

Agilent G1978B Multimode Source for 6100 Series Single Quad LC/MS

Set-Up Guide



Agilent Technologies

Notices

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CAUTION

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A **WARNING** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a **WARNING** notice until the indicated conditions are fully understood and met.

In This Guide

This guide explains how to install, maintain and troubleshoot your nanoelectrospray 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.

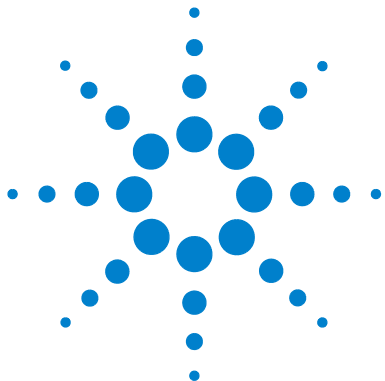
3 Methods

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

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This chapter contains instructions to install the multimode source on a 6100 Series Single Quad LC/MS system, and also to remove and replace the source.



Installation

This section explains how to install the G1978B multimode source on a 6100 Series Single Quad LC/MS instrument. The G1978A multimode source is not supported on these instruments.

The 6100 Series Single Quad LC/MS with multimode source is supported on ChemStation B.03.01 or higher, or the ChemStation B.01.03 with a patch.

Step 1. Prepare to install

The Multimode Enablement Kit, G1978-60451, is shipped with the multimode source. This kit needs to be installed before the multimode source is used.

Note that the multimode source and its accessories are to be installed by an Agilent Customer Engineer.

1 Check that the Multimode Enablement Kit contains the following parts:

- Multimode Bd HV cable, p/n G1960-60858
- Multimode HV PCA, p/n G1960-61015
- Multimode Bd Power/Data Cable, p/n G1960-60873



Figure 1 From left to right: G1960-60858, G1960-61015 and G1960-60873

2 Install the APCI Enablement Kit, G1947-60451, which is shipped with the multimode source.

The APCI Enablement kit contains the following parts:

- Fast APCI HV Supply, p/n G1946-80058

Step 2. Install the HV control PCA and cables

- Valve BD-APCI supply, p/nG1960-60802
- Valve BD-APCI Needle Interlock, p/n G1960-60856



Figure 2 From left to right: G1946-80058, G1960-60802 and G1960-60856

Step 2. Install the HV control PCA and cables

- 1 Turn off the system power and remove the system power cord.

Keep the power cord intact if the vacuum control switch box is used. The switch box is intended to keep the vacuum on while a CE works on the electronics. The switch box is for CE use only.

- 2 Remove the CDS cover, top, side, front, and the Aux Module cover.
- 3 Disconnect the ribbon cable that connects the valve PCA to the Vcap/Vchamber power supply. Then disconnect the Vcap and Vchamber cable from the power supply.

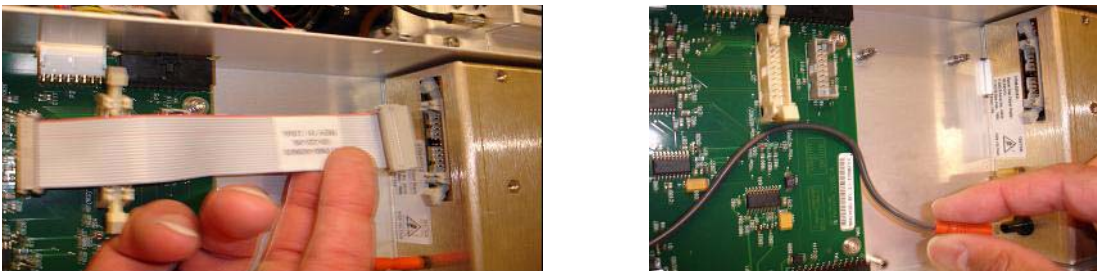


Figure 3 Disconnecting the Vcap/Vchamber power supply from the valve PCA (left) and the Vcap/Vchamber.

- 4 Place the multimode HV power supply PCA in the slot between the valve PCA and the Vcap/Vchamber. Secure the board by pressing it down into its slot and then attach it with two screws.
- 5 Connect the short gray cable from valve PCA to the multimode HV power supply.

1 Installation

Step 2. Install the HV control PCA and cables



Figure 4 Connecting the valve PCA to the multimode HV power supply.

- 6 Install the APCI HV power supply. The APCI HV power supply is located at the end of the AUX Module.
- 7 Connect ribbon cable between valve PCA and Vcap/Vchamber power supply.



Figure 5 Connecting the valve PCA to the Vcap/Vchamber power supply.

- 8 Connect the Vcap and Vchamber cables to the Vcap/Vchamber power supply.

Step 2. Install the HV control PCA and cables



Figure 6 Connecting the Vcap and Vchamber cables to the power supply.

