands on the supply of carrier gas. Without electronic pneumatic control this would affect the column head pressure, resulting in large differences in the retention time of eluting compounds run to run.

Section 2.5 shows the schematics illustrating the flow protocol within the TT24-7 for each sampling mode within each hardware configuration.

The carrier gas supply entering the TT24-7 originates from the associated GC system and in particular from the electronic flow module normally supplying gas to a split / splitless injection port. This gas supply is now redirected from the capillary inlet into the TT24-7.

An important aspect associated with the control of the carrier gas is called closed loop feedback. This relates to controlling the capillary column head pressure so that whatever flow demands are made by the various processes occurring within

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the TT24-7, the column pressure remains at the defined value determined by the GC method. Normally this is a constant pressure value, however in certain circumstances pressure or flow programming may be required.

By controlling the pressure at the head of the column, the retention time of compounds eluting from the column will be fixed. Chromatographically this is highly advantageous as it produces reproducible retention times for each compound in all analyses. This ultimately leads to the generation of absolute retention time databases for compound identification.

Closed loop feedback is particularly important when the sample is being split. The split flow passes through the rear heated valve and out via a needle valve for control as discussed in section 2.3.3. This is immediately adjacent to the point where the transfer line leading to the capillary column enters the TT24-7, and without electronic control would directly affect the column head pressure i.e. as the split flow increased the head pressure would decrease and compound retention times would vary accordingly.

The pressure at this point is monitored by the pressure sensor within the GC flow module, this sensor subsequently controls the total flow being delivered into the TT24-7. This process is closed loop feedback. If the split flow is increased the pressure at this point would normally drop, however the sensor compensates for this by increasing the total flow into the TT24-7 to provide for the extra split flow and maintain the column pressure.

Most GC manufacturers have an LCD built into the instrument and information relating to the GC inlet flows can normally be observed. One such parameter is the "Total Flow" delivered by the inlet into the capillary injection port. When the inlet flow module is configured with the TT24-7 this "Total Flow" parameter is now the flow entering the thermal desorption system. This provides a mechanism to observe the change in total flow as the split flow is varied, i.e. as the split flow increases this value will also increase in direct proportion.

When the analysis is splitless (i.e. no split flow), the flow rate through the trap is equivalent to the column flow which is typically between 3 to 10 mL/min. Once again the flow is controlled by the column head pressure and the total flow demand now is approximately equal to the column flow. The value may be slightly higher than the exact column flow if a septum purge flow is running.

2.4.6.1 Carrier gas connections

Manufacturers of GC systems which have the capability of electronic pneumatic control typically have three pneumatic lines leading from the electronic module into the capillary injection port. These consist of the carrier gas supply line, a septum purge line and a split vent line.

When this module is connected to the TT24-7 system only the carrier supply line and septum purge line are used, the split vent line is redundant. For correct closed loop feedback control the pressure sensor for the inlet must be connected into the septum purge line.

The carrier, septum purge and split vent lines usually connect into the electronic module by means of a single manifold (e.g. Agilent technologies) or individual connectors (e.g. Thermo Electron). To protect the original gas lines (for future

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re-assembly of the capillary inlet) it is recommended that these lines are replaced with additional gas lines rather than being cut.

Figure 13 shows the back panel on the TT24-7 for connection of the carrier gas supply (carrier inlet) and pressure monitoring line (pressure outlet).

Figure 13. Carrier gas supply connections on the back panel of the TT24-7

A flow diagram showing the additional electronic flow module and gas lines to the TT24-7 is shown in figure 14 and further details are given in section 2.5.

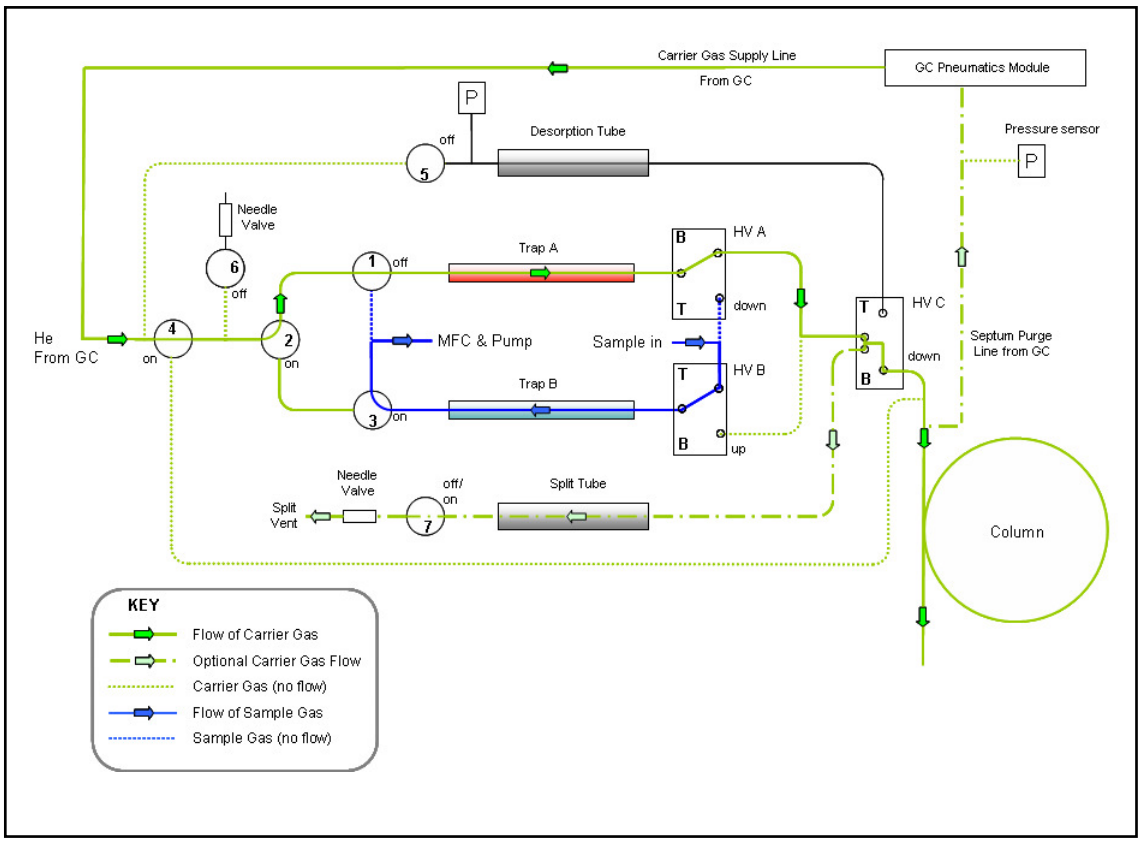

Figure 14. TT24-7 flow schematic (e3 configuration) showing the additional gas lines associated with the GC EPC module

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The pressure sensing Septum Purge line connecting to the TT24-7 may have carrier gas flowing. In some GC systems this is variable. If maximum sensitivity is required then this flow should be stopped or a small sample splitting will occur. This can be achieved by either switching the flow off within the instrument (or software), or simply blanking the exit line with a closed Swagelok nut.

2.4.6.2 Connection to Agilent Technologies 6890GC / 6850 GC

For correct operation of the Agilent Technologies EPC system with the TT24-7, a 6890 GC currently requires firmware revision of A.03.08 or N.04.09. For the 6850 GC serial numbers upwards of US10243001 will be able to provide the same functionality as the 6890 but may require a flashable firmware upgrade.

Note: You should confirm these firmware requirements with your local Agilent Technologies representative.

The Injection Port Mode of the EPC module now has to be configured.

Using either the GC keyboard (or hand held controller (6850GC)) or Chemstation™ software, access the injection port mode. This consists of Split, Splitless, Pulsed Split, or Pulsed Splitless. Select the "Splitless" mode of injection and set the purge time to 999.99 minutes exactly. This is required to prevent the EPC module resetting itself at the completion of each run which is not required with the TD system.

Note: This exact time value is very important for EPC control of the TT24-7.

The EPC module is physically connected to the TT24-7 using either the original gas lines (which must be cut) connecting to the split / splitless inlet, or preferably by using a separate pair of gas lines connecting into their own manifold (Agilent Part Number G2131-80500), which replaces the original three gas line manifold. This type of connection enables the original split / splitless inlet to be reconfigured very easily if required.

If the capillary injection port is not required, then it can also provide an easy access route for the heated transfer line to enter the GC oven for both the 6890 and 6850 models. See section 3.3.1 for further details.

When using the 6890 GC, the total flow of carrier gas entering the TT24-7 can be observed from the keyboard, by selecting the appropriate inlet (i.e. front or rear) and scrolling down to the line "Total Flow".

The "Total Flow" consists of column flow, septum purge flow (if applicable) and the split flow within the TT24-7.

For example in splitless injection mode (TT24-7) if the column flow rate is 3mL /min and the septum purge approximately 3 mL/min then the "Total Flow" will show as approximately 6 mL/minute on the 6890 keyboard.

In split sampling mode (TT24-7) if the split flow is for example 50 mL/min then the "Total Flow" reading will appear as approximately 56 mL/min. This value can be seen to provide an additional electronic readout of the split flow used by the TT24-7 at trap fire.

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2.4.6.3 Connection to Thermo Electron Trace / FOCUS GC

For correct operation of the Thermo Electron DPFC system with the TT24-7 the GC requires an available inlet configured for back pressure regulation.

The injection port mode of the DPFC module to be used has to be configured for split operation but the split flow has to be set to zero (off).

The DPFC module is physically connected to the TT24-7 using the gas lines on the GC that are used for supplying the split / splitless inlet. The carrier supply and septum purge lines should be cut and extended, using the $^{1}/_{8}$-inch unions and green PEEK tubing supplied in the TT24-7 shipping kit, so that they can be connected onto the back panel of the TT24-7. The carrier supply line should be connected to the carrier inlet of the TT24-7 while the septum purge line should be connected to the pressure outlet (see figure 13).

If the capillary injection port is not required, then it can also provide an easy access route for the heated transfer line to enter the GC oven. See section 3.3.1 for details.

2.4.6.4 Connection to other GC systems

Electronic pneumatic control using Shimadzu GC systems is also possible. The carrier gas and septum purge line are configured similarly to both the Agilent and Thermo Electron systems. For more information please contact Markes International Ltd.

2.5 TT24-7 flow schematics

This section reviews the flow schematics for the TT24-7 for the three hardware configurations available (TT24-7e1, TT24-7e2 and TT24-7e3). These will identify the direction of flow of carrier gas, sampling gas, and dry-purge gas for two sampling modes Continuous Sampling and Tube Desorb.

Each section shows the chronological order of events during the thermal desorption process.

The solenoid valves are labelled with a numerical SV designation, going from SV1 through to SV7. The status of each solenoid valve is either ON or OFF.

SV1, SV2, SV3 and SV4 are three-way valves whose ON / OFF status controls the direction of carrier gas flow.

SV5, SV6 and SV7 (where fitted) are two-way ON / OFF solenoids.

The heated (PTFE) valves are labelled HVA, HVB and HVC (where fitted). Each valve is in an UP or DOWN status depending on the two pin position of each valve.

Total (carrier) gas flow into the TT24-7 is controlled by the electronic pneumatic control module (EPC) of the associated GC system. This demand will be based on the carrier gas flow into the capillary column and any split flow or purge gas requirements which are configured and specified during the process. This includes the needle valve regulated split flow and dry-purge flow values.

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2.5.1 TT24-7e1 hardware configuration

TT24-7e1 can only be operated in the continuous sampling mode.

2.5.1.1 Standby

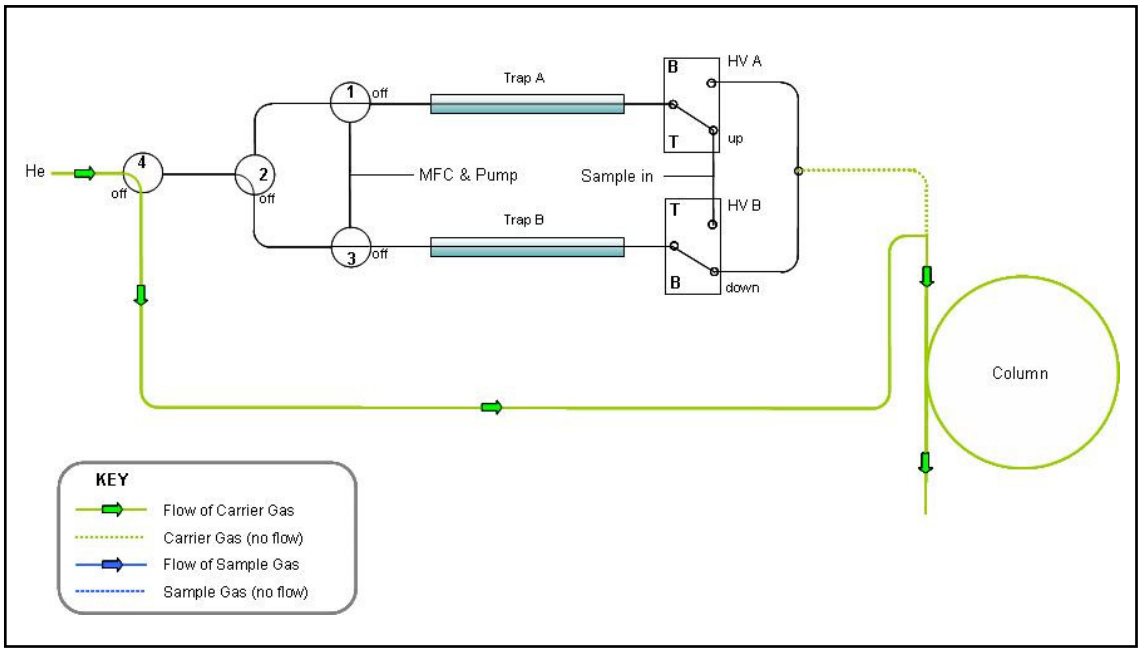

Figure 15. TT24-7e1 - Standby

In this non sampling mode, carrier gas alone is continuously supplied to the capillary column. No other gas flow occurs within the TT24-7 unit during this time.

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2.5.1.2 Trap A sampling, trap B purging

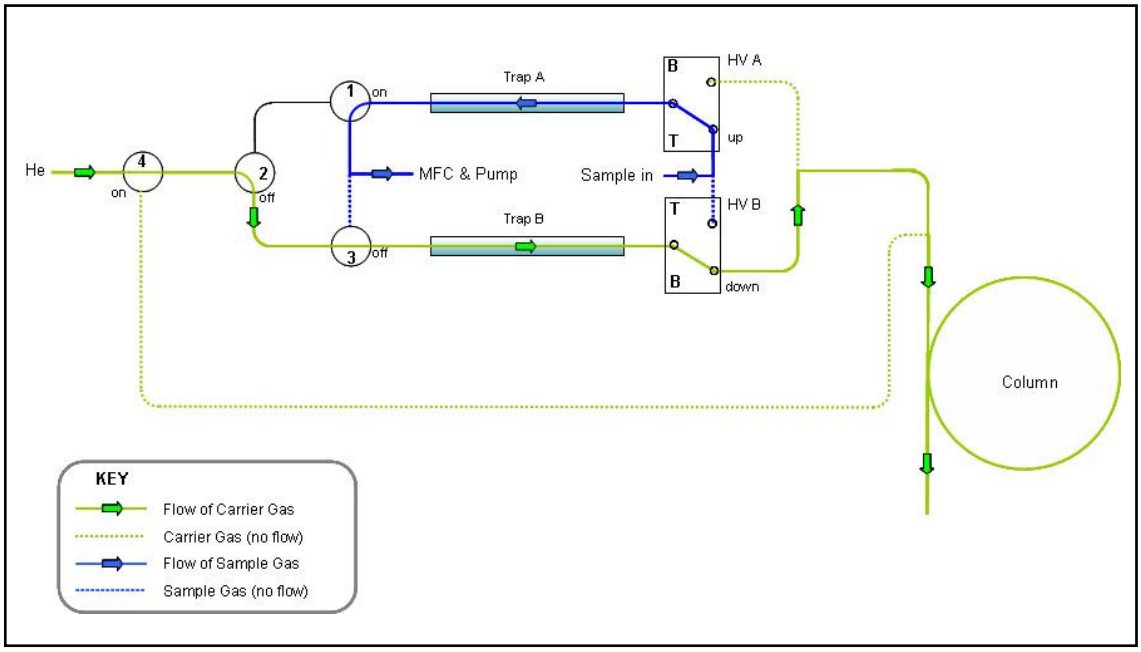

Figure 16. TT24-7e1 - Trap A sampling, trap B purging

By starting the TT24-7 software the continuous sampling process commences. The date and time that the sampling started, and the trap that is being sampled are reported as an information message in the reporting module of the software (see section 2.9.1.7). e.g.

```
05/12/2005 10:49:31    [ INFO] Sampling Started on Trap A
```

The sample passes initially onto trap A, while trap B pre-purges with carrier gas in the desorption direction. This removes air and moisture from the trap prior to firing. The pre-purge flow is determined by the column flow and **should be at least 3 mL/min**.

The sample gas flow rate passing into the TT24-7 is controlled by the internal mass flow controller (MFC).

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2.5.1.3 Trap A sampling, trap B desorbing

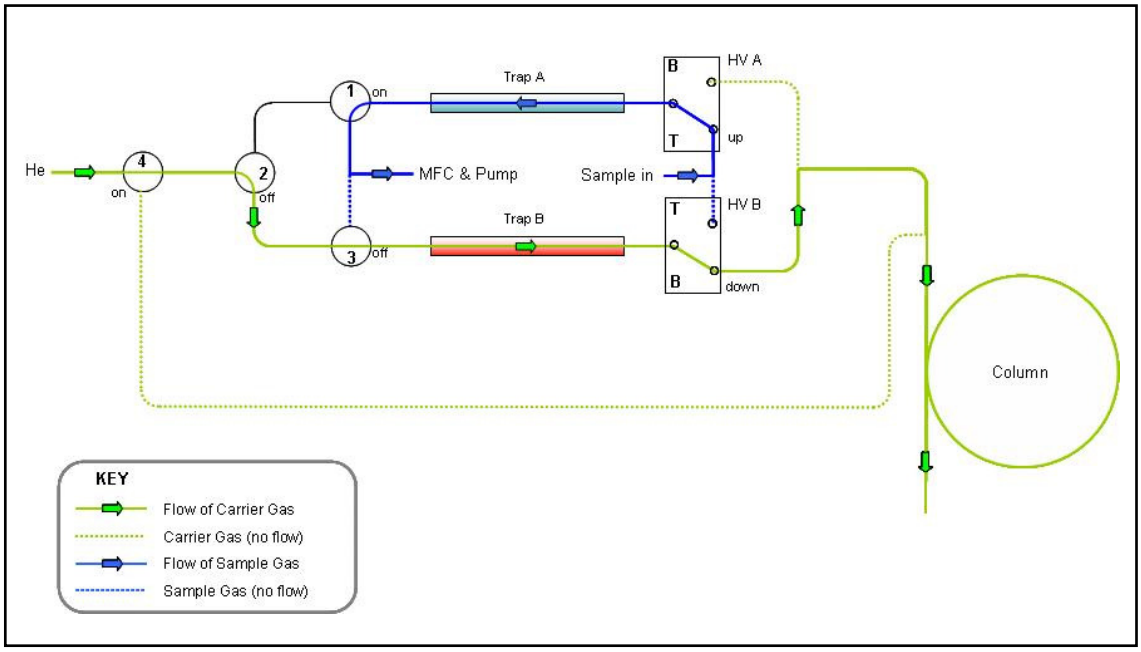

Figure 17. TT24-7e1 - Trap A sampling, trap B desorbing

After purging the trap (B) for the first time, trap fire occurs and flow is directed (backflushed) across to the capillary column. This first (B) trap fire prepares the trap for subsequent sampling. The flow rate through the trap is equal to the column flow rate and **should be at least 3 mL/min.**

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2.5.1.4 Trap A purging, trap B sampling

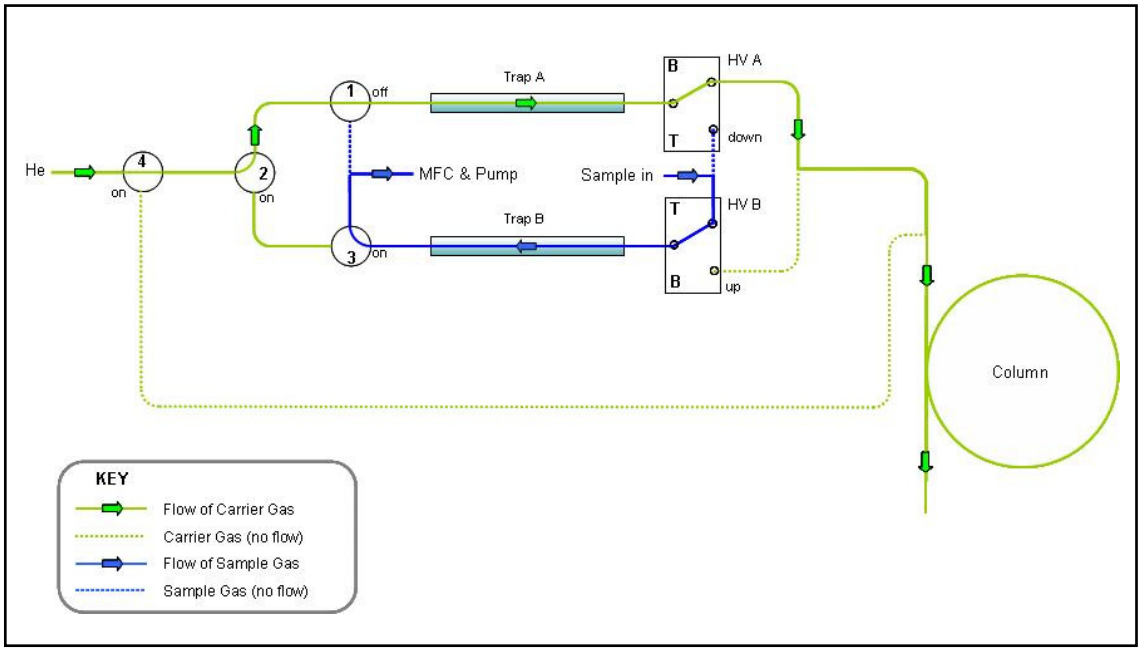

Figure 18. TT24-7e1 - Trap A purging, trap B sampling

After the sampling time for trap A has completed trap A switches into its pre-purge mode to sweep out air and moisture prior to trap fire. Having cooled to its starting temperature after the first clean up trap fire, trap B now goes into its sampling phase. The date and time that the sampling started, and the trap that is being sampled are reported as an information message in the reporting module of the software (see section 2.9.1.7). e.g.

```
05/12/2005 10:59:17    [ INFO] Sampling Started on Trap B
```


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2.5.1.5 Trap A desorbing, trap B sampling

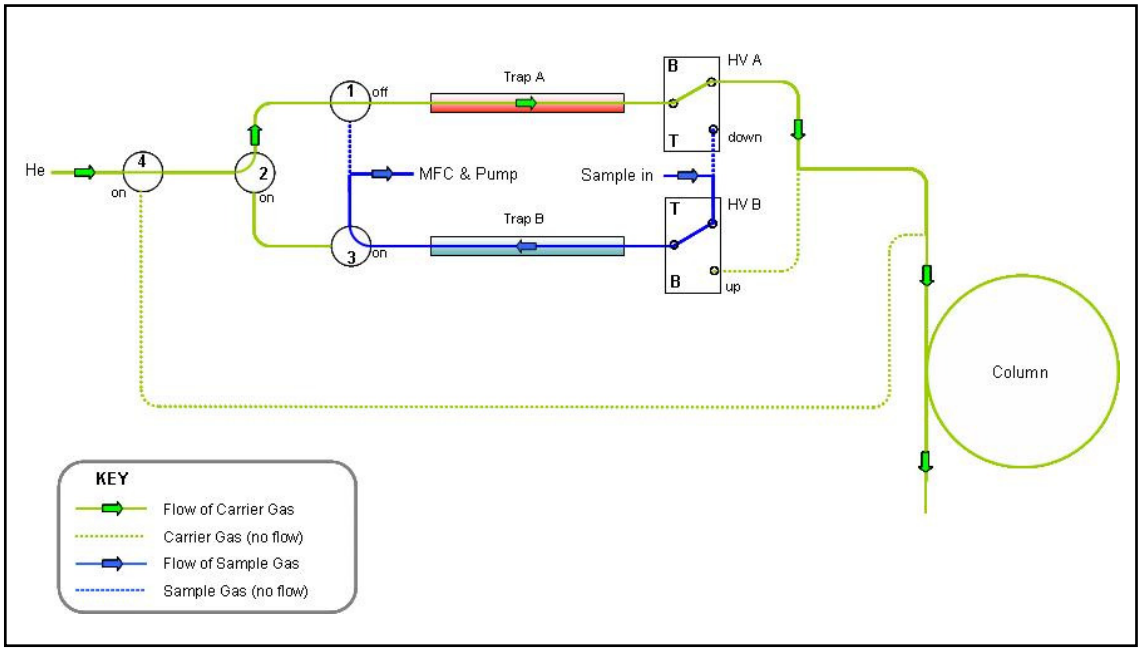

Figure 19. TT24-7e1 - Trap A desorbing, trap B sampling

Trap A now fires. If this is the first run for A this run is treated as a clean up run, however if this is a subsequent run then real sample will be transferred across to the capillary column for analysis.

To see how the time overlap of sampling, purging, trap fire and cooling occurs relative to the GC oven cycle time please refer to section 2.3.1.

The first two runs of the continuous sampling mode (i.e. trap A and trap B) would typically not be used for quantitative analysis of the sampled air but as a clean up process for both traps. After both traps have been fired once then the subsequent samples may be quantified.

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2.5.2 TT24-7e2 hardware configuration

TT24-7e2 may be operated in both sampling modes (continuous sampling and tube desorb).

2.5.2.1 Continuous sampling mode (TT24-7e2)

2.5.2.1.1 Standby

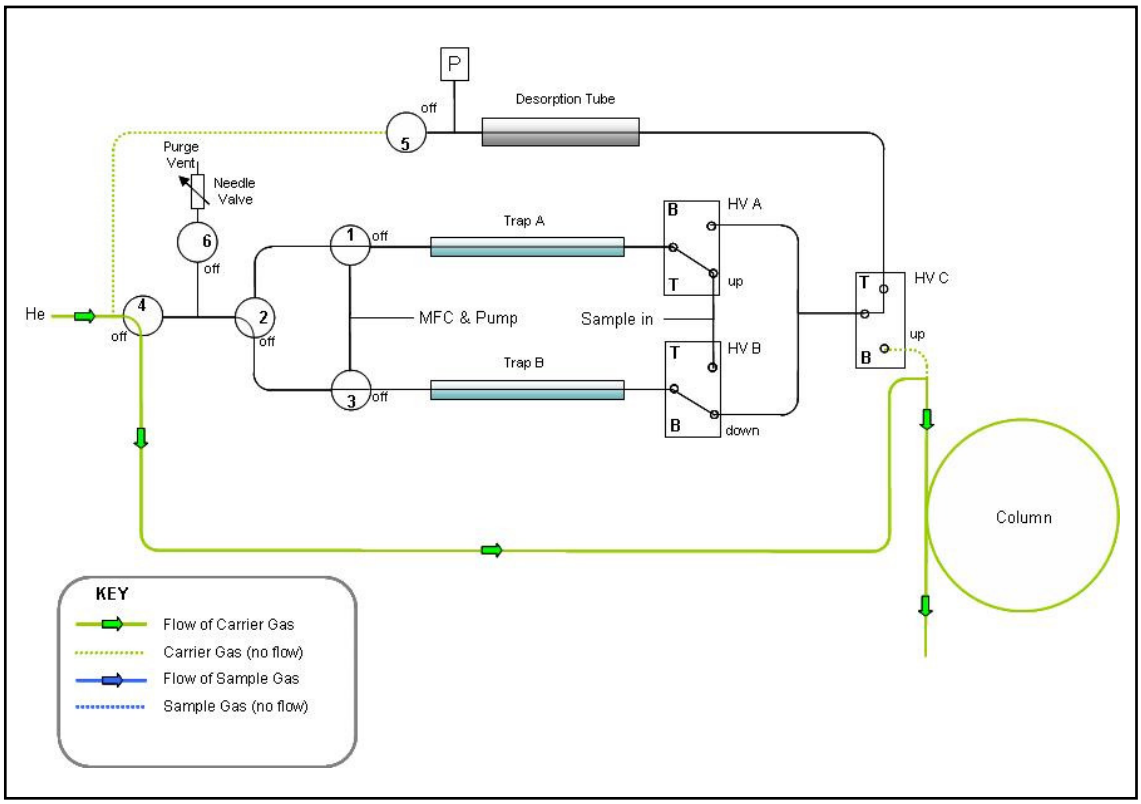

Figure 20. TT24-7e2 - Continuous sampling - standby

In this non-sampling mode, carrier gas alone is continuously supplied to the capillary column. No other gas flow occurs within the TT24-7 unit during this time.

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2.5.2.1.2 Trap A sampling, trap B purging

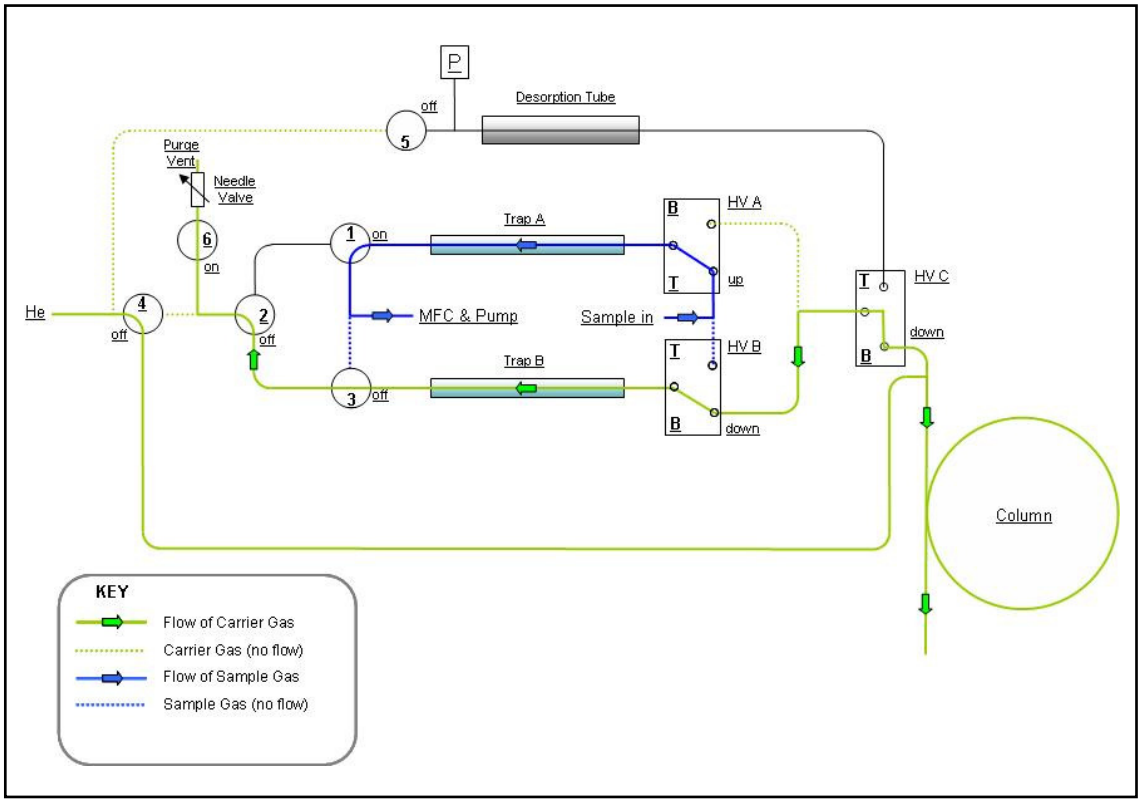

Figure 21. TT24-7e2 - Continuous sampling - trap A sampling, trap B purging

By starting the TT24-7 software the continuous sampling process commences. The sample passes initially onto trap A. The date and time that the sampling started, and the trap that is being sampled are reported as an information message in the reporting module of the software (see section 2.9.2.7). e.g.

```
05/12/2005 10:49:31    [ INFO] Sampling Started on Trap A
```

At the same time trap B dry-purges with carrier gas **in the sampling direction**. This removes air and moisture from the trap prior to firing. The dry-purge flow rate through the trap (B) at this time is controlled by a needle valve connected to SV6.

The sample gas flow rate passing into the TT24-7 is controlled by the internal mass flow controller (MFC).

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2.5.2.1.3 Trap A sampling, trap B desorbing

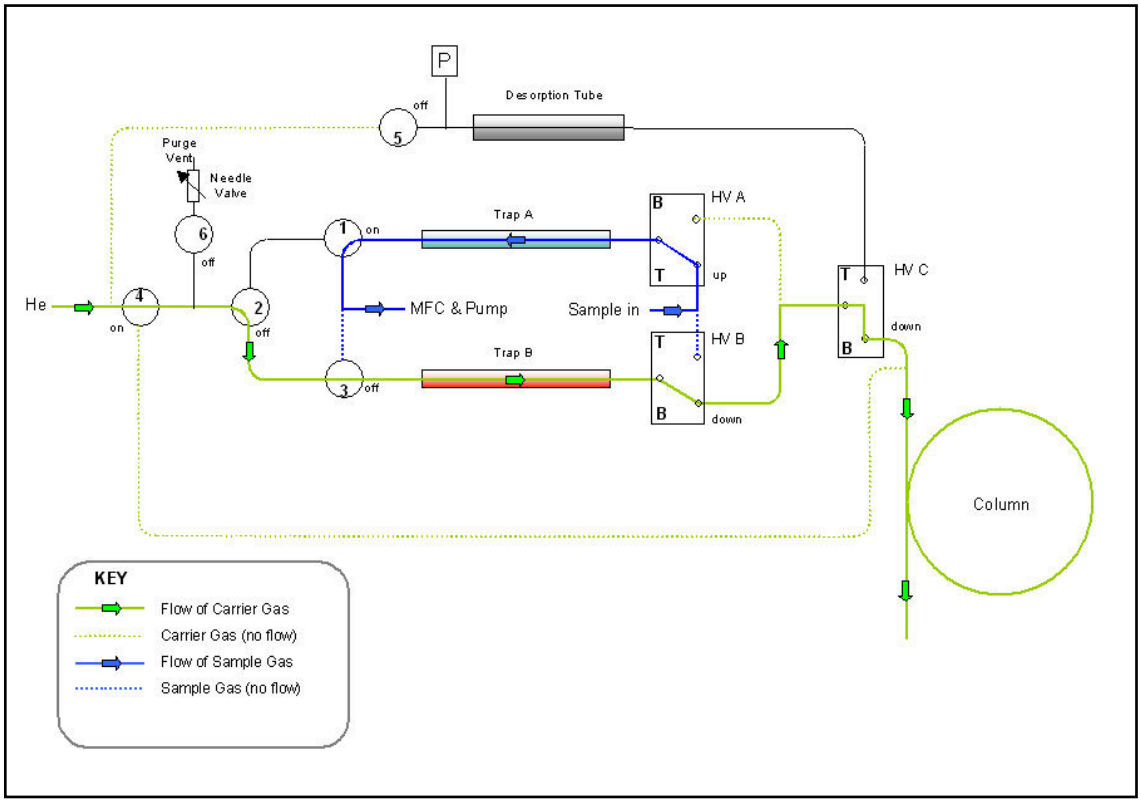

Figure 22. TT24-7e2 - Continuous sampling - trap A sampling, trap B desorbing

The first two runs of the continuous sampling mode (i.e. trap A and trap B) would typically not be used for quantitative analysis of the sampled air but as a clean up process for both traps. After both traps have been fired once then the subsequent samples may be quantified.

