Instruction Manual

PREP 9000

Micro-Preparative techniques allows you to .....

Separate complexe mixtures and structural isomeres

Isolate impurities for identification or for use as standards

Collect components for further use e.g. NMR

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PREP 9000 Instruction Manual

August 1999 Edition

Published by Brechbühler AG, Steinwiesenstrasse 3, CH-8952 Schlieren

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Read this first!

Unpacking

When receiving the product, inspect the packing for any damage.

If the external packing is damaged, make a note and pay particular attention during unpacking.

When the product has been unpacked, inspect it carefully for if any damage occurred.

If any damage is found at the Instrument, notify it immediately your Brechbühler AG representative, who will give you instruction accordingly.

Standard Outfit

Please check also carefully if the material delivered as standard outfit complies with the checklist enclosed in the package.

Operating Voltage

On the rear panel of the unit a label indicates the operating voltage of the unit. Two configurations are available, 230V and 120V.

Please make sure the voltage of the unit is suitable for the power supply you have at your disposal before removing the label. If the voltage is not correct, please contact your Brechbühler AG representative.

Warranty

Warranty

Brechbühler AG warrants all of its products to be free from defects in workmanship or material under normal use and service within the period of one year.

Exemption: 6 months warranty for the heated transfer line

The warranty does not cover consumables, damages caused by improper use of the instrument and damages due to transportation.

The warranty will be invalidated (specific agreements excepted) if the instrument is installed in the absence of a authorised and qualified Brechbühler AG service engineer.

Disclaimer

Brechbühler AG makes no warranty of any kind with regard to this material, including, but not limited to, warranties of merchantability and fitness for a particular purpose.

Brechbühler AG shall not be liable for errors contained in this document or consequential damages in connection with the furnishing, performance, or use of this information.

The information contained in this document is subject to change without notice. The contents of this document may be revised without prior notice.

Brechbühler AG also reserves the right to make changes and / or improvements to its products without incurring any obligation to incorporate such changes or improvements in units previously sold or shipped.

Brechbühler AG undertakes no responsibility to any defects of a Gas Chromatograph, in a caused manner trough inappropriate installation or disregard the Instruction Manual.

Safety Instructions

Precautions when using the unit

Warning!

The PREPr 9000 control unit is a sensitive analytical instrument. All electrical connections must be well grounded.

Poor grounding represents a danger to the operator and may be seriously affect the efficiency of the instrument.

Warning!

The PREP 9000 Interface controlled by the PREP 9000 control unit is heated during operation and can get very hot.

Do not touch the Interface during operation, you could get burned!

Using PREP 9000 on unknown samples

Warning!

GC-Preparative should not be conducted on unknown samples.

Safety and health of the person who is doing the Preparative analysis should be the primary concern before evaluating samples using the Preparative technique. Users conducting Preparative analysis should be aware about the nature of the samples they will be exposed to during analysis. Usually Laboratories do have their own internal regulations and safety standards for such a matter.

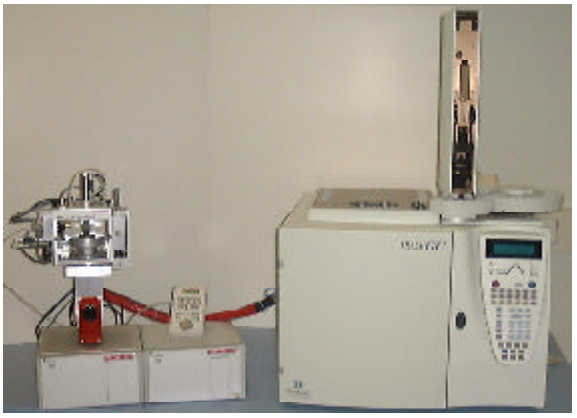
Brechbühler AG undertakes no responsibility to any incidental misuse by disregarding the safety regulations in your organisation.

Brechbühler AG will not be responsable for any accident created by analysing unknown sample mixtures.

Theoretical Aspects

Micro-Preparative techniques allows you to .....

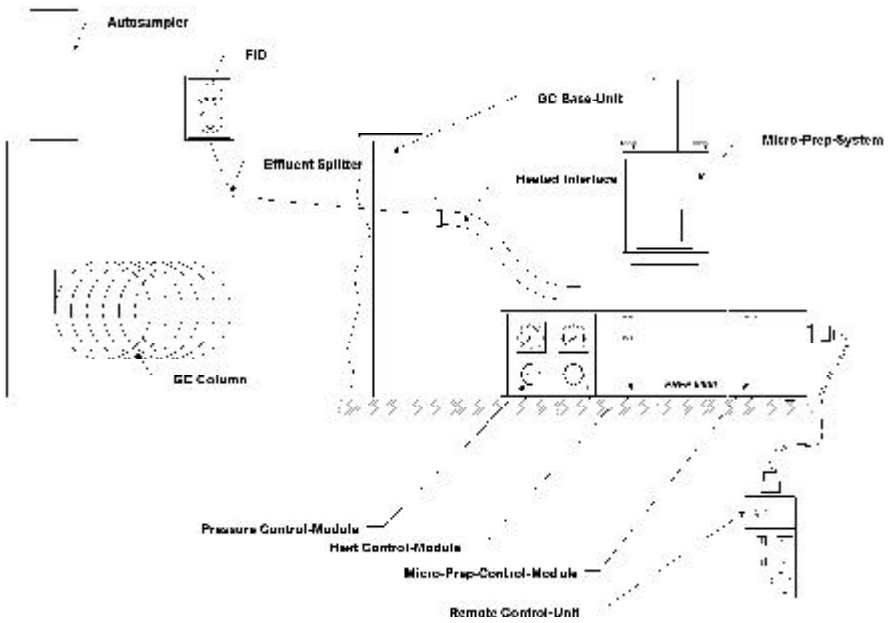
Separate complexe mixtures and structural isomeres
Isolate impurities for identification or for use as standards
Collect components for further use e.g. NMR



The new PREP9000 System based on the SNIFFER 9000 System has been completely redesigned in order to fit to the future needs. The system now is available as a stand alone unit to be installed to any GC available on the Market. The PREP9000 System has been designed to be a dedicated Preparative System connected to any GC available on the market by using a flexible heated interface line to the preparative unit. The new Electronic- and Pneumatic design , based on the new industry standard (LON) allows the system to be as flexible as possible for the future needs and to protect your investements in the Laboratory.

The Micropreparative technique

The enrichment procedure is based on a repetitive adsorption of eluting compounds after separation from a capillary column, by sucking them trough an adsorption tube mounted on the outlet splitter. Optimisation is easily performed during a supervised GC run in a semi automatic mode. Repetitive sample processing becomes possible today with the aid of computers. Using simple logical decisions, they are able to reliable supervise even complex systems. Based on the FID signal of a reference substance the system directs the column effluent at any given time (using the time events table of any Data Handling System) into the collecting system where the individual compounds of interest are adsorbed.



Principle of operation

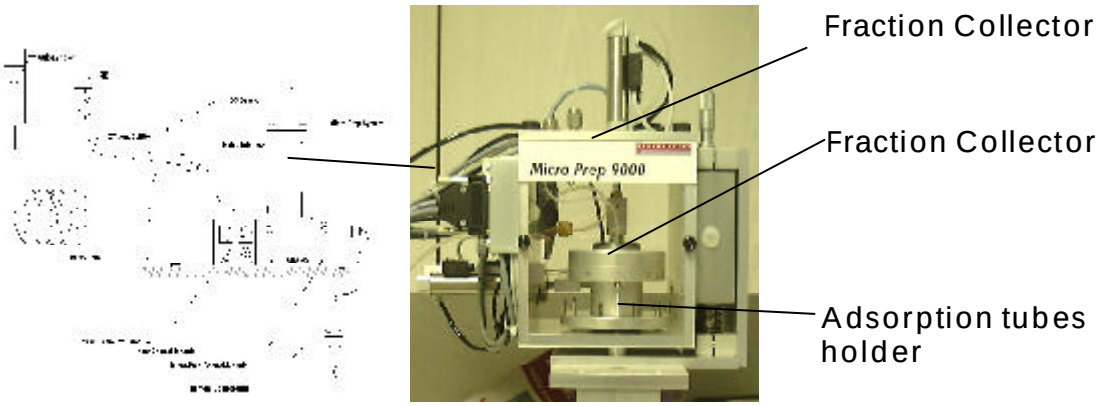
The mixture to be separated are filled into standard Autosampler Vials which are placed into a Automatic Sampler. The sample will be injected using the On-Column technique on conditioned 0.32 mm i.d. capillary columns.

Small glass tubes filled with an adsorbent are used for sample enrichment. They are fixed in a special designed revolving drum which is screwed on a additional detector base body heated by the standard detector heating block. One branch of an exit splitter leads through available PRES-FIT T-piece. During normal run conditions the whole column effluent flows into the detector. On changing to the switch condition the effluent can be directed completely or partially into the adsorption tubes. This is accomplished by the following mechanism: The adsorption tube placed directly above the splitter branch is connected to vacuum. The vacuum is activated byswitching a solenoid valve opening the circuit to the vacuum pump. On sucking, the whole effluent can be passed through the adsorption tube, so efficiently that the flame of the FID can even be extinguished. During normal operation the solenoid valve is closed. Thus a standing gas volume, serving as a gas barrier, is obtained in the splitter branch leading the adsorption tube. Therefore, the column effluent is forced to flow through the splitter branch leading to the FID and does not contaminate the adsorption tube.

Changing over from one fraction to another is performed by turning the pneumatically driven revolving fraction collector. This procedure needs about 2 seconds. Any loss of sample during this time can be neglected, particularly since it is preferable anyway to collect neighbouring peaks in such a way that the overlapping zone, which is generally larger than 2 seconds, is not collected. For resolved peaks, the sample loss between the peak valleys is negligible. Generally, the ratio between bleed and sample improves if the eluting sample is not collected at the bottom of either slope. Furthermore, the information gained from a spectrum of pure sample is more valuable than from a sample containing 10 to 20% impurities. Collected fractions can be stored or be desorbed with a suitable solvent for subsequent analysis, e.g. NMR, UV, IR, micro chemical reactions, and biological activity tests.

Fraction collecting system

Up to 10 adsorption tubes can be placed in to the fraction collector. Collecting times are controlled by any Data handling System which allows external events programming , using the Time events table (by switching the Digital Outputs on and off). This can be easily done by using a first reference Chromatogram and then using the Mouse pointer to set the collection time for the peaks of interest. If no Data handling system is available the PREP 9000 Hand Controller has a built in Time Table to enter the collecting times.

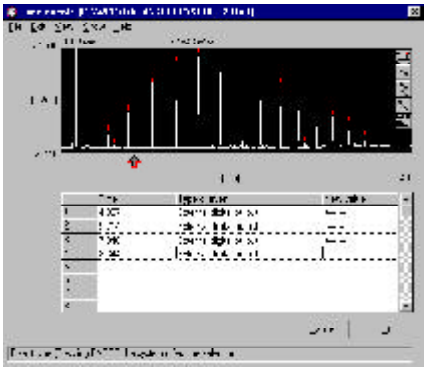


How to perform routine analysis....

