Agilent G1978B Multimode Source for 6410 Triple Quad LC/MS

User Guide

Agilent Technologies

Notices

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Safety Notices

CAUTION

A **CAUTION** 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 damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

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 multimode ion source.

1 Basic Operation and Maintenance

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

2 Installation

This chapter tells you how to install the multimode source.

3 Reference

This chapter contains an overview of the multimode source, safety precautions, and technical specifications.

Content

1
Basic Operation and Maintenance

This chapter describes the tasks that you need to operate and maintain the multimode source.

To set up a method to use the multimode source

1. In the MassHunter software, change the **Context** to **Acquisition**.
2. In the MS QQQ tab, set **Ion source** to **MMI** (see Figure 1 on page 9).
3. In the **Time Segments** table, chose an ionization mode from the **Ion Mode** list. You may set the ionization mode to one of the following:
   - ESI
   - APCI
   - ESI+APCI

   The Ion Mode selection ESI + APCI will specify a method for alternating ESI and APCI operation.

   Note that the Ion Mode selection is only visible if **Ion source** is set to **MMI**.
4. In the **Source** tab, set the desired source conditions. See the "Guidelines" on page 51 for suggested source conditions for the multimode source for the different ionization modes.
5. Make any other changes that are necessary for your method.
6. Save the method.

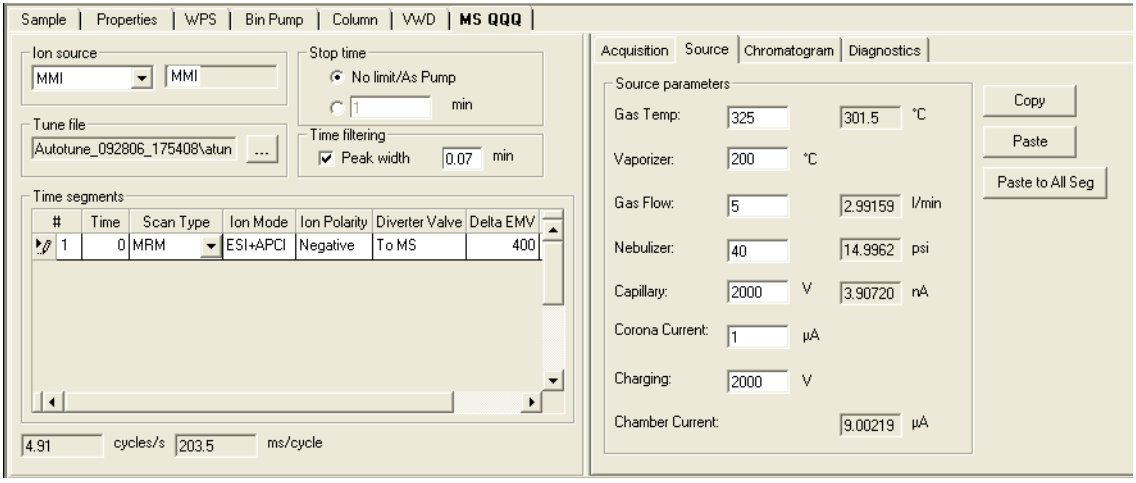


Figure 1 Multimode acquisition settings

WARNING **The LC/MS 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 open the multimode source

Open the multimode source to access the end cap and the capillary cap for cleaning and inspection.

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.**

1. Turn off the multimode source temperatures and flows:
   a. Change the **Context** view to **Acquisition**.
   b. Click the **MS QQQ** tab.
   c. Turn off all voltages and temperatures in the **Source** tab.
   d. Wait approximately 20 minutes for the source to cool down.
2. Open the spray chamber cover by pulling the latch.

   The high voltage automatically turns off when the chamber door is opened so that no high voltages are present within the chamber.
3. Check that the vaporizer temperature sensor is straight and extends 15mm from back of chamber.
4. Check that the separator is aligned vertically.
5. Check that the APCI corona needle is in and extends approximately 3mm from the corona guide.
6. Check that the source is clean.

To clean the multimode source daily

You should clean the multimode source daily or any time you suspect carryover (contamination) from one sample or analysis to another. The multimode source spray chamber is made of 316 stainless steel which is the same material used to make the spray shield.

Before you begin, check that you have:

- Abrasive paper, 8000 grit (p/n 8660-0852)
- Cloths, clean, lint-free (p/n 05980-60051)
- Cotton swabs (p/n 5080-5400)
- Gloves
- Mobile phase from the current method or clean isopropanol, reagent grade or better
- Wash bottle
- Water, reagent-grade or better

1 Turn off the spray chamber.

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

2 Remover the nebulizer and APCI corona needle.

3 Remove the cosmetic cover. The thermocouple probe will need to be removed if any wiping of the spray chamber will be done. Then, open the spray chamber.

4 Rinse the interior of the spray chamber using the wash bottle filled with the current mobile phase or with a mixture of isopropanol and water.

NOTE Recent residue should be soluble in the mobile phase. If you are not sure what mobile phase was used recently, a mixture of 50% isopropanol and 50% water works well as a general cleaning solution.

WARNING **Some mobile phases are hazardous chemicals. Use caution that is appropriate for the current mobile phase.**

5 Wipe the interior of the spray chamber with a clean, lint-free cloth.

WARNING **There are sharp edges inside the spray chamber such as the separator. Pay close attention when wiping the interior of the spray chamber.**

6 Rinse the area around the spray shield. Do not spray directly at the end of the capillary. This can cause pressure surges in the vacuum system.

7 Dampen a clean cloth with the mobile phase. Wipe the spray shield, field shaping electrodes and the area around the spray shield.

8 Replace the nebulizer and the APCI corona needle.

9 Install the thermocouple probe and adjust it so that it protrudes 15 mm from the inner spray chamber wall. See Figure 2.

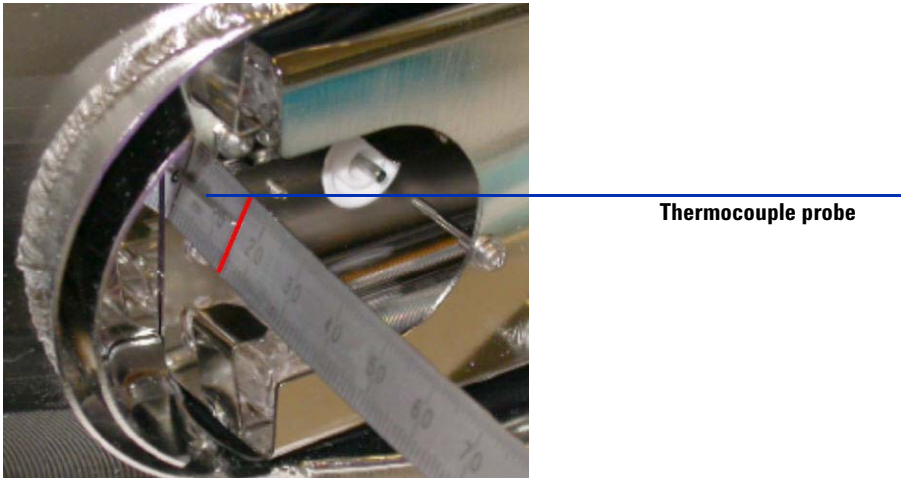


Figure 2 Installing the thermocouple probe and 15 mm adjustment

10 Replace the cosmetic cover.

11 Close the spray chamber.

NOTE If symptoms of contamination persist, or if the spray shield or capillary cap show significant discoloration that cannot be removed by the regular, daily cleaning, use the weekly cleaning procedure.

