Scientific Instruments Manufacturer GmbH
D-46149 Oberhausen • ☎ +49-208-94 10 78-0 • www.sim-gmbh.de

Agilent Technologies
Channel Partner

SERVICE MANUAL

SIM Direct Inlet Probe

for

Agilent 5975 MSD

and

Agilent 7000 Triple Quadrupole

(Firmware V.1.10)

Document History
Edition 7075e 10/2010, from Firmware / Software Version V1.10 and dip.v.1.2.

The information contained in this document is subject to change without notice.

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

SIM GmbH shall not be liable for errors contained herein or for incidental or consequential damages in connection with the furnishing, performance or use of this material.

CONTENT

1 Parameter Menu of the Controller (Firmware V1.10)

Parameter	Assignment	min value	max value	default
0	1000 (Password)	10	0x7fff	1000
1	Temp. adjustment (0.1°C)	-200	200	-23
2	LCD contrast	0	50	20
3	push rod position outside (min pos)	-400	-100	-136
4	push rod position ion source (max pos)	300	550	467
5	cooling position	100	500	300
6	max temperature	350	500	400
7	xp*0.1	10	1000	150
8	tn*0.1	1	100	40
9	inj-position	-400	-100	-135
10	ion source position	300	500	444
11	temp correction (*0.001)	-200	200	-115
12	damp down heating oscillations td*0.1	0	30	10
13	O-ring change position	-400	0	-115
14	Option bits: 1=useExStart, 2=ready position inside the DIP, 4=90°position, 8=continuous operation, 16=smoothing of temperature, 32=early start, 64= CO_2 cooling	-0x8000	0x7fff	23
15	push rod horizontal position	-0x8000	0x7fff	75
16	light barrier change position	-	-	160
17	tilt angle adjustment	-	-	12
18	dwell time (in valve after opening to the ion source; 1min =100, default = 6 s)	0		-001
19	position of light barrier for tube test (-97 = activated, -1 = deactivated)			-97

Additional remarks to **parameter 14**:

You can combine the items by adding the numbers, e. g. “23” (1+2+4+16) for DIP without autosampler or “31” (1+2+4+8+16) for DIP with autosampler, “95” ((1+2+4+8+16+64) for DIP with autosampler and CO_2 cooling

This menu is reserved to qualified service engineers only and needs a password.
Select the parameter menu of the controller in the following way:

1. Press <Enter> and keep it pressed
   while pressing <arrow up><arrow down><arrow up><arrow down>
   then let off the <Enter> key so that the diagnostic menu appears

   press <arrow right> and <Enter> so that the cursor in the parameter menu appears:
   the first two numbers represent the parameter number, the selected number is taken with <Enter>
   then the belonging value (to this parameter) will be shown and can be changed
2. Select parameter “00” and enter the password “1000”
3. Change the values of the designated parameter.

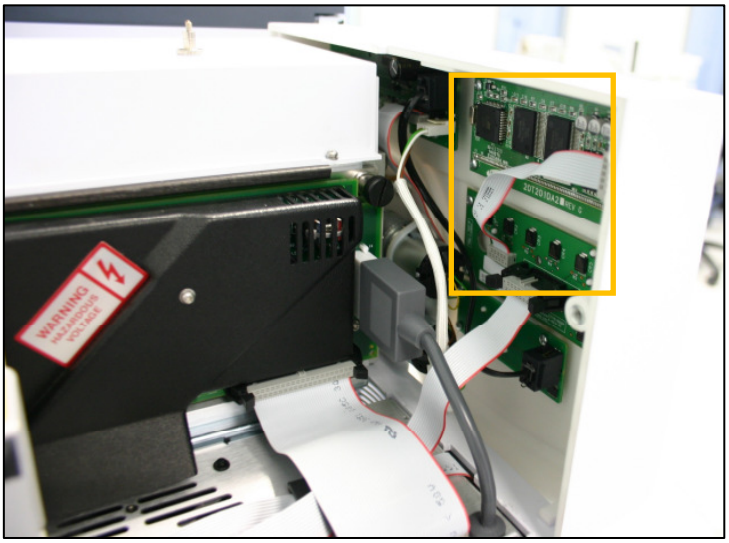
2 DIP Installation Procedure

2.1 Replacing the ion source

Please follow the instructions given in the corresponding Agilent Manual to replace the existing ion source with the provided modified ion source (EI or CI).

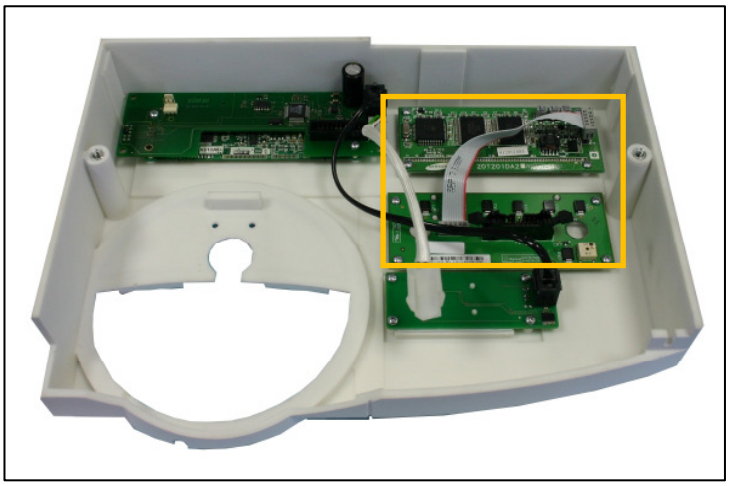
2.2 Installing the DIP on the Agilent 5975 MSD

Before installing the DIP System to the Agilent 5975 MSD you have to uninstall the MSD front panel:



1. After you have disconnected the required cables, remove the MSD front panel carefully. Please follow the instructions given in the 5975 MSD manual.

