Agilent G1978A Multimode Source for G1946/G1956 LC/MSD

Set-Up Guide

Agilent Technologies

Notices

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CAUTION

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In This Guide

This guide explains how to install, maintain and troubleshoot your nanoelectospray ion source.

1 Installation

This chapter tells you how to install the multimode ion source.

2 Verification

This chapter tells you how to install the multimode ion source.

3 Methods

This chapter describes basic operation and maintenance for the multimode ion source.

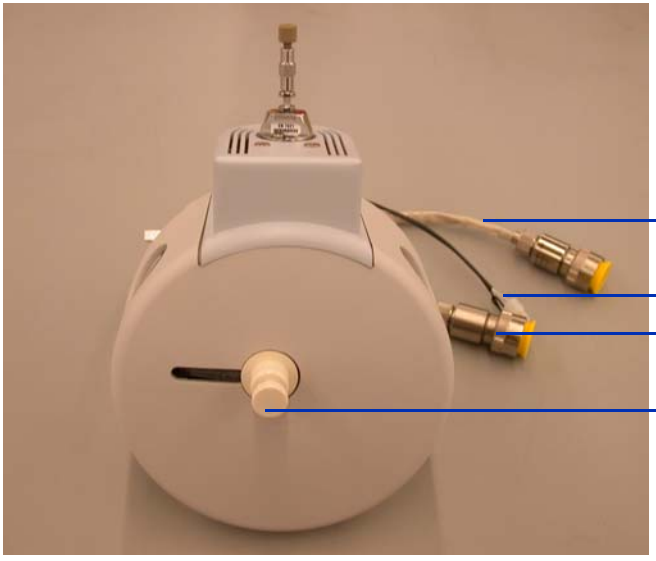
Content

Contents

1 Installation

This chapter contains instructions to install the G1978A multimode source on the G1946 and G1956 LC/MSD instruments. (The G1978B source is not supported on the G1946/G1956 instruments.)

The instructions of this manual are supported with LC/MSD ChemStation Release B.01.01 or B.01.03, with G1978-10002 update patch or higher. The patch installation CD contains both a B.01.01 and B.01.03 folder, each containing a **setup.exe** file. Run the appropriate **setup.exe** file, depending on your ChemStation release number.


APCI High voltage
HV cable +/- 2kv
Vaporizer Cable
APCI Corona receptacle

Installation

This section contains instructions to install the G1978A multimode source onto the G1946 or G1956 LC/MSD instrument.

Step 1. Prepare to install

Before you install the multimode source, check that you have the appropriate parts and tools.

1 Check that you have these parts:

- Bundled LC/MSD Multimode ESI/APCI Source (p/n G1978A)
- LC/MSD Multimode ESI/APCI Source (p/n G1978-65239)
- Multimode HV Module Assembly (p/n G1978-60050)
- LCMSD MM ESI/APCI Enablement Kit (p/n G1978-60150)
- Firmware Upgrade Kit, MM (p/n G1978-60156)
- ChemStation B.01.01 or B.01.03 or greater
- Patch (p/n G1978-10002). The software patch is included in G1978A and is required for both B.01.01 and B.01.03.

NOTE All G1946B/C/D and G1956A/B MSD instruments that were installed before the multimode source was released will need to be on ChemStation B.01.01 or greater to install the multimode source hardware, firmware, new PID values, and software patch.

2 Check that you have these tools, supplies and chemicals. The items in this list are not provided with your multimode source.

- Cloths and gloves, clean, lint-free
- Water and organics, such as acetone, methanol, acetonitrile or isopropyl alcohol, all HPLC grade
- ¼ inch open-end wrench
- Torx drive T10

Step 2. Check the instrument board revisions

The software allows you to check to see if the instrument boards have been updated.

Analyzer 3 board

Do the following steps to determine if you have an Analyzer 3 board installed.

1 In the Method and Run Control view on the command line, type
   - `pat$=nvrAnRev$()`

2 On the command line type
   - **`Print pat$`**

The part number for the analyzer board will print on the message line. If the part number is ***G1946-60250***, the board that is installed is an Analyzer 3 board. If a different part number is printed, you will need to upgrade to the Analyzer 3 board.

Power distribution board (PDB)

The Power distribution board on all G1956A/B & G1946B/C/D are already the correct board (p/n G1946-60002).

Step 3. Turn off the instrument

- Refer to the instructions for your instrument to properly turn off the instrument.

NOTE Shut down the instrument completely if not a bundled installation. This includes G1946B/C/D and pre-multimode G1956A/B instruments. These instruments are considered upgrades for use with the G1978A source.

Step 4. Change chips on electronic boards (CE Only)

The Main PLCC Firmware chip U129 must be changed on the Analyzer 3 PCA for source identification (new, p/n G1978-80067). Two chips on the power distribution board U6 (new, p/n G1978-80100) Programmed ROM MM LON and U18 Chip EPROM (new, p/n G1978-80200) will also be changed. These steps only need to be done the first time that the multimode source is installed.

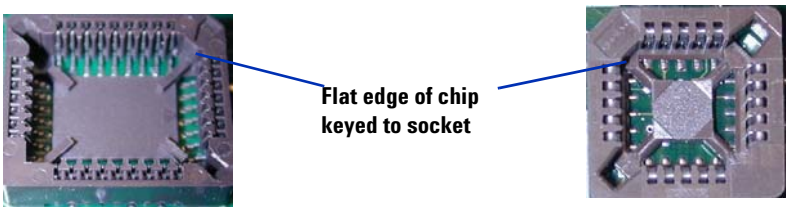


Figure 1 Socket with keyed spot for chips U6 and U18 on the power distribution board (left) and socket with keyed spot for chip U129 on the Analyzer 3 board (right). Note that the keyed spot is in the upper left corner for the Analyzer board, and on the opposite side (upper right) for the power distribution board.

CAUTION Make sure that the flat edge of the chip is aligned with the flat edge of the keyed spot on the socket. Improper insertion can damage the chip when the power is turned on. The keyed spot on the socket for the power distribution board is different than on the analyzer board.

You do not need to completely remove the power distribution board from instrument during chip replacement. However, doing so prevents the chip or tool from being dropped into the instrument.

CAUTION The following steps are to be done only by an Agilent trained CE. Damage to the chip can happen at power on if these steps are not properly done.

1 Verify the instrument is powered off. Refer to the instrument user guide.

2 Remove the Analyzer 3 PCA board from the tub assembly.

Figure 2 Analyzer 3 PCA board (p/n G1946-65250)

3 Use the chip removal tool provided with the upgrade to remove the chip.

Figure 3 Chip removal tool and Analyzer 3 PCA board (p/n G1946-65250), keyed in the upper left corner.

CAUTION Be careful when you remove the chip with the chip removal tool. The tool can be inserted too far and can crack the socket if forced.

4 Replace the PLD chip on the Analyzer 3 PCA board with the one provided with the multimode source.

5 With the analyzer board still out, install the 10 MΩ ACPI high voltage cable. See Figure 4.

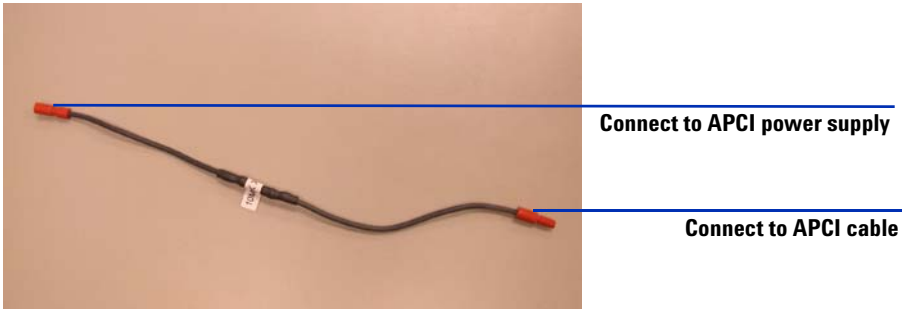


Figure 4 10 MΩ ACPI high voltage cable (p/n G1978-60806). In the next steps, make sure that the appropriate ends are connected to the APCI power supply and APCI cable.

6 Disconnect the APCI high voltage cable from the APCI power supply. See Figure 5.

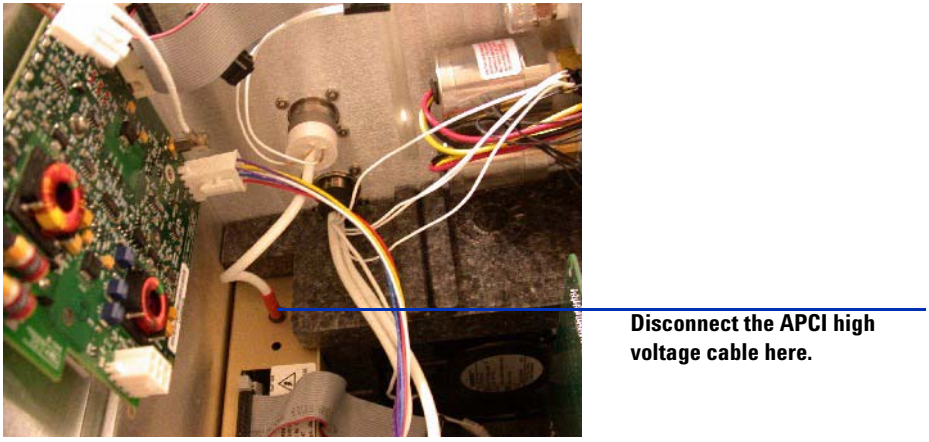


Figure 5 Disconnecting the APCI high voltage cable.

7 Install the 10 MΩ cable in series with the APCI high voltage cable and reconnect to APCI power supply. See Figure 6.

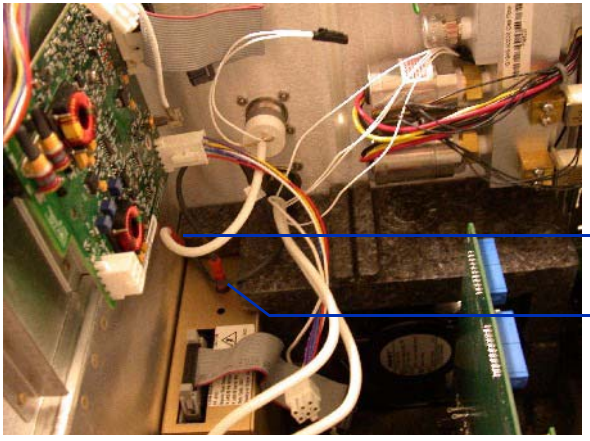


Figure 6 10 MΩ high voltage cable connected to the APIC high voltage cable and APCI power supply.

8 Remove the power distribution board from instrument during chip replacement to prevent dropping the chip or tool into the instrument.

NOTE The two chips can be replaced on the power distribution board without removing the board from the instrument, but be careful to not drop the tool or chip into the instrument.

