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Agilent Technologies
Channel Partner

MANUAL

SIM Direct Inlet Probe for Agilent 7000 Triple Quadrupole

from version 1.10e

Document History
Edition 7000e 10/2010, from Firmware / Software Version V1.10 and dip.v.1.2.

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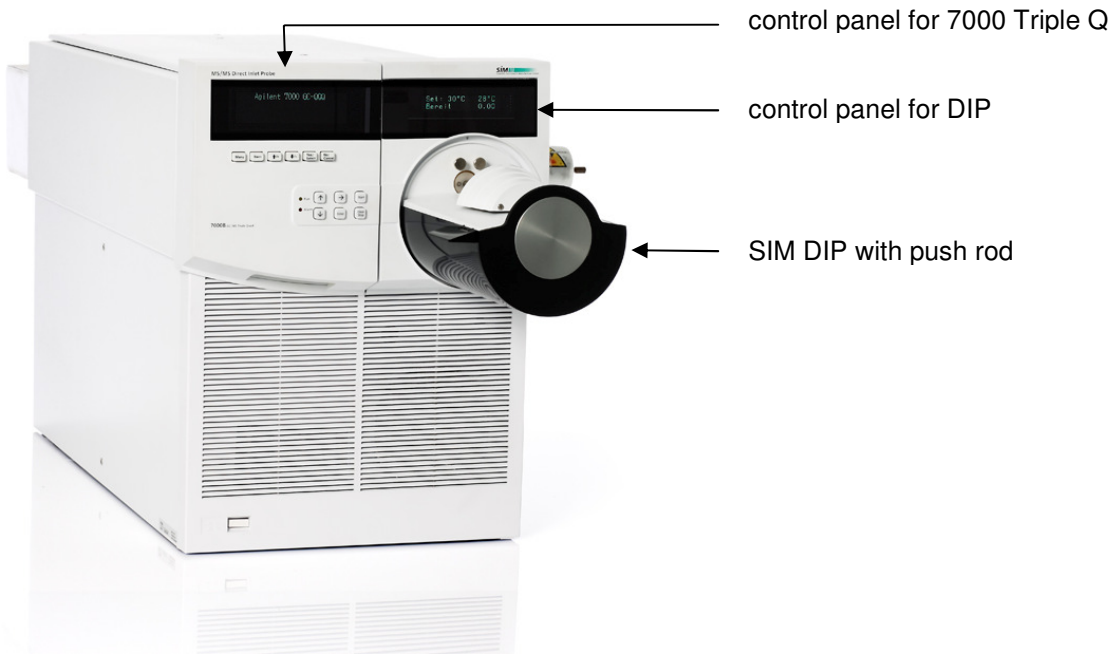
CONTENT

1. System Information

The **Direct Inlet Probe** (**DIP**) **System** consists of a modified Agilent Technologies 7000 Triple Quadrupole and a push rod to insert samples directly to the ion source of the mass spectrometer. It enhances the application range of the MS because determination of mass spectra is possible without using the gas chromatograph. The DIP system has been carefully optimized for the 7000 Triple Q: A funnel to the ion source as well as special seals along the vacuum lock ensure proper system performance while maintaining a high vacuum in the quadrupole. In delivery condition, the system is equipped with a probe tip for liquid samples which can be changed easily to the probe tip for solid samples.
Moreover, time consuming changeover from GC/MS to DIP/MS and vice versa is not necessary – the GC/MS interface need not to be uncoupled for DIP/MS operation.
For data analysis the Agilent Mass Hunter software is used, already existing MS searching libraries can be applied.



1.1 Mode of operation

Fig. 1 DIP system

A push rod is used to transfer the sample from atmospheric conditions through a vacuum lock into the high vacuum ion source (EI or CI) of the mass spectrometer:
In the “ready” mode the push rod tip is inside the DIP in front of the valve (Fig.1). For sample loading, the push rod moves into the 90° position (Fig. 2, left) outside the DIP.
The sample is placed into the sample vessel of the probe tip. Then the push rod is introduced automatically step-by-step into the high vacuum of the mass spectrometer up to the funnel to the ion source. When the tip of the push rod is directly in front of the ion source funnel, the tip is heated corresponding to the temperature program (temperature program with 3 ramps, heating rate 0.1 to 2.0 °C/s); this permits the thermal separation of the various analytes in the sample so that the delayed recording of the mass spectral data is possible.

The automatic insertion of the sample into the ion source is controlled by a microprocessor minimizing the change of accidental venting of the mass spectrometer.
The microprocessor also enables heating and cooling of the probe tip, namely heating by an heating element and cooling by compressed air.

