Varian, Inc.
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4000 GCMS SYSTEM

Installation Procedures

Contents

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Introduction

This installation procedure represents a stepwise guide to setting up the 4000 GCMS system, meeting product specifications, and carrying out basic customer familiarization.

The 4000 MS has several modes of operation. It is not desirable to test all of these modes at installation. The customer needs to decide what will be the standard mode or preferred mode of operation when the system is sold. The system will be shipped in the preferred mode. Install and test the system using the preferred mode. Additional ionization mode demonstrations may be sold. If so, these ionization modes should also be demonstrated. Under normal circumstances, the 4000 MS installation should be complete within 2-3 days on site. Demonstration of additional ionization modes will increase the time needed to install a system.

A Typical MS Installation

1. System unpacking, setup and checking	2 hours
2. Power up, pump down, initial customer familiarization	4 hours
3. Verification of GC/MS System Operation	2 hours
4. EI ionization mode demo	1 hours
5. Additional customer familiarization	4 hours

Option Installation

1. Autosampler installation, check out, familiarization	1.5 hours
2. CI ionization mode demo, familiarization	1.5 hours

Installation results should be entered into the Installation Tracking Database.

The following should be determined before arriving at an installation site:

The CSR should be aware of whether the customer has met the important site requirements outlined in the Pre-Installation Document. **A phone call should be placed with the customer to pre-qualify the site.** The critical site requirements are bench space and load, power, temperature, humidity, carrier gas purity (helium) and CI gas purity (external only, methane). The specific requirements are clearly outlined in the 4000 Pre-Installation Document.

It is also important to determine, before going on site, whether the customer desires to use the existing system PC or the PC included with the MS upgrade. **If the customer provides the PC, then the set up of this computer should be a billable call, and not charged to installation.** In addition, Varian assumes no liability for configuration or operational problems associated with the customer's computer.

NOTE: Test samples are not shipped overseas with the system. Before going on site to do an installation, ensure you have all the necessary samples, or that they have been shipped to the customer from a local Varian office. Installation engineers outside of the USA should confirm the availability of test samples from their home office as much as 2 weeks in advance. For US shipments the samples are shipped with the system.

Section 1. System Unpacking / Checking / Set Up

1.1 Unpacking / Parts Checking

Unpack the entire system, and check thoroughly for missing parts and shipping damage. Use caution when unpacking the MS - the transfer line is not a handle, **lifting the instrument using the transfer line can result in damage to the analyzer**.

NOTE: The Cal Gas (FC-43 P/N 03-920353-00) is shipped uninstalled in an ampule. The Cal Gas must be installed during the installation. Aliquot approximately 0.5 mL of the FC-43 into the Cal Gas vial and install into the MS. The remaining FC-43 can be stored in the vials provided in the accessory kit.

Do not overfill the Cal Gas vial installed in the MS. Overfilling can result in FC-43 contamination that interferes with Cal Gas adjustments.

Before setting up and starting the GC/MS system, check that all parts and kits have been delivered, and are complete. Review with the customer the contents of the accessory kits and their functions.

Report all shipping damage or missing parts to the Customer Service department (Phone: 925-939-2400, Fax: 925-945-2360, email: orders.csb@varianinc.com), Varian, 2700 Mitchell Drive, Walnut Creek, CA, 94598.

Shipping damage or missing items should also be noted on the installation checklist and in the Installation Tracking Database.

1.2 System Set Up On Bench

Set the 3800 GC and 4000 MS onto the bench. The GC and MS should initially be well separated to allow easy access. Remove the turbo pump shipping restraint and analyzer shipping restraints from the 4000 MS. Remove shipping restraints from the GC such as the metal dowel at the rear of the GC. The removed restraints and brackets should be retained in case the system needs to be transported in the future.

Connect the roughing hose to the roughing pump. Connect the roughing pump power cord to the LINE VOLTAGE PUMP ONLY connector at the rear of the MS. To minimize vibration, the roughing pump should not be on the bench with the GCMS.

Connect the GC-to MS sync cable under the GC top cover at J6 ANALOG OUT and at the rear of the MS at J9 SYNC.

Connect the USB cable to the rear of the MS at J7 USB and to a USB port on the PC.

Connect the Ethernet cable to the GC and to the PC. Be sure to connect the Ethernet cable using the side connector of the Tee and NOT the terminal end.

Install the new column shipped with the system into the 1177 injector (or 1079 if no 1177 is installed) but to prevent contamination do not connect to the MS transfer line until after initially conditioning the column in the GC oven.

Uninstall the empty Cal Gas vial from behind the MS front door by loosening the two mounting screws and twisting the vial while pulling down. Fill the vial with approximately 0.5 mL of the FC-43 (03-920353-00) provided in the 4000 MS Accessory Kit. Re-install the partially (less than half) filled vial into the MS and tighten the mounting screws. The vial must be inserted far enough into the pneumatic block to engage the o-ring. Store the remaining FC-43 in the vials provided in the accessory kit. Be careful not to overfill the Cal Gas vial to avoid FC-43 contamination that can interfere with system performance.

1.3 GCMS Plumbing and Purging

Use 1/8” brass tubing to plumb 99.995% helium to the back of the GCMS system. Install the Gas Clean Filter assembly. For an external mode 4000 MS, Tee the helium output line after the Gas Clean Filter assembly so helium can be delivered to the GC and the MS. The 4000 MS (in External Mode only) uses helium Damping Gas that will need to be plumbed into the MS. This MS Damping Gas is typically installed by teeing off the helium line to the GC and MS after the Gas Clean Filter assembly. Verify that the helium line pressure is set to 80 psi. at the cylinder regulator. Ensure the cylinder contains at least 500 psi. of helium. Before connection to the GC/MS, purge the helium line from the cylinder for 10 minutes at maximum flow. Connect one end of the Tee to the inlet on the back of the GC, the other to the inlet on the back of the MS. The GC pneumatics need to purge for at least 1/2 hour to remove any water or air that may have entered during shutdown.

NOTE: The MS pneumatics (**in External Mode**) will also need to be purged as described in Section 2 of this document

The column shipped with the system must be installed to achieve the best results for acquisitions associated with the installation. If an 1177 injector is installed on the GC, use the 1177 to pass the sensitivity specifications. Condition the injector and column one time before installing into the MS to prevent column bleed from contaminating the MS. Condition the injector at 230 °C with a high split ratio (e.g., 50:1). Condition the column starting at 50 °C, ramping at 5 degrees/minute up to 300 °C, and hold for 60 minutes unless this exceeds the maximum allowable operating temperature for the column phase.

1.4 Column Installation Into the MS

Column installation into the 4000 MS transfer line depends on the ionization mode of the MS and is described in the 4000 Hardware Operation Manual. Note the ionization mode in which the instrument was shipped and install the column accordingly.

NOTE: Be sure to leave a significant service loop of column detached from the column cage so the GC and MS can be easily separated.

At this point the GC and MS can be pushed together on the bench. Ensure that the transfer line column nut is visible inside the GC oven and the transfer line rubber boot is inside the hole on the GC side panel.

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Section 2. GC/MS Power Up

Ensure that the GC and MS are plugged into separate isolated outlets as specified in the 4000 Pre-installation Document.

2.1 Initial Power Up

Turn on the GC. Open System Control and bring the GC online.

Turn the Service Switch underneath the MS top cover to ON. Turn on the main circuit breaker at the rear of the MS. The mechanical pump will run loudly initially, but should run quietly within 30 seconds. If the mechanical pump continues to run loudly, turn off the main circuit breaker and check the system for obvious leaks. Common sources of large leaks at this point are the vacuum hose connections to the turbo or roughing pumps, the manifold or transfer line O-rings, and the column connection into the transfer line.

Found New Hardware Wizard

Because the PC has not been connected to the MS the Windows XP operating system may launch the Found New Hardware Wizard when first turning on the MS. Configure the MS as new hardware by clicking through the pages of this wizard as shown in the following screen captures.

Select **No, not this time** and click **Next**.