To clean the multimode source weekly

The cleaning procedure for cleaning the multimode source weekly is similar to the procedure for cleaning the source daily. The main difference is that the multimode source is removed from the instrument.

Before you begin, check that you have:

- Abrasive paper, 8000 grit (p/n 8660-0852)
- Cloths, clean, lint-free (p/n 05980-60051)
- Cotton swabs (p/n 5080-5400)
- Gloves
- Mobile phase from the current method, or clean isopropanol, reagent grade or better
- Wash bottle
- Water, reagent-grade or better

1 Do the steps in "To remove the multimode source" on page 21.

2 Fill the spray chamber with clean mobile phase, or with a mixture of isopropanol and water.

NOTE Recent residue should be soluble in the mobile phase. If you are not sure what mobile phase was used recently, a mixture of 50% isopropanol and 50% water works well as a general cleaning solution.

WARNING **Some mobile phases are hazardous chemicals. Use caution that is appropriate for the current mobile phase.**

3 Scrub the corona insulator and the interior of the spray chamber with a clean cotton swab.

4 Empty the spray chamber.

5 Wipe the interior of the spray chamber with a clean, lint-free cloth.

WARNING **The inside of the spray chamber has sharp edges, such as on the separator. Pay close attention when you wipe the interior of the spray chamber.**

6 Remove the spray shield. Use abrasive paper to gently clean the end of the capillary cap.

7 Dampen a clean cloth and wipe the end of the capillary cap.

8 Reinstall the spray shield.

9 Use abrasive paper to gently clean the spray shield. Dampen a clean cloth and wipe the spray shield.

10 Rinse the area around the spray shield. Then wipe the area around the spray shield.

11 Reinstall the spray chamber on the instrument.

12 Replace the nebulizer and APCI corona needle.

13 Install the thermocouple probe and adjust it so that it protrudes 15 mm from the inner spray chamber wall. See Figure 2 on page 12.

14 Replace the cosmetic cover.

15 Close the spray chamber.

To autotune with the multimode source

1 Remove the G1948B Electrospray source and install the G1978B multimode source.

2 Pour the Electrospray or APCI calibrant back into its original bottle or another suitable container, rinse the calibrant bottle with acetonitrile, pour the MMI-L Low Concentration Tuning Mix (G1969-85020) calibrant into the calibrant bottle, and attach the calibrant bottle back onto the CDS.

3 Set the **Context** view to **Tune** in the MassHunter Workstation program.

4 Load an autotune file that was generated with the G1948B Electrospray source.

5 Run an Autotune using the G1978B multimode source.

2
Installation

This chapter contains instructions to install the multimode source on a 6400 Series Triple Quad LC/MS system, and also to remove and replace the source.

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 3 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
- Valve BD-APCI Supply Cable, p/nG1960-60802
- Valve BD-APCI Needle Interlock Cable, p/n G1960-60856

Figure 4 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.

The power cord should be kept intact if the vacuum control switch box is used. The switch box is intended to keep the vacuum on while a service engineer works on the electronics. The switch box is for service engineer 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.

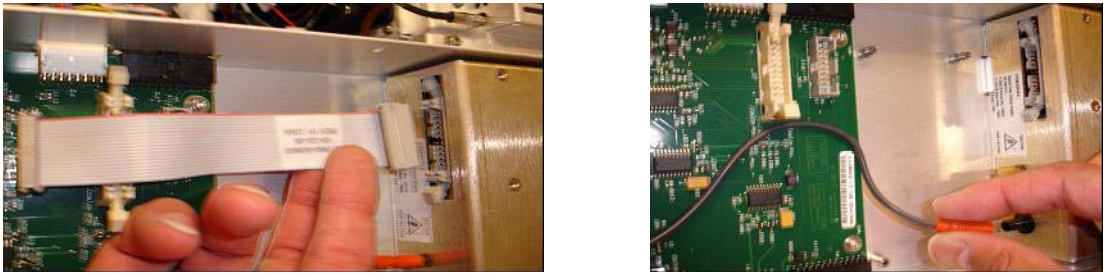


Figure 5 Disconnecting the Vcap/Vchamber power supply from the valve PCA (left) and the Vcap/Vchamber.

4 Place the MM HV power supply PCA in the slot between the valve PCA and the Vcap/Vchamber power supply. Secure the board by pressing it down into its slot and then attach it with two screws.

5 Connect the short gray cable from the valve PCA to the multimode HV power supply.

Figure 6 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 the valve PCA and Vcap/Vchamber power supply.

Figure 7 Connecting the valve PCA to the Vcap/Vchamber power supply.

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

Figure 8 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 9 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 (see Figure 10).

Figure 10 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.

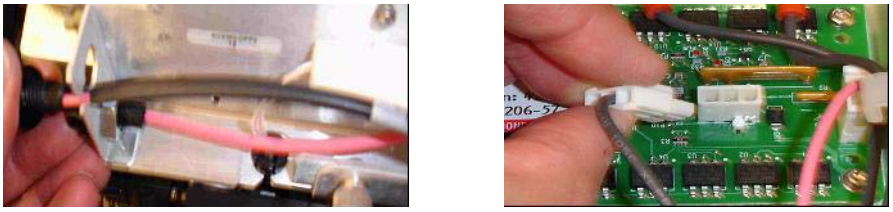


Figure 11 Connecting the HV PCA to the chassis.

12 Close the AUX Module cover and reconnect all cables.

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

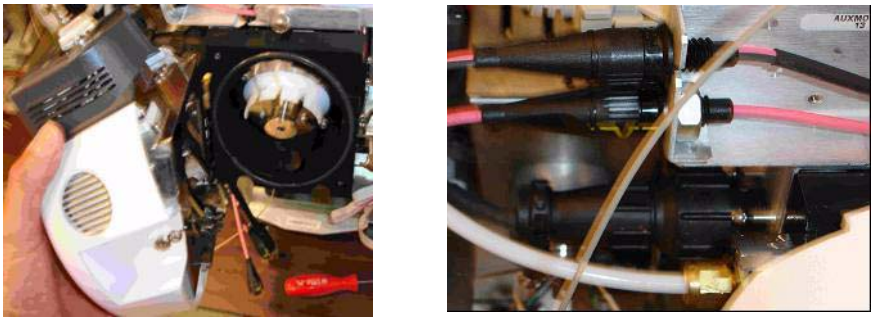


Figure 12 Installing the multimode source (left) and connecting all connectors.

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 MassHunter Workstation program and verify that the software recognizes the source.

17 Set the **Context** view to **Tune**, and in **Manual Tune**, verify that the system can generate the proper tune peaks.

To remove the multimode source

Do the following steps to remove the multimode source.

1 Turn off the multimode source temperatures and flows:

   a Change the **Context** view to **Acquisition**.

   b Click the **MS QQQ** tab.

   c Turn off all voltages and temperatures in the **Source** tab.

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.

10 Place the source shipping cover on the source.

To convert from multimode to ESI or APCI

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. Unscrew and remove the multimode spray shield with the field shaping electrodes.
2. 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.
3. For an APCI ion source, connect the vaporizer heater cable and the APCI high voltage cable.
4. For all sources, reconnect the nebulizer gas line tubing and the LC/MS sample tubing.