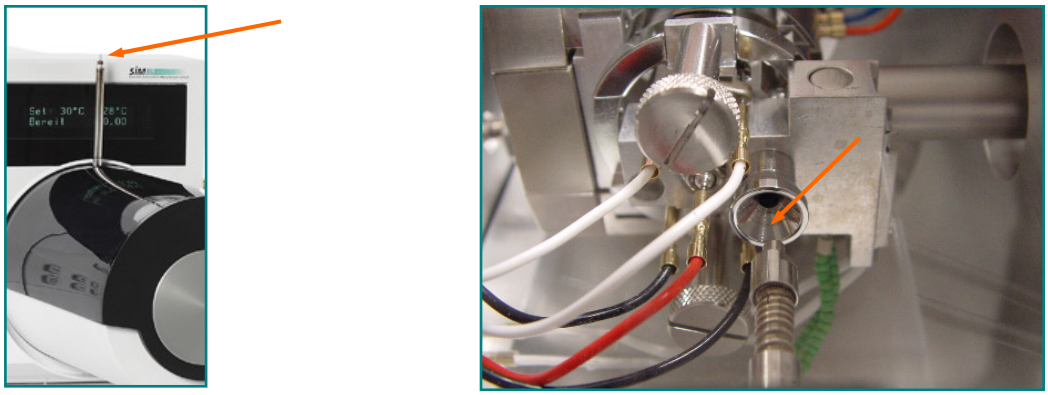


Fig. 2 Details of the DIP
Sample vessel for liquid samples at the push rod tip (on the left, sample vessel see arrow) and the probe tip on the verge of the ion source funnel (on the right, probe tip see arrow)

1.2 Control panel

Fig. 3 Control panel of the MS Direct Inlet Probe

The control panel acts as local user interface to the DIP. All control commands (general operation, maintenance) can be entered at this panel. You can perform some basic tasks such as start of determination and entering heating rates from the control panel as well as from the computer (see 1.3).

1.3 Controlling software

Using the software “DIP”, you can enter the temperature program user-friendly at the PC. The keys “Start”, “Stop” and “Clear” are consistent with the keys of the control panel:
The values of the temperature program are entered into the belonging data fields. Total run time will be calculated and displayed.
The status display shows the ready-signal, run time, actual temperature and position of the push rod. The last line shows the position of the push rod together with the valve status.

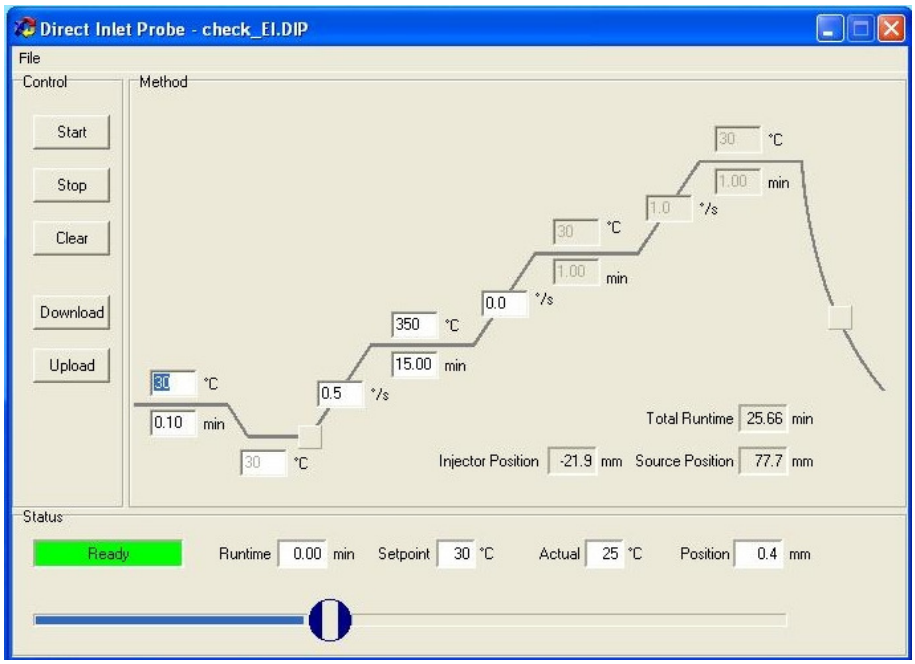


Fig. 4 DIP-Software

For GC/MS operation and general operation of the EI and CI MS see “Hardware Manual of the Agilent 7000 Triple Quadrupole GC/MS”. Special tasks referring to the DIP are described below (**see 2-4!**).

2 To vent the MS

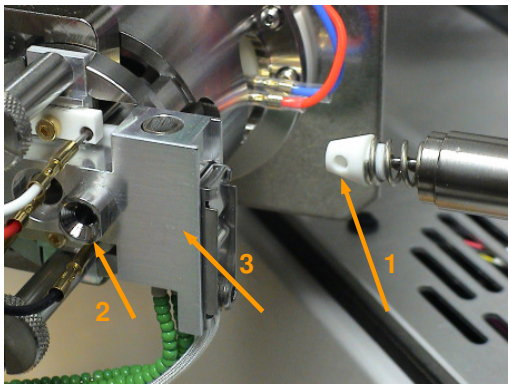
Please follow the instructions of the Agilent Hardware Manual (but you cannot use the vent valve knob!):
Go to the MS Tune section and select the tab “Manual Tune”. In Vacuum control you have to push the “Vent” button. The vent program cools the GC/MS interface heater and the GC oven. The program turns off the analyzer heaters and the high vacuum pump. The software will prompt you when it is safe to switch off the power.

