

# Chapter

## Optimizing the MS Detector with Your Analyte in APCI/MS Mode

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This chapter provides information on optimizing the tune of your MS detector in the APCI/MS high flow mode. It is not necessary to recalibrate the MS detector when you switch to APCI/MS operation. You can use the calibration settings you obtained from the successful automatic calibration procedure you performed in the ESI/MS mode.

For APCI/MS operation you simply open a default tune method located in your *C:\Xcalibur\methods* directory, in this case *APCIhighflow.LCQTune*. From this starting point, you optimize automatically the tube lens offset voltage, capillary voltage, and ion transfer capillary temperature for your particular analyte.

**Note.** The following procedures assume that you are familiar with your LCQ Advantage instrument and the Tune Plus window. If you need information, refer to the LCQ Advantage online Help, *LCQ Advantage Getting Connected*, and/or *LCQ Advantage Hardware Manual*.

Ensure that you have completed the procedures in the topics **Tuning and Calibrating Automatically in the ESI/MS Mode** and **Setting Up to Acquire Data in the APCI/MS Mode**.

This chapter includes the following topics:

- Optimizing the Tune of the MS Detector Automatically in APCI/MS Mode
- Saving the APCI/MS Tune Method
- Cleaning the Spray Shield after Optimization

## 7.1 Optimizing the Tune of the MS Detector Automatically in APCI/MS Mode

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You can optimize the tune of the MS detector automatically for APCI operation.

The most important parameters that affect the signal quality during APCI/MS operation are the vaporizer temperature, ion transfer capillary temperature, capillary voltage, tube lens offset voltage, gases, and solution flow rate. If any one of these parameters is changed, you need to reoptimize MS detector parameters. (You can use the Semi-Automatic tune procedure to tune the MS detector on individual parameters.)

Use the following procedure to optimize the MS detector automatically on the reserpine peak at  $m/z$  609.2 at your particular flow rate, for example, 1 mL/min. (Refer to Table 1-2 for guidelines about flow rates and temperatures.)



1. Display the automatic tuning page in the Tune dialog box, as follows: Click on the Tune button on the Control/Scan Mode toolbar. Then, click on the Automatic tab.



2. Specify that you want to tune on the peak at  $m/z$  609.2, as follows: Double-click on the Mass spin box, then type **609.2**.
3. Ensure that the Divert/Inject valve is in the Detector position, as follows
  - a. Click on the Divert/Inject button to open the Divert/Inject Valve dialog box.
  - b. Select the Detector option button.
  - c. Click on **Close**.

4. Start the automatic tuning procedure from the Tune dialog box, as follows:
  - a. Click on **Start**. A message box displays the following message:  
*Please ensure that the 500 microliter syringe is full.*  
Ensure the syringe pump contains at least 420  $\mu\text{L}$  of the 1 ng/ $\mu\text{L}$  reserpine tuning solution.
  - b. Close the message box, and return to the Tune Plus window: Click on **OK**.



5. Display the Graph view, as follows: Click on the Graph View button in the File/Display toolbar.
6. Observe the Tune Plus window and the Tune dialog box. While automatic tuning is in progress, LCQ Advantage displays various tests in

the Spectrum and Graph views in the Tune Plus window and displays various messages in the Status group box in the Tune dialog box. Your Tune Plus window should now look similar to the one shown in Figure 7.2.

You have now successfully tuned the MS detector in APCI/MS mode for the compound reserpine (or your analyte of interest). Leave the LC pumps on (with a flow rate of approximately 1 mL/min), and leave the Tune Plus window open with *APCIhighflow.LCQTune* to complete the next topic: **Saving the APCI/MS Tune Method.**