The PREP 9000 System can be installed to any GC available on the market. Using standard Autosamplers and any Windows based Chromatography data system the PREP9000 system can be piloted without any additional Software. However, if for any reason no Data handling system is available the PREP9000 system can be time programmed by it self using the internal time table built in the Hand Controller.

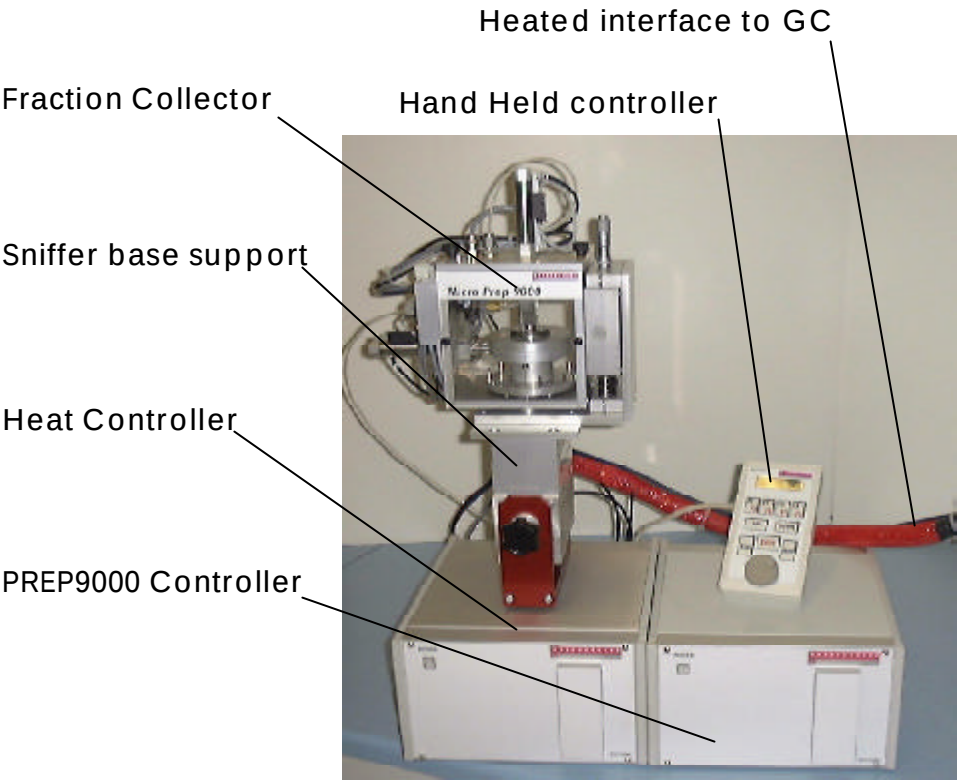
The Fraction Collecting System which is piloted by any Software (or the internal time programming table) to collect the peaks of interest, has a intelligent built in Electronic Circuit to ensure secure functionality during routine Analysis. As the User selects the Components of interest by using time events of a Data handling system (e.g 3 Peaks) the Electronic Circuit has to count how many samples has ben collected. At the end of the run (e.g. 3 Peaks) the revolving system moves to his correct start position and has to wait for the next injection.

Usually the user performs one or two reference Chromatogramm to ensure stability of the analytical conditions. After this procedure a time programmed table will be filled to set the desired collecting times.
The Hand-Held Remote Controller of the PREP9000 system can be upgraded by Software option to allow the system to be programmed as stand allone system, without using an external Data Handling system.



For Test purposes the Fraction Collector can be moved also manually to any desired Position to collect Peaks manually without using the routine procedure provided by a Data Handling system or the built in Time table.

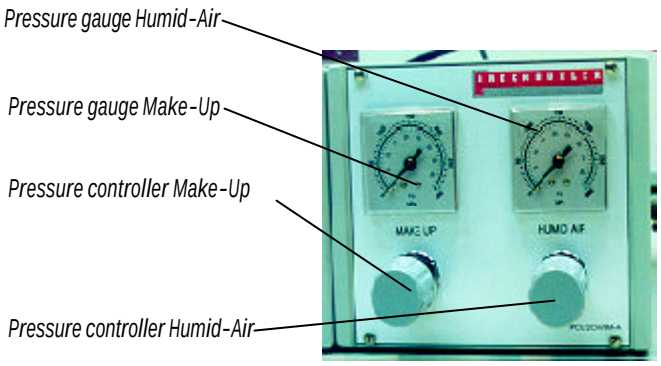
Instrument description....



Description Pneumatic controller

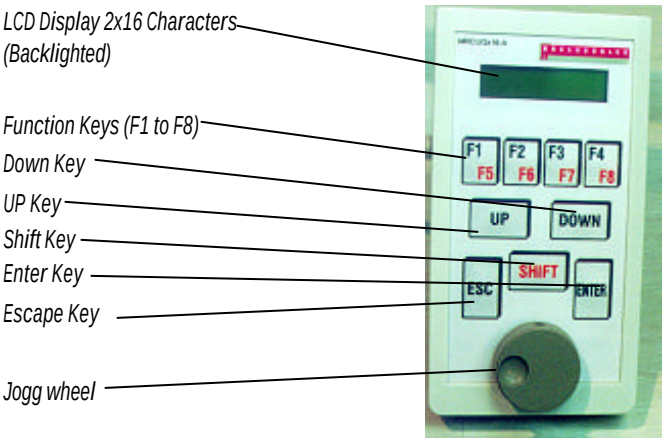
The hot, dry carrier gas carrying the GC effluent to the nose may be irritating, uncomfortable and drying mucous membrane at the users nose. To prevent this, a gentle stream of humidfied air may be used among the fused silica line to the outlet.

The Pneumatic pressure controller of the PREP9000 has implemented a Pressure regulator and a Pressure gauge for make-up gas and air humidification separately to allow the user to adjust the gas flows of both to his comfort.