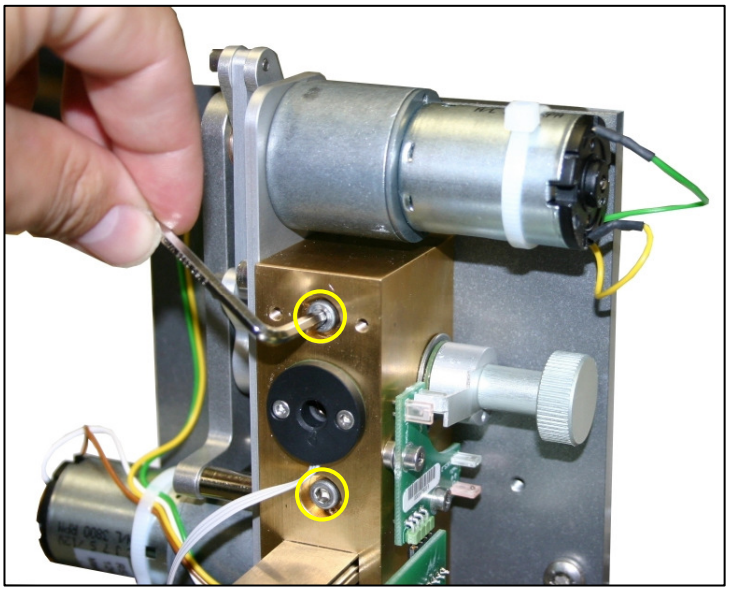
2. Demount the two boards for the MSD display (see marking on the left) and put the MSD front panel aside and



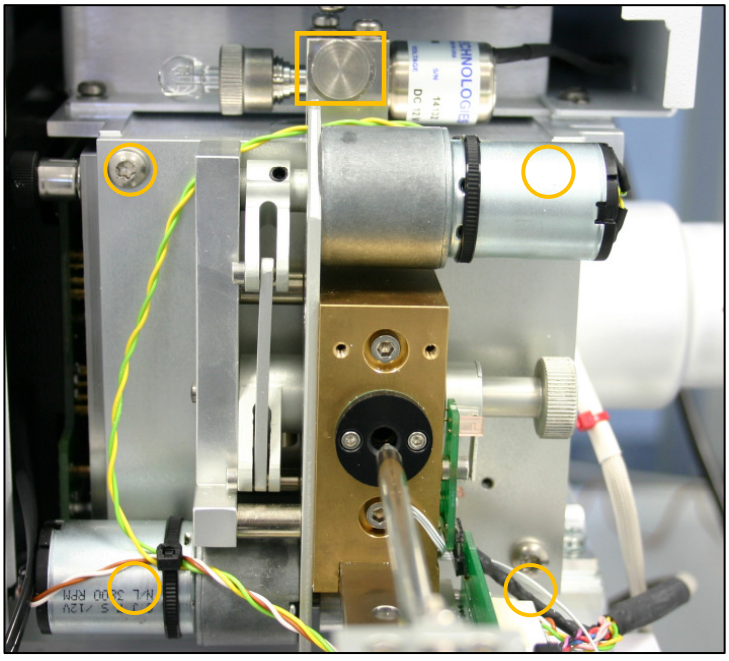
3. Remove the DIP front panel of the DIP System following the instructions given in the DIP manual (chapter 7.2).

4. Take the DIP front panel and mount the display boards of the MSD onto the preset positions (see left).

5. Before mounting the DIP system, remove the glass plate in front of the MSD ion source compartment.



6. To mount the DIP system in front of the MSD, first deinstall the valve unit by loosing the two screws (see marked circles on the left). Now the adapter plate is separated and the four screws are well accessible for the next step.



7. Take the adapter plate and mount it in front of the ion source compartment using the four screws (see marked circles on the left, the screws behind the motors are not visible on this photo).

8. Then retighten the valve unit of the DIP system on the adapter plate with the two screws according to the photo of step no. 6.

9. Replace the original knurled knob of the MSD valve with the provided knob for the DIP System (see marked rectangle in the photo left).

10. Mount the push rod cover by reversing the instructions in chapter 7.2 of the DIP manual. Make sure that all cables are correctly connected.

2.3 Installing the DIP on the Agilent 7000 Triple Q

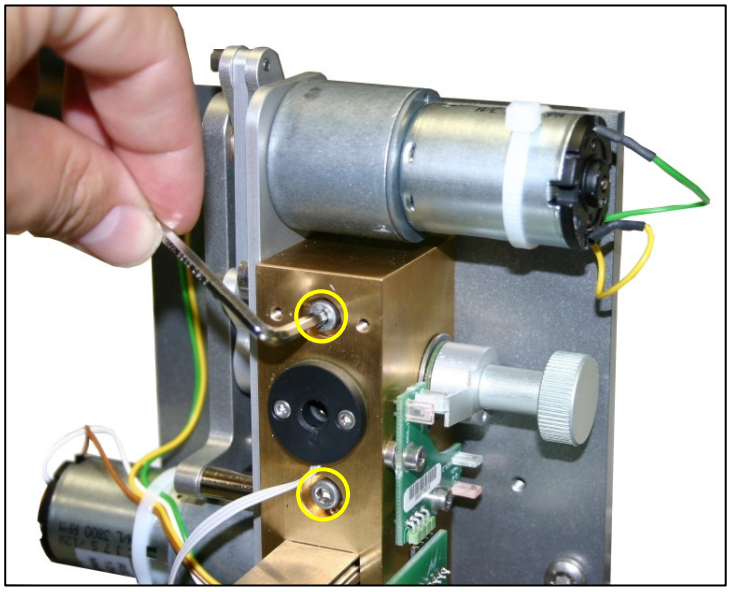
Before installing the DIP System to the Agilent 7000 Triple Q MS/MS you have to uninstall the MSD front panel. After you have disconnected the required cables, remove the MS front panel carefully (please follow the instructions given in the Agilent manual.)