Figure 2.1

Select **Install the software automatically (Recommended)** and click **Next**.

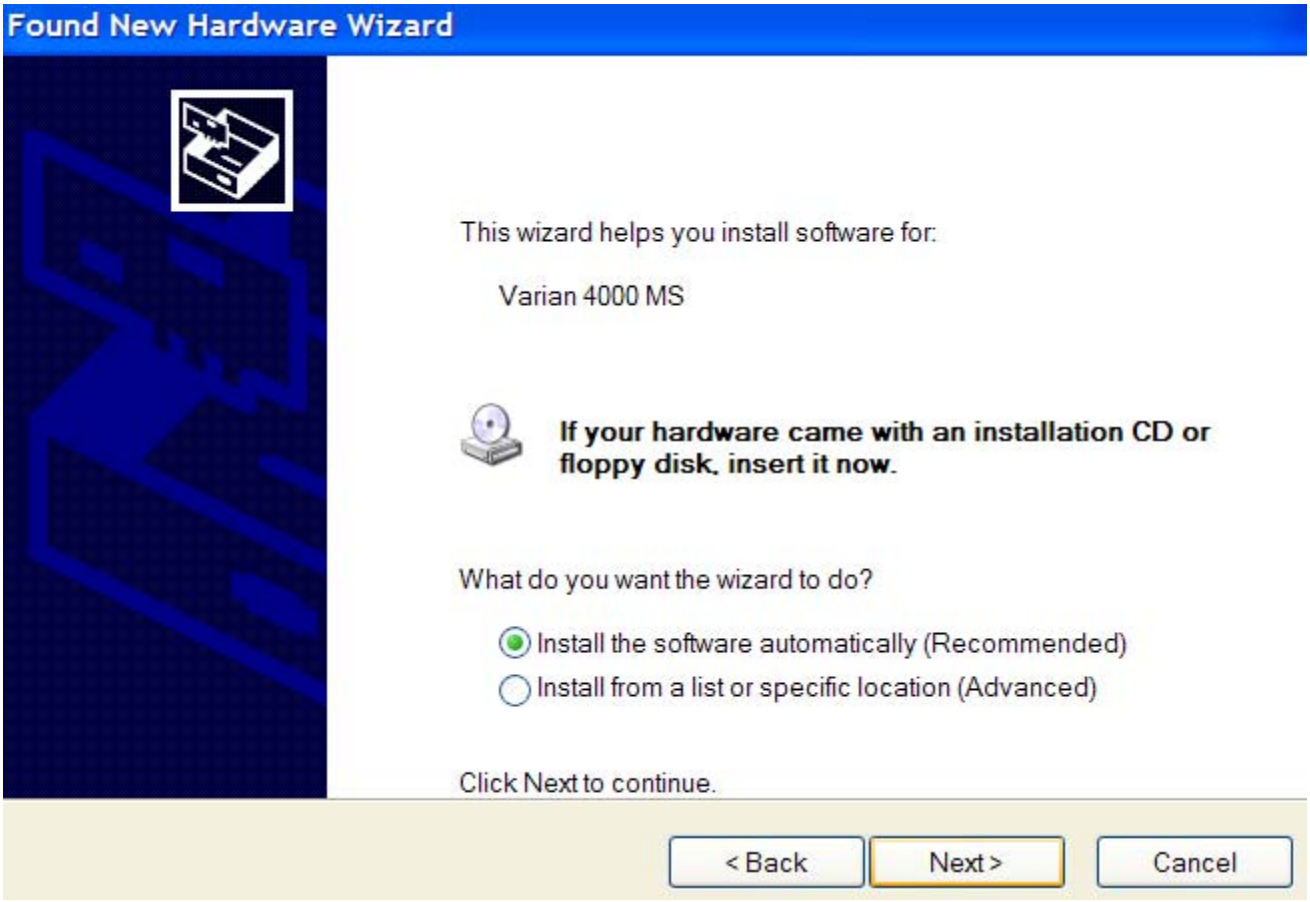

Figure 2.2

Click **Finish** to close the wizard.

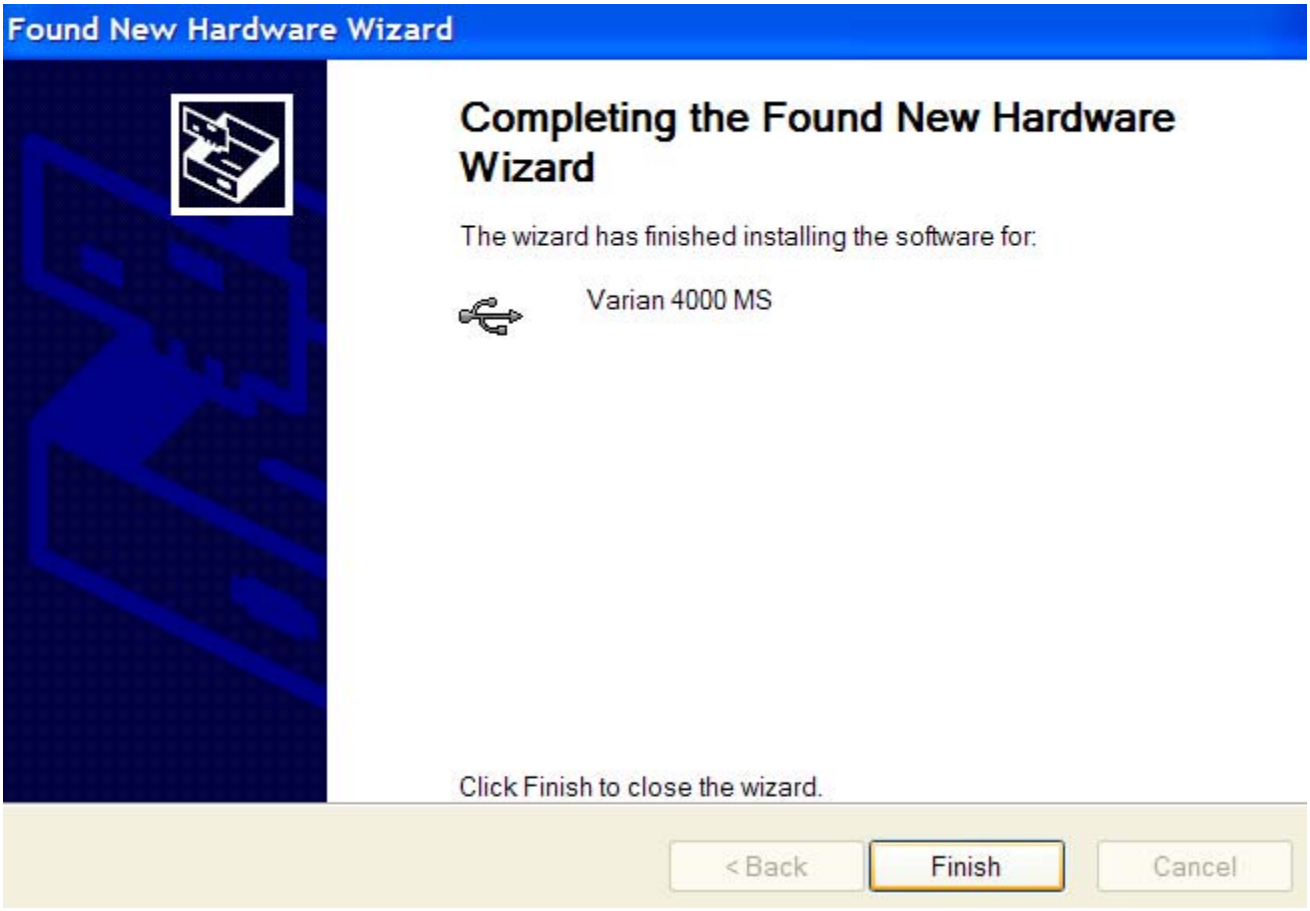

Figure 2.3

Open System Control

Open System Control and if necessary to start the pumps click the Start Up button on the Startup/Shutdown tab. Monitor the Pump Spin Speed and Current from Startup/Shutdown (refer to Figure 2.4). Normally, the turbo should run up to 100% in about 1 minute. Current is usually less than 300 mA when the turbo reaches 100%.