- 9** Connect the long ribbon cable, p/n G1960-60802, from the APCI HV power supply to the valve PCA.



Figure 7 Connecting the APCI HV power supply to the valve PCA.

- 10** Insert one end of the APCI Needle Interlock cable, G1960-60856, through the slot at the front of the system and then plug it to the APCI HV connector. Attach the other end to the chassis with the o-ring and the nut.

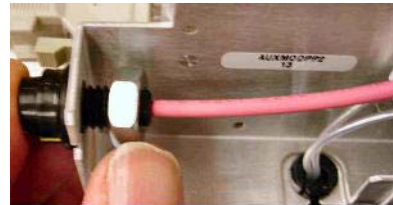


Figure 8 Connecting the APCI HV to the chassis.

- 11** Insert the cable, G1960-60858, to the top slot and attach it to the chassis. Plug the other two ends into the multimode HV PCA.

1 Installation

Step 2. Install the HV control PCA and cables



Figure 9 Connecting the HV PCA to the chassis.

12 Close the AUX Module cover and reconnect all cables.

13 Install the multimode source to the system and connect all connectors.

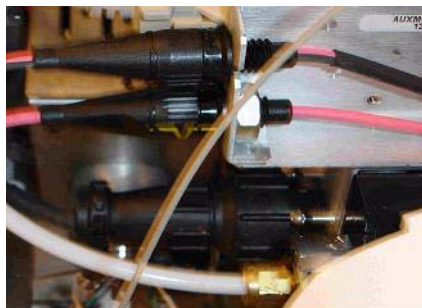


Figure 10 Installing the multimode source (left) and connecting all connectors.

14 Put back the side, top, front and CDS cover.

15 Plug the system power cord back on and turn the front switch on.

The pump down process will start.

16 Start the ChemStation program.

17 Click **Method and Run Control View > MSD > Spray Chamber** and verify that the source is an MM Source.

18 Go to MSD Tune window and start the Auto tune with a multimode source.

19 Verify that the multimode source is working correctly.

Changing Sources

To remove the multimode source

Do the following steps to remove the multimode source.

- 1 Set the source temperatures for vaporizer heater and drying gas heater 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 approximately 20 minutes or until the source is cool.
- 3 Open the CDS door at the front of the MS to access the cables.
- 4 Disconnect the ESI high voltage charging electrode cable.
- 5 Disconnect the APCI Needle Interlock and multimode HV cable.
- 6 Unscrew the nebulizer gas line from the nebulizer.
- 7 Unscrew the LC sample tubing from the nebulizer.
- 8 Open the latch on the source and open the source.
- 9 Remove the multimode source from the spray chamber mount.

1 Installation

To convert from multimode to ESI, APCI or APPI

10 Place the source shipping cover on the source.

To convert from multimode to ESI, APCI or APPI

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 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.
- 2 Unscrew and remove the multimode spray shield with the field shaping electrodes.
- 3 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.
- 4 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.
- 5 For all sources, reconnect the nebulizer gas line tubing and the LC/MS sample tubing.

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/MS 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 11](#).

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.

1 Installation

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

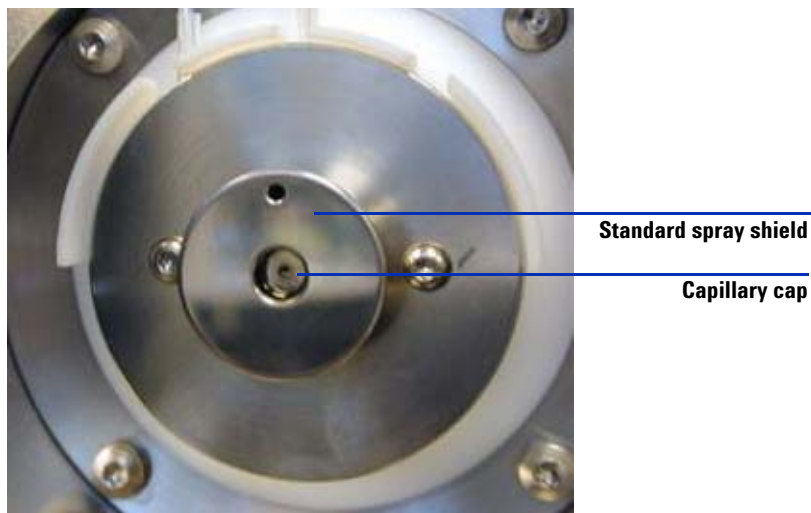


Figure 11 Standard spray shield and capillary cap for ESI or APCI

10 Remove the capillary cap. If needed, moisten a clean cloth with isopropyl alcohol and wipe the capillary cap. See [Figure 12](#).