To convert from ESI or APCI 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 15.

1. Turn off the multimode source temperatures and flows:
   a. Change the **Context** view to **Acquisition**.
   b. Click the **MS QQQ** tab.
   c. Turn off all voltages and temperatures in the **Source** tab.
2. Wait for the source to cool (until temperatures are at least below 100°C).
3. Disconnect the nebulizer gas tubing from the currently installed ion source.
4. Disconnect the LC/MS sample inlet tubing.
5. If the APCI source is installed, remove the APCI vaporizer heater cable and APCI high voltage cable.
6. Remove the currently installed ion source.
7. Unscrew and remove the spray shield. See Figure 13.

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.**

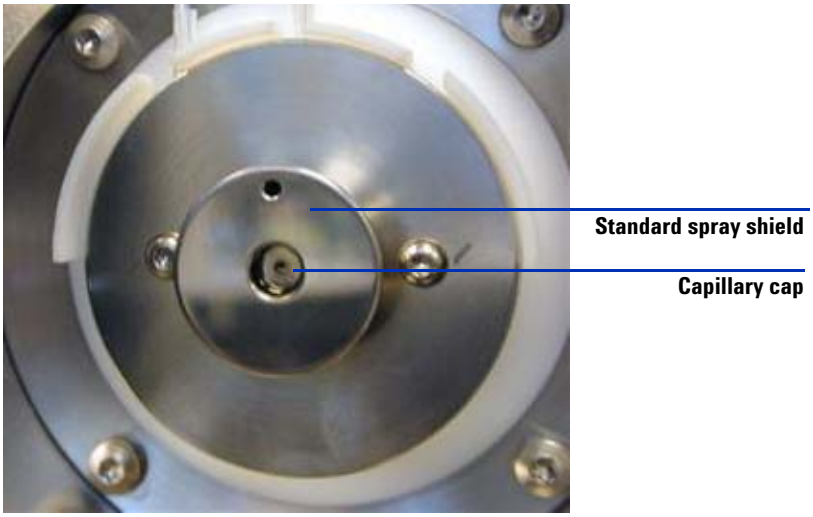


Figure 13 Standard spray shield and capillary cap for ESI or APCI

8 Remove the capillary cap. If needed, moisten a clean cloth with isopropyl alcohol and wipe the capillary cap. See Figure 14.

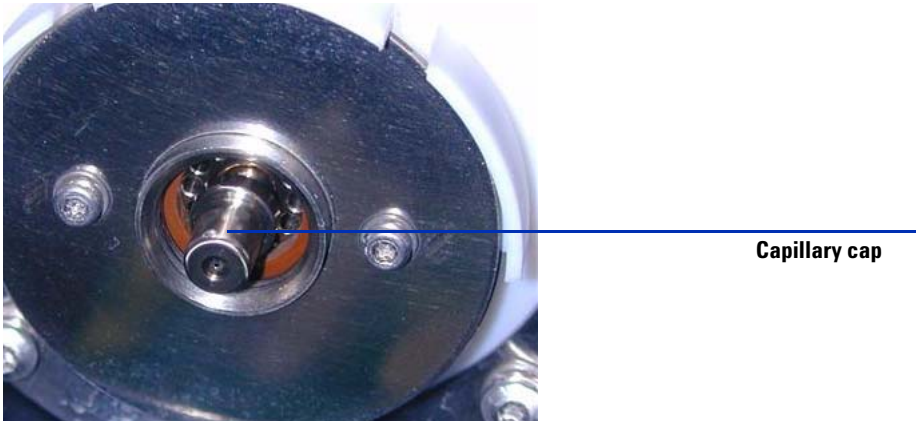


Figure 14 Spray shield removed.

9 Place the capillary cap back on the capillary.

10 Install the new spray shield with field shaping electrodes. See Figure 15.

Figure 15 Multimode spray shield

11 Screw the multimode spray shield into the holder for the spray shield. See Figure 16.

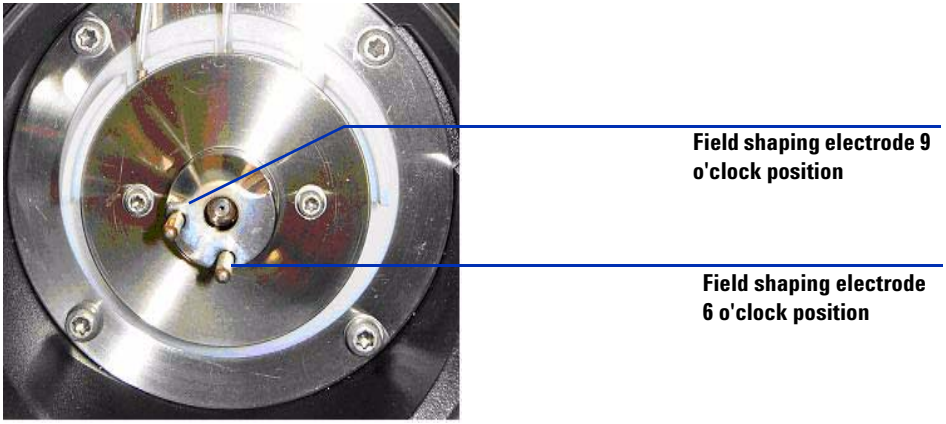


Figure 16 Multimode spray shield installed

NOTE

The field shaping electrodes should be in the nine o'clock and the six o'clock positions. Failure to place the field shaping electrodes in the correct positions will result in greatly reduced response by the multimode source. Loosen the end plate screws on each side to adjust the field shaping electrodes position.