1. When prompted, turn off the Triple Quadrupole MS power switch.
2. Select the service mode “Vent” at the DIP controller (see 5.4.2) instead of turning the vent valve knob to admit air into the vacuum manifold.

3 To prepare the system for CI operation

3.1 To install and to clean the CI ion source

Please follow the instructions of the Agilent Manual but pay attention to the difference in installing the **CI interface tip seal**, because this seal has a bore hole for the direct inlet:



1. Remove the seal from the ion source storage box.
2. Place the seal over the end of the interface so that the bore hole of the seal (1) is aligned to the ion source funnel (2) which is integrated within the CI source heater assembly (3).

Note: Pay attention with the DIP ion source funnel (arrow number “2” in the photo above)! Even if the enamel surface of the funnel is very robust, it should be checked visually for damage in case of ion source changing/installation.

4 To adjust the position of the push rod in the ion source

After changing or installation of a new ion source (but not after every cleaning procedure) or changing of the push rod tip, the ideal position of the push rod has to be adjusted in the ion source:

1. Select the service menu “Auto” at the controller (see 5.4.1).
2. Move the push rod into the ion source up to the ideal position by following the instructions given for the service menu “Auto”.

5 Controller with menu-driven handling

5.1 Control elements

On the left of the control panel (see marking in the photo above), there is a yellow light for “Run” and a red one for “Alarm”.

Meaning of the six keys at the control panel:

↑	to scroll forwards in the menu to increase a value to move forwards
↓	to scroll backwards in the menu to decrease a value to move backwards
→	menu selection forwards cursor to the right (resp. to the left by continuing pushing)
Enter	to acivate the entry (cursor appears) to confirm the entry
Start	to start the program
Clear Stop	to stop the program to reset the controller

5.2 Basic display

first line: temperature set value (left) and actual value (right)
second line: system status and actual run time

Note: Only the keys “arrow right” (menu selection), “start” and “clear/stop” are active in the basic display.

The system status is displayed during all actions relating to the push rod. Possible values are listed:

system status	**meaning**
Initialize	initialize
Fault	fault (see below)
Not Ready	not ready
Ready	ready, push rod in front of the valve (inside)
Idle Pos	horizontal position, push rod in front of the vacuum lock (outside)
Load Pos	90° position for sampling
Move out	push rod moves out
Clos.valve	valve is closed
Calibr.Pos	position for calibration
Pre-Heat	pre-heating of the sample vessel
Move in	push rod moves in
Open valve	valve is opened
Wait	wait
Ramp 1	temp. program is at ramp 1
Final 1	temp. program is at the final temp. of ramp 1 (dwell time)
Ramp 2	temp. program is at ramp 2
Final 2	temp. program is at the final temp. of ramp 2 (dwell time)
Ramp 3	temp. program is at ramp 3
Final 3	temp. program is at the final temp. of ramp 3 (dwell time)

In case of the system status “fault” the fault will be defined as follows:

fault status	**meaning**
Communicat.fault	communication fault
Manual stop	manual stop of the program
Drive1 blocked	drive 1 is blocked
Encoder fault	encoder fault
Drive1 overload	drive 1 is overloaded
Drive1 not oper.	drive 1 is out of service
Drive2 overload	drive 2 is overloaded
Drive2 not oper.	drive 2 is out of service
TC Fault	thermocouple fault
Heater fault	heater fault
Safety Switch	safety switch of the covering cap
Maintenance	maintenance operation
Probe missing	no sample tube in the push rod tip (see 6.3.2)
ProbeSensorFault	light barrier fault, please call service engineer

5.3 Main menu: temperature program

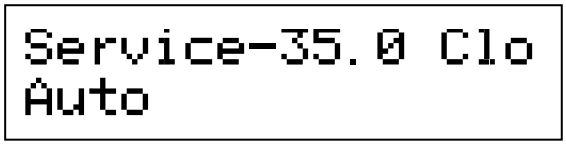
1. Select the main menu with a click on the “menu selection” key (arrow right).
2. Choose the selected parameter with the arrow keys “up” and “down”.
3. Activate the entry by pressing the “Enter” key and a blinking cursor will appear.
4. Enter the value with arrow keys “up” and “down”. The cursor can be moved with the arrow key “right”.
5. If the correct value is displayed, close the entry by pressing the “Enter” key again.
6. If the entered value is unallowable, the display returns to the default value.

Note: All changes of the parameter values take effect directly!
The display skips back to the basic display after a few seconds.

