

**HP 1100 Series
LC/MSD System**

Getting Started

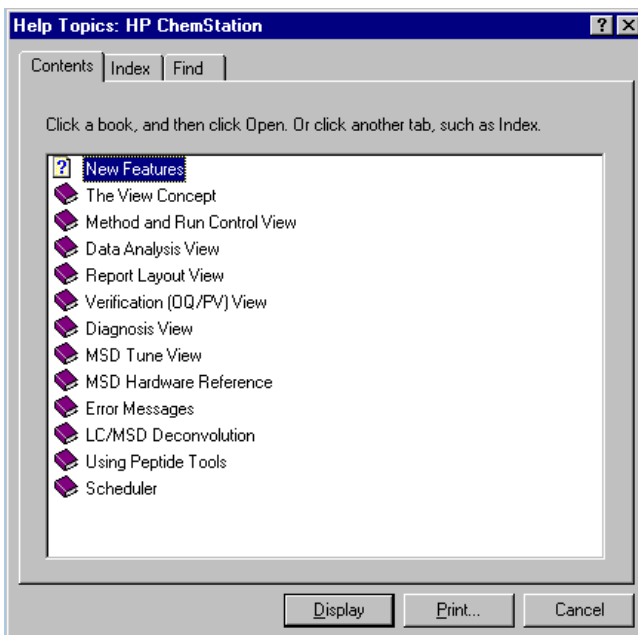


Using Online Help

To access the online help, select **Help Topics** from the Help menu in any window, or click the **Help** button on any dialog box.

When you jump to a help topic, it is displayed in another help window. Click the **Contents** button in that window to return to this list of help topics.

Item	Description
Contents	Displays the list of help topics (shown to the right).
Index	Lets you use keywords to search the help index for a particular topic.
Find	Lets you type a word or phrase and then displays a list of all the topics in the online help that contain those words.
Open	Opens the selected book or help topic. When you open a book or select a book that is already open, this button changes to Close .
Print	Prints the selected book or help topic for future reference. If you select a book, then all the help topics contained in the book are printed. To print a popup, click on it with the right mouse button and select Print from the menu.
Cancel	Closes the Help Topics window.



Help Icons



Indicates a book containing more help topics. To open a book, select it then double-click or click the **Open** button.



Indicates an open book of help topics. To close an open book, select it then double-click or click the **Close** button.

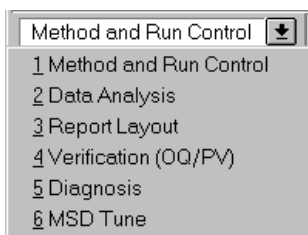


Indicates a help topic. To jump to a help topic, select it then double-click or click the **Open** button.

ChemStation Views

The ChemStation software consists of several views. The available items in the menus and toolbar depend on the current view. Each view allows you to perform a certain set of tasks.

You can switch views using the View menu or by using the pulldown menu in the upper left corner of the screen.



Each view is briefly described below and on the following pages.

Method and Run Control View

In this view, you can set up methods and adjust instrument parameters to inject samples and acquire data one sample at a time or in automated sequences.

Data Analysis View

In this view, you can perform various data evaluation tasks on chromatograms and spectra. You can view both mass spectral and UV signals simultaneously. Common tasks include integration, quantitation, checking peak purity, and deconvolution. Once you have analyzed the data, you can select one of the predefined report types.

Report Layout View

This view lets you design your own custom report templates to use with the data generated by your ChemStation.

Verification (OQ/PV) View

In this view you can determine if your system is operating in a predictable manner. This is useful to show Good Laboratory Practice (GLP) compliance. This may be required by some government agencies.

Diagnosis View

This view lets you run tests to diagnose instrument problems and access information on how to resolve these problems. The early maintenance feedback (EMF) feature can be used to notify you when it is time to perform system maintenance before a problem occurs. Videos of the various maintenance procedures are provided on the LC/MSD maintenance CD-ROM.

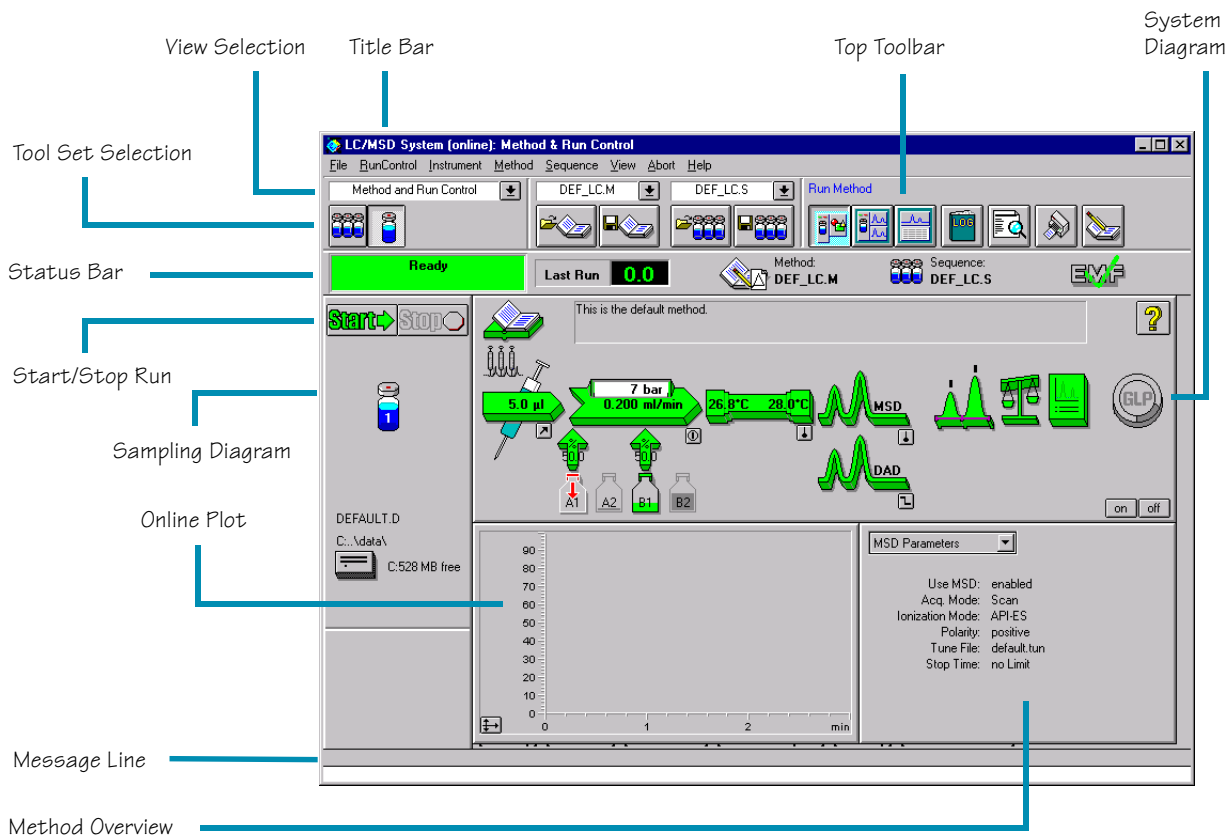
MSD Tune View

This view lets you calibrate your LC/MSD automatically. You can also set parameters manually for specific types of molecules.

HP LC/MSD Reference Collection CD-ROM

More information about the ChemStation views and how to use your system can be found in the ChemStation online help and on the HP LC/MSD Reference Collection CD-ROM.

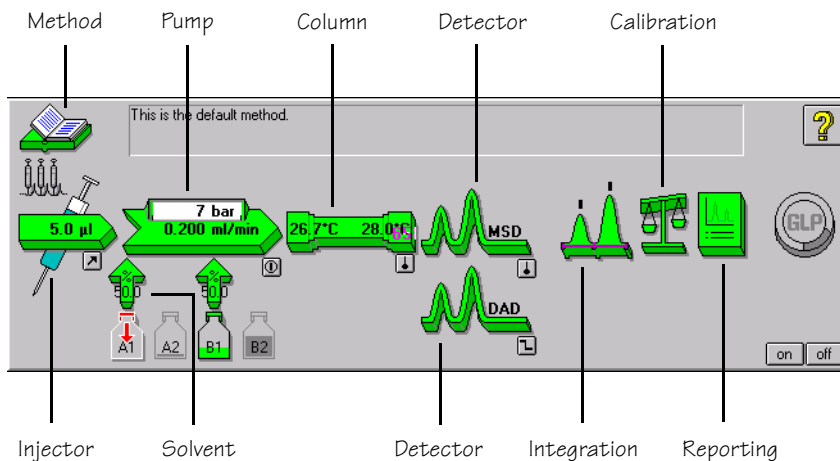
Method and Run Control View (configured with LC/MSD and DAD)



System Diagram

Each icon of the system diagram represents one component or module of your system.

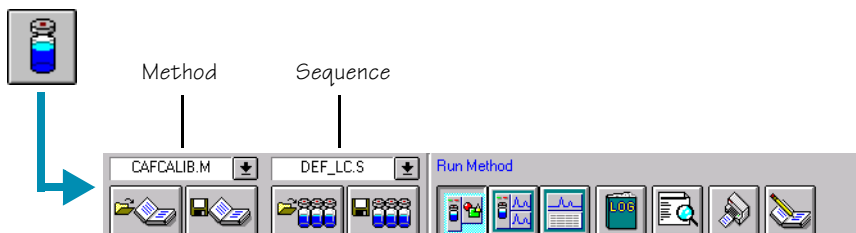
Click on an icon if you want to edit the method parameters or go to the online help for that particular component.



Single Sample Tool Set

Lets you work on methods and run an analysis for a single sample.

If you click the Single Sample icon, the toolbar changes as shown.