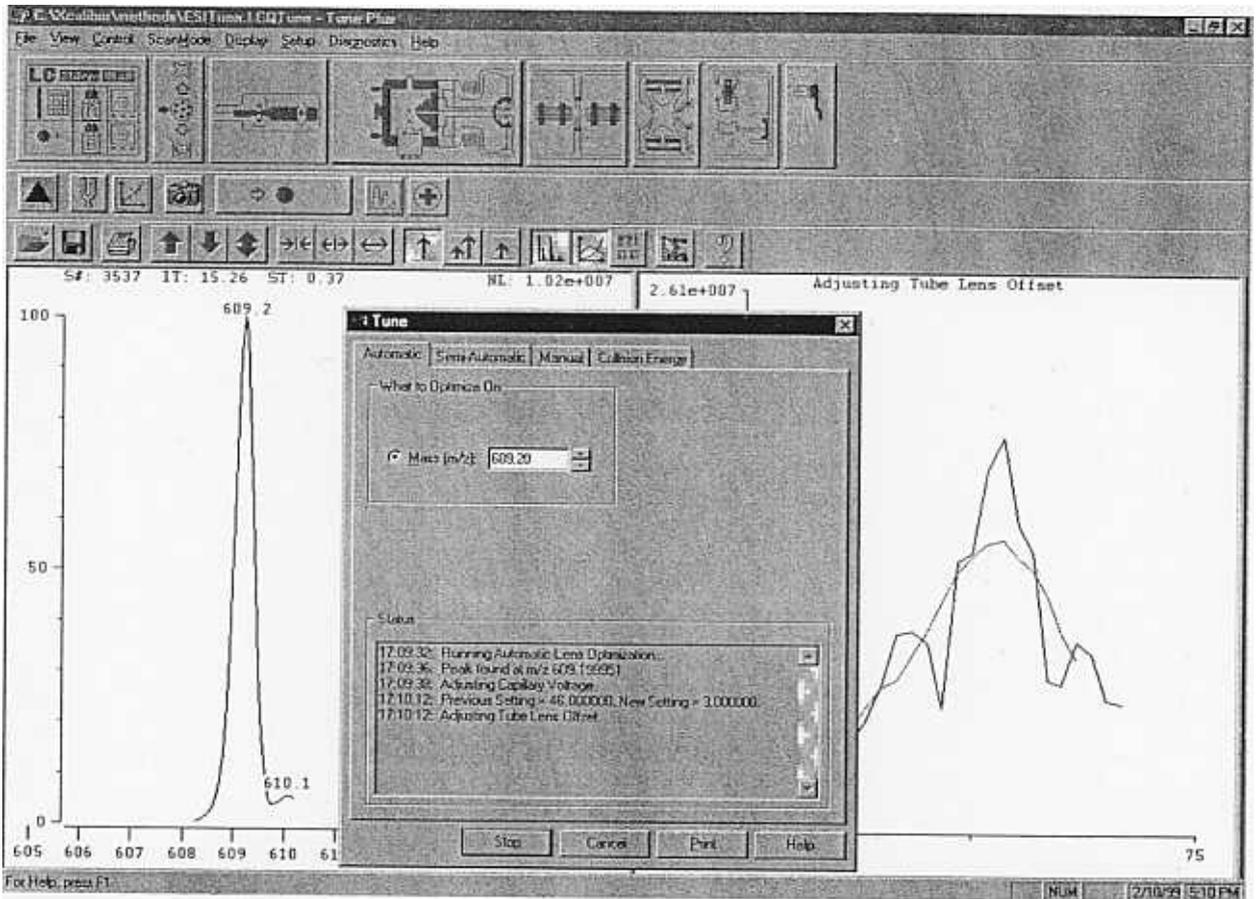


Figure 7-1. Tune Plus window with the Tune dialog box, showing the Automatic page