Parameter	Min	Max	Default.	Unit
Pre-Heat Temp	30	100	30	°C
Pre-Heat Time	0.00	99.99	1.00	min
Rate 1	0.1	2.0	1.0	°/s
Final Temp 1	30	400	150	°C
Final Time 1	0.00	99.99	1.00	min
Rate 2	0.0	2.0	1.0	°/s
Final Temp 2	30	400	250	°C
Final Time 2	0.00	99.99	1.00	min
Rate 3	0.0	2.0	1.0	°/s
Final Temp 3	30	400	350	°C
Final Time 3	0.00	99.99	1.00	min
Inject.Position	35.0	35.0	35.0	mm
Source Position	70.0	78.8	77.0	mm

5.4 Service menu

Select the service menu with a continuous pressing of the “menu selection” key (arrow right):
In case of starting the service menu, you will find the given setting “Auto” and “Clo”:



In addition to the menu mode (“Service”), the following information are given in the display:

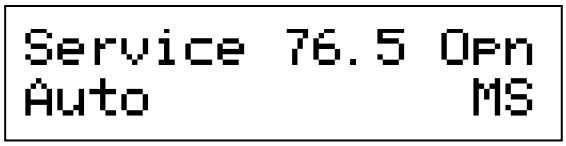
First line: actual position of the push rod in mm (here: -35.0) and
valve position (“Clo” – closed, “Opn” – opened, “---“ – in between)
Second line: mode of the service menu (see table below)

With a click on the “Enter” key, the cursor occurs and you can select one of the following service modes with the keys “arrow up” or “arrow down” and afterwards the “Enter” key:

mode	see at	meaning
Auto	5.4.1	step-by-step moving of the push rod and the valve
Vent	5.4.2	venting the instrument via the push rod
O-ring	5.4.3	positioning of the push rod for O-ring change
Pos.	5.4.4	operating the push rod
Valve	5.4.5	operating the valve

If a service mode is selected and an action is carried out, the controller changes the system status to “Maintenance”. That means, starting the instrument is not allowed until the key “Clear/Stop” is pressed which starts initializing the DIP. The maintenance status is saved even if the instrument is switched off.

5.4.1 Service mode “Auto”



This service mode is used for step-by-step moving of push rod and valve according to the usual measuring cycle. The safety lock is active all the time to prevent damaging of the instrument.
Select this mode if you want to **adjust the position of push rod in the ion source** (after changing of ion source or tip of the push rod):

1. Pass through the motion sequence with the keys “arrow up” and “arrow down” step by step until the push rod is in the correct position in the ion source:
   When the push rod is on the verge of the ion source, move it slowly into the ion source by tapping the arrow keys. If the value of the actual position of the push rod will not change even more (this is the correct position), tap the key once again (shortly and carefully!)
2. Confirm the position with the “Enter” key.
3. Press the “arrow right” key twice and then the “arrow up” key, then select the item “MS” with the “Enter” key (only possible if the push rod is within a permitted zone):
4. The position value is confirmed in the display (“source position value stored”) and saved even if the instrument is switched off.
5. Skip back to the basic display by pressing the “arrow right” key three times (“Maintenance” status).
6. Press the “Clear/Stop” key to leave the “Maintenance” status.

5.4.2 Service mode “Vent”

If the MS has to be vented use this service mode. First, heating elements and high vacuum pump have to be switched off by the MassHunter software. The software will prompt you when it is safe to switch off the power. After switching off, follow the instructions below:

1. Confirm the venting with two subsequent clicks on the “Enter” key (safety requests to prevent accidental venting of the MS: “Press enter to vent instrument” - “Vent Instrument? Press enter to confirm”).
2. The push rod is moved automatically in a position near the O-ring seal of the valve. Then the valve is opened and the push rod is moved slowly out of the valve. This procedure takes about 1.5 minutes (display: venting instrument).
3. At the end the service menu “Vent” returns to the display.
4. Skip back to the basic display by pressing the “arrow right” key three times.
5. Press the “Clear/Stop” key to leave the “Maintenance” status.

5.4.3 Service mode “O-ring”

This service mode has to be selected if the O-ring of the valve shall be replaced:

First, heating elements and high vacuum pump have to be switched off by the MassHunter software. The software will prompt you when it is safe to switch off the power. After switching off, follow the instructions below:

1. Select the service mode “O-ring” with “Enter” and confirm venting with two subsequent clicks on the “Enter” key (“Press enter to replace O-ring” – “Vent instrument? Enter to confirm”).
2. The push rod is moved automatically in the position near the O-ring seal of the valve. Then the valve is opened and the push rod is moved slowly out of the valve. This procedure takes about 1.5 minutes (display: “venting instrument”).
3. Then the push rod is automatically moved into the O-ring change position and the valve is turned into the correct positon for unplugging (see marker position at 7.3). The display skips to the start of the service menu “O-ring”:
4. O-ring change can be carried out (see 7.3).
5. After changing the O-ring, skip back to the basic display by pressing the “arrow right” key three times.
6. Press the “Clear/Stop” key to leave the “Maintenance” status.

5.4.4 Service mode “Pos.”

This service mode is used to move the push rod only. It should be reserved to the service engineers.

Note: This mode enables the deactivation of the safety lock.
If the safety lock is not active (“FREE”), risk of damaging the instrument is high!

5.4.5 Service mode “Valve”

This service mode is used turn the valve only. It should be reserved to the service engineers.

Note: This mode enables the deactivation of the safety lock.
If the safety lock is not active (“FREE”), risk of damaging the instrument is high!

5.5 Menu: Network configuration

If the instrument shall be embedded to a network, use this menu to enter the parameters for the network interface.

