



# CONCENTRATOR/HEADSPACE ANALYSIS INJECTOR

## Installation and operation Instructions

for kit Manual Control when used with Control Module

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## WARRANTY STATEMENT

This CHIS and Control Module is guaranteed against faults in materials or workmanship for a period of **twelve months** from the date of invoice.

This warranty implies free repair and or replacement of defective goods only, upon proper written proof and, where authorised, return of the defective product.

It does not cover 'disposables'.

THIS UNIT HAS BEEN DESIGNED TO FULFIL THE PURPOSE OF CONCENTRATED/HEADSPACE INJECTION AND THIS WARRANTY IS VOID IF THE INSTRUMENT IS USED FOR ANY UNRELATED PURPOSE. SGE RESERVES THE RIGHT TO REFUSE FREE SERVICE UNDER WARRANTY ON ANY UNIT WHICH HAS BEEN ABUSED OR TAMPERED WITH IN ANY WAY.

No other warranty or representation is expressed or implied by SGE for its products with respect to merchantability and fitness for any particular use or purpose or any other matter. SGE shall not under any circumstances, be liable for any incidental, consequential, or compensatory damages arising from use of or in conjunction with its products. The maximum liability for breach of warranty shall be the invoice price of said products.

SGE acknowledges that there are many service engineers in the field who have the expertise to service the CHIS or Control Module and to assist them we have enclosed the necessary data in this manual.

To protect your interest, however, we respectfully suggest that any failures during the warranty period be attended to by SGE service personnel or by SGE authorised service people.

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## 1.0 INTRODUCTION

The Concentrator/Headspace injector is a dedicated, capillary gas chromatography sample introduction system, designed for use with a wide range of air or aqueous samples.

Samples are drawn through the glass lined absorption tube packed with an adsorbent. Organic materials are trapped in the absorption tube, enabling transfer to the gas chromatograph.

The absorption tube is inserted into the special injector located in the heated injection block of the gas chromatograph. In "desorb" mode the sample components are thermally desorbed from the absorption tube and focused at the head of the capillary column.

As the sample is chromatographed in "run" mode, a reverse flow of carrier gas to vent, cleans and conditions the absorption tube ready for the next sample.

## SYSTEM REQUIREMENTS

The CHIS injector must be installed into a spare injection block position in the gas chromatograph. This may require the removal of an existing injector, or the injection block (HP5890), to make room for the CHIS. Removal and replacement of injectors is a simple procedure and should be done when the injector is cool.

A 1/16" carrier gas line is required for connection with the CHIS system. This line must originate after the carrier gas pressure regulator.

## ABSORPTION TUBES

The absorption tubes supplied with the CHIS Injection System are packed with Tenax™ TA, 60/80 mesh. Tenax is suitable for adsorbing compounds  $>C_6$  and  $C_1$  to  $C_5$  depending on functional group. The tubes can be emptied and repacked with other adsorbents if required. Appendix 1 lists some of the more common adsorbents and the application specifications.

## INSTALLATION OF INJECTOR ASSEMBLY

1. Remove the existing injector components from the injector heater block. If necessary refer to the instrument maintenance manual for information on injector removal procedure.

**NOTE:** In the case of the HP5890, (or gas chromatographs which require the CHISA 50 kit), the existing heater block should be removed to enable installation

### Additional Spares

Description	Used In	Part No.
0.8mm ID empty absorption tube for CHISA	CHISA	0932230
3.0mm ID empty absorption tube for CHISA	CHISA	0932232
0.8mm ID empty absorption tube for CHISB	CHISB	0932234
3.0mm ID empty absorption tube for CHISB	CHISB	0932236

### Additional Accessories

Description	Code	Part No.
CHIS A- Storage tube		0932260
CHIS B - Storage Tube		0932261
CHIS tube conditioning module		0932262
Detector connector kit for 1/4" fittings	DC-4	103462
Detector connector kit for 1/8" fittings	DC-8	103463
Detector connector kit for 5mm fittings	DC-05	103464
Detector connector kit for Varian 3700 and later GC models	DC-4V	103465
Detector connector kit for HP5890	DC-4HP5890	103467
Constant flow velocity connector Varian	DRC-Varian	1034681
Constant flow velocity connector HP	DRC-HP	1034683
CO <sub>2</sub> Cryogenic focusing system	CTS.LCO2	093346

### Trademarks

Tenax™Enka Research Institute  
 Carboxen™Supelco Inc.  
 Carbosieve™Supelco Inc.

of the SGE heater block supplied .  
 (Refer to Section 18 of the Hewlett Packard maintenance manual (Series I & II) for an exploded diagram of the heater block) the Heater Cartridge and Thermocouple should be reinstalled.