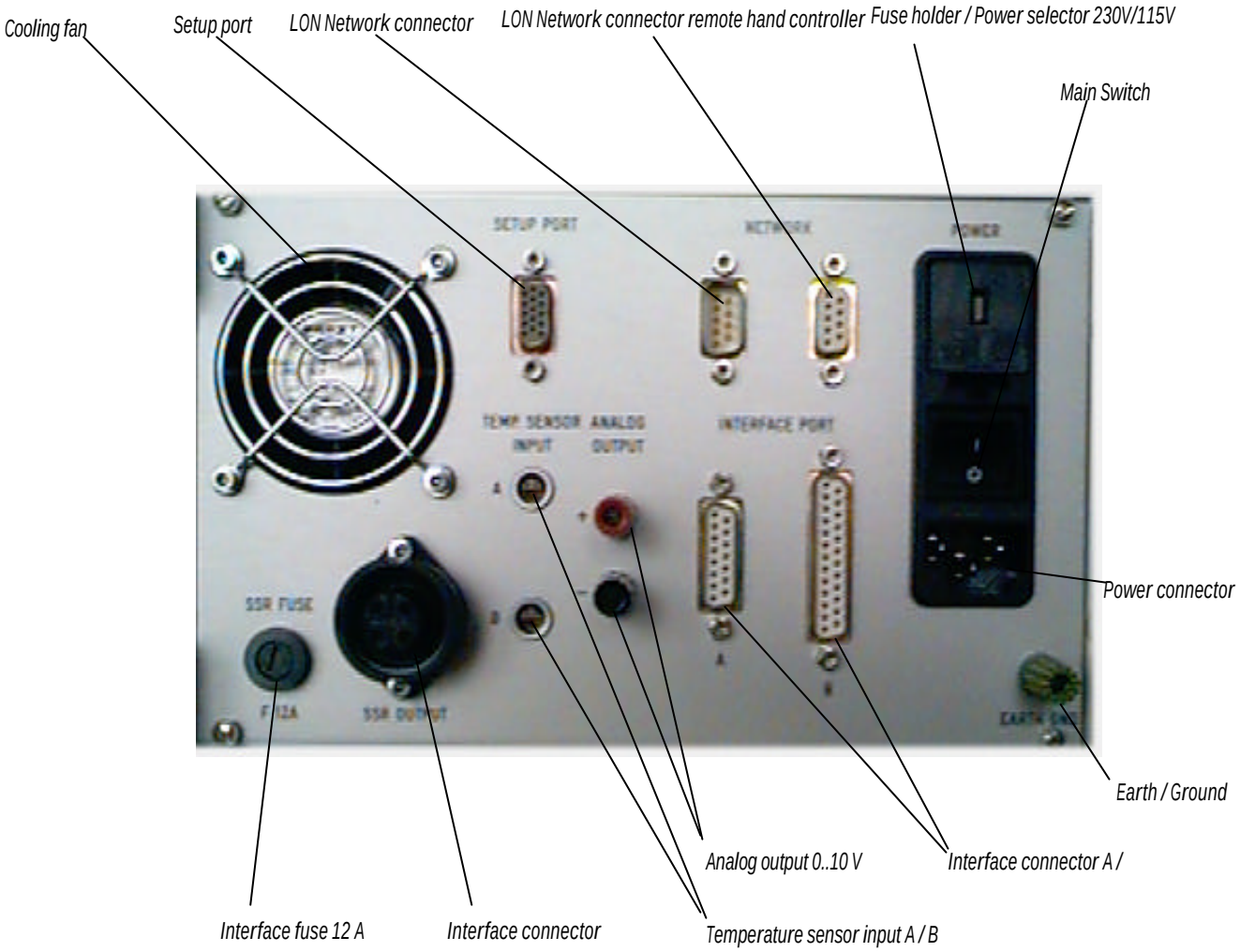


Description Remote Hand Controller

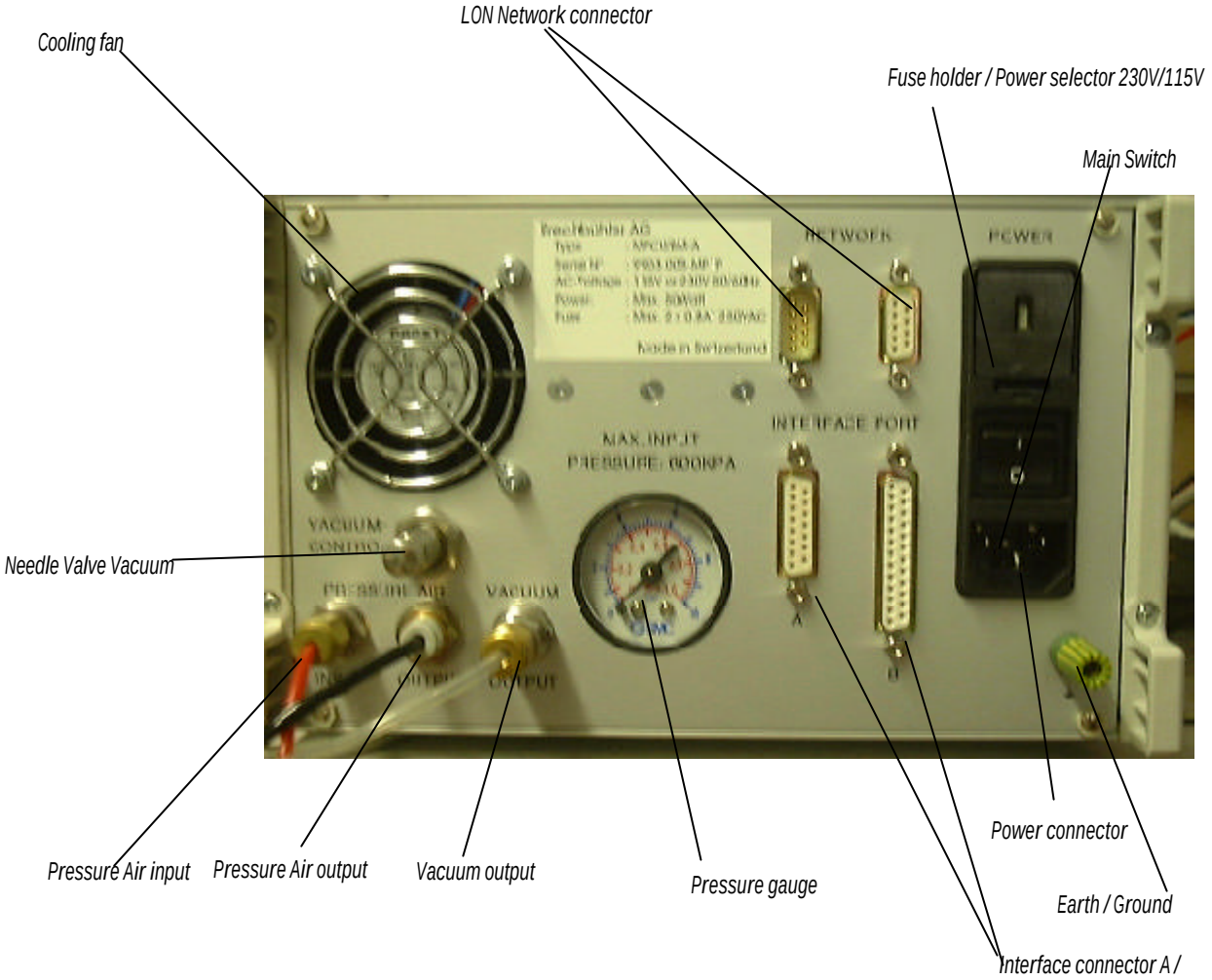
To add a maximum of additional comfort the remote hand Controller has been developed in a way to give the user access to all the necessary parameters like temperature, analogous outlet signal on a very comfortable way and even while seating.



Description Heat controller rear side

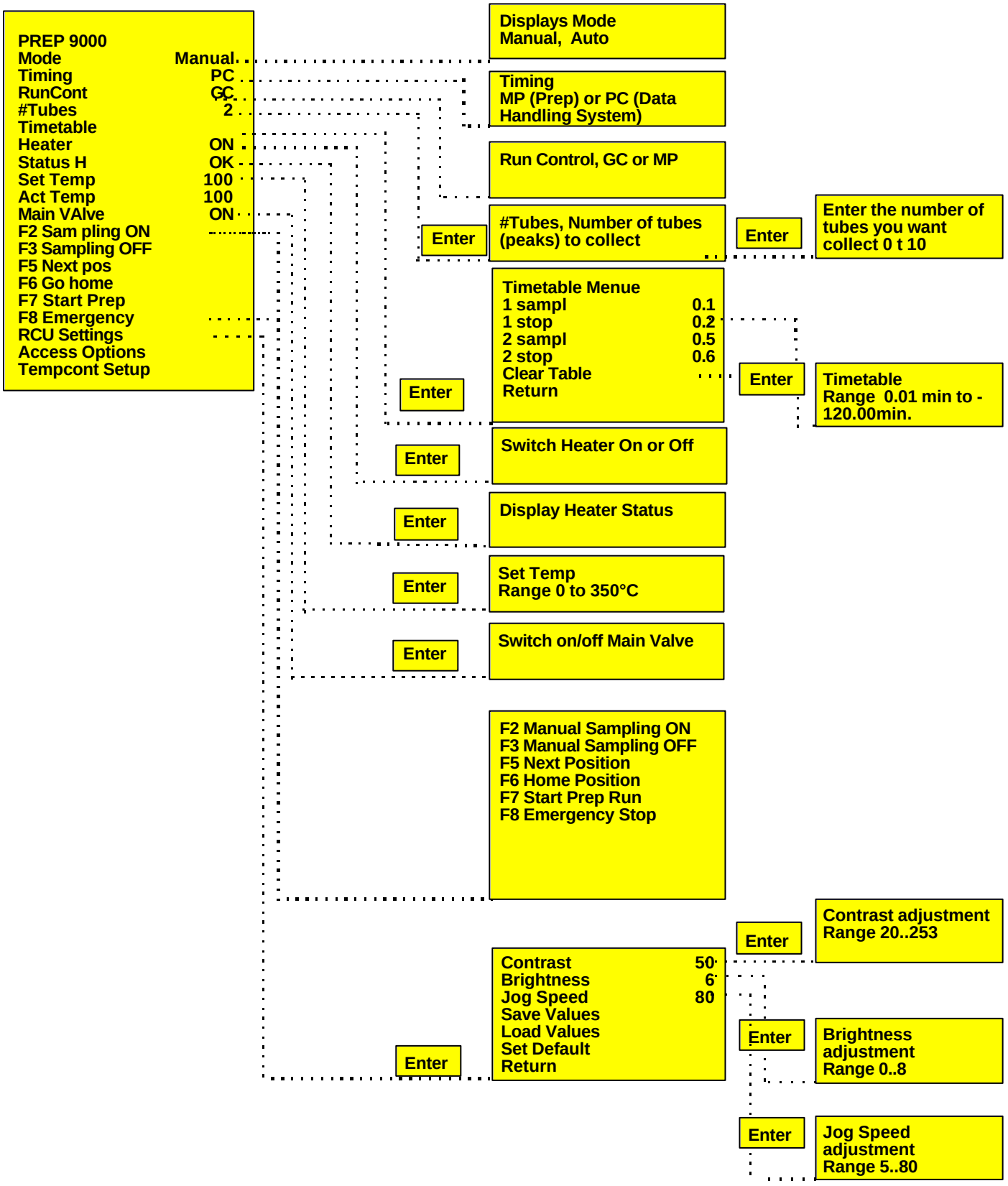


Description PREP controller rear side



Software description Remote Hand Controller

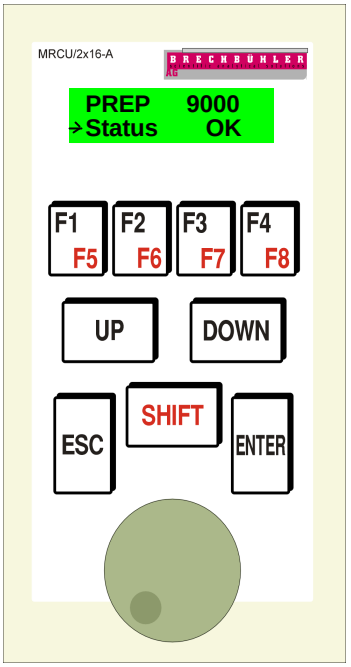
Menustructure Remote Hand Controller



How to use the PREP 9000 menu

The Software in the PREP 9000 hand controller has a built in menu structure which uses a Jogg wheel to move up and down in the different menu options.

How to move in the Menu.....

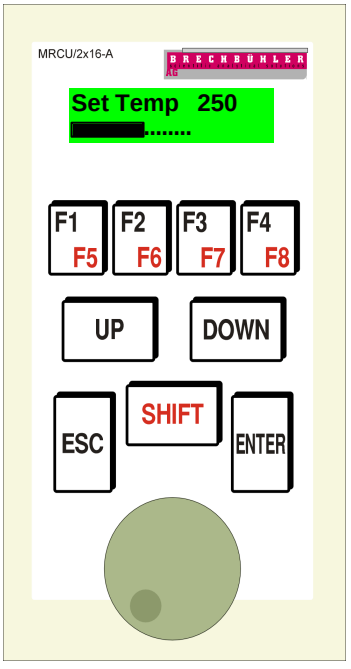


To move down in the menu turn the Jogg wheel clockwise and the menu scrolls upwards.

To move up in the menu turn the Jogg wheel counterclockwise and the menu scrolls downwards.

If you placed the cursor at the menu Option you want press ENTER to open this menu

How to change Values.....



To change Values turning the Jogg wheel clockwise will increase them, turning the Jogg wheel counterclockwise will decrease them.

Values increasing or decreasing resolution is set by default to 1 degree steps.

Pressing the UP key once changes the resolution to 2 degree steps,

pressing the UP key a second time changes the resolution to 5 degree steps,

pressing the UP key the third time changes the resolution to 10 degree steps,

pressing the UP key the fourth time changes the resolution to 20 degree steps,

pressing the UP key the fifth time changes the resolution to 50 degree steps

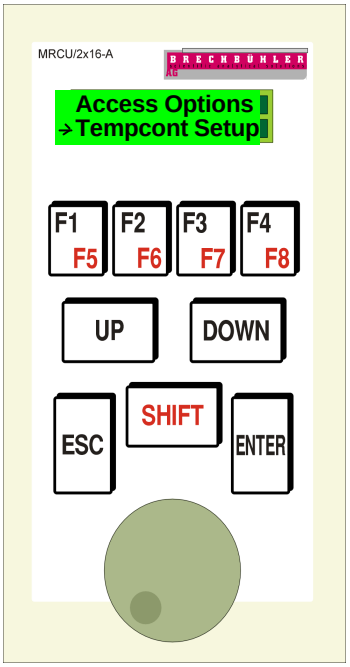
and pressing the UP key the sixth time changes the resolution to 100 degree steps.

To change back the resolution press the DOWN key in the same numbers you pressed the UP key.

F1 to F8 function buttons.....

To access the F1 to F4 press the appropriate function button.

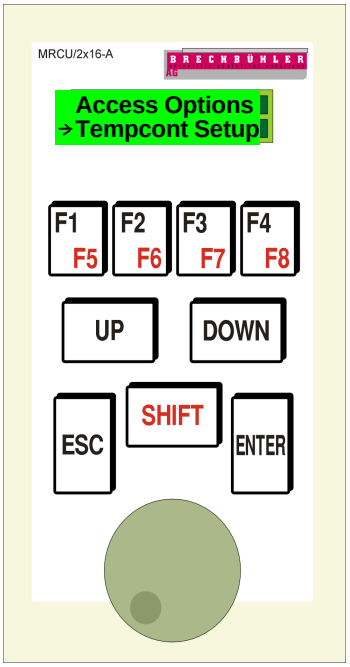
To access F5 to F8 use the red shift button together with the desired function button



The Tempcont Setup Menu.....

By default the Tempcont Setup Menu is disabled. You don't see this Menu point when the Instrument is delivered. The Tempcont Setup Menu is used for Service purposes only.