Parameter	Example
IP Address	10.1.1.103
Netmask	255.255.255.000
Gateway	192.168.001.001
DHCP Mode	Fixed IP, DHCP

1. Select this menu by pressing the “arrow right” key until the service menu appears and another click on the “arrow right” key.
2. Scroll with the “arrow up” and “arrow down” key in the parameters.
3. Select the parameter with the “Enter” key and change the value with the arrow keys.
4. Skip back to the basic display by pressing the “arrow right” key two times.

In the mode “DHCP” the assigned parameters of the DHCP server and the actual status of the DHCP client are displayed:

In case of a correct assignment of the parameters, “OK” is displayed instead of “Busy”.

Note: All changes of the parameter values take effect directly and are saved if you leave the configuration menu!

5.6 Menu: General Configuration

This menu is used to select the language (German or English).

Moreover, it is possible to change the operational mode which is reserved to the service engineers!

1. Select this menu (“General Config”) by pressing the “arrow right” key until the service menu appears and two subsequent clicks on the “arrow right” key.
2. Press “Enter” and select with the “arrow right” key
   a) the language (cursor is on the left side): with the arrow keys “up” and “down” you can change between “English” and “German”.
   b) the operational mode (cursor is on the right side): with the arrow keys “up” and “down” you can change between “Operate” (normal measuring mode) and “ValMode” (validation mode).
3. Skip back to the basic display by pressing the “arrow right” key.

Note: The validation mode “ValMode” enables the heating of the push rod outside the ion source!
Due to the risks of bodily injury and damaging the instrument, this mode is reserved to qualified service engineers!!!

6 Operating the DIP

6.1 Settings in the MassHunter Software

1. Click on the instrument icon to start up the MassHunter software.

2. Pump down the MS and perform a tune as described in the MassHunter software manual.

3. Enter a new MassHunter method for DIP measurements:
   Follow the instructions of the MassHunter manual and note that there are some different inputs to be made:
   • In the menu item “Inlet/Injection Types” select for “Sample Inlet” **Other**/**None**, for “Injection Source” **External Device**, for “Scan Type” **MS1 Scan** and enter the value for “Total Runtime” which is displayed in the DIP software (see figure in 6.2)

4. After loading this new MassHunter method, click the Start button (green arrow) in the Instrument Control view and specify all sample informations in the Sample Information panel.

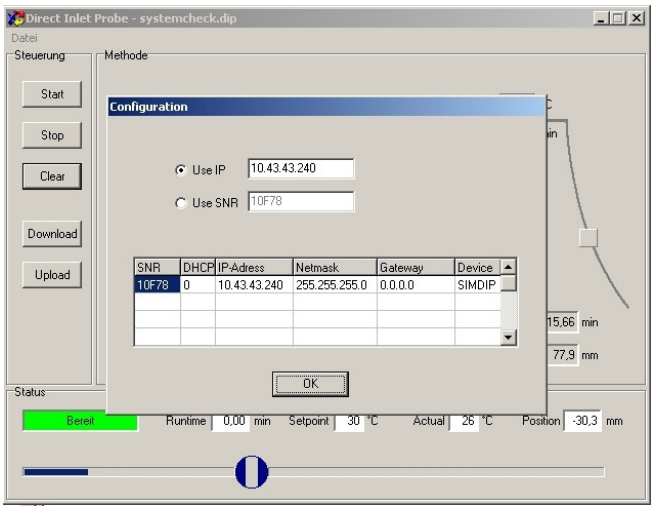
5. Click on “Start Run” and wait for the ready-signal of the MS (“Acquisition: Waiting for remote start …”).
   NOTE: The DIP has to be started using the controller or the “DIP” software (see .6.2).

6. When all settings for the DIP temperature program are done (see 6.2) and the sample is injected (see 6.3) and you can start the DIP at the controller or in the “DIP” software with the “Start” key.

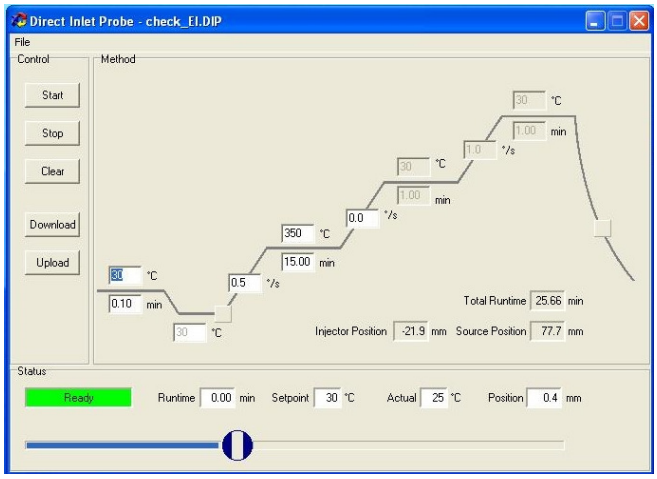
6.2 Settings for the SIM DIP Controller or the SIM “DIP” Software

Enter all values for the temperature program (up to three ramps) according to the main menu “Temperature program” at the control panel (see 5.3) or use the software “DIP”:

NOTE:
Please save/install all “DIP” method files (“**.**dip”) in the same folder that contains the DIP software file “DIP**.**exe”.