2. Locate and install the Instrument Specific brass Adaptor (6) in the instrument heater block.

3. Install the "screw threaded" Injector Body (5) through the top of the Instrument Specific Adaptor. The screw threaded end of the Injector Body should now be located in the gas chromatograph oven.

4. The CHIS injector is supplied with the pneumatic inlet Tee unions (10, 11, 12 & 13) assembled. (If re-fitting of these components is required, assemble the GLT interface tube (11), Vespel Sealing Ring (12) and Tee (13) onto the Union Tee (10) as shown in figure 1. (Note: the square end of the GLT interface tube is located into the Union Tee (10)).

5. Install a Graphitised Vespel Tube Seat (9) into the Union Tee (10) as shown ensuring the Seat locates into the positioning well at the base of the Tee.

6. Fit and finger tighten the SSN/4 Nut (7) onto the Union Tee (10) with the GVF/4 ferule (8) located as shown figure 1 (Taper up).

7. Locate and screw assembly Union Tee (10) onto the CHIS Body (5) until finger tight.

8. Spanner tighten the SSN/4 Nut (7) to obtain a leak tight seal with the CHIS body.

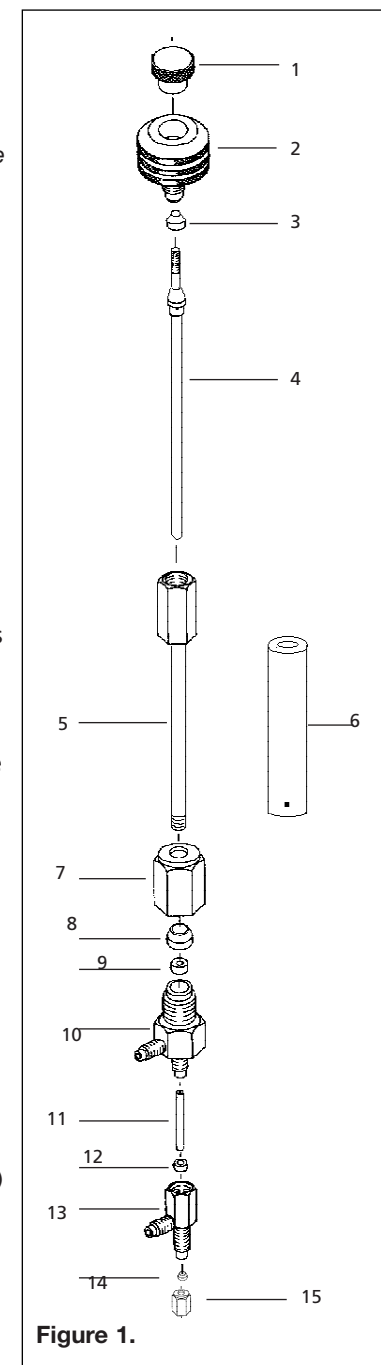


Figure 1.

## 2.0 KIT INSTRUCTIONS CARRIER GAS CONNECTIONS

See Figure 2.

9. Select a suitable position for the MTV-3 valve to be mounted onto the G.C. Cut a suitable length of 1/16" stainless steel tubing to make a connection from the carrier gas source, after the carrier gas pressure regulator, to the supplied AT-PCS absorption trap (A). From the outlet end of the AT-PCS connect another length of stainless steel tubing to the inlet side of the MTV-3 valve (B). (VSR/16 sealing rings can be used on all connections inside and outside the heated oven zone.)