After purging the trap (B) for the first time, trap fire occurs and flow is directed (backflushed) across to the capillary column. This first (B) trap fire prepares the trap for subsequent sampling. The flow rate through the trap is equal to the column flow rate and **should be at least 3 mL/min.**

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2.5.2.1.4 Trap A purging, trap B sampling

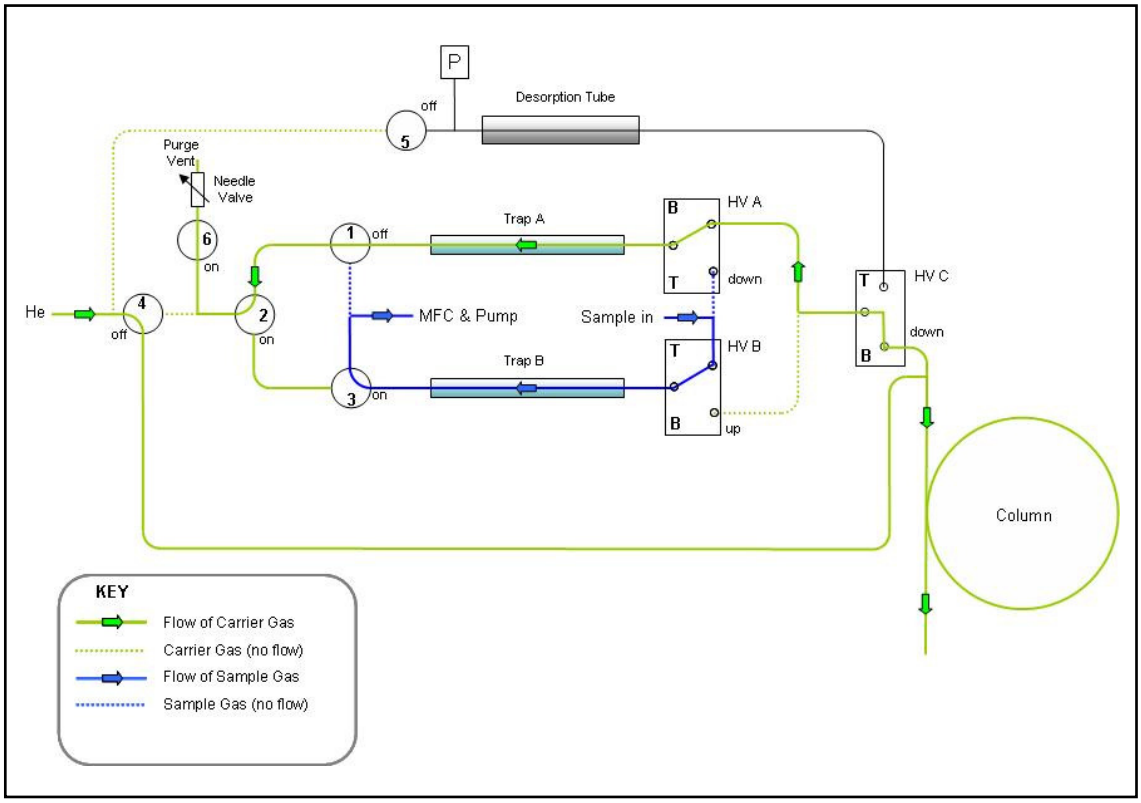

Figure 23. TT24-7e2 - Continuous sampling - trap A purging, trap B sampling

After the sampling time for trap A has completed trap A switches into its dry-purge mode to sweep out air and moisture prior to trap fire. Having cooled to its starting temperature after the first clean up trap fire, trap B now goes into its sampling phase. The date and time that the sampling started, and the trap that is being sampled are reported as an information message in the reporting module of the software (see section 2.9.2.7). e.g.

```
05/12/2005 10:49:31    [ INFO] Sampling Started on Trap B
```


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2.5.2.1.5 Trap A desorbing, trap B sampling

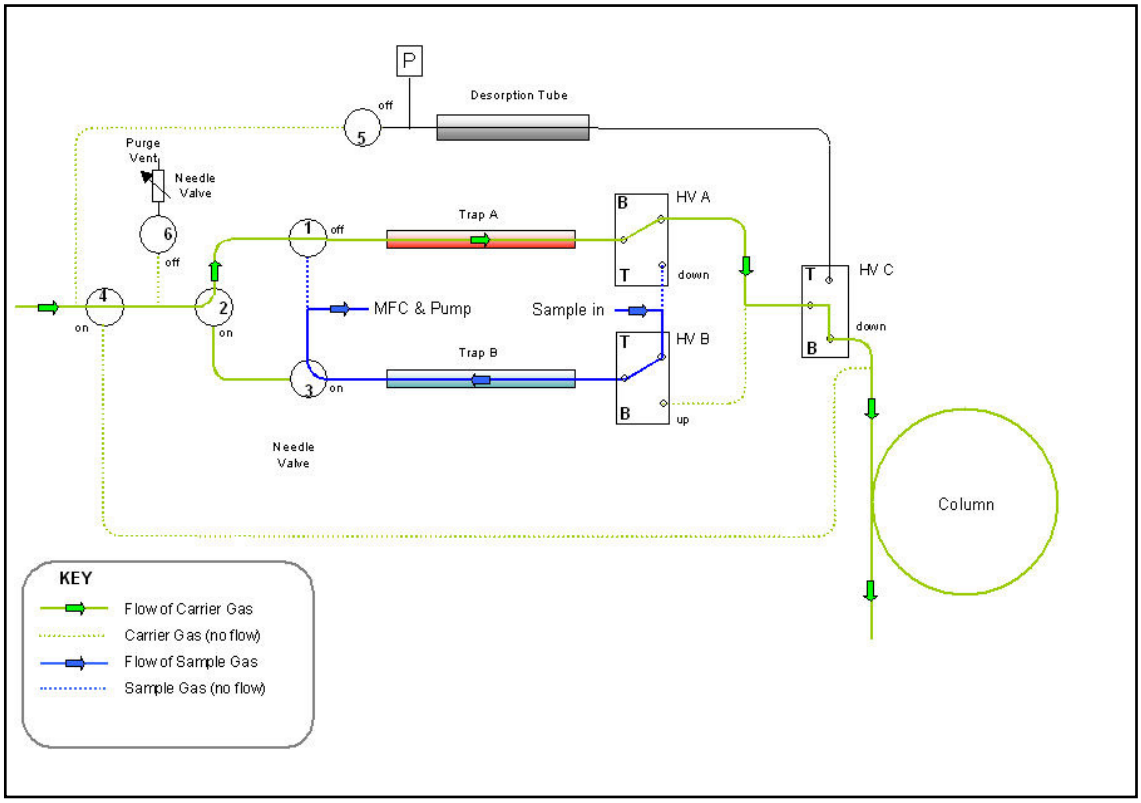

Figure 24. TT24-7e2 - Continuous sampling - trap A desorbing, trap B sampling

Trap A now fires. If this is the first run for A this run is treated as a clean up run, however if this is a subsequent run then real sample will be transferred across to the capillary column for analysis.

To see how the time overlap of sampling, dry-purging, trap fire and cooling occurs relative to the GC oven cycle time please refer to section 2.3.1.

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2.5.2.2 Tube desorb mode (TT24-7e2)

2.5.2.2.1 Standby

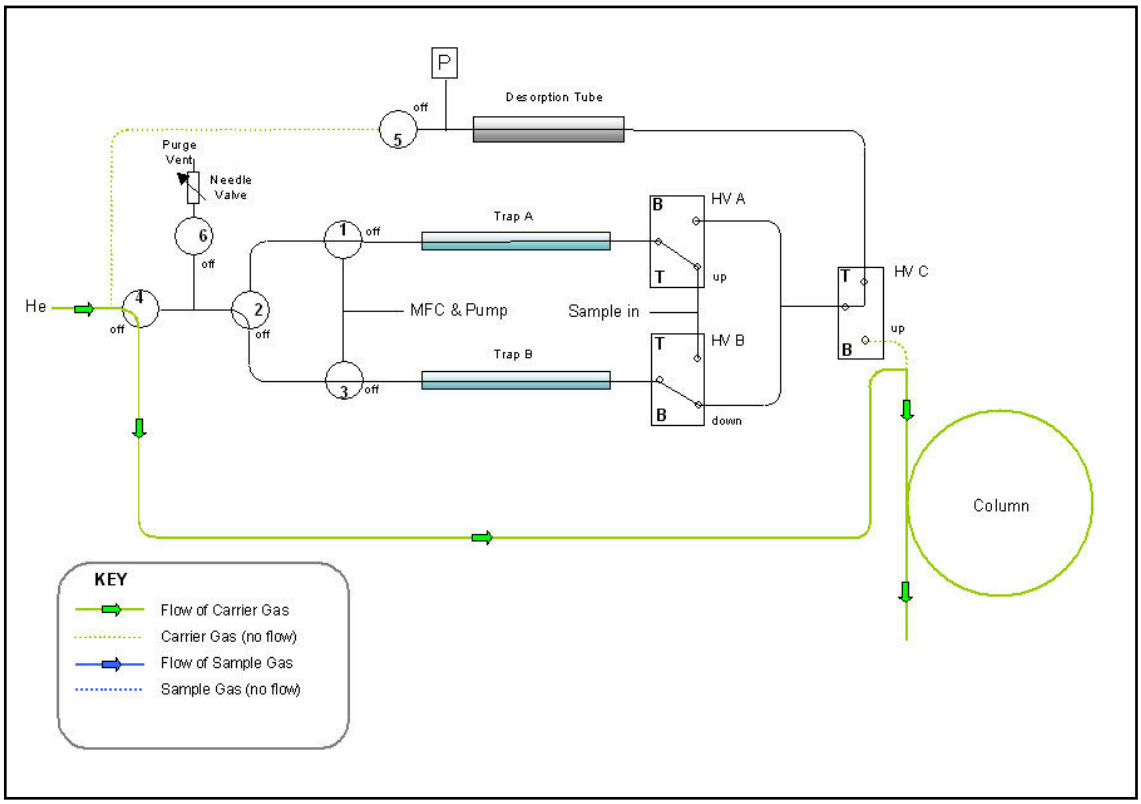

Figure 25. TT24-7e2 - Tube desorb mode - standby

In this non-sampling mode, carrier gas alone is continuously supplied to the capillary column. No other gas flow occurs within the TT24-7 unit during this time.

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2.5.2.2.2 Leak testing tube

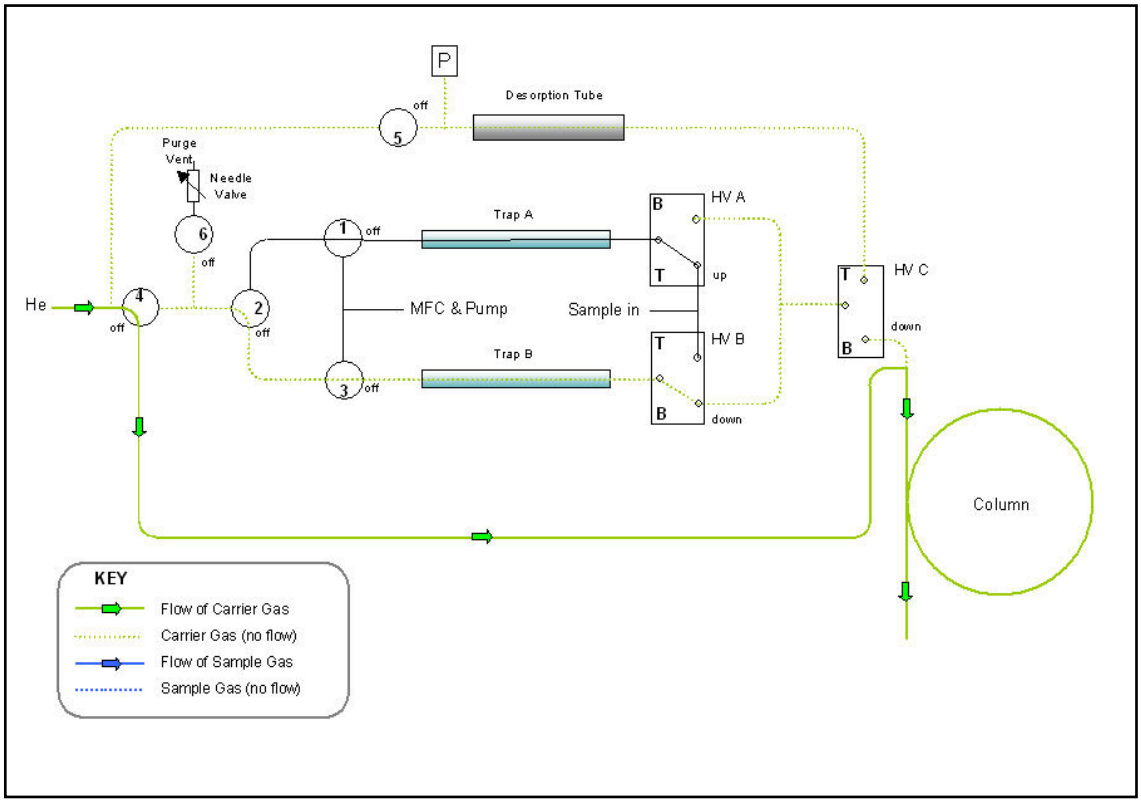

Figure 26. TT24-7e2 - Tube desorb mode - leak testing tube

The first stage in tube desorption is a tube leak test. This is essential as the tube has been inserted into the TT24-7 flow path and sealed with two retaining o-rings one at the front and back of the tube oven. See section 3.5 for tube installation instructions.

During this phase the tube desorption flow lines in the TT24-7 are initially pressurised with carrier gas and then solenoid SV5 is switched OFF to seal the system. The heated valves are configured so that there is no connection beyond HVC. The pressure sensor P adjacent to SV5 is monitored to measure the rate of pressure decay. If this is acceptable (< 5% drop in pressure within 30 seconds) the software moves on to the next phase in the tube desorption process.

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2.5.2.2.3 Leak testing trap A

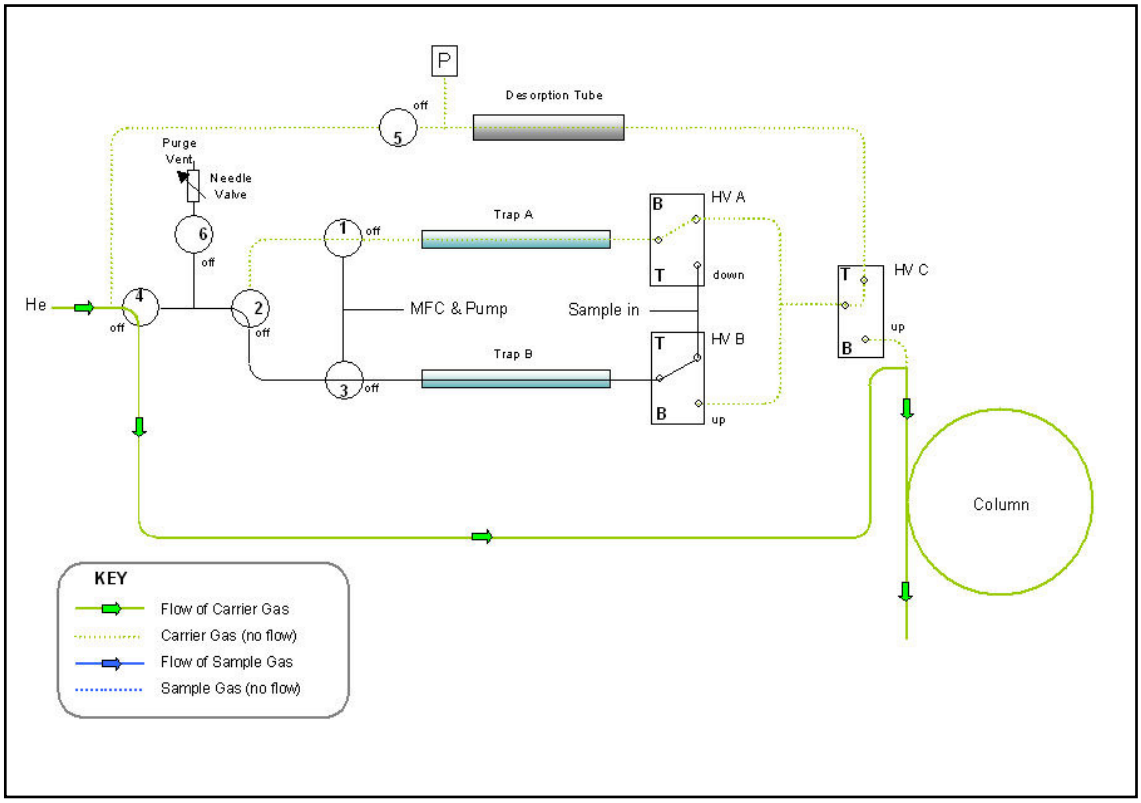

Figure 27. TT24-7e2 - Tube Desorb Mode - Leak testing trap A

After the tube leak test the selected trap also undergoes a further leak test. In the example above trap A is to be used. The heated valve HVC now switches to allow pressurisation (via SV5) of the line right through trap A, SV1 being switched OFF. Once again the pressure sensor next to SV5 is monitored for pressure decay.

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2.5.2.2.4 Tube purge

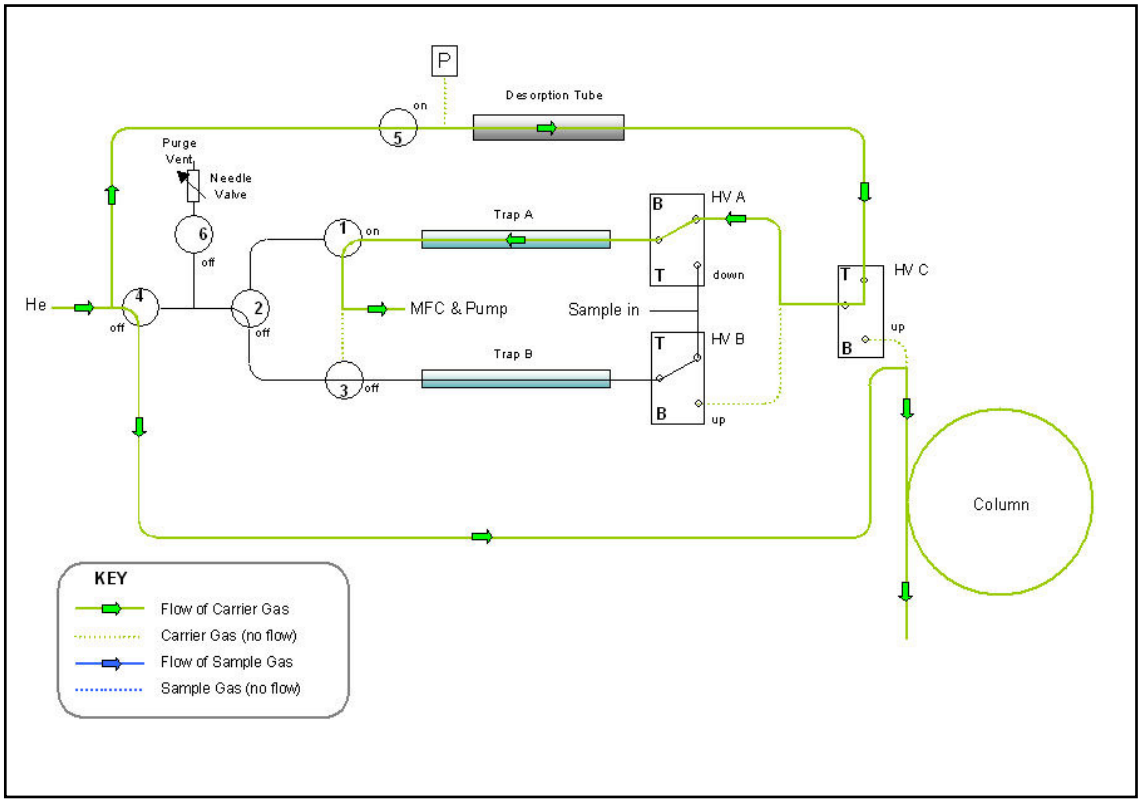

Figure 28. TT24-7e2 - Tube desorb mode - tube purge

Prior to the tube desorption (heating) phase, the air and moisture which will be present in the tube needs to be flushed out to preserve the integrity of the sorbent material. Carrier gas purges through the tube for a default time of 30 seconds at the tube desorb flow rate and residual air / moisture is then flushed onto the chosen trap.

The majority of the air will pass directly through the trap as it is unretained, however the trap is also dry-purged in the next phase (section 2.5.2.2.6) to remove any residual air and, depending on the sorbent phase, moisture as well.

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2.5.2.2.5 Tube desorption

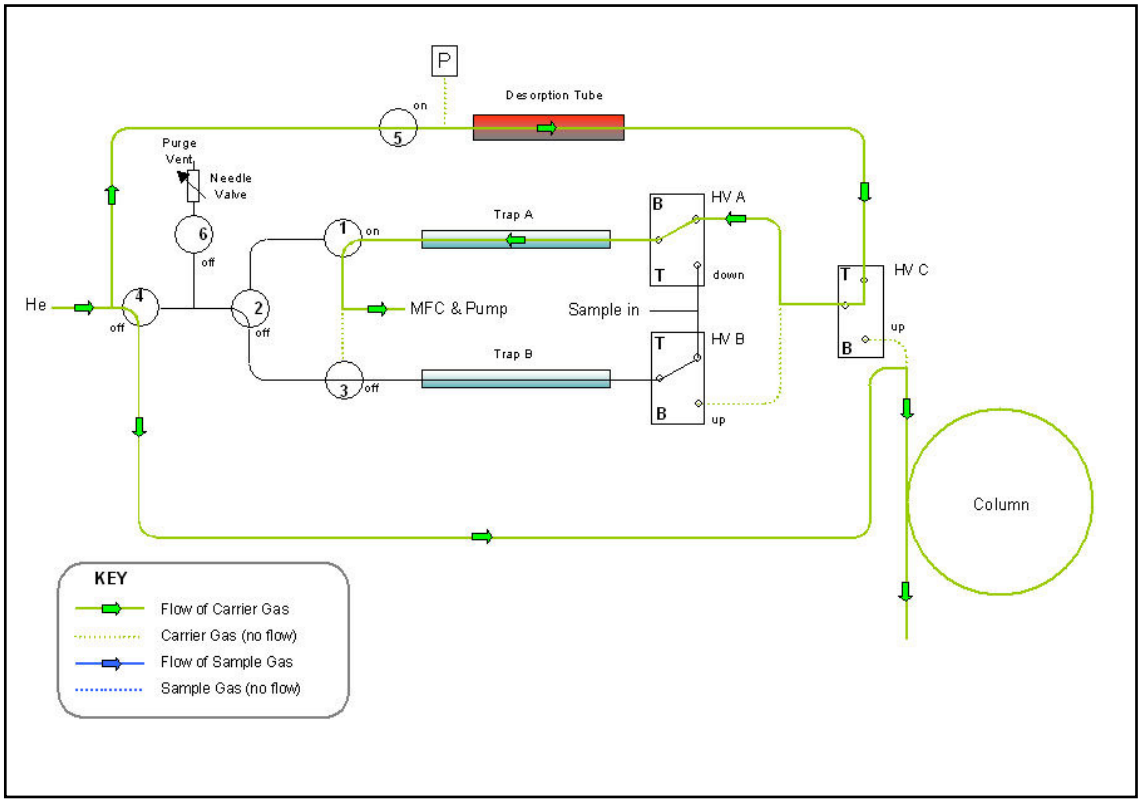

Figure 29. TT24-7e2 - Tube desorb mode - tube desorption

The tube is now heated to the temperature set as the 'Desorb Temp' within the TT24-7 method. The tube 'Desorb Flow' and 'Desorb Time' parameters control the desorption process. The set 'Desorb Temp' value will depend on the sorbent material and should not be set higher than that recommended for the sorbent(s) in the tube. See Appendix 3 for further details on sorbent selection and maximum recommended temperatures.

Sufficient flow and time should be maintained to completely desorb all analytes from the tube onto the specified trap A or B.

In the schematic above A is the chosen trap.

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2.5.2.2.6 Trap (A) purge

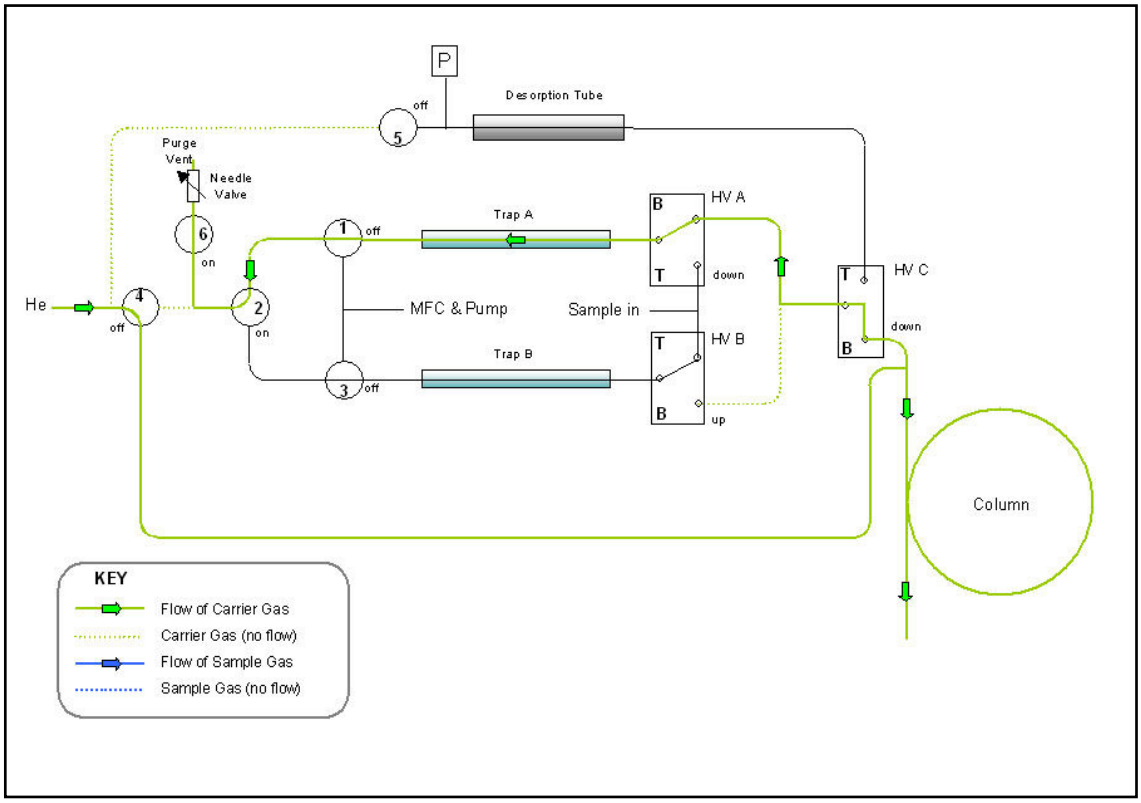

Figure 30. TT24-7e2 - Tube desorb mode - trap (A) purge

This is the dry-purge phase prior to trap fire, to remove any residual air and moisture coming from the tube. The flow is controlled by the needle valve attached to SV6.

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2.5.2.2.7 Trap (A) desorption

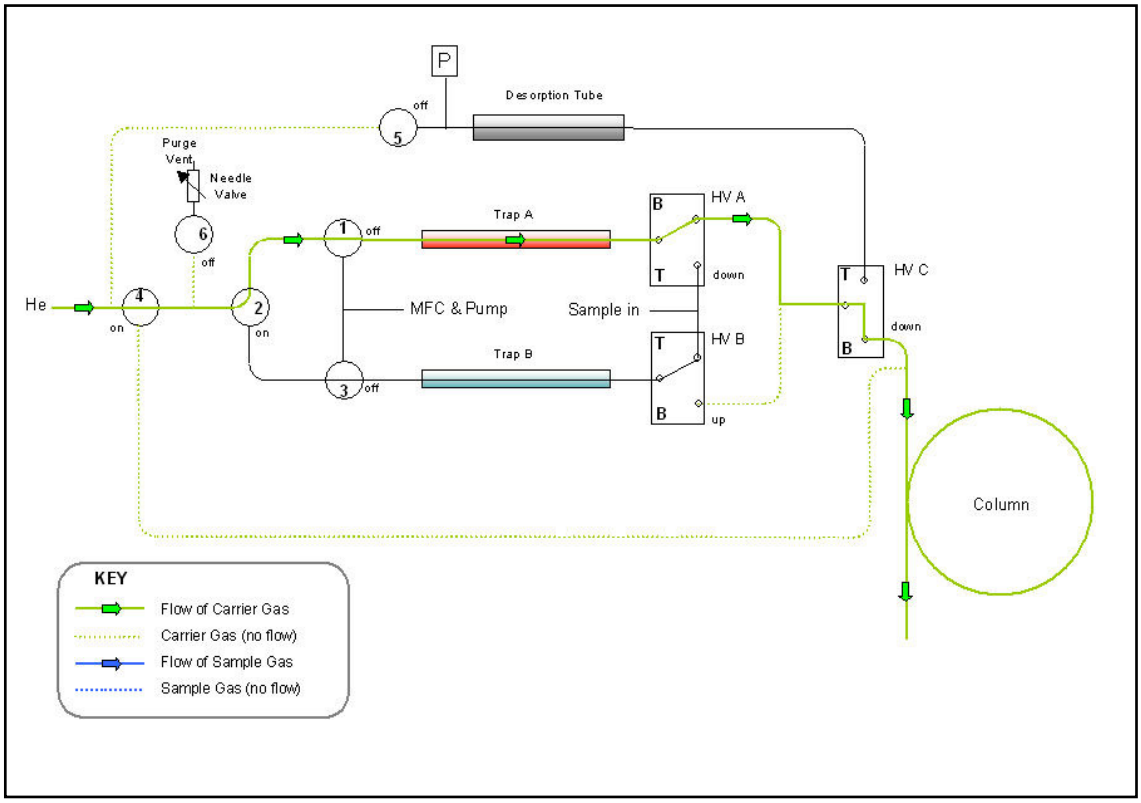

Figure 31. TT24-7e2 - Tube desorb mode - trap desorption

The selected trap is now rapidly heated and backflushed with carrier gas to remove retained analytes from the sorbent bed(s) into the capillary column. The flow rate through the trap is equal to the column flow and **should be at least 3 mL/min**.

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2.5.3 TT24-7e3 hardware configuration

TT24-7e3 includes the option to split sample and may be operated in both sampling modes - continuous sampling and tube desorb.

2.5.3.1 Continuous sampling mode (TT24-7e3)

2.5.3.1.1 Standby

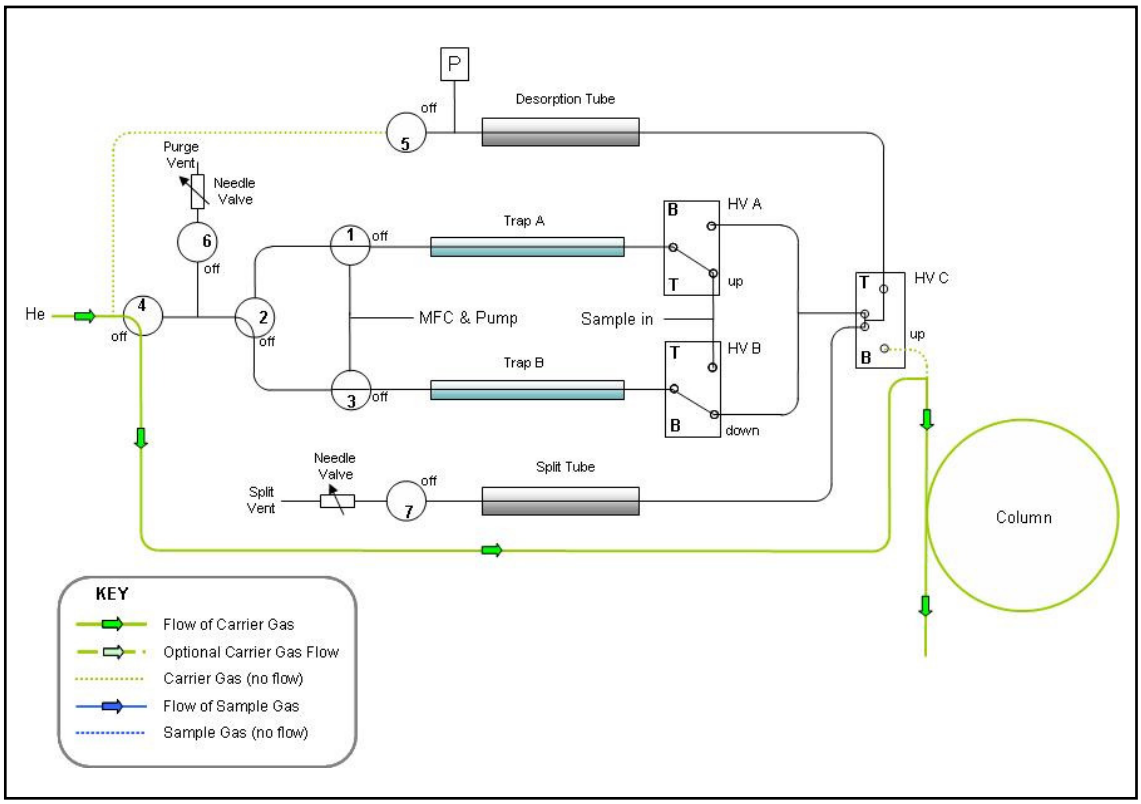

Figure 32. TT24-7e3 - Continuous sampling - standby

In this non-sampling mode, carrier gas alone is continuously supplied to the capillary column. No other gas flow occurs within the TT24-7 unit during this time.

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2.5.3.1.2 Trap A sampling, trap B dry-purging

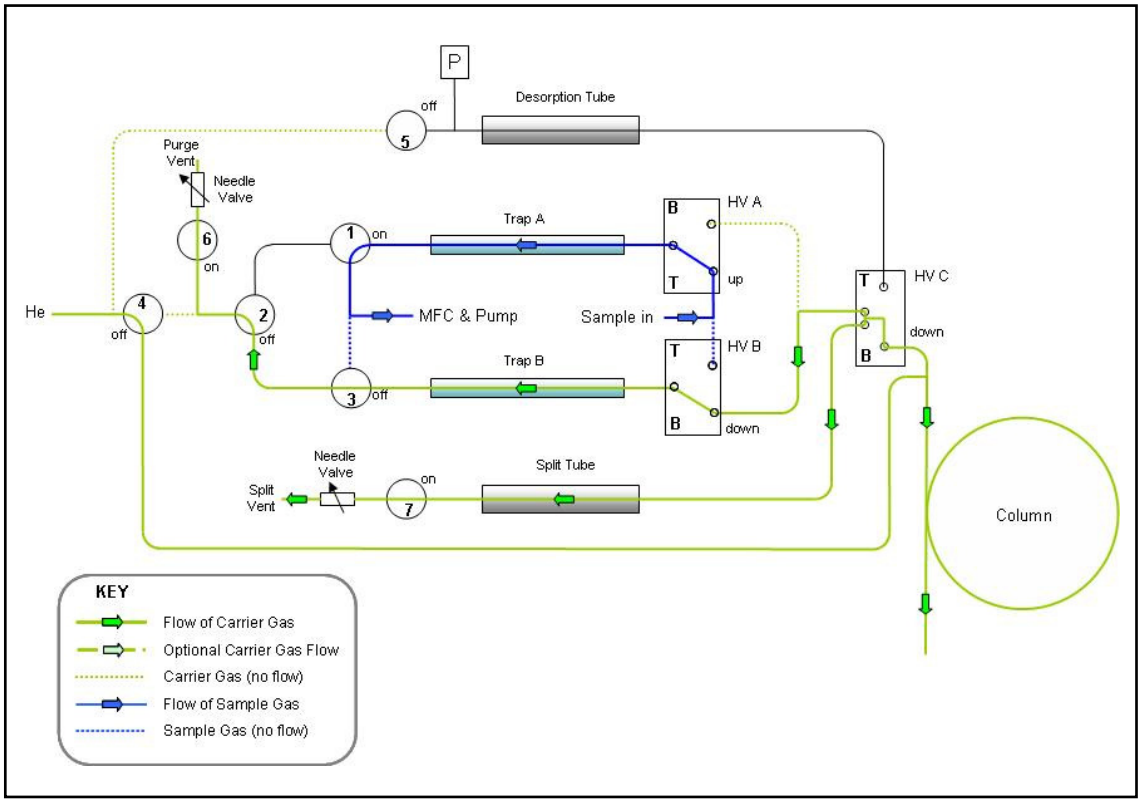

Figure 33. TT24-7e3 - Continuous sampling - trap A sampling, trap B dry-purging

By starting the TT24-7 software the continuous sampling process commences. The sample passes initially onto trap A. The date and time that the sampling started, and the trap that is being sampled are reported as an information message in the reporting module of the software (see section 2.9.2.7). e.g.

```
05/12/2005 10:49:31    [ INFO] Sampling Started on Trap A
```

At the same time trap B dry-purges with carrier gas in the sampling direction. This removes air and moisture from the trap prior to firing. The dry-purge flow rate through the trap (B) at this time is controlled by a needle valve connected to SV6.