9 Use the chip removal tool to remove both the U6 and U18 chips from the power distribution board. See Figure 7 through Figure 9.

CAUTION Make sure that the flat edge of the chip is aligned with the flat edge of the keyed spot on the socket. Improper insertion can damage the chip when the power is turned on.

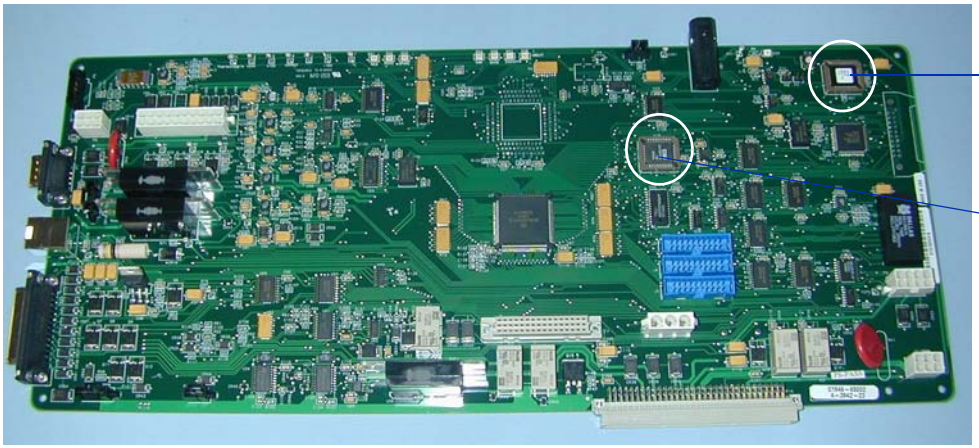


Figure 7 Two chips are to be replaced on the power distribution board (p/n G1946-65002).

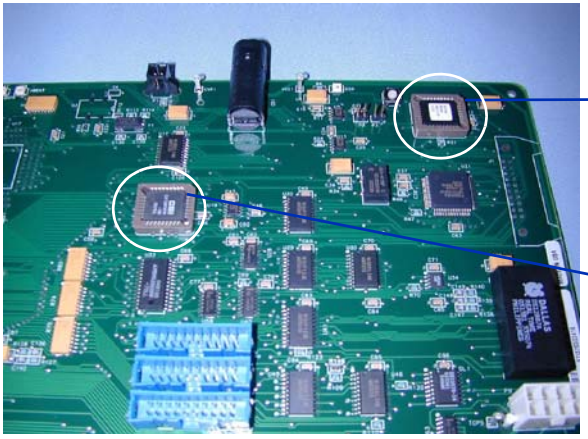


Figure 8 Two chips to be replaced on the power distribution board (p/n G1946-65002), keyed at the upper right corner of the socket.

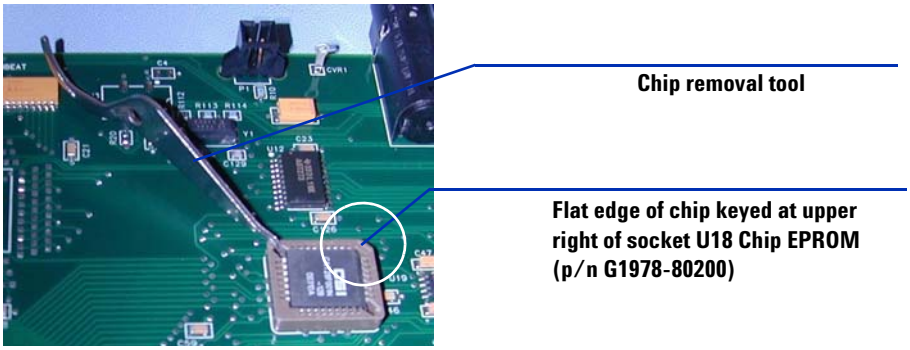


Figure 9 Flat edge of chip keyed at top right of socket

10 Replace the two chips on the power distribution board.

You can verify that the chips were replaced on both boards by following the steps in "Step 7. Check instrument boards" on page 19.

Step 5. Convert from ESI, APCI or APPI to multimode source

- Do the steps in "To convert from ESI, APCI or APPI to the multimode source" on page 23.

Step 6. Upgrade the software with the G1978-10002 patch

If you are running ChemStation B.01.01 or B.01.03, you need to install this patch. If you are running ChemStation B.03.1 or later, skip this step.

1. Place the G1978-10002 update disk into the CD drive.
2. Open the folder **B.01.01** or **B.01.03** (depending on your ChemStation release) and click **setup.exe**.

   The files replaced by this patch are backed up so the software patch can be uninstalled.

3 Click **Next** on the Welcome screen.

4 Click **Yes** to accept the software license agreement.

5 Click **Next** on the Readme screen.

6 Click **Next** on the Start Copying Files dialog box that allows you to review your setup choices.

NOTE You cannot select the installation directory since you are installing a patch to the current chemstation software.

7 Click **Yes** to upgrade the MS firmware.

WARNING **Do not interrupt this upgrade process. Do not start the MS ChemStation software or interrupt power to the instrument during this process. You will damage your instrument if you interrupt this process.**

8 Enter the instrument IP address and type **`yes`**. See Figure 10.

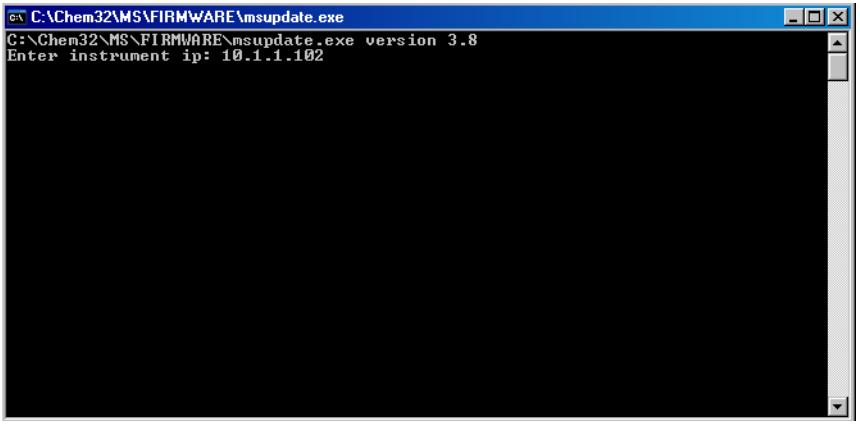


Figure 10 MSUpdate program

9 When prompted if it is OK to proceed with the hardware update, type **`Y`**. See Figure 11.

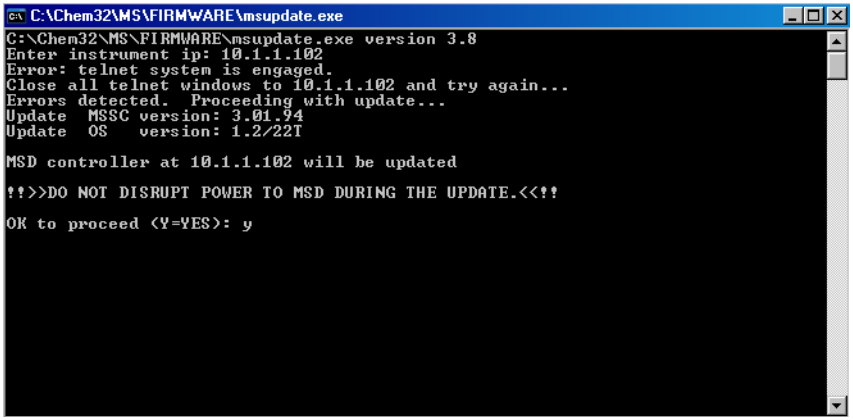
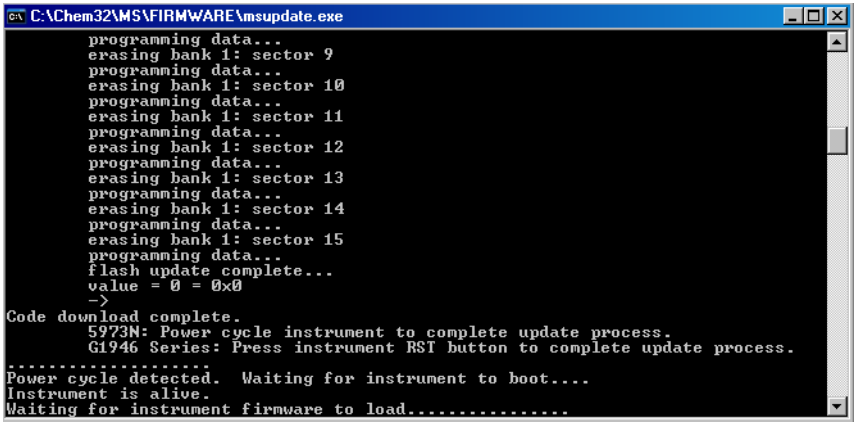


Figure 11 Update MSD firmware

10 When prompted, press instrument RST to complete the update process.

11 Two more message will be displayed in the command window.



WARNING **Do not interrupt this upgrade process. Do not start the MS ChemStation software or interrupt power to the instrument during this process. When the MSUpdate window goes away, you may launch the ChemStation program.**

After the command window disappears, the instrument has been updated.

Step 7. Check instrument boards

To validate that the new PID values were changed.

1 Launch the ChemStation. The **pid2.mac** changes the PID values automatically when the ChemStation starts up.

2 In the **Method and Run Control** view on the command line enter:

- `MSZONEPID 7`

The following information will be shown on the message line:

P = 2500

I = 1

D = 0

I_D = 1

To validate the changes to the chips on the Analyzer 3 and the Power distribution boards

Do the following steps to validate that the chips on the Analyzer 3 board (p/n G1946-65250) and the power distribution board (p/n G1946-65002) were updated to support the multimode source. If either the ChemStation version B.01.01 or B01.03 with G1978-10002 multimode update software disk has been loaded, the macro **mstnnvr.mac** will be updated and returns more information.

1 At the command line in the **Method and Run Control** view, type

```
mmcheck = MMBoardUpdate()
```

2 Type the command below at the command line, then press **Enter**.

```
Print mmcheck
```

If the variable ***mmcheck*** has the value 0, both boards have been updated.

If the variable ***mmcheck*** has the value 1, one board has not been updated.

If the variable ***mmcheck*** has the value 2, neither board has been updated.

3 To validate all firmware chip revisions:

- Type `readnvr` and press **Enter**.
- Type `shownvr` or `printnvr` and press **Enter**.