1. Open the program “DIP“ (DIP**.**exe).
2. When the program is started, data synchronization of controller’s IP address takes place. Choose “Datei – Configuration / File – Configuration” and select the IP address or serial number (SNR).



3. Now you can

   a) enter the temperature program by inscribing the values in the belonging fields und saving with “Datei – speichern/file - save”.

   b) select a saved temperature program with “Datei – öffnen/file - open”.

   c) assume the temperature program of the controller by clicking the key “Upload”.

NOTE: If you select 3a) or 3b), that means you enter the temperature program in the DIP software at the PC, it is necessary to transfer the data to the DIP controller by clicking the key “Download”!

4. The first temperature value (left position of the temperature profile, preheating temperature) is achieved in the “Load” position and can be used to evaporate the solvent.

5. The second position of the temperature profile showing a temperature value below the preheating temperature is not accessible in the standard version. Together with a CO_2 Cooling Kit (option) this temperature is used to cool the tip of the push rod while the push rod is inserted into the ion source.

6. If the “Ready” signal appears in the status display (green field), click the “Start” button so that the push rod moves into the sampling position (90 °) and the status display indicates “Loadpos”.

7. Now you can inject the sample according to 6.3.

6.3 Sample injection

6.3.1 for liquid samples (sample vessel B 9630 59 75)

1. The push rod is in the “Ready” position (push rod in front of the valve - inside). After choosing the temperature program (see 6.2) click the “Start” button.
2. For easy sampling, the push rod is tilted out of the slot in the covering cap into the 90° sampling position (see fig. on the left).
3. Use a syringe with a cone needle tip (0.47 or 0.64 mm OD) to inject the sample into the sample vessel (see arrow).
4. Again click on the “START” key of the controller or in the “DIP” program to start the measurement.
5. After the run, the push rod stops in the “Ready” position again.

6.3.2 for solid samples (sample tip B 0635 59 73)

If the push rod tip for liquid samples is still mounted, first change it according to this manual (see 7.4) and adjust the push rod again (see chapter 4).

1. The push rod is in the “Ready” position (push rod in front of the valve - inside). After choosing the temperature program (see 6.2) click the “Start” button.
2. For easy sampling, the push rod is tilted out of the slot in the covering cap into the 90° sampling position.
3. Fill the sample into the sample tube (B 0685 59 73).
4. Use the provided tweezers to put the tube into the probe tip with the hole outside (see detail photo).
5. Again click the “START” key of the controller or in the “DIP” program to start the measurement.
6. After the run, the push rod stops in the “Ready” position.
7. If there is no sample tube in the push rod tip during insertion in the ion source, the light barrier gives the fault “ProbeSensorFault”. Please press the buttons “Clear” and then “Start” so that the push rod moves into the 90° sampling position. Now follow the instructions from number 2 on (see above).

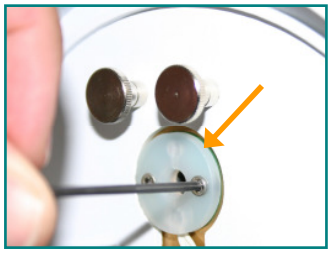
7 Maintenance

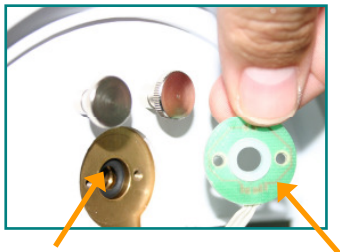
During analyzer maintenance procedures, care must be taken to avoid contaminating the analyzer or interior of the analyzer chamber. Clean gloves should be worn during all analyzer maintenance procedures.

7.1 To replace the O-ring seal of the vacuum lock chamber

Spare part: **B 0651 59 73** (O-ring seal for vacuum lock chamber)

1. Select the service mode “Auto” (see 5.4.1.1) and move the push rod out of the valve as far as possible (with the “arrow down” key) so that opening of the covering cap and replacing of the O-ring is possible.
2. Afterwards put off the controller and open the DIP covering cap.
3. Put a small sheet of paper beneath the push rod so that nothing can fall into the slot beneath the push rod. Loose the screws of the plastic disc and remove it carefully without damaging the cable of the light barrier (see photo on the bottom of this page, function of the light barrier see 6.3.2).
4. Behind the plastic disc you can see the O-ring seal of the direct inlet. This seal has to be removed with a small wooden rod to prevent scratches on the metallic surface.
5. Inspect the O-ring. If it does not appear as good as new, replace it.
6. Carefully refit the plastic disc (together with light barrier and cable, see photo on the bottom) with the screws.
7. After changing the O-ring, switch on the controller and skip back to the basic display by pressing the “arrow right” key three times (until “maintenance” appears).
8. Leave the “maintenance” status by pressing the key “Clear/Stop”.