Carrier gas also purges down the split line to purge any air from the line and to prevent ingress into the system.

The sample gas flow rate passing into the TT24-7 is controlled by the internal mass flow controller (MFC).

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2.5.3.1.3 Trap A sampling, trap B desorbing

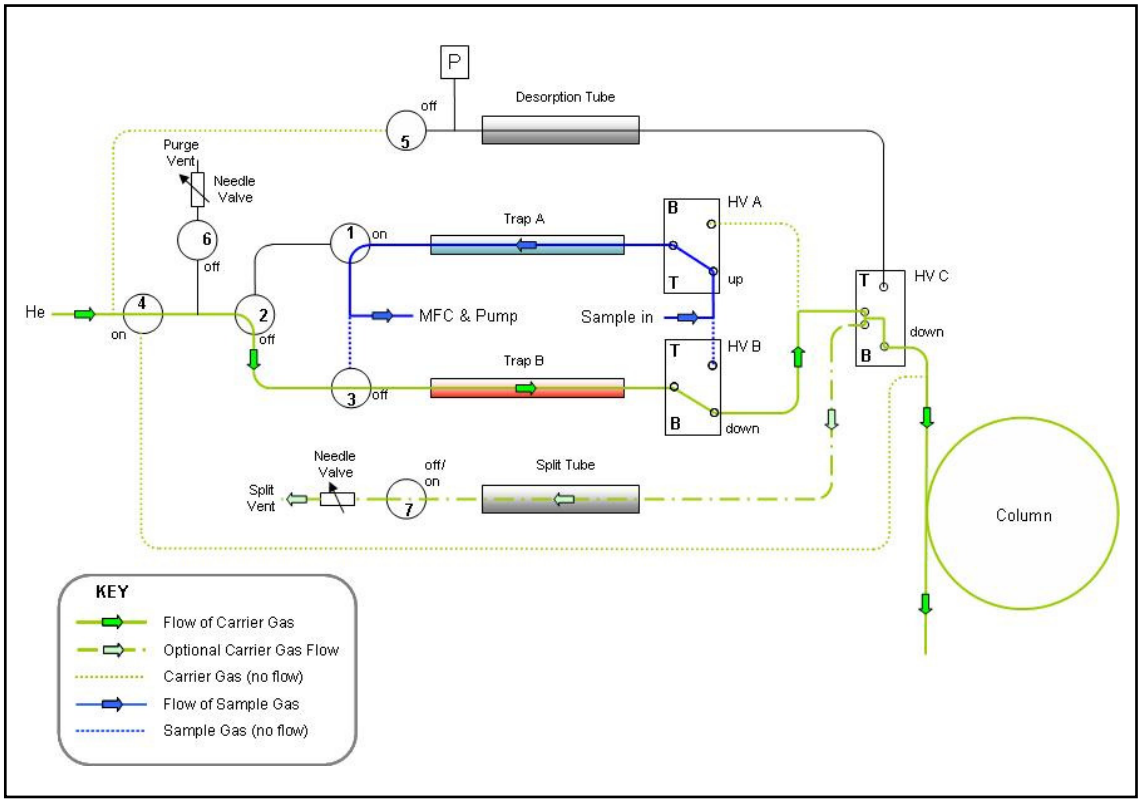

Figure 34. TT24-7e3 - Continuous sampling - trap A sampling, trap B desorbing

The first two runs of the continuous sampling mode (i.e. trap A and trap B) would typically not be used for quantitative analysis of the sampled air but as a clean up process for both traps. After both traps have been fired once then the subsequent samples may be quantified.

After purging the trap (B) for the first time, trap fire occurs and flow is directed (backflushed) across to the capillary column. This first (B) trap fire prepares the trap for subsequent sampling.

If the split function is enabled in the method - see section 2.9.2.1.14), then this will occur at trap fire (Trap B Desorbing). The flow rate through the trap is therefore equal to the column flow plus the split flow through SV7. The split ratio is therefore:

$$\text{Split Ratio} = \frac{\text{Column Flow}}{\text{Column flow + Split flow}}$$

If the split function is not enabled then the flow rate through the trap is equal to the column flow rate alone and **should be at least 3 mL/min.**

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2.5.3.1.4 Trap A purging, trap B sampling

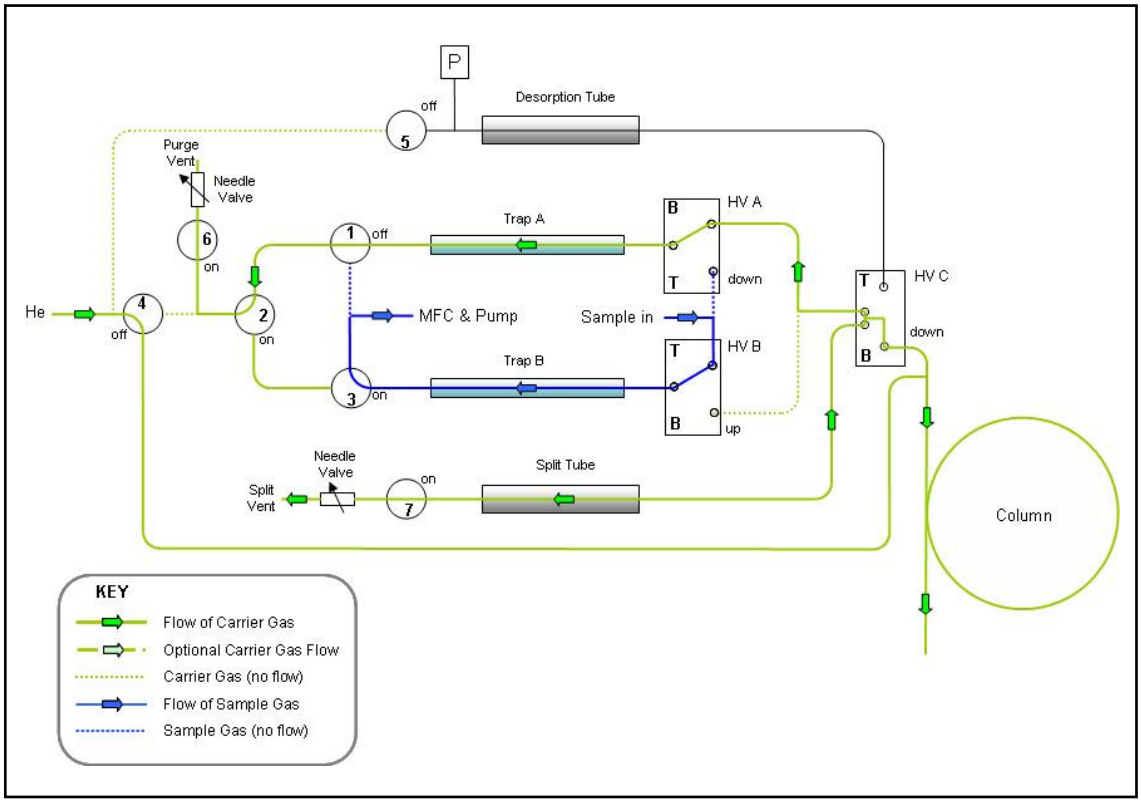

Figure 35. TT24-7e3 - Continuous sampling - trap A dry-purging, trap B sampling

After the sampling time for trap A has completed trap A switches into its dry-purge mode to sweep out air and moisture prior to trap fire. Having cooled to its starting temperature after the first clean up trap fire, trap B now goes into its sampling phase. The date and time that the sampling started, and the trap that is being sampled are reported as an information message in the reporting module of the software (see section 2.9.2.7). e.g.

```
05/12/2005 10:49:31    [ INFO] Sampling Started on Trap A
```

Carrier gas also purges down the split line to purge any air from the line and to prevent ingress into the system.

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2.5.3.1.5 Trap A desorbing, trap B sampling

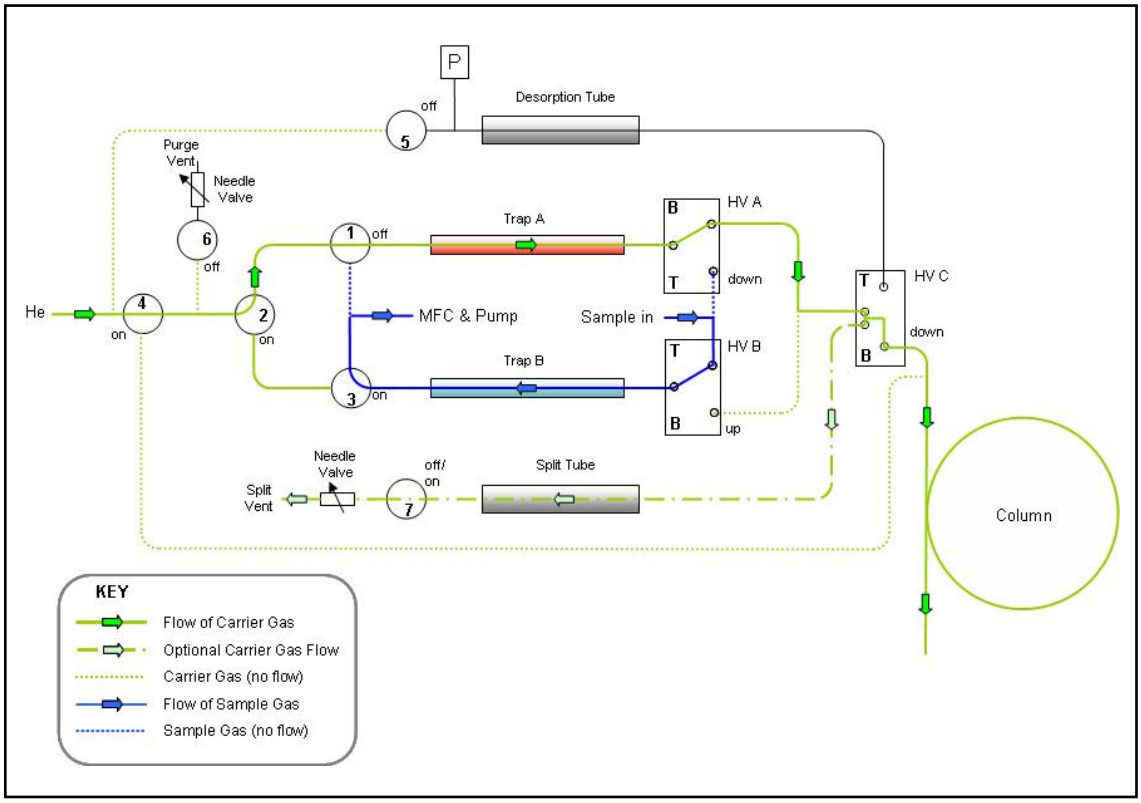

Figure 36. TT24-7e3 - Continuous sampling - trap A desorbing, trap B sampling

Trap A now fires. If this is the first run for A this run is treated as a clean up run, however if this is a subsequent run then real sample will be transferred across to the capillary column for analysis.

If the split function is enabled in the method - see section 2.9.2.1.14 then this will occur at trap fire (Trap A Desorbing). The flow rate through the trap is therefore equal to the column flow plus the split flow through SV7. The split ratio is therefore:

$$\text{Split Ratio} = \frac{\text{Column Flow}}{\text{Column flow + Split flow}}$$

If the split function is not enabled then the flow rate through the trap is equal to the column flow rate alone and **should be at least 3 mL/min.**

To see how the time overlap of sampling, dry-purging, trap fire and cooling occurs relative to the GC oven cycle time please refer to section 2.3.1.

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2.5.3.2 Tube desorb mode (TT24-7e3)

2.5.3.2.1 Standby

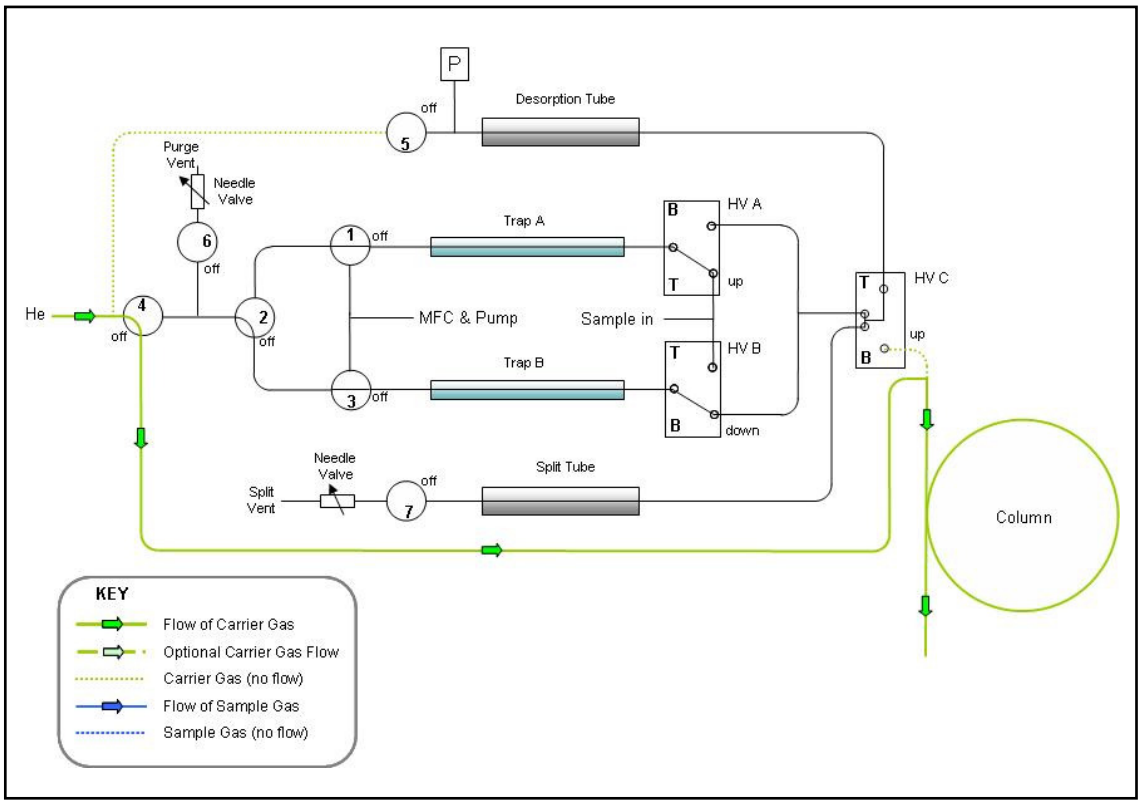

Figure 37. TT24-7e3 - Tube desorb mode - standby

In this non-sampling mode, carrier gas alone is continuously supplied to the capillary column. No other gas flow occurs within the TT24-7 unit during this time.

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2.5.3.2.2 Leak testing tube

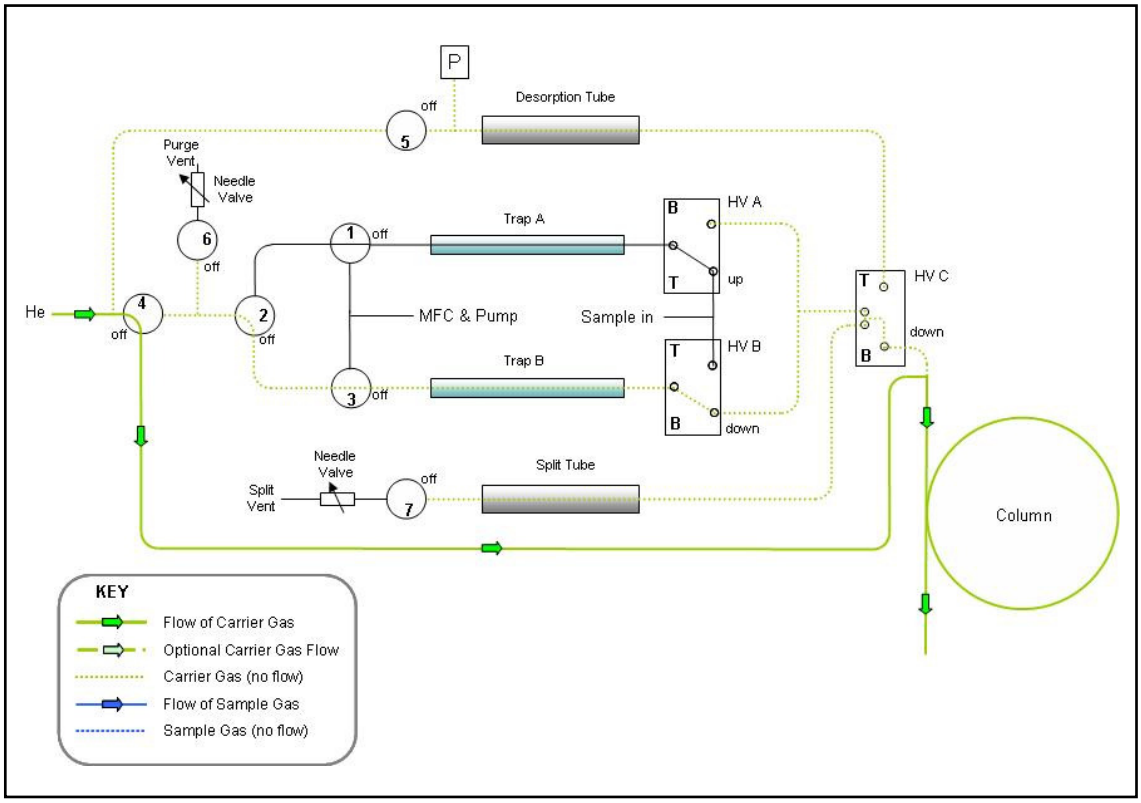

Figure 38. TT24-7e3 - Tube desorb mode - leak testing tube

The first stage in tube desorption is a tube leak test. This is essential as the tube has been inserted into the TT24-7 flow path and sealed with two retaining o-rings one at the front and back of the tube oven. See section 3.5 for tube installation instructions.

During this phase the tube desorption flow lines in the TT24-7 are initially pressurised with carrier gas and then solenoid SV5 is switched OFF to seal the system. The heated valves are configured so that there is no connection beyond HVC. The pressure sensor P adjacent to SV5 is monitored to measure the rate of pressure decay. If this is acceptable (< 5% drop in pressure within 30 seconds) the software moves on to the next phase in the tube desorption process.

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2.5.3.2.3 Leak testing trap A

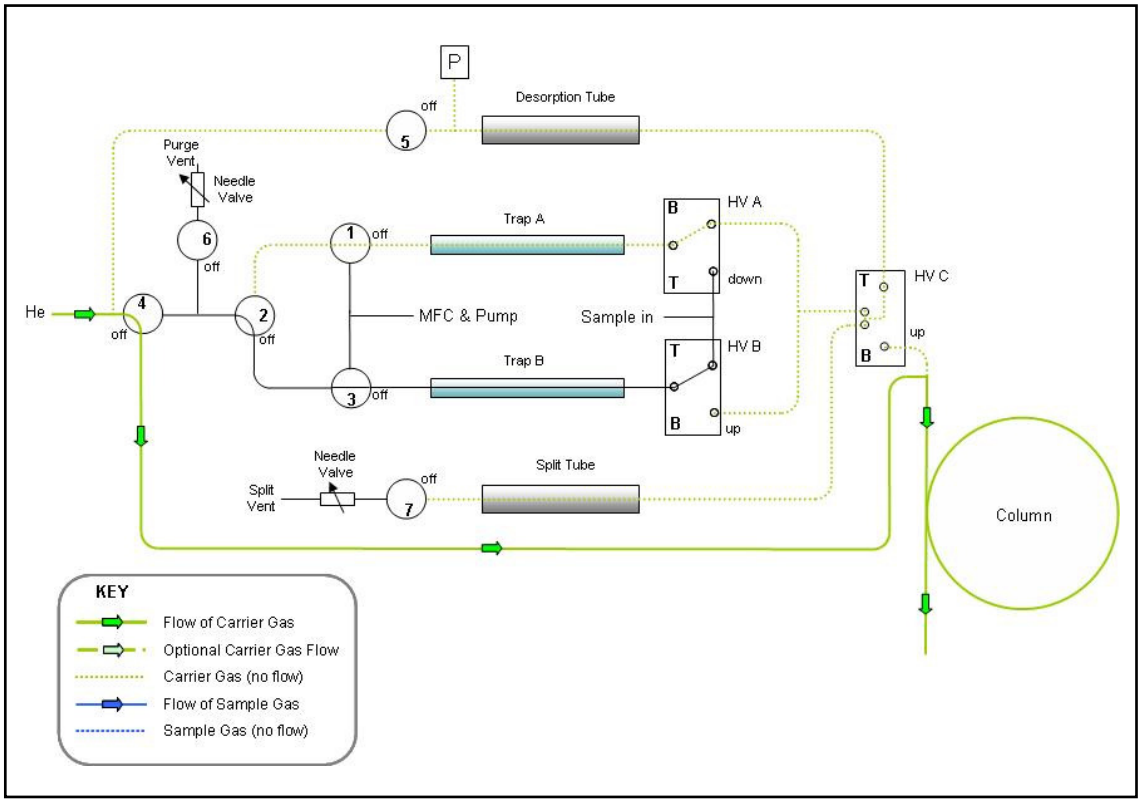

Figure 39. TT24-7e3 - Tube desorb mode - leak testing trap A

After the tube leak test the selected trap also undergoes a further leak test. In the example above trap A is to be used. The heated valve HVC now switches to allow pressurisation (via SV5) of the line right through trap A, SV1 being switched OFF. Once again the pressure sensor next to SV5 is monitored for pressure decay.

The split line pneumatics through to SV7 are also included within this leak test.

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2.5.3.2.4 Tube purge

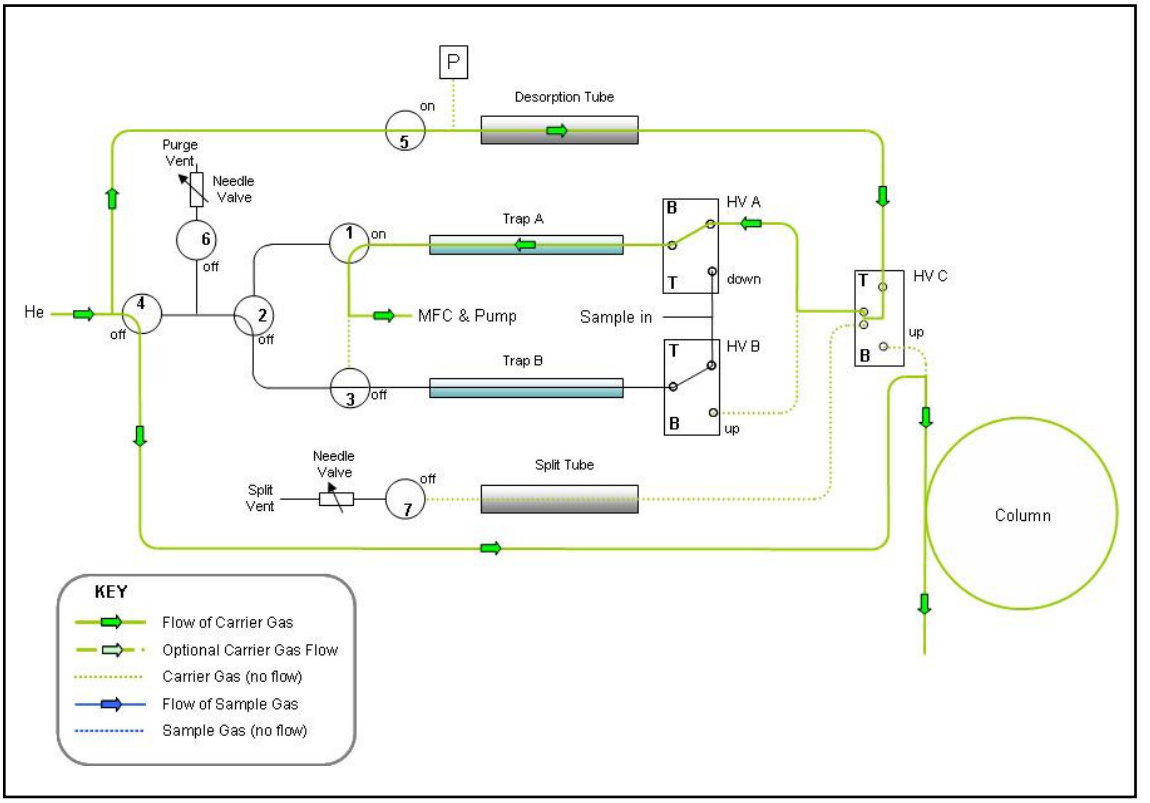

Figure 40. TT24-7e3 - Tube desorb mode - tube purge

Prior to the tube desorption (heating) phase, the air and moisture which will be present in the tube needs to be flushed out to preserve the integrity of the sorbent material. Carrier gas purges through the tube for a default time of 30 seconds at the tube desorb flow rate and residual air / moisture is then flushed onto the chosen trap.

The majority of the air will pass directly through the trap as it is unretained, however the trap is also dry-purged in the next phase (section 2.5.3.2.6) to remove any residual air and, depending on the sorbent phase, moisture as well.

2.5.3.2.5 Tube desorption

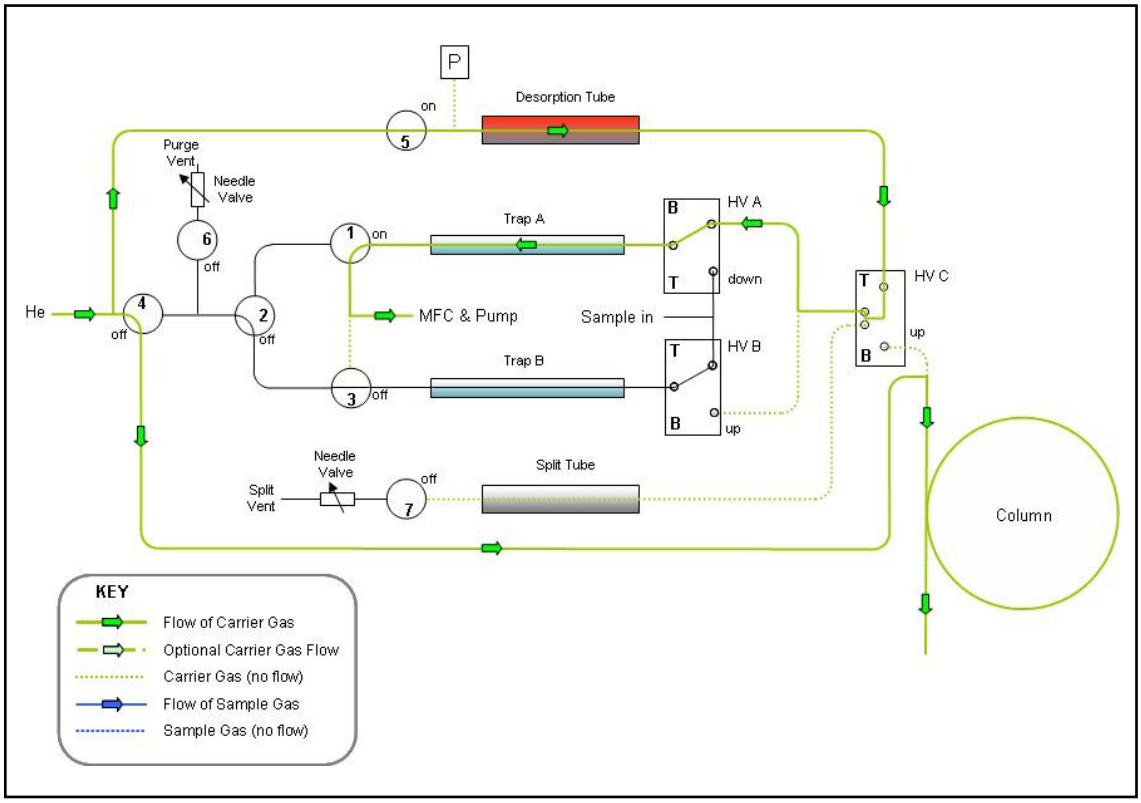

Figure 41. TT24-7e3 - Tube desorb mode - tube desorption

The tube is now heated to the temperature set as the 'Desorb Temp' within the TT24-7 method. The tube 'Desorb Flow' and 'Desorb Time' parameters control the desorption process. The set 'Desorb Temp' value will depend on the sorbent material and should not be set higher than that recommended for the sorbent(s) in the tube. See Appendix 3 for further details on sorbent selection and maximum recommended temperatures.

Sufficient flow and time should be maintained to completely desorb all analytes from the tube onto the specified trap A or B.

In the schematic above A is the chosen trap.

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2.5.3.2.6 Trap (A) purge

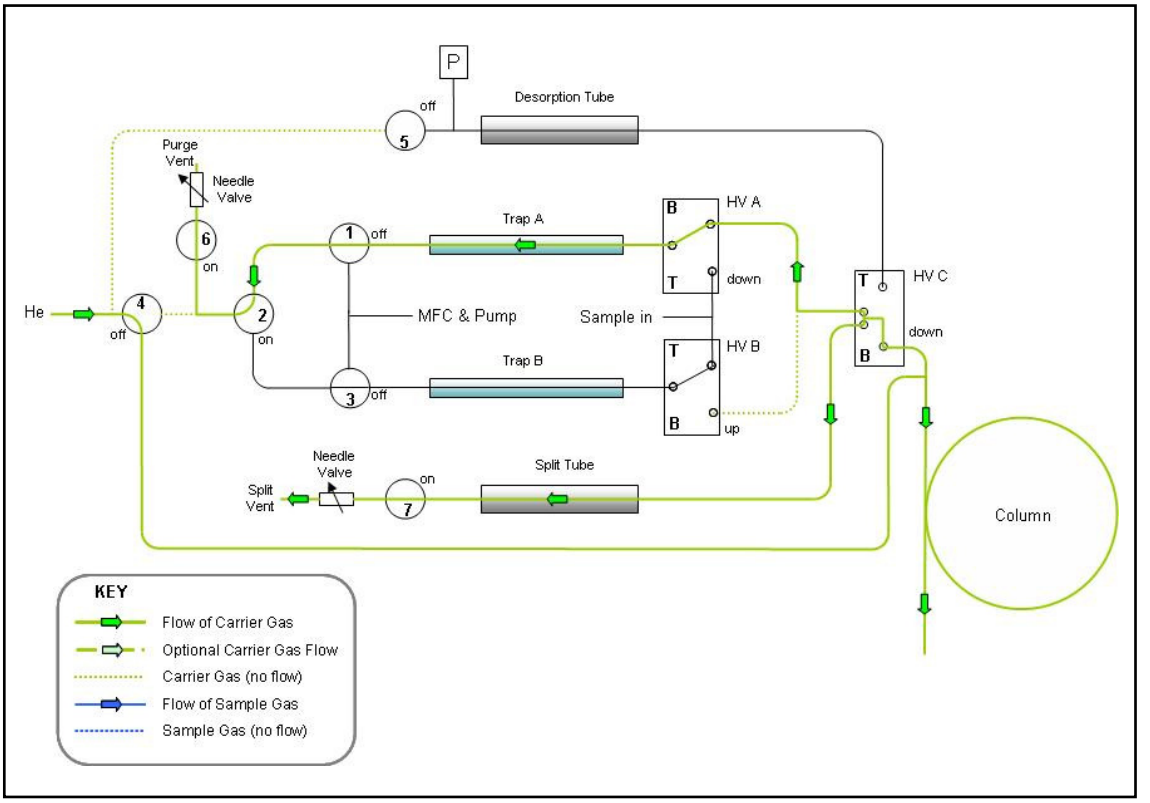

Figure 42. TT24-7e3 - Tube desorb mode - trap (A) purge

This is the dry-purge phase prior to trap fire, to remove any residual air and moisture coming from the tube. The flow is controlled by the needle valve attached to SV6.

The split line pnuematics through SV7 and its associated needle valve are also purged during this operation. The flow through this line is controlled by this SV7 needle valve.

2.5.3.2.7 Trap (A) desorption

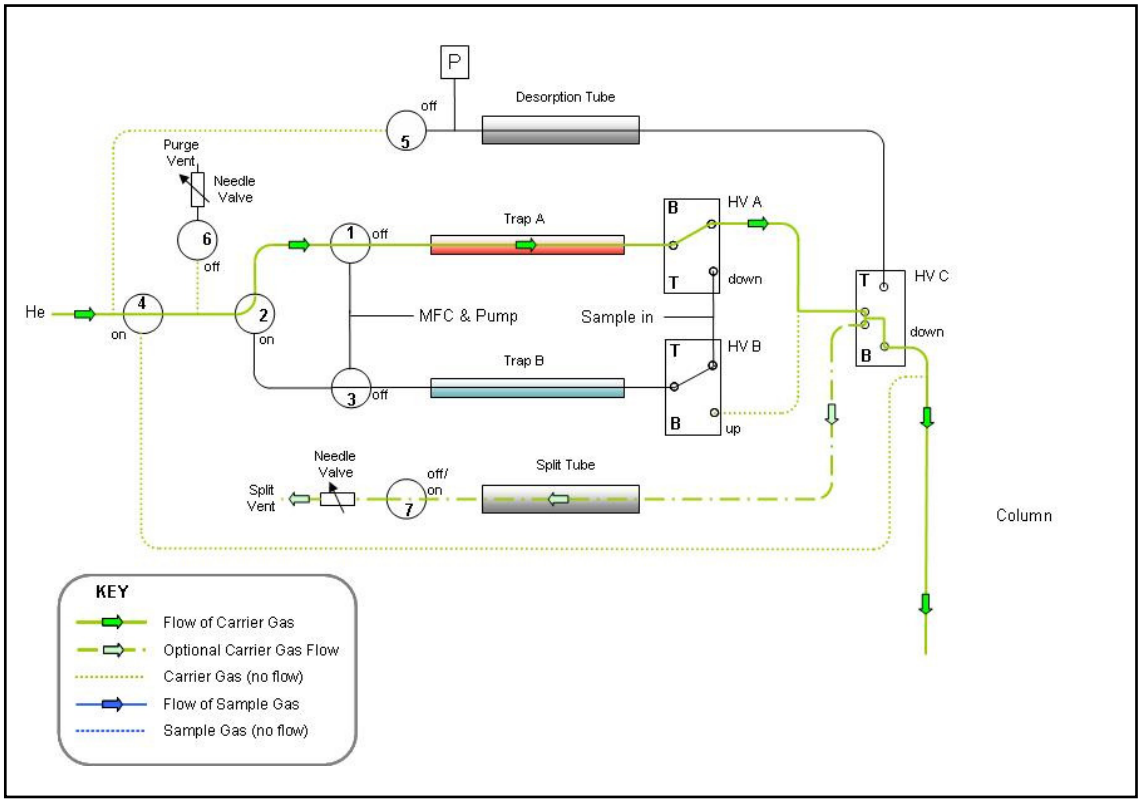

Figure 43. TT24-7e3 - Tube desorb mode - trap desorption

The selected trap is now rapidly heated and backflushed with carrier gas to remove retained analytes from the sorbent bed(s) into the capillary column.

If the split function is enabled in the method - see section 2.9.2.1.14, then this will occur at trap fire. The flow rate through the trap is therefore equal to the column flow plus the split flow through SV7. The split ratio is therefore:

$$\text{Split Ratio} = \frac{\text{Column Flow}}{\text{Column flow + Split flow}}$$

If the split function is not enabled then the flow rate through the trap is equal to the column flow rate alone and **should be at least 3 mL/min.**

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2.6 Software control and configuration

This section reviews the software control and configuration for each of the available TT24-7 hardware configurations. The user interface is discussed in detail.