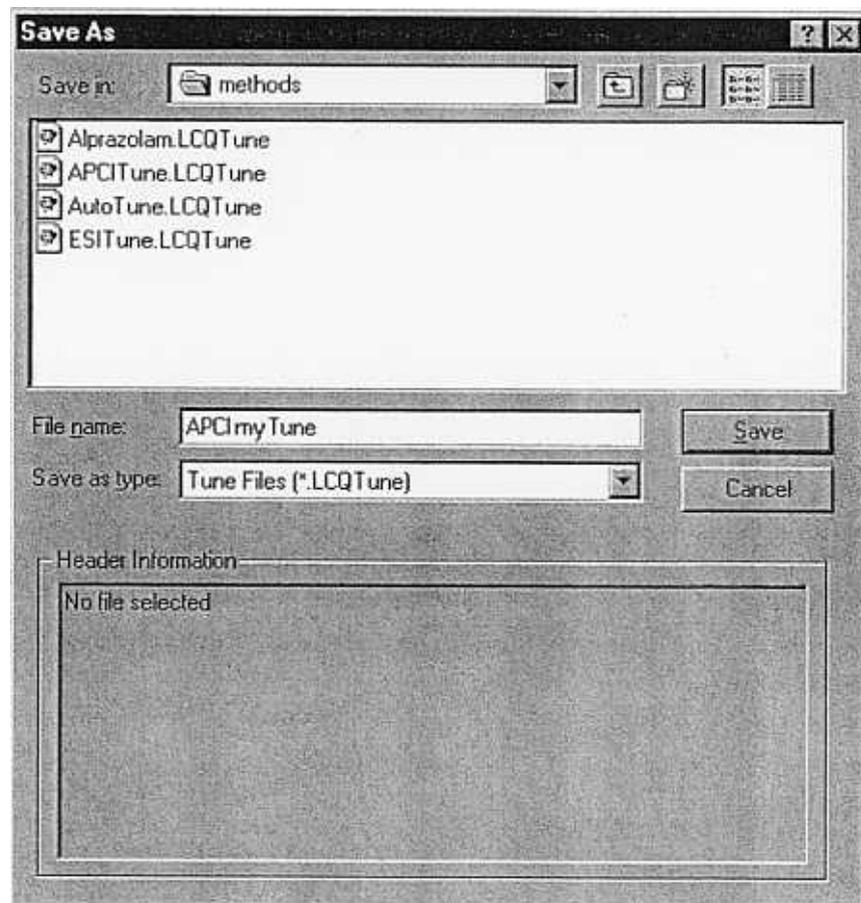
## 7.2 Saving the APCI/MS Tune Method

You can save the settings you just obtained in a tune method specific to your particular analyte and solvent flow rate. (In this case, you save settings obtained using reserpine.) You can recall the tune method and use it as a starting point for optimizing the MS detector on reserpine at a different flow rate.

**Note.** Save the Tune Method while the MS detector is On.

Save your APCI/MS tune method, as follows:

1. Display the Save As dialog box, as follows:  
Choose **File | Save As**. See Figure 7-2.



**Figure 7-2.** Save As dialog box, showing files in the directory *C:\Xcalibur\methods*

2. Select the *C:\Xcalibur\methods* directory.
3. Name the Tune Method, as follows:  
Click on the File Name text box, and type **APCImyTune**.
4. Save the Tune Method, close the dialog box, and return to the Tune Plus window, as follows:  
Click on **Save**. Note that the Tune Method is named *APCImyTune.LCQTune*.

Before you acquire data, go to the next topic: **Cleaning the MS Detector after Tuning in APCI Mode**.

## 7.3 Cleaning the MS Detector after Tuning in APCI Mode

Use the following procedure to clean the MS detector after tuning on your analyte of interest.



On

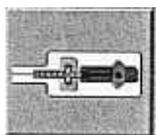


Standby

1. Put the MS detector in Standby, as follows:  
Click on the On/Standby button. When the MS detector is in Standby, LCQ Advantage turns Off the vaporizer heater and the corona discharge voltage. The MS detector stops scanning, and LCQ Advantage freezes the displays for the Spectrum and Graph views.



**CAUTION.** Always place the MS detector in Standby (or Off) before you open the API source to atmospheric oxygen. The presence of oxygen in the ion source when the MS detector is ON could be unsafe. (LCQ Advantage automatically turns the MS detector Off when you open the API source, however, it is best to take this added precaution.)



2. Turn Off the syringe pump to stop the flow of solution into the ion source, as follows:
  - a. Display the Syringe Pump dialog box, as follows:  
Click on the Syringe Pump button.
  - b. Select the Off option button in the Flow Control group box to turn off the syringe pump.
  - c. Click on **Close**.
3. Remove the syringe from the syringe holder, as follows:
  - a. Pull back on the silver syringe handle to free the syringe.
  - b. Remove the syringe from the holder.
  - c. Disconnect the tip of the syringe needle from the Teflon tubing.
4. Clean the syringe thoroughly, as follows:  
First, clean the syringe with a solution of 5% acetic acid in water. Then, rinse the syringe with a solution of 50:50 methanol:water. Finally, rinse the syringe with acetone.
5. Loosen completely the two APCI flange retainer bolts.
6. Slide the APCI flange out, away from the MS detector chassis, on the slide rails.

7. Flush the sample transfer line, sample tube, and APCI probe thoroughly with a solution of 5% acetic acid in water (or with another appropriate solvent), as follows:

**Note.** The solvent that you use to flush the sample transfer line, sample tube, and APCI probe assembly depends on the solvent system you use to dissolve your samples. For example, if you are using a buffered solution of a high concentration, an acidic solution is appropriate.

- a. Fill a clean, 250  $\mu$ L Unimetrics syringe with an appropriate solvent.
  - b. While holding the plunger of the syringe in place, carefully insert the needle of the syringe into the free end of the Teflon tube.
  - c. Flush the sample transfer line, sample tube, and APCI probe with the solution by slowly depressing the syringe plunger. Visually check that the solution is exiting the tip of the APCI probe on the inside of the probe assembly. Use a lint-free tissue to gently remove the excess solution as it exits the probe.
  - d. Remove the needle of the syringe from the Teflon tube.
8. Repeat step 7 with a solution of 50:50 methanol:water.
  9. Slide the APCI flange forward along the slide rails and into the ion source housing.
  10. Secure the APCI flange with the two flange retainer bolts.

If you plan to run analytical samples in APCI mode, go to the next chapter: **Acquiring APCI Sample Data Using the Tune Plus Window.**