O-Ring B 0651 59 73

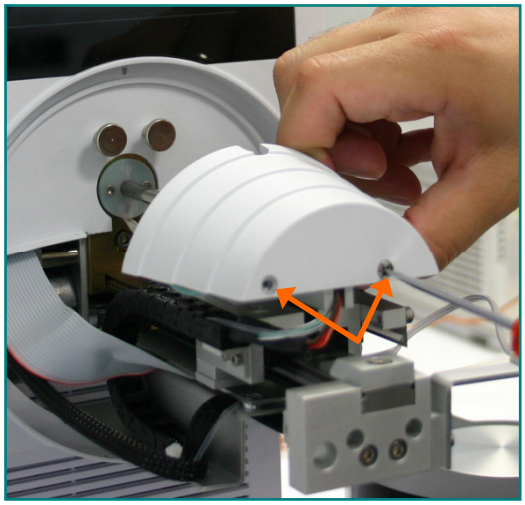
light barrier and cable on the back of the plastic disc

7.2 To replace the O-ring seal of the “Plug Shut Off Valve” of the vacuum lock

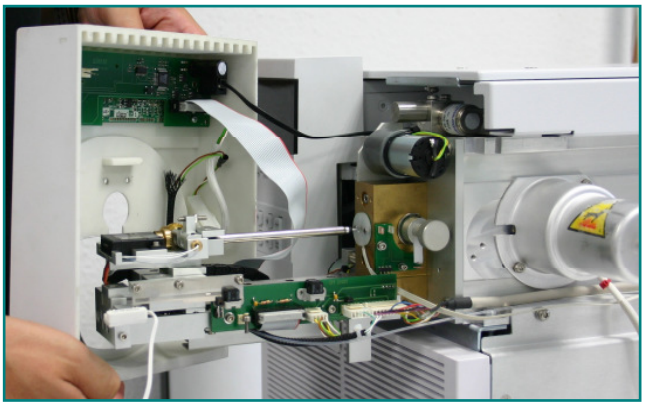
Spare parts: **B 0652 59 73** O-ring seal for valve vacuum lock
B 0653 59 73 O-ring seal for valve guide

Note: Before you attend the orders below, vent the MS (according to this manual text passage 2 and 5.4.2)!! and select the service menu “O-ring” at the controller (see 5.4.3) and perform the steps 1-3. Then follow the instructions given below.

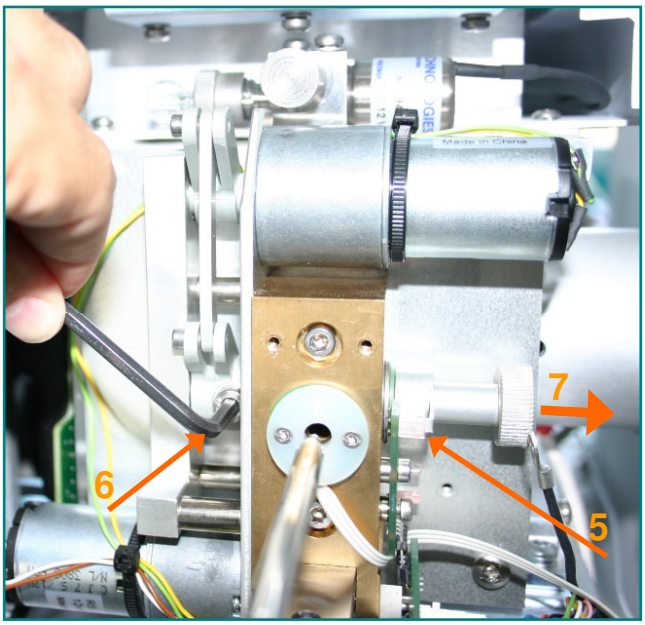
1. Switch off the controller when the push rod is in the position for O-ring change (see “Note” above).
2. Loose the screw on the bottom side of the push rod cover (see arrow).
3. Pull off the housing completely and put it aside.



4. Loose the two screws of the motor cover and remove it.



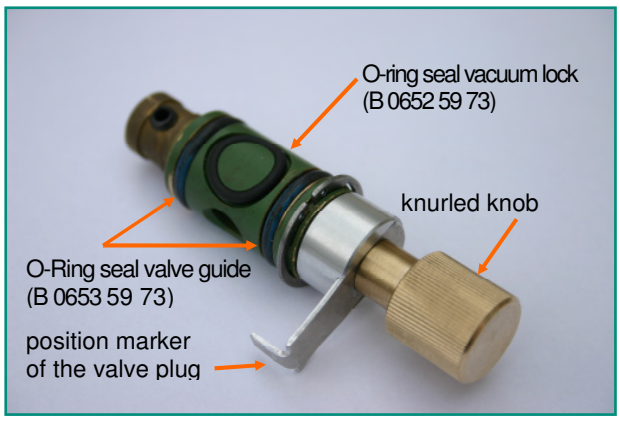
4. Carefully remove the front panel of the DIP by slightly rotating and tilting it, then disconnect all cables of the front panel and put the cover aside.