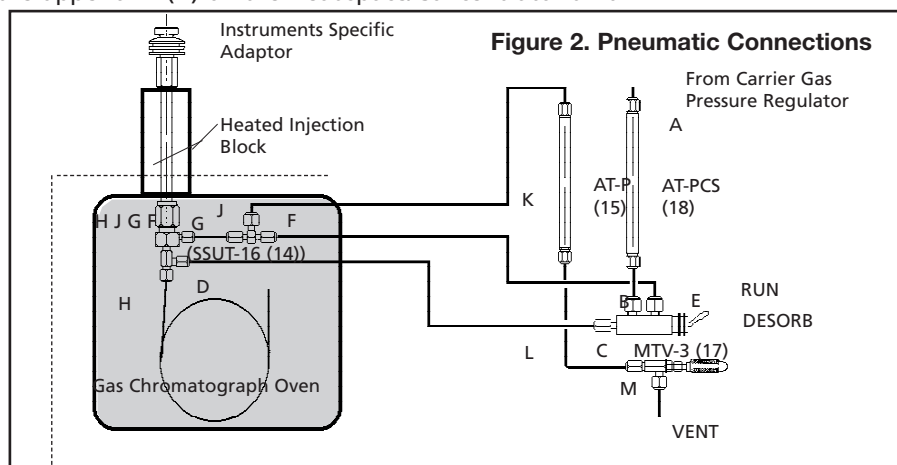
**CAUTION:** We advise the use of purified carrier gas supplies to prevent possible contamination of Tenax. AT-PCS purifier is a backup only and should only be used with a dedicated organics purifier.

10.

Connect a length of 1/16" O.D. stainless steel tubing from the MTV-3 rear outlet (C) to the lower arm (D) on the Headspace/Concentrator.

11. From the MTV-3 (17) valve outlet (E) connect 1/16" stainless steel tubing to one arm (F) of the SSUT-16 tee union inside the GC oven. **Note:** SSUT-16 must be situated inside the oven heated zone.

12. Connect a length of 1/16" stainless steel tubing from SSUT-16 (14) union (G) to the upper arm (H) on the Headspace/Concentrator unit.



13. From the remaining arm (J) of the SSUT-16 union connect another length of 1/16" stainless steel tubing to one end of the AT-P (15) mini filter (K).

## 7.0 SPARES LIST

No	Description	Packed Qty	Used In	Part No.
1	CHIS-LOCKNUT, Knurled Lock Nut	2	CHISA & B	0932258
2	CHIS-SEPCAP, Septum Cap	2	CHISA & B	0932259
3	CHIS-SR, Graph, Vespel Sealing Ferrule	5♦	CHISA & B	0932256
4	0.8mm ID Absorption Tube (Tenax TA)	2	CHISA CHISB	0932240 0932244
OR				
	3.0mm ID Absorption Tube (Tenax TA)	2	CHISA CHISB	0932242 0932246
5	CHIS-Body, Screw threaded CHIS body	1 1	CHISA CHISB	0932252 0932253
6	Instrument Specific Adaptor	1		
7	SSNE/4 Stainless Steel Nut1	CHISA & B	103407	
8	GVF/4, 1/4" Graphitised Vespel Ferrule	2 & 1♦	CHISA & B	072667
9	CHIS-S, Graphitised Vespel Tube Seat	2 & 1♦	CHISA & B	0932255
10	CHIS-TEE, Screw Threaded Union Tee	1	CHISA & B	0932254
11	CHIS GLT (1/16" x 0.810mm)	1	CHISA & B	
12	VSR-16, Vespel Sealing Ring	20 & 1♦	CHISA & B	072653
13	CHIS-TEE2, Lower Tee Union	1	CHISA & B	
14	SSUT/16, Union Tee	1	CHISA & B	103418
15	CHIT Sampling Adaptor	1	CHISA	0932250
	SST-16/0.8	1	CHISA & B	0624160
	Spanner 1/4" x 5/16" AF	2	CHISA & B	
	1/16" Allen Key	1		
	Kevlar Thermal Gloves	1		
	Instructions			

\* For CHISA-11 heater block suitable for HP5890 is supplied.

\* For CHISA-50 a heater and heater cartridge suitable for countries voltage is supplied.

♦ Installed in the units.