The TT24-7 software runs as a Windows® application using Windows 98™ onwards, however Windows 2000™ or Windows XP™ is the preferred version. If Windows NT™ is used no USB cable connection is possible as this is not supported by NT.

Ensure that you have one of these versions of Microsoft Windows installed on the PC prior to installation of the software.

The software consists of a top level screen containing a set-point user interface, an active flow schematic, a reporting module and a status bar showing set-point and actual values (figure 44).

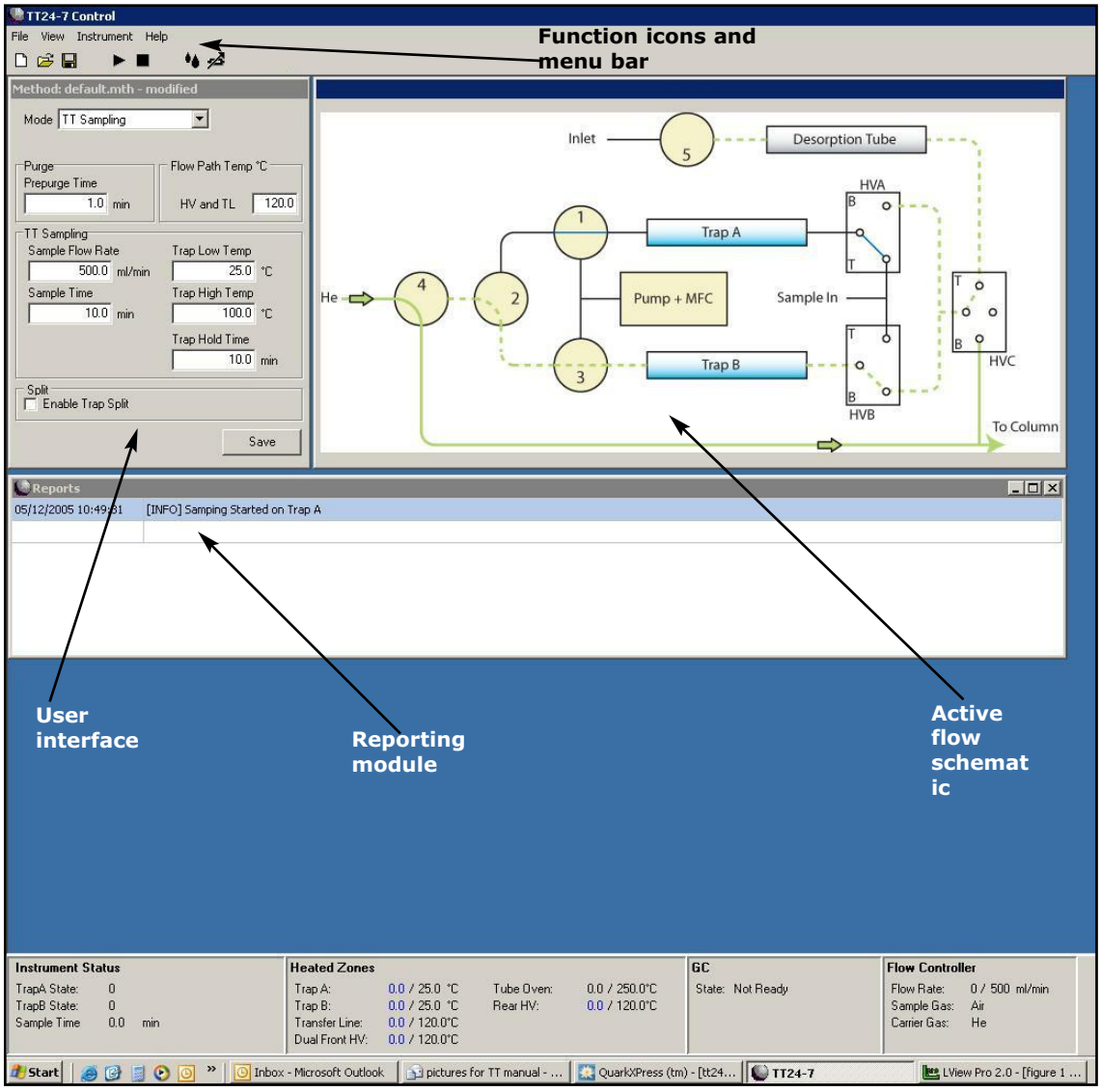

Figure 44. TT24-7 software - top level screen

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At the top of the screen are function icons and a menu bar for instrument configuration and control.

2.7 Software installation

Remove the software CD from its packaging, and insert into the CD drive of the PC.

Access the CD drive from Windows and click on the Setup icon. Follow the on screen instructions to complete the installation of the software.

2.8 Firmware download

After the software has been loaded onto the PC the firmware has to be downloaded into two separate boards within the TT24-7 system.

Firstly ensure that all the electrical connections between the TT24-7, the PC and the rest of the analytical system are in place. See section 3.1 for details.

On the front panel of the TT24-7 there are two LED display lights and an instrument activity switch. The LEDs refer to the status of the Sampler and MFC boards within the TT24-7 and show different colours depending on the download status of the firmware (figure 45).

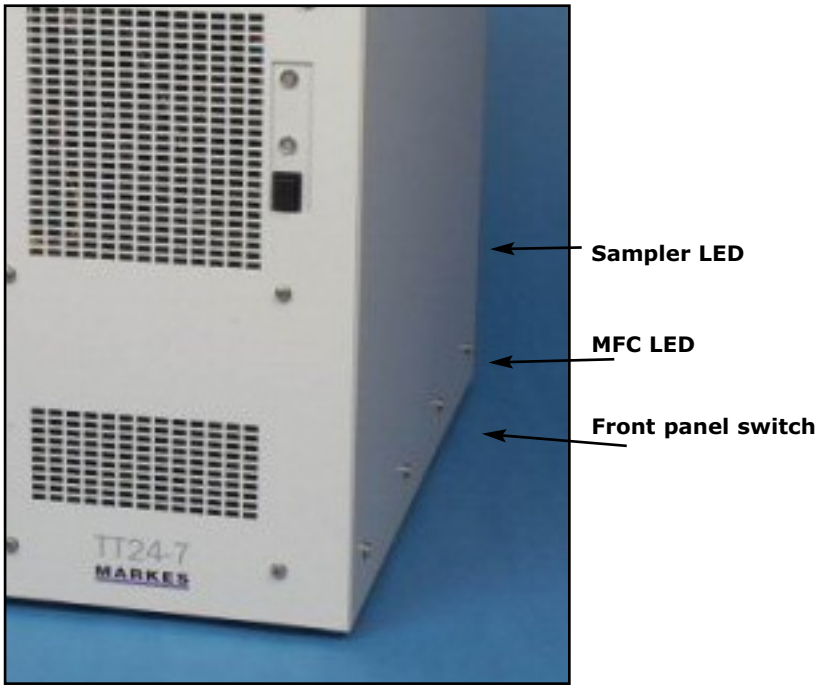

Figure 45. Location of status© LEDs and front panel switch on TT24-7

To download the firmware follow the stepwise procedure below.

Step 1: The TT24-7 should be switched on at the back of the instrument, with the front panel switch off (i.e. not depressed). This should cause the top LED to turn red, while the bottom LED remains OFF.

If however the front panel switch is inadvertently ON (i.e. depressed) at this

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time both LED's will appear red. Correct this by simply turning the front panel switch OFF before continuing.

Step 2: Start the TT24-7 software within the controlling PC. This is achieved by clicking on the system icon.  The software starts by attempting to detect the sampler board (top LED) and if successful will begin downloading the firmware. The top LED will turn amber at this time indicating the download is in progress.

If however the top LED remains red after approximately 10 seconds this indicates the sampler board was not detected. To correct this, turn the main (rear) power switch off, wait 10 seconds and switch on again. The TT24-7 software will continue to look for the sampler board, and assuming there is not a specific problem within the instrument, the firmware download will resume and the top LED turns amber.

Upon successful completion of the download process the top LED will turn from amber to green.

Step 3: If however the sampler board is still not detected, it may be due to an incorrect COM port designation, and eventually download activity within the software will time out. A dialogue box will then appear as shown in figure 46.

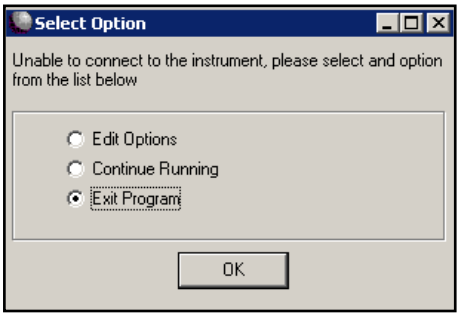

Figure 46. Download activity timeout dialogue box

From this dialogue box select the "Edit Options" configuration, this will launch the instrument options software page, and from this selection of the "Ports" tab enables the COM port settings to be reviewed (figure 47). Ensure that the correct COM port settings are specified. See section 2.9.1.5.2 for further details.

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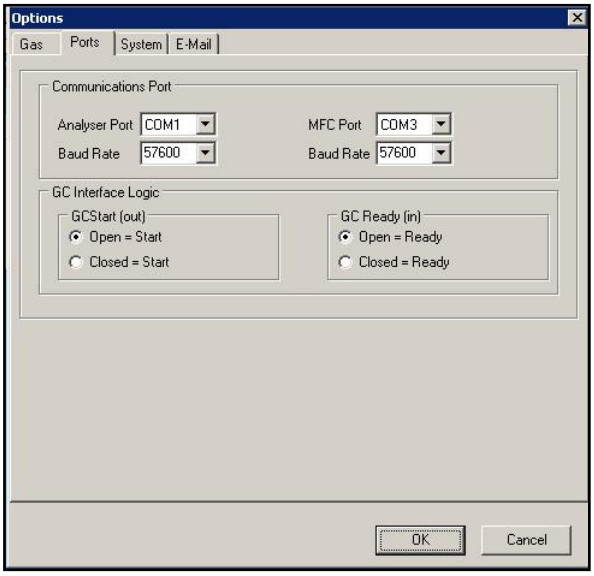

Figure 47. Ports tab in options configuration

With the correct COM port settings specified, return to Step 1 above and repeat the process.

Step 4: The next step is downloading firmware into the mass flow controller board (MFC) which is represented by the lower LED. To achieve this turn the front panel switch On (depressed), at which point the lower LED turns red. The software will automatically attempt to download the firmware into the MFC board and this is indicated by the LED turning amber.

As for the sampler board, if detection of the MFC board is unsuccessful, toggle the front panel switch off (wait 10 seconds) and on, and the software will re-attempt to download the firmware again.

On successful completion the lower LED will turn green.

If the download is still unsuccessful then check the COM settings for the MFC board as specified in Step 3 above.

At this point both LEDs should appear green, and the user interface should be loaded onto the PC display.

2.9 Software control - top level

The top level software screen is shown in figure 44 above.

Four separate windows are shown titled Method, Flow Display and Reports and, at the base of the screen, the Instrument Status display. Above the screen are seven graphical icons and a menu bar.

The software control and user interface will vary according to the hardware configuration installed. Ensure that you know which configuration you have prior to reading this manual (see Introduction).

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2.9.1 User interface for TT24-7e1 hardware configurations

Figure 48 shows the user interface for the TT24-7e1 hardware configuration. Only one sampling mode is available in this configuration - TT Sampling.

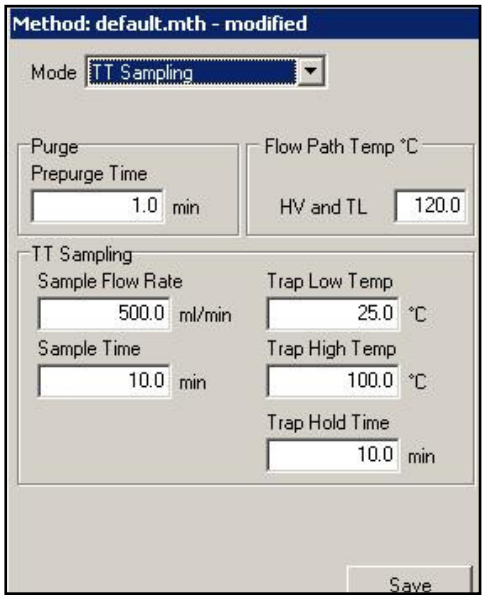

Figure 48. TT24-7e1 - User interface

2.9.1.1 Method parameters

At the top of the method window is shown the active method name (e.g. default.mth) and its status i.e. whether or not it has been modified from its original set-point values.

The set point values used in the interface are described below.

2.9.1.1.1 Mode

The sampling mode cannot be changed in the software - it is set to continuous sampling (TT Sampling), as this is the only mode of operation.

2.9.1.1.2 Prepurge time

This is the pre-purge time when carrier gas is passed through the trap just prior to trap fire. This purge (in the desorption direction) is to purge air / moisture from the trap after sampling and the flow is equivalent to the column flow (which should be at least 3 mL/min). The pre-purge can be set between 0 and 99.9 minutes in increments of 0.1 minute and is typically 0.5 to 1 minute, although this can be reduced where the cycle time of the TT24-7 system needs to be kept to a minimum.

2.9.1.1.3 Flow path temp

This is the set point temperature for the heated valves and the transfer line to the GC. The temperature range extends from ~100°C to 200°C. The temperature should be set high enough to prevent any condensation of sample within the valve / transfer line and values less than 120°C are rarely used. However this temperature value will also affect the lower trap temperature value.

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If the flow path temperature is set to its maximum of 200°C then the trap temperature cannot be controlled at values less than 25°C. If the flow path temperature is set to 150°C then a minimum trap temperature of ~15°C is possible.

2.9.1.1.4 Sample flow rate

This specifies the flow rate of sample vapour passing through the traps. The sample may be at atmospheric pressure, in which case a vacuum pump is required to pull sample into the system. The flow rate is then regulated by the internal mass flow controller (MFC). If the sample is at positive pressure (Max 50 psi), then the vacuum pump is not required, and again the MFC controls the flow through the trap.

Sample flow rates from 50 mL/min to 1 L/min are possible, with 0.1 mL increments, however the maximum flow rate is affected by the sorbent bed depth and mesh size. Where fine mesh size material is used (i.e. 80/100 mesh or finer) the maximum flow rate will be reduced. Ultimately the fastest flow rate achievable is determined by specifying the maximum flow rate of 1 L/min and observing the actual flow value on the status bar (see section 2.9.1.3) at the bottom right side of the user interface.

In certain applications where the analyte concentration is very low (i.e. sub ppb) and there is a time restriction on sampling (i.e. NRT) there is a temptation to set a very fast sampling flow rate to maximise the amount of analyte passing into the trap in unit time. For example sampling at 1 L/min for 10 minutes results in a sampling volume of 10L. If the analyte concentration is very low e.g. in the pg/L concentration (i.e. ppt), then a 10L volume would contain sufficient sample in theory for detection by selective GC detectors or by GCMS.

However sensitivity is very dependant on the signal to noise (S/N) ratio of the resultant chromatographic peak. If this is sharp i.e. peak widths <= 5 seconds then much better integration and detection is possible than for peak widths >=15 seconds.

In practice it has been shown that very fast sampling flow rates (>= 800 mL/min) with certain sorbent materials produces poor or broad peak shapes and this can compromise the minimum detection limit (MDL) for that compound. If the same compound is run at a lower flow i.e. 400 mL/min as opposed to 800 mL/min, this can result in a much sharper peak and a significantly better S/N ratio even though the amount of analyte trapped is half.

The science behind this effect is based on the depth the analyte passes into the sorbent, and as a consequence how easily it comes off at trap fire. It is therefore both compound and sorbent dependant.

2.9.1.1.5 Sample time

This specifies the time interval for sample to pass into the trap. This parameter is directly associated with the sampling flow rate as discussed above to determine the total amount of sample transferred. The range for sample time is from 0.1 to 99.9 minutes, however, values greater than 1 minute will typically be used.

The sampling time and therefore the sampling volume can be affected by the

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ready / not ready status of the GC system. At the completion of the defined set-point time value and prior to the system going into the pre-purge phase of the trap, the TT24-7 monitors the GC ready status. If the GC is ready then the pre-purge process commences. If however the GC is not ready then the sampling time is extended until the GC becomes ready, or a specified "time out" value (see section 2.9.1.5.3) is equalled at which point the system stops. The GC becoming not ready is typically due to the oven not cooling down as fast as normal and this affects the GC cycle time.

The sampling time can be fixed to the specified value i.e. preventing extended sampling within the TT24-7 software. Under these conditions if the GC is not ready then after the fixed sampling time the system then waits until the GC becomes ready or again the time out value stops the process. See section 2.9.1.5.3 for extended sampling and fixed time sampling.

2.9.1.1.6 Trap low temperature

This is the (lower) trap temperature value used to retain compounds on the sorbent bed. Values ranging from 15 to 50°C are possible. Sub ambient values are possible as a consequence of the Peltier cooling elements situated immediately below each trap.

The trap minimum value is sensitive to the flow path temperature described above, the flow rate of sample passing through the trap and the temperature of the air sample. For example if the flow path temperature is set to 200°C, with a fast flow rate (i.e. >= 700 mL/min), then the trap minimum value may be 25°C or higher at faster flows. If additionally the sample gas is above ambient then this minimum value will be higher again.

Using lower sampling flows will assist this value, and can indeed improve on signal to noise for the resulting chromatographic peak (See section 2.9.1.1.5 above).

Ultimately the minimum trap temperature achievable is determined experimentally by reviewing the relationship between the set-point and actual values as shown in the status bar (section 2.9.1.3) and this will be a function of the flow path temperature and the sampling conditions.

2.9.1.1.7 Trap high temperature

This is the upper temperature setting for the trap. Values ranging from 50°C to 400°C are possible. The upper trap temperature should be set to the optimum value which enables 100% recovery of analytes from the sorbent bed, **but does not exceed the maximum permissible temperature for that sorbent.** See Appendix 3. Keeping the maximum trap temperature as low as possible whilst still enabling 100% analyte recovery will extend trap lifetime.

Note: If you exceed the maximum sorbent temperature, the resulting breakdown of the sorbent may severely contaminate the flow path of your system. This may require the complete replacement of the flow path and associated components by a fully qualified service engineer and would not be covered by the instrument warranty.

When multi-bed traps are being used, the maximum temperature that can be set is that relating to the sorbent with the lowest allowable temperature.

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Consideration must then be taken of the recovery efficiency for analytes from sorbents which go to higher values.

2.9.1.1.8 Trap hold time

This is the time interval when the trap is held at its maximum value. Time intervals from 0.1 to 10 minutes are possible, however values in excess of ~ 0.5 minutes are typically used. This value must be long enough to ensure complete removal of analytes from the trap into the analytical column. Consideration of the trap flow conditions at trap fire are required, i.e. the flow through the trap at trap fire is equal to the column flow rate which could be as little as 3mL/min. In this case a time value less than 1 minute is not recommended.

Incorrect setting of this time can be the cause of poor sensitivity for certain compounds, as insufficient time will reduce the amount of sample leaving the trap.

2.9.1.1.9 Save

Once a method parameter has been changed then the word "modified" appears alongside the method name in the blue bar at the top of the method window. When the desired method parameters have been specified they can be saved directly into the active method by clicking on the SAVE button at the bottom of the user interface.

2.9.1.2 Flow display

The flow display window (figure 49) is designed to help the user see the flow direction process and the current run status of the TT24-7 system. The diagram shows the flow direction through the solenoid valves SV1, 2, 3 and 4, and the flow direction through the heated valves and traps.

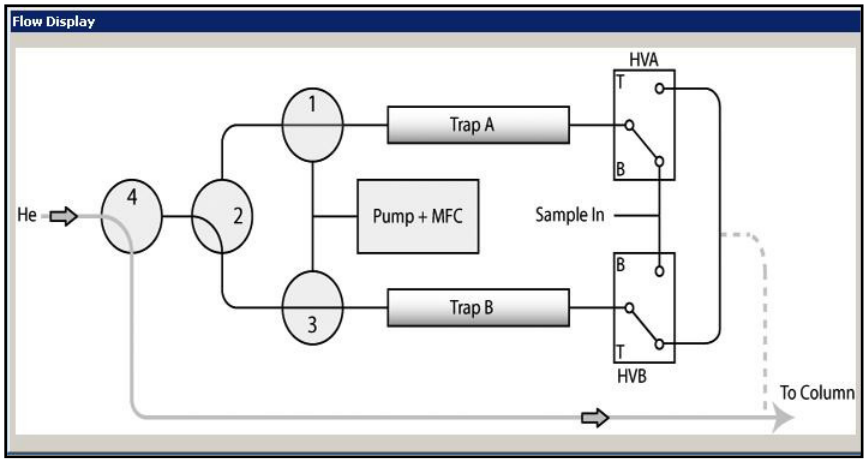

Figure 49. Flow display showing flow direction through solenoid valves

2.9.1.3 Instrument status bar

Figure 50. Instrument status bar (TT24-7e1)

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The instrument status bar (figure 50) is located at the bottom of the top level screen. It is divided into four sections, i.e. Instrument Status, Heated zones, GC and Flow Controller.

2.9.1.3.1 Instrument status

This shows the status of the continuous sampling run which is operating.

Instrument status: Indicates the current status of traps A and B. Examples of status messages are:

Sampling

Purging

Desorbing

Waiting for GC Ready

Equilibrating

Sample Time: How long the current trap has been sampling for (see section 2.9.1.1.5).

2.9.1.3.2 Heated zones

This section shows the heated zones monitored within the TT24-7.

Trap A

Trap B

Transfer Line

Dual front HV

The status bar shows two adjacent numbers. The first represents the actual value and the second the set-point value. If these two numbers are equal they appear in black font, if however the actual value is not at the set-point it will appear blue. If the TT24-7 is started when any one of these values is blue, then the software will enter an equilibrating mode and the run will only commence when the value becomes ready. These values have to be equal (±2°C tolerance value) for the system to become ready.

The transfer line and heated valves are controlled collectively by the flow path temperature (see section 2.9.1.1.3).

2.9.1.3.3 GC

The cable connecting the TT24-7 to the associated GC (see section 3.1), performs two functions. Firstly it monitors the GC ready status as specified in the GC interface logic of the TT24-7 software (see section 2.9.1.5.2). If the GC set-point values are correct and equilibrated, and the software for the GC is in a state to accept a start signal from the TT24-7, then the GC will be ready. This ready status is relayed to the TT24-7 and observed in the GC status box. If the GC is not ready, then a "Not Ready" comment appears.

When the GC is ready the second function for the cable is to send the start signal from the TT24-7 to the GC at the point of trap fire so it can begin its analytical process. If the GC is not ready at the end of the sampling time, then extended or fixed time sampling commences (see section 2.9.1.5.3).

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2.9.1.3.4 Flow controller

This component of the status box is not shown by default and requires configuring within the TT24-7 software (see section 2.9.1.5.3). When configured, following information is given.

Flow rate: the sampling flow rate through the trap

Sample gas: the composition of the sample gas (as configured in the options - section 2.9.1.5.1) is shown (typically air)

Carrier gas: the composition of the carrier gas (as configured in the options - section 2.9.1.5.1) is shown (typically Helium)

2.9.1.4. Software icons and menu items

In the header section of the top level software there is a menu bar and a series of icons as shown in figure 51.

Figure 51. Software icons and menu items for configurations e1

2.9.1.4.1 Software icons

Creates a new method which starts with default parameters

Opens the method subdirectory so that an existing method can be loaded into the TT24-7

Requests a method file name to be saved into the methods sub directory. No suffix is required as the "**.**mth" suffix is automatically added

Starts a TT24-7 run with the loaded method conditions

Opens the Stop Run dialogue box which asks whether you wish to "Stop Immediately" or "Continue running"

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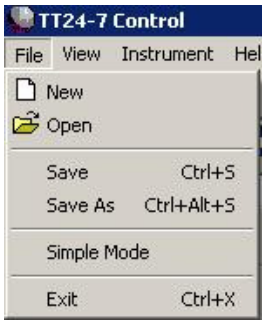
2.9.1.4.2 Menu bar

File



New: Creates a new method which starts with default parameters

Open: Opens the method subdirectory so that an existing method can be loaded into the TT24-7

Save: Saves the current TT24-7 parameters directly into the loaded method

Save As: Requests a method file name to be saved into the methods sub directory. No suffix is required as the .mth suffix is automatically added

Exit: Immediately closes down the TT24-7 software

View

Options: Accesses the TT24-7 configuration software (see section 2.9.1.5 for details)

Diagnostics: Accesses two levels of diagnostic software for the TT24-7: - flow only (user) diagnostics and full (service engineer) diagnostics (see section 2.9.1.6.)

Instrument

Run: Starts the TT24-7 with the loaded method conditions

Stop: Opens the Stop Run dialogue box which asks whether you wish to "Stop Immediately" or "Continue running"

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2.9.1.5 Options

Options is the principle configuration section for the TT24-7 system. It is accessed from the "View" menu item (section 2.9.1.4.2 above).

Within Options, there are three separate configuration screens.

2.9.1.5.1 Gas

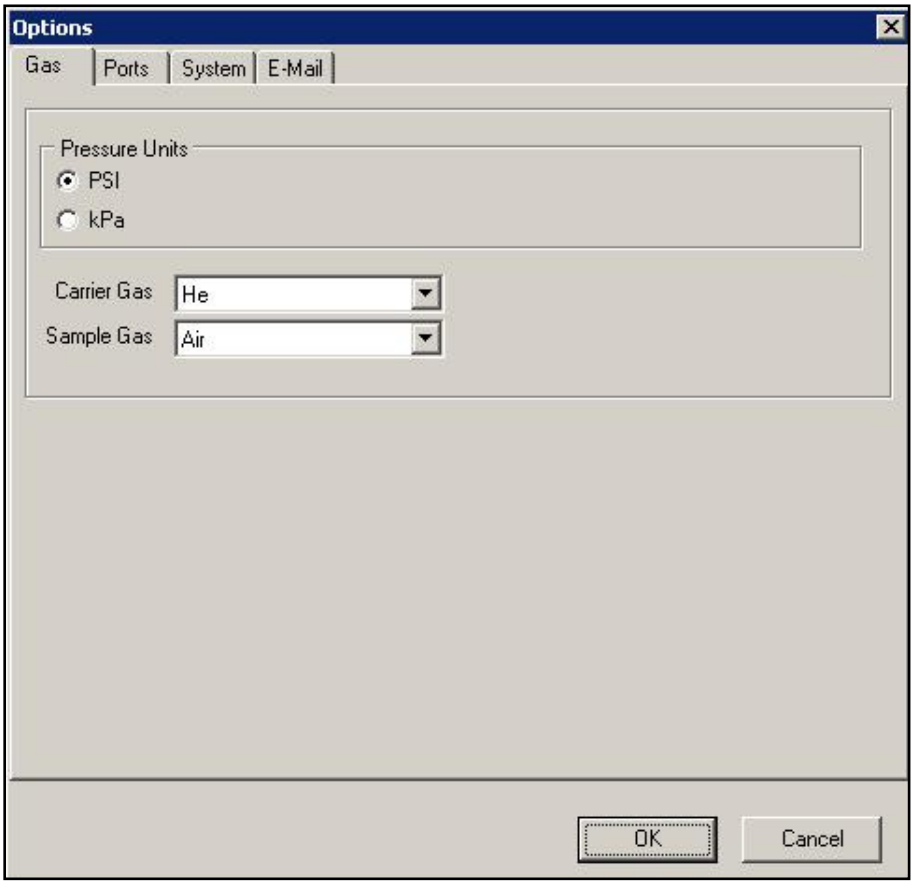

Figure 52. Options dialogue box - gas tab

This section defines the pressure units and the sample and carrier gas selection.

Pressure units:	The pressure unit selection is either psi, or kPa. Note 1 psi = 101.325 kPa. However note that there are no displays of pressure on the instrument status bar on a TT24-7e1 system.
Carrier gas type:	Choices of Helium (He), Nitrogen (N_2), Hydrogen (H_2), and diagnostic air are available.
Sample gas type:	Choices of Air, N_2, He, H_2 (safety considerations may apply using this gas), and Carbon Dioxide (CO_2) are available. This selection configures the flow calibration of the mass flow controller.

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2.9.1.5.2 Ports

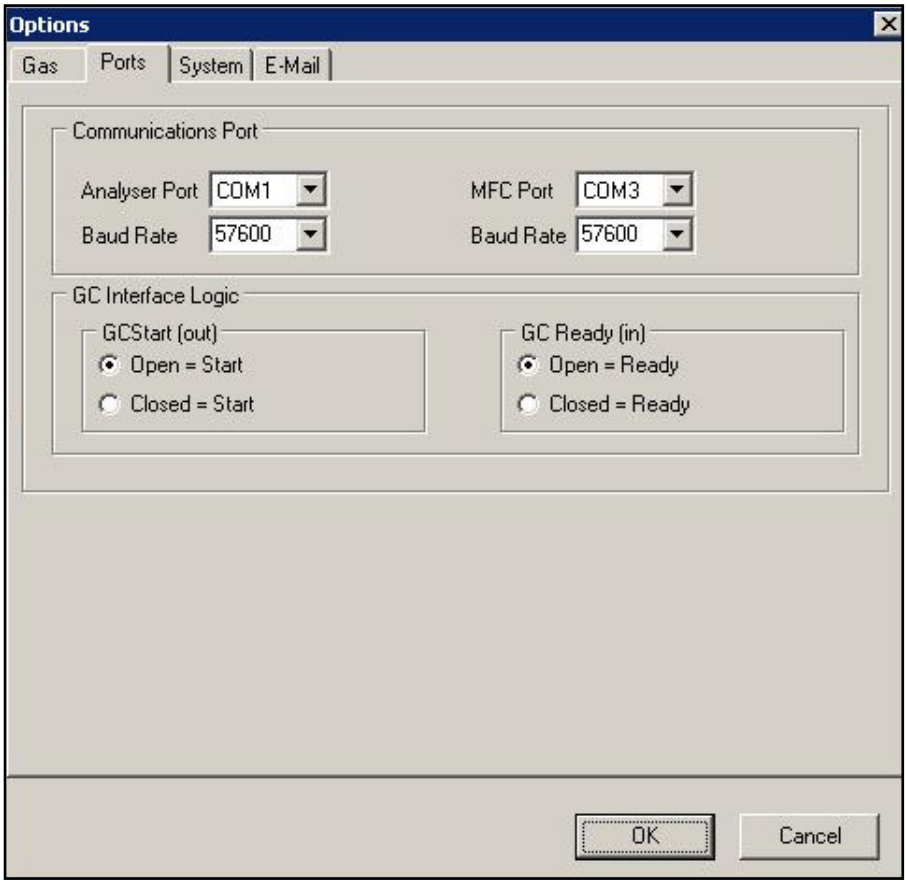

Figure 53. Options dialogue box - ports tab

This screen consists of two sections, the communications port, and the GC interface logic. These two sections control communication between the TT24-7 and the associated PC system, and the ready / not ready and start signal logic between the GC and the TT24-7.

Communications port:

The TT24-7 has two serial ports at the rear of the instrument, as shown in figure 67 section 3.1. These are the serial interfaces for the mass flow controller and the TT24-7 analyser.

Each port requires its own dedicated communications (COM port) connection from the PC. This can be provided in two ways, i.e. using two separate serial port cables from the PC system, or if two serial ports are not available using USB hub and USB to serial connection cables (N.B. Not supported by Microsoft Windows NT). See section 3.1 for more details.

Under normal circumstances the default baud rate values of 57600 should be used. However if there are communications problems then a lower baud rate be specified i.e. 38400.

GC Interface logic

This section of the software affects the start / stop interaction between

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the TT24-7 and the host GC / GCMSD system. The actual configuration is dependent on the model of GC / GCMSD used. For Agilent Technologies and Thermo Electron instrumentation the GC Start (out) and GC Ready (in) settings should be in the "closed" state.

For other GC manufacturers please contact Markes International for details.

2.9.1.5.3 System

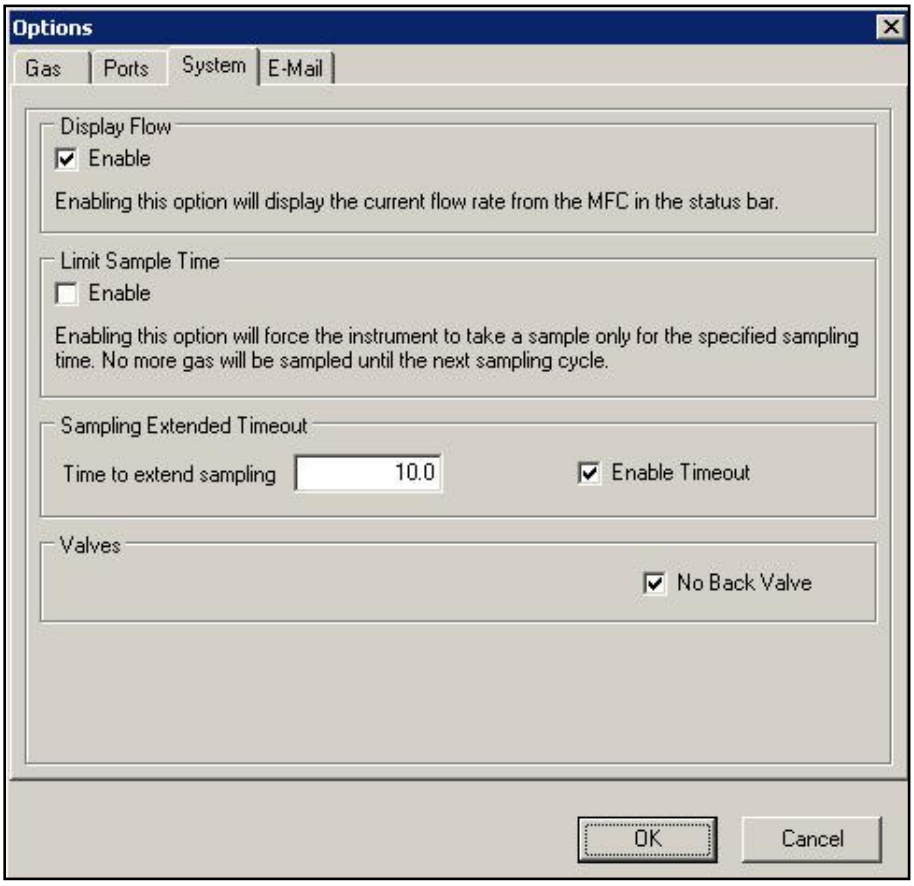

Figure 54. Options dialogue box - system tab

Display flow: If the enabling box is checked, the mass flow controller flow rate and associated values are displayed in the top level status bar (see section 2.9.1.3.4.

Limit Sample Time: This option fixes the sampling time in continuous sampling mode. No extended sampling will take place if the GC is not ready when this is active (see 2.9.1.1.5. for further information regarding sample time).

Sampling Extended Timeout: In continuous sampling mode, the TT24-7 checks on the GC ready status at the completion of the sampling time. If the GC is ready, then the system continues into dry purge and then trap fire. However if the GC system is not ready e.g. the oven has not cooled down to its set-point value and equilibrated, then the TT24-7 will automatically go into an extended sampling

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mode until the GC becomes ready. The extended sampling time can be limited to a fixed time period by entering a "Time to extend sampling" and ticking the "Enable Timeout" box.

As a result of extended sampling the amount of sample in the trap will be greater than that if extended sampling had not occurred. The analytical result will therefore be greater, and this may need to be compensated for in the final quantitative data.

Valves: The "No Back Valve" box should be checked - this configures an e1 system.