The following MSDNVRAM.TXT report indicates the correct firmware loaded. Check the entries that are bolded below to verify correct revision numbers:

```
G1946 LC/MSD Instrument Configuration
    9:46:07 AM    7/14/2005
    ------------------------------------------------------------------------------
    Instrument Name                 : Instrument 1
    Serial Number                   : MS1202
    Product Number                  : product1                          Exp in G1946
    Mfg Date                        : 04/01/96                          Exp <> 04/01/96
    Quad Serial Number              : quad56                            Exp <> quad56
    MS Inject Valve Present         : 1
    ------------------------------------------------------------------------------
    ChemStation Rev                 : Rev. B.01.01 [164] or B.01.03 [203]
    SmartCard Rev                   : 3.02.01
    Analyzer Board FW Rev           : G1946-60250MM
    PDB HW Rev                      : PPHA.01.00
    PDB FW Rev                      : PRS2.03.00                        Exp = PRS2.02.00
    PDB 68332 FW Rev                : 1.63                              Exp = 1.58
    SICB-LON HW Rev                 : PRH1.00.01
    SICB-LON FW Rev                 : PRS1.01.01
    IO Board FW Rev                 : 6.2; 6.3
    Turbo Pump Ctrl HW Rev          : TURB1.0.00
    Turbo Pump Ctrl FW Rev          : PRSW1.1.02
    Convect. Gauge HW Rev           : 011411-102
    Convect. Gauge FW Rev           : PP11520109
    Ion Gauge HW Rev                : 0115-27103
    Ion Gauge FW Rev                : PR11616115
    Log Amp ID                      : LOG01,CAL
    ------------------------------------------------------------------------------
    Quad Frequency                  : 1001200.0010
    Pos Ion Quad Polarity           : 0
    Neg Ion Quad Polarity           : 1
    ------------------------------------------------------------------------------
    Stdby Quad Temp                 : 100
    Stdby Drying Gas Temp           : 300
    Stdby Drying Gas Flow           : 3.000
    Stdby Nebulizer Press           : 20.0
    Stdby Vaporizer Temp            : 325
    ------------------------------------------------------------------------------
    Quad Temp PIDs                  : P=3000;I=0;D=0;ID=1
    Drying Gas Temp PIDs            : P=165;I=2;D=1024;ID=1
    Vaporizer Temp PIDs             : P=2500;I=1;D=0;ID=1       Exp = P=512;I=2;D=0;ID=1
    Drying Gas Flow PIDs            : P=10;I=1;D=10;ID=1        Exp = P=10;I=1;D=1;ID=1
    Nebulizer Pres PIDs             : P=10;I=1;D=10;ID=1
    ------------------------------------------------------------------------------
    Quad Temp Timeout               : 88.8
```

```
Drying Gas Temp Timeout           : 12.3
Vaporizer Temp Timeout            : 1.6                          Exp = 4.4
Drying Gas Flow Timeout           : 13.7
Nebulizer Pres Timeout            : 13.7
-------------------------------------------------------------------------------
CDS Leak Sensor Calibration       : 0                            Exp <> 0
CDS On Purge Time                 : 30
CDS Off Purge Time 1              : 75
CDS Off Purge Time 2              : 60
CDS On Delay                      : 30
-------------------------------------------------------------------------------
Mass Axis Lag D Coeff 0           : -0.0274494
Mass Axis Lag D Coeff 1           : 0.000127939
Mass Axis Lag D Coeff 2           : 2.65427e-09
-------------------------------------------------------------------------------
Std EMV                           :-------------------------------------------------
EMV Gain Coeff 0                  : 8.202345
EMV Gain Coeff 1                  : -59.097311
EMV Gain Coeff 2                  : 0
-------------------------------------------------------------------------------
Default Analog Out                : 0
Default Fraction Collection Relay : 0
Default Aux Relay                 : 0
-------------------------------------------------------------------------------
Polarity Switching Delay          : 200
Signal Switching Delay            : 0
-------------------------------------------------------------------------------
EMF limit: Calibrant A hrs        : 0
EMF limit: Calibrant B hrs        : 0
EMF limit: Pump Oil hrs           : 0
EMF limit: Gas Conditioner hrs    : 0
EMF limit: Ion Optics hrs         : 0
EMF limit: SSV Cycles             : 0
EMF limit: EM Current             : 0
-------------------------------------------------------------------------------
Last Backup Date                  : 5/5/2005 3:38:05 PM
Last Restore Date                 :
NVR Macro Revision                : 1.14
```

Step 8. Verify performance of the multimode source

Before using your system, verify the performance of your system.

1 Start the ChemStation software.

2 Do the steps in "To do an autotune" on page 56.

3 Bake out the instrument. Refer to the multimode *Maintenance Guide*.

4 Do the steps in "To prepare performance evaluation samples" on page 45.

NOTE These verification methods are to be used for sensitivity verification for bundled instruments shipped with a multimode source only.

5 Do the steps in "To verify multimode source operations" on page 51.

Changing Sources

This section includes tasks that you will need to do change the source on your instrument.

To convert from ESI, APCI or APPI to the multimode source

CAUTION If you are installing this source on this instrument for the first time, follow the steps in "Installation" on page 7.

1. Switch to the **MSD Tune** view.
2. Select **Instrument/Set Spray Chamber** and set all gas flows and temperatures to 0.
   - Drying Gas (L/min)
   - Nebulizer Pressure (psig)
   - Drying Gas Temperature (°C)
   - Vaporizer Temperature (APCI source only)
   - Lamp Off (APPI source only)
3. Wait for the source to cool (until temperatures are at least below 100 °C).
4. Disconnect the nebulizer gas tubing from the currently installed ion source.
5. Disconnect the LC/MSD sample inlet tubing.
6. If the APCI or APPI source is installed, remove the APCI vaporizer heater cable and APCI high voltage cable.
7. If the APPI source is installed, remove the serial port B RS-232 cable.
8. Remove the currently installed ion source.

9 Unscrew and remove the spray shield. See Figure 12.

WARNING **Do not touch the multimode source or the capillary cap. They may be very hot. Let the parts cool before you handle them.**

WARNING **Do not insert fingers or tools through the openings on the multimode chamber. When in use, the capillary and capillary cap are at high voltages up to 4 kV.**

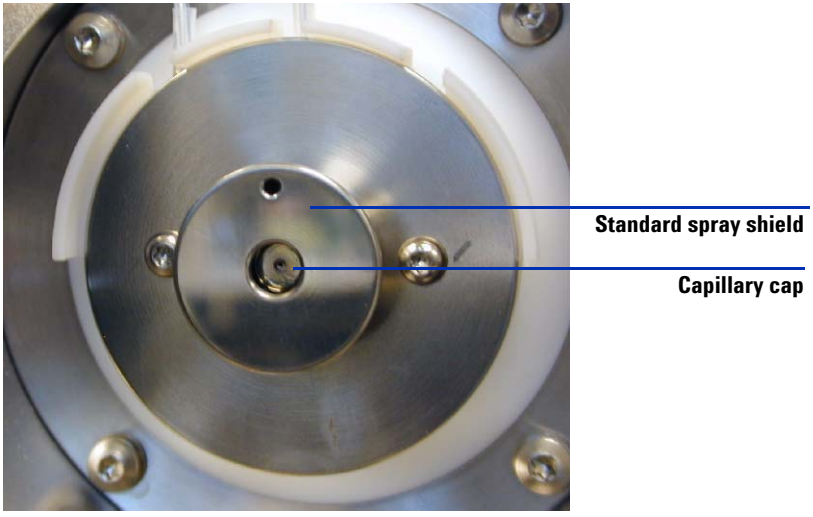


Figure 12 Standard spray shield and capillary cap for ESI or APCI

10 Remove the capillary cap. If needed, moisten a clean cloth with isopropyl alcohol and wipe the capillary cap. See Figure 13.

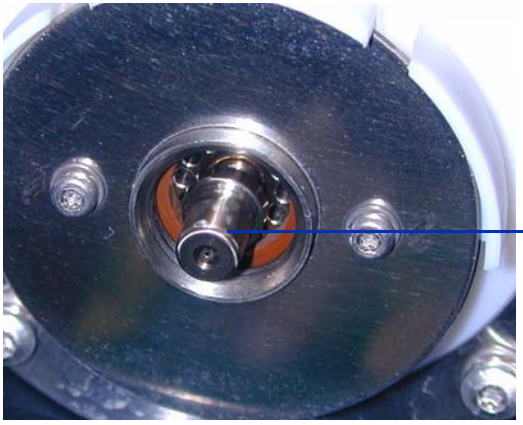


Figure 13 Spray shield removed.

11 Place the capillary cap back on the capillary.

12 Install the new spray shield with field shaping electrodes. See Figure 14.

Figure 14 Multimode spray shield

13 Screw the multimode spray shield into the holder for the spray shield. See Figure 15

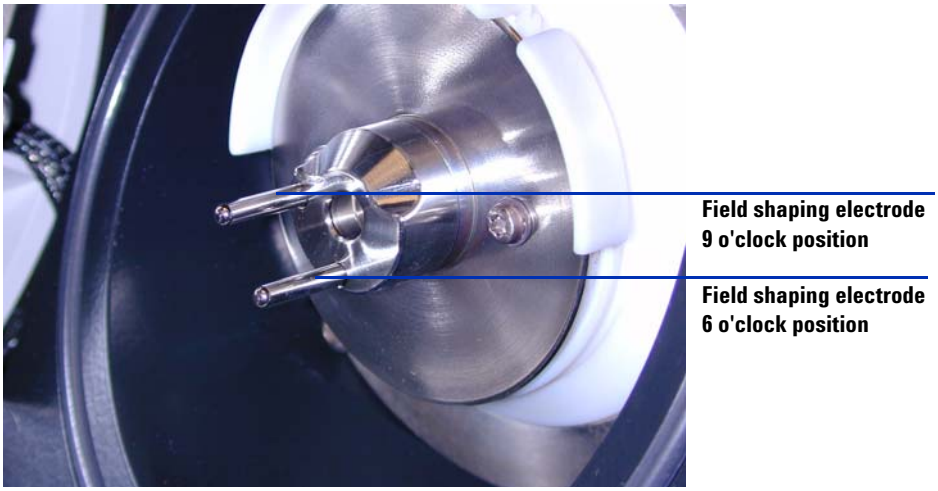


Figure 15 Multimode spray shield installed

NOTE The field shaping electrodes should be in the nine o'clock and the six o'clock position. Loosen the end plate screws on each side to adjust the field shaping electrodes position.