1. Demount the two boards of the MS/MS front panel and then put this two-part front panel aside.

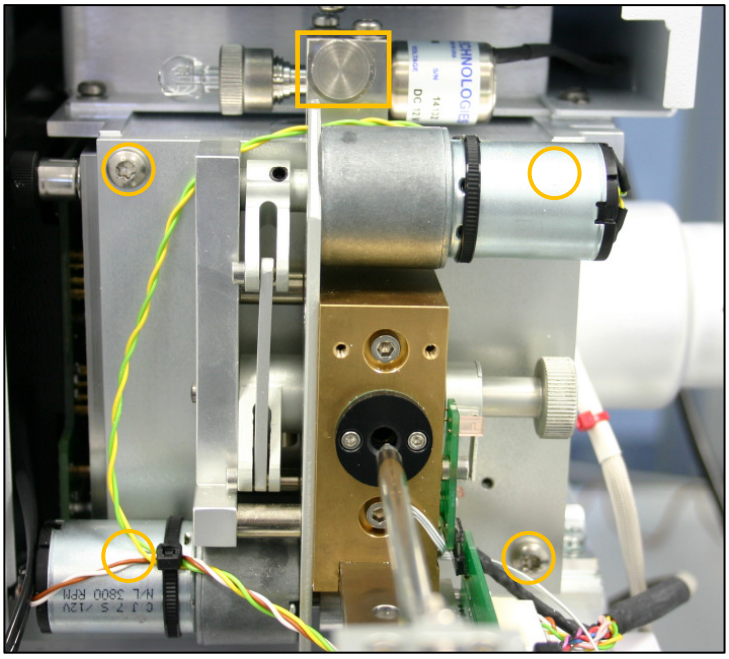
2. Remove the front panel of the DIP System following the instructions given in the DIP manual (chapter 7.2).

3. Mount the two boards onto the preset positions of the DIP front panel (see marking on the right photo)

4. Before mounting the DIP system, remove the glass plate in front of the MS/MS ion source compartment.



5. To mount the DIP system in front of the MS/MS, first deinstall the valve unit by loosing the two screws (see marked circles on the left). Now the adapter plate is separated and the four screws are well accessible for the next step.



6. Take the adapter plate and mount it in front of the ion source compartment using the four screws (see marked circles on the left, the two screws behind the motors are not visible on this photo).

7. Then retighten the valve unit of the DIP system on the adapter plate with the two screws according to the photo of step no. 5.

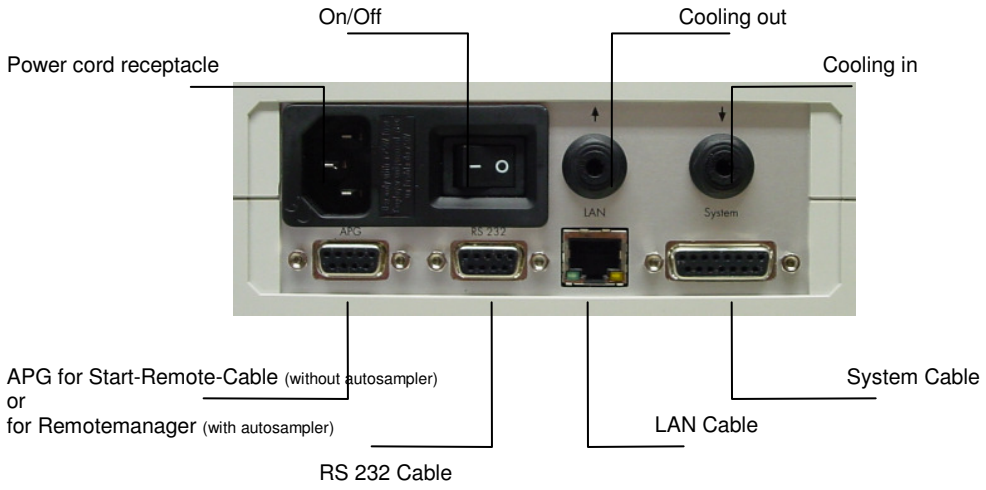
8. Replace the original knurled knob of the MSD valve with the provided knob for the DIP System (see marked rectangle in the photo left).

9. Mount the push rod cover by reversing the instructions in chapter 7.2 of the DIP manual. Make sure that all cables are correctly connected.

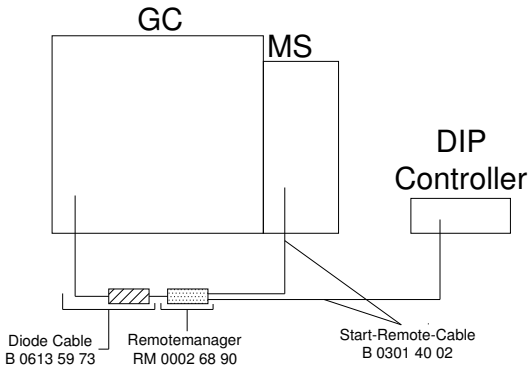
2.4 Connecting the DIP and the GC

Please follow the instructions of the Agilent Manual but pay attention to the difference in installing the **DIP controller** as you can see in the figures below:

DIP controller rear panel view:



connecting diagram GC-MS-DIP Controller



2.5 Configuration of the DIP-Controller

The IP adress is assigned in the menu “Network configuration “ of the DIP controller (see MSD DIP Manual, 5.5 Network configuration):

1. Turn on the DIP controller.
2. Select the menu “Network configuration” and scroll with the “arrow up” and “arrow down” key in the parameters.
3. Set the parameter “DHCP Mode” to “Fixed IP”.
4. Select the parameter “IP adress” with the “Enter” key and change the numbers with the arrow keys.
5. Skip back to the basic display by pressing the “arrow right” key two times.

Or the IP-adress is assigned automatically if the modus “DHCP” is selected (see MSD DIP Manual, 5.5):

1. Turn on the DIP controller.
2. Select the menu “Network configuration” and scroll with the “arrow up” and “arrow down” key in the parameters.
3. Set the parameter “DHCP Mode” to “DHCP” (see).
4. Skip back to the basic display by pressing the “arrow right” key two times.

2.6 Installing the DIP Software

Note: You can find all files with software and default methods on the disc enclosed.