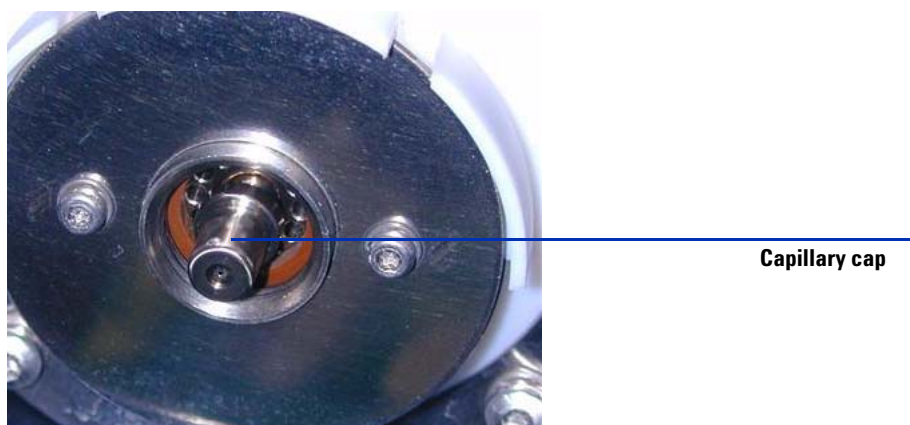


Figure 12 Spray shield removed.

11 Place the capillary cap back on the capillary.

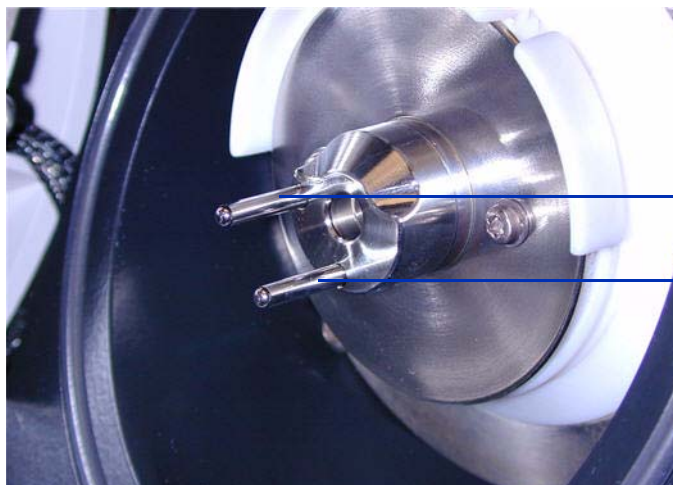
12 Install the new spray shield with field shaping electrodes. See [Figure 13](#).

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



Figure 13 Multimode spray shield

13 Screw the multimode spray shield into the holder for the spray shield. See [Figure 14](#).



Field shaping electrode
9 o'clock position

Field shaping electrode
6 o'clock position

Figure 14 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.