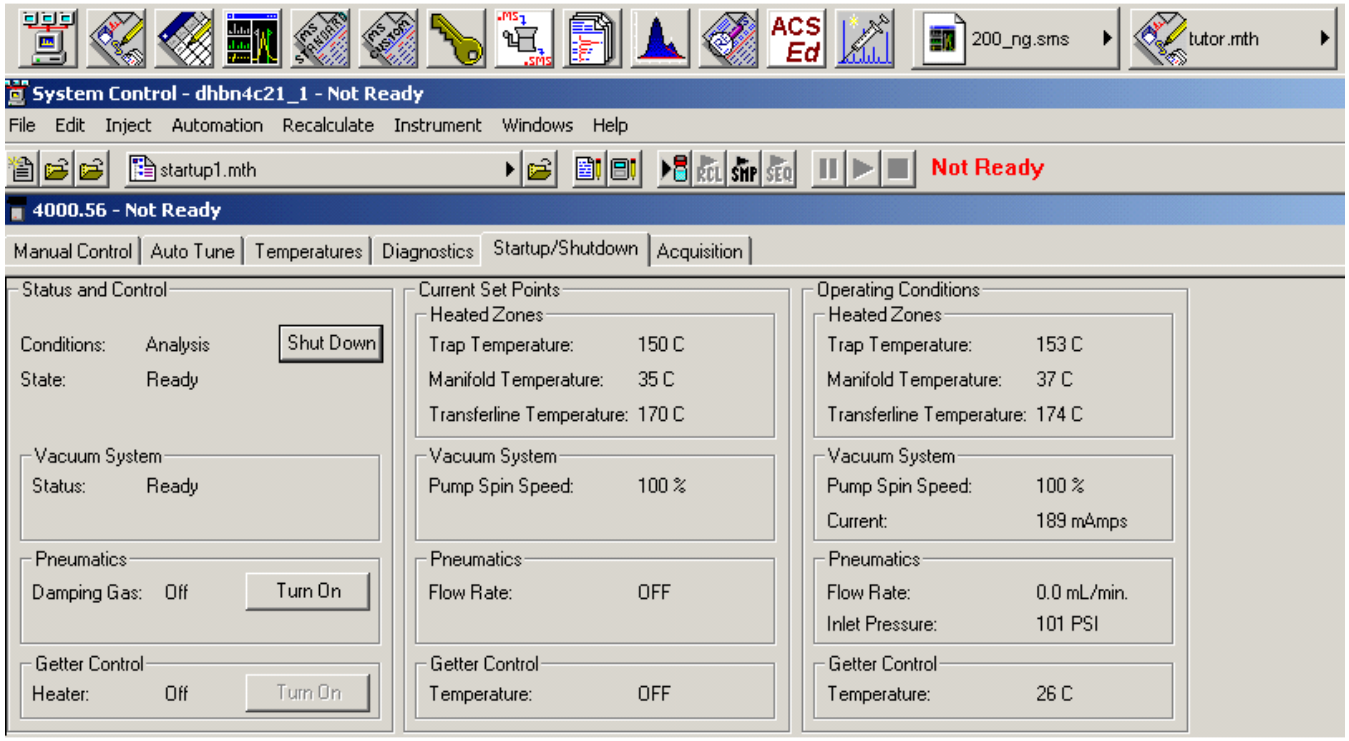

Figure 2.4

4000 MS in External Mode

The 4000 MS in External Mode is equipped with Damping Gas. Once the vacuum is established but before heating up the Getter, the MS pneumatics should be purged by turning on the Damping Gas flow in Startup/Shutdown (refer to Figure 2.4, click Turn On for Pneumatics). Set the Damping Gas Flow to 4 mL/min on the Module Attributes tab in Manual Control (refer to Figure 2.5, enter 4.0 mL/min and click **Activate Changes**) and purge the MS for approximately 20 minutes. After purging the MS pneumatics, set the Trap Damping Gas Flow as appropriate for the external EI acquisition, for the external PCI acquisition, or the external NCI acquisition. The appropriate Damping Gas flow rates can be found embedded in the names of the test data files from the Test Files CD included with the system, and also in the Module Attributes section of the Data File Information dialog of each external data file. After purging and setting the analytical damping gas flow rate, turn on the Getter heater (click Turn On for Getter Control).

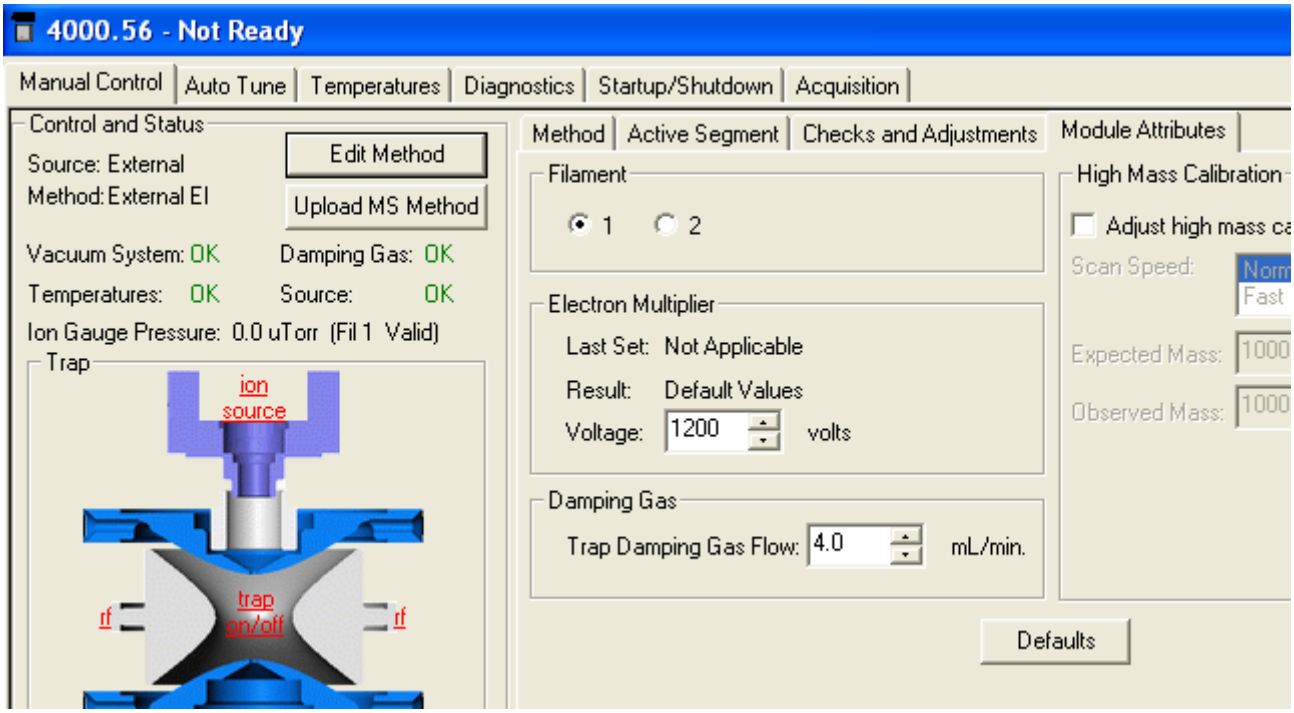

Figure 2.5

2.2 Quick Check of Air / Water

It is important to monitor the progress of the manifold pump down before initiating a bakeout. Once the Pump Spin Speed reaches 100%, check for the presence of a massive water peak (18 and 19 m/z, unlikely to be resolved) and the absence of a nitrogen peak (28 m/z). **Only turn the trap on for a short period of time (<5 seconds) so the filaments and multiplier are not adversely affected.**

NOTE: This check of air water may not give usable results until after auto tuning the system.