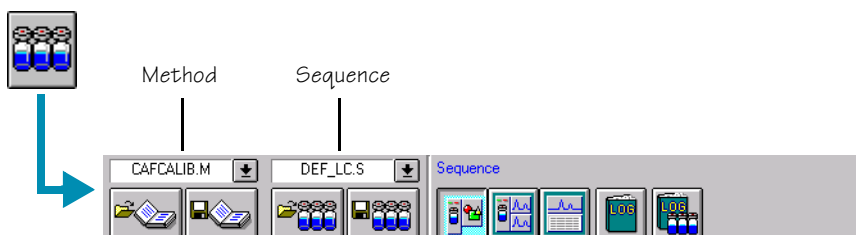


Move the cursor over individual icons or screen items to display descriptive information on the message line.

Sequence Tool Set

Lets you work on sequences and run automated analyses of multiple samples.

If you click the Sequence icon, the toolbar changes as shown. The sampling diagram now shows the sample tray.



Move the cursor over individual icons or screen items to display descriptive information on the message line.

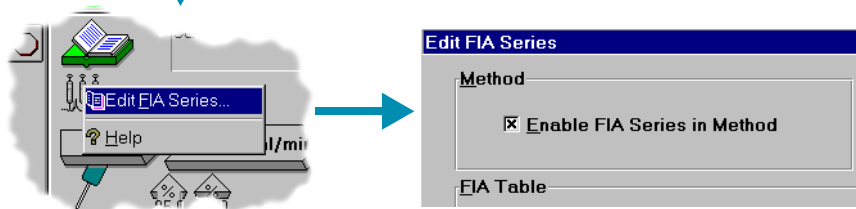
Flow Injection Analysis

Lets you inject multiple samples directly into the detector, bypassing the chromatographic column. The results are sent to a single data file. FIA can be used for method development or for applications that do not require chromatography.



To enable FIA from the system diagram:

- Click the FIA icon.
- Select **Edit FIA Series**.
- Check the **Enable FIA Series in Method** box and edit the FIA Table.



Data Analysis View

The screenshot shows the 'Data Analysis' window of the LC/MSD System. The window title is 'LC/MSD System (online): Data Analysis'. The menu bar includes File, Graphics, Integration, Calibration, Report, Spectra, Batch, View, Abort, and Help. The toolbar contains icons for Data Analysis, CAFCAL02.D, CAFCALIB.M, Integration / Report, and GLP+Short. A dropdown menu shows 'All Loaded Signals'. The main plot area displays two chromatograms: 'DAD1 A, Sig=272.4 Ref=460.80 (MSDEMO\CAFCAL02.D)' and 'MSD1 TIC, MS File (MSDEMO\CAFCAL02.D)'. The DAD plot shows a peak at approximately 2.5 minutes labeled 'caffeine'. The TIC plot shows a peak at approximately 2.5 minutes labeled 'caffeine'. A vertical toolbar on the right contains various tool icons. Callouts on the left point to 'View Selection', 'Title Bar', 'Tool Set Selection', 'Signal View Selection', 'DAD Signal', 'LC/MSD Signal', and 'Message Line'. Callouts on the top right point to 'Cursor Tools' and 'Graphics Tools'.

Integration Tool Set

Lets you perform integration and reporting tasks on a chromatogram.

The screenshot shows a close-up of the 'Integration / Report' toolbar. A callout points to the 'Integration' icon (a grid with a curve) and says 'Click the Integration icon to display this toolset.' The toolbar includes dropdown menus for 'Data Analysis', 'CAFCAL04.D', 'DEF_LC.M', and 'Integration / Report'. A 'Short' button is also present. Below the toolbar is a 'Message Line' with a scroll bar. Callouts point to 'Data File' and 'Method'. A text box on the right says 'Move the cursor over individual icons or screen items to display descriptive information on the message line.'


Calibration Tool Set

Lets you perform calibration tasks for quantitation.

Click the Calibration icon to display this toolset.

Data File: CAFCAL04.D
Method: DEF_LC.M

Move the cursor over individual icons or screen items to display descriptive information on the message line.



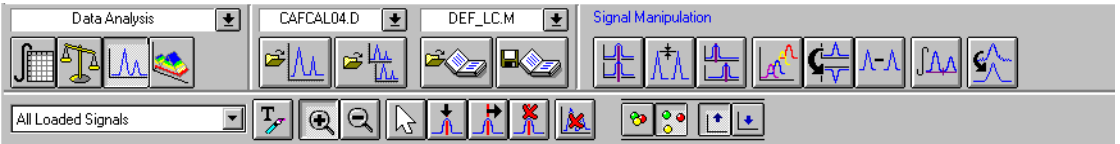
Signal Tool Set

Lets you graphically work with the UV or MS signal.

Click the Signals icon to display this toolset.

Data File: CAFCAL04.D
Method: DEF_LC.M

Move the cursor over individual icons or screen items to display descriptive information on the message line.




Spectral Tool Set

Lets you perform spectra evaluation tasks.

Click the Spectral icon to display this toolset.

Data File: CAFCAL04.D
Method: DEF_LC.M

Move the cursor over individual icons or screen items to display descriptive information on the message line.












Data Analysis Tools

Integration Tool Set

Lets you perform integration and reporting tasks on a chromatogram.






-  Integrate all chromatograms
-  Find suitable integration events
-  Define integration events
-  Specify a report
-  View a report on screen
-  Print a report

The following cursor tools are for manual integration and working in the signal window:









-  Graphics tools (available in all tool sets)
-  Zoom in (available in all tool sets)
-  Zoom out (available in all tool sets)
-  Pointer tool (available in all tool sets)
-  Draw baseline
-  Draw baseline for negative peaks
-  Tangent skim
-  Split peaks
-  Delete peak from integration results

Calibration Tool Set

Lets you perform calibration tasks.









-  Create a new calibration table
-  Recalibrate using current chromatogram
-  Add calibration levels to calibration table
-  Add new peaks to your calibration table
-  Define signal options

The following cursor tools are for manual calibration and signal actions.









-  Delete peaks from the calibration table
-  Add peaks to the calibration table
-  Recalibrate peaks in the calibration table
-  Set calibration table options
-  Show/hide current chromatogram
-  Zoom in on MS window
-  Extract ion chromatogram
-  Hide extracted ion chromatogram

Signal Tool Set

Lets you graphically work with a chromatogram.




-  Align the x-axis of multiple signals
-  Align the y-axis of multiple signals
-  Reset the alignment of your signals
-  Create a 3D overlay of signals
-  Mirror signals
-  Subtract signals
-  Integrate all chromatograms
-  Smooth all signals

The following cursor tools are for working with alignment markers and the signal displays.










-  Set a time reference point
-  Move a time reference point
-  Delete a time reference point
-  Delete object from chromatogram display
-  Display signals overlaid
-  Display signals separated
-  Display the signals in full scale
-  Display all signals in the same scale

Spectral Tool Set

Lets you perform spectral evaluation tasks.

-  Set spectral options
-  Open a spectral library
-  Save a spectral library

The following cursor tools are for spectral selection tasks.

-  Select spectrum
-  Select peak apex spectrum
-  Select average spectrum
-  Average spectra across a peak
-  Select peak for purity evaluation
-  Select 1st reference spectrum
-  Select 2nd reference spectrum
-  Zoom in on MS window
-  Extract ion chromatogram

Report Layout View

The screenshot shows the 'Report Layout' window in the LC/MSD System. The window title is 'LC/MSD System [online]: Report Layout'. The menu bar includes 'File', 'Edit', 'View', 'About', and 'Help'. The main interface is divided into several sections:

- View Selection:** A dropdown menu currently showing 'Report Layout'.
- Title Bar:** The top bar of the window containing the title and standard window controls.
- Calculation Technique:** A dropdown menu showing 'DEF_LCM'.
- Report Template:** A dropdown menu showing 'SIMPLE.FRP'.
- Top Toolbar:** A row of icons for various functions like file operations and printing.
- Report Section:** A vertical list on the left side of the main content area, currently showing 'Header' and 'General'. A blue triangle points to 'Header'.
- Message Line:** A horizontal bar at the bottom of the window.


The main content area displays a report layout template with the following text:

Example of a simple Report Layout

Data File name: [] Raw Data File Name []
 Method name: [] Method []

Header
General

To test this template load a calibrated method

 Replace the logo by inserting your company logo as WMF graphic and insert your company name as a "constant text" here.

The general section comprises elements that are valid for the complete data file. The elements below were inserted using the text and number tools.

data acquired by: Acq. Operator []
 on: Injection Date []

New Section Menu







Report Section

Insertion Triangle

Click a blue triangle to activate the new section menu (triangle turns red)

Message Line

Report Layout Tips

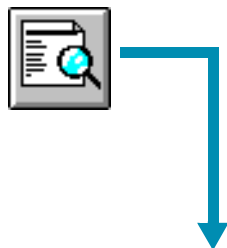
-  Pointer Tool
-  Graphics Tool
-  Table Tool
-  Text Tool
-  Number Tool
-  Page Break Tool

- Move the cursor over the individual icons or screen items to display descriptive information on the message line.
- Edit or create a report layout as described in the online help.
- To test your report layout, select a calculation technique from the drop-down list box to define how the results should be calculated.
- Load a data file. The results are loaded into the report template using the selected calculation.
- Select **File / Add to Report Styles** to add your completed report template to the list of available report styles. Now you can use your customized report within a method.

Sample Report Layouts (in \hpchem\repstyle)

Template	Description
SIMPLE.FRP	Includes a chromatogram and calibration curves per calibrated compounds.
NESTED.FRP	Shows how to nest subsections.
SPECTRA.FRP	Shows how to include spectra.
PURITY.FRP	Shows how to include peak purity data.
ESTD.FRP	Uses the same elements as the standard ESTD reports so you can customize your external standard reports.
AREAPCT.FRP	Uses the same elements as the standard area percent report so you can customize your area percent reports.
LIBRARY.FRP	A customized report that uses library search results. You must have a method loaded that specifies the spectra library.