1. Copy the files “DIP.exe, dip.enu, dip.deu” into the new folder “DIP”.
2. Copy the default methods in the folder “DIP\Methods”
3. Open the “DIP”-Software.
4. Click on “File” and open the menu “Config.”.
5. Select the DIP controller with a click on the IP adress.

2.7 Insert the ChemStation/MassHunter methods for the DIP

Note: You can find all the folders with the default methods on the disc enclosed.

2.7.1 5975 MSD: ChemStation methods

1. Copy the folders “DIP_EI_Q1” and “DIP_CI_Q1” into the methods section of the MSD ChemStation under “Programme\MSDChem\1\Methods”.
2. optional (with autosampler):
   Copy the folders “Injection_GC” and “Injection_DIP” into the methods section of the MSD ChemStation.

2.7.2 7000 Triple Q MS/MS: MassHunter methods

1. Copy the folders “DIP_EI_Scan.M” (and possibly “DIP_CI_Scan.M”) into the methods section of the MassHunter software under “MassHunter\GCMS\1\Methods”.

2. optional (with autosampler):
   Copy the macros (cycles) “DIP-Liquid-Inj.cyx” and “DIP-Solid-Inj.cyx” into the PAL section under “MassHunter\GCMS\1\PALDATA”.

2.8 Adjustment of the CombiPAL to the MS-DIP System (optional)

1. Define two new DIP positions for a precise co-operation of PAL and DIP (see DIP-PAL Manual 6.4.3).
2. Create a macro and a new method in the Cycle Composer Software of the autosampler (see DIP Manual 6.4.4 and 6.4.5) or copy the files of the folder “Cycle Composer” into
   ”C:\Programs\PAL\Cycle Composer\Combi PAL”.

2.9 Push rod performance test

1. Select the method “drive.dip” in the DIP-Software and click“OK”.
2. Check the correct insertion of the push rod into the MSD ion source and the operational sequence backwards in real and even at the status display of the software.
3. Select the method “heat.dip” in the DIP-Software and click “OK”. The push rod is inserted into the ion source and heated up to 400 °C. This temperature is maintained for 1 minute.
4. Check the correct temperature run via the status display.

2.10 Tuning of the DIP-MS System

2.10.1 Autotune 5975 MSD with DIP

1. Verify that the system has been pumping down for at least 2-4 hours and that the DIP controller is turned on. The DIP push rod status is “Ready”.
2. In the “Instrument Control view”, select “Perform MS Autotune” from the instrument menu.
   The software will perform an autotune and print out the report.

3. When the autotune has completed, save it as “ATUNE.U”, and then select Tune Evaluation from the Qualify menu. The software will evaluate the last autotune and print a System Verification – Tune report.
4. Verify that there is no air and water in the system.
   (If there are peaks at 18, 28, and 32 amu, there may be an air leak in the system.)

2.10.2 Autotune 7000 TripleQ MS/MS with DIP

1. Verify that the system has been pumping down for at least 2-4 hours and that the DIP controller is turned on. The DIP push rod status is “Ready”.
2. In the “Instrument Control view”, select “MS Tune” from the instrument menu and select the items “print autotune report” and “save tune file when done”. The software will perform an autotune, save it as “atunes.ei.tune.xml” (default file name) and print out the report.
3. Go to “Manual Tune – Vacuum Control” and select “Air And Water-Check”. Print the screen to document the result.
4. Verify that there is no air and water in the system.
   (If there are peaks at 18, 28, and 32 amu, there may be an air leak in the system.)

2.11 Controller: Update of the firmware

1. Switch on the controller.
2. Open the program “CHIPTOOL”
   (download link : http://www.beck-ipc.com/bc/download/index.asp?sp=en)
   that will find all installed chips with their ID number.
3. Select the chip of the SIM DIP controller and click at the right mouse button: Now you can select the FTP server and enter the following items and finish this procedure with the “Connect” button:
   User: ftp; Password: ftp, Representation Type: Binary (Image)
4. Now you can see the contents of the chip at one side of the screen (Drive A), and the contents of one of the other drives at the other side of the screen. Select the drive with the update-files of the new firmware version.
5. Copy the update files “_t.exe” (for SIM controller) and/or “MSDDIP.hex” (for display controller) into the A directory of the chip and close the FTP server.

If you have updated the MSDDIP.hex file, you have to contiunue with the following procedure:

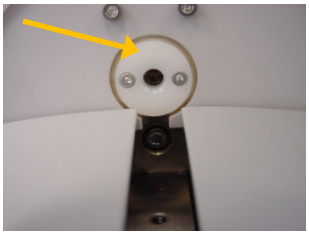
6. Select the SIM DIP controller and click at the right mouse button.
7. Now you can choose “Telnet” as connection type .Enter the following items and finish the procedure with the “Connect” button:
   User: tel; Password: tel; Automatically login
8. In this “TELNET session”, break “_t.exe” with the shortcut “Ctrl Z”.
9. Type “dir” so that the directory of A is displayed.
10. Type “d” and “Enter” into the next command line (A:\>d) so that program “D.exe” is started (D.exe runs the download of MSDDIP.hex from the DIP controller into the display controller)
11. When this download is done, the update is finished. Reboot the controller for correct working.