14 Remove the shipping cover from the multimode source spray chamber.

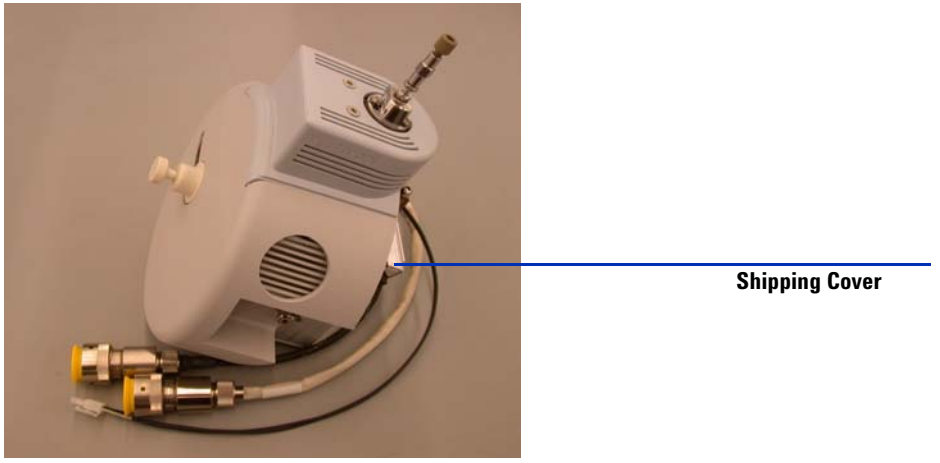


Figure 16 Multimode Spray Chamber with shipping cover

15 Install the spray chamber on the spray chamber mount.

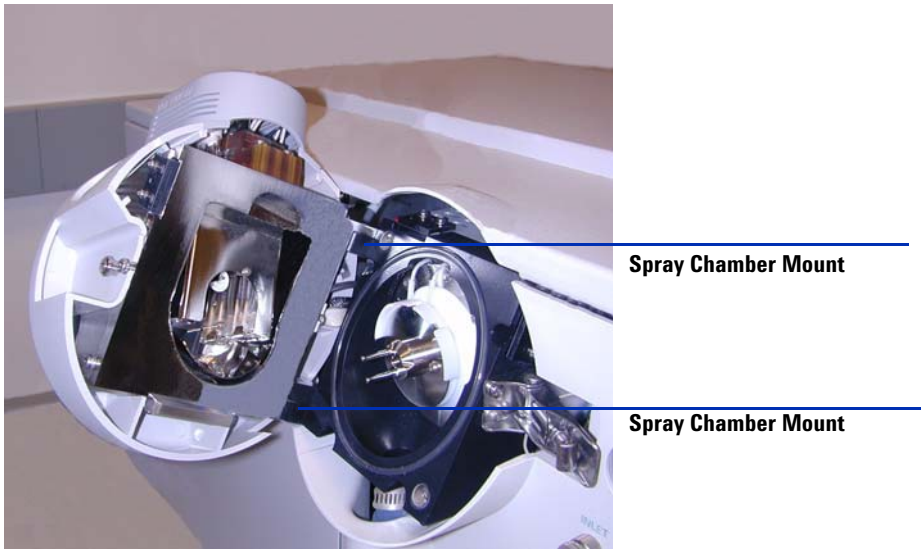


Figure 17 Multimode source installed on the spray chamber mount

16 Install the nebulizer on the multimode source spray chamber.

Figure 18 No nebulizer on top of the multimode source

17 Connect the 1/8-inch nebulizer gas tubing from the LC/MSD mainframe to the nebulizer gas fitting. See Figure 19.

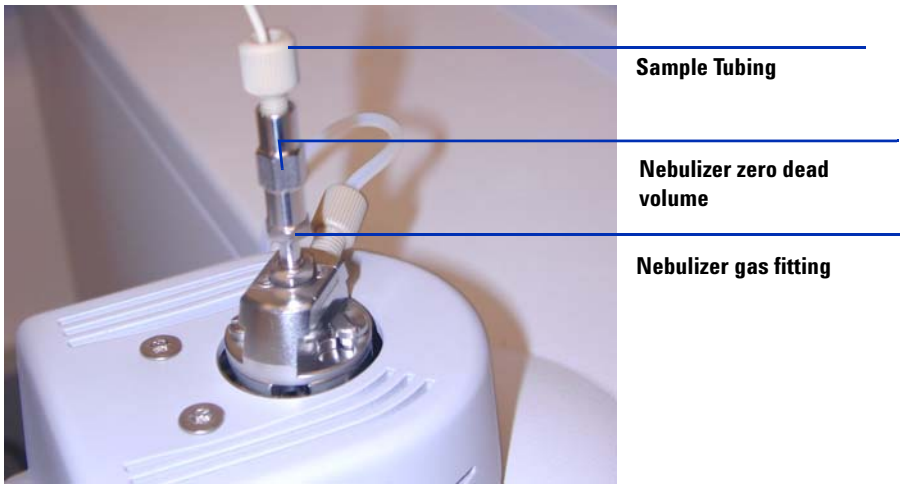


Figure 19 Nebulizer with gas tubing connected

18 Connect the LC/MSD sample tubing to the LC/MSD diverter valve inlet filter. See Figure 20 on page 29.

WARNING **The Agilent 1100 Series LC/MSD Liquid Chromatograph diverter valve is an integral part of the G1978A safety system. The LC mobile phase flow must always be connected to the diverter valve inlet filter. Never bypass the diverter valve and connect directly to the nebulizer. If the diverter valve is used in a manner not specified by Agilent Technologies, the protections provided by the diverter valve may be impaired.**

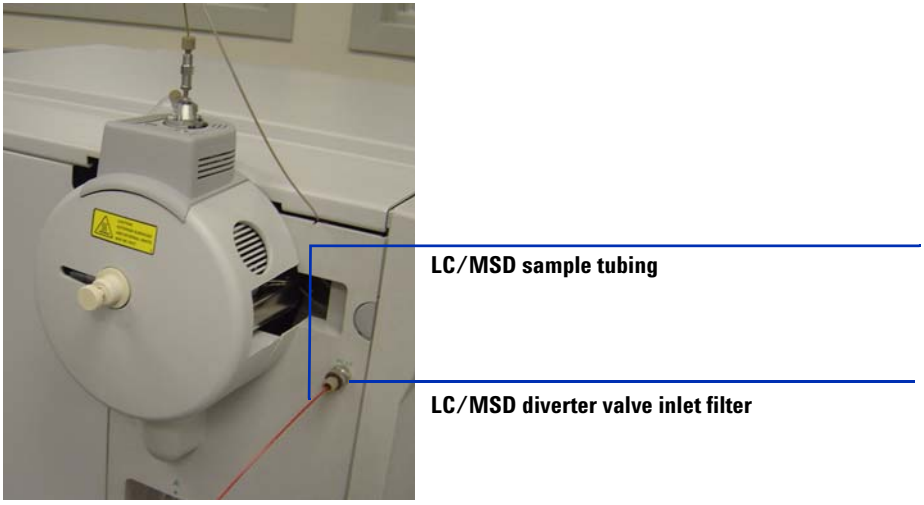


Figure 20 LC/MSD sample tubing connected to LC/MSD inlet filter

19 If you are installing the multimode source for the first time, follow the steps in "To install the HV control PCA" on page 30.

20 Follow the steps in "To connect multimode source cables" on page 34.

21 If you are installing the source for the first time, return to section "Step 6. Upgrade the software with the G1978-10002 patch" on page 16.