Print Preview button



Report Example of a simple Report Layout

Data File name: C:\HPCHEM\1\DATA\MSDEMO\CAFCAL02.D
Method name: C:\HPCHEM\1\METHODS\CAFCALIB.M

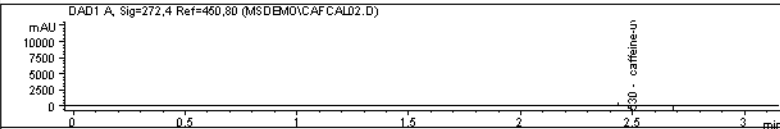
To test this template load a calibrated method

 **HEWLETT PACKARD** Replace the logo by inserting your company logo as WMF graphic and insert your company name as a "constant text" here.


The general section comprises elements that are valid for the complete data file. The elements below were inserted using the text and number tools.

data acquired by: mcintyre
on: 1/6/97
vial # 2.000

DAD1 A, Sig=272,4 Ret=460.80 (MSDEMO\CAFCAL02.D)



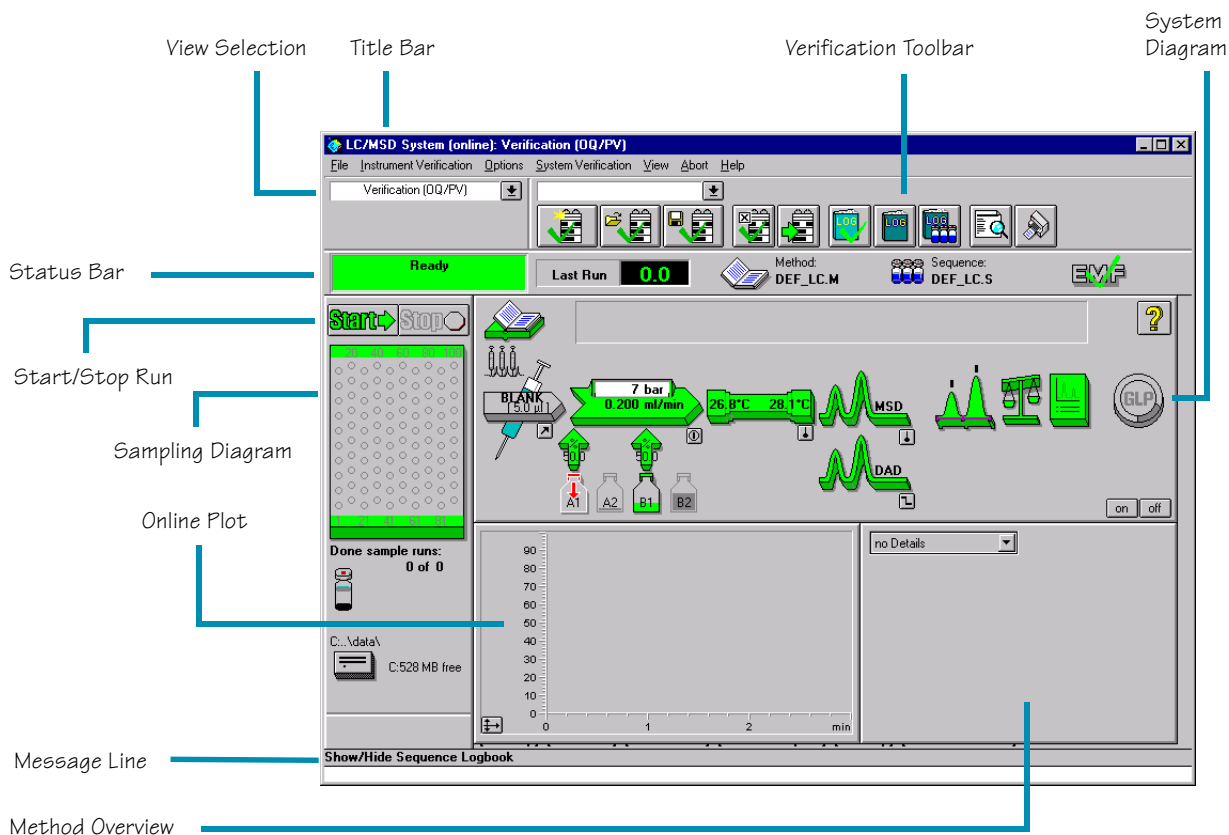
MSD1 TIC, MS File (MSDEMO\CAFCAL02.D)



HP LaserJet 4Si MX on LPT1:
Letter 8 1/2 x 11 in/Portrait

Close normal Page 1 of 2 Prev Next Print Help

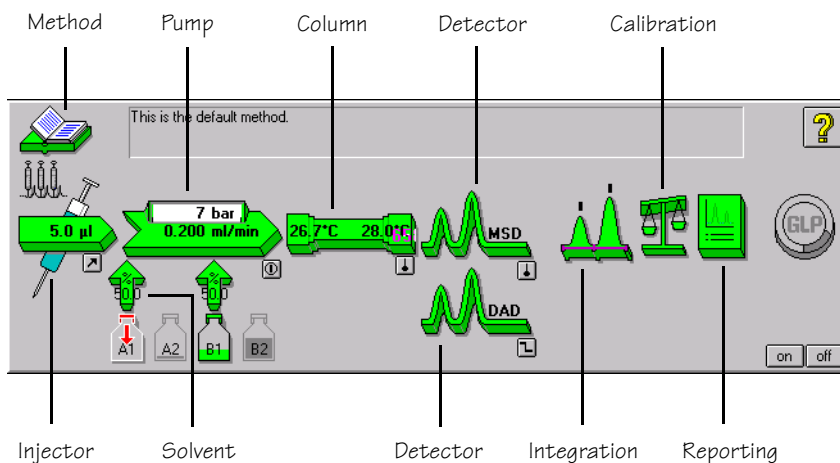
Verification (OQ/PV) View



System Diagram

Each icon of the system diagram represents one component or module of your system.

Click on an icon if you want to edit the method parameters or go to the online help for that particular component.



Verification Toolbar

The Verification Toolbar, which is located at the top of the Verification screen, is displayed when you select **Show Top Toolbar** from the **View** menu.



Set up a new verification.



Load a previously-stored instrument verification.



Save the current instrument verification to disk.



Edit the current instrument verification.



Run the current instrument verification.



Toggle the display of the Verification Logbook.



Toggle the display of the current online Logbook.



Toggle the display of the current Sequence Logbook.



Display the Instrument Verification Test Report.



Print a report of the current instrument verification results.

About This View

The Verification (OQ/PV) view lets you test whether your analytical instruments and the ChemStation software are operating correctly according to predefined performance criteria.

• Operation Qualification (OQ)

Operation Qualification is the documented verification that the equipment-related system or subsystem performs as intended throughout representative or anticipated operating ranges.

• Performance Verification (PV)

Performance Verification is the documented verification that the process and or the total process-related system performs as intended throughout all anticipated operating ranges.

Available Tests

The following verification tests are supplied with your ChemStation software. Refer to the online help for more information on how to run, edit, or create new verification tests.

- VWD Wavelength Accuracy
- Intensity
- Holmium
- Temperature Accuracy
- Noise, Flow, Temperature
- DAD Wavelength Accuracy
- Injector Precision*
- Detector Linearity/Carry-over*
- Injector Linearity*
- Gradient Composition

* These tests can be used to verify LC/MSD performance.

Diagnosis View

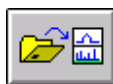
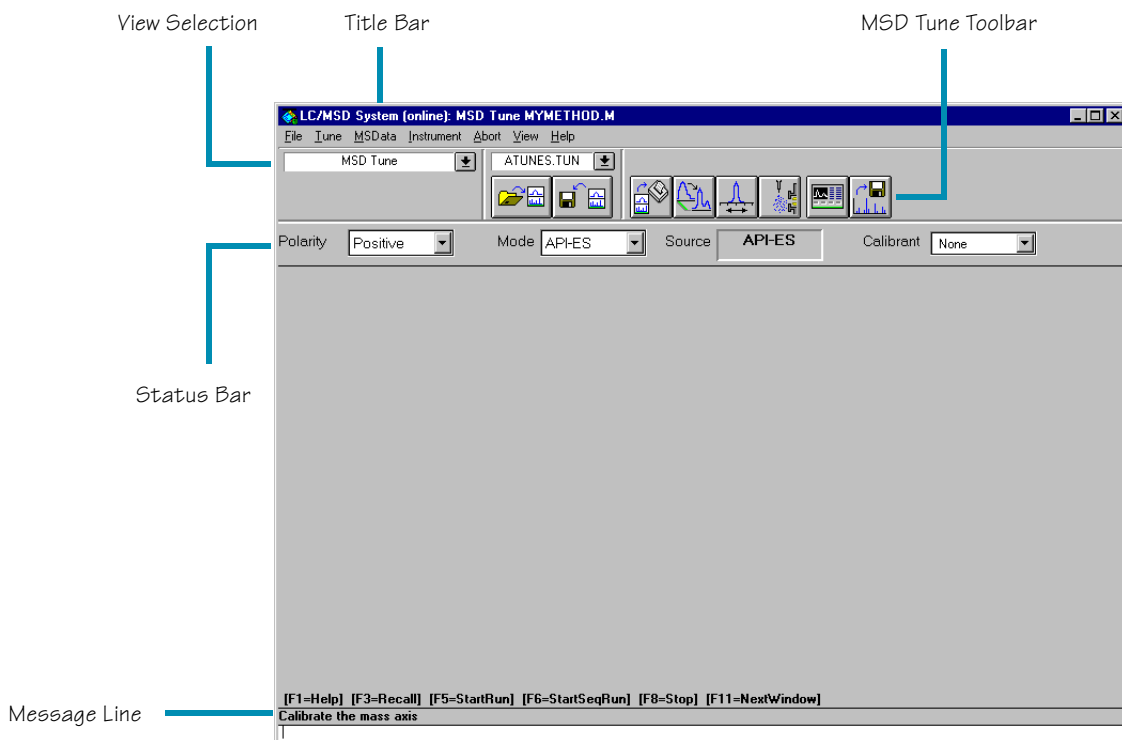
The screenshot shows the 'Diagnosis View' window for the 'LC/MSD System (online)'. The window title is 'Diagnosis_DEF_LC.M_DEFAULT.D_SMAL0303.TUN'. The menu bar includes 'File', 'Diagnosis', 'Maintenance', 'View', 'Abort', and 'Help'. The main interface is divided into several sections:

- View Selection:** A dropdown menu currently set to 'Diagnosis'.
- Title Bar:** The top window title bar.
- Symptoms:** A section containing 'Thermostat Temperature' and 'Defective Heater' dropdown menus, along with 'OQ/PV' and 'Analytic' buttons.
- Possible Causes:** A section with 'Test Results/Logbooks', 'Observed Symptoms', and 'Possible Causes' buttons.
- Instrument Panel:** A central area with a schematic diagram of the system components. Below the diagram are status indicators for 'Binary Pump', 'ALS', 'Thermostat', 'DAD', and 'MSD', all of which are currently green.
- Variables Display:** A section titled 'MSD Generic Information' containing a table of system data:

Product No.	G1946A	Serial No.	US78912345
FW Rev. Sbc Lon	PRS1.01.01	MSD On Time	0.00 h
Manufacturing Date	04/01/96	FW Rev. SmartCard	2.06.88
HP-IB Address	0		
- Message Line:** A status bar at the bottom left with the text '[F1=Help] [F3=Recall] Print test results or logbooks'.
- Cause Information Pad:** A right-hand panel titled 'Memo Pad' and 'Defective Heater' containing a 'Thermostat Test' section with a 'Start' button and energy consumption data:

Thermostat Test	Start
Left Energy Cons.	1871.75 kW/s
Right Energy Cons.	1105.28 kW/s
- Help:** A yellow question mark icon in the bottom right corner of the Cause Information Pad.
- Link to Maintenance CD-ROM:** A CD-ROM icon in the bottom right corner of the Cause Information Pad.

MSD Tune View



Load an LC/MSD tune file.



Calibrate the mass axis.



Save the current tune file.



Change spray chamber parameters.



Generate a profile and scan report.



Edit LC/MSD parameters for manual tuning.



Autotune the LC/MSD and print a tune report.



Acquire multiple scans and send results to data file.

To Start Up and Shut Down the System

To Start Up the System

This procedure assumes the system is under vacuum and the LC and LC/MSD are properly connected. It also assumes the liquid flow path is properly set up.

- 1 From the Method and Run Control view, click the **On** button in the system diagram.
- 2 Set up the LC conditions (pump, column heater, and detector).
- 3 Set up the LC/MSD spray chamber conditions. If you are using normal flow rates, the following values are typical.

Allow 15 minutes for the system to warm up.

Electrospray

The screenshot shows the 'MSD Spray Chamber' configuration window. The 'Ionization Mode' is set to 'AP-ES'. The 'Installed Spray Chamber' section is titled 'Temperatures, Pressure, and Flow' and contains a table with columns for 'Actual', 'Setpoint', and 'Maximum'. The 'Parameters' section is below the table.

	Actual	Setpoint	Maximum
Drying Gas Flow (l/min):		12.0	13.0
Nebulizer Pressure (psig):		50	60
Drying Gas Temperature (°C):		300	350
Vaporizer Temperature (°C):		N/A	N/A

Parameters

Capillary Voltage (V):	4000
Corona Current (µA):	N/A

APCI

The screenshot shows the 'MSD Spray Chamber' configuration window. The 'Ionization Mode' is set to 'APCI'. The 'Installed Spray Chamber' section is titled 'Temperatures, Pressure, and Flow' and contains a table with columns for 'Actual', 'Setpoint', and 'Maximum'. The 'Parameters' section is below the table.

	Actual	Setpoint	Maximum
Drying Gas Flow (l/min):		6.0	13.0
Nebulizer Pressure (psig):		50	60
Drying Gas Temperature (°C):		300	350
Vaporizer Temperature (°C):		350	500

Parameters

Capillary Voltage (V):	4000
Corona Current (µA):	4.0

To Put the System in Standby Mode

Put the system in standby mode overnight or whenever you won't be analyzing samples for an extended time. The standby state for the LC/MSD leaves the nebulizer and drying gas on at low flow.

- 1 Flush the system with pure mobile phase to ensure the flow path is clear of buffers. This avoids plugging the nebulizer. A plugged nebulizer can cause a high back pressure which can damage the LC flow cell.

It is good practice to flush the flow path (including the column) for 5-10 minutes with a mobile phase without buffers such as 50:50 water/acetonitrile.

- 2 Click the **Off** button in the system diagram. All modules will now be set to standby mode (indicated by the gray color in the system diagram).

Standby for the LC/MSD is 20 psi for the nebulizer, 3 L/min for drying gas, 300°C for drying gas temperature and, if present, 325°C for the APCI vaporizer. Also, the MS stream selection valve is set **LC to Waste**.

To Tune the LC/MSD

What is Tuning?

When the LC/MSD is used as a detector for the HPLC, a mass spectrum is associated with each data point in the LC chromatogram. To obtain high quality, accurate mass spectra, the LC/MSD must be optimized to:

- Maximize sensitivity
- Maintain acceptable resolution
- Ensure accurate mass assignment

Tuning is the process of adjusting LC/MSD parameters to achieve these goals. After the LC/MSD parameters have been optimized, they must be saved in a tune file (.tun). This tune file is then specified in the method that is used to acquire data for your samples.

Frequent tuning, automated or manual, is not required. Once tuned, the LC/MSD is very stable. Tuning should generally not be needed more often than monthly, or at most weekly.

Wait at least 4 hours after pumpdown before tuning or operating your LC/MSD. It takes the analyzer at least 4 hours to reach thermal equilibrium. Tune files created or data acquired before the LC/MSD is at thermal equilibrium may have incorrect mass assignments and other inaccuracies.

Using Autotune

Use autotune for automated adjustment of the LC/MSD performance.

- 1 From the MSD Tune view, select **Tune / Autotune** or click the **Autotune** icon.
- 2 Review the tune report which is printed automatically when tuning is completed.

Using Check Tune

Check Tune lets you quickly determine whether the LC/MSD is correctly tuned without performing a complete autotune. It performs a single profile scan of the tune masses and compares the peak widths and mass axes with target values.

- 1 Select **Tune / Check Tune**.
- 2 Review the Check Tune report. If any values are outside of acceptable ranges, Check Tune will suggest adjustments.

Using Manual Tune

Use Manual Tune when you want to:

- Achieve maximum sensitivity by sacrificing some resolution
- Tune specifically for the very low end (<150 amu) of the mass range
- Tune with a compound other than the standard calibrants

Manual tuning involves 4 steps:

- 1 Optimizing ion transmission through the source ion optics (fragmentor, skim 2, lens 1, lens 2, octapole peak and octapole knee).
- 2 Setting the desired mass resolution (adjusting width gain and width offset).
- 3 Calibrating the mass axis (adjusting mass gain and mass offset).
- 4 Adjusting the signal strength (setting iris and adjusting the multiplier gain).

Note that fragmentor and gain are method parameters. The fragmentor affects ion transmission and fragmentation. For more information, see the online help.

To Acquire LC/MS Data

Modes of Acquisition

There are three modes of acquiring data:

- Running a method for a single sample
- Running a sequence for multiple samples
- Running an FIA series

Note the following about acquiring data:

- All three acquisition modes require an appropriate method.
- Samples may be injected either manually or with an ALS.
- A run must always be started from the software.
- An FIA method cannot be used in a sequence.

To Edit a Method and Start a Run

Once you know the acquisition mode you want to use, you need to set up an appropriate method. Methods are set up in the Method and Run Control view. New methods are created by editing existing ones.

- 1 Select **Method / Load Method** or click the Open Method button on the toolbar. Choose a method from the list.
- 2 Select **Method / Edit Entire Method**. This menu item is also available when you click the method icon on the system diagram.

Once you select to edit a method, a series of dialog boxes will be displayed that let you set up your method and instrument parameters.

Click the **Help** button on any of the following dialog boxes for descriptive information on the items available.

- 3 Select the method sections you want to edit (select all sections to become familiar with the method parameters that are available).
- 4 Add any method comments you want to appear on your reports.
- 5 Set up the instrument parameters:
 - set up the pump parameters
 - set up the injector parameters
 - set up the DAD (or VWD) parameters
 - set up the column thermostat parameters
 - set up the MSD signals
 - set up the MSD spray chamber
- 6 Set up the Data Analysis parameters:
 - set up the signal details
 - edit integration events
 - specify report parameters
 - select the instrument curves
 - select the calibration curves
 - set up the calibration table
 - find the ion parameters
- 7 Complete the Run Time Checklist.
- 8 Save the method using a different name. Select **Method / Save Method As** or click the Save Method button on the toolbar.






Once you are familiar with the options that are available, you can use the system diagram menus for quick access to particular method parameters, rather than using the edit entire method process.
- 9 When you are ready to begin a run, click the **Start** button.

To Use Deconvolution


Deconvolution is a process that transforms mass spectra from multiply-charged ions into a calculated molecular weight.

To Deconvolute a Mass Spectrum

This is a generalized procedure; you may need to do some fine tuning.


- 1 In the Data Analysis view, load an electrospray MS data file. The TIC will be displayed.
- 2 Generate the MS spectrum that you want to deconvolute (select the Spectral task tool set and use the spectrum selection tools to pick the spectrum of interest). Note that these operations are performed on data acquired in full scan mode.
- 3  Click the **Enter Deconvolution Tool** button to set up the deconvolution display area.
- 4  When the spectrum is displayed, click the **Find Ions** button to locate masses that will be used in deconvolution.
- 5  Examine the ions that were found. Very noisy data or data with unresolved regions often need special settings to find ions. You can use the **Find Ions Options** button to optimize the set of ions found.
- 6  Click the **Edit Deconvolution Parameters** button to change any of the parameters.
- 7  Click the **Run Deconvolution** button to begin the deconvolution process. Once the deconvolution is done, the Components will be displayed in the upper right window. The Charge States will be displayed in the bottom right window.