Go to Manual Control and click the **Active Segment** tab. Enter a Mass Range of 15 to 45 m/z (refer to Figure 2.6, Full Scan Parameters tab). Also enter a Fixed Ionization Time of 100 microseconds for internal mode or 5000 microseconds for external mode (refer to Figure 2.7, Ionization Control tab). Click **Activate Changes**. Alternatively, one can use segment 1 of C:\VarianWS\4000Service\4000 MS (Int or Ext) Service.mth.

This is a quick check to confirm the absence of a m/z 28 signal. The m/z 28 signal should not be present due to the massive intensity of the 18/19 signal before bakeout. Presence of a m/z 28 signal can indicate a leak or contaminated gas.

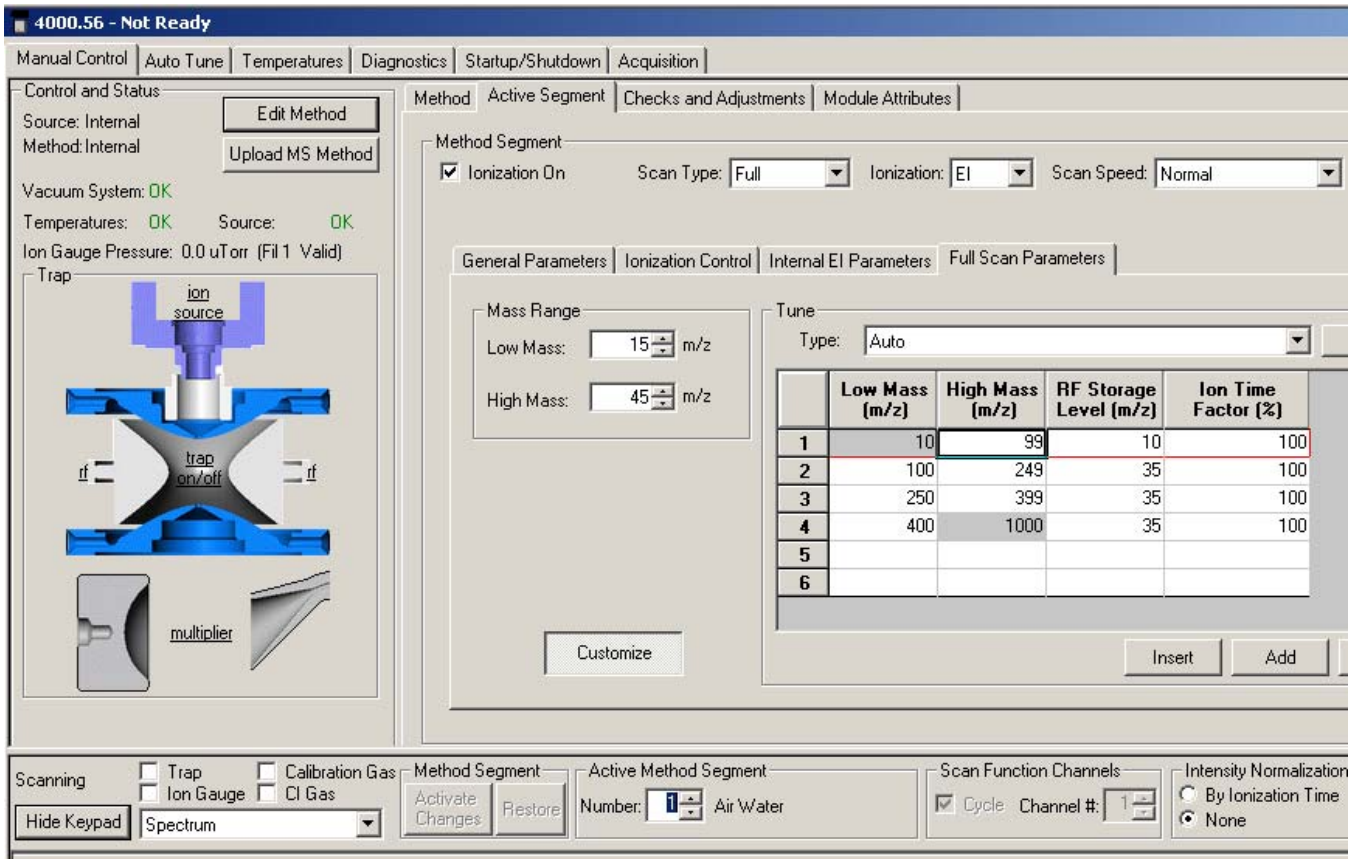
4000.56 - Not Ready
Manual Control
Auto Tune
Temperatures
Diagnostics
Startup/Shutdown
Acquisition
Control and Status
Source: Internal
Method: Internal
Edit Method
Upload MS Method
Vacuum System: OK
Temperatures: OK
Source: OK
Ion Gauge Pressure: 0.0 uTorr (Fil 1 Valid)
Trap
ion source
rf
trap on/off
rf
multiplier
Method
Active Segment
Checks and Adjustments
Module Attributes
Method Segment
Ionization On
Scan Type: Full
Ionization: EI
Scan Speed: Normal
General Parameters
Ionization Control
Internal EI Parameters
Full Scan Parameters
Mass Range
Low Mass: 15 m/z
High Mass: 45 m/z
Tune
Type: Auto
Low Mass (m/z)
High Mass (m/z)
RF Storage Level (m/z)
Ion Time Factor (%)
1 10 99 10 100
2 100 249 35 100
3 250 399 35 100
4 400 1000 35 100
5
6
Customize
Insert
Add
Scanning
Trap
Ion Gauge
Calibration Gas
Cl Gas
Hide Keypad
Spectrum
Method Segment
Activate Changes
Restore
Active Method Segment
Number: 1
Air Water
Scan Function Channels
Cycle
Channel #: 1
Intensity Normalization
By Ionization Time
None