1 Installation

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



Figure 15 Multimode Spray Chamber

15 Install the spray chamber on the spray chamber mount.

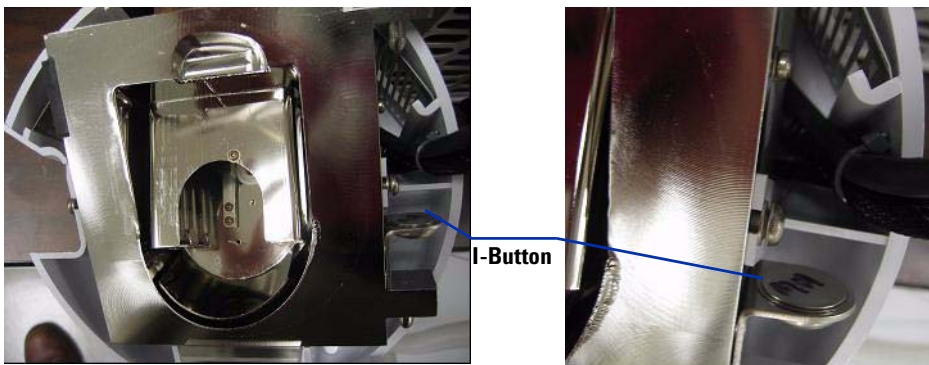


Figure 16 Multimode source with I-Button

16 Install the nebulizer on the multimode source spray chamber.

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



Figure 17 No nebulizer on top of the multimode source

17 Connect the 1/8-inch nebulizer gas tubing from the LC/MS mainframe to the nebulizer gas fitting. See [Figure 18](#).

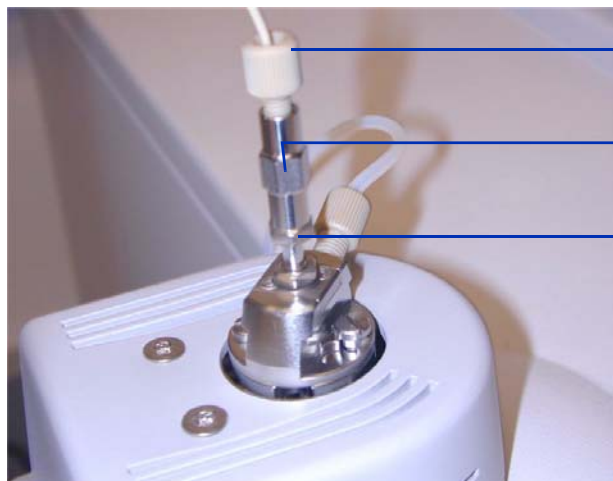


Figure 18 Nebulizer with gas tubing connected

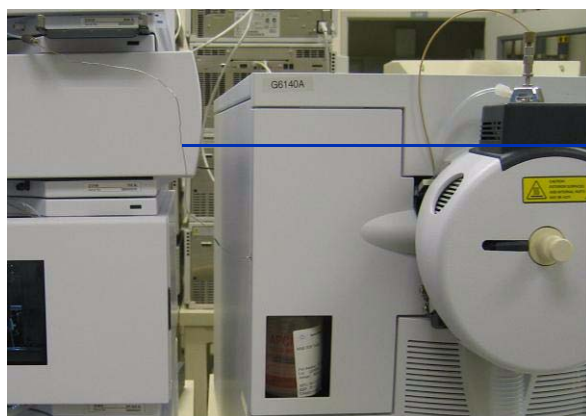
1 Installation

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

18 Connect the LC/MS sample tubing to the LC/MS diverter valve inlet filter.
See [Figure 19](#) on page 20.

WARNING

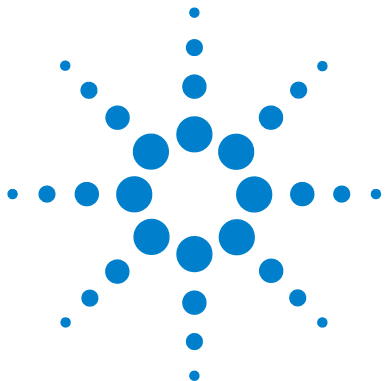
The LC/MS Liquid Chromatograph diverter valve is an integral part of the G1978B safety system. The LC mobile phase flow must always be connected to the diverter valve inlet filter. Never bypass the diverter valve and connect directly to the nebulizer. If the diverter valve is used in a manner not specified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



LC/MS sample tubing

Figure 19 LC/MS sample tubing connected to LC/MS inlet filter

19 If you are installing the multimode source for the first time, follow the steps in [“Step 2. Install the HV control PCA and cables”](#) on page 9.



2 Verification

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To verify sensitivity for 6130 and 6140 Quads - G1978B Multimode, Positive SIM Modes [24](#)

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To verify sensitivity for the G1978B Multimode using Multiple FIA method in Mixed Mode operation [30](#)

To do an autotune [32](#)

Example of multimode verification report [33](#)

This chapter contains instructions to verify your G1978B multimode a 6100 Series Single Quad LC/MS instrument.



2 Verification

To verify sensitivity for 6120 Quad - G1978B Multimode, Positive SIM Modes

To verify sensitivity for 6120 Quad - G1978B Multimode, Positive SIM Modes

NOTE

The G6110A instrument does not support the multimode source without the Selection valve. Use the option kit G2735H to enable the G6110A to work with the multimode source.

Preparation

- 1 Transfer 1 mL of 5 ng/ μ L 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 0.1% formic or acetic acid.
- 3 Transfer 1 mL of the first dilution to a second 50 mL volumetric flask. Use a clean graduated pipette.
- 4 Dilute to the 50 mL mark with 75:25 methanol/water with 0.1% formic or acetic acid.

This provides the final 2 pg/ μ L reserpine concentration required for performance verification.

- 5 Transfer approximately 1 mL of the second dilution to a vial for use in the LC autosampler.

Table 1 6110 and 6120 Quad Performance Verification Summary Table

	G1978B ES Positive SIM Mode	G1978B APCI Positive SIM Mode
Sample	Reserpine, 5 ng/ μ L	Reserpine, 5 ng/ μ L
Concentration after dilution	2 pg/ μ L	2 pg/ μ L
Injection volume	5 μ L	5 μ L
Total sample amount injected	10 pg	10 pg
Sample order number	G2423A	G2423A
Solvent	75:25 methanol/water with 0.1% formic or acetic acid	75:25 methanol/water with 0.1% formic or acetic acid

To verify sensitivity for 6120 Quad - G1978B Multimode, Positive SIM Modes

Table 1 6110 and 6120 Quad Performance Verification Summary Table

	G1978B ES Positive SIM Mode	G1978B APCI Positive SIM Mode
Method name	6110SIMES_MM.M (6110 Quad)	6110SIMCI_MM.M (6110 Quad)
	6120SIMES_MM.M (6120 Quad)	6120SIMCI_MM.M (6120 Quad)
Performance Specification	20: 1 pk-pk 100: 1 rms	10: 1 pk-pk 50: 1 rms

Sensitivity Verification for Multimode in ESI Positive SIM Mode

- 1 Start up the ChemStation software.
- 2 Change the view to **MSD Tune**, and start an Autotune.