- 8 You can look at individual components or groups of components by selecting specific components from the Component list.

- 9  Optionally, click the **Preview Deconvolution Report** button



or click the **Print Report** button.

- 10  Optionally, select components from the Component List box and then click the **Delete Components** button. This action removes the peaks for the selected components from the original spectrum. You can then continue with step 4 above, using this new spectrum.

- 11 Repeat the process.

Deconvolution Report

The Deconvolution report contains summary information about each component selected, along with detailed information on how each peak in a component contributes to the component's molecular weight. In the first part of the report, the components are ranked by percent relative abundance, which is useful for estimating the percent of impurities.

Note that the actual molecular weight may differ from the computed molecular weight even if the data fit a Gaussian curve perfectly, due to other errors such as errors in mass axis assignment or unresolved chemical impurities.

The Deconvolution software is optional and is part of the HP G2720AA Bioanalysis Software package.

Operating Tips

- ❑ Back up your data and methods **regularly** to avoid loss of data if the files are accidentally overwritten, deleted, or if a hardware problem develops with your disk drive.
- ❑ Put the system in standby mode overnight or whenever you won't be analyzing samples for an extended time.
- ❑ Make sure the tune file you are using is appropriate for your samples.
- ❑ Save Tune reports in an MS Logbook for future reference.
- ❑ Regular system maintenance can reduce problems. Maintenance tasks are described on the HP 1100 Series LC/MSD Maintenance CD-ROM. Keep a maintenance record.
- ❑ Use the Maintenance Logbook and EMF features (in the Diagnosis view) to help you keep track of when maintenance is needed and to keep an online maintenance record.
- ❑ Flush the sample path and clean the spray chamber, capillary tip, and spray shield daily or at the end of each shift. Check the pump fluid level every week.
- ❑ The spray chamber vent hose must be connected to a lab vent that is used **only** for the source (completely separate from the vent hose for the foreline pump). Otherwise, waste products can migrate into the spray chamber vent producing chemical noise.
- ❑ Samples need to be filtered. They should be salt and detergent-free if no chromatography is used.
- ❑ Use only filtered, HPLC-grade mobile phase.
- ❑ If a UV detector is available, use it in series with the LC/MSD. Try to minimize chromatographic peak broadening by using low dispersion tubing.
- ❑ To avoid chromatographic band broadening, make sure all tubing connections are free of dead volume. Use zero dead-volume (ZDV) fittings when possible. If using fingertight fittings, force tubing into unions and tees while tightening the fittings.
- ❑ Use the following table as a guide to using SIM, condensed scan, and full scan acquisition modes.

Task	Mode
Acquire electrospray data for samples containing large, multiply-charged analytes.	Full Scan
Analyze a mixture with unknown components (small molecules).	Scan
Analyze a mixture with known components in unknown amounts (quantitate).	Scan or SIM
Identify the presence of a few known compounds at low levels within a mixture.	SIM

- ❑ If you are using APCI, the optimal flow rate is 1.0 ml/min. The range is 0.5 to 1.5 ml/min.

Troubleshooting Tips

No peaks

- Make sure there is spray from the nebulizer.
- Make sure the capillary voltage is set correctly.
- Make sure the LC/MSD is tuned correctly.
- Make sure LC/MSD pressures are within normal ranges.
- Check the drying gas flow and temperature.
- Make sure the fragmentor is set correctly.

Poor mass accuracy

- Recalibrate the mass axis.
- Make sure the ions used for tuning span the mass range of the sample ions and that they show strong, stable signals.

Low signal

- Check the solution chemistry. Make sure the solvent you're using is appropriate for your sample. Mixed samples can exhibit signal suppression of one or more components.
- Make sure the sample is fresh and has been stored correctly.
- Make sure the LC/MSD is tuned correctly.
- Check the nebulizer condition.
- Clean the capillary entrance.
- Check the capillary for damage and contamination.

Unstable signal

- Make sure the drying gas flow and temperature are correct for the solvent flow you are using.
- Make sure the solvent is thoroughly degassed. Do **not** use ultrasonic degassing with protein samples.
- Make sure the LC backpressure is steady; this indicates a steady solvent flow.

High spectral noise

- Use appropriate mass filter values.
- Check the spray shape. Nebulizer may be damaged or incorrectly set.
- Make sure drying gas flow and temperature are correct for the solvent flow you are using.
- Make sure the solvent is thoroughly degassed. Do **not** use ultrasonic degassing with protein samples.
- Make sure the LC backpressure is steady; this indicates a steady solvent flow.
- If you are using water as part of the mobile phase, make sure it is de-ionized (>18M Ω).

Droplets, not spray, exiting the nebulizer

- Make sure the nebulizing gas pressure is set high enough for the LC flow being used.
- Check the position of the needle in the nebulizer.
- Stop the solvent flow and remove the nebulizer assembly. Use a magnifying glass to examine the end of the nebulizer for damage.

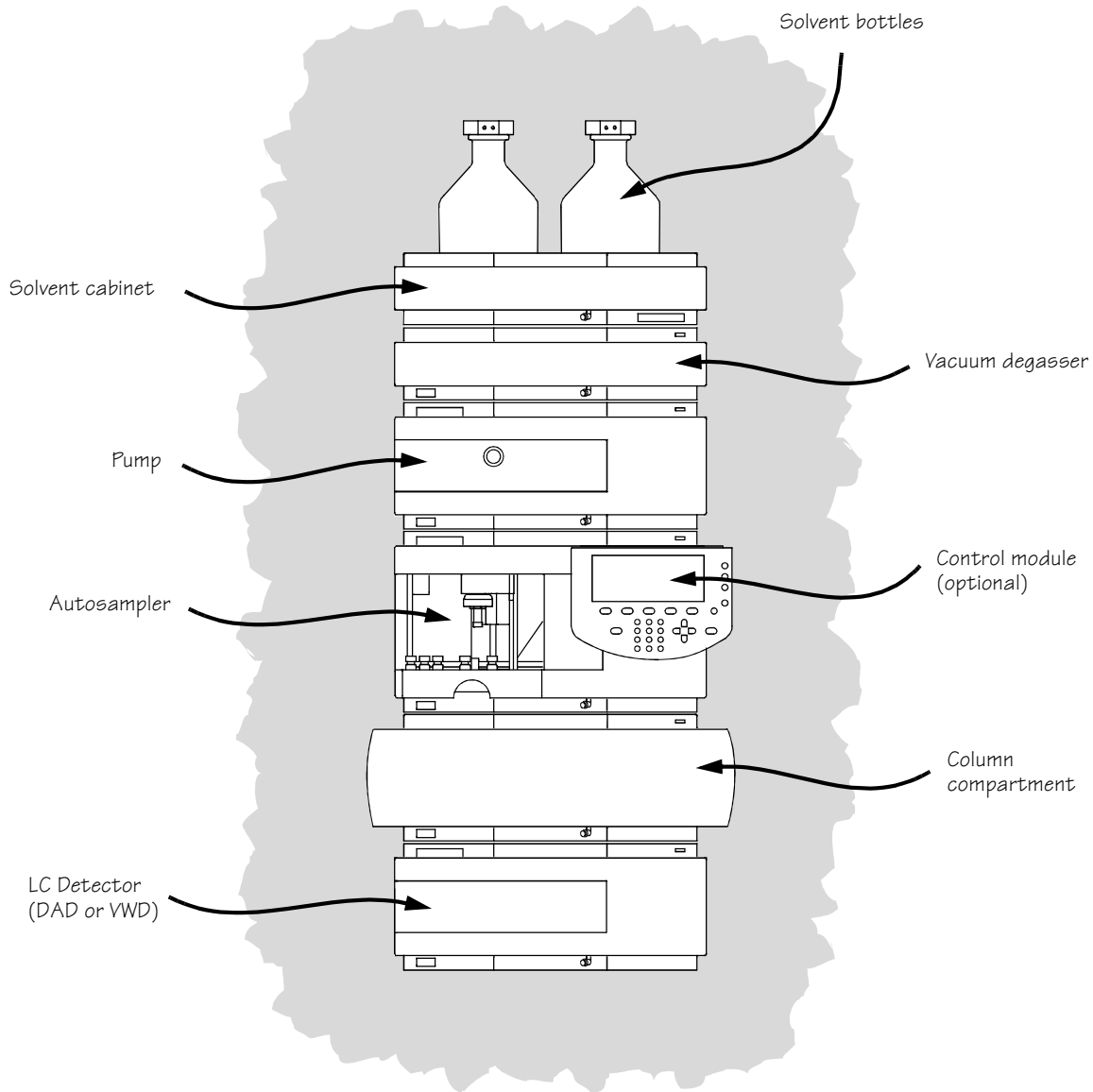
No flow

- Make sure the LC is on and there is sufficient solvent in the correct bottle.
- Check for LC error messages.
- Check for blockages. Repair or replace any blocked components.
- Check for leaks.
- Make sure the MS stream selector valve is set to **LC to MSD**.