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

Figure 17 Multimode Spray Chamber

13 Install the spray chamber on the spray chamber mount.

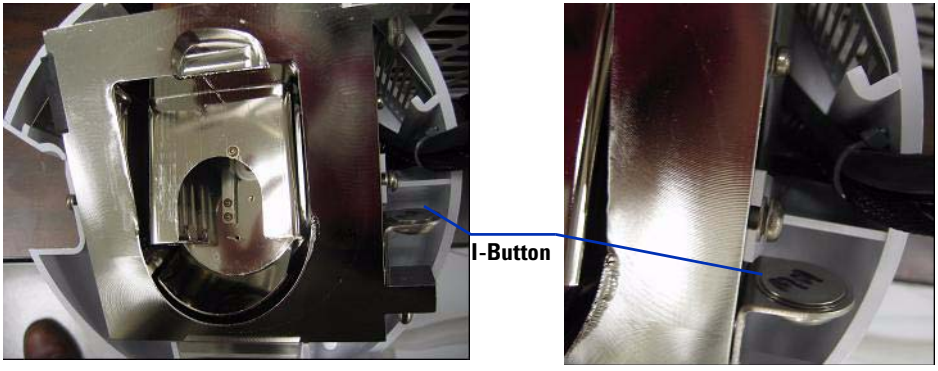


Figure 18 Multimode source with I-Button

14 Install the nebulizer on the multimode source spray chamber.

Figure 19 No nebulizer on top of the multimode source

15 Connect the 1/8-inch nebulizer gas tubing from the LC/MS mainframe to the nebulizer gas fitting. See Figure 20.

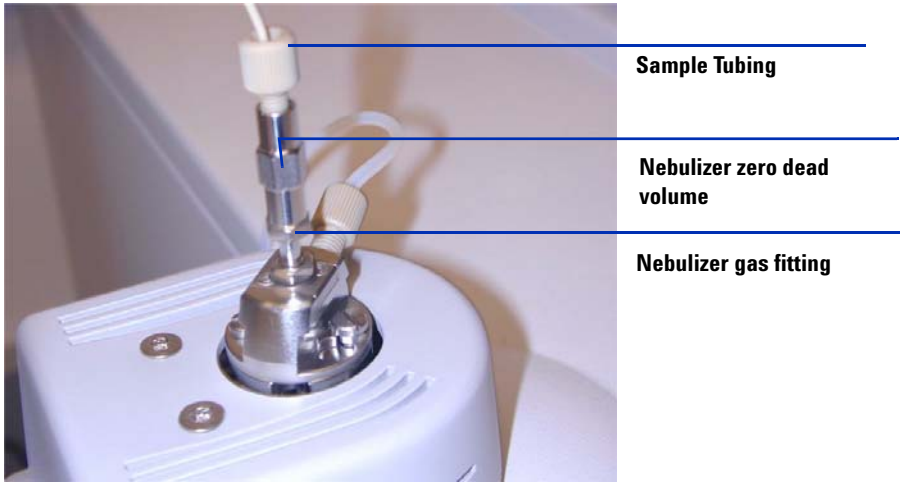


Figure 20 Nebulizer with gas tubing connected

16 Connect the LC/MS sample tubing to the LC/MS diverter valve inlet filter.

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.**

17 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 17.

3 Reference

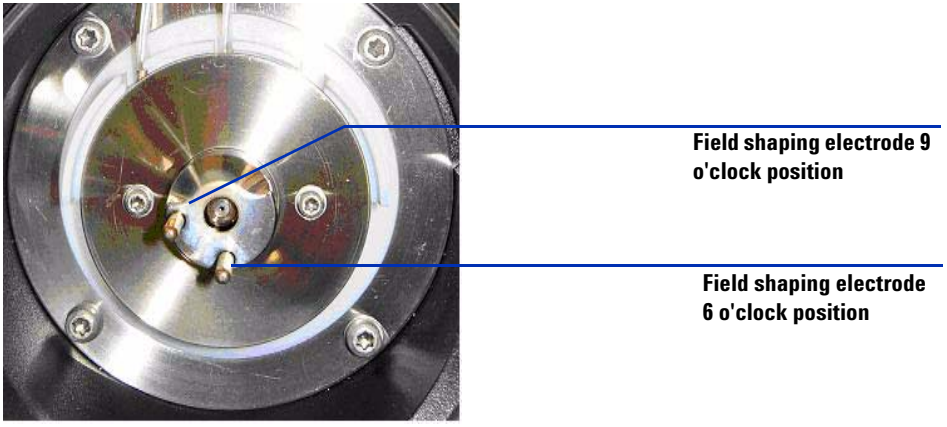
This chapter contains reference information to help you run your 6400 Series Triple Quad LC/MS with a multimode source.

Tips for Successful Operation

The following tips are helpful to set up a multimode source for operation.

Check the position of the field shaping electrodes

As specified on page 25, the field shaping electrodes on the multimode's spray shield should be at the 6 o'clock and 9 o'clock positions. If these field shaping electrodes are in an incorrect position, you may see reduced response and poor peak shape.



Check the position of the nebulizer needle

Use the longer ES Nebulizer Assembly (P/N G1946-60098) on the multimode source, and not the APCI/APPI Nebulizer Assembly. Check that the Nebulizer Needle (P/N G1946-20177) is flush with the end of the Nebulizer Body. If the Nebulizer Needle is withdrawn slightly during tuning, the calibrant flow (at 13 psi) can be restricted by the nebulizer gas flow (at 30 psi). The erratic spray and “sputtering” that may result can affect the quality of peaks.

To adjust the nebulizer position, see the video and procedure on the *Agilent 1100 Series HPLC Maintenance & Repair* CD-ROM (P/N 01100-60008).

Clean out old calibrant used by other sources

Calibrant used by other sources can cause interference problems if it is not properly cleaned out before tuning with the multimode source. For example, betaine (trimethylglycine) is the first positive mode peak (m/z 118) for the ES, ES-L Low Concentration, and ES-Trap Tuning Mixes.

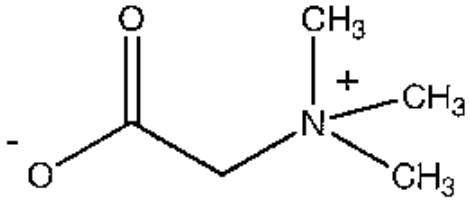


Figure 21 Betaine

If a system contains substantial amounts of betaine, and you convert to the MMI-L Tuning Mix, you may have problems. The first positive mode peak is m/z 121, so there may be interference with 118. When tuning the instrument with the Widest resolution setting (e.g. FWHM is equal to 2.5 amu) the 121 peak may appear unstable and split.

Because betaine is rather polar, it can be cleaned out using deionized water or a 50:50 water:acetonitrile mixture. Use the procedures on the *Agilent 1100 Series HPLC Maintenance & Repair* CD-ROM to remove the spray chamber and wash it out three times. Wipe down the spray shield, making sure you do not get solvent into the capillary. Finally, to clean out the tubing from the selection valve to the nebulizer and the capillary, lower the drying gas to 15 psi and 3 L/min and pump solvent at 1 mL/min for one half-hour through the nebulizer into the spray chamber.

Safety and Specifications

This section describes safety, safety symbols, and technical specifications.

Safety

Some of the procedures in this chapter require access to parts of the instrument and multimode source while it is in Shutdown state or shortly after it is turned off. If you do not perform these procedures correctly, you are exposed to dangerous temperatures, voltages, and chemical hazards. This topic describes the potential dangers.

Nebulizer Needle Hazard

- The nebulizer needle tip is very fragile. Do not touch the tip to any objects, such as the capillary cap or spray chamber. If you accidentally touch the nebulizer needle tip, replace the needle.
- The vaporizer temperature sensor is very sharp and can pierce your skin. Do not touch the tip, especially when you analyze toxic substances or when you use toxic solvents.
- Use care when you adjust the nebulizer needle. Do not damage the end of the needle.

High temperatures

Most parts in the multimode source operate at or reach temperatures high enough to cause serious burns. These parts include, but are not limited to the capillary, capillary cap, spray shield, vaporizer temperature sensor, APCI corona needle, counter electrode, source hinge and spray chamber.

Do not touch these parts.

- Certain parts remain hot for many minutes after the instrument is shut down or turned off. In particular the spray shield and the capillary cap could be very hot after working with APCI, ESI or MM-ES+APCI. Use extreme care when you work on an instrument that has recently been turned off.

- The Infrared medium-wave twin tube emitters are made of quartz glass. The IR radiation is not hazardous and is radiated as heat.
- Do not touch any surfaces in the source spray chamber. The spray chamber in most cases will be very hot

Hazardous voltages

Whenever the instrument is not in Standby, hazardous voltages are present on one or more interior parts. Parts that use hazardous voltages include, but are not limited to, the capillary cap and counter electrode.

These parts are usually covered or shielded. As long as the covers and shields are in place, you will not make contact with hazardous voltages.