2.9.1.5.4 eMail

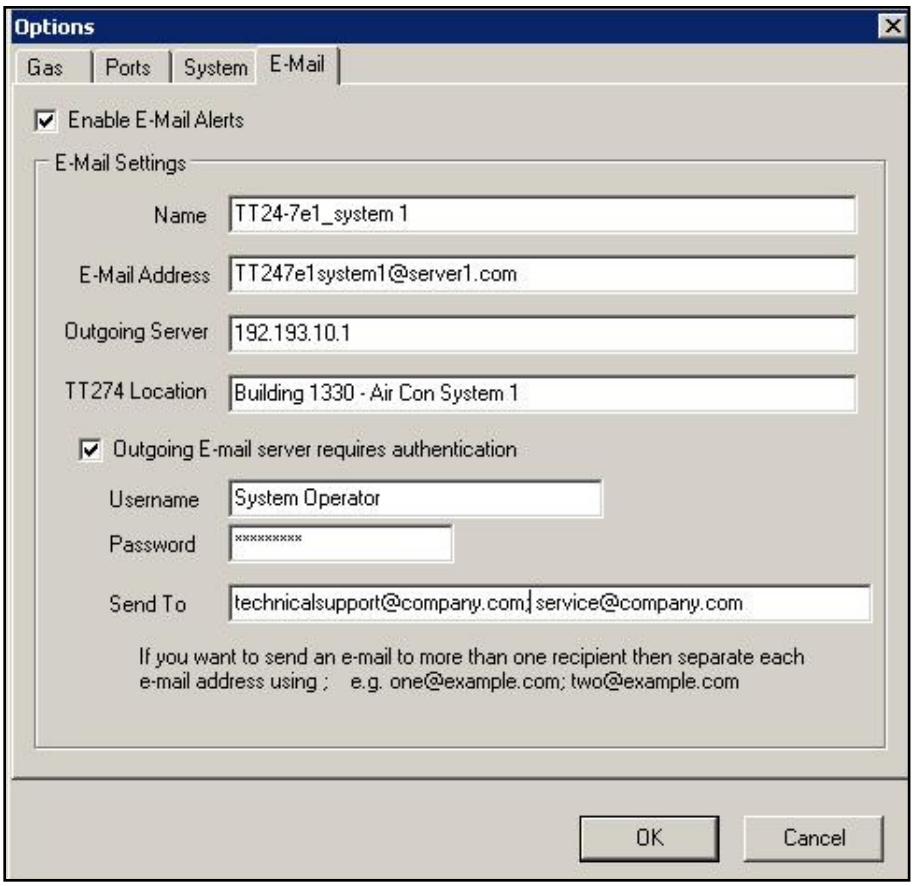

Figure 55. Options dialogue box - email tab

This screen enables alert emails to be generated should the TT24-7 system encounter a problem. This could be due to a set-point value not being controlled accurately due to a system fault. The nature of the fault, date and time it occurred appear in the Report deviation screen of the top level software.

The email alert facility has to be enabled by checking the "Enable eMail Alerts" box.

Note: Some of the items in this section of the software may have to be

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provided by the corporate IT department.

eMail Settings

Name: This can be any descriptive name for the system in use. Where multiple TT24-7 systems are employed, a unique identifying name per system is advised for accurate tracking.

email Address: This is the email address of the PC system connected to the TT24-7.

Outgoing Server: The Internet Protocol (IP) address of the server used by the PC must be specified here.

TT247 Location: This is an important item, as it identifies the exact location of the TT24-7 which has sent the email alert. It is recommended to make this section as clear and descriptive as possible.

Outgoing E-mail server requires authentication: If the Outgoing E-mail server requires authentification, the box is checked as shown above. However the alert email will not be sent unless a Username and Password are typed. If the box is unchecked i.e. no authentification is required, then the alert email is automatically generated and sent.

Send to: This defines to whom the email is sent. This could be a single person or consist of multiple email addresses. Each address has to be separated by a semi colon (;).

eMail alert message

The email alert contains the following type of information:

This is an automated message which has been sent as an error has occurred with the TT24-7. Please see the details below.

Problem: Transfer Line Over Temperature

Date: 14/02/2005 10:45:41

Name: TT24-7e1_system 1

Location: Building 1330 – Air Con System 1

This provides sufficient information to identify the nature of the problem, the system at fault, and its precise location.

As the PC system connected to the TT24-7 could be part of a company / organisation internal network, then remote access software could be initiated at this point, and the diagnostic section of the TT24-7 software accessed. This could provide a clearer insight into the problem, before dispatching repair personnel.

2.9.1.6 User diagnostics

User diagnostics allows the user to manually manipulate the valves - both heated valves and solenoid valves - in order to assist in tracking down a leak in the system (see section 4.1). It is accessed from the "View" menu item (section 2.9.1.4.2 above).

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Selecting "Diagnostics" opens a password Dialogue Box - enter the password "flowonly" and this will open the diagnostics screen - figure 56. Clicking on any of the valves will cause the valve to move (e.g. move UP from DOWN or OFF from ON) allowing different parts of the flow path to be leak checked with an external device such as a helium leak detector (see Section 4.1).

Before exiting the flow only diagnostic section, click the "standby" button (top left hand side), this returns all valves to the standby position ready for system operation.

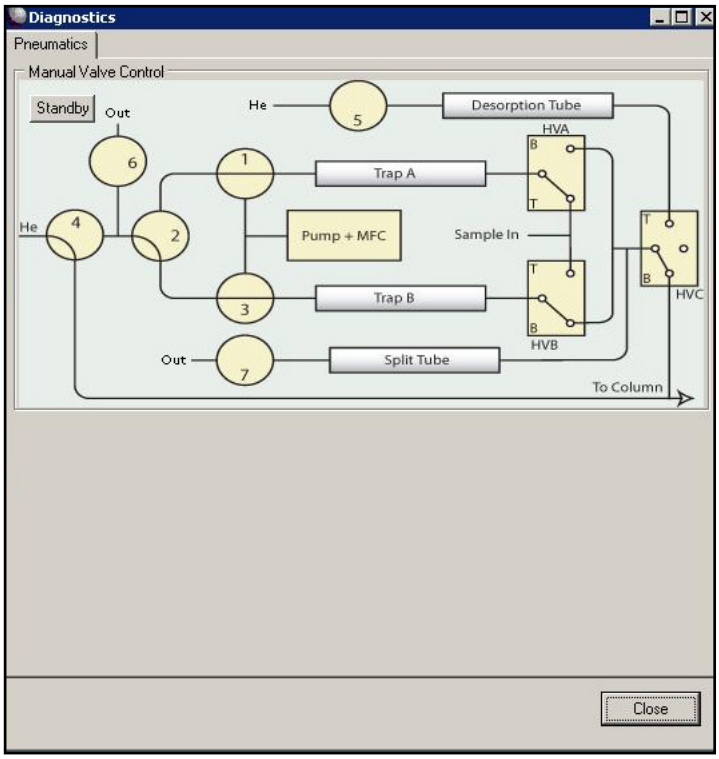

Figure 56. Flow diagnostics screen

2.9.1.7 Reporting module

The reporting module (figure 57) displays a number of different Information, Warning or Error messages.

All messages detailed below will appear for either Trap A or Trap B if relevant. Trap A is used as an example below. All error messages are date and time stamped e.g. 05/12/2005 10:34:54

Figure 57. Reporting module

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2.9.1.7.1 Information messages

[INFO] Sampling started on Trap A

- date and time stamped when sampling started

[INFO] Sampling has been extended on Trap A

- reported when samping time exceeded the expected time set in the method, this is typically due to the GC Ready signal being delayed - perhaps due to the oven taking longer than expected to cool down.

Information messages do not affect the operation of the system in any way.

2.9.1.7.2 Warning messages

[WARNING] Sampling volume outside of expected range

- this warning message is given when the total volume sampled is not what would be expected from the method. For example this could be because the sampling time was extended (see section 2.9.1.5.3) or because the flow controller was unable to pull the required flow rate over the sampling time (due to a failing cold trap or other problem).

A warning message does not affect the operation of the TT24-7 system but gives information about something which may affect the quantitation and the results.

2.9.1.7.3 Error messages

[ERROR] Trap A not heating

[ERROR] Valve not heating

[ERROR] Line not heating

[ERROR] Trap A over temperature

[ERROR] Valve over temperature

[ERROR] Line over temperature

If one of the above errors occur then the TT24-7 system will shut down and await operator / service engineer intervention.

2.9.2 User interface for TT24-7e2 and TT24-7e3 hardware configurations

2.9.2.1 Method parameters

At the top of the method window is shown the active method name (e.g. default.mth) and its status i.e. whether or not it has been modified from its original set-point values.

The set point values used in the interface are described below.

2.9.2.1.1 Mode

Hardware configurations e2 and e3 allow two different sampling modes - each with a slightly different user interface.

Figure 58 shows the two user interfaces for the different sampling modes.

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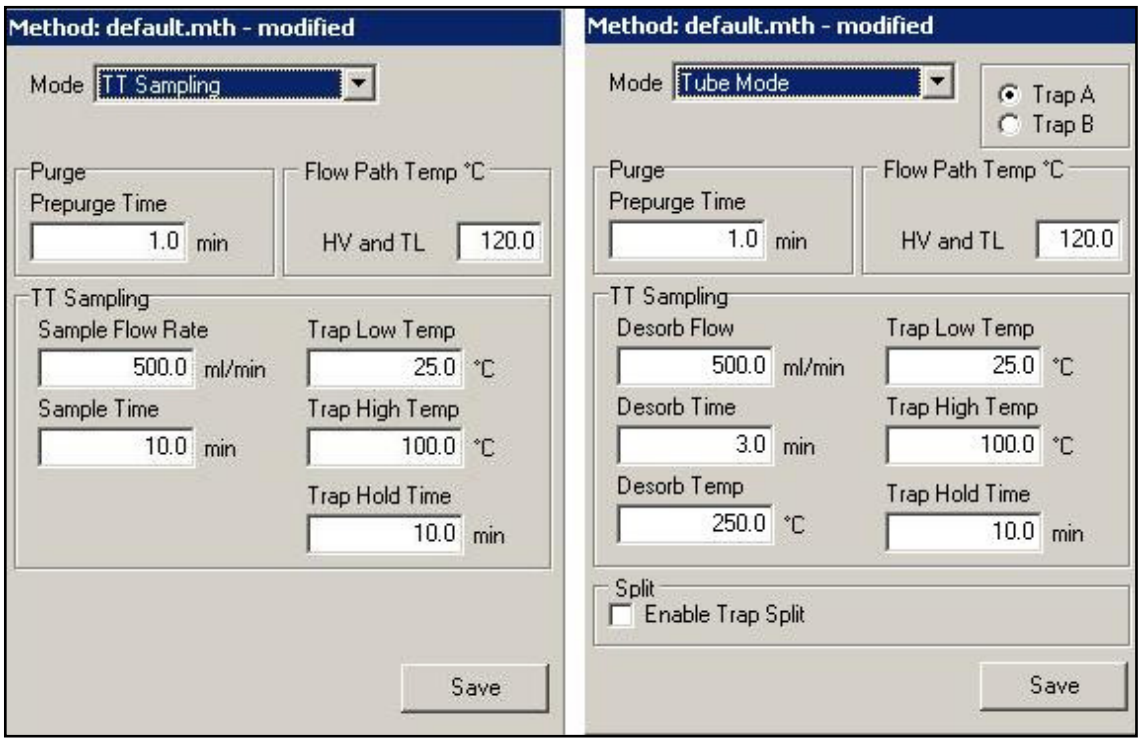

Figure 58. User interface for each sampling mode

TT Sampling: for continuous sampling from a stream of gas/air (see section 2.3.1)

Tube Desorb: allows the desorption of a sorbent tube (see section 2.3.2)

2.9.2.1.2 Prepurge time - *available in both sampling modes*

This is the time that the **trap** is dry-purged (carrier gas passed through the trap in the sampling direction) just prior to trap fire. This dry-purge removes air / moisture from the trap after sampling and the flow is controlled by a needle valve SV6 as shown in figure 21. The flow should be typically set to a value of ~ 50 mL/min. The pre-purge can be set between 0 and 99.9 minutes in increments of 0.1 minute and is typically 0.5 to 1 minute, although this can be reduced where the cycle time of the TT24-7 system needs to be kept to a minimum.

2.9.2.1.3 Flow path temp - *available in both sampling modes*

This is the set point temperature for the heated valves and the transfer line to the GC. The temperature range extends from ~100°C to 200°C. The temperature should be set high enough to prevent any condensation of sample within the valve / transfer line and values less than 120°C are rarely used. However this temperature value will also affect the lower trap temperature value.

If the flow path temperature is set to its maximum of 200°C then the trap temperature cannot be controlled at values less than 25°C. If the flow path temperature is set to 150°C then a minimum trap temperature of ~15°C is possible.

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2.9.2.1.4 Sample flow rate - *available in TT sampling mode*

This specifies the flow rate of sample vapour passing through the traps. The sample may be at atmospheric pressure, in which case a vacuum pump is required to pull sample into the system. The flow rate is then regulated by the internal mass flow controller (MFC). If the sample is at positive pressure (Max 50 psi), then the vacuum pump is not required, and again the MFC controls the flow through the trap.

Sample flow rates from 50 mL/min to 1 L/min are possible, with 0.1 mL increments, however the maximum flow rate is affected by the sorbent bed depth and mesh size. Where fine mesh size material is used (i.e. 80/100 mesh or finer) the maximum flow rate will be reduced. Ultimately the fastest flow rate achievable is determined by specifying the maximum flow rate of 1 L/minute and observing the actual flow value on the status bar (see section 2.9.2.3) at the bottom right side of the user interface.

In certain applications where the analyte concentration is very low (i.e. sub ppb) and there is a time restriction on sampling (i.e. NRT) there is a temptation to set a very fast sampling flow rate to maximise the amount of analyte passing into the trap in unit time. For example sampling at 1 L/min for 10 minutes results in a sampling volume of 10L. If the analyte concentration is very low e.g. in the pg/L concentration (i.e. ppt), then a 10L volume would contain sufficient sample in theory for detection by selective GC detectors or by GCMS.

However sensitivity is very dependant on the signal to noise (S/N) ratio of the resultant chromatographic peak. If this is sharp i.e. peak widths <= 5 seconds then much better integration and detection is possible than for peak widths >=15 seconds.

In practice it has been shown that very fast sampling flow rates (>= 800 mL/min) with certain sorbent materials produces poor or broad peak shapes and this can compromise the minimum detection limit (MDL) for that compound. If the same compound is run at a lower flow i.e. 400 mL/min as opposed to 800 mL/min, this can result in a much sharper peak and a significantly better S/N ratio even though the amount of analyte trapped is half.

The science behind this effect is based on the depth the analyte passes into the sorbent, and as a consequence how easily it comes off at trap fire. It is therefore both compound and sorbent dependant.

2.9.2.1.5 Sample time - *available in TT sampling mode*

This specifies the time interval for sample to pass into the trap. This parameter is directly associated with the sampling flow rate as discussed above to determine the total amount of sample transferred. The range for sample time is from 0.1 to 99.9 minutes, however, values greater than 1 minute will typically be used.

The sampling time and therefore the sampling volume can be affected by the ready / not ready status of the GC system. At the completion of the defined set-point time value and prior to the system going into the pre-purge phase of the trap, the TT24-7 monitors the GC ready status. If the GC is ready then the pre-purge process commences. If however the GC is not ready then the

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sampling time is extended until the GC becomes ready, or a specified "time out" value (see section 2.8.1.5.3) is equalled at which point the system stops. The GC becoming not ready is typically due to the oven not cooling down as fast as normal and this affects the GC cycle time.

The sampling time can be fixed to the specified value i.e. preventing extended sampling within the TT24-7 software. Under these conditions if the GC is not ready then after the fixed sampling time the system then waits until the GC becomes ready or again the time out value stops the process. See section 2.9.1.5.3 for extended sampling and fixed time sampling.

2.9.2.1.6 Desorb flow - *available in tube desorb mode*

This is the flow rate of carrier gas which passes through the tube during its primary desorption phase when the tube is heated to transfer the retained sample into the selected trap - either A or B (see 2.9.2.1.11). Flow values ranging from 50 mL/min to 1000 mL/min are possible and typically flow rates of ~100 mL/min are used.

2.9.2.1.7 Desorb time - *available in tube desorb mode*

This is the primary desorption time interval, values ranging from 0.1 to 10 minutes are possible. Within this time frame sample from the tube must pass into the selected trap and the tube must reach and maintain its upper temperature value (section 2.9.2.1.8). Values of less than three minutes are therefore not recommended unless the required desorb temperature is very low. The heating rate for the tube is much slower than that of the trap so sufficient time must be given for the tube oven to reach its desired set-point and stay at this value for at least 1 minute.

2.9.2.1.8 Desorb temp - *available in tube desorb mode*

This is the upper tube set-point temperature. Values ranging from 50°C to 350°C are possible, however the upper value should not exceed the maximum temperature for the sorbents used. If a mixed bed sorbent tube is used the upper value should not exceed the lowest of the maximum temperatures. See Appendix 3 for details.

Note: If you exceed the maximum sorbent temperature, the resulting breakdown of the sorbent may severely contaminate the flow path of your system. This may require the complete replacement of the flow path and associated components by a fully qualified service engineer and would not be covered by the instrument warranty.

2.9.2.1.9 Trap A/B - *available in tube desorb mode*

This selects which trap is used for sampling i.e. onto which trap A or B the desorbed or injected sample is passed.

2.9.2.1.10 Trap low temperature - *available in both sampling modes*

This is the (lower) trap temperature value used to retain compounds on the sorbent bed. Values ranging from 15 to 50°C are possible. Sub ambient values are possible as a consequence of the Peltier cooling elements situated immediately below each trap.

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The trap minimum value is sensitive to both the flow path temperature described above, the flow rate of sample passing through the trap, and the temperature of the air sample. For example if the flow path temperature is set to 200°C, with a fast flow rate (i.e. >= 700 mL/min), then the trap minimum value may be 25°C or higher at faster flows. If additionally the sample gas is above ambient then this minimum value will be higher again.

Using lower sampling flows will assist this value, and can indeed improve on signal to noise for the resulting chromatographic peak (see section 2.9.1.1.5 above).

Ultimately the minimum trap temperature achievable is determined experimentally by reviewing the relationship between the set-point and actual values as shown in the status bar, and this will be a function of the flow path temperature and the sampling conditions.

2.9.2.1.11 Trap high temperature - *available in both sampling modes*

This is the upper temperature setting for the trap. Values ranging from 50°C to 400°C are possible. The upper trap temperature should be set to the optimum value which enables 100% recovery of analytes from the sorbent bed, **but does not exceed the maximum permissible temperature for that sorbent.** See Appendix 3. Keeping the maximum trap temperature as low as possible whilst still enabling 100% analyte recovery will extend trap lifetime.

Note: If you exceed the maximum sorbent temperature, the resulting breakdown of the sorbent may severely contaminate the flow path of your system. This may require the complete replacement of the flow path and associated components by a fully qualified service engineer and would not be covered by the instrument warranty.

When multi bed traps are being used, the maximum temperature that can be set is that relating to the sorbent with the lowest allowable temperature. Consideration must then be taken of the recovery efficiency for analytes from sorbents which go to higher values.

2.9.2.1.12 Trap hold time - *available in both sampling modes*

This is the time interval when the trap is held at its maximum value. Time intervals from 0.1 to 10 minutes are possible, however values in excess of ~ 0.5 minutes are typically used. This value must be long enough to ensure complete removal of analytes from the trap into the analytical column. Consideration of the trap flow conditions at trap fire are required, i.e. the flow through the trap at trap fire is equal to the column flow rate which could be as little as 3mL/min. In this case a time value less than 1 minute is not recommended.

Incorrect setting of this time can be the cause of poor sensitivity for certain compounds, as insufficient time will reduce the amount of sample leaving the trap.

2.9.2.1.13 Save - *available in both sampling modes*

Once a method parameter has been changed then the word "modified" appears alongside the method name in the blue bar at the top of the method window.

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When the desired method parameters have been specified they can be saved directly into the active method by clicking on the SAVE button at the bottom of the user interface.

2.9.2.1.14 Split - *available in both sampling modes*

Note: The "Enable Trap Split" checkbox is only available with TT24-7e3 hardware configurations and when the split has been configured in Options (section 2.9.2.5).

When enabled, this function opens the split solenoid valve SV7 (see figure 37 in section 2.5.3.2.1) at the point of trap fire and splits the sample in proportion to the split flow rate and column flow rate (see section 2.3.3).

2.9.2.2 Flow Display

The flow display window (figure 59) is designed to help the user see the flow direction process and the current run status of the TT24-7 system. The diagram shows the flow direction through the solenoid valves SV1, 2, 3, 4, 5 and 6, the flow direction through the heated valves (HVA, HVB, HVC), the flow through the traps A and B, and the flow through SV7 solenoid valve in a TT24-7e3 configuration (if the split is enabled - see 2.8.2.1.14).

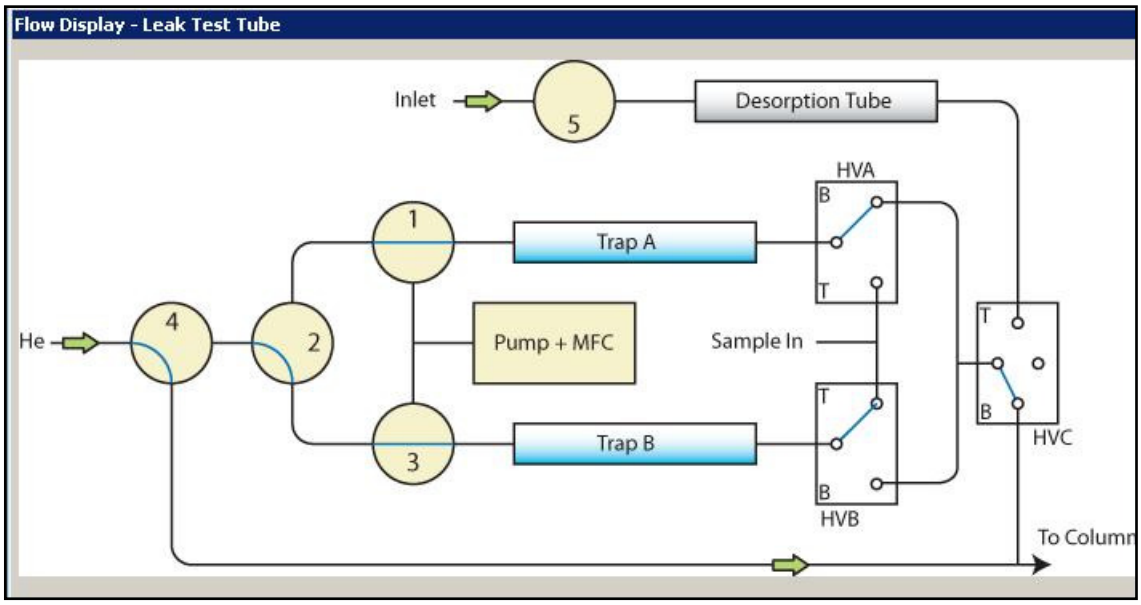

Figure 59. Flow Display showing flow direction through solenoid valves

2.9.2.3 Instrument status bar

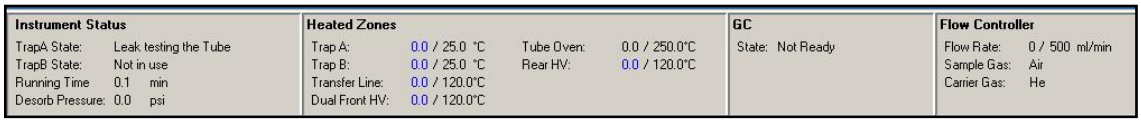

Figure 60. Instrument status bar - e2/e3 configurations

The instrument status bar (figure 60) is located at the bottom of the top level screen. It is divided into four sections, i.e. Instrument Status, Heated zones, GC and Flow Controller.

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2.9.2.3.1 Instrument Status

This shows the status of the sampling run which is operating.

Instrument status:

Indicates the current status of traps A and B e.g. sampling, desorbing, leak testing tube etc.

Sample Time: *(TT Sampling mode)*

How long the current trap has been sampling for

Running Time *(Tube Desorb mode)*

The run time associated with the current operational phase i.e. desorbing the tube, leak test, dry-purge etc.

Desorb Pressure *(Tube Desorb mode)*

Equivalent to the column head pressure at the point of tube desorption

2.9.2.3.2 Heated Zones

This section shows the heated zones monitored within the TT24-7.

Trap A

Trap B

Transfer Line

Dual Front HV

Tube Oven

Rear HV

The status bar shows two adjacent numbers. The first represents the actual value and the second the set-point value. If these two numbers are equal they appear in black font, if however the actual value is not at the set-point it will appear blue. If the TT24-7 is started when any one of these values is blue, then the software will enter an equilibrating mode and the run will only commence when the value becomes ready. These values have to be equal (±2°C tolerance value) for the system to become ready.

The transfer line and heated valves are controlled collectively by the flow path temperature (see section 2.9.2.1.3).

2.9.2.3.3 GC

The cable connecting the TT24-7 to the associated GC (see section 3.1), performs two functions. Firstly it monitors the GC ready status as specified in the GC interface logic of the TT24-7 software (see section 2.9.2.5.2). If the GC set-point values are correct and equilibrated, and the software for the GC is in a state to accept a start signal from the TT24-7, then the GC will be ready. This ready status is relayed to the TT24-7 and observed in the GC status box. If the GC is not ready, then a "Not Ready" comment appears.

When the GC is ready the second function for the cable is to send the start signal from the TT24-7 to the GC at the point of trap fire so it can begin its analytical process. If the GC is not ready at the end of the sampling time, then extended or fixed time sampling commences (see section 2.9.2.5.3).

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2.9.2.3.4 Flow controller

This component of the status box is not shown by default and requires configuring within the TT24-7 software (see section 2.9.2.5.3). When configured, following information is given.

Flow rate: the sample flow as follows

Trap A / B *TT Sampling mode*

Tube desorb flow *tube desorb mode*

Sample gas: the composition of the sample gas (as configured in the Options - section 2.9.2.5.3) is shown (typically air)

Carrier gas: the composition of the carrier gas (as configured in the Options - section 2.9.2.5.3) is shown (typically He)

2.9.2.4 Software icons and menu items

In the header section of the top level software there is a menu bar and a series of software icons (figure 61).

Figure 61. Software Icons and menu bar for configurations e2/e3

2.9.2.4.1 Software icons

Creates a new method which starts with default parameters

Opens the method subdirectory so that an existing method can be loaded into the TT24-7

Requests a method file name to be saved into the methods sub directory. No suffix is required as the **.**mth suffix is automatically added.

Starts a TT24-7 run with the loaded method conditions

Opens the Stop Run dialogue box which asks whether you wish to "Stop Immediately" or "Continue running"

Initiates a leak test procedure. The first function is to perform a leak test of the tube, followed by a leak test of trap A and then trap B

Allows manual (needle valve) control of the split flow through SV7 (if enabled - e3 systems) and dry purge flow through SV6

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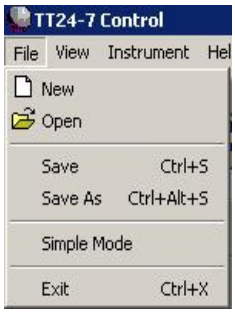
2.9.1.4.2 Menu bar

File



New: Creates a new method which starts with default parameters

Open: Opens the method subdirectory so that an existing method can be loaded into the TT24-7

Save: Saves the current TT24-7 parameters directly into the loaded method

Save As: Requests a method file name to be saved into the methods sub directory. No suffix is required as the .mth suffix is automatically added

Exit: Immediately closes down the TT24-7 software

View

Options: Accesses the TT24-7 configuration software (see section 2.9.1.5 for details)

Diagnostics: Accesses two levels of diagnostic software for the TT24-7: - flow only (user) diagnostics and full (service engineer) diagnostics (see section 2.9.2.6.)

Instrument

Run: Starts the TT24-7 with the loaded method conditions

Stop: Opens the Stop Run dialogue box which asks whether you wish to "Stop Immediately" or "Continue running"

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2.9.2.5 Options

Options is the principle configuration section for the TT24-7 system. It is accessed from the "View" menu item (see 2.9.1.4.2).

Within options, there are four separate configuration screens.

2.9.2.5.1 Gas

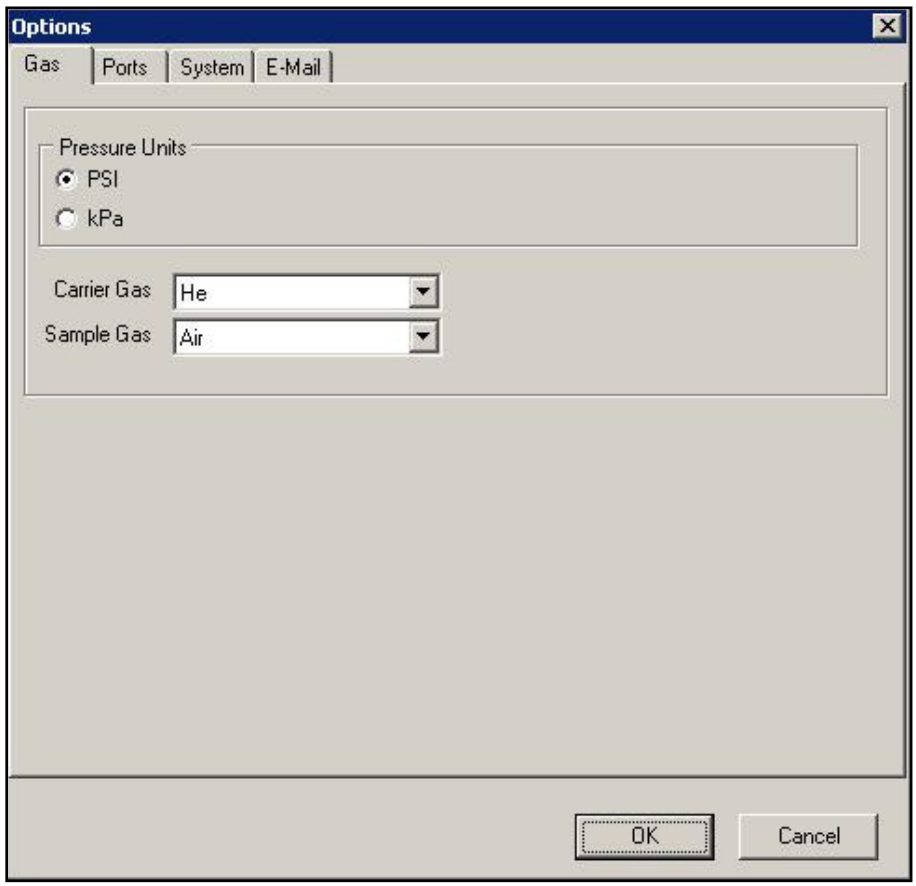

Figure 62. Options dialogue box - gas tab

This section defines the pressure units and the sample and carrier gas selection.

Pressure units: The pressure unit selection is either psi, or kPa.

Note 1 psi = 101.325 kPa.

In Tube Desorb mode the desorb pressure is displayed on the instrument status bar (see section 2.9.2.3.1)

Carrier gas type: Choices of Helium (He), Nitrogen (N_2), Hydrogen (H_2), and diagnostic air are available. The selection made here is shown in the flow controller status box as part of the instrument status. See section 2.8.1.3.4

Sample gas type: Choices of Air, N_2, He, H_2 (safety considerations may apply using this gas), and Carbon Dioxide (CO_2) are available. This selection configures the flow calibration of the mass flow controller. The selection made here is also shown in the flow controller status box.

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2.9.2.5.2 Ports

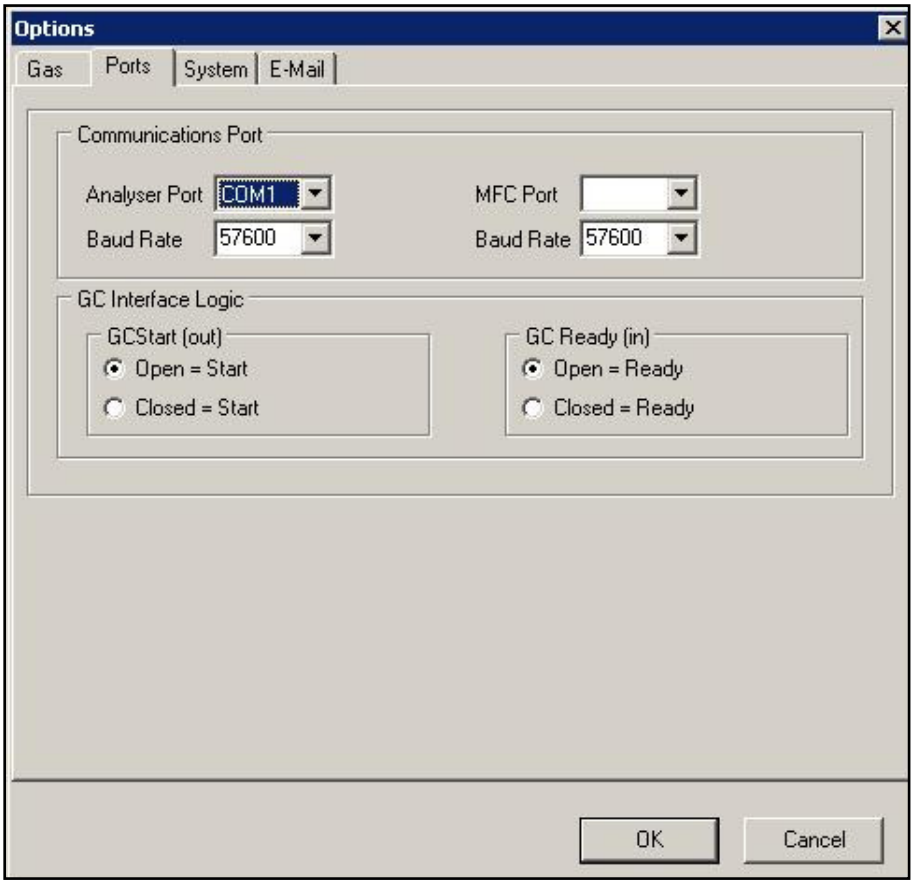

Figure 63. Options dialogue box - ports tab

This screen consists of two sections, the communications port, and the GC interface logic. These two sections control communication between the TT24-7 and the associated PC system, and the ready / not ready and start signal logic between the GC and the TT24-7.

Communications port:

The TT24-7 has two serial ports at the rear of the instrument, as shown in figure 67 section 3.1. These are the serial interfaces for the mass flow controller and the TT24-7 analyser.

Each port requires its own dedicated communications (COM port) connection from the PC. This can be provided in two ways, i.e. using two separate serial port cables from the PC system, or if two serial ports are not available using USB hub and USB to serial connection cables (N.B. Not supported by Microsoft Windows NT). See section 3.1 for more details.

Under normal circumstances the default baud rate values of 57600 should be used. However if there are communications problems then a lower baud rate be specified i.e. 38400.

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GC Interface logic

This section of the software affects the start / stop interaction between the TT24-7 and the host GC / GCMSD system. The actual configuration is dependent on the model of GC / GCMSD used. For Agilent Technologies and Thermo Electron instrumentation the GC Start (out) and GC Ready (in) settings should be in the "closed" state.

For other GC manufacturers please contact Markes International for details.

2.9.2.5.3 System

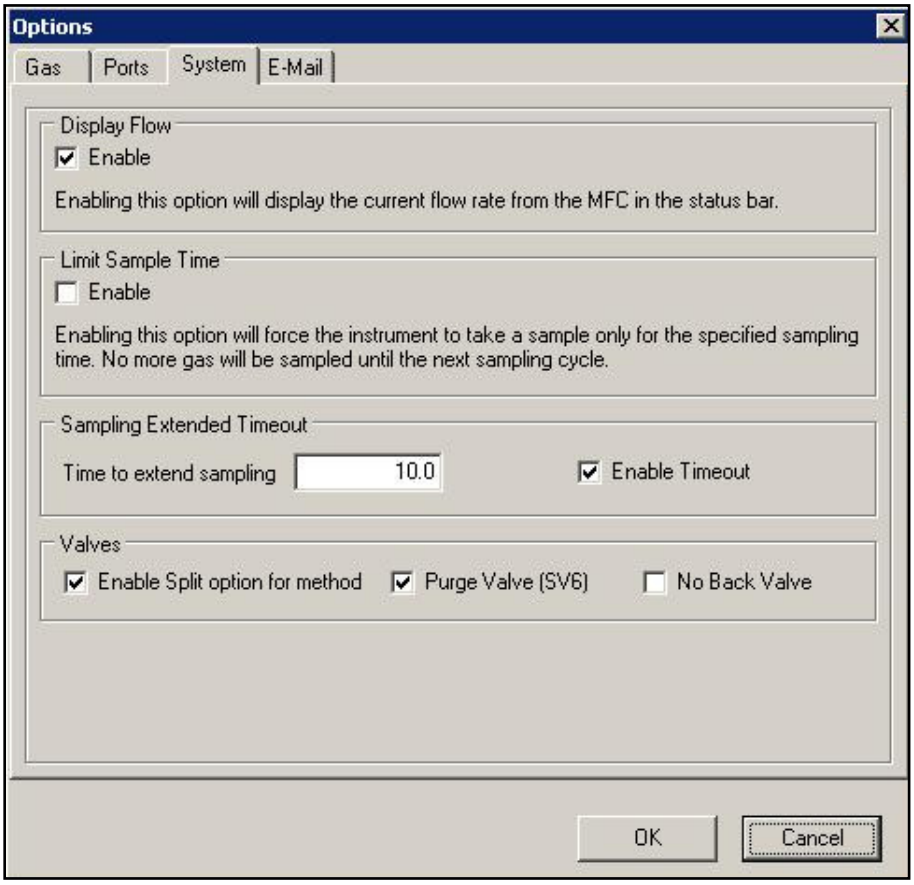

Figure 64. Options dialogue box - system tab

Display flow: If the enabling box is checked, the mass flow controller flow rate and associated values are displayed in the top level status bar (see section 2.8.1.3.4.