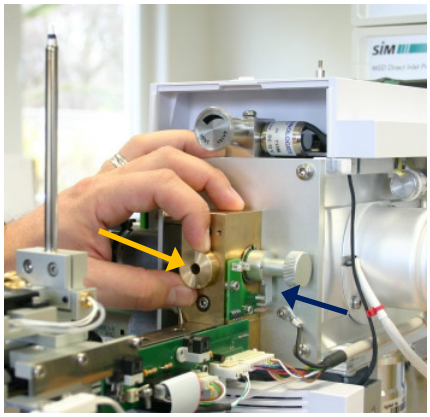
3 Exact Positioning of the Push Rod in the Ion Source

This chapter is valid for all DIP systems for the Agilent 7000 Triple Q and for those for the 5975 MSD starting from serial number SN1938 (December 2009) because these systems are not equipped with an inspection glass any longer:
In case of maintenance work at the push rod or for installing purposes it can be necessary to reposition the push rod in the ion source. Then use the following procedure:

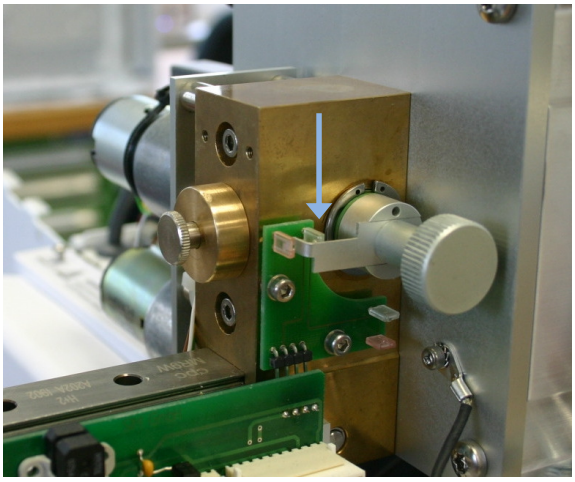
Note: Vent the MS before starting the following procedure (i.e. turbopump is off and ion source temperature < 100 °C)

1. Turn on the foreline pump to get the prevacuum.
2. Remove the housing of the push rod and the front panel (see DIP user manual, 7.2.) but do not disconnect the two cables to the front panel because you have to move the push rod with the display keys later on!
3. Fix the transportation safety screw completely (shown in the figure next to step no.11, see arrow).
4. Move the push rod out of the parking position in the valve by pressing the arrow key “up” (front panel) once. Manually bring the push rod into the vertical (90°) position.





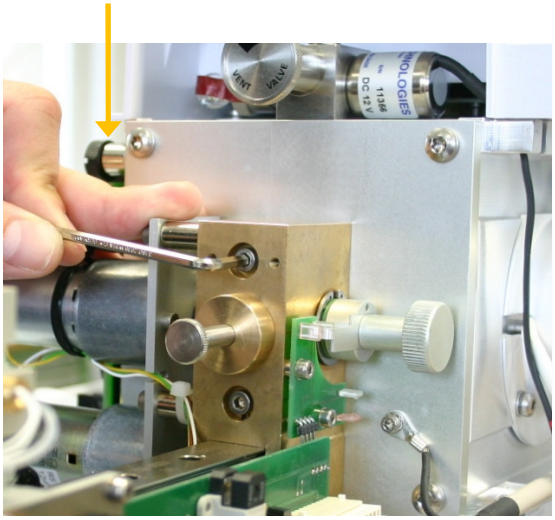
5. Remove the Teflon disc (left figure) and replace it by a brass one (right figure). The marker of the valve plug should be directed down to the bottom (see blue arrow in the right figure)..
6. Carefully take the front panel (still connected to the DIP system) and select the service mode “Valve” (see DIP user manual, 5.4.).
7. Without the push rod (still in the vertical position), select “valve free” with the subsequent clicks on the arrow keys “right” and “up” and “enter” for confirmation.



and you can skip the steps 11 - 12.

.

transportation safety screw



8. Press the arrow keys to open the valve (control it by watching the marker of the valve plug which is directed forwards to the observer when the valve is open, see blue arrow in figure on the left).

9. Take the dummy push rod for the alignment procedure. While the original push rod is already in the vertical position, insert the dummy into the valve as far as possible. If this is consistent with the position in the figure left, positioning of the push rod in the ion source is correct

10. Otherwise please loose the screws of the brass block slightly. Now you can cautiously move the valve block until you are able to insert the push rod completely.

11. Fix the screws to adjust the correct position of the push rod.

12. Remove the dummy and exchange the brass disc with the Teflon disc.

13. Click the push rod down while the valve is still open (marker forwards).

14. Mount the push rod cover and the front panel onto the MSD again and press the arrow “right” three times so that the valve is closed (!) and initializing of the DIP is started.

15. Check the exact position of the push rod by using the service mode “Auto”.

16. Loose the transportation safety screw (see figure above). Now you can pump down the MS and then start running the DIP.

4 Temperature Calibration of the Controller

4.1 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!!!

4.2 Temperature Calibration

1. Select "ValMode" at the DIP Controller (see above and DIP Manual 5.6)
2. Put the temperature sensor of a calibrated thermometer (indicates the actual value) into the push rod tip and isolate these both.
3. Select the parameter menu of the controller:
   Press <Enter> and keep it pressed
   while pressing <arrow up><arrow down><arrow up><arrow down>
   then let off the <Enter> key so that the diagnostic menu appears

   press <Arrow right> and <Enter> so that the cursor in the parameter menu appears:
   the first two numbers represent the parameter number, the selected number is taken with <Enter>
   then the belonging value (to this parameter) will be shown and can be changed
4. Select parameter "00" and enter the password "1000" (now all parameter values may be changed)
5. Select parameter "01" and calibrate the controller to room temperature:
   example: actual value (thermometer): 20 °C controller-value: 22 °C
   change the value to "-20" and press <Enter>
   (algebraic sign "-" because the controller value is higher than the actual value, otherwise do not use a sign)
6. Load the DIP method "check_temp.dip." and start it:
   The tip of the push rod is successively heated to 30/150/250/350°C. Please note each temperature of the controller display at the end of the dwell time.
7. Calculate the deviation percentage of each temperature and average over all temperatures (for example 8%).

8. Select parameter “11” in the parameter menu and enter the calculated value in the following manner:
   ”-80” and <Enter> (for the above mentioned example, if the value of the controller is higher than the actual value).
   ”80” and<Enter> (for the above mentioned example, if the value of the controller is lower than the actual value)

9. Press <Arrow right> to return to the diagnostic menu and leave it with <Enter>.
10. Change the validation mode (ValMode) to the normal measuring mode (Operate) in the General Configuration Menu (see above and Manual 5.6).