- e. After the appropriate "desorption" time as determined by experiment, the module is returned to the "RUN" mode during which time the sampling tube is reconditioned while the chromatography continues.
- f. At the end of the chromatographic run, switch on the cooling gas to the cooling receptacle by moving the "Purge Gas" toggle valve to the "on" position.
- g. Remove the sampling tube from the injector and immediately insert it into the cooling receptacle located in the top of the control module.
- h. Insert either the next sample tube or a "blank" tube into the injector to maintain the column head pressure.

## 6.0 APPENDIX 1

### TRAPPING MATERIAL

### PROPERTIES

Tenax™ - TA	Traps C6 and greater, C1 to C5 depending on functional groups. Does not trap low-boiling solvents and highly volatile compounds.
Carboxen™ - 563	Traps C3 and greater, C1 to C2 depending on functional groups. Halogenated compounds in water.
Molecular Sieve 5A**	Traps C3 and greater.
Activated Charcoal	Traps C3 and greater, C1 to C2 depending on functional groups.
Carbosieve™ S-111	Traps C2 and greater. Excellent for compounds such as ethane and ether.

#### NOTE:

*Tenax TA is particularly useful for the analysis of high boiling compounds such as alcohols, polyethylene glycols, diols, phenols, monoamines, diamines, ethanalamines, aldehydes, ketones and chlorinated aromatics.*

*\*\* The capacity of Molecular Sieves is depressed by the adsorption of Carbon Dioxide and water vapour. Condition in excess of 300 °C to remove these.*

14. Connect a further 1/16" stainless steel line between the AT-P (15) (L) and the BMCV-1 (16) (M).

15. Connect the capillary column into the tee union (D) using the supplied SSNE16-012 nut (15) Fig. 1. It is important to insert the column so that it terminates just into the vespel seat (9). The distance from the bottom of the tee union (13) to the seat is approximately 24mm.

## 3.0 THE CONTROL MODULE

### INTRODUCTION

This control module is designed to provide all the pneumatic controls to allow conversion of your gas chromatograph to perform capillary column analysis of samples obtained by absorption into the sample tube of the SGE Concentrated Headspace Injection System, here in referred to as CHIS.

#### A. FRONT PANEL

**Purge:** Controls the rate of back flush of the sample tube after injection.

**Purge Outlet:** Allows measurement when required, of the back flush rate.

**Cooling Gas:** The action of desorbing and backflushing the sample tube also cleans and conditions it ready to be used for taking a new sample. To prevent the ingress of "contaminated" laboratory air during cooling, the tube is removed from the injector and immediately inserted in a receptacle within the body of the control module, which is purged with clean compressed gas. This valve turns on/off the gas as required, during this process.

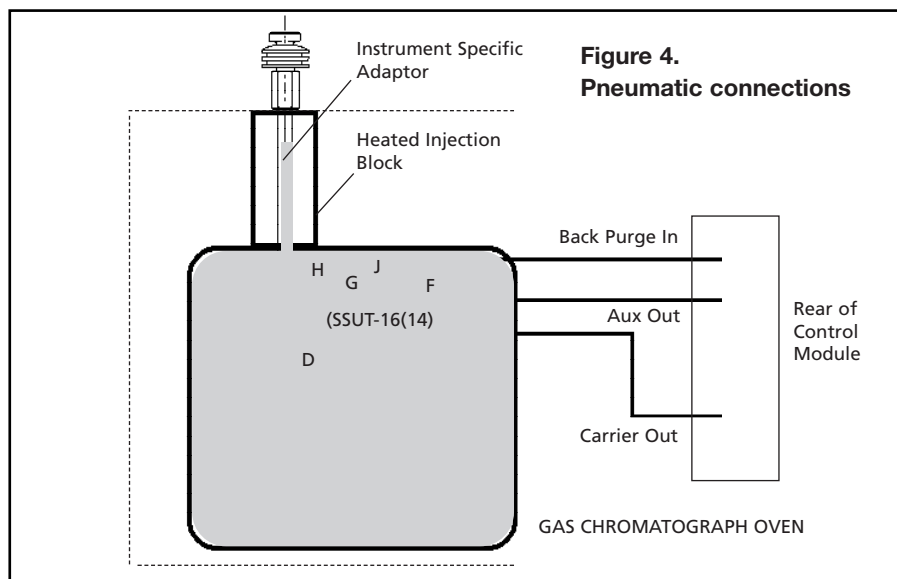
**Run - Desorb:** During the insertion and removal of the sample tube, the injector is open to the air and carrier gas must be maintained to the column. During the desorption of the sample the carrier must be redirected down the absorption tube.