Figure 2.6

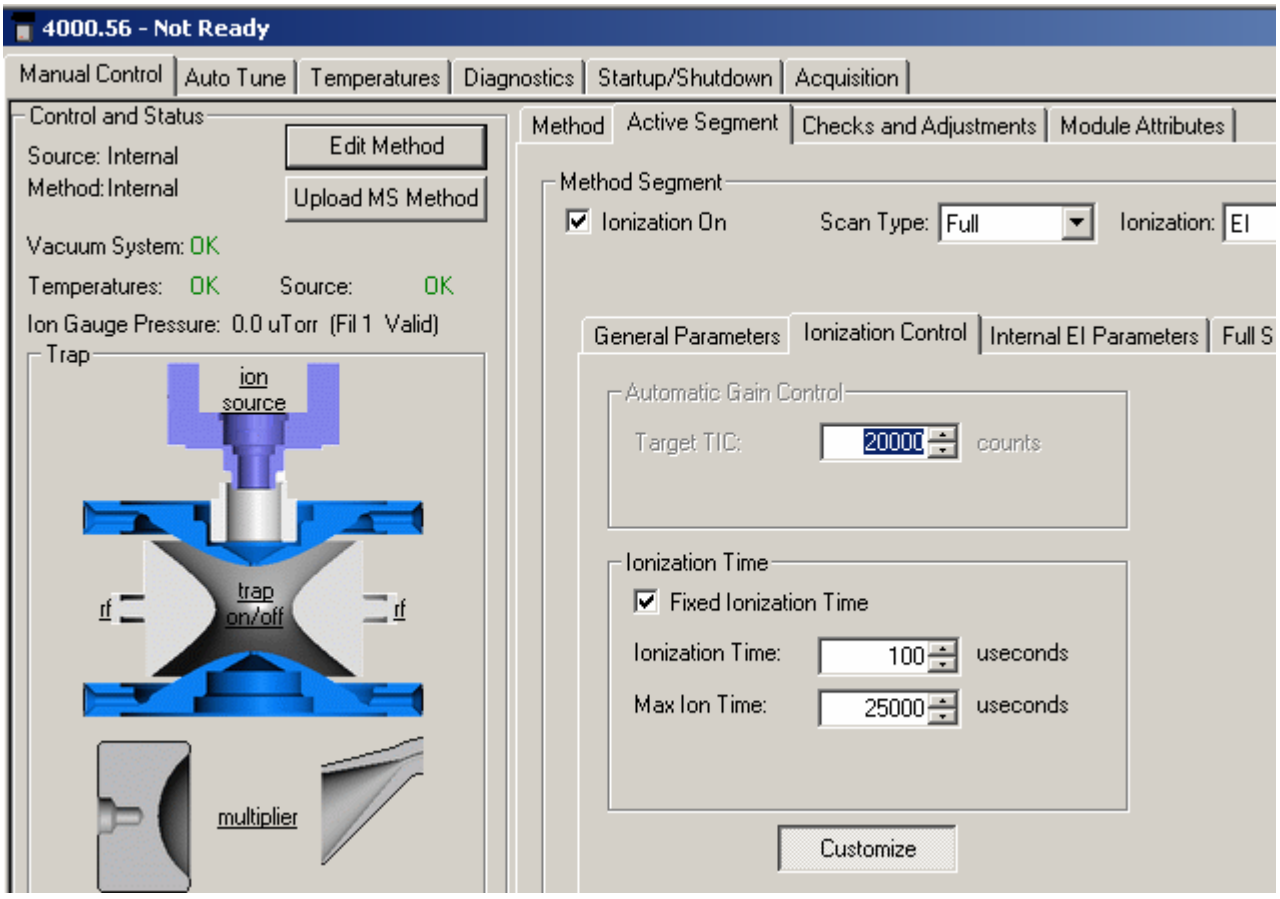
4000.56 - Not Ready
Manual Control
Auto Tune
Temperatures
Diagnostics
Startup/Shutdown
Acquisition
Control and Status
Source: Internal
Method: Internal
Edit Method
Upload MS Method
Vacuum System: OK
Temperatures: OK
Source: OK
Ion Gauge Pressure: 0.0 uTorr (Fil 1 Valid)
Trap
ion source
rf
trap on/off
rf
multiplier
Method
Active Segment
Checks and Adjustments
Module Attributes
Method Segment
Ionization On
Scan Type: Full
Ionization: EI
General Parameters
Ionization Control
Internal EI Parameters
Full S
Automatic Gain Control
Target TIC: 20000 counts
Ionization Time
Fixed Ionization Time
Ionization Time: 100 useconds
Max Ion Time: 25000 useconds
Customize

Figure 2.7

Turn on the ion trap **briefly**, take a quick glance at the spectrum, and then turn off the ion trap. If the system is pumping down as expected, there should be one large off scale peak on the display centered around 19 m/z and a smaller (typically 10-50% full scale) peak around 32 m/z. There should not be a significant 28 m/z peak visible. (Refer to Figure 2.8.) If there is the system has a leak which needs to be found and fixed. If the system has a significant leak, the baseline will be flat, or there will be one large, broad peak from about 15 - 35 m/z. If this is the case, then the leak should be found and corrected before progressing any further with the installation. After the system has been pumping for > 30 minutes the intensity of the 18/19 m/z signal will decrease and the 28 m/z signal should appear.

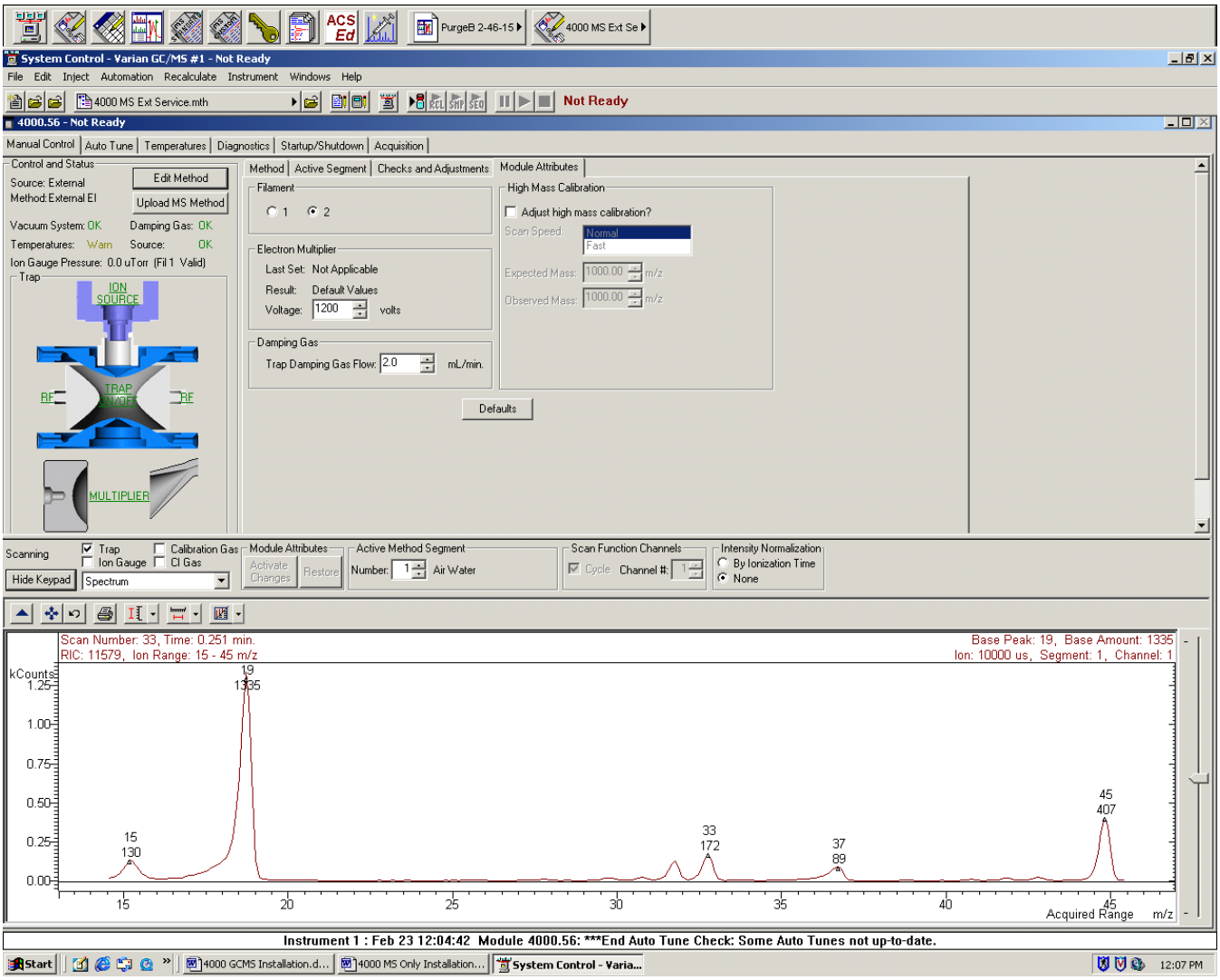

Figure 2.8

2.3 Checking for High Mass Noise

Before baking out the system it should be checked for high mass noise (HMN).

NOTE: This check for HMN may not give usable results until after auto tuning the system.

On the Full Scan Parameters tab in Manual Control (refer to Figure 2.9) enter a Mass Range of 50 to 1000 m/z. On the Ionization Control tab turn off the fixed ion time option (turned on in section 2.2) by un-checking the Fixed Ionization Time check box (refer to Figure 2.7) and click **Activate Changes**. Alternatively, one can use segment 2 of the C:\VarianWS\4000Service\4000 MS (Int or Ext) Service.mth. Turn on the trap and click on the words **Ion Source** to turn off the ion source. The RF and Multiplier should remain on (green is on, black is off). In this configuration (refer to Figure 2.9) the Base Amount (monitored in the upper right corner of the spectral window) should equal 1 or 2. In addition, no spiking above the baseline should be observed. If the Base Amount is significantly > 2 and spiking is observed, the system should be shut down and trap should be blown free of debris using a compressed inert gas. In a persistent case, the trap cavity may require repeated efforts to free it of debris. If so, the surrounding area, including the vacuum manifold and the center tube of the transfer line may also need to be blown free of debris using a compressed inert gas.