5 DIP Test Procedures

DIP Temperature Accuracy Test	Evaluates the DIP tip temperature using a probe with a calibrated thermometer.
Air/Water Check during DIP operation	Evaluates the DIP for leaks.
Coupling Test: CombiPal with MSD-DIP	Evaluates the function of the DIP (45°version) and the GC together with the CombiPal
DIP EI Performance Test	Evaluates the performance of the MSD (EI) DIP using a traceable standard.
PCICH4.U Test (if applicable)	Evaluates the performance of the MSD (PCI) using a traceable calibration standard.
DIP PCI Performance Test (if applicable)	Evaluates the performance of the MSD (PCI) DIP using a traceable standard.

5.1 DIP Temperature accuracy test

This procedure uses a traceable digital thermometer to determine the temperature accuracy of the push rod tip at the DIP system.

CONDITIONS
- MS – “Ready”
- DIP – “Ready/Bereit”

PROCEDURE
1. With the push rod outside, select the “Validation Mode” of the DIP controller: Go to the menu “General Configuration” (see DIP manual, 5.6 Menu General Configuration) and change the operate modus to the validation modus “ValMode” with the arrow keys “up” or “down”.
2. Put the temperature sensor of the calibrated digital thermometer into the tip of the push rod.
3. Isolate the tip of the push rod together with the thermometer and shut the cap of the DIP.
4. In the DIP software, select the method “check_temp.dip” and start the temperature program.
5. When the first setpoint is reached, observe the calibrated thermometer’s display until the reading stabilizes.
6. Record the thermometer’s reading at the end of the 5 minute’s dwell time in the table of the Results section.
7. Repeat no. 5 and 6 for the second and third setpoint.
8. In the DIP controller’s “General Configuration” menu change the mode from “ValMode” to “Operate”.

RESULTS

DIP Temperature Accuracy Test				
Setpoint [°C]	Measured Value [°C]	Typical Range of Operation	Pass/Fail	Verified By/Date
30		30 ± 5		
150		150 ± 5		
250		250 ± 7		
350		350 ± 7		

5.2 Leak test

This procedure consists of the Tune evalutation results (MS ChemStation) or the Air/Water Check (MassHunter software) described in section 2.10 (DIP in “Ready” status with the push rod outside the MS ion source and the results of an Air/Water check while the push rod is located in the ion source. The leak test of DIP/MS-System is passed, when the results of both measurements do not essentially differ.

REQUIREMENTS
- an autotune and tune evalutation test (for 5975 MSD) or “Air And Water Check” (for 7000 Triple Q) have just been performed.

CONDITIONS
- MS – “Ready”
- DIP – “Ready/Bereit”

PROCEDURE

1. Start the DIP Software and load the method “*a_w_check.dip*”.
2. Start the DIP (start button in the software or controller): The push rod will be inserted into the ion source, heated up to the source temperature (230 °C) and be kept there for 10 minutes.
3. When the setpoint is reached and the reading stabilizes, perform a tune evaluation or an “air/water check” (5975 MSD: go to “Instrument / Edit MS Tune Parmeters” and select “Air/water check”; 7000 Triple Q MS/MS: go to “Manual Tune / Vacuum Control” and select “Air and Water check”.
4. Compare this result with the tune evaluation report (5975 MSD)/air and water check (7000 Triple Q MS/MS)when the push rod was in the “Ready” position outside the MS ion source (see this manual, section 2.10) and complete the table in the Results section.

RESULTS

LEAK TEST					
Item	Air/Water Check [%], push rod outside ion source	Air/Water Check [%], push rod inside ion source	Limit for deviation [percentage points]	Pass/Fail	Verified By/Date
Abundance Ratio 18/69			< 5		
Abundance Ratio 28/69			< 2		

5.3 Coupling test: CombiPAL with MSD-DIP and GC (optional, for DIP - 45° type with 5975 MSD)

This procedure checks the function of the CombiPAL autosampler by alternating injections into the DIP system and the GC.

REQUIREMENTS
- Two additional DIP positions have been defined in the CycleComposer software according to the DIP manual.
- Cycle Composer: Verify that the methods “GC-Injection” and “DIP-Injection”, the macros “Injection DIP” and “Injection GC” and the sequence “Injection_GC-DIP” (see “Cycle Composer” on the disc enclosed) are copied into “Programs\PAL\Cycle Composer\ Combi PAL.
- MSD ChemStation: Verify that the folders “Injection_GC” and “Injection_DIP” (see “MSChemstation” on the disc enclosed) are copied into the methods section of the MSD ChemStation under “Programme\MSDChem\1\Methods“. Moreover, copy the sequence “Injection GC-DIP” into the sequence section of the MSD ChemStation.

CONDITIONS
- MSD – “Ready”
- MSD-DIP – “Ready/Bereit”
- CombiPAL – Power on

PROCEDURE
1. Position an empty vial on position 1 at tray 1 of the CombiPAL.
2. Open the MSD ChemStation and load the sequence “ Injection GC-DIP (possibly check it with “edit sequence”).
3. Open the “Cyle Composer with Macro Editor” and load the sample list “Injection_GC-DIP”.
4. Open the DIP software and download the method “drive.dip”.
5. Switch to the MSD ChemStation window and start the sequence (“run sequence”).
6. Switch to the Cycle Composer window and start the sample list (“start”).
7. Watch the alternating injections into the DIP and the GC (3 times each) and complete the table below.

The sequence has to run completely with the needle in the correct positions.

5.4 DIP (EI) Performance test

This test uses a standard solution with two components to determine the correct performance of the DIP. Perform this procedure on both types of probe tips (for liquid and for solid samples).