After the autotune completes, you may need to wait up to 30 minutes to allow for the calibrant solution to be pumped out of the Quad instrument. This minimizes any background signal resulting from the calibrant.
- 3 Change the view to **Method and Run Control**, and load the method **6120SIMES_MM.M** (6120 model).
- 4 Edit the method to ensure that 75:25 methanol/water with 0.1% formic or acetic acid is selected as the LC solvent.
- 5 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/ μ L)
- 6 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.

You may need to further optimize the nebulizer pressure by running FIA experiments to achieve maximum instrument sensitivity.
- 7 Review the results.

2 Verification

To verify sensitivity for 6130 and 6140 Quads - G1978B Multimode, Positive SIM Modes

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. The blank-subtracted average signal-to-noise value must be greater than or equal to 20:1 peak-to-peak (100:1 RMS).

To verify sensitivity for 6130 and 6140 Quads - G1978B Multimode, Positive SIM Modes

Preparation

- 1 Transfer 1 mL of 5 ng/ μ L 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 1 pg/ μ L reserpine concentration required for performance verification.

- 5 Transfer approximately 1 mL of the second dilution to an autosampler vial.

Table 2 6130 and 6140 Quad Performance Verification Summary, SIM Mode

	G1978B ES Positive SIM Mode	G1978B APCI Positive SIM Mode
Sample	Reserpine, 5 ng/ μ L	Reserpine, 5 ng/ μ L
Concentration after dilution	1 pg/ μ L	1 pg/ μ L
Injection volume	1 μ L	1 μ L
Total sample amount injected	1 pg	1 pg
Sample order number	G2423A	G2423A

To verify sensitivity for 6130 and 6140 Quads - G1978B Multimode, Positive SIM Modes

Table 2 6130 and 6140 Quad Performance Verification Summary, SIM Mode

	G1978B ES Positive SIM Mode	G1978B APCI Positive SIM Mode
Solvent	75:25 methanol/water with 5mM ammonium formate	75:25 methanol/water with 5mM ammonium formate
Method name	6130SIMES_MM.M (6130 model)	6130SIMCI_MM.M (6140 model)
	6140SIMES_MM.M (6140 model)	6140SIMCI_MM.M (6140 model)
Performance Specification	20: 1 pk-pk 100: 1 rms	10: 1 pk-pk 50: 1 rms

Sensitivity Verification for Multimode in ESI, Positive SIM Mode

- 1 Start up the ChemStation software.
- 2 Change the view to **MSD Tune**, and start an Autotune.

After the autotune completes, you may need to wait up to 30 minutes to allow for the calibrant solution to be pumped out of the Quad instrument. This minimizes any background signal resulting from the calibrant.
- 3 Change the view to **Method and Run Control**, and load the method **6130SIMES_MM.M** (6130 model) or **6140SIMES_MM.M** (6140 model).
- 4 Edit the method to ensure that 75:25 methanol/water with 5mM ammonium formate is selected as the LC solvent.
- 5 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/ μ L)
- 6 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.

You may need to further optimize the nebulizer pressure by running FIA

2 Verification

To verify sensitivity for 6130 and 6140 Quads - G1978B Multimode, Positive SIM Modes

experiments to achieve maximum instrument sensitivity

7 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. The blank-subtracted average signal-to-noise value must be greater than or equal to 20:1 peak-to-peak (100:1 RMS).

Sensitivity Verification for Multimode in APCI, Positive SIM Mode

1 Start the ChemStation software.

2 Change the view to **MSD Tune**, and start an Autotune.

After the autotune completes, you may need to wait up to 30 minutes before you continue, to allow for the calibrant solution to be pumped out of the Quad instrument. This minimizes any background signal resulting from the calibrant.

3 Change the view to **Method and Run Control**, and load the method **6130SIMCI_MM.M** (6130 model) or **6140SIMCI_MM.M** (6140 model)

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

5 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/ μ L)

6 Run the method.

You may need to further optimize the nebulizer pressure by running FIA experiments to achieve maximum instrument sensitivity

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.

7 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. The blank-subtracted average signal-to-noise value must be greater than or equal to 10:1 peak-to-peak (50:1 RMS).

To verify sensitivity for 6130 and 6140 Quads - G1978B Multimode, Positive Scan Modes

Preparation

- 1 Transfer 1 mL of 5 ng/ μ L 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/ μ L reserpine concentration required for performance verification.