Limit Sample Time: This option fixes the sampling time in continuous sampling mode. No extended sampling will take place if the GC is not ready when this is active (see 2.8.1.1.5. for further information regarding sample time).

Sampling Extended Timeout: In continuous sampling mode, the TT24-7 checks on the GC ready status at the completion of the sampling time. If the GC is ready, then the system continues into dry purge and then trap fire. However if

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the GC system is not ready e.g. the oven has not cooled down to its set-point value and equilibrated, then the TT24-7 will automatically go into an extended sampling mode until the GC becomes ready. The extended sampling time can be limited to a fixed time period by entering a "Time to extend sampling" and ticking the "Enable Timeout" box.

As a result of extended sampling the amount of sample in the trap will be greater than that if extended sampling had not occurred. The analytical result will therefore be greater, and this may need to be compensated for in the final quantitative data.

The total sampling volume is reported in the reports deviation screen, which allows the re calculation of the correct amount of analyte retained on the trap.

Valves: There are three tick boxes that may be checked

Enable Split option for method

For TT24-7e3 systems configured with the split option this box should be checked to include this capacity in the method

Purge Valve SV6

For TT24-7e2/e3 hardware configurations the SV6 valve box should be checked to configure the purge valve and allow dry purging of the traps

No Rear Valve

This box should be unchecked for e2 and e3 configurations.

2.9.2.6 User diagnostics

User Diagnostics allows the user to manually manipulate the valves - both heated valves and solenoid valves - in order to assist in tracking down a leak in the system (see section 4.1). It is accessed from the "View" menu item (section 2.9.1.4.2 above).

Selecting "Diagnostics" opens a password Dialogue Box - enter the password "flowonly" and this will open the diagnostics screen - figure 65. Clicking on any of the valves will cause the valve to move (e.g. move UP from DOWN or OFF from ON) allowing different parts of the flow path to be leak checked with an external device such as a helium leak detector (see Section 4.1).

Before exiting the flow only diagnostic section, click the "standby" button (top left hand side), this returns all valves to the standby position ready for system operation.

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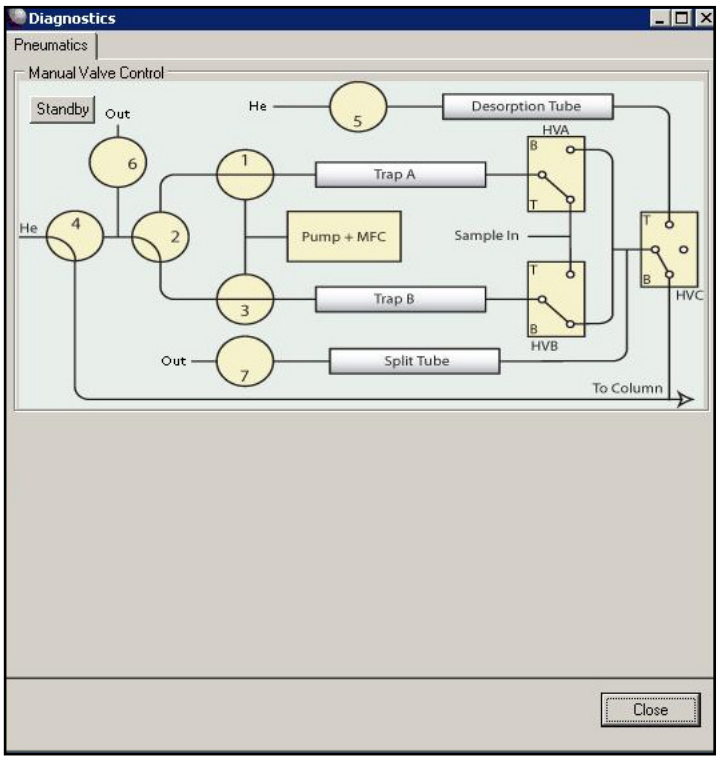

Figure 65. Flow diagnostics screen

2.9.2.7 Reporting module

The reporting module (figure 66) displays a number of different Information, Warning or Error messages.

All messages detailed below will appear for either Trap A or Trap B if relevant. Trap A is used as an example below. All error messages are date and time stamped e.g. 05/12/2005 10:34:54

Figure 66. Reporting module

2.9.2.7.1 Information messages

[INFO] Sampling started on Trap A

- date and time stamped when sampling started

[INFO] Sampling has been extended on Trap A

- reported when samping time exceeded the expected time set in the method, this is typically due to the GC Ready signal being delayed - perhaps due to the oven taking longer than expected to cool down.

Information messages do not affect the operation of the system in any way.

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2.9.2.7.2 Warning messages

[WARNING] Sampling volume outside of expected range

- this warning message is given when the total volume sampled is not what would be expected from the method. For example this could be because the sampling time was extended (see section 2.9.1.5.3) or because the flow controller was unable to pull the required flow rate over the sampling time (due to a failing cold trap or other problem).

A warning message does not affect the operation of the TT24-7 system but gives information about something which may affect the quantitation and the results.

2.9.2.7.3 Error messages

[ERROR] Trap A not heating

[ERROR] Valve not heating

[ERROR] Line not heating

[ERROR] Trap A over temperature

[ERROR] Valve over temperature

[ERROR] Line over temperature

If one of the above errors occur then the TT24-7 system will shut down and await operator / service engineer intervention.

3.0 User Installed Components and Maintenance

3.1 System cabling TT24-7 / PC / GC

This section describes the cabling connections required to correctly operate and control the TT24-7 system. The TT24-7 has electrical connections with both the associated GC, and the controlling PC. A power supply is also required (100 - 240V).

There are four cables attached to the back of the TT24-7 system. These consist of:

i) and ii) Two (9 pin "D") serial (RS232) communication cables (P/N Z-0189)

iii) A single 25 pin remote (GC) start/stop cable, which is instrument specific

Agilent GC P/N UTD-5098

Thermo Electron GC P/N UTD-5108

any other GC system uses UTD-5095

iv) A power cable (P/N Z-0024).

These four cables must be connected correctly for successful operation of the TT24-7 system and remote starting of the associated GC.

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3.1.1 TT24-7 power cable - ON/OFF switch

The power cable is inserted into the power supply socket at the rear of the TT24-7 and the system is switched on using the adjacent ON/OFF switch. The location of this connection and switch is shown in figure 67. The power is supplied to the system when the ON/OFF switch is in the depressed state ▬ To turn the TT24-7 OFF press the ON/OFF switch so the depressed state is **O**.

3.1.2 Communications and Remote start cabling

Figure 67 shows the rear of the TT24-7 illustrating the location of the sockets for the two RS232 communication cables, the remote start cable and the power cable.

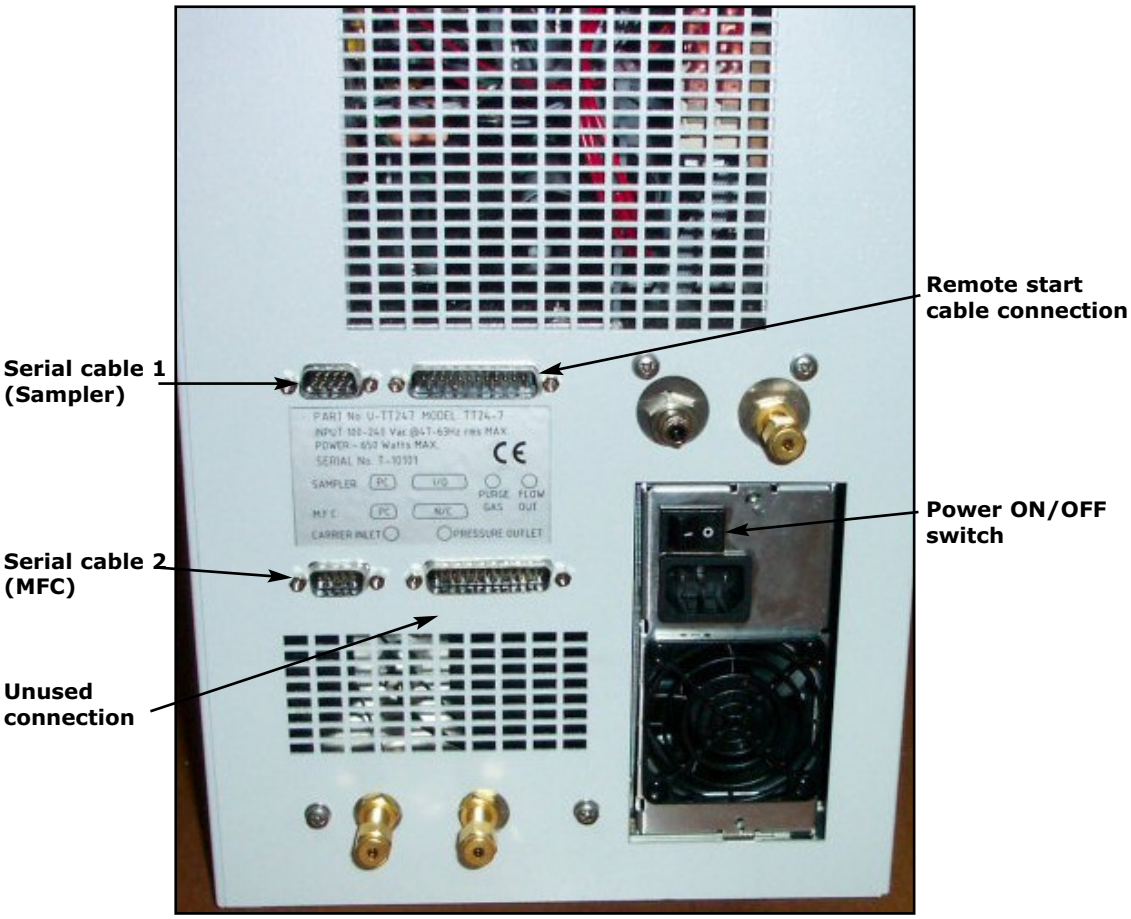

Figure 67. Rear view of TT24-7 showing system cabling connections

Figure 68 shows a close up of the labelling on the back of the TT24-7. (Note that the label also shows the pneumatic connections into the TT24-7.)

Additional useful information such as the power requirements and instrument serial number for the TT24-7 are also shown here.

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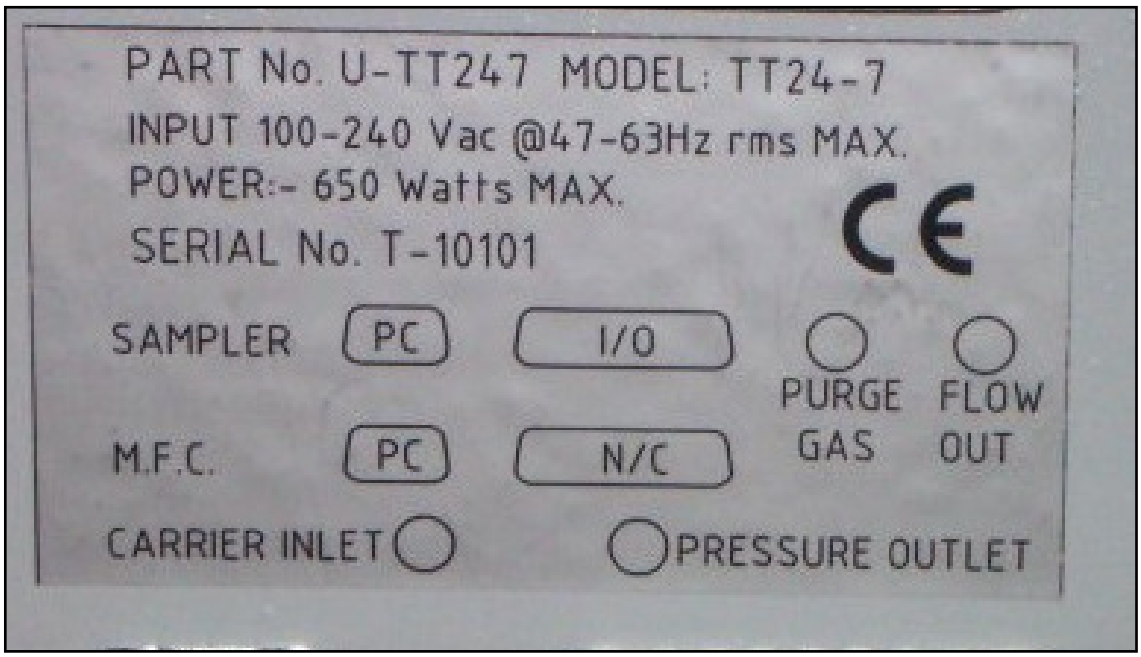

Figure 68. TT24-7 labelling

One of the 9 pin serial (RS232) cables connects into the Sampler port of the TT24-7. This communicates with the analyzer part of the TT24-7 for instrument control. This cable leads back to the PC, either directly into a serial communication port (COM1, COM2 etc.) in the PC or via a USB hub. The COM port assignment is selected within the TT24-7 software (see section 2.9.1.5.2/2.9.2.5.2 Ports).

The second serial cable connects into the MFC connector of the TT24-7. This is the mass flow controller connection port and all flow related values within the TT24-7 system are controlled and monitored via this cable connection to the PC. As for the Sampler connection described above this cable also leads back to the PC, either directly into a serial communication port (COM2, COM1) in the PC or via a USB hub.

The COM port assignment is selected within the TT24-7 software (see section 2.9.1.5.2 (e1) and 2.9.2.5.2 (e2/e3)). Note that the COM port selection for the MFC and Sampler must be different.

If the two serial cables become disconnected from the back of the TT24-7, ensure that the correct serial cable is returned to its allocated socket as per the COM settings. If the cables are crossed over the TT24-7 will not operate correctly.

Connection of the 25 pin Remote start cable is made into the I/O socket of the TT24-7. This cable is also connected into the associated GC, and particularly into the GC Remote start socket. Because GC manufacturers have different connectors for a Remote Start cable, different cables are required for each GC.

The N/C socket is currently not used.

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3.1.3 Serial and / or USB Cable connections

The TT24-7 requires two serial connections with the controlling PC. Direct serial connection therefore requires two spare COM ports in the PC. However many modern PC systems do not have these serial ports available and may require upgrading with an additional serial card.

When using Microsoft Windows™ 98™, 2000™ or XP™, an extra serial port can be generated using an available USB port and a USB to serial port conversion cable (P/N U-USBSR).

Note: USB communication is **NOT** possible with Windows NT™

The standard 9-pin RS232 serial cable is connected between the TT24-7 and the USB / Serial conversion cable. The conversion cable may be connected directly to a USB port on the PC or, if there are insufficient spare USB ports, into a USB hub (U-USBHB) which can accommodate up to four connections. The hub is then connected directly to one USB port on the PC.

Figure 69 shows a cabling schematic using direct serial connection from the TT24-7 and the PC, and the remote start connection.

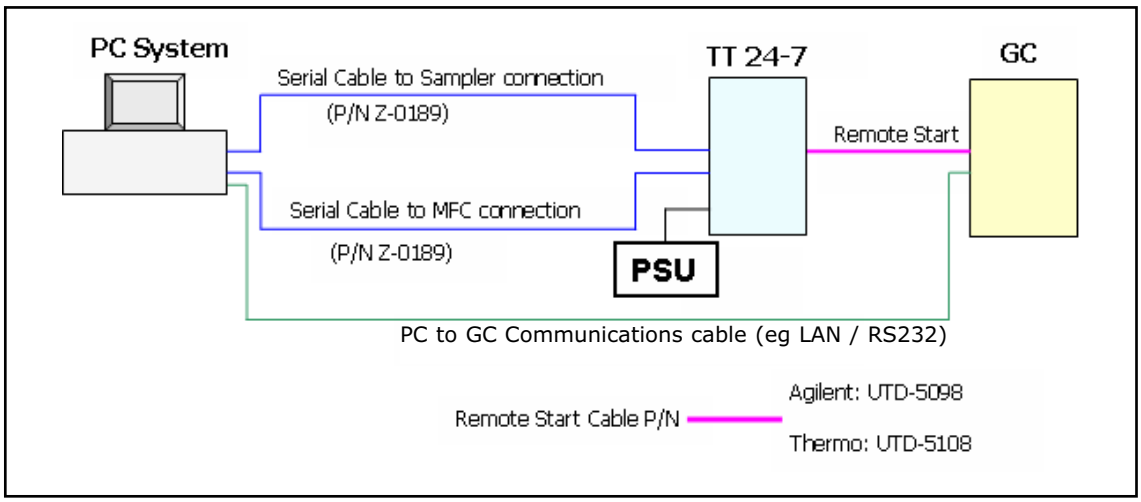

Figure 69. Direct serial connection

Figure 70 shows a similar schematic but in this case USB communication is used.

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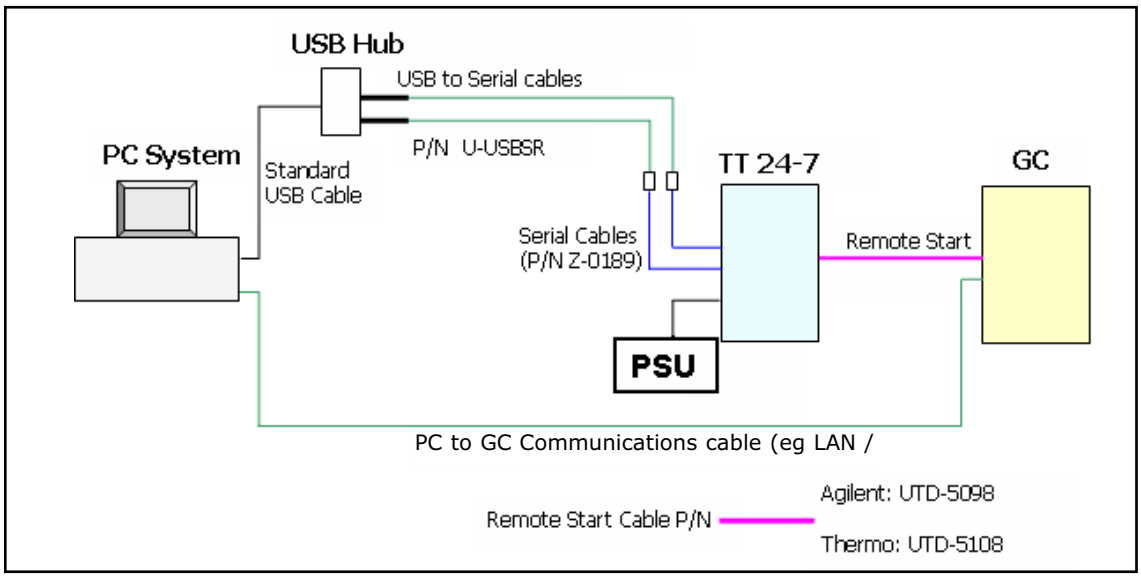

Figure 70. USB connection

It is possible to have a combined configuration in which one of the serial communication cables is connected directly to the PC from the TT24-7, and the other connects via USB.

3.1.4 Power recycle box

Remote system control of the TT24-7 and associated GC or GCMS can be very useful. Where access to the system is difficult e.g. remote location, high security, worker exposure safety etc, the ability to run and monitor the respective instruments remotely is very advantageous. This is particularly evident where the continuous sampling mode is being used.

Remote PC to PC control is achieved using commercially available software packages (an example of this is VPN).

A specific use of this is in the case of power failure. Power failures will close down both the TT24-7 and the GC system, however the controlling PC will typically have a battery backup for a few hours.

When the power returns the GC will switch on and reset to its original method values, however the TT24-7 requires the firmware to be downloaded again. This requires the TT24-7 software to be closed as it will still be active due to the battery backup in the PC. If the power failure extends beyond the battery backup of the PC, then the PC software will have to be rebooted from scratch. User name login and passwords will be required to access the Microsoft Windows desktop, followed by rebooting of both the TT24-7 and GC software.

To successfully download the firmware into the TT24-7 it is recommended that the instrument is switched OFF and then ON (at the back of the instrument). This will turn both LED lights to red as discussed in section 2.8. The TT24-7 is now ready to receive the firmware download and this is achieved by rebooting the software as described in section 2.8.

However where remote system control is active, the ON / OFF switch at the rear

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of the instrument cannot be physically accessed, so an alternative mechanism is required.

This is achieved using a power recycle box (P/N U-RCYBX). The power recycle box is activated when the TT24-7 software is initiated, this electronically switches the system OFF and then back ON. After this has occurred the firmware is automatically downloaded into the TT24-7. The desired sampling method can then be started again.

The recycle box has two power (IN / OUT) supply sockets, and two (IN / OUT) serial ports. The power supply to the TT24-7 now passes through the recycle box which in turn connects into the power socket of the TT24-7. The power cable connecting the recycle box to the TT24-7 has a female socket at either end (P/N Z-0207).

The serial port connection to the Sampler socket in the TT24-7 also passes through the recycle box and it is this connection which initiates a power cycle when the software is downloaded.

Figure 71 shows a schematic of this configuration.

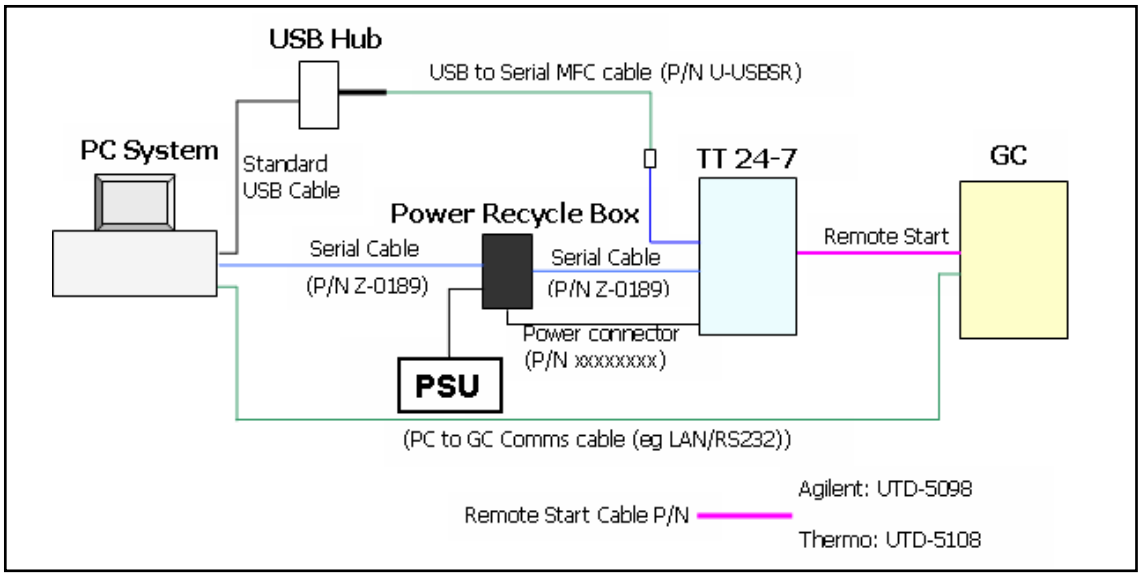

Figure 71. Cable connections including power recycle box

3.2 Gas requirements

The gases required by the TT24-7 system are

Carrier Gas

Sample gas (continuous sampling mode)

Trap box purge gas and heated valve actuator pneumatics

The quality of both the carrier gas and the purge gas is an important consideration.

For the carrier gas the quality must be 5.0 grade (99.999%) or better. This can be achieved directly from the supply tank or achieved using gas filters. Typically

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this will include a Moisture trap (P/N C-MSTRP), Oxygen trap (P/N C-O2TRP) and Hydrocarbon trap (P/N C-HCTRP).

The external supply pressure of carrier gas needs to be ~ 10 psi greater than the capillary column head pressure requirements. If the column is operating in constant pressure mode this is a simple addition, i.e. for a head pressure of 30 psi the supply pressure needs to be ~ 40 psi etc. However if constant flow conditions are being used then the supply pressure needs to be calculated at the oven maximum value when the pressure demand by the column is at its greatest. Connection of the carrier gas to the TT24-7 is discussed in section 2.4.6.1.

The trap box purge and valve actuator gas is typically dry air, nitrogen, or carrier gas (He only). This needs to be supplied at a pressure between 50 and 70 psi and must have a dew point lower than -35°C. The trap box purge gas is essential to prevent moisture condensation and subsequent icing within the Peltier cooled trap box. If this occurs, the lower (i.e. sub ambient) temperatures will not be achievable.

The dry gas supply must always be on when the TT24-7 is being used and secondary pressure regulation of this gas supply is required in addition to the laboratory control.

Markes International can supply a secondary pressure regulating accessory (P/N U-GAS01), which regulates both the dry gas and the carrier gas pressure.

The dry gas is attached at the rear of the instrument to the connector labelled "Purge Gas".

The sample gas (typically air) will contain both oxygen and moisture. The air is removed from the trap just prior to trap fire using the dry-purge / pre-purge gas flow. Moisture can also be removed by prudent selection of sorbent material, e.g. Tenax does not retain moisture (see Appendix 3). If a hydrophilic sorbent is used the water will be transferred across to the analytical system. Split mode sampling (where available with e3 systems) will reduce this volume.

3.2.1 User maintenance for gas requirements

Standard good practice should be observed for the maintenance of the gas lines serving the TT24-7 and in particular the replacement / regeneration of any filters on the system (de-oxo filters, moisture traps, hydrocarbon traps etc.). Attention should be paid to the manufacturers instructions of these filters with respect to frequency of regeneration / replacement.

3.3 Installation (and removal) of heated transfer line

3.3.1 Connecting the transfer line to the gas chromatograph

The TT24-7 is supplied with a universal transfer line to deliver desorbed analytes from TT24-7 to a gas chromatograph or other analytical system. The sample path utilises a deactivated fused silica line (0.25mm I.D. and 0.35mm O.D. (P/N UTD-5093)) heated over its entire length by means of a distributed heater and at the GC end by heat conduction from the GC oven.

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The line is 1 m long, which is sufficient to reach most gas chromatographs even when a mass spectrometer is attached.

Most GCs have access points built into the oven region by means of holes in the side, top or back of the oven skin, with "knock out" sections in the outer casing, and one of these should ideally be utilised to install the transfer line.

If all such access points are already in use it is possible to gain entry via an unused injector or detector port with or without a heated zone.

The general approach is illustrated in the three diagrams, figures 72, 73 and 74.

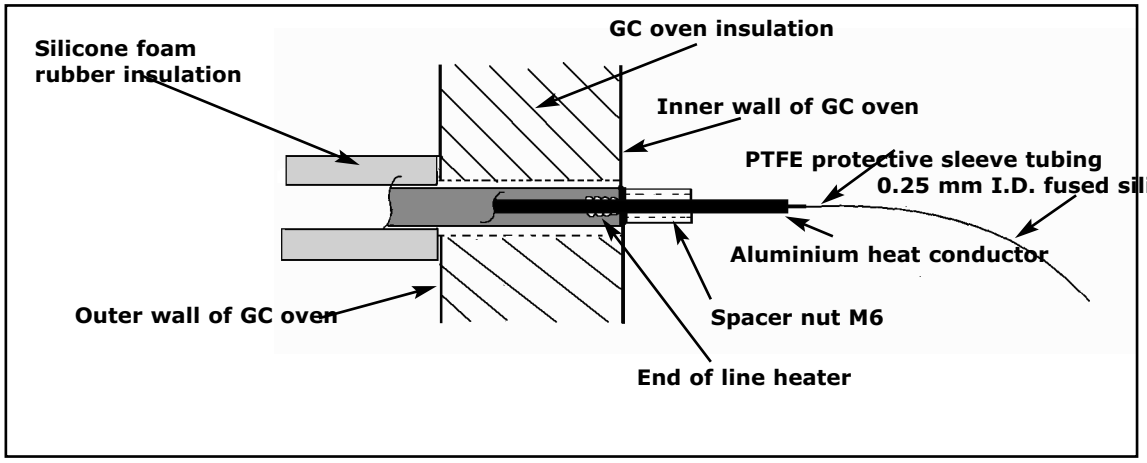

Figure 72. Installation of transfer line through inner wall of GC oven

Note: In all cases the fused silica and PTFE sleeve tubing are fitted as the final operation.

Locate a hole in the inner oven wall with a corresponding hole leading to the outside of the instrument. It is usually necessary to displace the oven insulation material to enable the flexible metal line to be pushed against the outside of the inner oven wall.

If the GC oven wall insulation is particularly thick it may be necessary to shorten the silicone foam rubber insulation sleeve, which is intended to rest against the outer wall of the GC oven.

The M6 spacer nut (attached to a ¼-inch spacer tube on the transfer line) secures the line casing to the oven wall allowing the ⅛-inch aluminium sleeve to protrude into the oven. If the hole in the inner oven wall is larger than the end of the line, fit one of the large metal washers from the shipping kit at this point.

In figure 73 the entry to the GC oven is through the fan protection grill. In this situation the ¼-inch spacer tube attached to the transfer line prior to the spacer nut is used to extend the line and a special U-shaped metal support bracket is pushed through adjacent holes in the grill to press against the oven inner wall.

Note: The line must not be secured with a nut against the fan grill as this could be distorted causing it to hit the fan.

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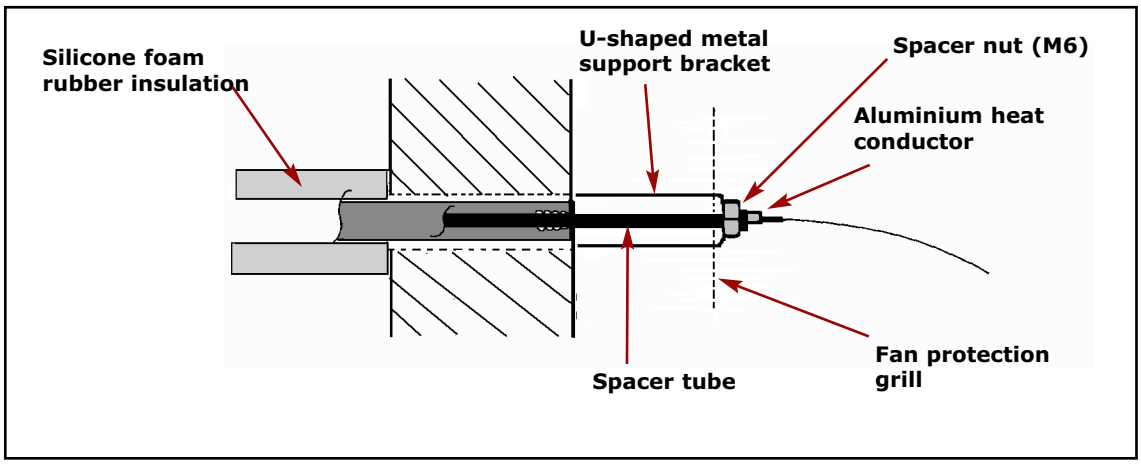

Figure 73. Installation of transfer line through fan protection grill

Figure 74 shows installation via a heated zone block. As the entry hole will generally be larger than the diameter of the metal line sleeve, one or more of the large washers supplied will be needed. If the heated zone block is particularly deep both the M6 spacer nut and spacer tube will needed as shown.

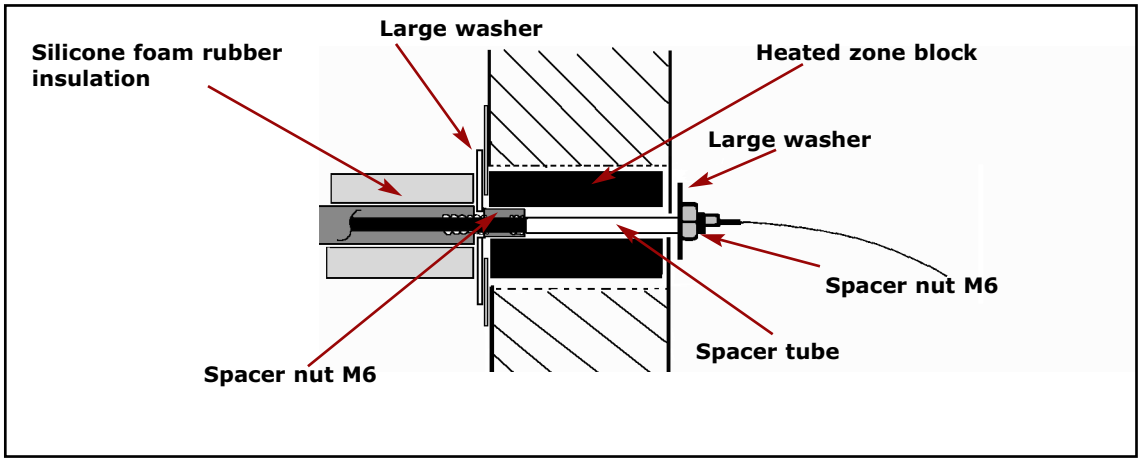

Figure 74. Installation of transfer line through heated zone block

This part of the line derives its heat from the heated zone block which should be set to run at a conveniently high temperature, preferably 50°C above the line setting but not above 250°C as the silicone foam rubber insulation will be damaged.

The parts supplied can be used in other combinations to suit particular instrument configurations.

3.3.2 Installing the fused silica transfer line insert

Once the heated line has been fitted to the GC, the fused silica plus associated PTFE sleeving (P/N UTD-5093 see Appendix 1) are pushed from the GC end, along the 1/8-inch aluminium tube until they protrude from the other (TT24-7) end of the transfer line.

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3.3.3 Connecting the transfer line to the TT24-7

Place the TT24-7 on the bench on the most convenient side of the GC. Ensure that the transfer line will reach from the back of the TT24-7 to the selected entry point into the GC oven.

3.3.3.1 e1 systems

For e1 systems the transfer line inlet is situated behind the front two heated valves and the transfer line is installed vertically down into the union.

To obtain access to the installation point, first remove the heated valve cover as follows.

Loosen the two screws holding the back panel in place (figure 75) and remove the panel.