To install the HV control PCA

1 Remover the cover from the source HV and control PCA power supply. See Figure 21.

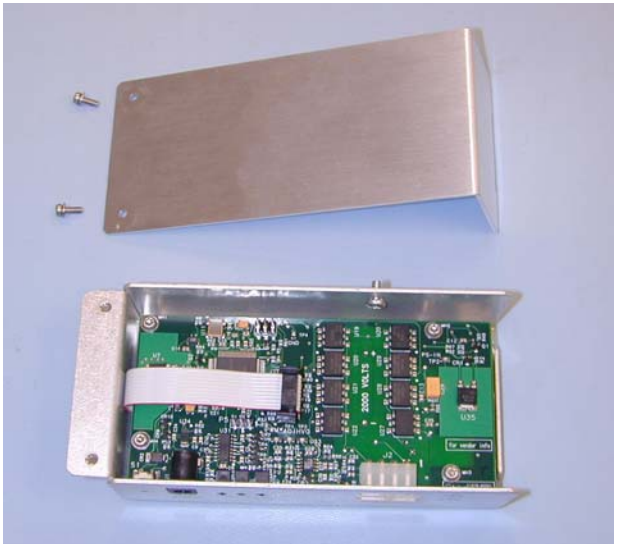


Figure 21 Cover removed from the source HV and control PCA power supply

2 Attach the RS-232 cable to the HV and control PCA power supply RS-232 connector. See Figure 22.

Figure 22 Attaching the RS-232 cable

3 Remove the instrument front cover, top cover, safety cover with magnet, and side panel access door.

4 Remove the plastic cable clamp from the desolvation heater cable See Figure 23.

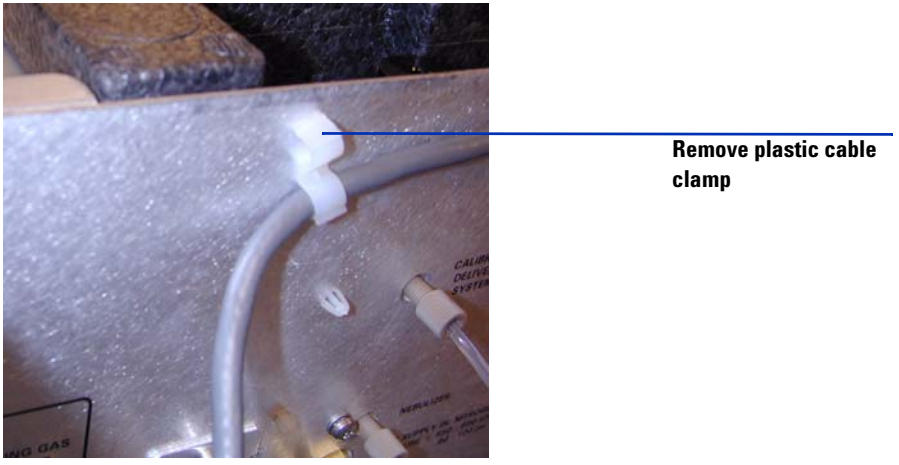


Figure 23 Cable clamp removal

5 Re-route the cable under the Calibrant Delivery System gas line. See Figure 24.

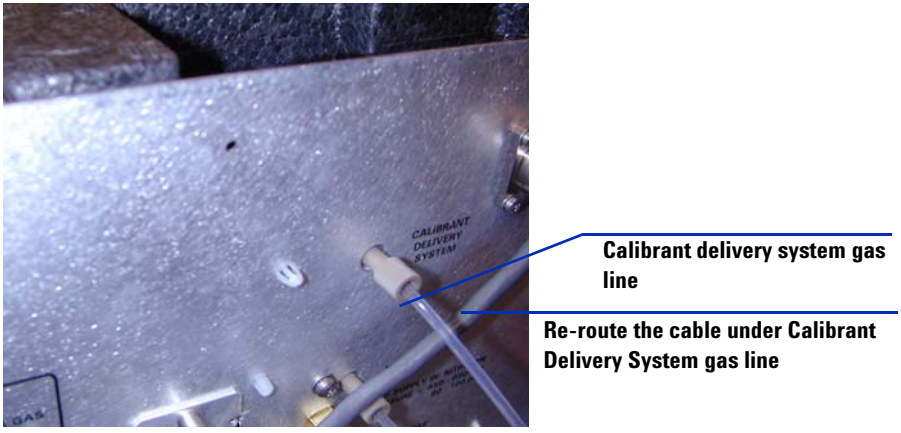


Figure 24 Calibrant delivery system gas line

6 Attach the HV and control PCA power supply to the tub with the self-trapping screw supplied. See Figure 25.

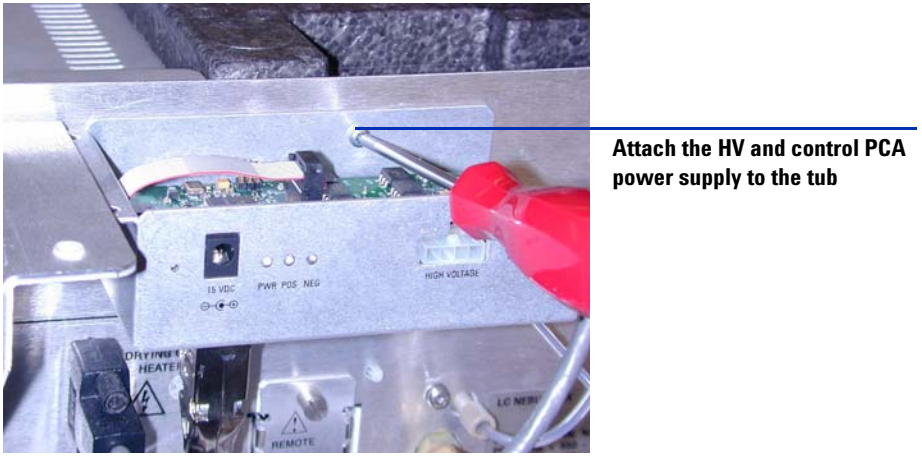


Figure 25 Attach the HV and control PCA power supply

7 Clamp the top cover of the HV and control PCA power supply with screws provided to the support bracket. See Figure 26.

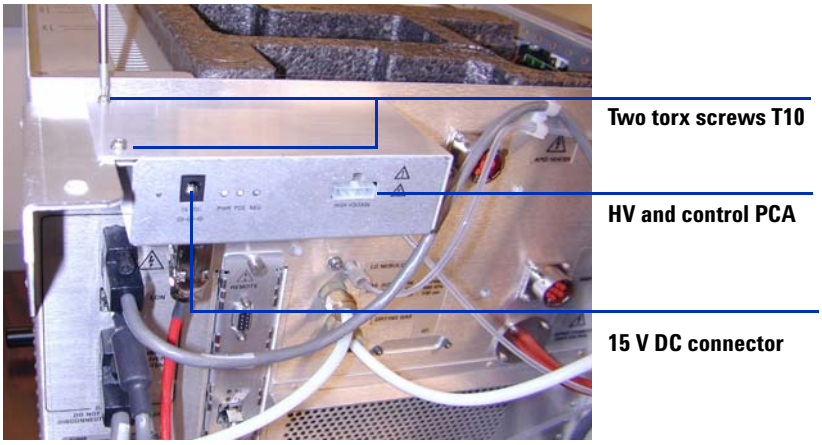


Figure 26 Clamping to support brackets

8 If you are installing the HV control PCA as part of a conversion to the multimode source, return to **"To convert from ESI, APCI or APPI to the multimode source"** on page 23.

To connect multimode source cables

1 Connect the RS-232 cable to the Serial B connector on the Smart Card 3 interface, which is located on the left side of the instrument chassis. See Figure 27.

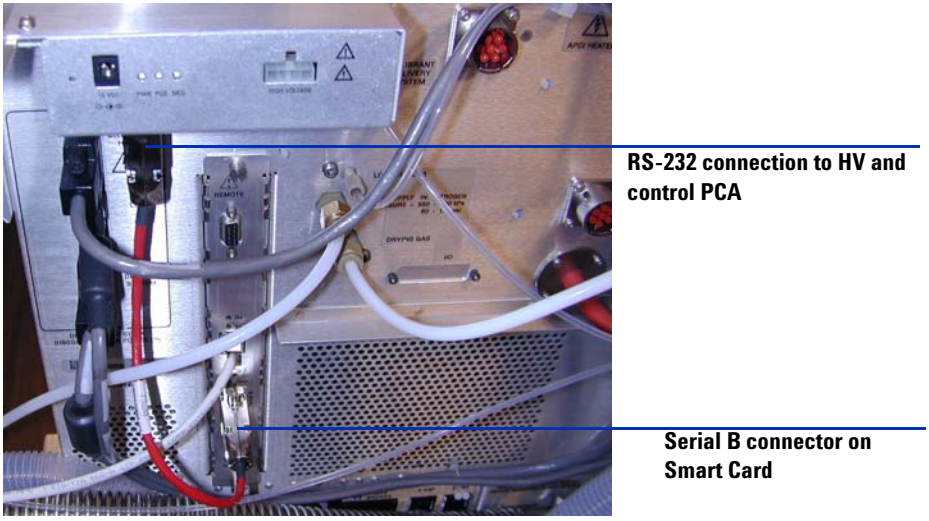


Figure 27 RS-232 cable connections

2 Connect the 15 vdc power supply to the HV and control PCA. See Figure 28.

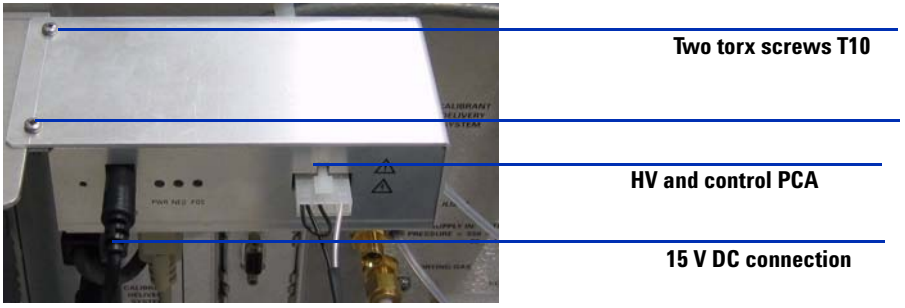


Figure 28 HV and control PCA

3 Connect the other end of the 15 V DC power supply into an 110vac outlet using the power cord supplied with the 15 V DC power supply. See Figure 29.

Figure 29 Power cord and 15 VDC supply

4 Use a cable-tie to +15 V output power cable of the power supply (p/n 0950-4581) to the RS-232 cable of the Multimode HV Module Assembly (p/n G1978-60050.) See Figure 30.

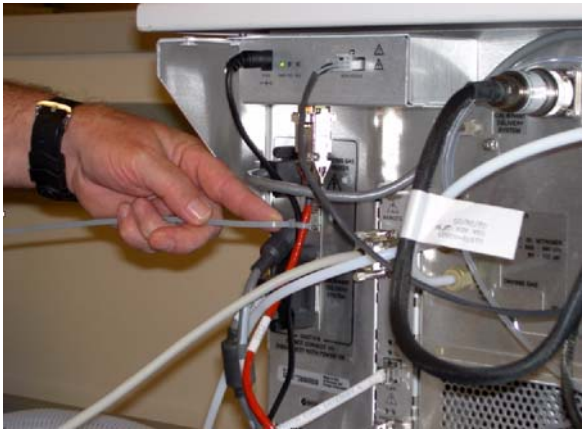


Figure 30 Cable-tie attaching the power cable to the RS-232 cord.

5 Connect the vaporizer heater, APCI high voltage, and HV and control PCA cables. The APCI heater connector, APCI high voltage connector, and HV

and control PCA connector are located on the left side of the instrument chassis. See Figure 31.

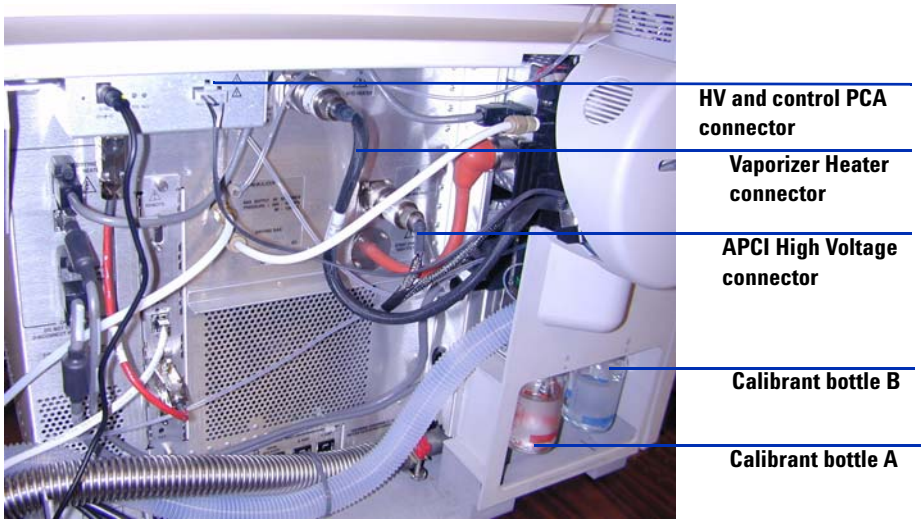


Figure 31 Multimode source cable connections

6 Verify that the multimode source bottle A of the calibrant delivery system (CDS) has a sufficient amount of APCI/APPI calibrant solution (100 mL) (G2432A). See Figure 31.

7 Verify that the multimode source bottle B of the calibrant delivery system (CDS) has a sufficient amount of ES calibrant solution (100 mL)(G2421A). See Figure 31.

8 Close service panel door and validate all covers are in place. See Figure 32.

9 Return to the section "To convert from ESI, APCI or APPI to the multimode source" on page 23.

Figure 32 Multimode source with covers installed

To remove the multimode source

Do the following steps to remove the multimode source.

1 The source temperatures for vaporizer heater and drying gas heater need to be set to minimum values to cool the source. Use the **Tune > Instrument > Edit Spray Chamber** menu item to display the Edit Spray Chamber dialog box. Set the drying gas flow, nebulizer gas flow, drying gas temperature and the vaporizer temperature to minimum values.

WARNING **Do not touch the multimode source or the capillary cap. They may be very hot. Let the parts cool before you handle them.**

WARNING **Never touch the source surfaces, especially when you analyze toxic substances or when you use toxic solvents. The source has several sharp pieces which can pierce your skin including the APCI corona needle, vaporizer sensor and counter current electrode.**

WARNING **Do not insert fingers or tools through the openings on the multimode chamber. When in use, the capillary and capillary cap are at high voltages up to 4 kV.**

2 Wait around 20 minutes until the source is cool.

3 Open the service door on the left side of the MSD to access the cables. See Figure 33.

Figure 33 Instrument with multimode source installed

4 Disconnect the ESI high voltage charging electrode cable. See Figure 34.

5 Disconnect the APCI heater (vaporizer) cable and APCI High voltage cable. See Figure 34.

6 Unplug the 15 V DC connection to the multimode electronics module. See Figure 34.

NOTE If you do not remove the power to the multimode electronics module, the new source will be identified as unknown source.