This Menu point is only accessible by entering a password and has to be used by qualified Service Engineers only.



Note:

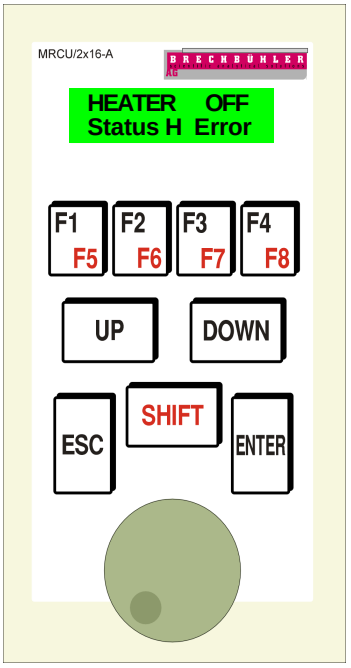
The Tempcont Setup menu allows the Service Engineer to set different values for Testpurposes and allows some Diagnostics in the field.

Switch on the PREP 9000

The PREP 9000 system has a built in Power On check routine which does during power on cycle a internal test to assure that all the components are working correct.

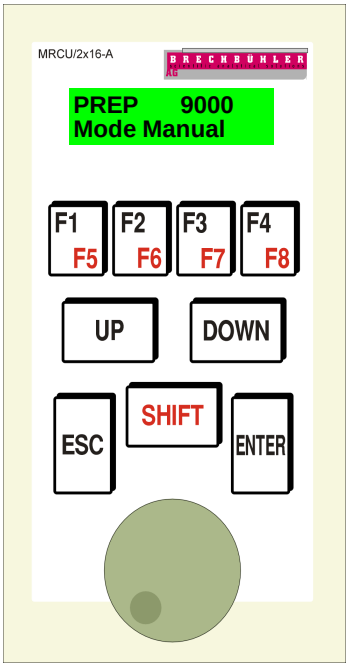
Switch On .....

After switching on the Heater Control unit the display at the hand controller shows for about 30 seconds “PREP 9000”, “Status Error”. During this time the system performs a selftest routine to check if the heater and the thermocouple is present.



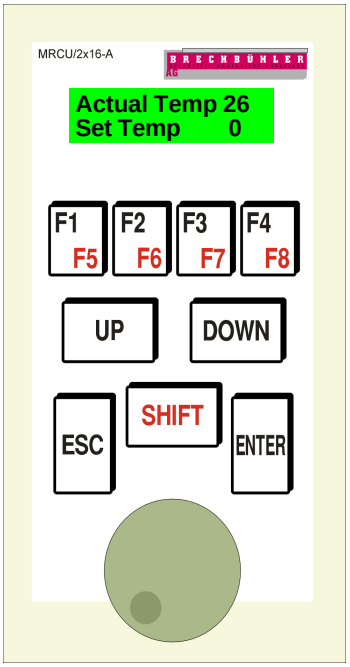
Selftest successfully performed.....

After the self test has been performed and all the checks passed successfully the display at the hand controller shows “PREP 9000”, “Status H OK”. Now the system is ready to start working



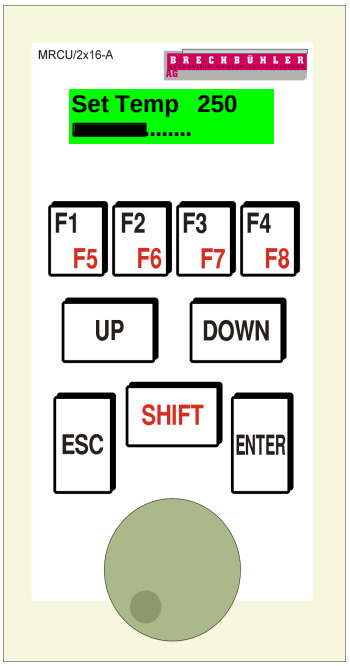
Set Temperature.....

To move the cursor down to the menu “Set Temp” turn the jog wheel to the right side the menu scrolls up and you may point the cursor to the menu “Set Temp”. Value 0 on “Set Temp” means heater is off.



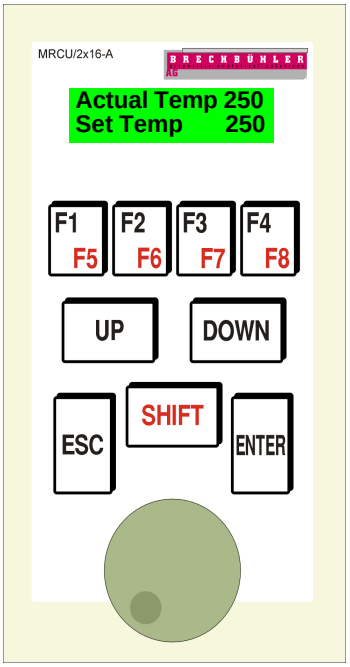
Set Temperature.....save value.....

If you moved the cursor to “Set Temp” press “Enter” to enter in to the Menu “Set Temp”. To increase the set value turn the jog wheel clockwise, to decrease the set value turn it counter clockwise. If you reached the desired value press Enter again to save the value.



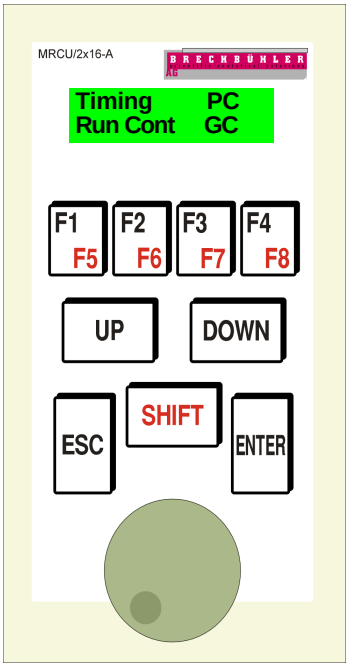
Show actual temperature.....

The system returns to the Main menu, where you now can se the “Act Temp” increasing and the “Set Temp” Value is at 250.



Set Timing....

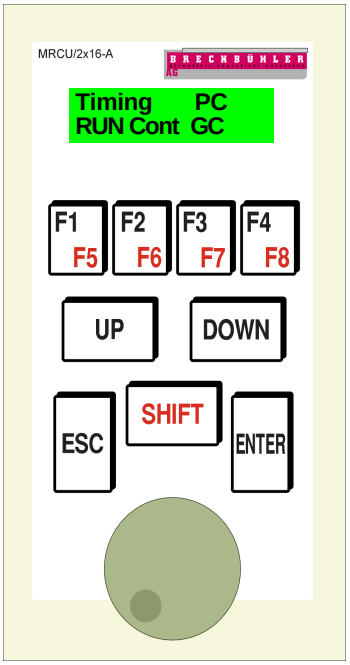
The PREP 9000 allows you to choose the timing source from PC (uses a time event table on your data handling system) or from MP (using the internal time table).



Set timing to PC....

If Timing is controled by PC the collecting times have to be entered at your timed even table on your data handling system.

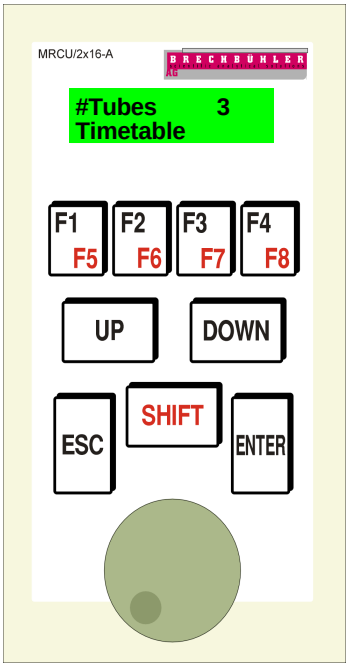
RUN Cont GC means the PREP 9000 system waits for the start and stop signal comming from the GC.



Set timing to MP.....

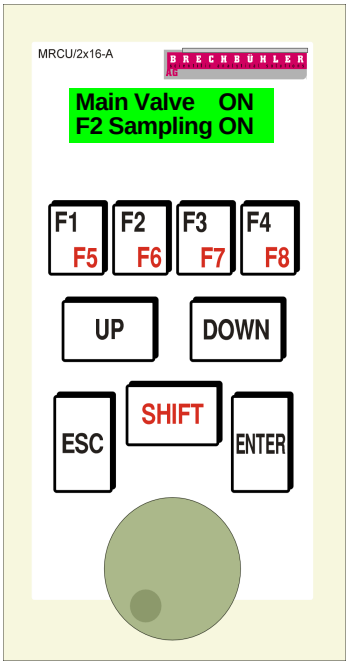
If timing is set to MP the menu will open two additional options.

#tubes	where you can enter the number of peaks you want to collect.
Timetable	where you can enter the appropriate times when your peaks have to be collected.



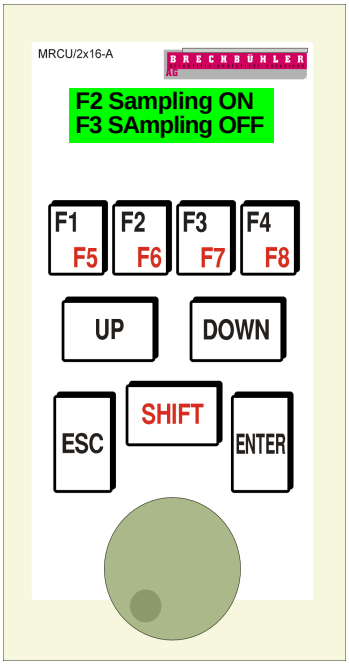
Set Main Valve Off or On.....

If tubes have to be changed the system allows you to switch the Main Valve OFF to assure that you do not have any pressure on the system. In that case the Revolving head can be dismounted.



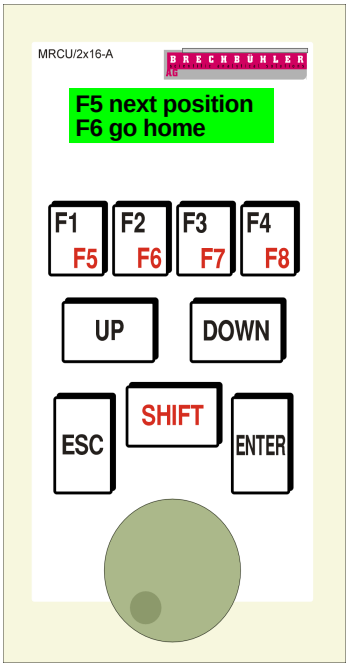
Manual Collecting.....

If you want to collect peaks manually you may choose the F2 key to switch on the collecting valve (Vacuum) or F3 to switch it off again.