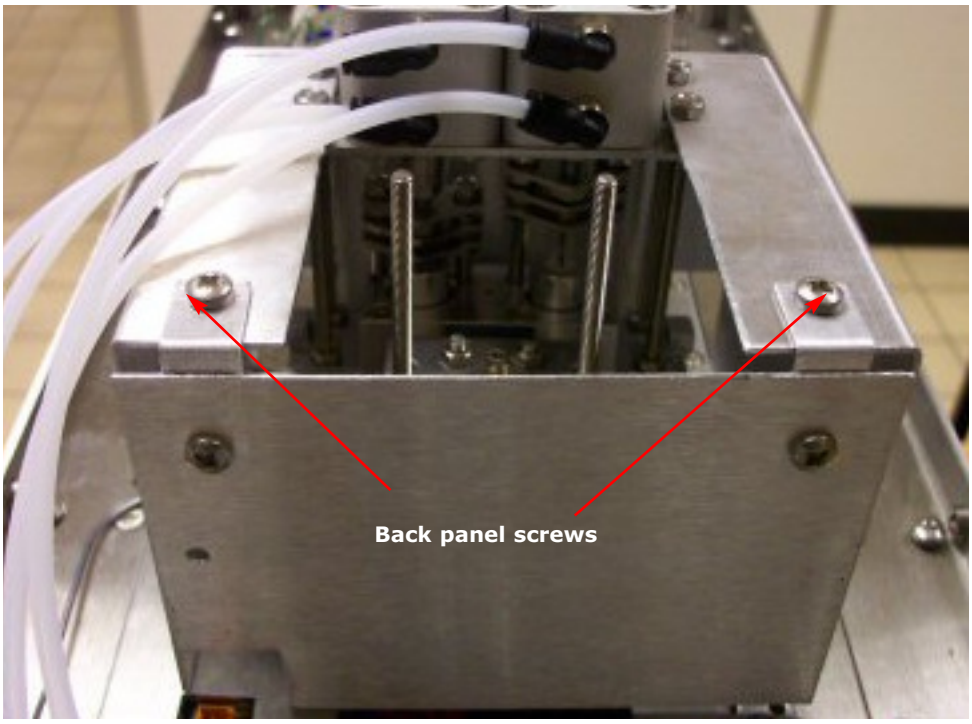

Figure 75. Loosen the back panel screws

Remove the four M4 nuts at the front of the valve box and lift the box off (figure 76)

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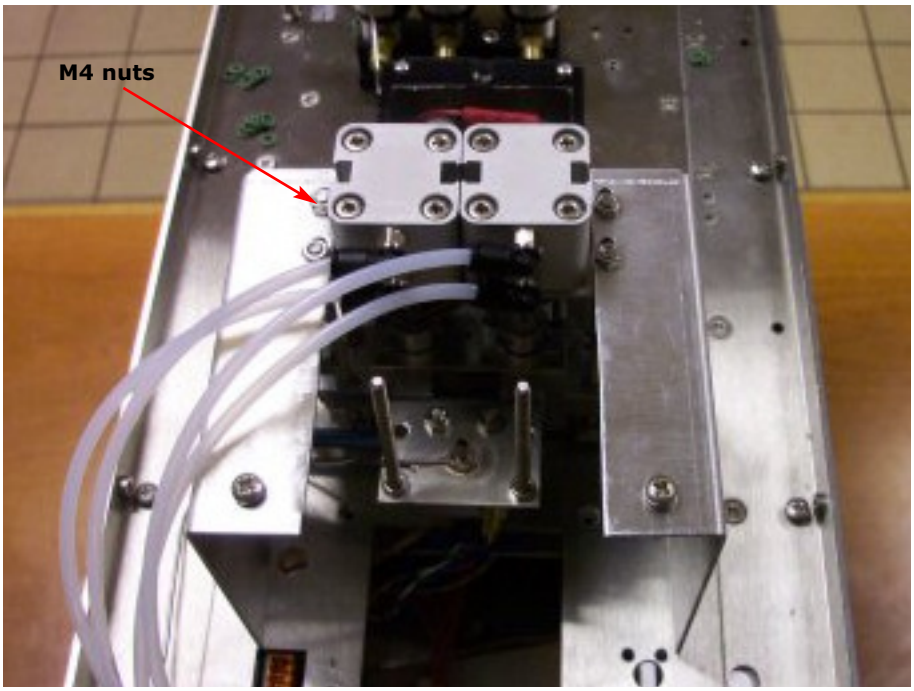

Figure 76. Remove the M4 nuts

Figure 77shows the 1/16-inch stainless steel union into which the fused silica transfer has to be fitted. Figure 78 shows a partly sectioned view of this 1/16-inch union giving an indication of where to position of the end of the fused silica tubing.

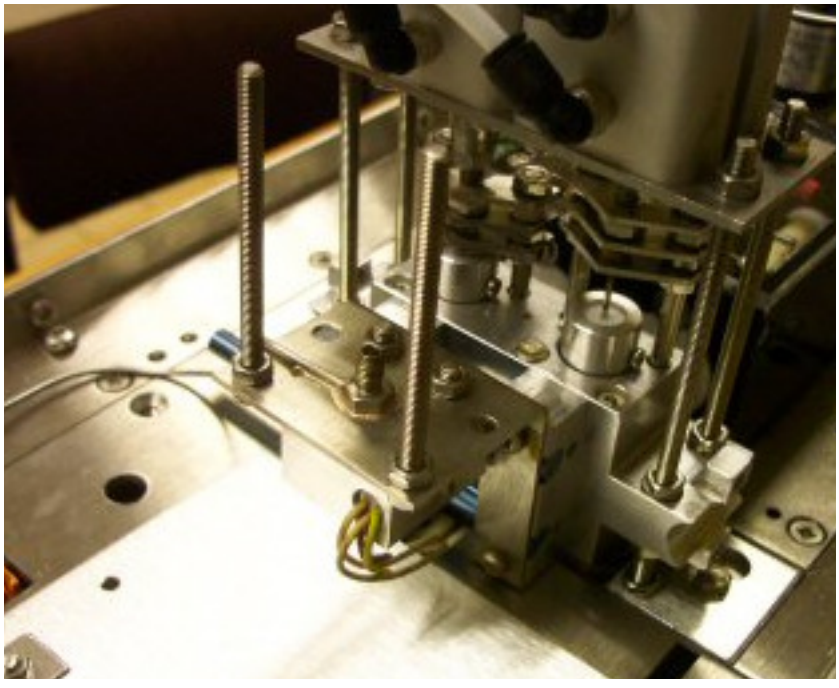

Figure 77. Transfer line connection

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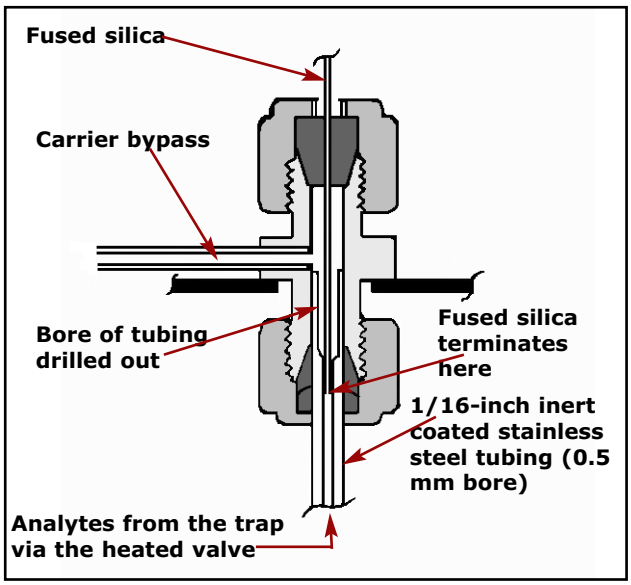

Figure 78. Precise positioning of end of fused silica transfer line

If the fused silica terminates in the space above the end of the inert coated stainless steel tube, some analytes can diffuse into the side arm causing measurable peak tailing. The 0.25 mm I.D. fused silica transfer line must be installed through the union and on into the narrow bore part of the inert coated stainless steel tube.

When connecting the transfer line to the TT24-7e1, pull about 20 cm of fused silica from the PTFE line casing. Slide a 1/16-inch stainless steel Swagelok nut and a 1/16-inch x 0.4mm ferrule (supplied with shipping kit, see Appendix 1) onto the end of the fused silica and then cut off the first few mm of fused silica. Feed the fused silica into the top of the union and slide the ferrule into position. Screw the nut onto the union and, with the fused silica still loose, position it so that the fused silica is inserted around 20 mm into the union. Tighten the nut to trap the fused silica and then tighten a further half turn using one of the 8 mm wrenches (spanners) provided in the shipping kit. Do not over-tighten or the ferrule will become distorted.

Carefully bring the clamp plate, PTFE plate and shield tube down into position (shown in figures 79 & 80) with the shield tube covering the union nut. The shield tubing should be positioned such that the 1/16-inch side tubing projects through one of the cutouts. As the transfer line is lowered into position the clamp plate and PTFE plate should fit onto the two exposed threads.

Use two M4 nuts (supplied with shipping kit) to lock the PTFE plate onto the exposed threads.

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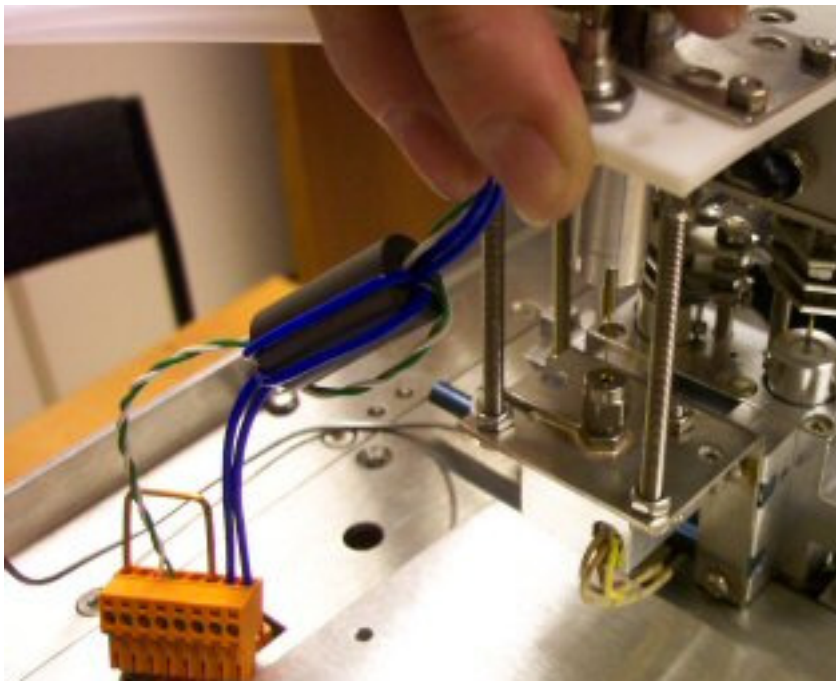

Figure 79. Moving the clamp plate, PTFE plate and shield tube down into position

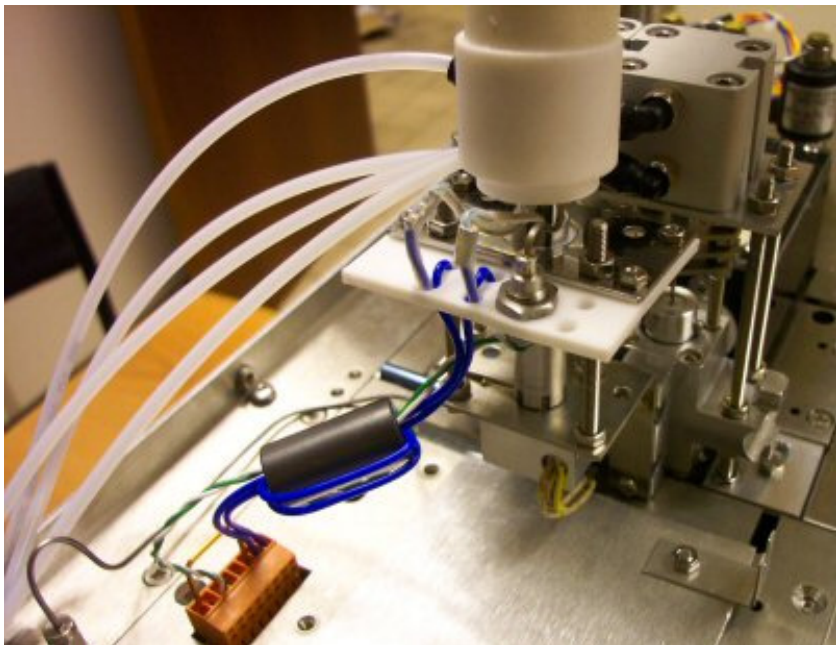

Figure 80. The transfer line installed and the 8-way orange connector plugged in

Plug the orange 8-way connector into the socket adjacent to the transfer line connection. See figure 80 for the correct orientation of this connector.

Replace the heated valve box and M4 nuts and re-fix the back panel by reversing the procedure above.

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3.3.3.2 e2/e3 systems

For e2 / e3 systems the transfer line inlet is situated at the back of the third heated valve and the transfer line is installed horizontally into this union.

Figure 81 shows the $^{1}/_{16}$-inch stainless steel union into which the fused silica transfer has to be fitted. Figure 82 shows a partly sectioned view of this $^{1}/_{16}$-inch union giving an indication of where to position of the end of the fused silica tubing.

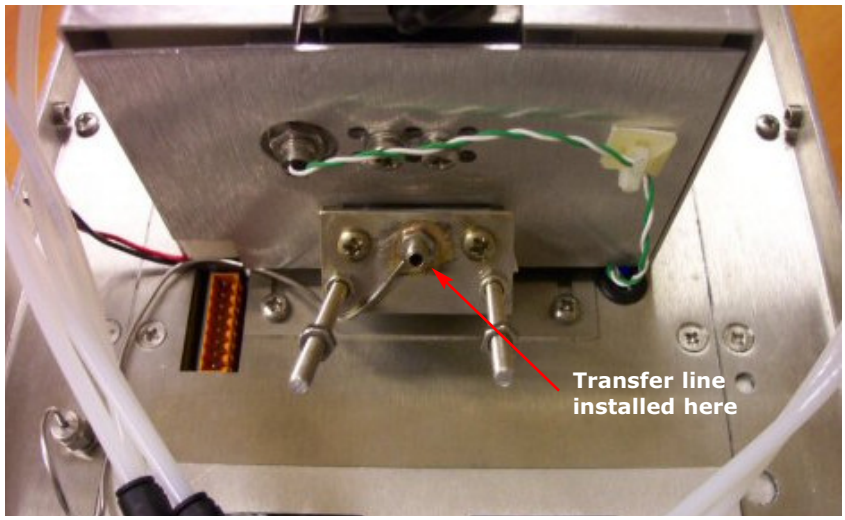

Figure 81. Installation of fused silica tubing

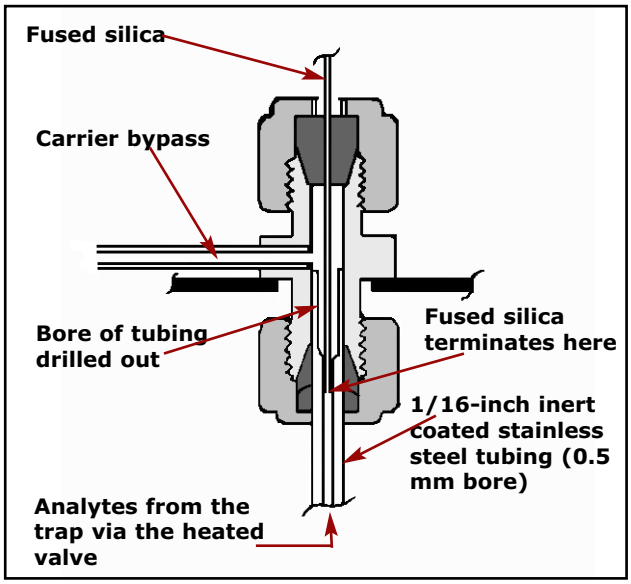

Figure 82. Precise positioning of end of fused silica transfer line

If the fused silica terminates in the space above the end of the inert coated stainless steel tube, some analytes can diffuse into the side arm causing measurable peak tailing. The 0.25 mm I.D. fused silica transfer line must be installed through the union and on into the narrow bore part of the inert coated stainless steel tube.

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When connecting the transfer line to the TT24-7e2/e3, pull about 20 cm of fused silica from the PTFE line casing. Slide a 1/16-inch stainless steel Swagelok nut and a 1/16-inch x 0.4mm ferrule (supplied with shipping kit, see Appendix 1) onto the end of the fused silica and then cut off the first few mm of fused silica. Feed the fused silica into the top of the union and slide the ferrule into position. Screw the nut onto the union and, with the fused silica still loose, position it so that the fused silica is inserted 50 mm into the union. Tighten the nut to trap the fused silica and then tighten a further half turn using one of the 8 mm wrenches (spanners) provided in the shipping kit. Do not over-tighten or the ferrule will become distorted.

Carefully bring the clamp plate, PTFE plate and shield tube down into position (shown in figures 83 & 84) with the shield tube covering the union nut. The shield tubing should be positioned such that the 1/16-inch side tubing projects through one of the cutouts. As the transfer line is lowered into position the clamp plate and PTFE plate should fit onto the two exposed threads.

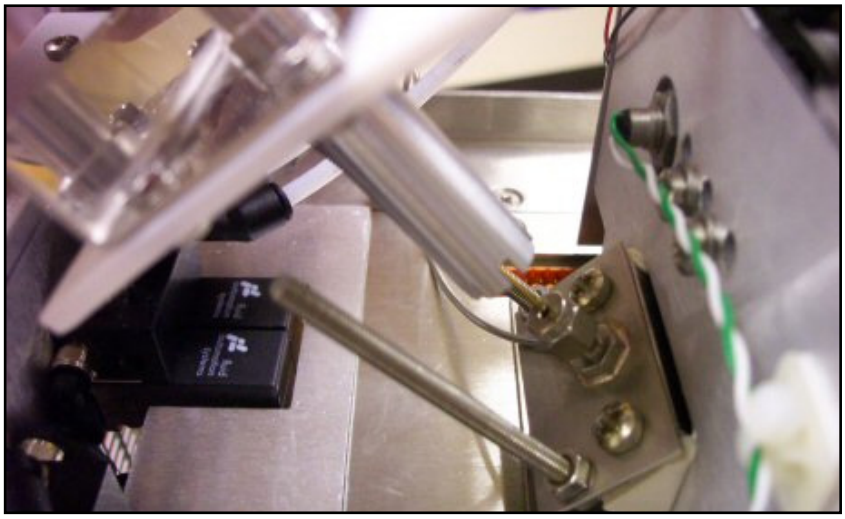

Figure 83. Aligning the clamp plate, PTFE plate and shield tube into position

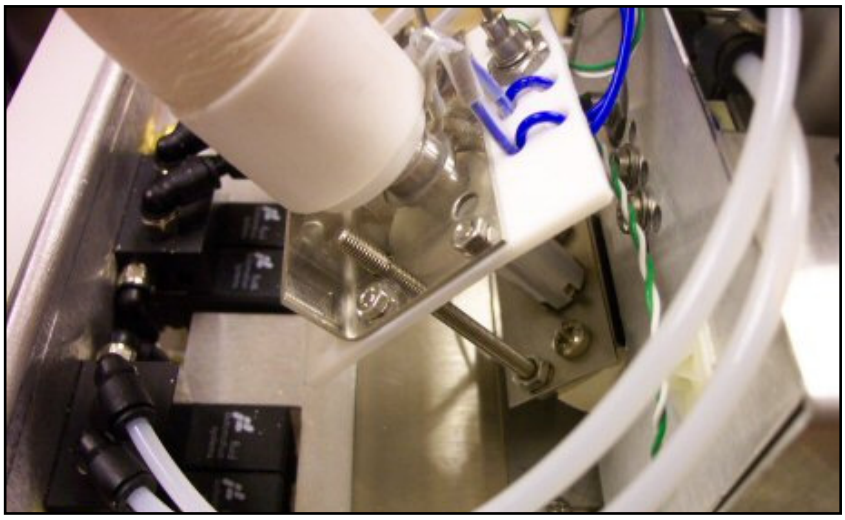

Figure 84. Clamp plate, PTFE plate and shield tube in position

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Use two M4 nuts (supplied with shipping kit) to lock the PTFE plate onto the exposed threads.

Plug the orange 8-way connector into the socket adjacent to the transfer line connection. See figure 85 for the correct orientation of this connector.

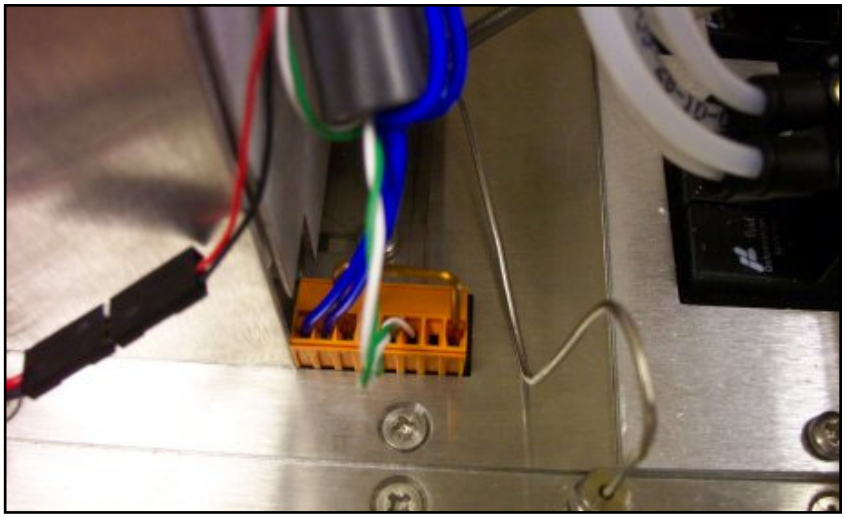

Figure 85. Plugging in the 8-way connector

3.3.4 Coupling the fused silica transfer line to the GC column

Return to the GC and connect the column to the end of the transfer line using the quick seal column connector provided with the shipping kit. Instructions are included with the connectors. Alternatively you may use a 1/16-inch zero dead volume connector with the appropriate ferrules.

3.3.5 User maintenance of transfer line

The quick seal column connector should be replaced every time the seal between the fused silica transfer line and the GC column is broken. If a zero dead volume connector is being used the ferrules should be replaced as necessary when the connection is broken.

The fused silica transfer line itself (and its accompanying PTFE sleeve) should be replaced as part of a scheduled program of maintenance. Frequency of changing the transfer line will depend entirely on the usage level of the system and the nature of the compounds being analysed. In any event it is advised to replace the fused silica transfer line at least annually.

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3.4 Installation and removal of Cold Traps

Note: Never turn on the power to TT24-7 without the cold traps installed

3.4.1 Installing cold traps

Refer to Figure 86.

- Loosen the locating screw on the trap pneumatics before sliding the pneumatics forwards.
- Slide the narrow bore end of the cold traps into the trap box until they touch the o-rings in the heated valves (3.5 x 1.5 mm o-ring- P/N U-COV35).
- Switch on the instrument and download the firmware (see section 2.8).
- Allow the heated valves to reach their temperature set-point.
- Push the cold traps the remaining 2-3 mm into the heated valve o-rings.
- Fit the spacer o-rings (P/N U-COV39) onto the end of the trap.
  - For collared traps use two o-rings pushed up against the collar
  - For old-style uncollared traps use a sufficient number of o-rings to fill the space between the trap valve connector and the trap guides (see figure 86)
- Replace the trap pneumatics at the front of the instrument and carefully slide the pneumatics towards the traps until the trap valve connectors (P/N TTD-5032) have sealed onto the traps.

Note: It is easier to locate the trap tube links on the cold traps if they are gently rotated as they are pushed onto the cold traps.

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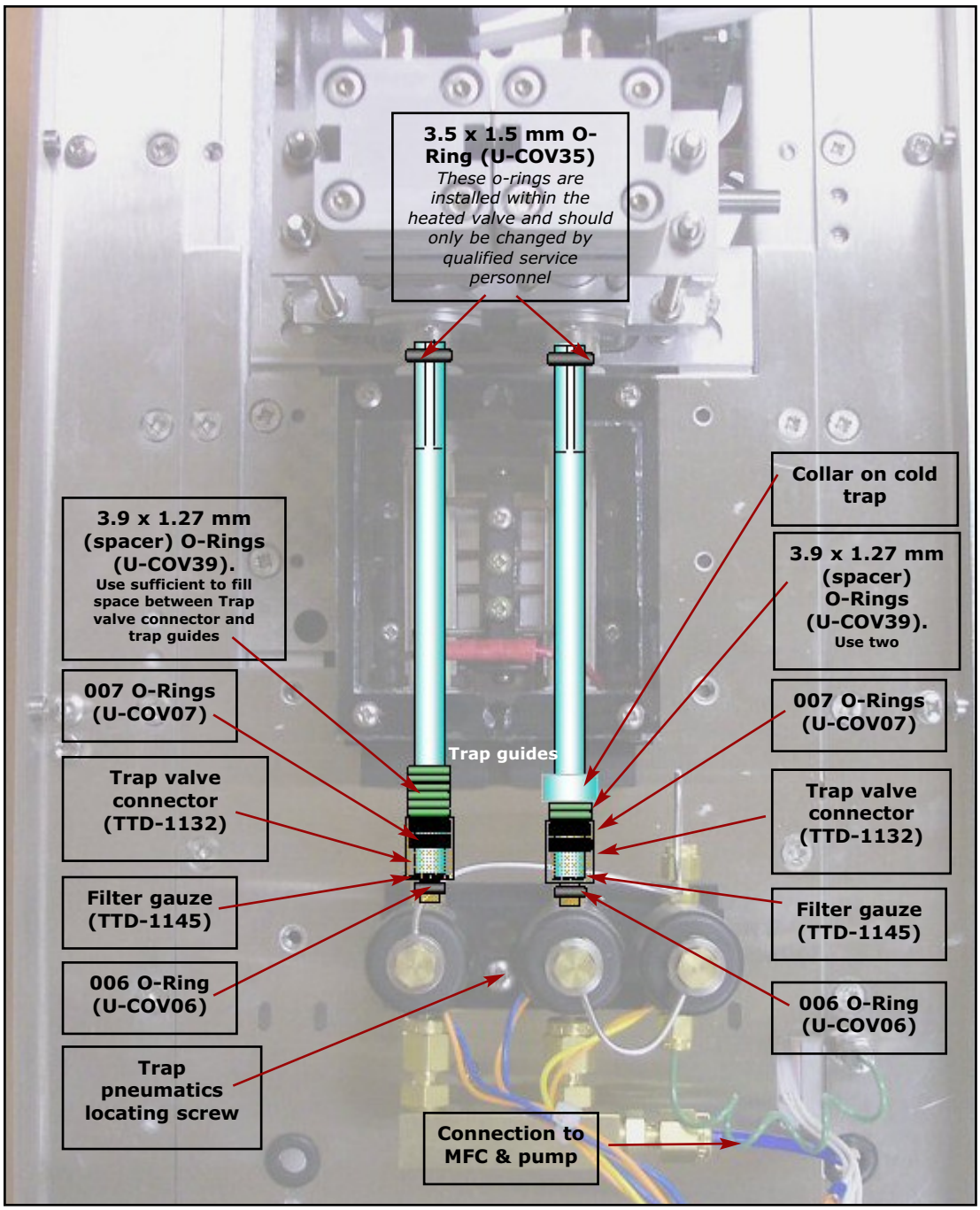

Figure 86. TT24-7e1 with overlaid schematic showing trap positions and user serviceable parts

3.4.2 Removing the cold traps

Refer to Figure 86

- Ensure that the instrument is in standby.
- Remove the trap pneumatics by loosening the locating screw and carefully sliding the pneumatics forward.

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Note: Take care not to rotate the trap pneumatics as they are moved to avoid breaking the cold traps.

- Once clear of the cold traps lift the trap pneumatics clear of the traps and move to the RHS of the instrument.
- Close the TT24-7 software.
- Switch the instrument off.

Collared Traps

- Place the trap extraction tool (TTD-5032) over the cold trap so that the notch of the tool sits directly behind the cold trap collar.
- While holding the tool in place begin to unsdrew the knob at the front of thre trap extraction tool which will extract the cold trap from the heated valve o-ring.
- Once fully unscrewed the knob can then be pulled forward to extract the cold trap from its housing.

Note: Cold traps are much easier to remove whilst the o-rings in the heated valves are still hot.

Uncollared Traps

- Carefully pull each of the cold traps out

Note: Using a latex glove will provide better grip on the traps to aid removal.

Note: Cold traps are much easier to remove whilst the o-rings in the heated valves are still hot

- If the cold traps remain difficult to pull out then switch the instrument back on and re-download the firmware, allow the heated valves to attain their temperature set-point, switch the instrument off and repeat the process.

3.4.3 Available Cold Traps

A range of pre-packed TT24-7 cold traps are available from Markes as follows:

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Part No	Description
T-1VX	TT24-7 cold trap optimised for the sampling of CW agents (specifically VX)
T-2HD	TT24-7 cold trap optimised for the sampling of CW agents (specifically Mustard (HD))
T-3GBGE	TT24-7 cold trap optimised for the sampling of CW agents (specifically Sarin (GB) and the G-analogue of VX (GE))
T-6EMP	TT24-7 empty cold trap for packing by user
T-7CUS	TT24-7 cold trap custom packed to user specification

3.4.4 User maintenance of TT24-7 cold traps

The quartz cold traps are fragile and packing them is a skilled task that should be undertaken with care. Both traps in the TT24-7 should be identically packed and should be flow tested prior to use to ensure that they have similar impedences. For these reasons we recommend the use of Markes pre-packed TT24-7 cold traps, either one of the application specific traps, or cold traps which are custom packed for your specific application.

3.4.4.1 Packing cold traps

If you wish to pack your own traps Markes supply empty traps for this purpose. The traps should be packed from the wider bore end using the following procedure.

Insert a small plug of quartz or glass wool (depending on the application), using a suitable flexible tool.

Pour in the required amount of sorbent(s).

If multiple sorbents are to be used, the weakest sorbent should be inserted first, followed by the stronger sorbent. Sorbents should be separated by small plugs (~2 mm) of quartz / glass wool as above.

Finish the trap with a 5 mm plug of quartz / glass wool.

A 6 cm length of the trap, measured from the point of bore restriction, is subjected to full heating / cooling power. All the trap packing / wool plugs (except the final wool plug) should be within this 6 cm length.

Note: Care should be taken when using tools / funnels etc. to insert wool and sorbent that the edge surface of the traps are not damaged or chipped in any way as this will prevent them sealing correctly and the TT24-7 will not function.

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3.4.4.2 Cold trap lifetime

Cold trap lifetime is dependent upon a number of factors.

- the sorbent(s) type used in the trap (porous polymers have a lower lifetime than graphitised carbon sorbents for example)

- the maximum temperature that the trap is routinely heated to during trap fire, and the length of time it is held at this temperature (operating your traps close to the maximum temperature of the sorbents within them will reduce trap lifetime)

- the nature of the compounds being analysed

When the performance of the trap starts to deteriorate it is time to replace **both** traps.

In any event it is recommended that cold traps are replaced annually.

3.5 Installation / removal of sampling tube (e2/e3)

The sampling tube is located on the left hand side of the TT24-7 when looked at from the front of the instrument (figure 87).

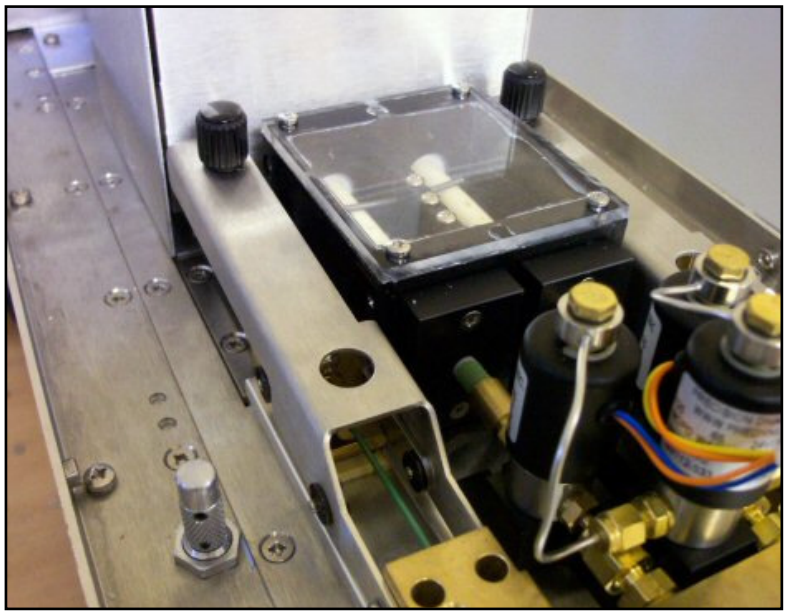

Figure 87. TT24-7e2/e3 system with Desorb Tube option on LHS

To access the tube oven which houses the sampling tube the sealing mechanism lever should be lifted (see figure 88).

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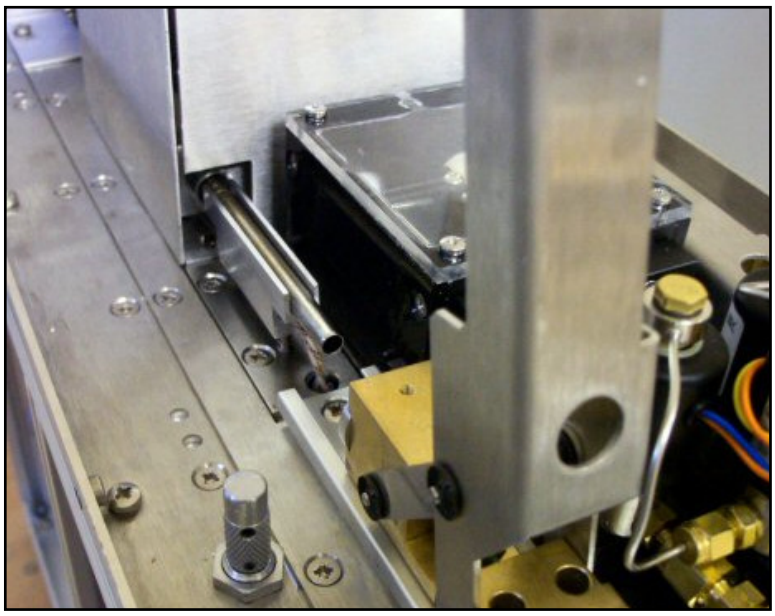

Figure 88. TT24-7e2/e3 system - tube sealing mechanism lever lifted up

With the lever in the up position a tube can be positioned into the oven. It is important that the tube is orientated correctly (the grooved end of the tube should be towards the back of the instrument). With the tube in place the sealing mechanism lever can be lowered which seals the tube into the flow path. When sealing the tube into place take care that the tube is aligned with the sealing o-rings to prevent any o-ring damage being incurred. Any damage to the o-rings could lead to a tube leak test failure.

Removal of the tube is a very similar process. First, lift the sealing mechanism lever to provide access to the tube and then pull the tube out by hand from the sealing o-ring (see figure 89). If the tube is difficult to extract from the sealing o-ring then the tube extractor can be used to provide extra grip.

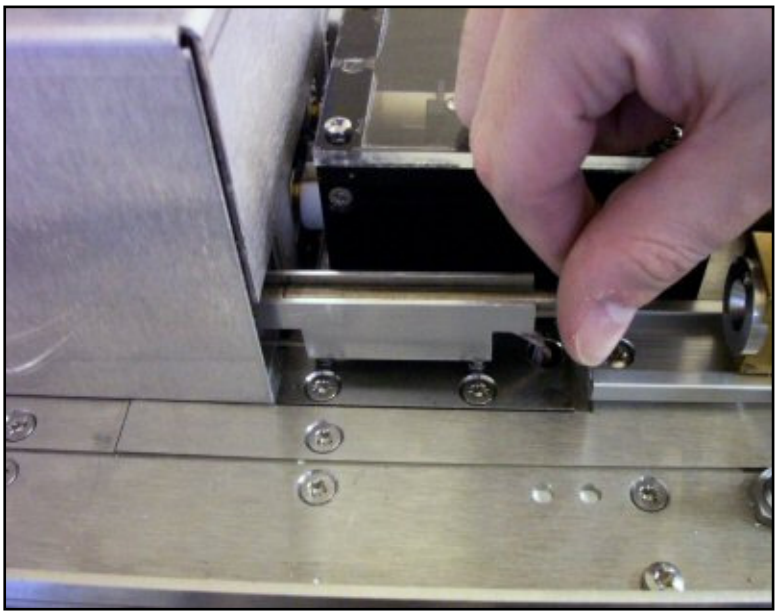

Figure 89. TT24-7e2/e3 system removing desorb tube

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3.6 Installation / removal of split tube (e3)

The split tube is located on the right hand side of the TT24-7 when looked at from the front of the instrument (figure 93). The split tube is either a charcoal split tube which traps any split volatiles and prevents them from being passed back into the laboratory atmosphere. Or alternatively, when in Tube Desorb mode, the split tube can be replaced by a conditioned sorbent tube and used to re-collect the split portion of the sample for method / data validation and repeat analysis (SecureTD-Q™). For further information regarding SecureTD-Q and sample re-collection please see the Markes International brochure "Validation of Thermal Desorption featuring SecureTD-Q - Quantitative sample re-collection for thermal desorption".