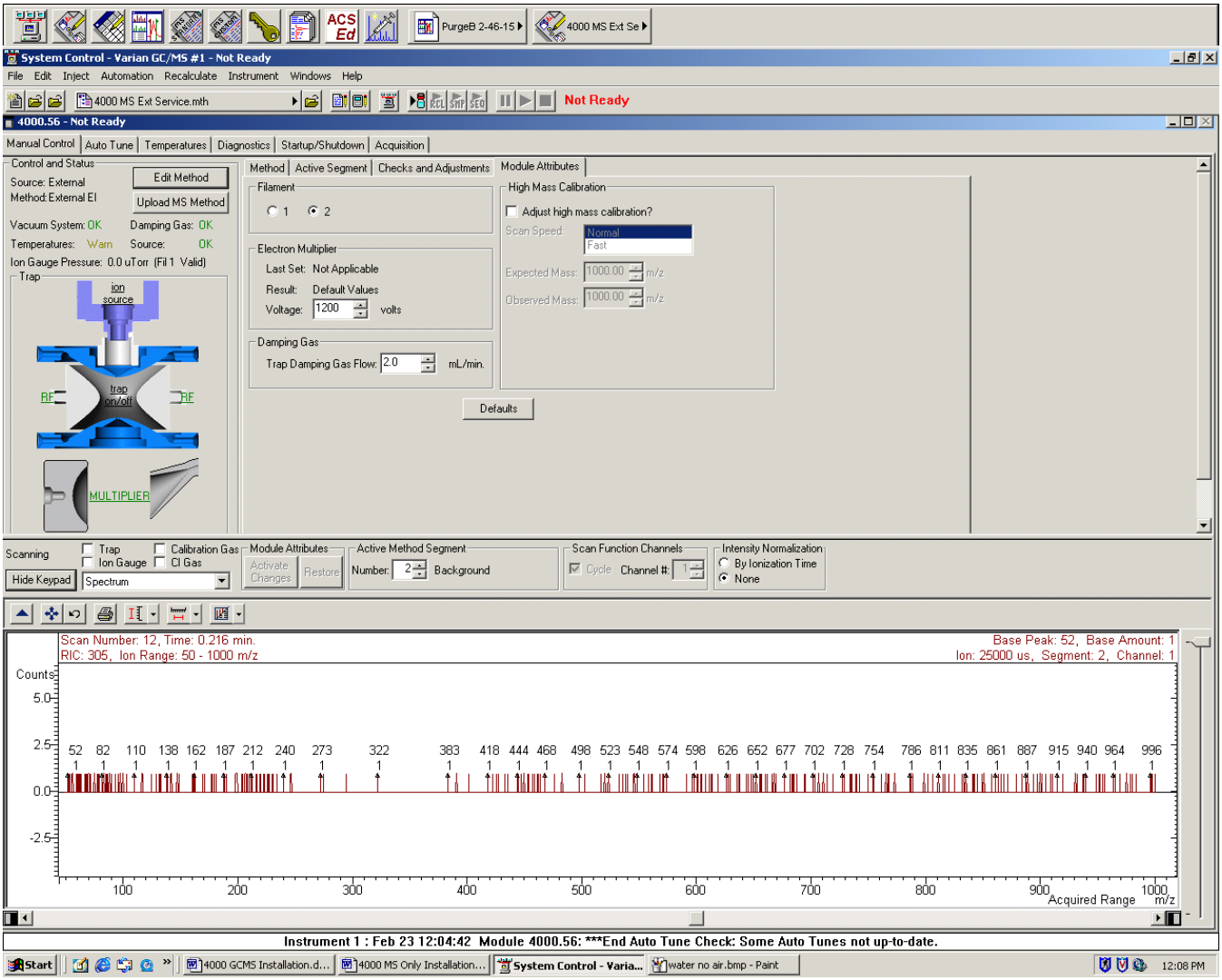

Figure 2.9

2.4 System Conditioning

Ensure that the Trap and Source (**external mode only**) temperatures are at least 150 °C and the Manifold temperature is at least 50 °C before heating up the transfer line, GC column oven, and injector so column stationary phase does not collect on low temperature MS components. Condition the injector at 230 °C with a high split ratio (e.g., 50:1 or 100:1). Condition the column starting at 50 °C, ramping at 5 degrees/minute up to 300 °C (or a temperature appropriate for the column stationary phase), and hold for 60 minutes.

From the Analysis tab on the Temperatures page set the Trap to 150 °C, Manifold to 40 °C, Transfer Line to 230 °C, and the Source to 150 °C (**external mode only**). These MS Analysis temperatures will be restored after the bakeout period and are appropriate for the injections associated with an installation. Click the Bakeout tab and enter temperatures of 220 °C for the Trap, 110 °C for the Manifold, 250 °C for the Transfer Line, and 200 °C for the Source (**external mode only**). Set a Hold Time that allows the bakeout to end about 2 hours before arriving the next day and click on **Start Bakeout**. Be sure to leave System Control open. If you do not, the bakeout will be cancelled.

The system should be conditioned for at least 10 - 24 hours, before proceeding with meeting system specifications. Ideally, the system should be conditioned overnight. Hydrocarbon and water background levels can be expected to continue to decrease even after the initial bakeout. During conditioning, some familiarization could be done with the operator.

NOTE: After bakeout, reset the manifold bakeout temperature to 80 °C and click **Save**. A manifold bakeout temperature of 110 °C is appropriate for the initial bakeout that lowers the water level in the system. Additional bakeouts will likely be done to lower the level of volatiles in the system and this can be achieved with a manifold temperature of 80 °C. Repeated manifold bakeouts at higher temperatures are to be avoided as they can potentially degrade system performance.

Section 3. Preliminary Tuning

Before preliminary tuning, be sure the MS has returned to Analysis temperatures and the GC column flow rate is set to 1 mL/min.

3.1 System Monitoring

After bakeout, go to Startup/Shut Down (shown in Figure 2.1) and monitor the following:

- *Heated Zones*: Operating Conditions should be close to Current Set Points.
- Vacuum System: Status should be Ready, Pump Spin Speed should be 100%, Current should be significantly less than 300 mAmps, dependent on damping gas flow rate.
- Pneumatics (external mode only): Damping Gas On, Flow Rate 1.0 - 4.0 mL/min (should match the set point).
- Getter C*ontrol*: Heater On, Temperature > 300 °C

Next, go to Diagnostics, select the Ion Source, Multiplier/Dynode, and Ion Gauge check boxes (refer to Figure 3.1) and monitor the following:

- *Vacuum System*: Power should be 8-12 Watts with no damping gas, 12-15 Watts with damping gas flow.
- *Ion Gauge*: Pressure should read back a µTorr value that will vary dependent on column flow, damping gas flow, air/water levels, etc. This is the manifold pressure.
- *Trap*: Multiplier voltage operating condition should be displayed, Dynode should be approximately 10,000V.
- *Ion Source (**external mode**)*: operating conditions should be displayed, check for no read backs equal to zero. (NOTE: un-checking the Ion Source box should cause Emission to read back < 5 µAmps or there may be a current leakage issue indicating a need to clean the filament and lens areas.)
- *Roughing Line Pressure*: should be significantly <100 mTorr.
- *Deviation*: should be less than 3 µA after auto tuning the electron and ion lenses.