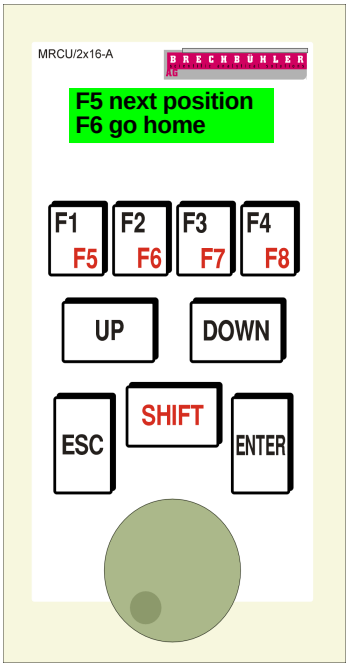
Move to next Position.....

If you want to move the collecting head to the next position use the F5 Key. The collecting head will then change to the next position.



Move to home position.....

If you want to move the collecting head to his home position use the F6 key. The collecting head will then move to his home position (tube number 1)

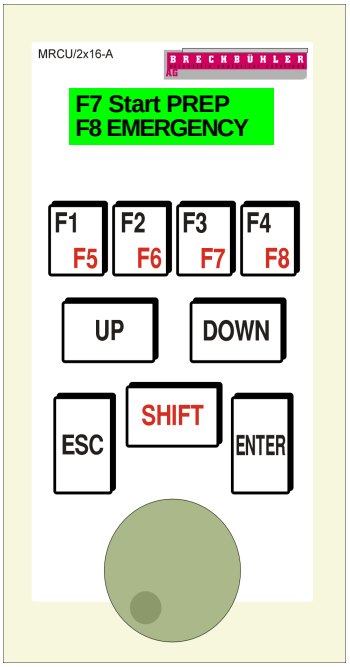


Start PREP Run.....

If you want to start a PREP run press F7. The PREP run will start according to the settings you have choosen in the Timing and Run Cont menu.

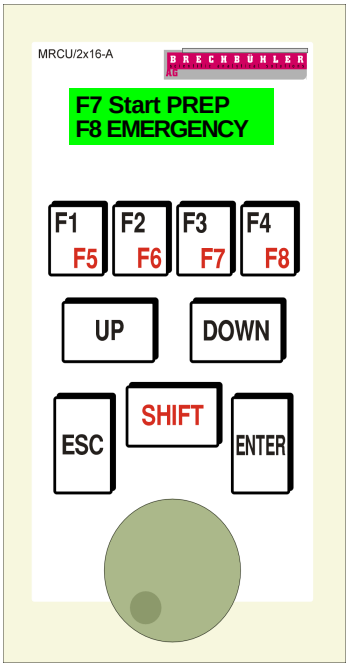
If Timing is MP and Run Cont is MP the system acts as a manual system. In that case you may choose manually the GC start and stop function and the internal time table is used to collect the peaks.

If Timing is MP and Run Cont is GC the system wait for the GC start signal and for the GC stop signal and the internal time table is used to collect the peaks.



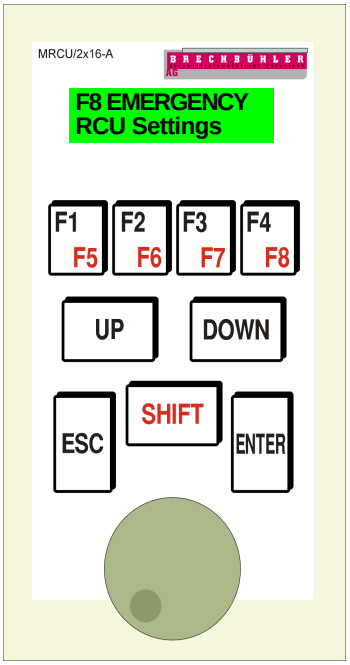
Emergency stop.....

If for any reason you have to stop the system you can use the F8 Emergency stop key. The system will shut down and closes the Main Valve in that case there is no pressure at the pneumatic cylinder.



RCU Settings.....

To enter in to the RCU Settings menu move the cursor down to “RCU Settings” and press “ENTER”. In this menu you may adjust the Contrast and Brightness of the display, the speed of the jog wheel and you may set all the values to default parameters. To exit this menu move the cursor down to “Return” and press “ENTER”. The menu points “Save Values” and “Load Values” are not active. These menus are for future extensions.



Error Messages

Exeptional Error Messages.....
These error Messages should not apply if system is correct handled

Error	Description
1	Voltage Problem at the MOCU inputs
2	One or both of the Carusell sensors are defective. Sys tem shuts down. Main Valve will be closed.
3	Error during sampling. System shuts down. Main valve will be closed.
4	Carusell not in down position. System shuts down. Main valve will be closed.
5	Carusell up then down. Should be in down position. Sys tem shuts down. Main Valve will be closed.
6	Carusell down then up. Should be in up posoition. sys tem shuts down. main Valve will be closed.
7	Rotating cylinder not back. System shuts down. Main Valve will be closed.
8	Rotating cylinder not forward. System shuts down. Main valve will be closed.
9	Wrong Sensor. system shuts down. Main valve will be closed. Power On Reset necessary.
10	Carusell not up. System shuts down. Main valve will be closed.
11	Time out during movement.
12	Comunication time out. Network is disconnected.

Manipulation Error Messages.....

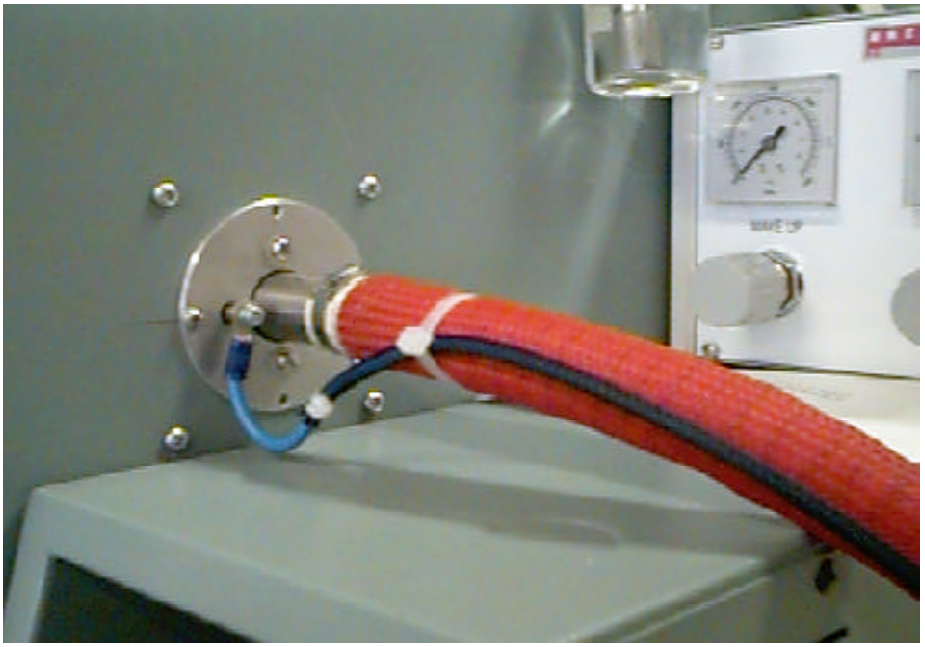
Error	Description
100	Main valve not open.
101	#tubes are not added. Please enter the appropriate num bers of tubes.
102	Heater error. The Heater is off or the set temperature is not achieved.
103	Sampling is not possible
255	EMERGENCY was pressed or a unknown error hap pened. System shuts down. Main valve will be closed.

Installation of the PREP 9000

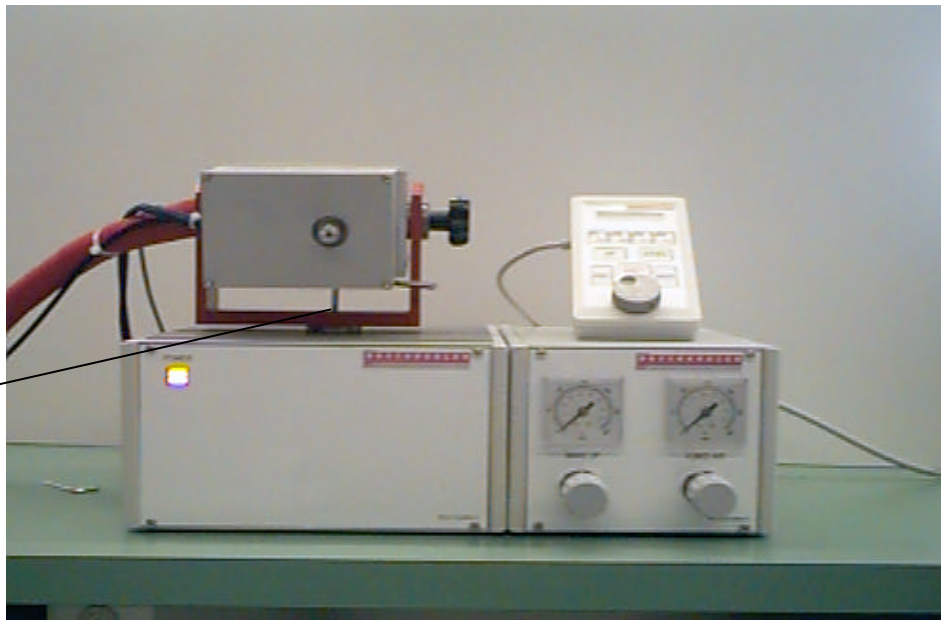
Mechanical installation.....

The PREP 9000 may be installed to any GC available on the market. The most GC's in the field do have on the left or on the right side already a whole to enter with a interface. You may choose the appropriate whole which fits to the needs of the flexible heated interface of the PREP 9000 or you have to drill a new whole to install the flexible heated interface to your GC.