- Never open the multimode spray chamber while the instrument is in the operation or the HV voltages are turned on.
- Do not insert fingers or tools through the openings on the multimode spray chamber. During operation the capillary and capillary cap are at high voltage up to 4 kV.
- If you connect the instrument to an ungrounded or improperly grounded power source, you create a shock hazard for the operator and can damage the instrument. Intentional interruption of instrument grounding is strictly prohibited.

Biohazardous residue

The multimode interface does not ionize all of the sample and solvent. The vacuum pumps of the instrument remove the sample that is not ionized and solvent. The exhaust from these pumps can contain traces of sample and solvents. Vent all pump exhaust outside the building or into a fume hood. Comply with your local air quality regulations

- The exhaust fumes from the vacuum system and spray chamber contains trace amounts of the chemicals you analyze. Health hazards include chemical toxicity of solvents, samples, buffers, and pump fluid vapor, as well as potentially biohazardous aerosols of biological samples. Vent all exhausts out of the building where they cannot be recirculated by environmental control systems. Do not vent exhausts into your laboratory. Comply with your local air quality regulations.

- Fluid drained from the multimode chamber is made of solvent and sample from your analyses. The fluid in the mechanical pump collects traces of samples and solvents. In addition, unnebulized solvent and sample collects at the bottom of the spray chamber. Connect the drain on the bottom of the spray chamber to a closed container.
- Handle and dispose of all fluids using precautions appropriate for their biohazardous and biological content. Comply with local environmental regulations.
- Handle all used pump fluid as hazardous waste. Dispose of used pump fluid as specified by your local regulations.

Environmental Conditions

This equipment must be installed in an environment of Category II installation as defined in IEC 664. Check that the supply voltage does not fluctuate more than +10% or -10% of rated voltage.

Equipment Class Class 1 Laboratory Equipment

Pollution Degree 2

Installation Category II

Environment Indoor Use

Altitude Not to exceed 2300 m

Electrical supply 100 - 240 V AC, 50/60 Hz, 1.2 A

Mains supply voltage Fluctuations not to exceed 10% of nominal supply voltage

Operating Temperature 15 to 35°C (59 to 95°F)

Humidity < 90% RH at 35°C

Operational Conditions

If the G1978B is used in a manner not specified by Agilent Technologies, the protections provided by the G1978B may be impaired.

Cleanliness

Cleanliness and the prevention of accidental contamination during maintenance are very important. Contamination of the interior of the vacuum system or the sample path can affect the results of your analyses.

- Always wear clean gloves when handling parts that come in contact with the sample path. Oil from your fingers is difficult to remove.
- When you set parts down, place them on clean, lint free cloths or clean aluminum foil, not directly on the laboratory bench.
- Keep parts covered so they do not get dirty.
- If possible, maintain a separate set of tools that have been thoroughly cleaned. Use these tools only when working on clean assemblies.
- With open ion sources, such as API, avoid dusty and fibrous environments. Dust particles can enter MS ion source and deposit on ion optics, causing sensitivity loss.

Safety Symbols

NOTE

This symbol is placed on the product where it is necessary for you to refer to the **manual** in order to understand a **hazar**d.

WARNING CAUTION

This symbol is placed on the product within the area where **hazardous voltage** is present or shock hazard can occur. Only trained service persons should perform work in this area.

WARNING

This symbol is placed on the product within the area where **hot parts and surfaces** are present. Allow the product to cool before performing work in this area.

WARNING

This symbol is placed on the product within the area where **biohazards** are present. Handle these areas with the respective care.

Technical Specifications

Size

Height	Length	Width
17 cm	18 cm	9.5 cm
6.8 in	7.1 in	3.7 in

Weight 2.27 kg / 5 lbs.

Power source Power 60 VA

Primary

Voltage	100 V to 240 V
Current	0.85 A to 0.40 A
Frequency	50 Hz / 60 Hz

Secondary

Voltage	12 V
Current	3.3 A

Operating Temperature 15°C to 35°C (59°F to 95°F) (Analytical specifications will be met only within a temperature range of 21°C ± 3°C (70°F ± 6°F).

Operating Humidity 15% to 95% (non condensing at 35°C)

Operating Altitude < 2300 meters / < 7500 feet

Overview

Benefits of a multimode source

Figure 22 Multimode source

The multimode source is an ion source for LC/MS that can operate as an APCI, ESI or simultaneous APCI/ESI source. See Figure 22. The source is intended to operate at normal chromatographic flow rates 50 to 2000 µL /min. The multimode source can be run in three different modes of operation (MM-ES, MM-APCI or MM-ES+APCI).

Why use the multimode source?

- A single ion source with a single nebulizer performing MM-ES, MM-APCI, or MM-ES+APCI
- MM-ES to MM-APCI without source exchange
- Operates in MM-ES only, MM-APCI only, or MM-ES+APCI mixed mode
- Operates at typical HPLC flow rates up to 2 mL/min, even in MM-ES mode
- Multimode source provides higher throughput for data requiring validation in both MM-ES and MM-APCI
- More universal response

- Permits analysis of fast-eluting peaks
- Operation at flow rates used for high-throughput analysis without splitting
- Operation at lower flow rates for higher-sensitivity analysis

The design uses a single nebulizer through which the LC effluent flows. You do not need to switch back and forth between APCI and ESI modes. While this option will be available, the great value of the source is its ability to produce APCI and ESI generated ions simultaneously. This ability increases the effective duty cycle of the mass spectrometer. Another key feature is the much greater operational flow rate that is available in this design.

Ionization Mode	Polarity
APCI	positive
APCI	negative
ESI	positive
ESI	negative
Multimode	positive
Multimode	negative

Mixed mode operation is generally a balance between optimal ESI and APCI conditions. Changing the vaporizer temperature, the nebulizer pressure, and the corona current alters the balance between the ionization modes.

Parameter adjustment	APCI response in mixed mode generally	ESI response in mixed mode generally
Higher vaporizer temperature	Increases	Stays the same
Higher nebulizer pressure	Decreases	Increases
Higher corona current	Increases	Decreases

Multimode source operational description

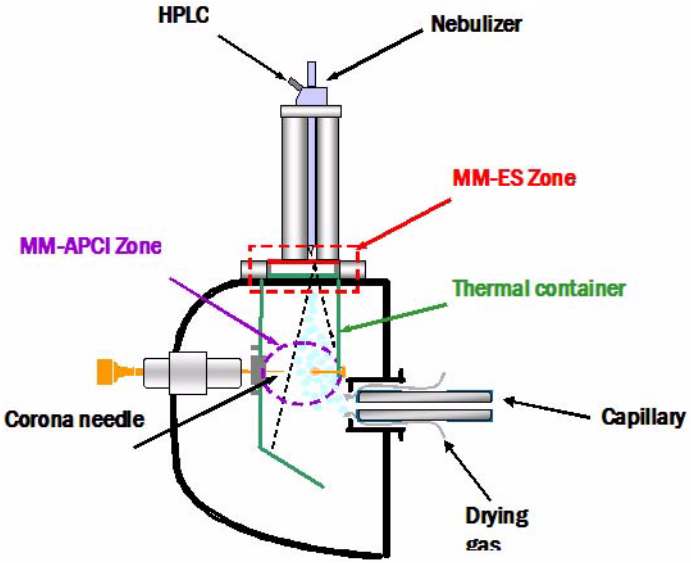


Figure 23 Graphical representation of multimode source

1. Liquid enters the grounded nebulizer
2. A charged aerosol is made in the ESI Zone
3. The aerosol is dried by IR lamps
4. Neutral analytes and ESI charged analytes pass through the APCI Zone
5. ESI and APCI ions enter the capillary