REQUIREMENTS

- An autotune has been performed.
- A new method for DIP EI measurements has been created in the MS ChemStation software:
  Follow the instructions of the MSD ChemStation 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 (DIP method “*check_EI.dip*”, see DIP manual).
- Optional: CombiPAL is installed correctly on the GC/MSD-DIP system (see DIP manual)

CONDITIONS for DIP/5975 MSD

DIP	Ready / Bereit
DIP method	check_EI.dip (30°C for 0.1 min, 30-350°C at 0.5°/s, 350°C for 5 min)
MSD	Ready
MSD method	DIP_EI_Q1.M
MSD aux temp	280°C
Tune file	ATUNE.U
Aquisition mode	SCAN
Run length	15.50 min
Column flow	0.5 ml/min
Solvent delay	0.10 min
EMV	Relative ± 0
Mass range (low)	100.0
Mass range (high)	600.0
Threshold: Inst	150
Sampling:	2
MS quad temp:	150°C
MS ion source temp :	230°C
Optional : CombiPAL PAL method	DIP-injection.PME

Sample	triglyceride standard
Push rod tip	A: for liquid samples, B: for solid samples
Sample volume	1 µl

CONDITIONS for DIP/7000 Triple Q MS/MS

DIP	Ready / Bereit
DIP method	check_EI_scan.dip (40°C for 1 min, 40-350°C at 1.0°/s, 350°C for 5 min)
MS/MS	Ready
MS method	DIP_EI_Scan.M
MS aux temp	280°C
Tune file	atunes.ei.tune.xml
Ion Sorce	EI
Aquisition mode	SCAN
Scan Type	MS1 Scan
Stop Mode	By Stop Time
Stop time	10 min
Column flow	0.5 ml/min
Solvent delay	0.3 min
Time filter enabled	yes
Time filter peak width	0.7 s
Use gain	no
EMV	Use tune setting (Delta EMV=0)
Mass range (low)	100.0
Mass range (high)	600.0
Scan time	100 ms
Threshold	0
Step size	0.1 amu
MS quad temp (Q1 + Q2)	150°C
MS ion source temp :	230°C

Sample	triglyceride standard
Push rod tip	A: for liquid samples, B: for solid samples
Sample volume	1 µl

PROCEDURE
Set up the Methods

1. Load method DIP_EI_Q1.M for the 5975 MSD (MS ChemStation) or DIP_EI_Scan.M for the 7000 Triple Q MS/MS.
2. Select the actual autotune using Instrument/Select MS Tune File (ATUNE.U or atunes.ei.tune.xml).
3. Save the method to "…\1\METHODS\" as "DIP_EI_Q1.M" resp. "DIP_EI_Scan.M"
4. Print the method and verify that method and conditions are the same.
5. Load method "*check_EI.dip*" for the DIP-system (via DIP software, see DIP manual).

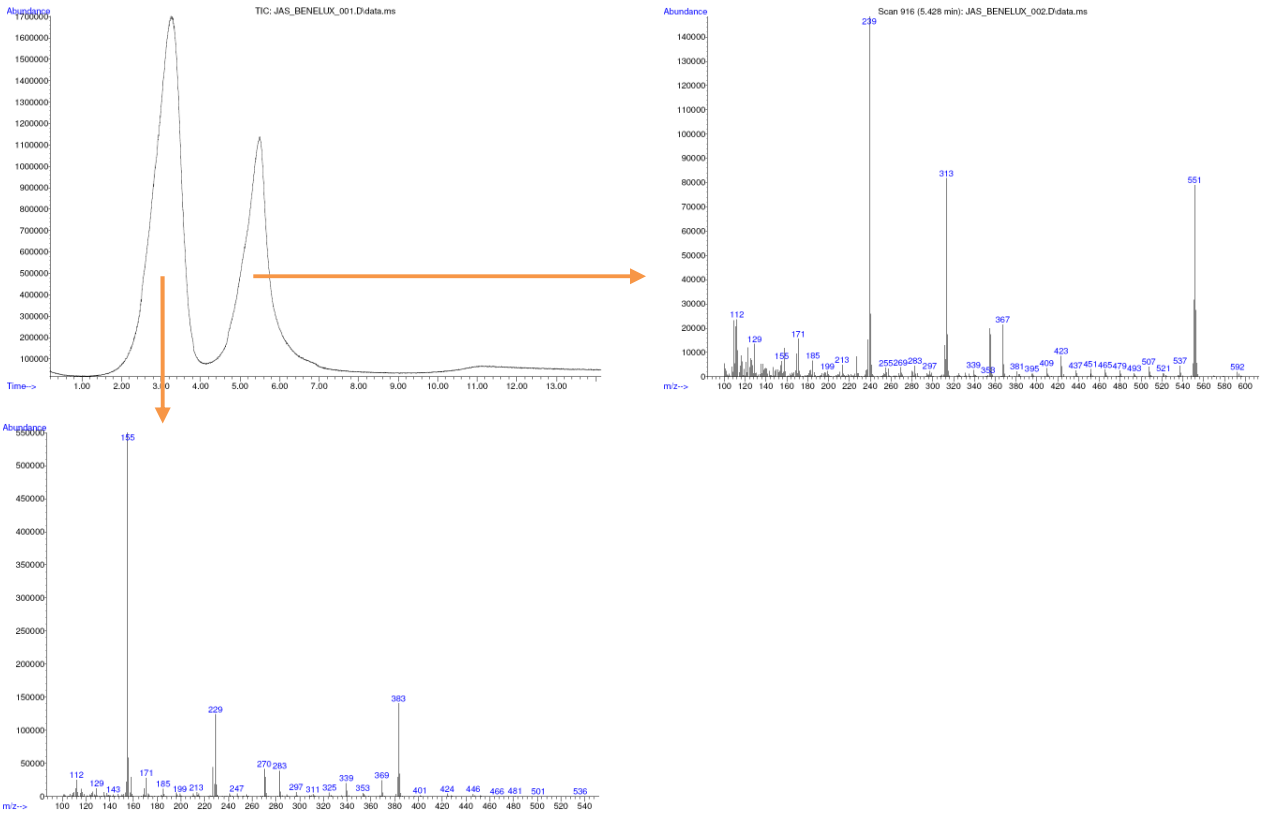
The following steps have to be performed with the push rod tip for liquid samples (A) and the tip for solid samples (B):

1. After loading the DIP method, click the Start button (green arrow) in the Instrument Control view (of the MS software) and specify all sample information in the Sample Information panel.
2. Click on “Start Run” and wait for the Ready-signal of the MS (“Acquisition: Waiting for remote start …”).
3. If the Ready-signal appears in the status display (green field), you can inject the sample according to the DIP manual (see 6.3) by pressing the “START” key to move the push rod into the “Load” position.
4. Start the DIP using the controller or the “DIP”-software (see DIP manual, 6.2): Click on the “START” key of the controller or in the “DIP” programm to start the measurement.