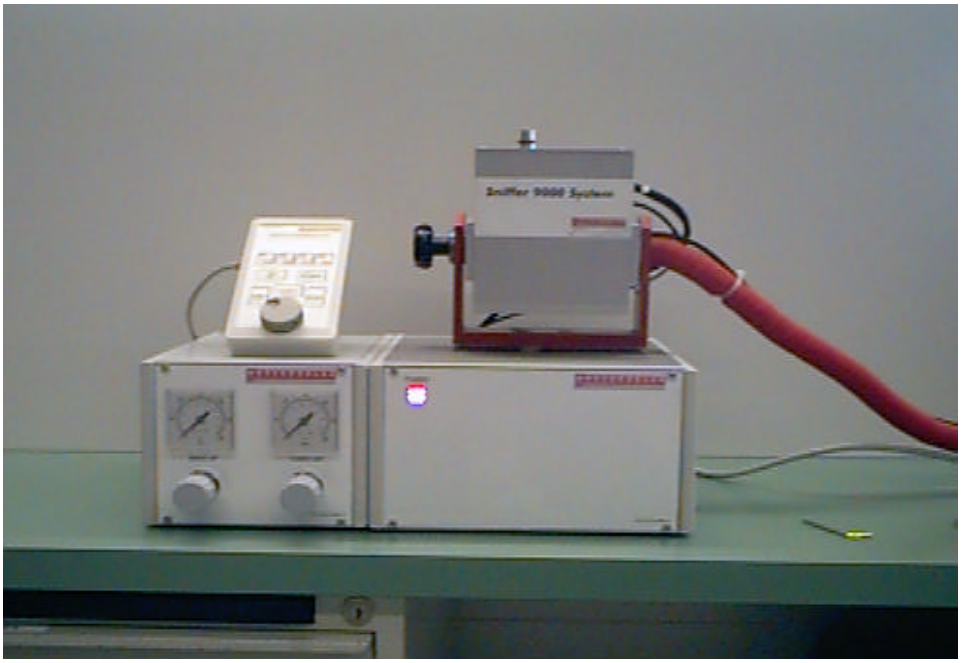
You may fix the interface by using the aluminium support and the included screws in the standard outfit as you see on the picture. Please note that the interface is using a direct heating system. Therefore it is mandatory that you use only the aluminium support which is delivered with the PREP 9000 system otherwise you may have a short circuit to the GC which may result in damaging the heat controller of the PREP 9000 system



To change the installation side from the left to the right side (or vice verca) turn the Interface outlet support in a 90 degree angle to the front. Use the include Inbus key to loosen the fixing screw then turn the complete support to the desired position and fix the screw again

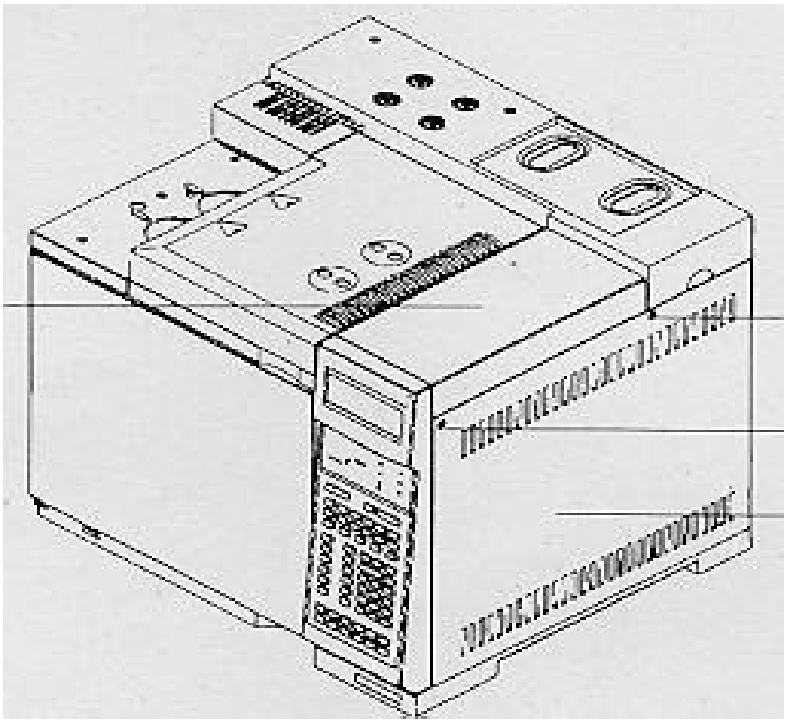


After you changed the Interface direction to the desired position and you fixed again the support you may install the Interface to the GC.



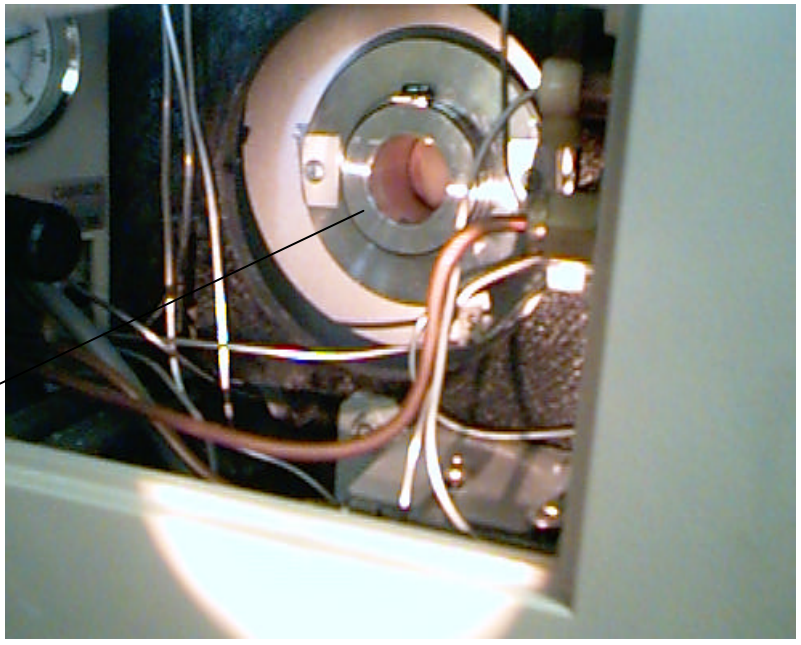
Installation on a HP 5890 / 6890.....

On HP 5890/6890 the PREP9000 may be installed on the left or on the right side. It depends what type of hyphenated detectors are installed to the Instrument. If there is no IRD or MSD installed on the left side of the GC usually the PREP9000 will be installed at the left side using the existing hole for the MS Interface.



Installation on a HP 5890 (left side).....

If your GC HP 5890 is not equipped with a MSD you may enter with the PREP9000 interface from the left side of the GC oven. Mount the included interface support at the outer oven wall and the you may easy insert the Interface in to the support and fix it with the screw at the interface support.



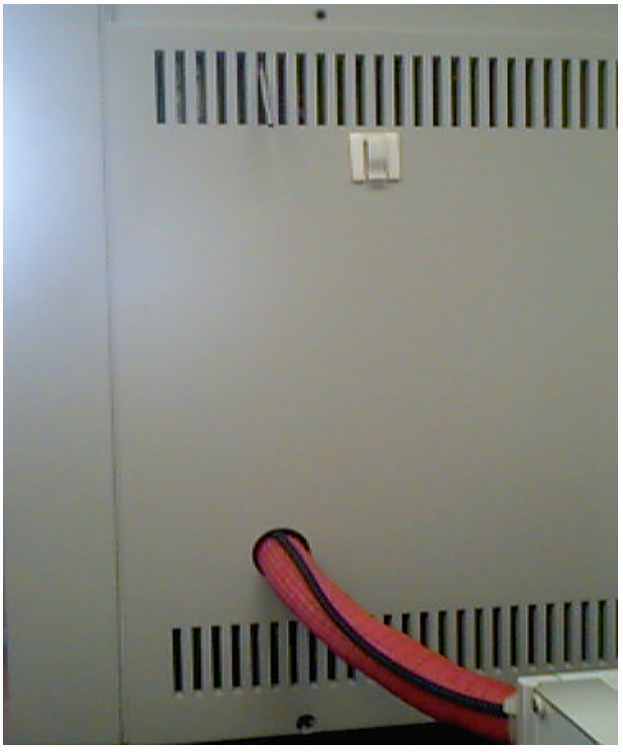
Installation on a HP 5890 (right side).....

If your GC HP 5890 is equipped with a MSD you must enter with the PREP9000 interface from the right side of the GC oven.

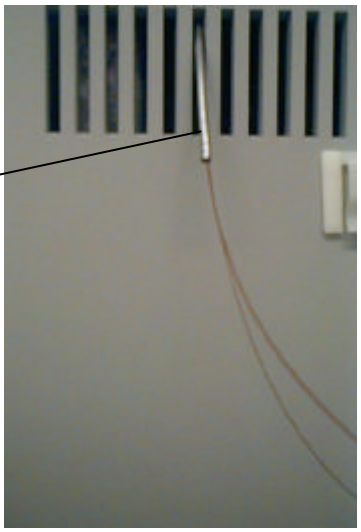
Drill a hole of 40 mm of diameter trough the entire oven wall below the electronic and mount the included interface support at the outer oven wall and the you may easy insert the Interface in to the support and fix it with the screw at the interface support.

Aluminium Interface support

After fixing the aluminium support you have also to drill a hole in to the outer plastic cover of the GC.



You can install a 1/8” stainless steel tube as guide for the fused Silica lines coming from the Pressure controller fore make-up gas and humid Air to enter easy in to the oven



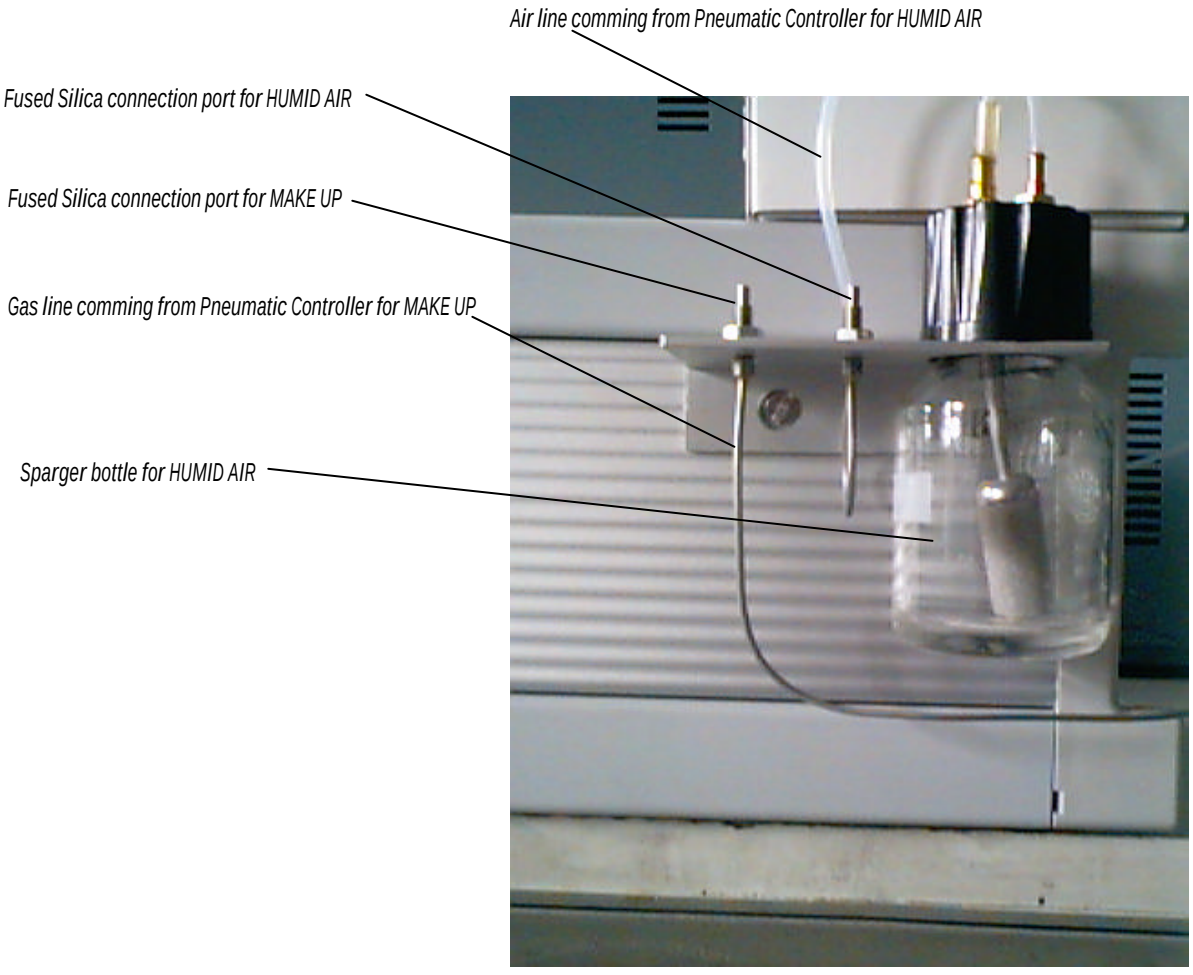
Stainless steel tube going in to the GC oven used as guide for the fused silica lines.