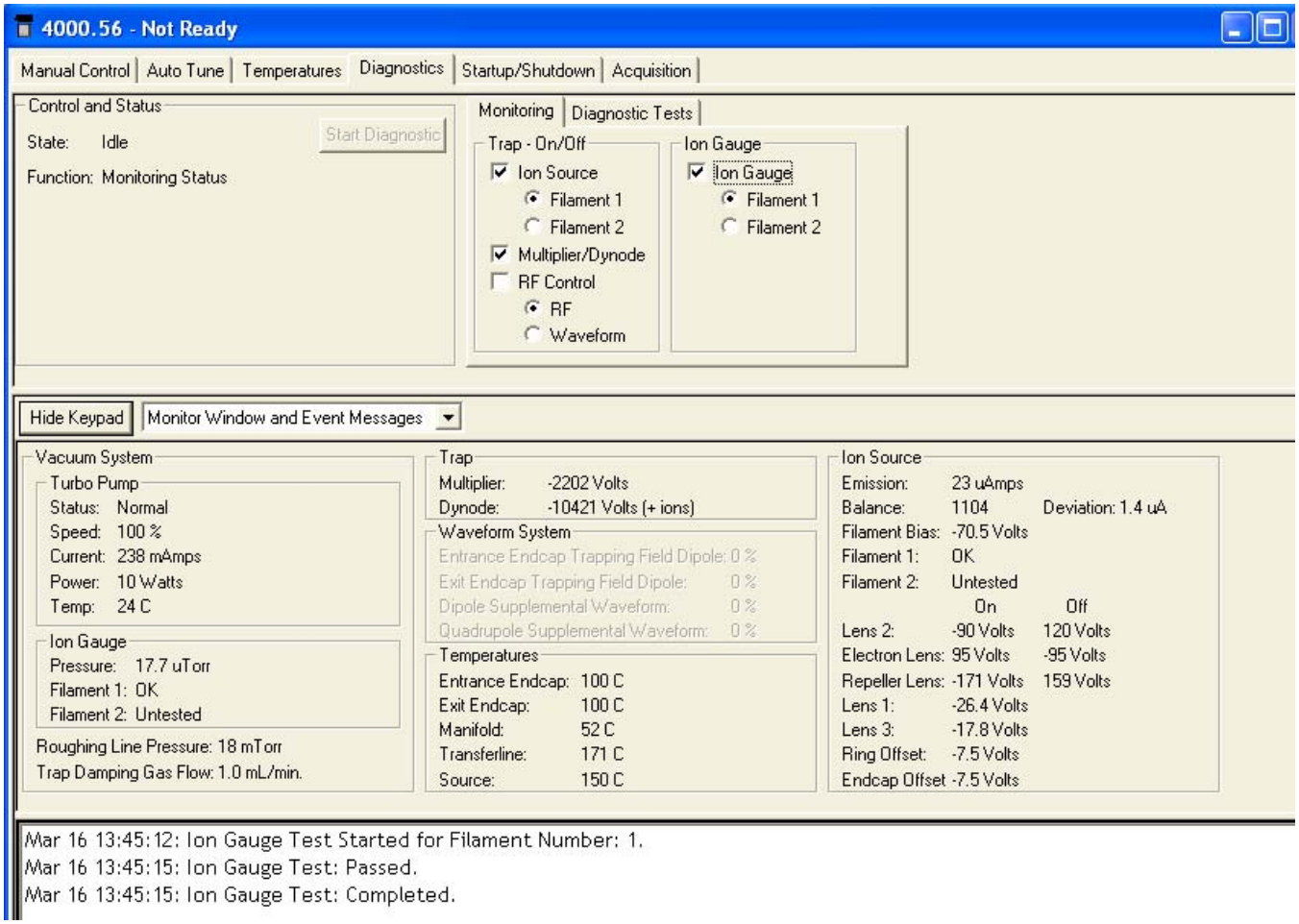

Figure 3.1

3.2 RF Ramp Adjustment

Go to the Checks and Adjustments tab in Manual Control, highlight RF Ramp Adjustment and click **Start** (refer to Figure 3.2). Adjust the RF tuning rod as shown in Figure 3.3 to minimize the Base Amount value displayed in the upper right corner of the spectral window. The graphic display can also be monitored for minimizing the intensity of the high end. Click **Done** when finished.

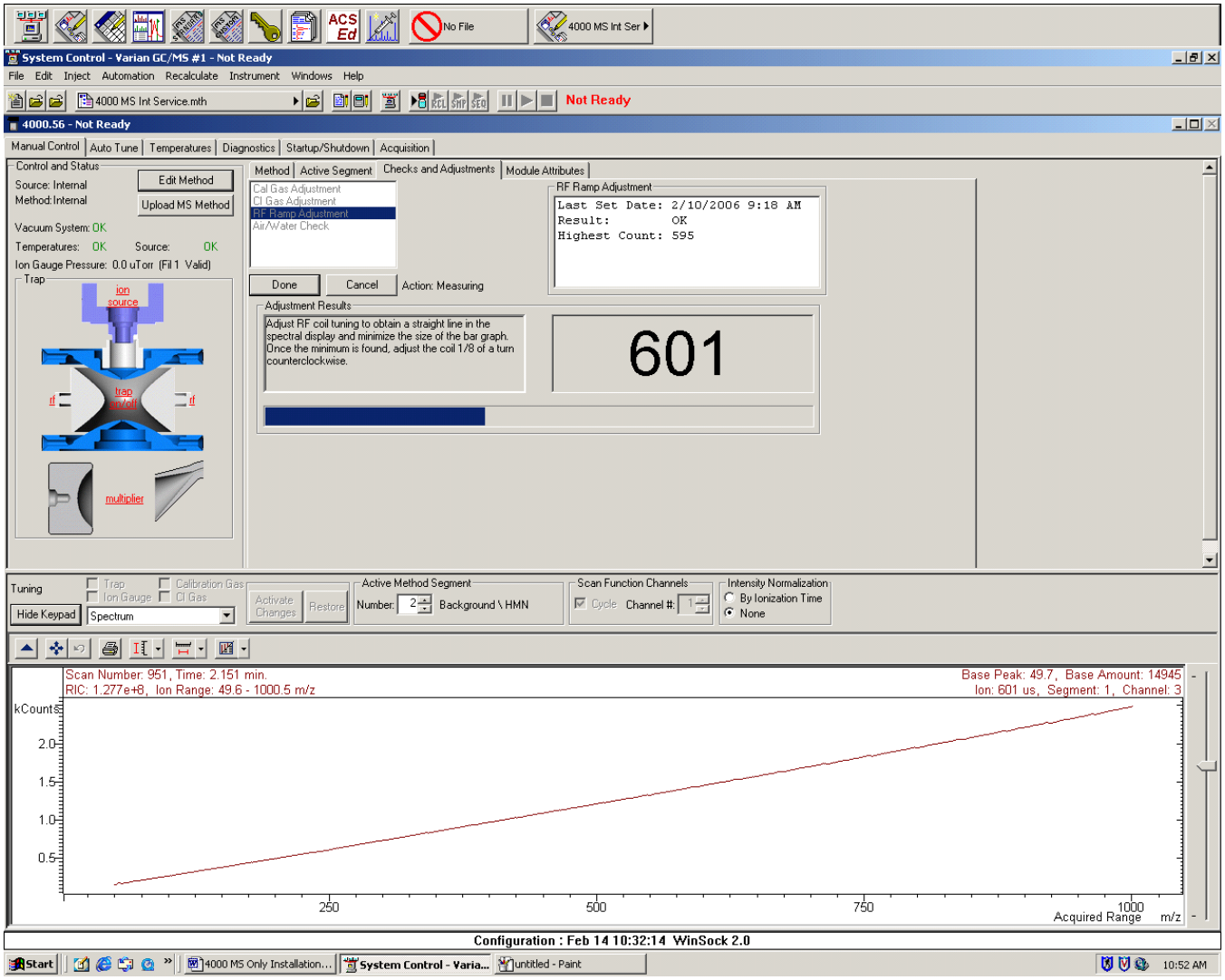
System Control - Varian GC/MS #1 - Not Ready
File Edit Inject Automation Recalculate Instrument Windows Help
4000 MS Int Service.mth
Not Ready
4000.56 - Not Ready
Manual Control | Auto Tune | Temperatures | Diagnostics | Startup/Shutdown | Acquisition
Control and Status
Source: Internal
Method: Internal
Edit Method
Upload MS Method
Vacuum System: OK
Temperatures: OK
Source: OK
Ion Gauge Pressure: 0.0 uTorr (Fil 1 Valid)
Trap
ion source
trap on/off
multiplier
Method | Active Segment | Checks and Adjustments | Module Attributes
Cal Gas Adjustment
CI Gas Adjustment
RF Ramp Adjustment
Air/Water Check
RF Ramp Adjustment
Last Set Date: 2/10/2006 9:18 AM
Result: OK
Highest Count: 595
Done
Cancel
Action: Measuring
Adjustment Results
Adjust RF coil tuning to obtain a straight line in the spectral display and minimize the size of the bar graph. Once the minimum is found, adjust the coil 1/8 of a turn counterclockwise.
601
Tuning
Trap
Ion Gauge
Calibration Gas
CI Gas
Hide Keypad
Spectrum
Activate Changes
Restore
Active Method Segment
Number: 2
Background \ HMN
Scan Function Channels
Cycle
Channel #: 1
Intensity Normalization
By Ionization Time
None
Scan Number: 951, Time: 2.151 min.
RIC: 1.277e+8, Ion Range: 49.6 - 1000.5 m/z
Base Peak: 49.7, Base Amount: 14945
Ion: 601 us, Segment: 1, Channel: 3
kCounts
2.0
1.5
1.0
0.5
250
500
750
1000
Acquired Range m/z
Configuration : Feb 14 10:32:14 WinSock 2.0
Start
4000 MS Only Installation...
System Control - Varia...
untitled - Paint
10:52 AM