5. Make sure that the marker of the valve plug (see photo below and arrow no. 5 on the photo left) is directed forwards to the observer (normal position during the service menu “O-Ring”)!!

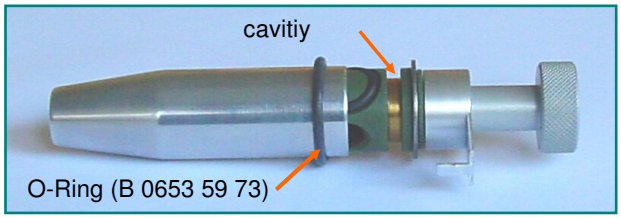
6. Loose the screw of the shaft to collar connection with the provided wrench (see photo, arrow no. 6) by turning the wrench ¼ till ½ to the right.

7. Remove the valve plug by pulling it rightwards at the knurled knob (without twisting the plug!, arrow no. 7)



8. Carefully remove the O-ring seal of the valve plug (B 0652 59 73) to prevent scratches.

9. Inspect the O-ring. If it does not appear as good as new, replace it:
Use a clean, lint-free cloth or glove to spread a thin coat of high vacuum grease on the surface of the O-ring (if the O-ring looks shiny, wipe away excess grease).



10. To replace the O-rings for the valve guide (B 0653 59 73) use the added auxiliary tool (see fig.) to slide the O-ring into the particular cavity.

11. Push the valve plug back into the valve in the correct position (see 7.).
12. Fix the right screw (see step no. 6.) by slightly swaying the plug at the knurled screw to reach the correct position of shaft to collar connection.
13. Reverse the steps 1-4 to mount the push rod cover onto the MS.
14. Switch on the controller and press “Clear/Stop” at the control panel to start initializing of the DIP.

7.3 To clean and to replace the probe tip

Spare parts: **B 9630 59 75** (liquid sample vessel, complete with guide ring);
B 0635 59 73 (solid sample tip, complete with guide ring)

1. Bring the push rod into the “Load” position by pressing the “Start” button of the controller.
2. If the sample vessel has to be cleaned or replaced, twist off the probe tip (see bracket 1) of the push rod.
3. Try to clean the sample vessel with a suitable solvent in an ultrasonic bath.
4. Screw the purified or a new probe tip into the push rod so that the sample vessel looks upwards.
5. Press “Clear/Stop” twice so that the push rod moves into the “Ready” position (inside the DIP in front of the valve).

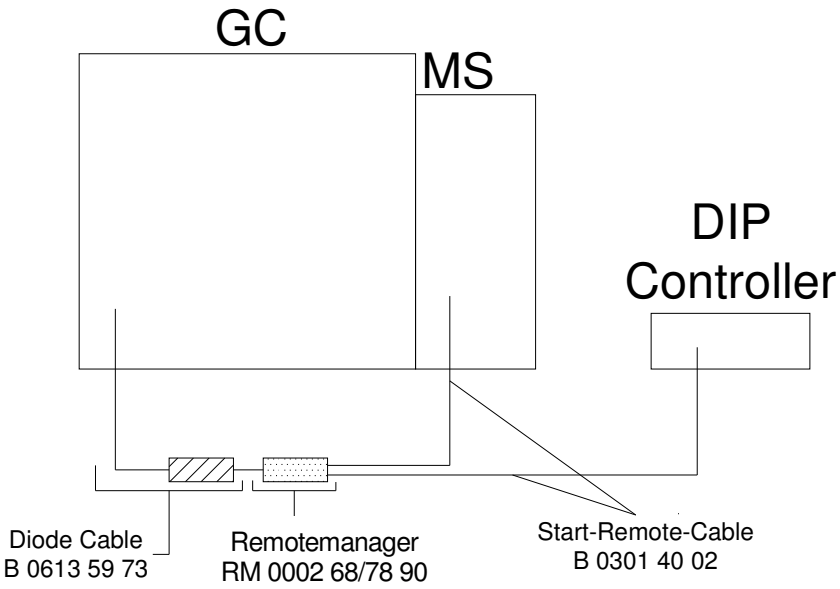
7.4 To replace the thermocouple

The thermocouple in the push rod has to be replaced at the SIM GmbH. If a failure occurs relating to the heating system of the push rod (error message: “TC fault”), please contact your SIM service engineer.

8 Spare Parts

Article	order no.
Seal assembly for vacuum lock chamber	B 0650 59 73
O-ring seal for vacuum lock chamber	B 0651 59 73
O-ring seal for valve vacuum lock	B 0652 59 73
O-ring seal for valve guide	B 0653 59 73
Sample vessel for liquid samples, complete with guide ring	B 9630 59 75
Sample tip for solid samples, complete with guide ring	B 0635 59 73
Tubes for solid samples (100 pieces)	B 0685 59 73
Push rod with thermocouple	B 0640 59 75
Lock chamber core, complete	B 0670 70 00

9 Connecting diagram



10 Technical data

10.1 Controller

power: 230/110 VAC, 50-60 Hz ,0.2 A

10.2 Direct Inlet Probe

power consumption: max. 2.4 W (max. 12 V, max. 0.2 A)