Pneumatic installation.....

The Pressure control unit is equipped with Pressure regulators and pressure gauges for make-up gas as well as for the humidification air. The PREP 9000 uses only the Make-Up line. On the rear side of the pressure control unit there are the connectors described as INPUT to connect the PREP 9000 to a existing gas supply in the lab.

Make-Up:	Nitrogen max. pressure 300 kpa
Humid-Air:	Synthetic Air max. pressure 300 kpa

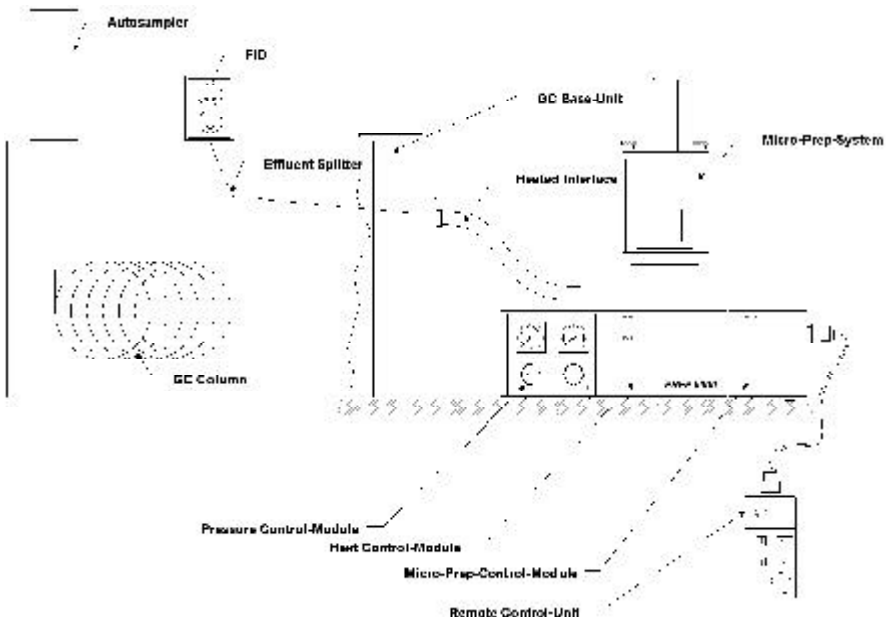
The OUTPUT Connectors are usually factory installed to the support mounted on the left or right side of the Pressure Controller. These lines goes to the appropriate fused silica line connectors described as MAKE-UP and HUMID AIR.

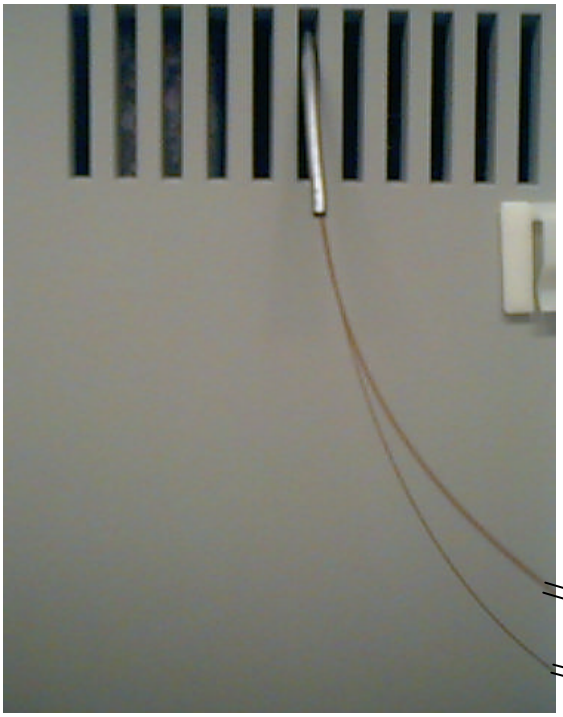


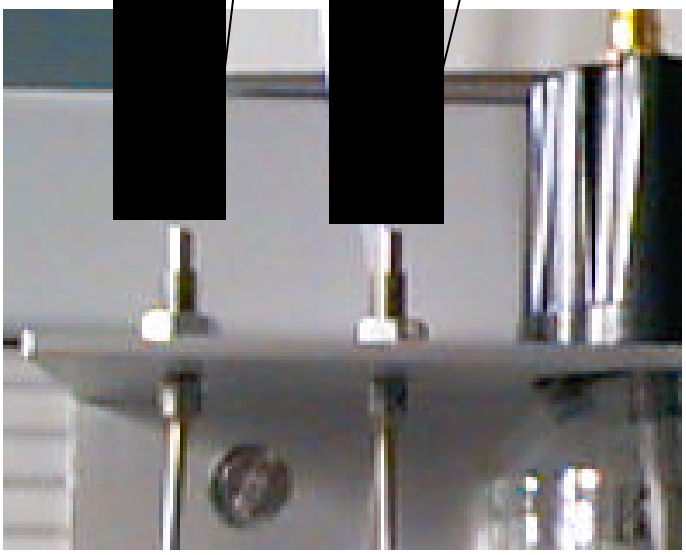
Analytical installation.....

After the PREP 9000 is physically connected to the GC we need to install the 4-way splitter inside the oven, which allows us to connect the make-up gas. The 4-way splitter usually is a pressfit connector which allows us to install fused silica- lines coming from the pressure controller of the PREP 9000.

At the left or right side of the pressure controller we have a special designed support where the fused silica lines going to the GC oven may be connected. Inside the GC oven we have a pressfit 4-way splitter where we connect the make-up gas line. Additionally we do have a special designed metallic piece which allows the connection of the hunid air line.







Fused Silica line ID 0.32 MAKE UP (going to GC Oven)

Nut 303 M4 (cod. 35032423)

Ferrules ID 0.45mm (cod. 29013487)

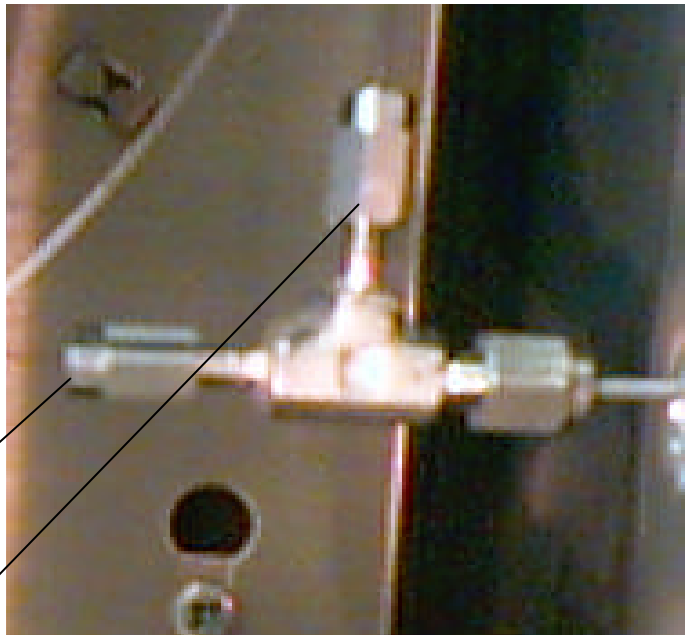
Fused Silica line ID 0.53 HUMID AIR (going to GC Oven)

Nut 303 M4 (cod. 35032423)

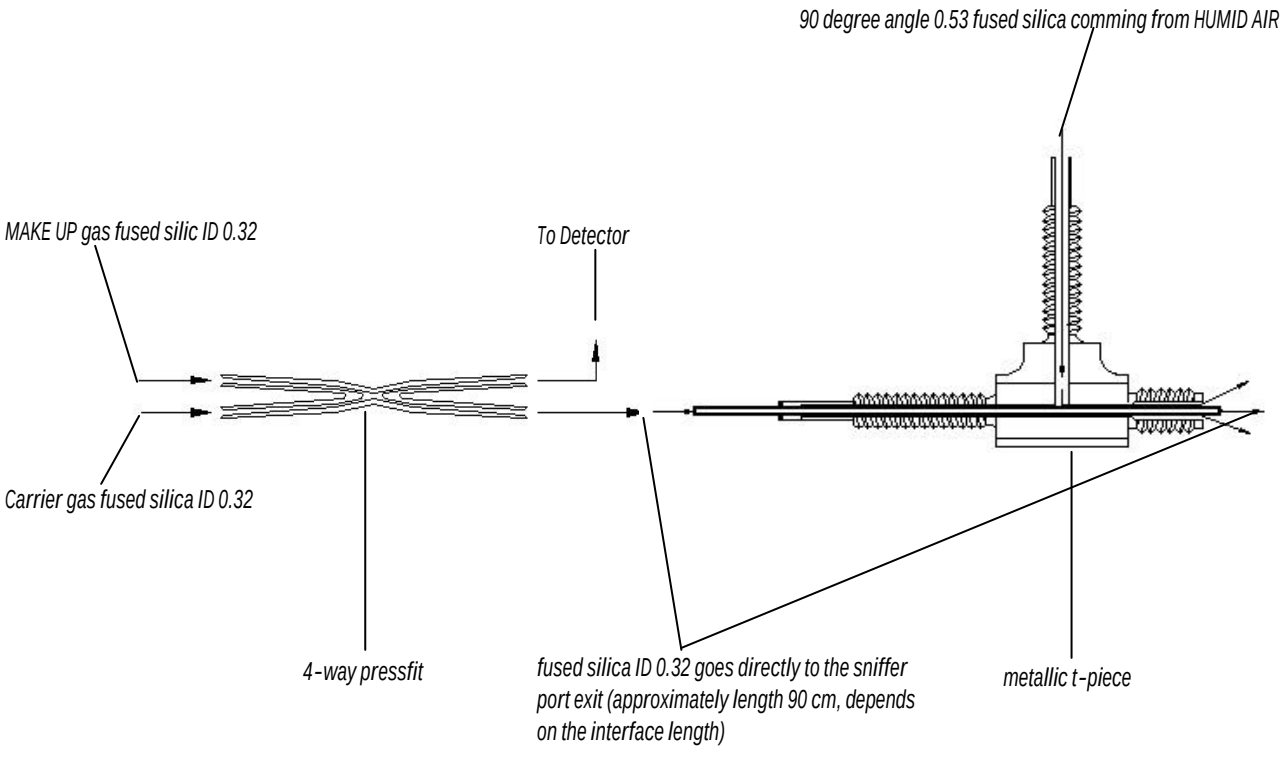
Ferrules ID 0.80mm (cod. 29013486)

Important!!!.....
Please take care that the Interface metallic part (including the metallic t-piece) does not make any contact to the GC oven wall. Due to the fact that the dedicated heated interface is using direct current to the interface metallic tube, you may create a short circuit, which may influence the temperature regulation.