- 5 Transfer approximately 1 mL of the second dilution to an autosampler vial.

Table 3 6130 and 6140 Quad Performance Verification Summary, Scan Mode

	G1978B ES Positive Scan Mode	G1978B APCI Positive Scan Mode
Sample	Reserpine, 5 ng/ μ L	Reserpine, 5 ng/ μ L
Concentration after dilution	10 pg/ μ L	10 pg/ μ L
Injection volume	5 μ L	5 μ L
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

2 Verification

To verify sensitivity for 6130 and 6140 Quads - G1978B Multimode, Positive Scan Modes

Table 3 6130 and 6140 Quad Performance Verification Summary, Scan Mode

	G1978B ES Positive Scan Mode	G1978B APCI Positive Scan Mode
Method name	6130SCNES_MM.M (6130 Quad)	6130SCNCI_MM.M (6130 Quad)
	6140SCNES_MM.M (6140 Quad)	6140SCNCI_MM.M (6140 Quad)
Performance Specification	20: 1 pk-pk 100: 1 rms	10: 1 pk-pk 50: 1 rms

Sensitivity Verification for Multimode in ESI, Positive Scan Mode

- 1 Start the ChemStation software.
- 2 Change the view to **MSD Tune**, and start an Autotune.

After the autotune completes, you may need to wait up to 30 minutes to allow for the calibrant solution to be pumped out of the Quad instrument. This minimizes any background signal resulting from the calibrant.
- 3 Change the view to **Method and Run Control**, and load the method **6130SCNES_MM.M** (for 6130) or **6140SCNES_MM.M** (for 6140).
- 4 Edit the method to ensure that 75:25 methanol/water with 5mM ammonium formate is selected as the LC solvent.
- 5 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/ μ L)
- 6 Run the method.

You may need to further optimize the nebulizer pressure by running FIA experiments to achieve maximum instrument sensitivity.

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.
- 7 Review the results.

To verify sensitivity for 6130 and 6140 Quads - G1978B Multimode, Positive Scan Modes

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. The blank-subtracted average signal-to-noise value must be greater than or equal to 20:1 peak-to-peak (100:1 RMS).

Sensitivity Verification for Multimode in APCI, Positive Scan Mode

1 Start the ChemStation software.

2 Change the view to **MSD Tune**, and start an Autotune.

After the autotune completes, you may need to wait up to 30 minutes to allow for the calibrant solution to be pumped out of the Quad instrument. This minimizes any background signal resulting from the calibrant.

3 Change the view to **Method and Run Control**, and load the method **6130SCNCL_MM.M** (6130 model) or **6140SCNCL_MM.M** (6140 model).

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

5 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)

6 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.

You may need to further optimize the nebulizer pressure by running FIA experiments to achieve maximum instrument sensitivity

7 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. The blank-subtracted average signal-to-noise value must be greater than or equal to 10:1 peak-to-peak (50:1 RMS).

To verify sensitivity for the G1978B Multimode using Multiple FIA method in Mixed Mode operation

Preparation

- 1 Do an autotune if necessary.

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 Quad instrument. This minimizes any background signal from the calibrant.

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

- 2 Load the method **MMCheckSL_ES.M** and:
 - a Edit the method to ensure that 65:35 methanol/water with .2% acetic acid is selected as the LC solvent.
 - b Save the method.
- 3 Repeat for **MMCheckSL_CI.M** and **MMCheckSL_MX.M**.
- 4 Place the vial into the LC autosampler.
 - Position 21: vial with the ESI + APCI LC Demo Sample (p/n G1978-85000)

Create and run a Multiple FIA Method Sequence

- 1 Select **RunControl > Run Multiple FIA Methods**.
- 2 In the Run Multiple FIA Methods dialog box, click **Group > Add Group**.
- 3 Type in a unique directory name for the data files to be stored.
- 4 In the Run Multiple FIA Methods dialog box, click **Methods > Add Method**, select **MMCheckSL_ES.M** and click **OK**.
- 5 Repeat to add **MMCheckSL_CI.M**.
- 6 Repeat to add **MMCheckSL_MX.M**.
- 7 Click **Data File** for the **MMCheckSL_ES.M** data file, then:
 - a Change the **Subdirectory** value to a unique subdirectory name in which to store the data files.
 - b Change **Data File** to **Multi_ES**.
 - c Change **Operator** to the user's name or identification code.

To verify sensitivity for the G1978B Multimode using Multiple FIA method in Mixed Mode operation

- d Click **OK**.
- 8 Repeat for the **MMCheckSL_CI.M** data file. Use the same subdirectory and change **Data File** to `Multi_CI`.
- 9 Repeat for the **MMCheckSL_MX.M** data file. Use the same subdirectory and change **Data File** to `Multi_MX` as the data file name.
- 10 Click **Run** to start the sequence.
- 11 Review the results.