This switching valve directs the carrier gas in the appropriate direction as required. In the CHIS-2 version of the Control Module this switching valve is replaced by an electric solenoid valve (see under "Remote" in the description of the Rear Panel).

**Carrier Control:** This pressure regulator and gauge provides the carrier gas control for capillary chromatography.

## 4.0 CONTROL MODULE INSTALLATION INSTRUCTIONS

### CHIS 1 Control Module - Standard Version



1. Position the unit alongside the gas chromatograph.
2. Connect the carrier gas source into the rear of the Control Module.
3. Using a suitable length of 1/16" stainless steel tubing connect the Carrier Out port of the Module to inlet D of the injector. See Figure 2.
4. Similarly connect a second piece of tubing from the "Aux Out" port to point F on the tee piece (SSUT-16, # 103418) located inside the oven.
5. Connect point G on the tee piece to point H on the injector using a short piece of stainless steel tubing.
6. Connect point J on the tee piece to "Back Purge In" port on the rear of the module.
7. Connect Make-Up gas of choice into the "Make Up In" port on rear of the control module.

#### Aux In

Allows introduction of the "cooling gas" (see front panel description).

#### Aux Out

As outlined in the front panel description under "Run-Desorb", carrier gas must be supplied to the injector from two sources to allow switching of the direction. This is the alternative source to that provided from the "Carrier Out" port.

#### Cool Out

Not used.

#### Remote

Provides the electrical connection to the solenoid in the CHIS 2 version. This solenoid, which is driven from a data station or instrument panel control, performs the same function as the toggle valve labelled "Run - Desorb" on the front panel of the manual (CHIS 1) version.

#### Carrier Out

Allows the injector to be supplied with pressure controlled carrier gas.

#### Carrier In

Allows input of carrier gas from pressurised cylinders or tanks. **NB.** This carrier gas should be scrubbed free of oxygen, moisture and organic contaminants. Ensure line traps are in good condition.

#### Make Up Out

Allows make-up gas to be supplied to the detector connector of the GC to allow optimisation of the particular detector.