RESULTS

EI PERFORMANCE						
Item	**t_{max} (TIC)** [min]	**Typical value** [min]	**Main signals** [m/z]	**Typical values** [m/z]	**Abundance (TIC, t_{max})** [counts]	**Typical value** [counts]
Tricaprin		1.5-4.0		155/229/383		> 1 000 000
Tripalmitin		4.5-7		239/313/551		> 600 000

Example for DIP/5975MSD System:



5.5 DIP (PCI) Performance test for DIP/5975 MSD Systems

This test uses a traceable standard solution to determine the correct performance of the DIP. Perform this procedure on both types of probe tips (for liquid and for solid samples).

REQUIREMENTS

- An autotune has been performed.
- A new method for DIP CI measurements has been created in the MS ChemStation software:
  Follow the instructions of the MSD ChemStation 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**, and for “Injection Source” **External Device** and enter the value for **“Total Runtime”** (take the value displayed in the DIP software minus 1.5)(see DIP manual)

CONDITIONS

DIP	Ready/Bereit
DIP method	check_CI.dip (30°C for 0.1 min, 30-350°C at 1.0°/s, 350°C for 5 min)
MSD	Ready
MSD method	DIP_CI_Q1.M
MSD aux temp	280°C
Tune file	PCICH4.U
Aquisition mode	SCAN
Run length	10.00 min
column flow	0.5 ml/min
Solvent delay	0.02 min
EMV	PCICH4.U + 400 V
Mass range (low)	50.0
Mass range (high)	550.0
Threshold: Inst	150
Sampling	2
MS quad temp	150°C
MS ion source temp.	250°C

Sample	DBP
Push rod tip	A: liquid samples, B: solid samples
Sample volume	1 µl

PROCEDURE

Set up the Methods

1. Load method DIP_CI_Q1.M for the 5975 MSD (MS ChemStation).
2. Select PCICH4.U using Instrument/Select MS Tune File.
3. Save the method to "…\1\METHODS\DIP_CI_Q1.M"
4. Print the method and verify that method and conditions are the same
5. Load method "check_CI.dip" for the DIP-system (via DIP software, see DIP manual).

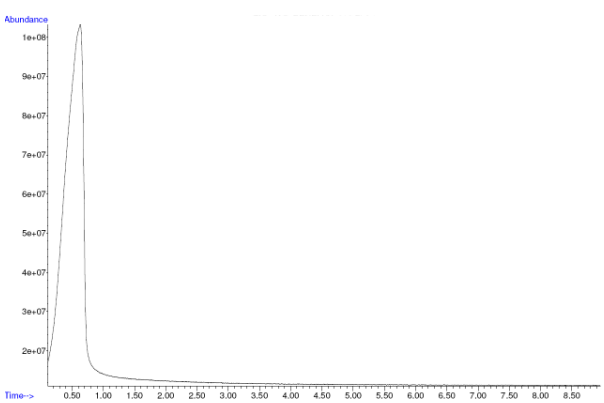
The following steps have to be performed with the push rod tip for liquid samples (A) and the tip for solid samples (B):

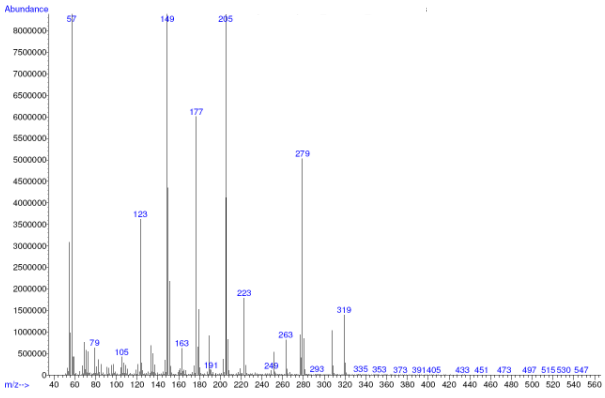
6. After loading the DIP method, click the Start button (green arrow) in the Instrument Control view (of the MS Software) and specify all sample information in the Sample Information panel.
7. Click on "Start Run" and wait for the Ready-signal of the MSD ("Acquisition: Waiting for remote start …").
8. If the Ready-signal appears in the status display (green field), you can inject the sample according to the DIP manual (see 6.3) by pressing the "START" key to move the push rod into the "Load" position.
9. Start the DIP using the controller or the "DIP"-software (see DIP manual, 6.2): Click on the "START" key of the controller or in the "DIP" program to start the measurement.

RESULTS

CI PERFORMANCE						
Item	**t_{max} (TIC)** [min]	**Typical value** [min]	**Main signals** [m/z]	**Typical values** [m/z]	**Abundance (TIC, t_{max})** [counts]	**Typical value** [counts]
DBP		0.3-1.4		149/205		> 1 000 000

Example for DIP/5975MSD System:





APPENDIX

DIP-Methods

drive.dip	30°C for 0.1 min, 30-35°C at 0.5°/s, 35°C for 0.05 min
heat.dip	30°C for 0.1 min, 30-400°C at 2.0°/s, 400°C for 1 min
a_w_check.dip	30°C for 0.1 min, 30-230°C at 1.0°/s, 230°C for 15 min
check_temp.dip	30°C for 2 min, 30-150°C at 1.0°/s, 150°C for 2 min, 150-250°C at 1.0°/s, 250°C for 2 min, 250-350°C at 1.0°/s, 350°C for 2 min
check_EI.dip	30°C for 0.1 min, 30-350°C at 0.5°/s, 350°C for 5 min
check_CI.dip	30°C for 0.1 min, 30-350°C at 1.0°/s, 350°C for 5 min