MM-ES Zone

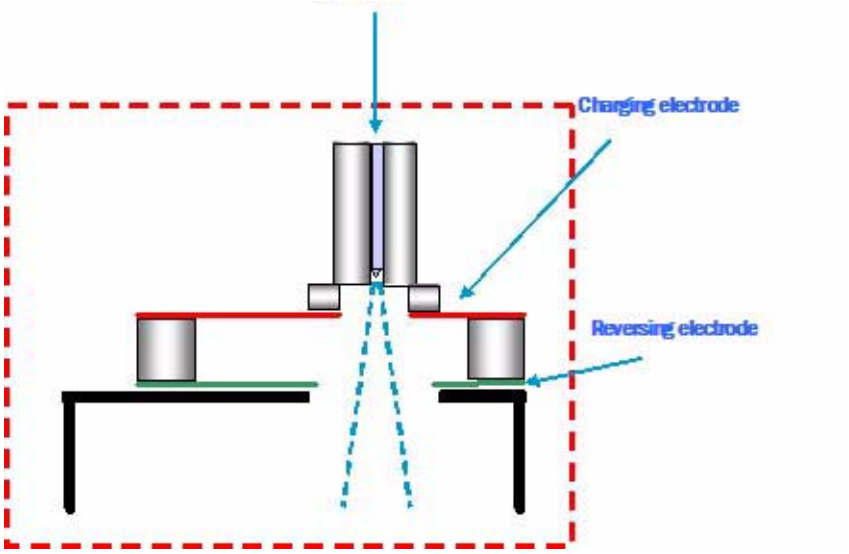


Figure 24 Graphical representation of MM-ES zone

1. Liquid enters the grounded nebulizer.
2. The charging electrode charges the liquid
3. Nebulizing gas pushes the charged aerosol past the charging and reversing electrodes
4. The reversing electrode separates the ESI formation from the APCI formation

Infrared Drying

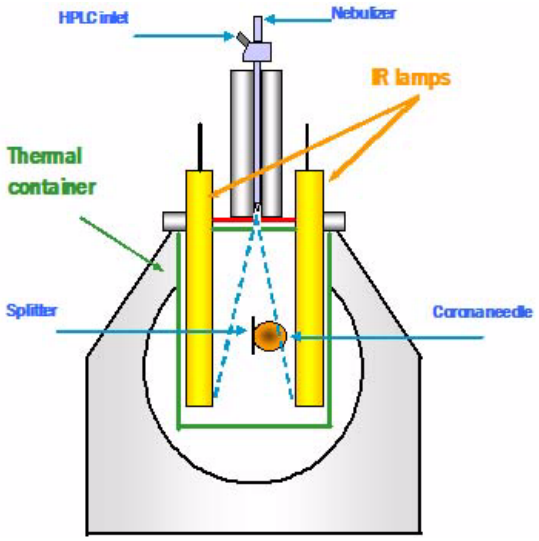


Figure 25 Graphical representation of infrared drying

1. The charged aerosol passes the reversing electrode and enters the thermal container.
2. The thermal container confines the heat and aerosol
3. Infrared lamps dry the charged aerosol on the way to the APCI Zone

MM-APCI Zone

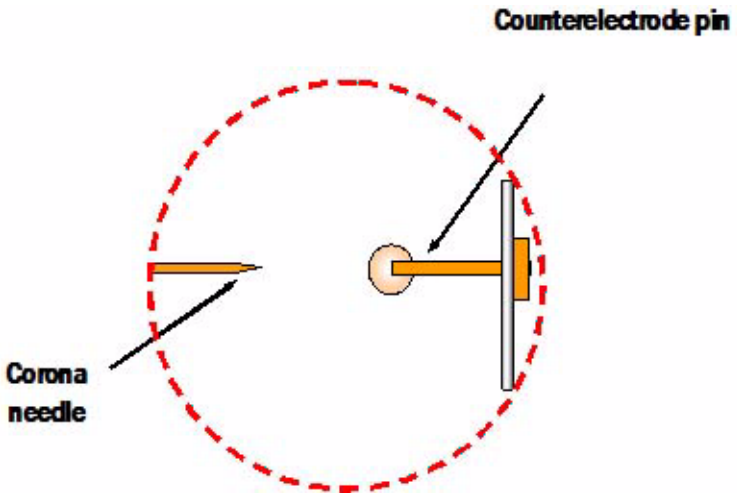


Figure 26 Graphical representation of MM-APCI zone

1. Neutral analytes and MM-ES charged analytes pass through the MM-APCI Zone.
2. A corona is formed between the APCI corona needle and the pin.
3. Neutral analytes in or very near this gap are ionized.
4. MM-ES and MM-APCI ions are merged and follow the electric field into the mass spectrometer.

Multimode source side view

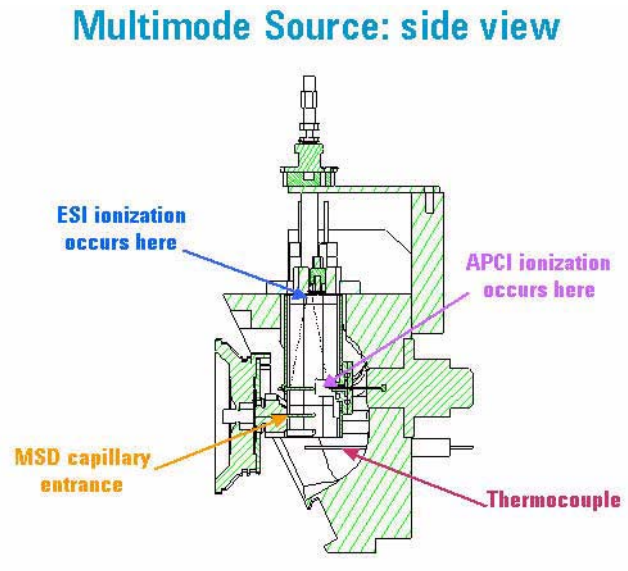


Figure 27 Graphical representation of a side view of the multimode source

Multimode source top down view

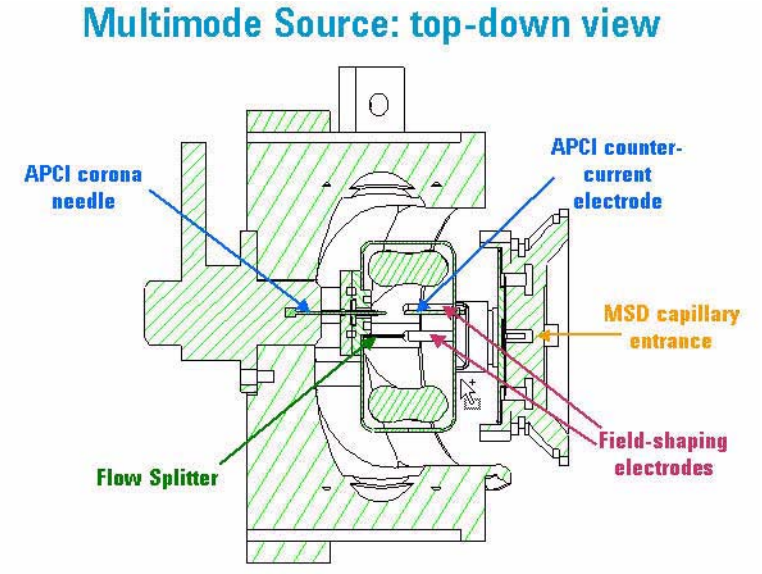


Figure 28 Graphical representation of a top view of the multimode source

Mobile phase considerations when using the multimode source

Flow rate

In general, once the analyte response is optimized, the flow rate or mobile phase composition may be changed without having to re-optimize the vaporizer temperature or drying gas temperature. The IR emitters have a feedback control that is provided by the vapor temperature sensor. Power to the IR emitters is dynamically changed to maintain the vapor temperature at the desired setpoint regardless of mobile phase changes.