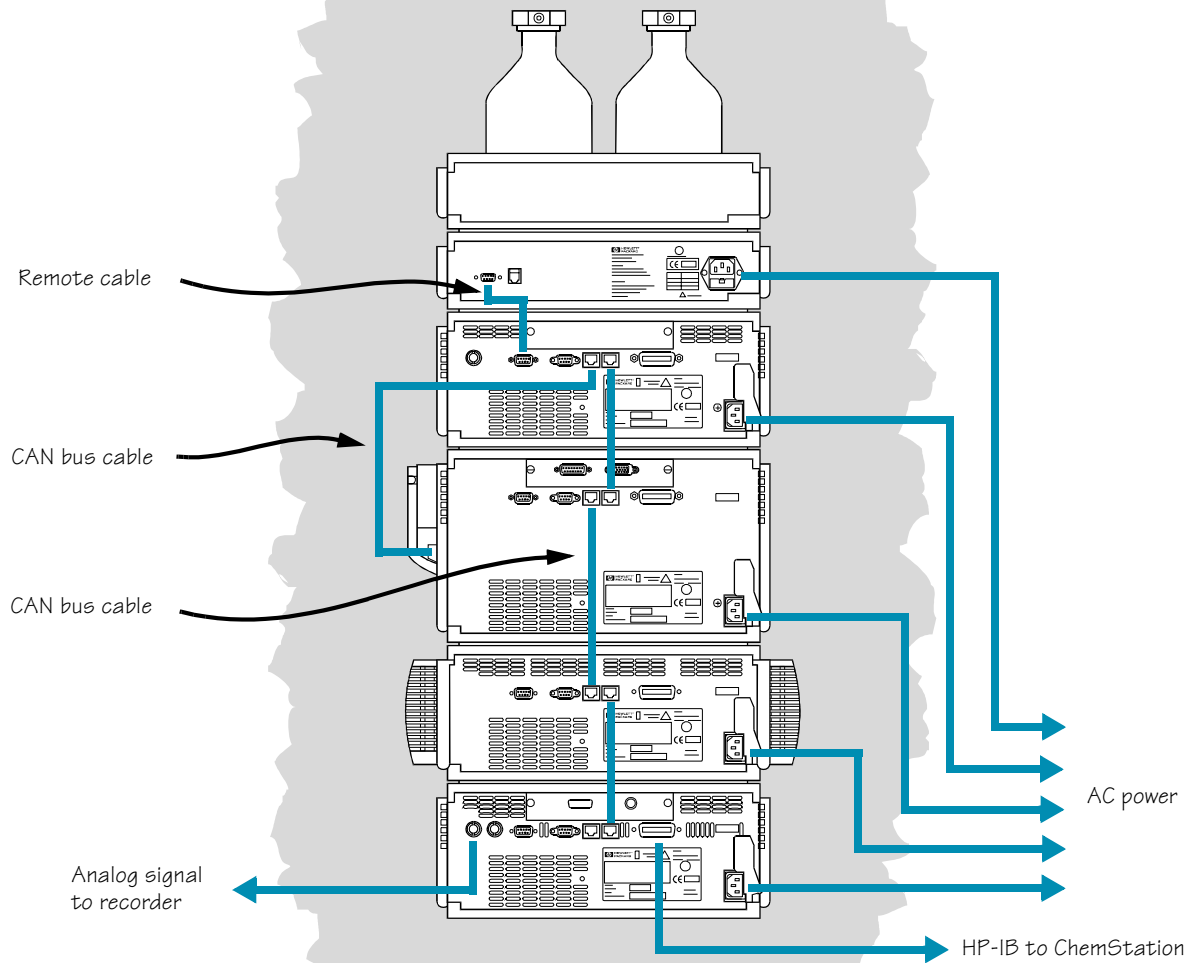
Undesired fragmentation

- Fragmentor is set too high.
- Ionization is causing fragmentation (APCI vs. Electrospray).
- APCI temperature is too high.