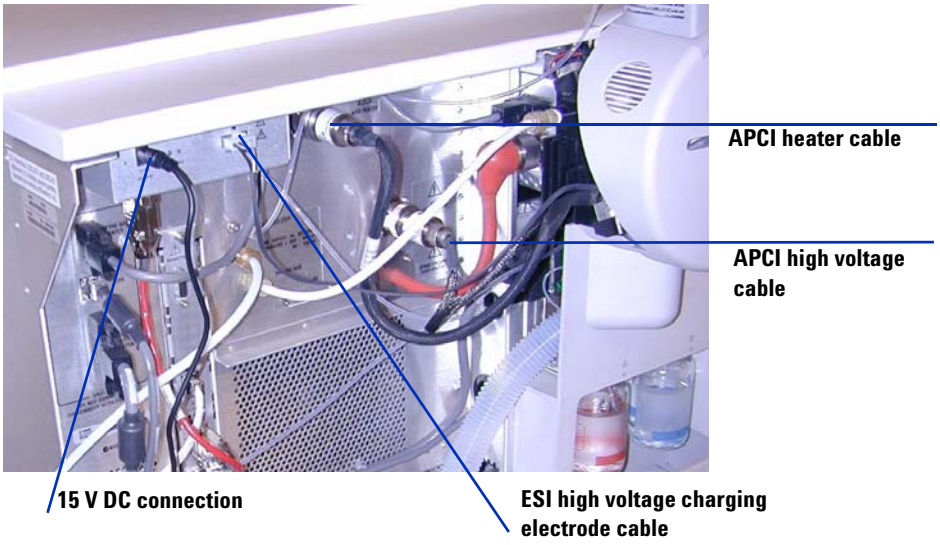


Figure 34 Instrument with service door open

7 Unscrew the nebulizer gas line from the nebulizer.

8 Unscrew the LC sample tubing from the nebulizer.

9 Open the latch on the source and open the source.

10 Remove the multimode source from the spray chamber mount.

11 Place the source shipping cover on the source.

12 If you were converting from a multimode source, continue in the section "To convert from multimode to ESI, APCI or APPI" on page 41.

13 If you were cleaning the multimode source, continue in the section "To clean the multimode source weekly" in the *Maintenance Guide*.

To convert from multimode to ESI, APCI or APPI

WARNING **Do not touch the multimode source or the capillary cap. They may be very hot. Let the parts cool before you handle them.**

WARNING **Never touch the source surfaces, especially when you analyze toxic substances or when you use toxic solvents. The source has several sharp pieces which can pierce your skin including the APCI corona needle, vaporizer sensor and counter current electrode.**

1 Do the steps in "To remove the multimode source" on page 38.

2 If the source to be installed is an APPI source, disconnect the multimode high voltage PCA RS-232 serial cable from the serial port B connector of Smart Card.

3 Unscrew and remove the multimode spray shield with the field shaping electrodes.

4 Install the new source and the standard spray shield, making sure that the hole in the spray shield is in the 12 o'clock position.

5 For APCI and APPI ion source, connect the vaporizer heater cable and the APCI high voltage cable. For the APPI source, connect the RS-232 cable to the serial port B connector of Smart Card.

6 For all sources, reconnect the nebulizer gas line tubing and the LC/MSD sample tubing.

2
Verification

This chapter includes the tasks that you need to do for verification of your multimode source and to validate the proper operation of your source.

To determine proper solvent mixture for performance verification

Solvent dilutions are given for all supported instruments with multimode source. The reserpine performance verification is only to be done on Bundled G1956A and G1956B instruments shipped with a multimode source.

Use the following information to determine the proper solvent mixture for your instrument model.

G1956A or G1946C LC/MSD VL

Any of the following organic solvents can be used: methanol, isopropanol or acetonitrile.

- 50:50 organic solvent/water

G1956B or G1946B/D LC/MSD SL

- 75:25 methanol/water with 5 mM ammonium formate.

  To make the 5mM ammonium formate solution, add 0.315g of ammonium formate to 1 liter of 75:25 methanol/water mobile phase. Use ammonium formate with 97% purity or better.

For both the VL and SL model of the LC/MSD

- Up to 0.2% acetic acid or 0.1% formic acid can be added for positive ion verification. Doing this is usually not necessary, but may be beneficial in overcoming ion suppression resulting from background contaminants in the mobile phase.
- Use solvents of at least HPLC grade. Solvents that are acceptable for most LC applications may contain high levels of background that are detectable by the more sensitive LC/MSD. Make sure LC solvents used with the LC/MSD are rated for both HPLC and pesticide, environmental, or GC/MS analyses. Use the highest purity solvents you can obtain. Acceptability of solvents must be empirically determined.

To prepare performance evaluation samples

NOTE This verification method may only be used on a bundled instrument shipped with a multimode source.

Before you begin, check that you have:

- 1 mL graduated pipette, p/n 9301-1423
- 50 mL volumetric flask (two each), p/n 9301-1424
- 100 mL volumetric flask, p/n 9301-1344
- Positive ion performance evaluation sample, p/n G2423A (for both interfaces)
- Plastic bottles for storing dilutions, p/n 9301-1433

A bundled instrument will come with the supplies listed above.

The supplied performance evaluation samples must be diluted to concentrations required for the LC/MSD system checkout. Refer to the section "To determine proper solvent mixture for performance verification" for more information.

NOTE Use the diluted samples within a day of dilution. Refrigerate the intermediate (first) dilution in the supplied bottles.

Tips

- Rinse the graduated pipettes and volumetric flasks thoroughly with deionized water before, in between, and after use.
- Use polypropylene labware for preparing performance evaluation samples, since glass vessels introduce unacceptable levels of sodium. Always rinse the autosampler vials and caps with the solvent mix used for sample dilution before filling them with the performance verification samples. Doing this minimizes any background contributed by the vials and caps. The vials may be run uncapped if the septa are found to be a source of background contamination.

Table 1 G1956A VL Performance Summary SIM, mode

	MM-ES Positive SIM Mode	MM-APCI Positive SIM Mode
Sample	Reserpine, 5 ng/ µL	Reserpine, 5 ng/ µL
Concentration after dilution	2 pg/µL	2 pg/µL
Injection volume	5 µL	5 µL
Total sample amount injected	10 pg	10 pg
Sample order number	G2423A	G2423A
Solvent	50:50 organic / water	50:50 organic / water
Method name	56VLSMES_MM.M	56VLSMCI_MM.M
Performance specifications	20: 1 pk-pk 100: 1 rms	10: 1 pk-pk 50: 1 rms

Table 2 G1956B SL Performance Summary SIM, mode

	MM-ES Positive SIM Mode	MM-APCI Positive SIM Mode
Sample	Reserpine, 5 ng/ µL	Reserpine, 5 ng/ µL
Concentration after dilution	1 pg/µL	1 pg/µL
Injection volume	1 µL	1 µL
Total sample amount injected	1 pg	1 pg
Sample order number	G2423A	G2423A
Solvent	75:25 methanol / water with 5mM ammonium formate	75:25 methanol / water with 5mM ammonium formate
Method name	56SLSMES_MM.M	56SLSMCI_MM.M
Performance specifications	20: 1 pk-pk 100: 1 rms	10: 1 pk-pk 50: 1 rms

Table 3 G1956B SL Performance Summary SCAN, mode

	MM-ES Positive SCAN Mode	MM-APCI Positive SCAN Mode
Sample	Reserpine, 5 ng/ µL	Reserpine, 5 ng/ µL
Concentration after dilution	10 pg/µL	10 pg/µL
Injection volume	5 µL	5 µL
Total sample amount injected	50 pg	50 pg
Sample order number	G2423A	G2423A
Solvent	75:25 methanol / water with 5mM ammonium formate	75:25 methanol / water with 5mM ammonium formate
Method name	56SLSCES_MM.M	56SLSCCI_MM.M
Performance specifications	20: 1 pk-pk 100: 1 rms	10: 1 pk-pk 50: 1 rms

Table 4 G1946C Performance Summary SIM, mode

	MM-ES Positive SIM Mode	MM-APCI Positive SIM Mode
Sample	Reserpine, 5 ng/ µL	Reserpine, 5 ng/ µL
Concentration after dilution	2 pg/µL	2 pg/µL
Injection volume	5 µL	5 µL
Total sample amount injected	10 pg	10 pg
Sample order number	G2423A	G2423A
Solvent	50:50 organic / water	50:50 organic / water
Method name	MSSUPRES_MM.M	MSSUPCI_MM.M
Performance specifications	10: 1 pk-pk 50: 1 rms	5: 1 pk-pk 25: 1 rms

Table 5 G1946D SL Performance Summary, SIM and SCAN mode

	MM-ES Positive SIM Mode Evaluation	MM-APCI Positive SIM Mode Evaluation	MM-ES Positive SCAN Mode	MM-APCI Positive SCAN Mode
Sample	Reserpine, 5 ng/ µL	Reserpine, 5 ng/ µL	Reserpine, 5 ng/ µL	Reserpine, 5 ng/ µL
Concentration after dilution	1 pg/µL	1 pg/µL	10 pg/µL	10 pg/µL
Injection volume	1 µL	1 µL	5 µL	5 µL
Total sample amount injected	1 pg	1 pg	50 pg	50 pg
Sample order number	G2423A	G2423A	G2423A	G2423A
Solvent	75:25 methanol / water with 5mM ammonium formate	75:25 methanol / water with 5mM ammonium formate	75:25 methanol / water with 5mM ammonium formate	75:25 methanol / water with 5mM ammonium formate
Method name	SLSIMES_MM.M	SLSIMCI_MM.M	SLSCNES_MM.M	SLSCNCI_MM.M
Performance specifications	10: 1 pk-pk 50: 1 rms	5: 1 pk-pk 25: 1 rms	10: 1 pk-pk 50: 1 rms	5: 1 pk-pk 25: 1 rms

Table 6 G1946B Performance Summary SIM, mode

	MM-ES Positive SIM Mode	MM-APCI Positive SIM Mode
Sample	Reserpine, 5 ng/ µL	Reserpine, 5 ng/ µL
Concentration after dilution	2 pg/µL	2 pg/µL
Injection volume	5 µL	5 µL
Total sample amount injected	10 pg	10 pg
Sample order number	G2423A	G2423A
Solvent	50:50 organic / water	50:50 organic / water

Table 6 G1946B Performance Summary SIM, mode

	MM-ES Positive SIM Mode	MM-APCI Positive SIM Mode
Method name	MSSUPRES_MM.M	MSSUPCI_MM.M
Performance specifications	10: 1 pk-pk 50: 1 rms	5: 1 pk-pk 25: 1 rms

G1946B/C Multimode Source, Positive SIM Mode Dilutions

1. Transfer 1 mL of 5 ng/µL reserpine (Agilent G2423A) to a 50 mL volumetric flask. Use a clean graduated pipette.
2. Dilute to the 50 mL mark with 50:50 organic solvent / water.
3. Transfer 1 mL of the first dilution to a second 50 mL volumetric flash. Use a clean graduated pipette.
4. Dilute to the 50 mL mark with 50:50 organic solvent / water. This provides the final 2 pg/µL reserpine concentration required for evaluation.
5. Transfer approximately 1 mL of the second dilution to a vial for use in the LC Autosampler.