ESI mode (or for compounds that have an ESI response in mixed mode)

Improved response will be obtained when lower amounts of electrolyte are used in the mobile phase. The recommended electrolyte concentration is lower, approximately one fifth that of prior recommendations. The need for lower electrolyte concentrations is due to the physics of ESI ion formation and charge separation in the multimode source. The following are example recommended electrolyte concentrations:

- 1 mM ammonium acetate instead of 5 mM
- 0.02% formic acid instead of 0.1%

NOTE The electrolyte-response effect is not present in the dedicated ESI source; continue to use higher electrolyte concentrations with the dedicated ESI source.

NOTE Do not use pure solvents (i.e., no electrolyte) as mobile phases for ESI work as this practice results in unpredictable analyte response.

APCI mode (or for compounds that have an APCI response in mixed mode)

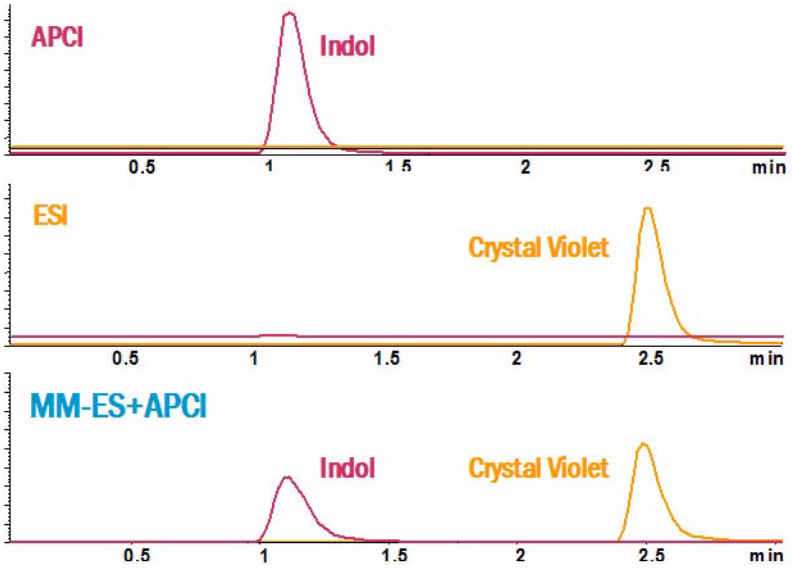
- In positive APCI mode, higher response is obtained using protic or neutral solvents as the organic mobile phase component instead of acetonitrile.
- Acetronitrile is a strong enough gas-phase base to deprotonate some analyte ions, leading to reduced response (30-fold reduction has been seen).
- Methanol, isopropyl alcohol, and acetone are preferred for positive mode APCI.

- In negative APCI mode, the use of acetonitrile has also been linked with a reduction in response of the analytes.

For electrolytes added to the mobile phase used for mixed mode operation, use care in selecting the anion as it may affect the response in negative APCI mode. For example, acetate is preferred over formate as it is more likely to accept a proton from the neutral analyte to produce a negatively-charged analyte ion under APCI conditions.

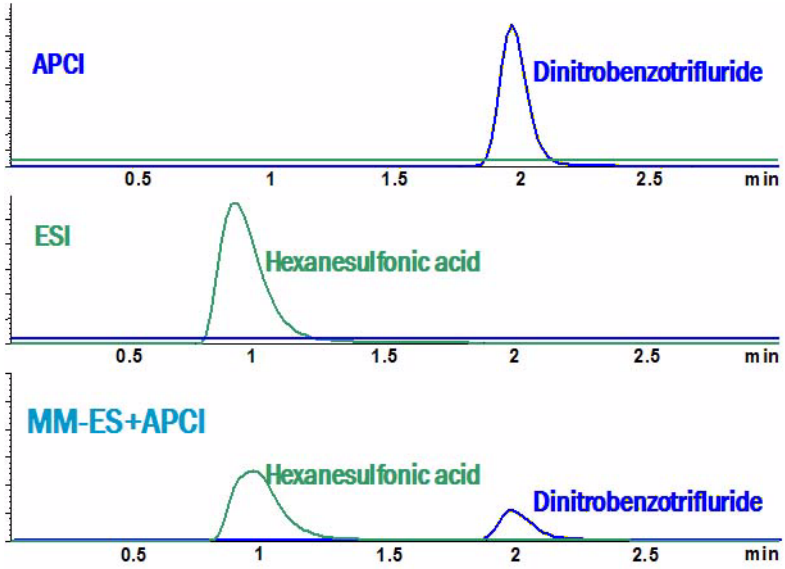
Source comparison in positive ion mode

Using the multimode source in MM-ES+APCI mode with positive polarity, you can acquire a single data file containing the two compounds, indol and crystal violet.



Source comparison in negative ion mode

Using the multimode source in MM-ES+APCI mode with negative polarity, you can acquire both hexanesulfonic acid and dinitrobenzotrifluride in the same file. Neither ESI nor APCI can acquire both of these compounds.



Guidelines

Guidelines for multimode source settings in MM-ES mode

Nebulizer pressure Always set to 60 psi.

Drying gas flow Always set to 5 L/min (IR does the drying).

Drying gas temperature Depends on the vaporizer temperature. A good starting temperature is 250°C. If running a cold vaporizer temperature, then set the drying gas temperature to vapor temperature or lower. If running the vaporizer hot (>175°C), then set the drying gas temperature to 350°C. Remember you want the vaporizer to control the drying and to be able to maintain the set point.

Vaporizer Start at 150°C. ESI works across a broad vaporizer range. If you are getting excessive sodium adducts, lower the temperature. You can run as low as 60°C. It is recommend to run 100°C or greater. Negative ion TFA adducts work best at lower temperature (<150°C).

Vcap Set to 2000 V. High mass ions (>1000) prefer a higher Vcap.

Charging Voltage Set to 200 V.

Guidelines for multimode source settings in MM-APCI mode

Nebulizer pressure Start at 20 psi. The signal decreases with higher nebulizer pressure.

Drying gas flow Always set to 5 L/min (IR does the drying)

Drying gas temperature Just set to 350°C. Remember you want the vaporizer to control the drying and to be able to maintain set point.

Vaporizer Start at 250°C. APCI is gas phase chemistry. A higher temperature usually works better. The vaporizer will compensate for flow rate or solvent composition.

Corona current Set to 5 µA positive or negative. The corona voltage will be ~4000 V at 6 µA.