T-Piece installation!!!.....
After you have installed your interface you have to connect the metallic t-piece to the interface outlet



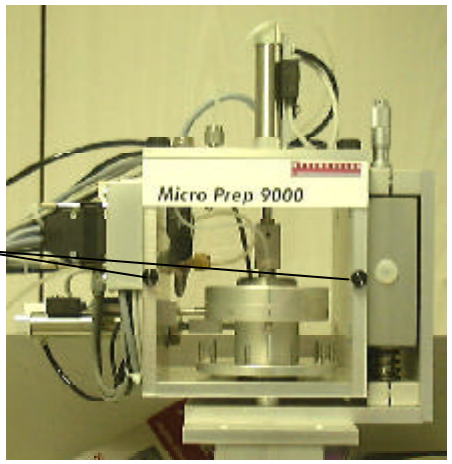
Schematic of the fused silica connections.....
Here below you see the schematics how to connect the fused silica lines to the PREP 9000 interface



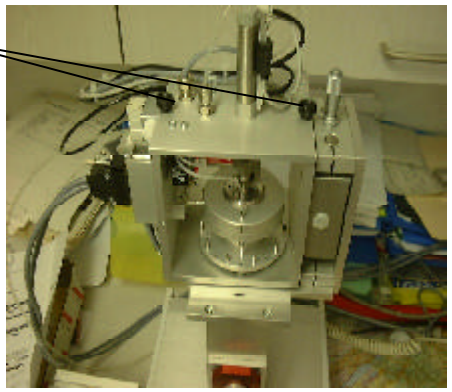
How to dismount the collecting head.....

To dismount the collecting head you have to switch OFF the Main Valve first (to assure that there is no pressure anymore at the pneumatic cylinders).

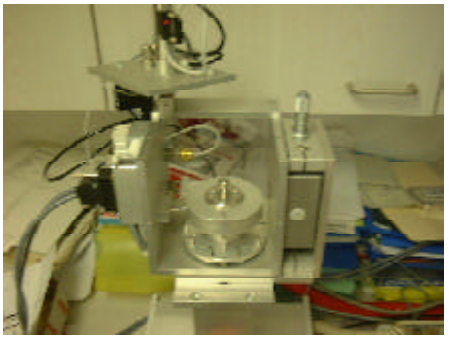
By loosen the two screws at the Plexi cover you can get access to the collecting head compartement.



Unscrew the two screws at the upper aluminium support.

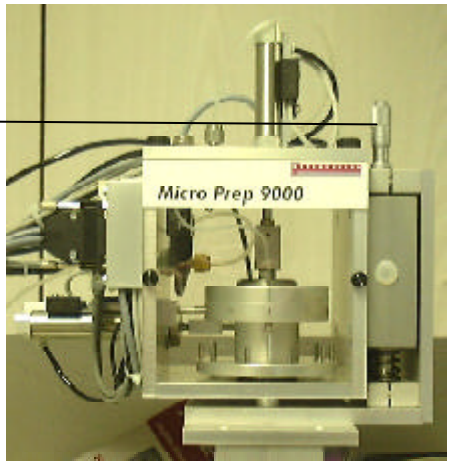


Move the upper aluminium support .



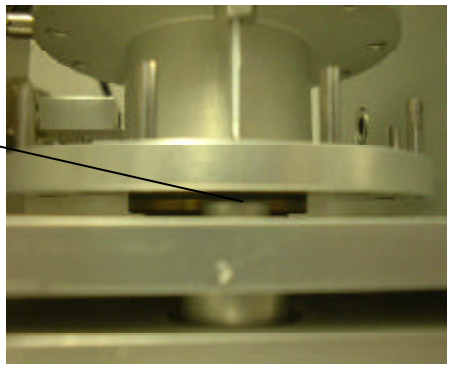
.....lift out the collecting head.

How to adjust the collecting head.....
To adjust the height of the collecting head use the micrometric screw on the right side.



Byturning the micrometric screw clockwise the head will move down and by turning it counter clockwise the head will move up.

be sure that the seals are thightening at the interface outlet.

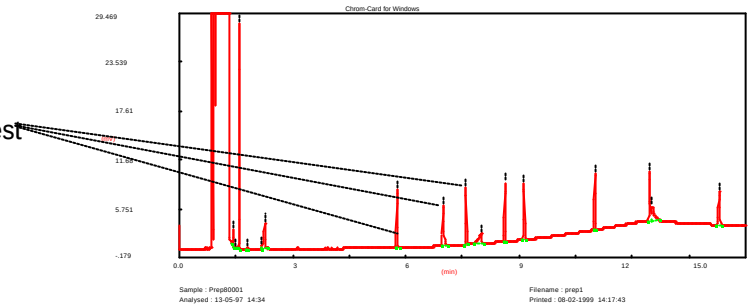


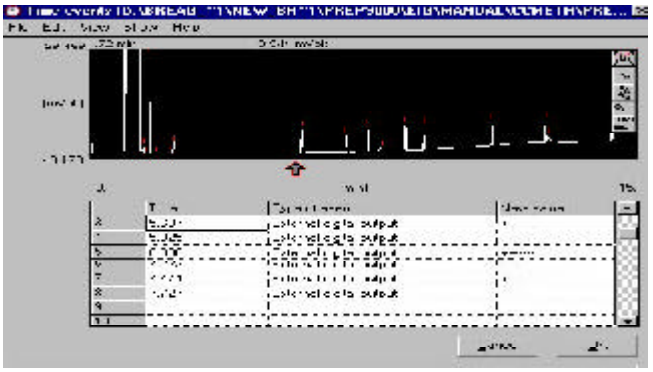
Analytical procedure

Reference Chromatogram.....
Inject a first refernce Sample to your GC to obtain a refernce Chromatogram.

Chosse the appropriate retention times for your peaks you want to collect

3 peaks of interest

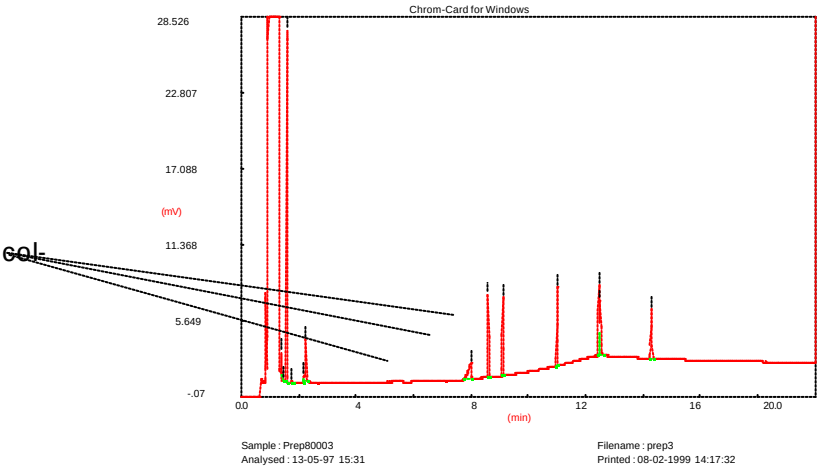




Choose collecting time.....
Enter the collecting times in to your time table of your data handling systemor in to the internal time table of the PREP 9000.

Collecting your peaks.....
After your time table has been set the automatic collecting of your peaks of interesst can beginn.

3 peaks of interest collected.



Interface connector description

Interface Port A (DSUB 15 Pol)

Name	Pin-Nr.	Description
11A Sink_A1	1	Lifting Valve (Up/Down Cylinder/ 24V)
12A Sink_A2	2	Rottating Valve (Forward/Back Cylinder/ 24V)
13A Sink_A3	3	Collecting Valve (24V)
23A INA_1	4	Sensor Home Position (24V SPS Input)
24A INA_2	5	Sensor Karussell Down (24V SPS Input)
25A INA_3	6	Sensor Karussell Up (24V SPS Input)
13C Relay_B1_NC	7	Sensor Rottating Cylinder Back (26A_INA_4/ SPS 24V Input)
15C Relay_B1_NO	8	Sensor Rottating Cylinder Forward (26A_INA_4/ SPS 24V In)
not connected	9	
not connected	10	
24V	11	Internal Powersupply
24V	12	Internal Powersupply
24V	13	Internal Powersupply
GND (24V)	14	Internal Powersupply
GND (24V)	15	Internal Powersupply

Interface Port B (DSUB 25 Pol)

Name	PIN	Description
23C Relay_B4_C	1	Gas-Chromatograph start signal
22C Relay_B4_NC	2	Gas-Chromatograph start signal
24C Relay_B4_NO	3	Gas-Chromatograph start signal
18A Sink_B4	4	Gas-Chromatograph start signal (5V Sink/ PGND internal)
32C TTL_OUT_B4	5	Gas-Chromatograph start signal (5V TTL Output/ PGND internal)
28A INB_1	6	GC/AS Start (5V TTL)
29A INB_2	7	GC/AS Stop (5V TTL)
30A INB_3	8	Collecting Control (PC/ Chrom Card/ 5V TTL)
TTL_V_EXT	9	
TTL_GND_EXT	10	
INB_GND_EXT	11	
not connected	12	
not connected	13	
not connected	14	
not connected	15	
VCC	16	5V Digital power source (galvanic isolatet) on DIOCU Board
GND	17	5V Digital power source (galvanic isolatet) on DIOCU Board
V_AUX	18	5V Power source on DIOCU Board
V_AUX	19	5V Power source on DIOCU Board
PGND	20	5V Power source on DIOCU Board
PGND	21	5V Power source on DIOCU Board
24V	22	Internal Powersupply max. current 0.4A
24V	23	Internal Powersupply max. current 0.4A
GND (24V)	24	Internal Powersupply max. current 0.4A
GND (24V)	25	Internal Powersupply max. current 0.4A

Ordering informations

Part No.	Description
	Base Unit
9 1005002	PREP 9000 Micro Preparative Collecting system includes: Dedicated flexible heated interface Heater control unit Hand held control unit Preparative fraction collector for up to 10 adsorption tubes Standard outfit, Manual

	Accessories / Spare parts
9 1000055	Flexible heated interface
9 1005110	Empty glass adsorption tubes (set of 10)
9 1005111	Empty glass adsorption tubes (set of 50)
9 1005120	Filled adsorption tubes Porapack Q (set of 10)
9 1005121	Filled adsorption tubes Porapack Q (set of 50)
9 1005130	Filled adsorption tubes Charcoal (set of 10)
9 1005131	Filled adsorption tubes Charcoal (set of 50)
9 1005140	Filled adsorption tubes TENAX (set of 10)
9 1005141	Filled adsorption tubes TENAX (set of 50)

Manufactured by:

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