G1956B or G1946D SL Multimode Source, Positive SIM Mode Dilutions

1. Transfer 1 mL of 5 ng/ µL reserpine (Agilent G2423A) to a 50 mL volumetric flask. Use a clean graduated pipette.
2. Dilute to the 50 mL mark with 75:25 methanol / water with 5 mM ammonium formate.
3. Transfer 1 mL of the first dilution to a 100 mL volumetric flask. Use a clean graduated pipette.
4. Dilute to the 100 mL mark with 75:25 methanol / water with 5 mM ammonium formate. This provides the final 1pg/ µL reserpine concentration required for evaluation.
5. Transfer approximately 1 mL of the second dilution to an autosampler vial.

G1956B or G1946D SL Multimode Source, Positive Scan Mode Dilutions

1. Transfer 1 mL of 5 ng/µL reserpine (Agilent G2423A) to a 50 mL volumetric flask. Use a clean graduated pipette.
2. Dilute to the 50 mL mark with 75:25 methanol / water with 5 mM ammonium formate.
3. Transfer 5 mL of the first dilution to a 50 mL volumetric flask. Use a clean graduated pipette.
4. Dilute to the 50 mL mark with 75:25 methanol / water with 5 mM ammonium formate. This provides the final 10 pg/µL reserpine concentration required for evaluation.
5. Transfer approximately 1 mL of the second dilution to an autosampler vial.

To verify multimode source operations

Use the methods specified below to verify the performance of the LC/MSD system for the multimode source purchased with the system. The performance verification methods require an Agilent 1100 LC with an autosampler.

NOTE Check that you have entered the custom tune parameters. Otherwise, you might not be able to tune the LC/MSD.

- G1956A Multimode Source Interface, Positive SIM
- G1956B Multimode Source Interface, Positive Scan
- G1956B Multimode Source Interface, Positive SIM
- G1956B LC/MSD SL model G1978A Interface in Mixed Mode operation
- Multiple FIA model G1978A Interface in Mixed Mode operation

Loading methods for G1956A Multimode Source Interface, Positive SIM

1. Load the method **56VLSMES_MM.M** for the G1956A.
2. Edit the method to ensure that 50:50 organic solvent / water is selected as the LC solvent. All other LC parameters correspond to the **56VLSMES_MM.M** method parameters.
3. Do an autotune with APCI multimode source calibrant.

NOTE After the autotune has completed, you may need to wait up to 30 minutes before continuing to allow the calibrant solution to be pumped out of the MSD. This minimizes any background signal from the calibrant.

NOTE You may need to further optimize the nebulizer pressure to achieve maximum instrument sensitivity.

4. Place the vials into the LC autosampler.
   - Position 1: empty, uncapped vial
   - Position 2: vial of the solvent used for dilution (solvent blank)
   - Position 3: vial with the reserpine sample (2 pg/µL)

5 Run the method.

The method performs an FIA run with one injection of the empty vial, five injections of the solvent blank, and five injections of the reserpine sample.

6 Review the results.

When the method is finished, a report prints showing the signal-to-noise ratio for the five blank and five sample peaks, and a blank-subtracted average of the sample peaks. This is to verify operation of the multimode source. The five sample peaks are visible in the EIC.

Loading method for G1956B Multimode Source Interface, Positive Scan

1 Load the method **56SLSCES_MM.M** for the G1956B.

2 Edit the method to ensure that 75:25 methanol / water with 5mM ammonium formate is used. All other LC parameters correspond to the **56SLSCES_MM.M** method parameters.

3 Do an autotune.

NOTE After the autotune has completed, you may need to wait up to 30 minutes before continuing to allow the calibrant solution to be pumped out of the MSD. This minimizes any background signal from the calibrant.

NOTE You may need to further optimize the nebulizer pressure to achieve maximum instrument sensitivity.

4 Place the vials into the LC autosampler.

- Position 1: empty, uncapped vial
- Position 2: vial of the solvent used for dilution (solvent blank)
- Position 3: vial with the reserpine sample (10 pg/µL)

5 Run the method.

The method performs an FIA run with one injection of the empty vial, five injections of the solvent blank, and five injections of the reserpine sample.

6 Review the results.

When the method is finished, a report prints showing the signal-to-noise ratio for the five blank and five sample peaks, and a blank-subtracted average of the sample peaks. This is to verify operation of the multimode source. The five sample peaks are visible in the EIC.

Loading methods G1956B Multimode Source Interface, Positive SIM

1 Load the method **56SLSM_MM.M** for the G1956B.

2 Edit the method to ensure that 75:25 methanol / water with 5mM ammonium formate is selected as the LC solvent.

All other LC parameters correspond to the **56SLSMES_MM.M** method parameters.

3 Do an autotune.

NOTE After the autotune has completed, you may need to wait up to 30 minutes before continuing to allow the calibrant solution to be pumped out of the MSD. This minimizes any background signal from the calibrant.

NOTE You may need to further optimize the nebulizer pressure to achieve maximum instrument sensitivity.

4 Place the vials into the LC autosampler.

- Position 1: empty, uncapped vial
- Position 2: vial of the solvent used for dilution (solvent blank)
- Position 3: vial with the reserpine sample (1 pg/µL)

5 Run the method.

The method does an FIA run with one injection of the empty vial, five injections of the solvent blank, and five injections of the reserpine sample.

6 Review the results.

When the method is finished, a report prints showing the signal-to-noise ratio for the five blank and five sample peaks, and a blank-subtracted average of the sample peaks. This is to verify operation of the multimode source. The five sample peaks are visible in the EIC.

Loading Multiple FIA method for G1978A Interface in Mixed Mode operation

1 Do an autotune if needed.

NOTE After the autotune has completed, you may need to wait up to 30 minutes before continuing to allow the calibrant solution to be pumped out of the MSD. This minimizes any background signal from the calibrant.

NOTE You may need to further optimize the nebulizer pressure to achieve maximum instrument sensitivity.

2 Load the method **MMCheckSL_ES.M**.

3 Edit the method to ensure that 65:35 methanol / water with 0.2% acetic acid is selected as the LC solvent. Save the method. Repeat for **MMCheckSL_CI.M** and **MMCheckSL_MX.M**.

NOTE If the instrument is a VL model, substitute methods **MMCheckVL_ES.M**, **MMCheckVL_CI.M**, and **MMCheckVL_MX.M** for the methods in steps 1 through 5.

4 Place the vial into the LC autosampler.

Position 21: vial with the ESI + APCI LC Demo Sample (p/n G1978-85000)

5 Set up a Multiple FIA Method sequence

a Select **RunControl > Run Multiple FIA Methods...** menu item.

b In the Run Multiple FIA Methods dialog box, use the **Group > Add Group** menu item. Type in a unique directory name for the data files to be stored in.

c In the Run Multiple FIA Methods dialog box, use the **Methods > Add Method** menu item. Select **MMCheckSL_ES.M** and use the **OK** button.

d Repeat to add **MMCheckSL_CI.M**.

e Repeat to add **MMCheckSL_MX.M**.

f Click on the **Data File** field for the **MMCheckSL_ES.M** data file.

- Edit the **Subdirectory** field to a unique subdirectory name in which to store the data files.
- Edit the **Data File** field to **Multi_ES**.

- Edit the **Operator** field with the users name or identification code.
- Click on the **OK** button.

g Repeat for the **MMCheckSL_CI.M** data file, using the same subdirectory as above and entering **Multi_CI** as the data file name.

h Repeat for the **MMCheckSL_MX.M** data file, using the same subdirectory as above and entering **Multi_MX** as the data file name.

i Click on the **Run** button to start the sequence

6 Review the results. When the last method has finished, the report "Multimode Verification Report" will print as shown in "Example of multimode verification report" on page 57.

To do an autotune

Tuning the multimode source is done in MM-APCI mode only. Autotune is done from the same menu as with all sources.

- From the **MSD Tune view**, select **Instrument > Autotune** menu item.

The tune report will have a header with either the title **MM-APCI Positive Mode - Standard Scan** or **MM-APCI Negative Mode - Standard Scan**. You can run the check tune after an autotune to validate that the instrument passes check tune criteria. Do the autotune after the system has had at least 8 hours to equilibrate vacuum and temperatures.

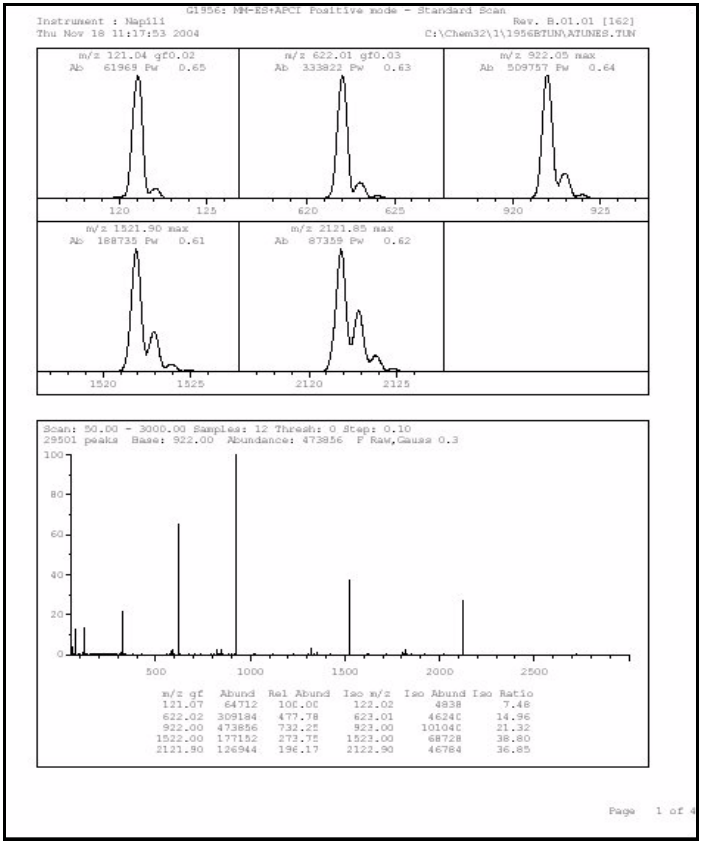


Figure 35 Autotune report

Example of multimode verification report

Multimode Ion Source Report

MSD type: G1956B Instrument name: Instrumen Operator name: pcormia

Acquisition date: 23-Feb-2005
Datafiles:
ESI mode : C:\Chem32\1\DATA\MMSTD_223\Multi_ES.d
APCI mode : C:\Chem32\1\DATA\MMSTD_223\Multi_CI.d
Mixed mode : C:\Chem32\1\DATA\MMSTD_223\Multi_MX.d

ESI Compound Results

Compound	m/z	Polarity	ESI mode	Mixed mode	Mixed:ESI ratio	Result
Crystal Violet	372.2	Positive	832925	541200	64.9 %	Pass
1-Hexanesulfonic acid	165.1	Negative	220506	181617	82.3 %	Pass

APCI Compound Results

Compound	m/z	Polarity	APCI mode	Mixed mode	Mixed:APCI ratio	Result
Carbazole	168.1	Positive	623026	225911	36.2 %	Pass
9-Phenanthrol	193.1	Negative	451189	254201	56.3 %	Pass

Passing criteria: Mixed mode response 20% or greater of single-mode response.