Vcap Set to 2000 V. The APCI response is usually better at the higher end of the Vcap range.

Charging Voltage Set to 2000.

NOTE

The above set of parameters is provided as a starting point and should yield good response for many analytes. To obtain a specific response, i.e., high sensitivity detection of a compound or class of compounds, or a balanced response to a wide variety of compound classes, these parameters and the LC mobile phase composition should be examined further using traditional techniques for optimizing a method. The four most important parameters and the suggested optimization order are: vaporizer temperature, then capillary voltage, then nebulizer pressure, and then corona current.

Guidelines for multimode source settings in MM-ES+APCI mode

Drying gas flow Always set to 5 L/min (IR does the drying)

Nebulizer pressure Start at 40 psi. You will use the nebulizer pressure and the corona current to balance ESI and APCI. A higher nebulizer pressure results in more ESI. A lower nebulizer pressure results in more APCI.

Drying gas temperature Set to 300°C. Remember you want the vaporizer to control the drying and to be able to maintain the set point.

Vaporizer Start at 200°C. A higher temperature usually works better for MM-APCI. MM-ESI is usually unaffected except for sodium adduction. The vaporizer will compensate for flow rate or solvent composition.

Capillary Voltage Set to 2000 V. You are balancing ESI and APCI response. A setting of 2000 is a good compromise.

Corona current Set to 1 µA for positive or negative polarity. More corona current will reduce the ESI signal and increase the APCI signal.

Charging Voltage Set the charging voltage to 2000 V.

NOTE

The above set of parameters is provided as a starting point and should yield good response for many analytes. To obtain a specific response, i.e., high sensitivity detection of a compound or class of compounds, or a balanced response to a wide variety of compound classes, these parameters and the LC mobile phase composition should be examined further using traditional techniques for optimizing a method.

Empirical Formulas for high resolution MS

G2421A MM-ES Calibration Ions

	Positive Ion (m/z)	Empirical Formula
Betaine+H	118.086809	C5.H12.O2.N
HP-0321+H	322.048699	C6.H19.O6.N3.P3
HP-0622+H	622.029499	C12.H19.O6.N3.P3.F12
HP-0922+H	922.010300	C18.H19.O6.N3.P3.F24
	1307.969049	C25.H17.O6.N3.P3.F40
HP-1521+H	1521.971900	C30.H19.O6.N3.P3.F48
fragment	1807.937049	C35.H17.O6.N3.P3.F60
HP-2121+H	2121.933500	C42.H19.O6.N3.P3.F72
fragment	2307.905049	C45.H17.O6.N3.P3.F80
HP-2721+H	2721.895100	C54.H19.O6.N3.P3.F96

	Negative Ion (m/z)	Empirical Formula
TFA-H	112.985039	C2.O2.F3
fragment	431.981740	C9.O.N3.F14
adduct	601.978370	C12.H.O.N3.F21
adduct	w 734.006709	C14.H18.O8.N3.P3.F15
adduct	1033.987509	C20.H18.O8.N3.P3.F27
fragment	1305.953400	C25.H15.O6.N3.P3.F40
adduct	1633.949110	C32.H18.O8.N3.P3.F51
fragment	1805.921399	C35.H15.O6.N3.P3.F60
adduct	2233.910709	C44.H18.O8.N3.P3.F75
fragment	2305.889400	C45.H15.O6.N3.P3.F80
adduct	2833.872308	C56.H18.O8.N3.P3.F99

w indicates a weak adduct signal.

NOTE Fragment ion signals are progressively stronger at higher m/z by increasing CID energy.

G2432A MM-APCI Calibration Ions

	Positive Ion (m/z)	Empirical Formula
Purine+H	121.051421	C5.H5.N4
HP-0321+H	322.048699	C6.H19.O6.N3.P3
HP-0622+H	622.029499	C12.H19.O6.N3.P3.F12
HP-0921+H	922.010300	C18.H19.O6.N3.P3.F24
fragment	1307.969049	C25.H17.O6.N3.P3.F40
HP-1521+H	1521.971900	C30.H19.O6.N3.P3.F48
fragment	1807.937049	C35.H17.O6.N3.P3.F60
HP-2121+H	2121.933500	C42.H19.O6.N3.P3.F72

	Negative Ion (m/z)	Empirical Formula
Purine-H	119.035771	C5.H3.N4
fragment	556.001426	C10.H15.O6.N3.P3.F10
fragment	805.985476	C15.H15.O6.N3.P3.F20
fragment	1305.953400	C25.H15.O6.N3.P3.F40
fragment	1805.921399	C35.H15.O6.N3.P3.F60

NOTE Fragment ion signals are progressively stronger at higher m/z by increasing CID energy.

Tuning Mix and Test Mix

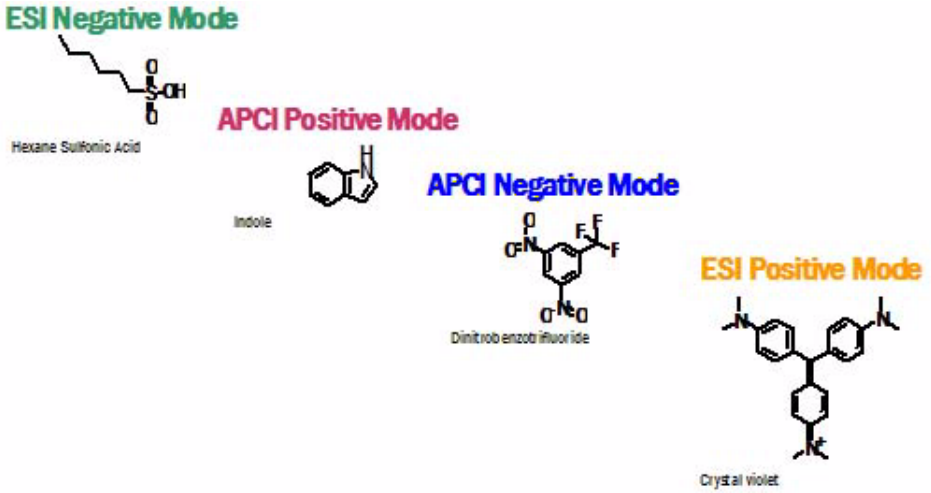
G1969-85020 MMI-L Low Concentration Tune Mix (100 mL)

Item-Description	Quantity
MMI tune mix label	1 each
plastic bottle	1 each
acetonitrile	74.67 g
HP-0321	<1 mg
HP-0621	<1 mg
HP-0921	<5 mg
HP-1521	<5 mg
HP-2121	<5 mg
HP-2721	<10 mg
HP-0585	<5 mg
betaine	<1 mg
tfa ammonium salt	26.2 mg

Trade Name	Chemical Description
HP-0321	Non-fluorinated triazatriphosphorine
HP-0621	Fluorinated triazatriphosphorine
HP-0921	Fluorinated triazatriphosphorine
HP-1521	Fluorinated triazatriphosphorine
HP-2121	Fluorinated triazatriphosphorine
HP-2721	Fluorinated triazatriphosphorine
HP-0585	Tris(heptafluoropropyl)-triazine

Chemical structures in the multimode test mix

The following diagram shows the chemical structure of the four components in the multimode test mix.



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In This Book

This book contains installation, operation, maintenance and troubleshooting instruction for the Multimode Source for 6410 Triple Quad LC/MS.

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