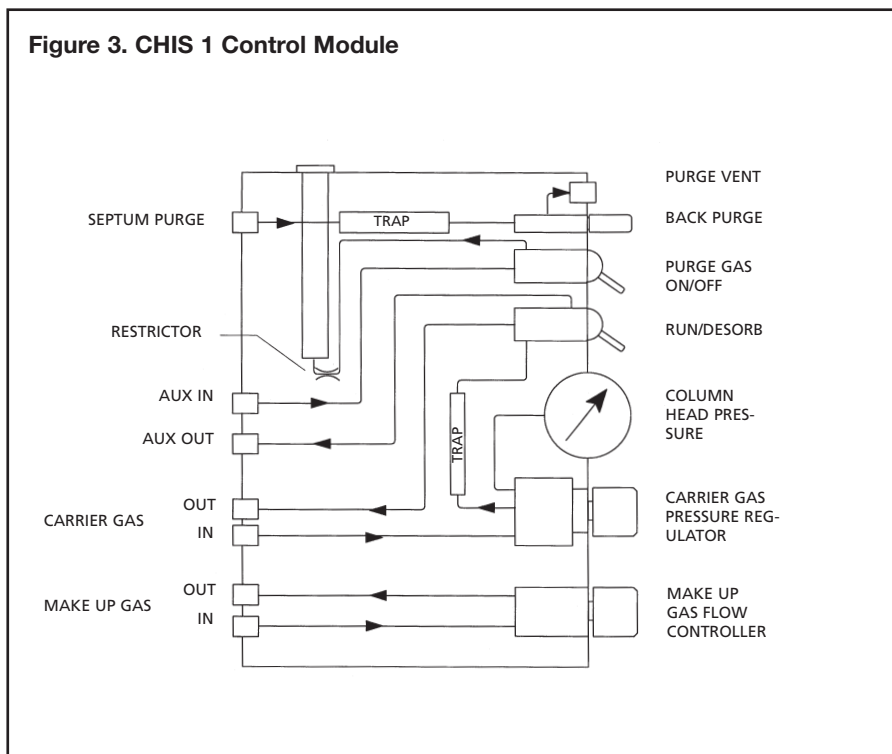
#### Make Up In

Allows introduction of the appropriate make-up gas from the cylinder or tank.



## Make-up Gas

GC detectors, particularly Flame Ionisation Detectors and Electron Capture Detectors are designed, under optimum conditions, to operate at much higher flows than are used with capillary gas chromatography. This control allows the addition of gas, after the column, to allow optimisation of the detector by making up the gas flow to the design specification. If the chromatograph is not provided with a make-up gas inlet to the detector, consult the SGE catalogue for details on our comprehensive range of detector connectors. Part numbers; 103462 to 103467, 1034681 and 1034683.



## B. BACK PANEL

### Back Purge In (Septum Purge Port)

After desorption, the sampling tube is back purged to clean and condition it for re-use. This port allows control of the purging rate.

### Split Vent

Not used.

using 1/16" OD stainless steel tubing.

**NB:**VSR 16 sealing rings can be used on all connections both inside and outside the heated oven zone.

9. Connect the capillary column into the tee union (D) using the SSNE16-012 nut supplied (15) Fig. 1. It is important to insert the column so that it terminates just into the vespel seat (9). Refer to figure 1. The distance from the bottom of the tee union (13) to the seat is approximately 24mm.

10. Assembly of Absorption Tubes.

The CHIS injector is supplied with prepacked Sampling Tubes.

If assembly of these tubes is required, follow the procedure below:

- Locate and fit the Vespel Sealing Ferrule (3) taper facing up.
- Fit the Septum Cap (2) and Lock Nut (1)
- Prior to using the Headspace/Concentrator System, check that all connections are secure and no leaks exist.

11. For the CHIS - 2 version of the injector, the 'Run-Desorb' toggle valve on the front panel is not present and is replaced by a solenoid valve which is driven from a data station, or a GC external events function.

DC power to the solenoid is supplied via the plug provided. Although the plug will only fit one way, the voltage does not need to be polarised.

**NB:**Check the rear of the module for the DC Voltage required. Power consumption

is 0.65 Watt at the specified voltage.

## 5.0 FUNCTIONS AND OPERATION OF THE CHIS INJECTOR

The following instructions outline the functions and operation of the Concentrator/Headspace system.

### FUNCTIONS

Headspace/Concentrator Operation Modes.

#### Run Mode

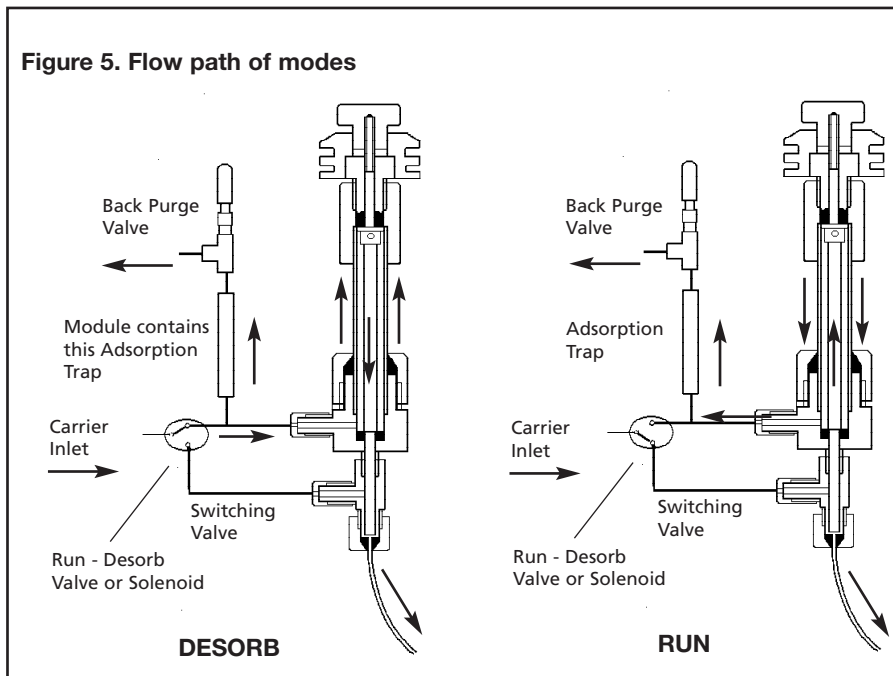
Allows a flow directly onto the column with a purge of carrier through the absorption tube to vent.