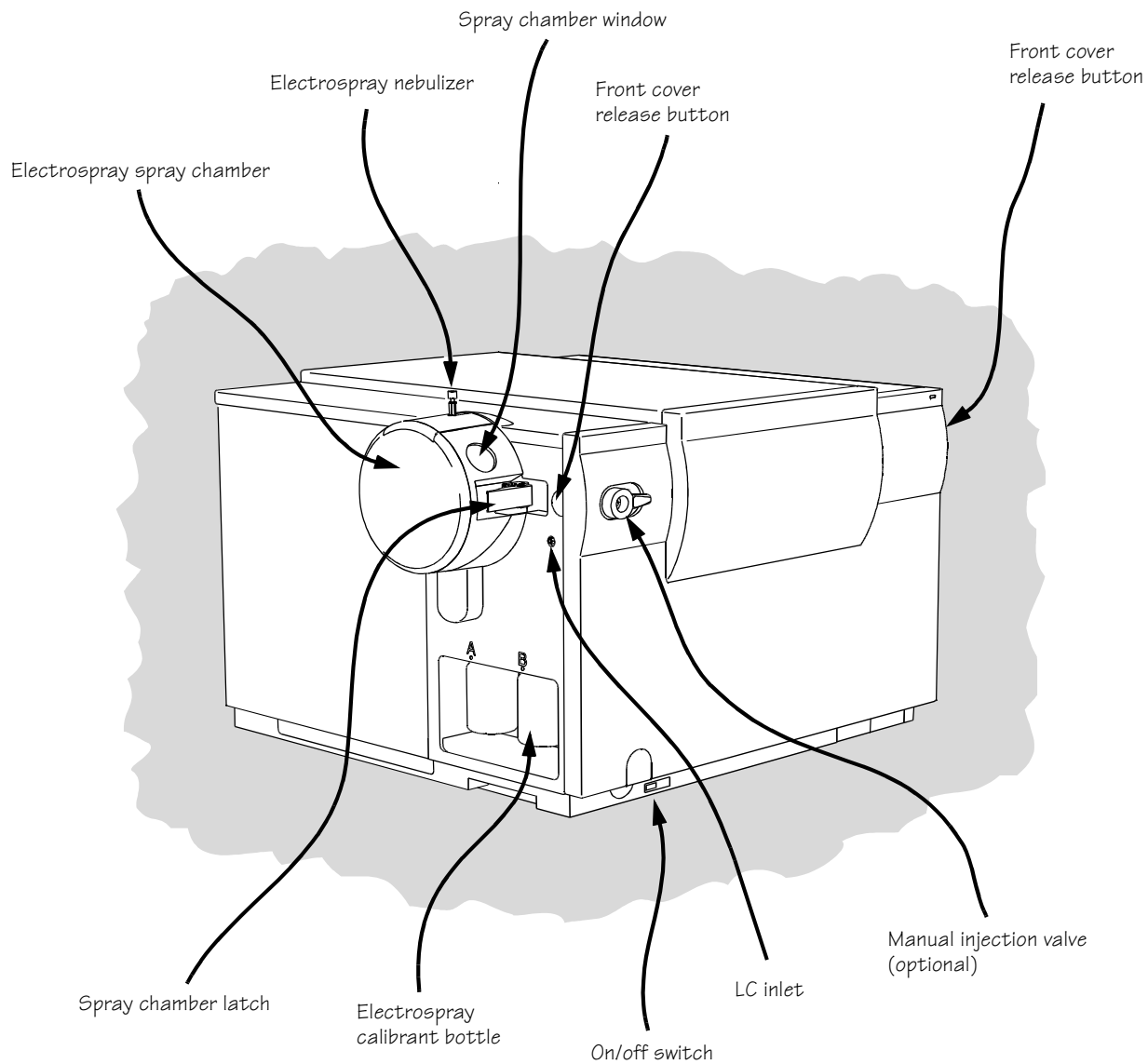
HP 1100 Series LC



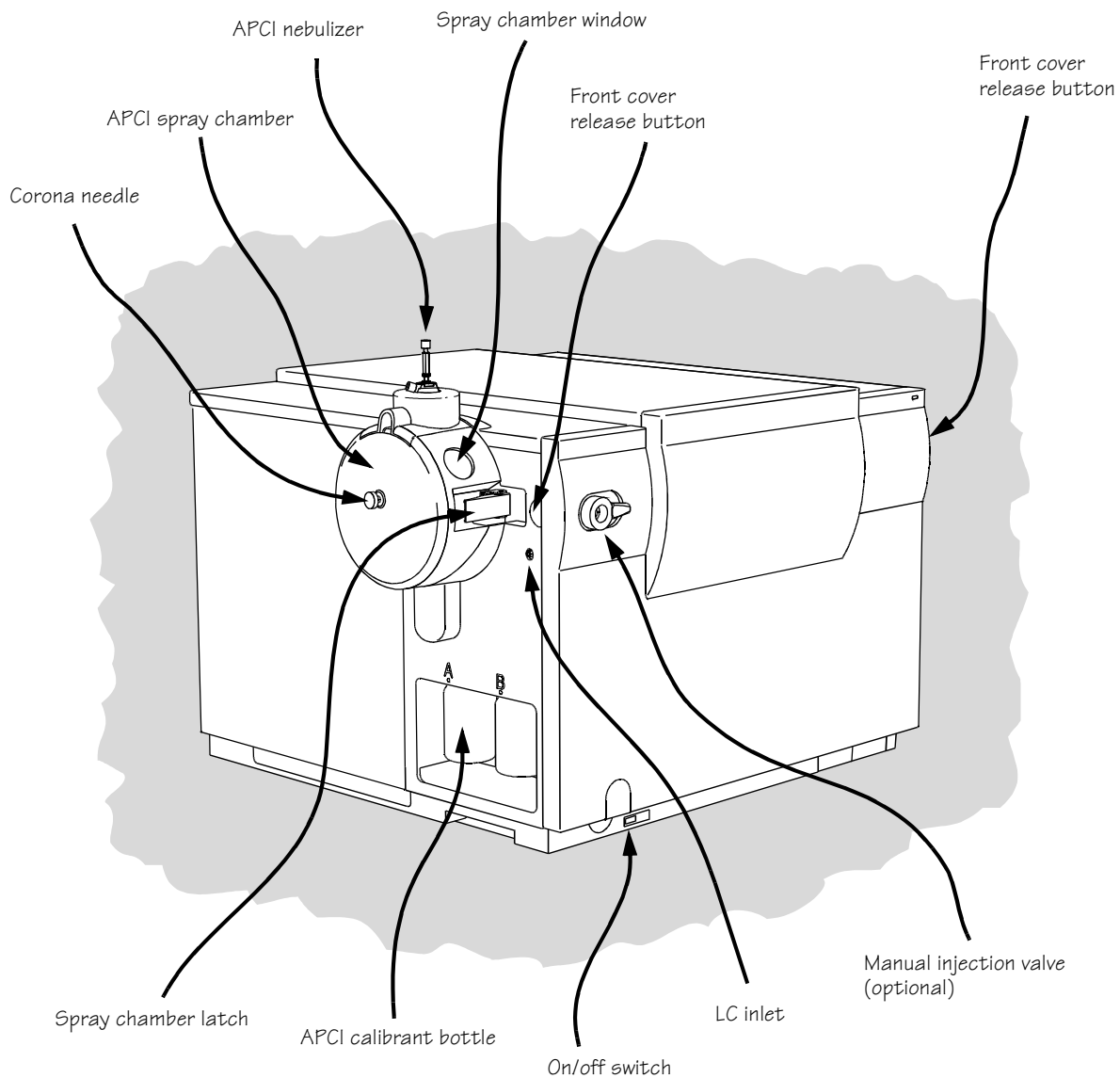
LC Cabling



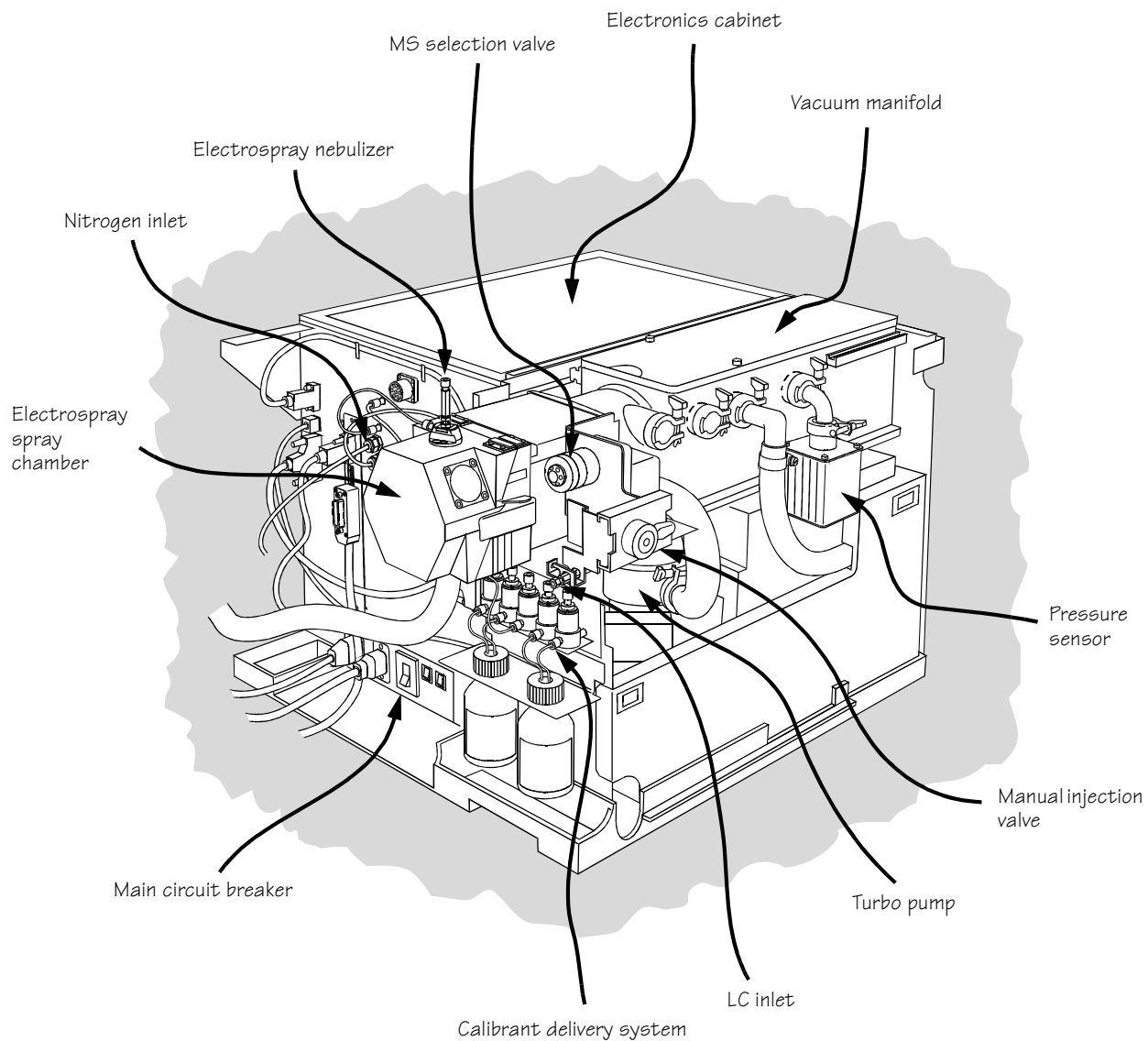
HP 1100 Series LC/MSD — Electrospray



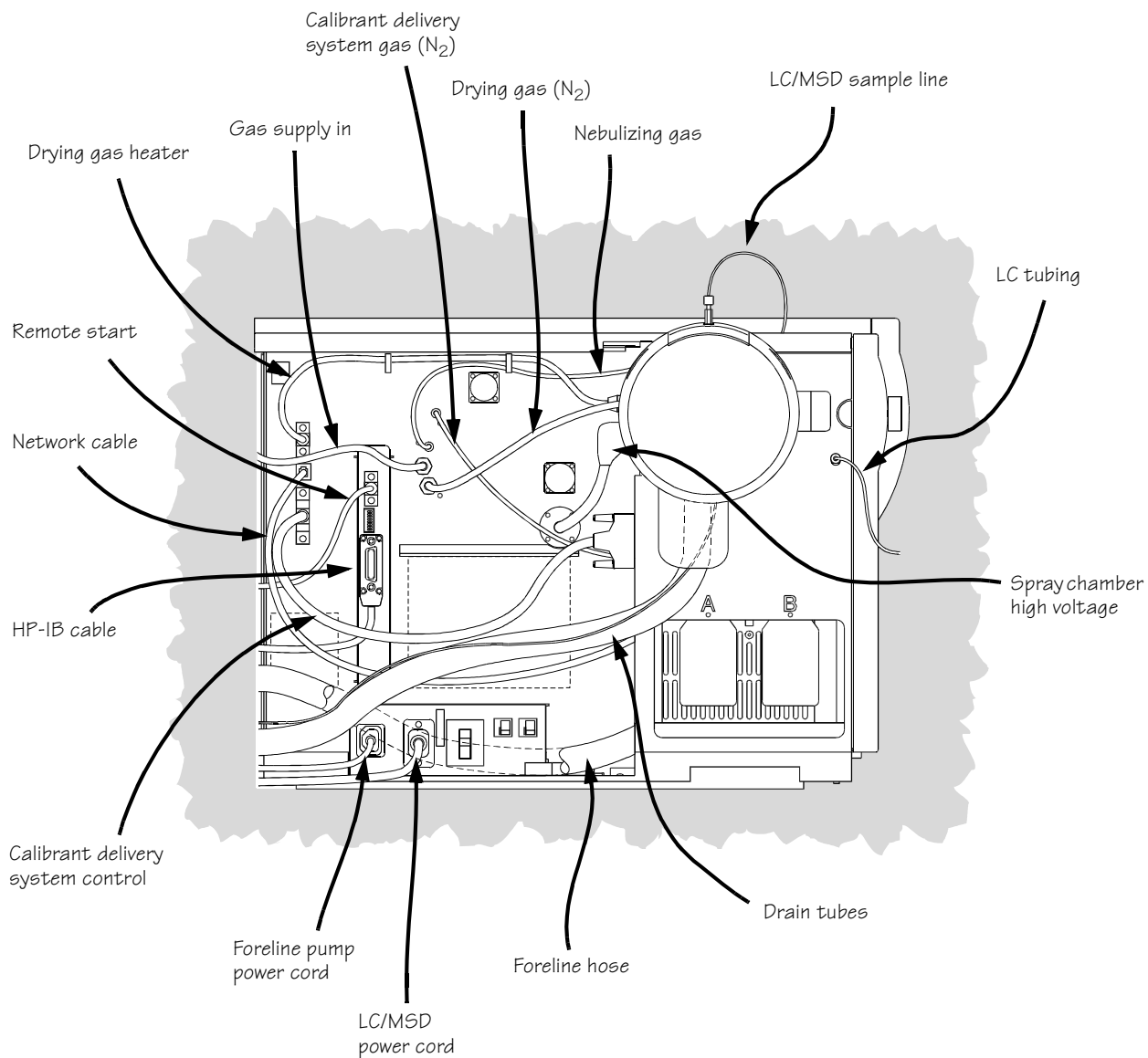
HP 1100 Series LC/MSD — APCI



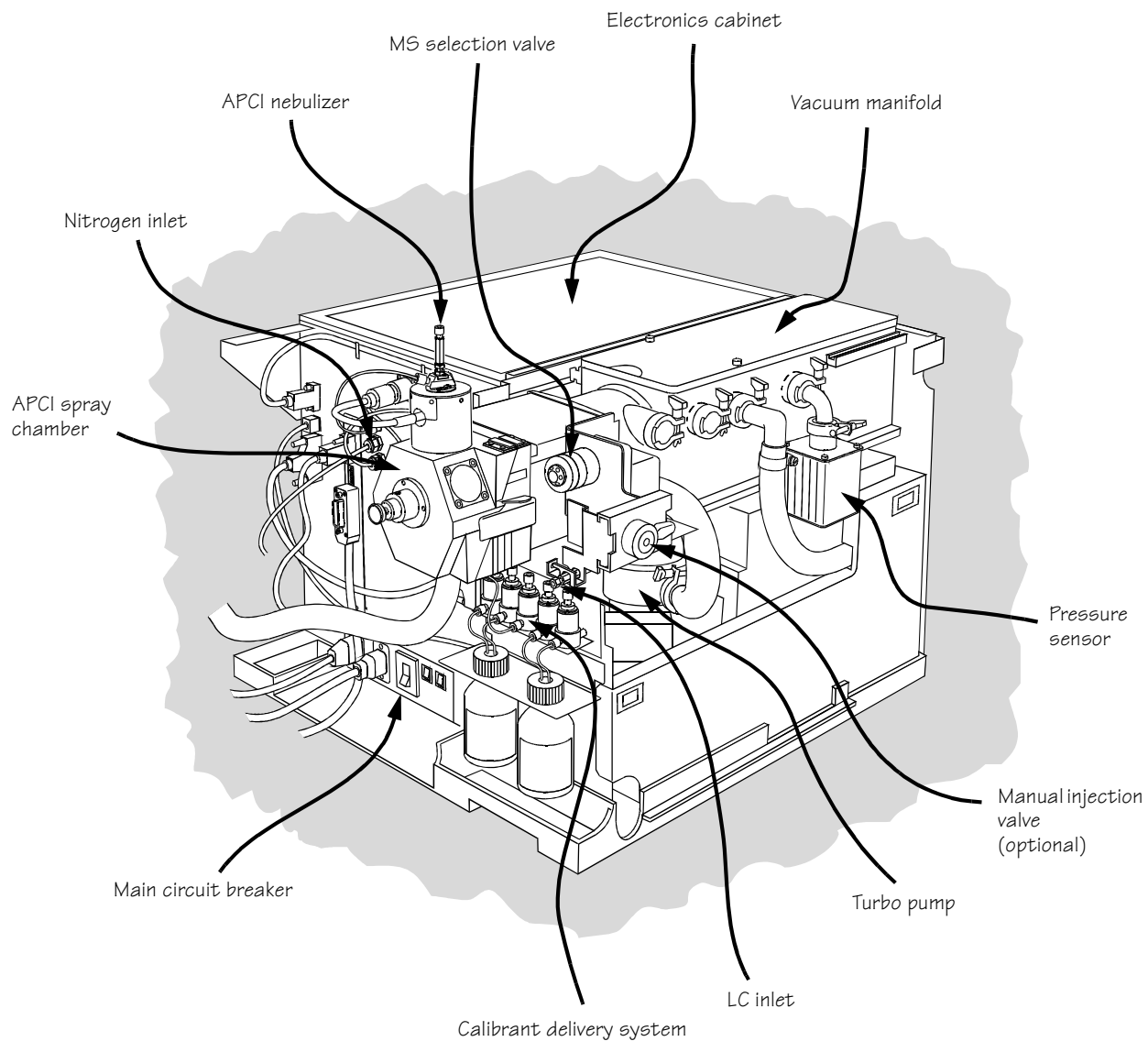
HP 1100 Series LC/MSD — Electrospray (with covers removed)



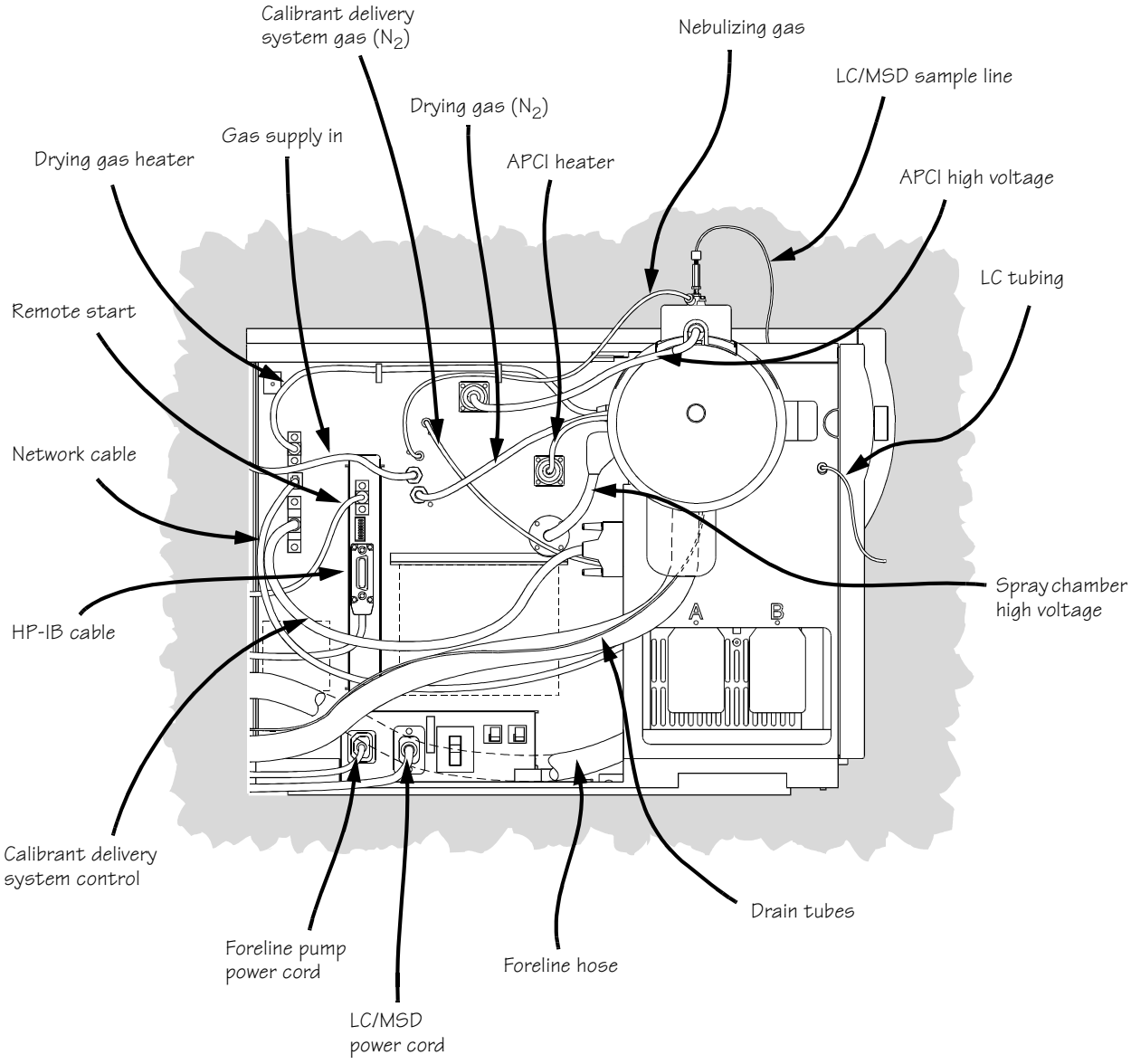
LC/MSD Cabling — Electrospray (side view, door open)



HP 1100 Series LC/MSD — APCI (with covers removed)



LC/MSD Cabling — APCI (side view, door open)



Commonly Used Parts — LC/MSD

Description	HP Part Number
<i>Chemical Consumables</i>	
Calibrant, Electrospray	G2421A
Calibrant, APCI	G2422A
Evaluation Sample, APCI Negative Mode	G2425A
Evaluation Sample, ES Negative Mode	G2424A
Evaluation Sample, Positive Mode	G2423A
Myoglobin Standard (Multiply-charged)	G2426A
OQ/PV Chemical Kit (Caffeine)	8500-6917
<i>Instrument Consumables</i>	
Capillary	G1946-80009
Corona Needle, APCI	G2429A
Electron Multiplier Replacement Horn	05971-801
Ferrule, Nebulizer Needle	G1946-20213
Fitting, F120 (Fingertight PEEK Fitting)	0100-1920
Front Capillary Bal Seal, 1/4" ID	0100-1516
Manual Sample Injector	0905-1475
Nebulizer Needle, Electrospray	0101-0941
Nebulizer Needle, APCI	G2427A
Nitrogen Gas Conditioner	G1946-80047
Tubing (PEEK) 0.005" ID, 1/16" OD	5182-3441
Vacuum Pump Fluid	0890-1915 6040-0834
<i>General Supplies</i>	
Abrasive Mesh (micro-grit paper)	8660-0852
Cloth, Clean	05980-60051
Nitrile Rubber Glove, Medium	9300-1751

Safety Warnings

Intended Purpose

Use of the HP 1100 Series LC/MSD in a manner not intended by the manufacturer is prohibited.

Safety Class

The HP 1100 Series LC/MSD is a Safety Class 1 instrument and has been designed and tested in accordance with IEC Publication 1010-1, Safety Requirements for Electrical Equipment for Measurement, Control, and Laboratory Use.