When the last method has finished, the report “Multimode Verification Report” prints.

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.

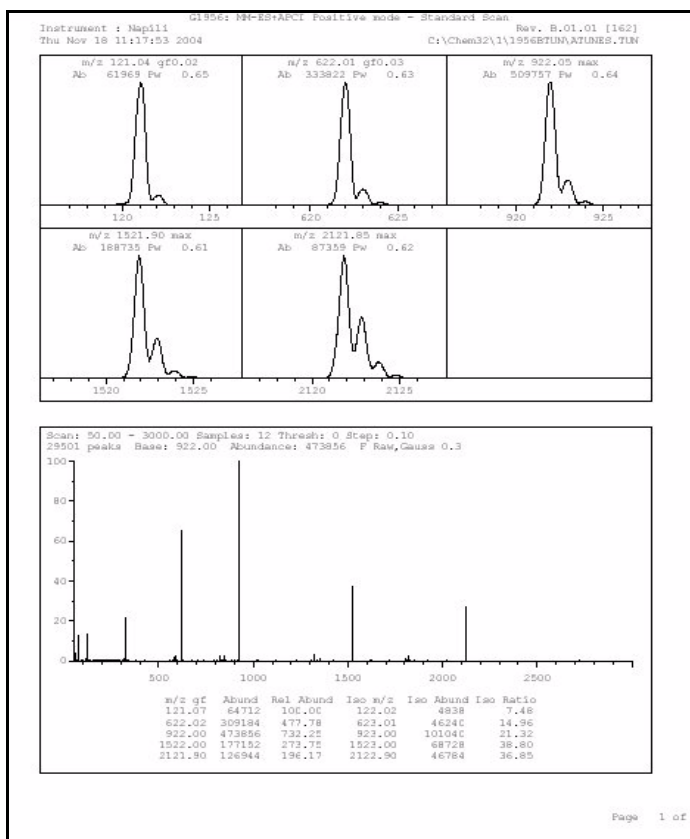


Figure 20 Autotune report

Example of multimode verification report

Multimode Ion Source Report

MSD type: G1956B Instrument name: Instrumen Operator name: pcorimia
 Acquisition date: 23-Feb-2005
 Datafiles:
 ESI mode : C:\Chem32\1\DATA\MMSTD_223\Multi_ESI.d
 APCI mode : C:\Chem32\1\DATA\MMSTD_223\Multi_CI.d
 Mixed mode : C:\Chem32\1\DATA\MMSTD_223\Multi_MX.d

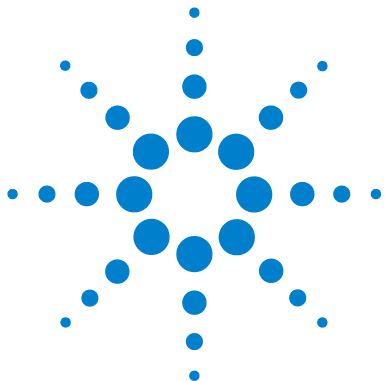
ESI Compound Results						
Compound	m/z	Polarity	ESI mode	Mixed mode	Mixed:ESI ratio	Result
Crystal violet	372.2	Positive	832925	541200	64.9 %	Pass
1-Hexanesulfonic acid	165.1	Negative	220506	181617	82.3 %	Pass

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.

2 Verification

Example of multimode verification report



3 Methods

- To setup a method to use the multimode source 36
- To create a method for positive/negative mixed mode operation 37
- To create a method for alternating ESI and APCI operation 39

This chapter describes the tasks that you need to set up methods for the multimode source. See “Guidelines” in the *Agilent G1978A/B Multimode Source Maintenance Guide* for suggested source conditions for the multimode source for the different ionization modes.



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 21](#).
- 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.