3 Methods

This chapter describes the tasks that you need to set up methods for the multimode source.

To setup a method to use the multimode source

To have your method use a multimode source, follow these steps:

1. Open the MSD Spray Chamber dialog box by clicking **Instrument > MSD Spray Chamber** in the **Method and Run Control** view.
2. Set **Method Spray Chamber** to **MM-ES+APCI**.
3. Verify that **Installed Spray Chamber** is set to **MM-ES+APCI**.
4. Make any other changes that are necessary for your method.
5. Click the **OK** button.
6. Open The Set up MSD Signals dialog box by clicking **Instrument > More > Set up MSD Signals** in the **Method and Run Control** view.
7. Choose the desired ionization mode from the **Ionization** list. This list is only visible if the Method Spray Chamber was set to **MM-ES+APCI**. You may set the ionization mode to one of the following:
   - **MM-ES**
   - **MM-APCI**
   - **MM-ES+APCI**
8. Make any other changes that are necessary for your method.
9. Click the **OK** button.

WARNING **The 6100 Series Single Quad LC/MS Liquid Chromatograph diverter valve is an integral part of the G1978B safety system. The LC mobile phase flow must always be connected to the diverter valve inlet filter. Never bypass the diverter valve and connect directly to the nebulizer. If the diverter valve is used in a manner not specified by Agilent Technologies, the protections provided by the diverter valve may be impaired and the system may catch fire.**

To create a method for positive/negative mixed mode operation

1. Open the MSD Spray Chamber dialog box by clicking the **Instrument > Set Up MSD Signals** in the **Method and Run Control** view.
2. Select **MM-ES_APCI** from the **Method Spray Chamber** drop-down list.
3. Check that **Installed Spray Chamber** is also set to **MM-ES+APCI**.
4. Make any other changes that are necessary for your method.
5. Click the **OK** button.
6. Open the Set up MSD Signals dialog box by clicking the **Instrument > MSD Spray chamber** in the **Method and Run Control** view.
7. Modify the settings so that Signal 1 has **Positive** polarity and Signal 2 has **Negative** polarity as shown in Figure 36.
8. Make any other changes that are necessary for your method.
9. Click the **OK** button.

Fast positive/negative polarity switching is a very useful technique but it requires time for the ion chemistry to be established and the optics path to refill with ions. The gas density plays a role in the speed of refilling the ion path. The gas density is affected by source temperature. For a method running positive/negative switching, use a lower vaporizer temperature (150 to 200°C) and a lower Vcap (approximately 1000 V). These will greatly affect the quality of the results in positive/negative switching experiments.

Polarity options for Signal 1 and Signal 2

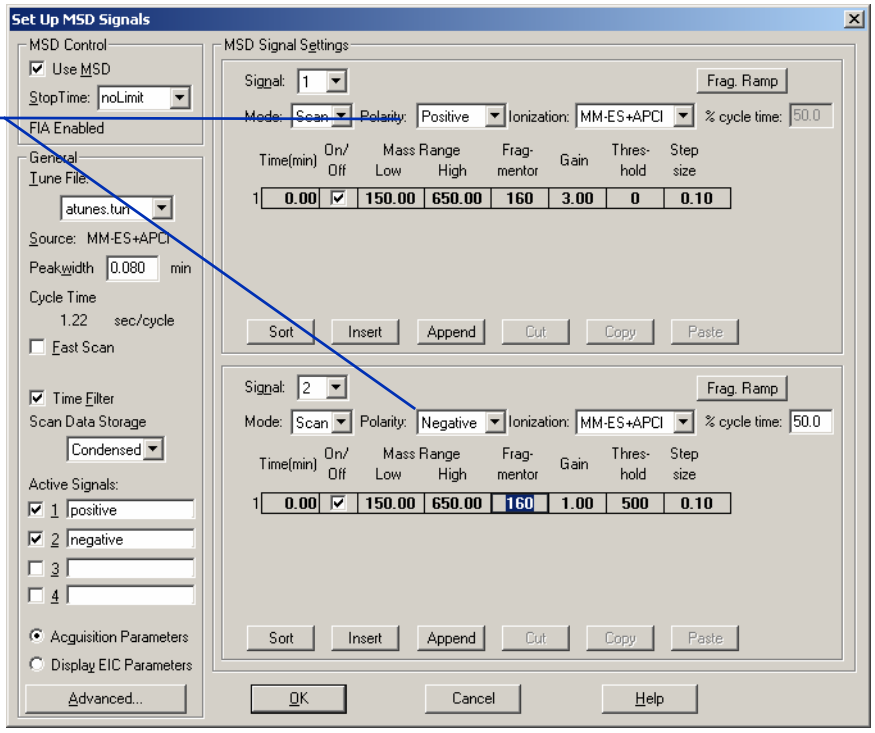


Figure 36 Positive/Negative polarity switching using Set Up MSD Signals dialog box

To create a method for alternating ESI and APCI operation

1 Open The MSD Spray Chamber dialog box by clicking **Instrument > MSD Spray Chamber** in the **Method and Run Control** view.

2 Set **Method Spray Chamber** to **MM-ES+APCI**.

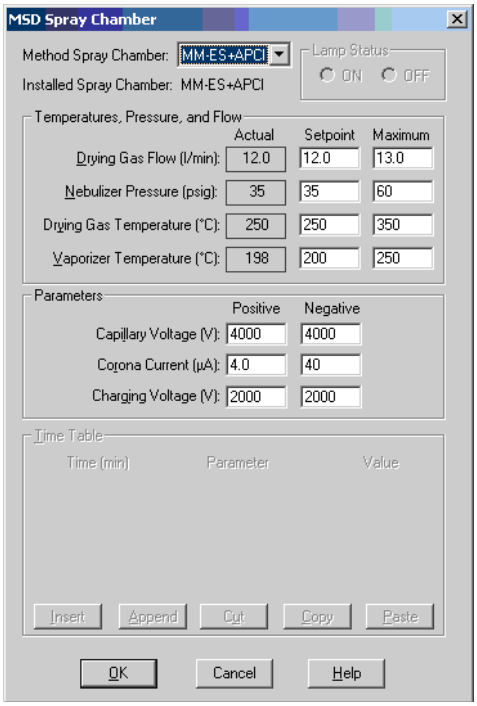


Figure 37 Method Spray Chamber set to MM-ES+APCI.

3 Check that **Installed Spray Chamber** is also set to **MM-ES+APCI**.

4 Make any other changes that are necessary for your method.

5 Click the **OK** button.

6 Open the Set up MSD Signals dialog box by clicking **Instrument > Set Up MSD Signals** in the **Method and Run Control** view.

7 Modify the settings so that Signal 1 has **Ionization** value of **MM-ES** and Signal 2 has **Ionization** value of **MM-APCI** as shown in Figure 38.

8 Make any other changes that are necessary for your method.

9 Click the **OK** button.

NOTE

In general, use the mixed mode operation (MM-ES+APCI setting with Signal 1) instead of alternating MM-ES (Signal 1) and MM-APCI (Signal 2) mode switching. Two times as many scans are obtained during elution of a chromatographic peak, and no delay is needed between scans, resulting in better data. It is rarely necessary to know whether a compound responds purely in ESI or APCI modes on a chromatographic time scale.

Ionization mode for Signal 1 and Signal 2

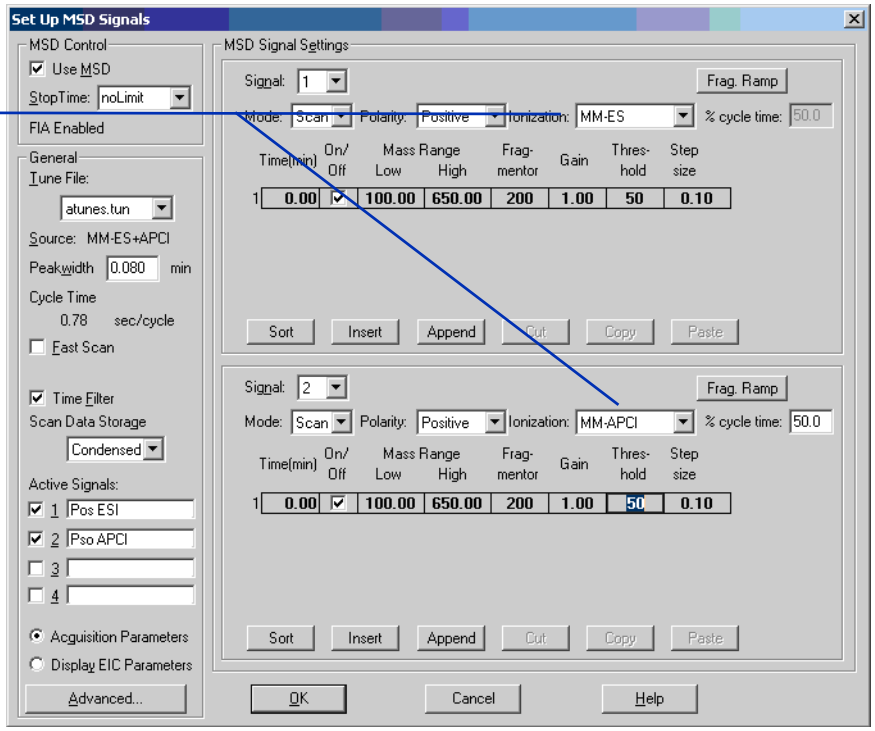


Figure 38 MM-ES and MM-APCI switching using Set Up MSD Signals dialog box

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In This Book

This book contains installation, operation, maintenance and troubleshooting instruction for the Multimode Source for G1946/G1956 LC/MSD.

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