Stack Configuration

WARNING

While it is possible to stack the HP 1100 Series LC modules on top of the LC/MSD without damaging it, this arrangement is potentially unstable and dangerous and is not recommended.

Placing the LC modules on top of the LC/MSD is also inconvenient. It puts the solvent bottles and some LC controls out of easy reach and requires the LC stack to be disassembled and removed for LC/MSD maintenance.

WARNING

Nitrogen is the only acceptable drying and nebulizer gas. Use of air, oxygen, or other gases, when combined with solvents and high voltages in the spray chamber, could result in an explosion.

Safety Symbols



Refer to operating instructions.



Indicates hazardous voltages.



Indicates hot surfaces.



Indicates earth (ground) terminal.

WARNING

Connecting the LC/MSD to a power source that is not equipped with a protective earth contact creates a shock hazard for the operator and can damage the instrument. Likewise, interrupting the protective conductor inside or outside the LC/MSD or disconnecting the protective earth terminal creates a shock hazard for the operator and can damage the instrument.

WARNING

Any adjustment, maintenance or repair of the opened instrument while it is connected to a power source should be avoided and, if required, should be performed only by trained persons who are aware of the hazards involved.

WARNING

The power switch on the front of the LC/MSD does not completely cut power to the instrument. You must also turn off the main circuit breaker and disconnect the power cord before performing maintenance on the electronics or turbo controllers.

Document History

First Edition, 5/98
HP G2710 LC/MSD ChemStation Software,
Rev. A.06.01 and later until superseded



Printed on recycled paper



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