3 Methods

To create a method for positive/negative mixed mode operation

Polarity options for
Signal 1 and Signal 2

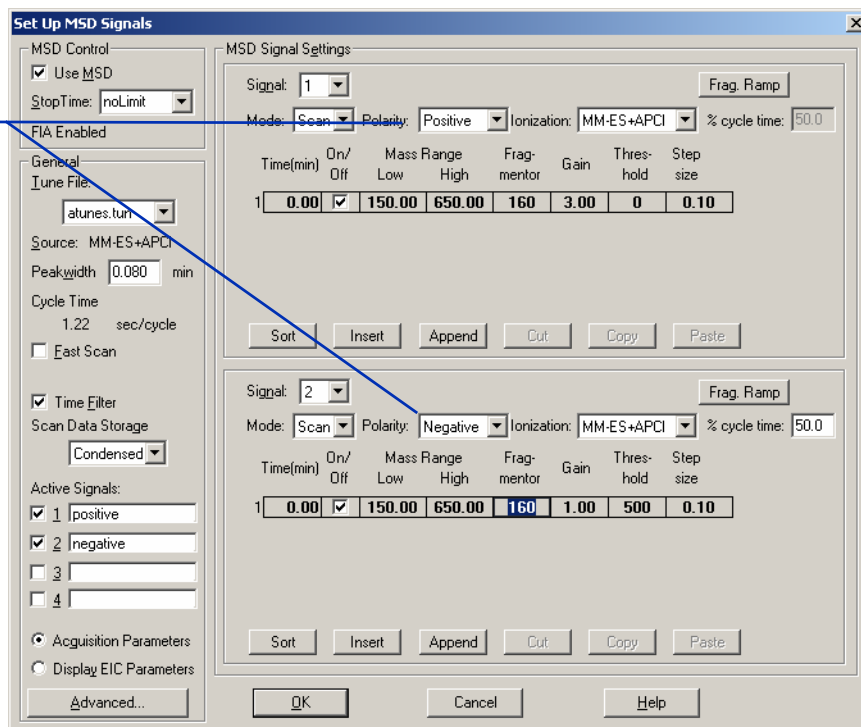


Figure 21 Positive/Negative polarity switching using Set Up MSD Signals dialog box

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**.

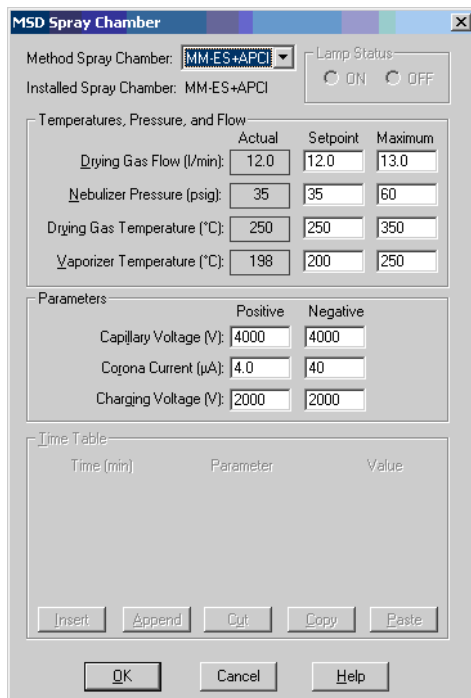


Figure 22 Method Spray Chamber set to MM-ES+APCI.

- 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 23](#).
- 8 Make any other changes that are necessary for your method.

3 Methods

To create a method for alternating ESI and APCI operation

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

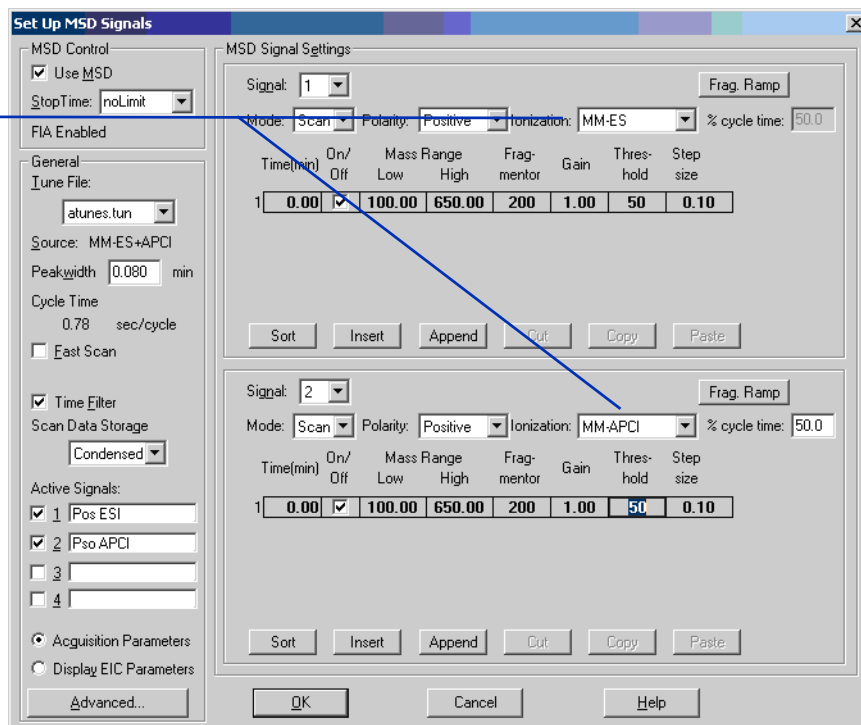


Figure 23 MM-ES and MM-APCI switching using Set Up MSD Signals dialog box

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In This Book

This book contains
installation, operation,
maintenance and
troubleshooting instruction
for the Multimode Source
for 6100 Series Single Quad
LC/MS.

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