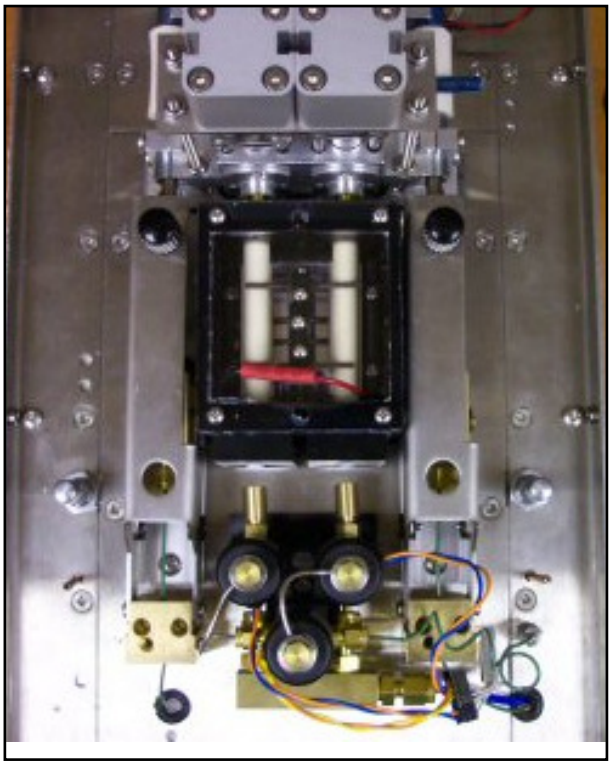

Figure 90. TT24-7e2/e3 system with Split tube option on RHS

To access the split tube the sealing mechanism lever should be lifted and the tube inserted / removed exactly as described for the sample tube above.

4.0 Troubleshooting

4.1 Leak testing the system

In any gas flow path leaks may occur due to worn or ill fitting o-rings, fittings etc. The TT24-7 flow path can be leak tested using the flow only diagnostics facility (see section 2.9.1.6 / 2.9.2.6). In order to leak test successfully, a leak detection system such as a helium leak detector (Markes p/n C-HEL23 / C-HEL11) should be used.

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Note: "wet" methods of leak testing (e.g. with bubble solution, SNOOP etc.) should be avoided at all costs.

Using diagnostics, individual valves may be switched in order to isolate different parts of the flow path and the helium leak detector applied to the appropriate connection point to ascertain if there is a gas leak.

In addition to the flow diagnostics, the TT24-7 software can perform a manual leak test of desorption tube, trap A and trap B by clicking on the appropriate icon in the software (see section 2.9.2.4.1).

4.2 Contamination - the presence of artifacts in the chromatogram

Artifacts are usually the result of either insufficient conditioning of sorbent from the cold traps (or sorbent tube where applicable (e2/e3 systems)), or contamination from the carrier gas or carrier gas supply equipment.

4.2.1 The carrier gas supply

The carrier gas supply is a common source of contamination in thermal desorption. Contaminants may derive from the gas itself, cylinder head regulators, gas lines or carrier gas filters.

If the contamination is of high boiling compounds then use the following procedure to establish whether the gas supply is at fault.

Immediately after a GC run, cool the GC oven down to ambient temperature and leave the TT24-7 in standby for a period of time (e.g. 10 minutes).

Carrier gas will now be passing directly into the GC column and because the column is at ambient temperature any high boiling contamination will be focusing on the front of the column.

After the selected time period, start the GC run by manually pressing RUN on the GC system.

Note the background contamination which is obtained.

Repeat the experiment but this time double the time period that the system is left in standby (e.g. 20 minutes).

Note the background contamination which is obtained.

If the contamination increases with the length of time that the instrument is in standby then the carrier gas supply is likely to be contaminated and individual components of the supply system should be checked.

If the contamination is of more volatile components then you will need to sample your carrier gas via the TT24-7 system itself using the following procedure.

Disconnect the carrier gas supply from the rear of the TT24-7 and attach a (clean) T-piece and short length of tubing so that the carrier gas can now be attached to both the rear of the TT24-7 and to the sample inlet of the TT24-7.

Set up a continuous sampling run on the TT24-7 to sample the carrier gas

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for a specified time period (e.g. 10 minutes).

Note the background contamination which is obtained

Repeat the above procedure but this time double the time period that the system is left in standby (e.g. 20 minutes).

Note the background contamination which is obtained.

If the contamination increases with the length of time that the instrument is in standby then the carrier gas supply is likely to be contaminated and individual components of the supply system should be checked.

To reduce the possibility of carrier gas contamination, ideally, each TT24-7 system should have its own independent carrier gas supply, separate from any other conventional chromatographs in the laboratory.

Note: TT24-7 is such a good concentrator of VOCs that normal laboratory gas lines, which perform perfectly well for conventional GC analyses, can produce artifacts on the system. It is recommended that the gas itself and gas line components meet the requirements stated in section 3.2.

4.2.2 Contamination from the cold trap

If the contamination is shown, from the experiment described above, not to be coming from the carrier gas, the next most likely candidate is the sorbent in the traps (or sorbent tube where applicable (e2/e3 systems)).

See Appendix 3 for information regarding sorbent conditioning / maximum sorbent temperatures etc.

To condition the traps set up a continuous sampling method with the following parameters:

- an extended pre-purge time (e.g. 5 minutes).

- a short sampling time (e.g. 30 seconds).

- a maximum trap temperature which is 10 - 20°C higher than that normally used for analysis - assuming that this does not exceed the sorbent maximum temperature (see Appendix 3) (typically to extend trap lifetime, your operating method should not require the trap to be used at its **maximum** temperature).

- an extended trap hold time (e.g. 5 minutes).

- run this method through both traps.

- you may need to run the method through each trap more than once depending on the contamination levels found.

4.2.3 Contamination from the sorbent tubes (e2/e3 systems)

If the contamination is shown, from the experiments described above, not to be coming from the carrier gas or the sorbent in the traps, the next most likely candidate is the sorbent in the tubes (e2/e3 systems).

Sorbent tubes should be thoroughly conditioned prior to use. Ideally - and

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certainly before use for the first time - this should be done **off-line** to avoid contaminating the TT24-7 flow path. This can be done using a tube conditioner such as the TC-20 from Markes International. Sorbent tubes should be conditioned at temperatures approximately 20 - 40 °C higher than those used for desorption of the samples - **assuming** that this does not exceed the sorbent maximum temperature. See Appendix 3 for information regarding sorbent conditioning / maximum sorbent temperatures etc.

4.2.4 Other potential sources of contamination

Unsilanized glass or quartz wool should be used as standard in the cold traps and should be conditioned at high temperatures before use. Silanized glass wool can be used but is only recommended for the analysis of labile compounds.

Note: NEVER heat silanized glass wool above 250°C, even during system conditioning, as the silylating reagent will break down and may irreversibly contaminate your sample flow path.

4.3 Poor peak shape / peak splitting

Peak broadening, particularly of early eluting components, is often an early indication that the cold trap packing needs changing. The trap sorbent is subjected to rapid heating during the analysis of every sample and should therefore be replaced regularly.

Normal aging or the desorption of samples containing aggressive compounds can produce activity in the transfer line or in the analytical column itself. This results in peak broadening or tailing. If this occurs, the capillary column or the fused silica insert inside the transfer line should be replaced.

A poor connection between the transfer line and the analytical column will also distort peak shapes. To avoid this, the connecting ends of both the column and the transfer line should be cut cleanly using a fused silica column cutting tool. The union or connector assembly should be a quartz quick seal connector or an inert, zero dead volume fitting recommended for butt connecting capillary tubing.

Broad peaks can also result from the selection of too strong an adsorbent in the cold trap or from low carrier gas flows through the trap during desorption. The gas flow through the cold trap during secondary desorption (i.e. the column flow) should be at least 3 ml/min for optimum peak widths.

If a cold trap is loaded with relatively large quantities (>1 mg) of water or solvent, flash vapourisation of the solvent or water may result in a temporary pressure surge causing peak splitting or discrimination as seen on conventional GC injectors. In these cases, reduce the amount of water or solvent retained by the cold trap (e.g by raising the cold trap temperature or by using a longer pre-purge time).

If the GC analytical column is overloaded this will cause band broadening. High resolution capillary columns work at optimum with analyte masses in the order of 20-200 ng.

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4.4 Carryover of components of interest

Carryover is usually caused by incomplete desorption and is usually addressed either by using more stringent desorption conditions (higher trap desorption temperature where possible or longer trap hold time), or by selecting a weaker sorbent for collecting the samples.

The gas flow rate is also critical and should be at least 3 mL/min.

If the sorbent in the trap is over-heated in error, this may have caused high boiling materials to deposit in the internal flow-path of the TT24-7.

Note: If you exceed the maximum sorbent temperature, the resulting breakdown of the sorbent may severely contaminate the flow path of your system. This may require the complete replacement of the flow path and associated components by a fully qualified service engineer and would not be covered by the instrument warranty.

4.5 Poor recovery / loss of sample

Recovery of labile components may often be improved by increasing the trap hold time and column gas flow rate while reducing the desorption and flow path temperatures. Many volatile labile analytes will pass successfully through TT24-7 with flow path temperatures as low as 50°C.

For the analysis of extremely labile, relatively involatile components (bp> n-C12), silanized glass or quartz wool alone should be used as the cold trap packing material where possible.

If a multibed cold trap is being used, ensure that the different sorbents are kept in discrete beds separated by unsilanised glass wool / quartz wool plugs and arranged in order of increasing sorbent strength - i.e. weak to strong from the sampling .

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Appendix 1. Packing List and Routine Maintenance Spares

The following items are included with your TT24-7 system, please check carefully and inform your distributor if there are any shortages. Items marked with a * are consumable items and may require changing at regular intervals - the commercial re-order number is given in the description and detailed in the routine maintenance spares section.

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Packing List - common parts (e1, e2 and e3 systems)

Part No	Description	Qty
SERUTD-1125	Autosystem clamp	1
SERTTD-1085	Tool Kit (TT24-7) consisting of: 2 x 7/16" x 1/2" wrench, 1 x posidriver No 2., 2 x 7 mm x 8 mm wrench	1
SERUTD-5093	Fused silica transfer-line insert 1.5 m & PTFE sleeve	1
SERZ-0050	Union reducer 4 mm x 1/8" brass	1
SERZ-0055	Tubing plastic 4 mm	1m
U-COV39	Pk 10 Size 3.9 mm x 1.27 mm O-Ring	2
U-COV06	Pk 10 Size 006 O-Ring	1
C-QSC10	Pk 10 Quick Seal connector & instructions	2/10
SERZ-0145	Tube copper 1/8" x 3 m	1
SERZ-0157	Nut 1/16" St st Swagelok	1
SERZ-0189	GC PCB Interface cable	2
U-FV001	Pk 10 Ferrule 1/16" Graph Vesp 0.4 mm	2/10
SERZ-0372	Washer 1/4" x 1 1/2"	2
SERZ-0371	Washer 1/4" x 1"	2
SERZ-0449	Washer 1/4" x 2"	1
SERZ-NM4FSS	Nut M4 St St	4
SERZ-0533	Latex Glove	2
SERZ-0567F	1/4" Elbow Assembly Coated	1
SERZ-WM3CSS	M3 Washer Crinkle St St	2
SERZ-SM308PPSS	M3 x 8 Pozi Pan St St	2
U-COV07	Pk 10 Size 007 O-Ring	1
SERZ-0026	Union brass 1/8" x 1/8"	2
U-FV003	Pk 10 Ferrule 1/8"x 1/16"	5/10
SERZ-0108	Tube PEEK 1/16" OD x 0.03" bore	3m
U-FV005	Pk 10 Ferrule 1/8" x 2 mm Graph Vesp	1/10

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Packing Lists - GC Cable Options

Your TT24-7 system comes with one of the following cable options:

SERUTD-5098 PCB GC Interface for **Agilent 6890/6850** GC

or

SERUTD-5095 PCB GC Interface - general purpose

or

TGK-6000 Thermo GC Installation Kit including:

SERUTD-5108	PCB GC Interface for **Thermo GC**	
SERTTD-1109	Transfer line termination	x 1
SERZ-0119	Brass union $^{1}/_{16}$"-$^{1}/_{16}$"	x 1
SERZ-0401	Plug cap $^{1}/_{16}$"	x 1
U-COV08	Pk 10 Size 008 O-Ring	x $^{2}/_{10}$
SERUTD-1036	8 mm Retaining ring	x 2

Packing Lists - e1 system specific parts

Part No	Description	Qty
QUI-1016	TT24-7 User Manual	1
QUI-1000	TT24-7 software CD	1
PUB-0001	Brochure pack	1
QQR-0024	Installation report back form	1
SERTTD-5008	Transfer line	1
SERZ-0024	Mains cable	1
ITS015	Quick Start Guide for TT24-7 software	1
SERTTD-1029	e1 Inlet support bracket	1
ITS012	TT24-7e1 Quick Reference Guide	1
	TT24-7 Trap tube packed with sorbent & certificate	1

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Packing Lists - e2 specific parts

Part No	Description	Qty
QUI-1016	TT24-7 User Manual	1
QUI-1000	TT24-7 software CD	1
PUB-0001	Brochure pack	1
QQR-0024	Installation report back form	1
SERTTD-5008	Transfer line	1
SERZ-0024	Mains cable	1
ITS015	Quick Start Guide for TT24-7 software	1
SERTTD-1044	e2/e3 Inlet support bracket	1
ITS013	TT24-7e2 Quick Reference Guide	1
	TT24-7 Trap tube packed with sorbent & certificate	1
SERUTD-5105	Conditioned Tenax Sampling tube with Brass Caps	1
U-DISK3	Pk 10 Disc Sintered PTFE 6.3 mm	1
U-DISK1	Pk 10 Disc Sintered PTFE 5.1 mm	1
U-COV10	Pk 10 Size 010 O-Ring	1
SERZ-0285	O Ring Insertion Tool	1
SERZ-0351	O Ring Extraction Tool	1
SERUTD-5062	Tube Extractor	1

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Packing Lists - e3 specific parts

Part No	Description	Qty
QUI-1016	TT24-7 User Manual	1
QUI-1000	TT24-7 software CD	1
PUB-0001	Brochure pack	1
QQR-0024	Installation report back form	1
SERTTD-5008	Transfer line	1
SERZ-0024	Mains cable	1
ITS015	Quick Start Guide for TT24-7 software	1
SERTTD-1044	e2/e3 Inlet support bracket	1
ITS014	TT24-7e3 Quick Reference Guide	1
	TT24-7 Trap tube packed with sorbent & certificate	1
SERUTD-5105	Conditioned Tenax Sampling tube with Brass Caps	1
U-DISK3	Pk 10 Disc Sintered PTFE 6.3 mm	1
U-DISK1	Pk 10 Disc Sintered PTFE 5.1 mm	1
U-COV10	Pk 10 Size 010 O-Ring	1
SERZ-0285	O Ring Insertion Tool	1
SERZ-0351	O Ring Extraction Tool	1
SERUTD-5062	Tube Extractor	1
SERUTD-5065	Split filter tube packed	1

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Routine Maintenance Spares - common parts (e1, e2 and e3 systems)

Part Number	Description
U-COV06	Pack 10 006 o-rings (cold trap assembly)
U-COV07	Pack 10 007 o-rings (cold trap assembly)
U-COV39	Pack 10 3.9 mm x 1.27 mm o-rings (cold trap ass'y)
U-FV001	Pk 10 ferrule, 1/16" graphitized vespel, 0.4 mm id hole (to connect fused silica transfer line to TT24-7
U-FV002	Pk 10 ferrules, 1/16" x 1/16" graphitized vespel (use with PEEK tubing i.e. connection of carrier gas lines.)
U-FV003	Pk 10 ferrules, 1/8" x 1/16" graphitized vespel
U-FV005	Pk 10 ferrules, 1/8" graphitized vespel, 2 mm id hole
TTD-1132	Trap valve connector
UTD-5093	0.25 mm id uncoated, deactivated fused silica transfer line insert with PTFE sleeve
C-PEEKG	1 m green PEEK tubing
C-QSC10	Pk 10 Universal glass quick seal connectors
T-1VX	TT24-7 cold trap optimised for high boiling point reactive compounds such as the chemical warfare Agent VX
T-2HD	TT24-7 cold trap optimised for the chemical warfare agent mustard (HD)
T-3GBGE	TT24-7 cold trap optimised for the more volatile chemical warfare agents such as GB and GE (the G-analog of VX)
T-6EMP	TT24-7 empty cold trap
T-7CUS	TT24-7 cold trap custom packed to your specification

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Appendix 2: Dynamic Data Exchange (DDE) software control

Dynamic data exchange (DDE) provides a mechanism to control and monitor the TT24-7 externally to the standard system software. The section below supplies the control and status commands to achieve this.

DDE commands are typically embedded in macro programs within the (GC / GCMS) host software, and enable that software to take control of the TT24-7. This is usually implemented to add some custom functionality to the system. An example of this would be to modify a method parameter or entire method based on an analytical result.

The DDE functionality is designed for customers with a strong background in software programming.

TT24-7 DDE Interface.

To create the connection to the DDE interface you will need to use the following settings:

Service Name:	Topcat
Topic:	DDEInterface

Information Requests

The following information can be returned from the TT24-7 software.

The Request item names are case sensitive.

State

Returns: A comma separated list of values in the order shown below

<mode>	Operating Mode
<state>	Standby, Running or LeakTest
<TAL>	Trap A Leak Error (1 or 0)
<TBL>	Trap B Leak Error (1 or 0)
<TAH>	Trap A Not Heating (1 or 0)
<TBH>	Trap B Not Heating (1 or 0)
<FVH>	Front Valve Not Heating (1 or 0)
<TLH>	Transfer Line Not Heating (1 or 0)
<TOH>	Tube Oven Not Heating (1 or 0)
<RVH>	Rear Valve Not Heating (1 or 0)
<TAT>	Trap A Over Temperature (1 or 0)
<TBT>	Trap B Over Temperature (1 or 0)
<FVT>	Front Valve Over Temperature (1 or 0)
<TLT>	Transfer Line Over Temperature (1 or 0)
<TOT>	Tube Oven Over Temperature (1 or 0)

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<RVT>	Rear Valve Over Temperature (1 or 0)
<split>	Split Valve Open (1 or 0)
<flow>	Current Flow Rate
<error>	General Instrument Error occurred (1 or 0)

Temperatures

Returns: A comma separated list of values in the order shown below

<TA>	The Temperature of Trap A
<TB>	The Temperature of Trap B
<TL>	The Temperature of the Transfer Line
<FV>	The Temperature of the Front Heated Valve
<RV>	The Temperature of the Rear Heated Valve
<TO>	The Temperature of the Tube Oven

CurrentMethod

Returns: The name of the currently active method

FlowRate

Returns: The current Flow value

ActiveTrap

Returns: Which trap is currently active

SampleTime

Returns: The current sample time

Commands

The following commands can be sent to the TT24-7 Software.

Command: **Start**

Parameters: *None*

Send a command to the TT24-7 to start run.

Command: **Stop**

Parameters: *None*

Send a command to the TT24-7 to stop the current run.

Command: **LeakTest**

Parameters: *None*

Send a command to the TT24-7 to start a leak test. Command will not work unless instrument is in a standby state.

Command: **LoadMethod**

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Parameters: *Method Filename*

Change the current loaded method on the TT24-7. If the instrument is in standby the method will be changed immediately. If the instrument is running the method will be changed at the end of sampling or at the end of the current run.

Command: **Split**

Parameters: *ON* | *OFF*

Open or Close the split valve on the TT24-7.

Command: **Mode**

Parameters: *TUBEA* | *TUBEB* | *INJECTA* | *INJECTB* | *CONTINUOUS*

Change the mode of operation of the TT24-7. Changing the mode will not take effect if the instrument is in running mode.

Command: **StopFlow**

Parameters: *None*

Sending command will set the sample flow rate to 0.

Command: **ResumeFlow**

Parameters: *None*

Sending command will return the sample flow to the value specified in the method file.

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Appendix 3: Sorbent specifications

1. Carbotrap C™ (20-40 mesh) / Carbopack C™ (60-80 mesh) / Carbograph 2TD (range of mesh sizes)

Sorbent Strength:	Very weak
Specific Surface Area (m^2/g):	~12
Approximate analyte volatility range:	n-C_8 to n-C_{20}
Example Analytes:	Alkyl benzenes, hydrocarbons to n-C_{20}
Sorbent Maximum Temperature:	>400°C
Recommended Conditioning Temperature:	350°C to 400°C
Recommended Desorption Temperature:	300°C to 350°C
Notes:	Hydrophobic
	Minimal (<0.1 ng) artefacts
	Some activity with labile compounds
	Friable

2. Tenax TA™ or GR™ (range of mesh sizes)

Sorbent Strength:	Weak
Specific Surface Area (m^2/g):	~35
Approximate analyte volatility range:	n-C_7 to n-C_{30}
	Bpt. 100°C to 450°C
Example Analytes:	Aromatics (except benzene), apolar components bpt>100°C, polar components bpt >150°C, PAHs/PCBs.
Sorbent Maximum Temperature:	350°C
Recommended Conditioning Temperature:	325°C
Recommended Desorption Temperature:	Up to 300°C
Notes:	Hydrophobic
	Low inherent artefacts (<1ng)
	Inert – suitable for labile compounds
	Graphitised form best for PAHs/PCBs
	Efficient desorption
	Use 35-60 mesh to minimise fines and eliminate "leakage" through conventional sorbent retaining gauzes

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3. Carbotrap™ (20-40 mesh) / Carbopack B™ (60-80 mesh) / GCB1™ (range of mesh sizes) / Carbograph 1TD (range of mesh sizes)

Sorbent Strength:	Medium / Weak
Specific Surface Area (m^2/g):	~100
Approximate analyte volatility range:	n-$C_{5/6}$ to n-C_{14}
Example Analytes:	Ketones, alcohols, aldehydes and apolar components within the above volatility range.
	Perfluorocarbon tracer gases
Sorbent Maximum Temperature:	>400°C
Recommended Conditioning Temperature:	350°C to 400°C
Recommended Desorption Temperature:	300°C to 350°C
Notes:	Hydrophobic
	Low artefacts (<0.1 ng)
	Some activity with labile compounds
	Friable

4. Carbopack X™ (40/60 and 60/80 mesh) / Carbotrap X™ (20/40 mesh)

Sorbent Strength:	Medium-Strong
Specific Surface Area (m^2/g):	~240
Approximate analyte volatility range:	n-$C_{3/4}$ to n-$C_{6/7}$
	Boiling point 50°C to 150°C
Example Analytes:	Light hydrocarbons, BTX for 2 week diffusive exposure
Sorbent Maximum Temperature:	>400°C
Recommended Conditioning Temperature:	350°C to 400°C
Recommended Desorption Temperature:	350°C to 400°C
Notes:	Hydrophobic
	Low artefacts (<0.1 ng)
	Some activity with labile compounds
	Friable

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5. Carbograph 5TD (various mesh sizes)

Sorbent Strength:	Medium-Strong
Specific Surface Area (m^2/g):	~560
Approximate analyte volatility range:	n-$C_{3/4}$ to n-C_8
	Boiling point 50°C to 150°C
Example Analytes:	Light hydrocarbons
Sorbent Maximum Temperature:	>400°C
Recommended Conditioning Temperature:	350°C to 400°C
Recommended Desorption Temperature:	350°C to 400°C
Notes:	Hydrophobic
	Low artefacts (<0.1 ng)
	Some activity with labile compounds
	Friable

6. Chromosorb 102™ (range of mesh sizes available)

Sorbent Strength:	Medium
Specific Surface Area (m^2/g):	~350
Approximate analyte volatility range:	Boiling point 50°C to 200°C
Example Analytes:	Alcohols, oxygenated compounds, haloforms less volatile than methylene chloride
Sorbent Maximum Temperature:	250°C
Recommended Conditioning Temperature:	225°C to 250°C
Recommended Desorption Temperature:	No higher than 220°C
Notes:	High artefacts (=10 ng)
	Hydrophobic
	Inert – suitable for labile compounds
	For trace level analysis condition at 225°C & desorb sample tubes no higher than 200°C to reduce background

7. Porapak Q (range of mesh sizes available)

Sorbent Strength:	Medium
Specific Surface Area (m^2/g):	~550
Approximate analyte volatility range:	n-C_5 to n-C_{12}

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	Boiling point 50°C to 200°C
Example Analytes:	VOC's within volatility range above, oxygenated compounds
Sorbent Maximum Temperature:	250°C
Recommended Conditioning Temperature:	225°C to 250°C
Recommended Desorption Temperature:	No higher than 225°C
Notes:	High artefacts (=10 ng)
	For trace level analysis condition at 225°C desorb sample tubes no higher than 200°C to reduce background levels
	Low maximum temperature - repack tubes after 50 thermal cycles
	Inert

8. Porapak N (range of mesh sizes available)

Sorbent Strength:	Medium
Specific Surface Area (m^2/g):	~300
Approximate analyte volatility range:	n-C_5 to n-C_8
	Boiling point 50°C to 150°C
Example Analytes:	Volatile nitriles, e.g. acrylonitrile, acetonitrile, propionitrile. Pyridine, volatile alcohols, ethanol, methyl ethyl ketone
Sorbent Maximum Temperature:	190°C
Recommended Conditioning Temperature:	180°C to 190°C
Recommended Desorption Temperature:	No higher than 180°C
Notes:	Hydrophobic
	High artefacts (=10 ng)
	For trace level analysis condition at 180°C and desorb sample tubes no higher than 160°C to reduce background levels

9. HayeSep D (range of mesh sizes available)

Sorbent Strength:	Medium
Specific Surface Area (m^2/g):	~795
Approximate analyte volatility range:	n-C_5 to n-C_{12}

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	Boiling point 50°C to 200°C
Example Analytes:	VOC's within volatility range above.
Sorbent Maximum Temperature:	290°C
Recommended Conditioning Temperature:	280°C to 290°C
Recommended Desorption Temperature:	No higher than 290°C
Notes:	Non polar, requires extensive conditioning, high artefact background.

10. Chromosorb 106™ (range of mesh sizes available)

Sorbent Strength:	Medium
Specific Surface Area (m^2/g):	~750
Approximate analyte volatility range:	n-C_5 to n-C_{12}
	Boiling point 50°C to 200°C
Example Analytes:	Hydrocarbons, benzene, volatile oxygenated compounds
Sorbent Maximum Temperature:	225°C to 250°C
Recommended Conditioning Temperature:	225°C to 250° C
Recommended Desorption Temperature:	No higher than 200°C
Notes:	High artefacts (=10 ng)
	For trace level analysis condition at 225°C, desorb sample tubes no higher than 200°C to reduce background
	Hydrophobic
	Inert – suitable for labile compounds

11. Spherocarb™ / UniCarb™ (60 - 80 mesh only)

Sorbent Strength:	Strong
Specific Surface Area (m^2/g):	~1200 – also operates on molecular sieve principle
Approximate analyte volatility range:	C_3 to n-C_8
	Boiling point -30°C to 150°C
Example Analytes:	Very volatile compounds e.g. VCM, ethylene oxide, carbon disulphide, dichloromethane, chloromethane. Volatile polar compounds e.g. methanol, ethanol, acetone

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Sorbent Maximum Temperature:	>400°C
Recommended Conditioning Temperature:	350°C to 400°C
Recommended Desorption Temperature:	300°C to 350°C
Notes:	Some hydrophillicity
	Low artefacts (<0.1 ng)
	Inert – suitable for labile compounds
	Excellent batch-to-batch reproducibility
	Non-friable
	Easily contaminated by higher boiling components - protect with front bed of weaker sorbent

12. Carbosieve SIII™ (60 - 80 mesh only)

Sorbent Strength:	Very Strong
Specific Surface Area (m^2/g):	~800 - but primarily operates on molecular sieve principle
Approximate analyte volatility range:	Ethane to n-C_5
	Boiling point -60°C to 80°C
Example Analytes:	Ultra volatile hydrocarbons
Sorbent Maximum Temperature:	>400°C
Recommended Conditioning Temperature:	350°C to 400°C
Recommended Desorption Temperature:	300°C to 350°C
Notes:	Some hydrophillicity
	Low artefacts (<0.1 ng)
	Easily and irreversibly contaminated by higher boiling components - protect with front bed of weaker sorbent

13. Carboxen 1000™ (range of mesh sizes)

Sorbent Strength:	Very Strong for small molecules
Specific Surface Area (m^2/g):	>1200 – also operates on molecular sieve principle
Approximate analyte volatility range:	permanent gases and light hydrocarbons (C_2, C_3)
	Boiling point -60°C to 80°C
Example Analytes:	Ultra volatile hydrocarbons

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Sorbent Maximum Temperature:	>400°C
Recommended Conditioning Temperature:	350°C - 400°C
Recommended Desorption Temperature:	300°C - 350°C
Notes:	Significantly hydrophilic - do not use in humid conditions. High artefacts (>10 ng), Easily & irreversibly contaminated by higher boiling components

14. Molecular Sieve 5Å

Sorbent Strength:	Very strong for small molecules
Approximate analyte volatility range:	Boiling point -60°C to 80°C
Example Analytes:	nitrous oxide
Sorbent Maximum Temperature:	350°C - 400°C
Recommended Conditioning Temperature:	300°C - 350°C max (increase temperature gradually from 100°C)
Recommended Desorption Temperature:	To suit analyte
Notes:	Significantly hydrophilic - do not use in humid conditions. High artefacts (>10 ng) Easily & irreversibly contaminated by higher boiling components

15. Molecular Sieve 13X

Sorbent Strength:	Very strong for small molecules
Approximate analyte volatility range:	Boiling point -60°C to 80°C
Example Analytes:	1, 3-butadiene
Sorbent Maximum Temperature:	350°C - 400°C
Recommended Conditioning Temperature:	300°C - 350°C max (increase temperature gradually from 100°C)
Recommended Desorption Temperature:	To suit analyte
Notes:	Significantly hydrophilic - do not use in humid conditions. High artefacts (>10 ng) Easily & irreversibly contaminated by higher boiling components

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Retention Volumes and Safe Sampling Volumes

The most accurate method of ensuring a particular sorbent tube will quantitatively retain a particular analyte is to determine its retention volume for that analyte. Retention volumes are usually quoted in litres per gram of sorbent and must be extrapolated to determine the retention volume on the mass of sorbent in a standard tube. They are always quoted at a set temperature - usually 20°C.

A comprehensive list of retention volumes for different analytes on several sorbents packed into industry standard 3.5" x ¼" OD tubes, is given in MDHS 72 - Volatile Organic Compounds in Air. (The MDHS (Methods for the Determination of Hazardous Substances) series comprises validated methods produced by the United Kingdom Health and Safety Executive for occupational hygiene work. Copies of these publications may be ordered from The Stationary Office, Holburn Book Shop, 59-60 Holburn Viaduct, London, EC1A 2FD, UK.)

It is possible to measure retention volumes in the laboratory by the following procedure using a gas chromatograph configured with a packed column.

Pack the chromatographic column with a known weight of the sorbent of interest

Operate the column at a range of temperatures between 150°C and 250°C

Inject the analyte in question and note the retention time and column flow at each temperature

From this information, calculate the specific retention volume in litres per gram for each temperature

Plot the log of the specific retention volume against the reciprocal of the absolute column temperature which gives a linear relationship

The slope of the graph may be extrapolated to give the log of the retention volume at 20°C

The retention volume may then be calculated

In order to ensure that there is absolutely no chance of any breakthrough occurring during a sampling procedure if, for example, the ambient temperature were to rise, a Safe Sampling Volume (SSV) is usually quoted as being half the retention volume. A sampling strategy which limits the volume of air sampled to the SSV or less is therefore considered to be prudent.

Note also that safe sampling volumes on some sorbents, particularly those with some hydrophillicity, are significantly impacted by high atmospheric humidity. For example SSVs on Spherocarb, UniCarb and Carbosieve SIII should typically be reduced by a factor of 10 at 80% RH and above.

Sampling in a mixed atmosphere

It is often the case that the list of analytes to be monitored requires more than one sorbent. For example, if both toluene and methanol are to be monitored simultaneously two sorbents - one medium / weak for toluene and one strong for methanol - should be used. For diffusive monitoring, this can only be achieved by using two or more tubes in parallel and by stringently conditioning the tube packed with the stronger sorbent before re-use. Pumped monitoring requires

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samples to be drawn through the two or more sorbents in series. Sampling onto sorbents in series can be achieved in one of two ways:

1. Linking together two tubes containing the sorbents required
2. Packing both sorbents as two separate beds in a single tube

In method 1 tubes are connected together using ¼ - ¼" brass couplings fitted with PTFE combined ferrules. The tubes must be connected in series such that the tube containing the weakest sorbent is at the front of the sampling train and all tubes must be oriented such that the sample passes through from the sampling (grooved) end of each tube. This ensures that the higher boiling components in the mixture are adsorbed by the weaker sorbent and eliminated from the sample stream before reaching the strong sorbent.

In method 2 small plugs of glass wool or sorbent retaining gauzes separate the 2 or three sorbents in a single tube. The weaker sorbent is packed at the front, sampling end of the tube followed by a plug of glass wool then the stronger sorbent. Again this ensures that when air is drawn through the tube the mixture reaches the weaker sorbent first where the higher boiling components are trapped.

Note that, if sampled tubes are to be stored for some time, for transportation or other purposes then method 1 is preferred. After sampling the tube train can be dismantled and each tube capped with long term storage caps. Long term storage of tubes containing two or more sorbents is not recommended as higher boiling components may migrate from the weaker to the stronger sorbent over time and this can cause incomplete desorption when the tubes are eventually analysed. If the multi-sorbent tubes are to be stored for longer than one week then the following procedure must be undertaken:

take the samples in the normal way and cap with ¼" brass storage caps in the field as usual

place the tubes in an air tight container - e.g. Tupperware box or clean tin can with lid - and place in a clean refrigerator

when the tubes have reached the 'cold' temperature, briefly remove them from the refrigerator and check and re-tighten the caps (due to the difference in the thermal properties of brass and stainless steel the caps may have become slightly loose)

return the samples, in their container, to the refrigerator

immediately prior to analysis, remove the tubes from the refrigerator and allow them to come to room temperature before analysing – this is vital to avoid any problems with condensation

Note also that if 2 or more sorbents are to be packed into a single tube, all the sorbents must have similar maximum temperatures. If one or more of the sorbents has a significant lower maximum temperature than the other(s) in the tube, it will not be possible to stringently condition the more stable sorbents without exceeding the temperature limitations of the less stable material. For this reason Chromosorb and Porapak sorbents are not recommended to be used in mixed sorbent bed tubes.

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Long term storage of clean and sampled tubes

Conditioned or sampled sorbent tubes should always be stored using ¼" brass Swagelok-type screw caps fitted with combined PTFE ferrules. It is recommended that these be tightened by hand plus a further quarter turn using conventional spanners / wrenches or, ideally, a Markes International CapLok™ tool. The Cap-Lok tool was invented by scientists at the French Environmental Centre - INERIS - and prevents over tightening and distortion of the PTFE ferrules.

It is not necessary to store capped tubes in refrigerated conditions – except as stated above. If refrigeration is to be used, caps must be retightened (approximately a quarter turn) using the CapLok tool once they have reached their storage temperatures.

N.B. There are other implications associated with storing tubes under refrigerated conditions. Many laboratory fridge / freezer atmospheres are highly contaminated with volatiles from other samples or from the refrigeration system itself.

N.B. If the tubes are to be transported in such a way as to be exposed to very cold temperature i.e. in an aircraft hold, by rail / road overnight during cold weather, it is advisable to follow the above retightening procedure by cooling the tubes (by placing outside, or in a clean refrigerator) prior to shipment.

When monitoring trace level atmospheric components, conditioned and sampled tubes can be wrapped in uncoated aluminium foil and / or placed in a sealed, non-outgassing container, such as an uncoated tin, during transportation and storage.

For further information on minimising artifacts - see TDTS19

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