#### Desorb Mode

The adsorbed sample is thermally desorbed and carried onto the capillary column.

Figure 5 illustrates the flow path of these two modes.

Figure 5. Flow path of modes



## OPERATION

### 1. Conditioning of Absorption Tubes

The four absorption tubes supplied are packed with Tenax TA adsorbent material. Prior to initial use, each absorption tube will require conditioning.

Conditioning of the absorption tubes should be done in the injector at 300°C for two to three hours with the headspace/concentrator in RUN mode. A vent flow out of the purge outlet 10-25ml/min. is recommended. During this procedure a column should be installed in the gas chromatograph, or a piece of fused silica or stainless steel plumbing between the injector and detector, to prevent build up of carrier gas inside the oven especially when Hydrogen is being used.

As an alternative to conditioning tubes on a "one-off" basis, the SGE CHIS Tube Conditioning Module allows for six tubes to be prepared simultaneously without using the injector at all. (Part number 0932260 CHIS Tube Conditioning Module).

### 2. Vent Flow Setting for Operational Modes

A vent flow of 10-25ml/min is recommended during standard operation of the CHIS.

### 3. Sample Collection

Sample collection into the absorption tube may be performed two ways:

- By connecting the absorption tube to a vacuum source using the sample adaptor.
- OR
- By pushing the sample through the absorption tube. The tube is connected into the adaptor as per Figure 6.

### 4. Desorbing the Sample

(Refer Fig. 1).

Once a sample has been adsorbed into an absorption tube, chromatography of the adsorbed components is carried out as follows:

- Set injection port temperature and wait for it to stabilise.
- Ensure that CHIS is switched to the "RUN" position using the toggle valve or solenoid.
- Insert the absorption tube into the main body (5) complete with cap (2) and lock nut (1) assembly and secure so that the tube is sealed.

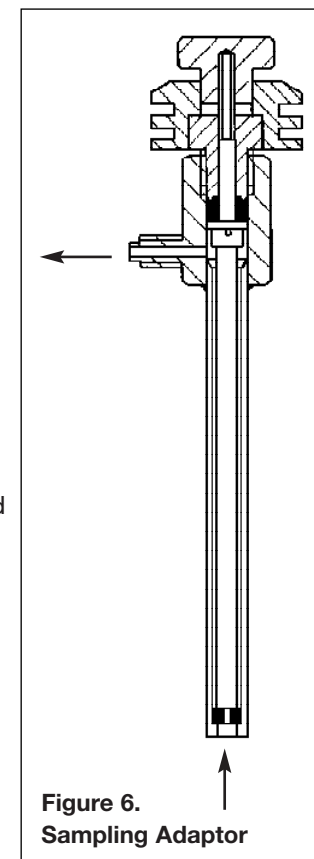


Figure 6. Sampling Adaptor

**CAUTION: To prevent contaminants from hands take care not to handle the outside of the adsorption stem.**

- The valve is then switched into DESORB mode for the time needed to desorb the sample. (Experiments should be performed to determine required desorption times. Begin with 2 minutes and vary the times both longer and shorter).

**NOTE:** The use of Cryogenic focusing is recommended for applications involving volatile samples. Using this method reduces peak tailing and band broadening due to the sample not being deposited in a narrow band at the beginning of the column.

SGE's cryogenic focusing system (optional) (CTS.LCO2 Part Number 093346) is recommended for this purpose.