Figure 3.2

Figure 3.3

3.3 Dipole Adjustment

Go to Diagnostics. Check the RF Control check box and select the RF radio button from the Monitoring tab as shown in Figure 3.4. Adjust the trapping field dipole inductors on the Upper Manifold PWA through the holes in the Manifold Electronics Enclosure Cover (as shown in Figure 3.5) so the Entrance and Exit Endcap Trapping Field Dipole values equal 100%.

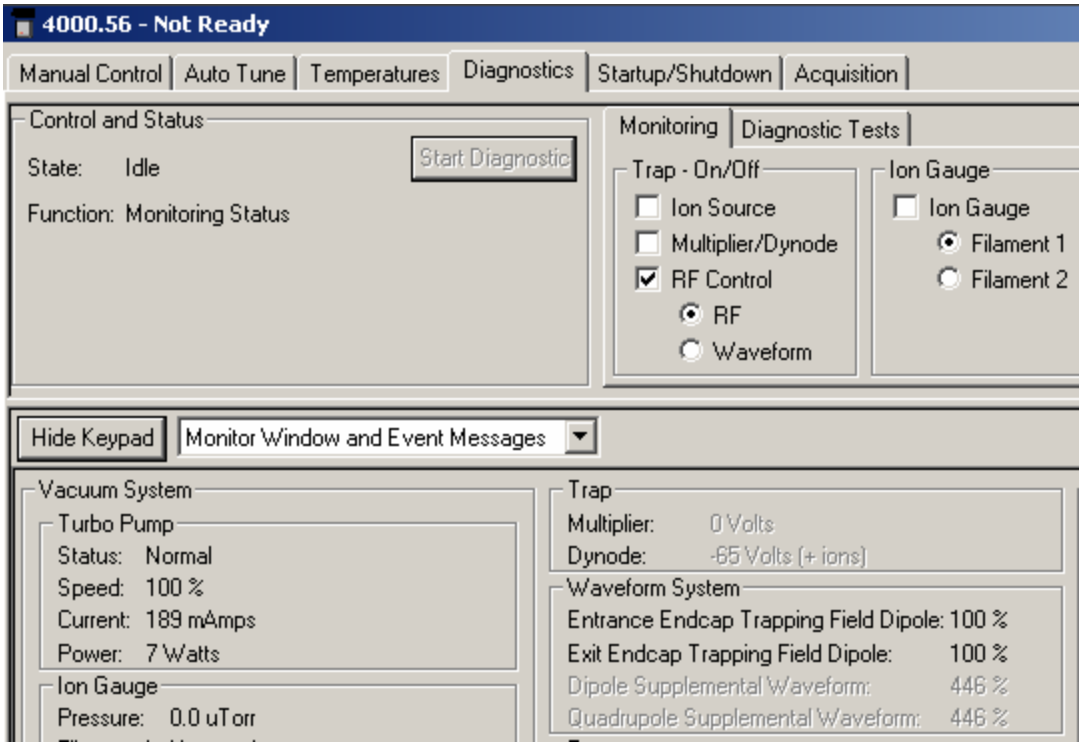

Figure 3.4

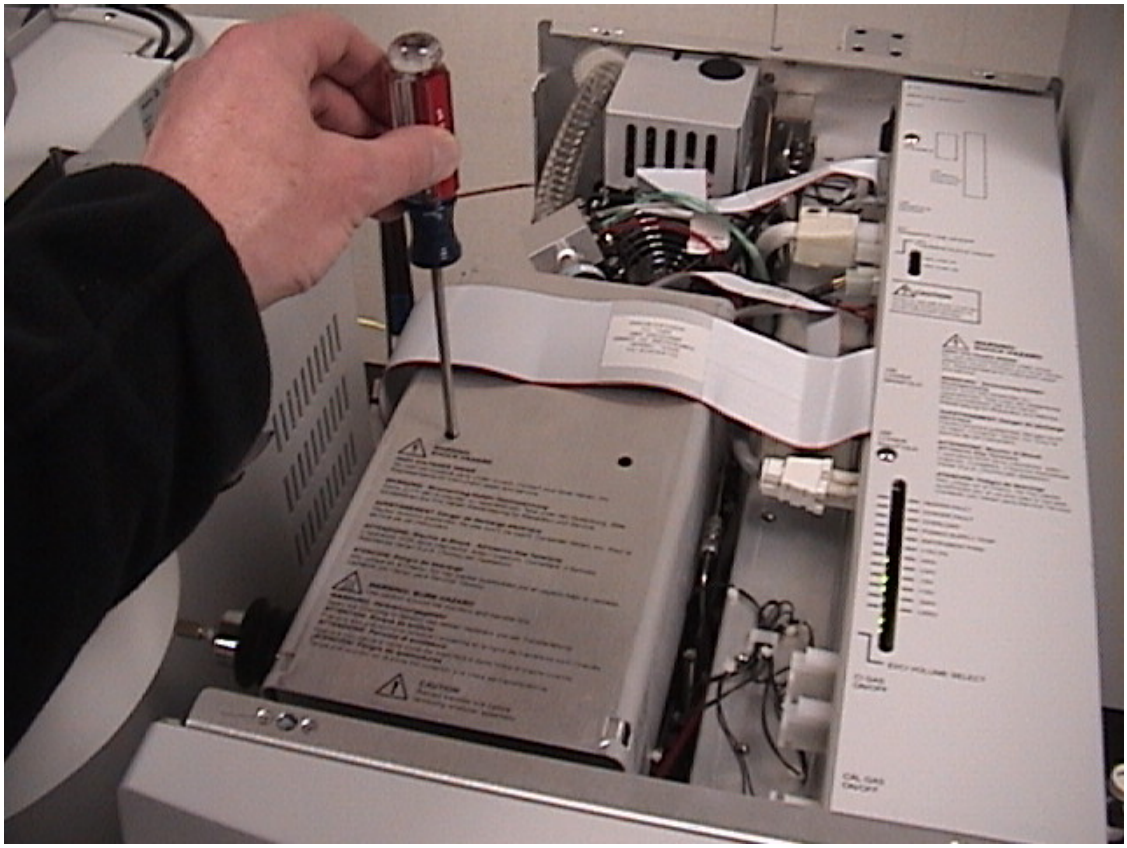

Figure 3.5

3.4 Air Water Check

From the Checks and Adjustments tab in Manual Control highlight Air/Water Check and click **Start** (refer to Figure 3.6). Click **Done** when finished. If the m/z 19 to m/z 18 ratio is greater than 50% or the peak width at 10% peak height of the m/z 28 signal is greater than 1 Dalton, the cause should be investigated before proceeding with the installation.

NOTE: This air/water check may not give usable results until auto tuning the system.

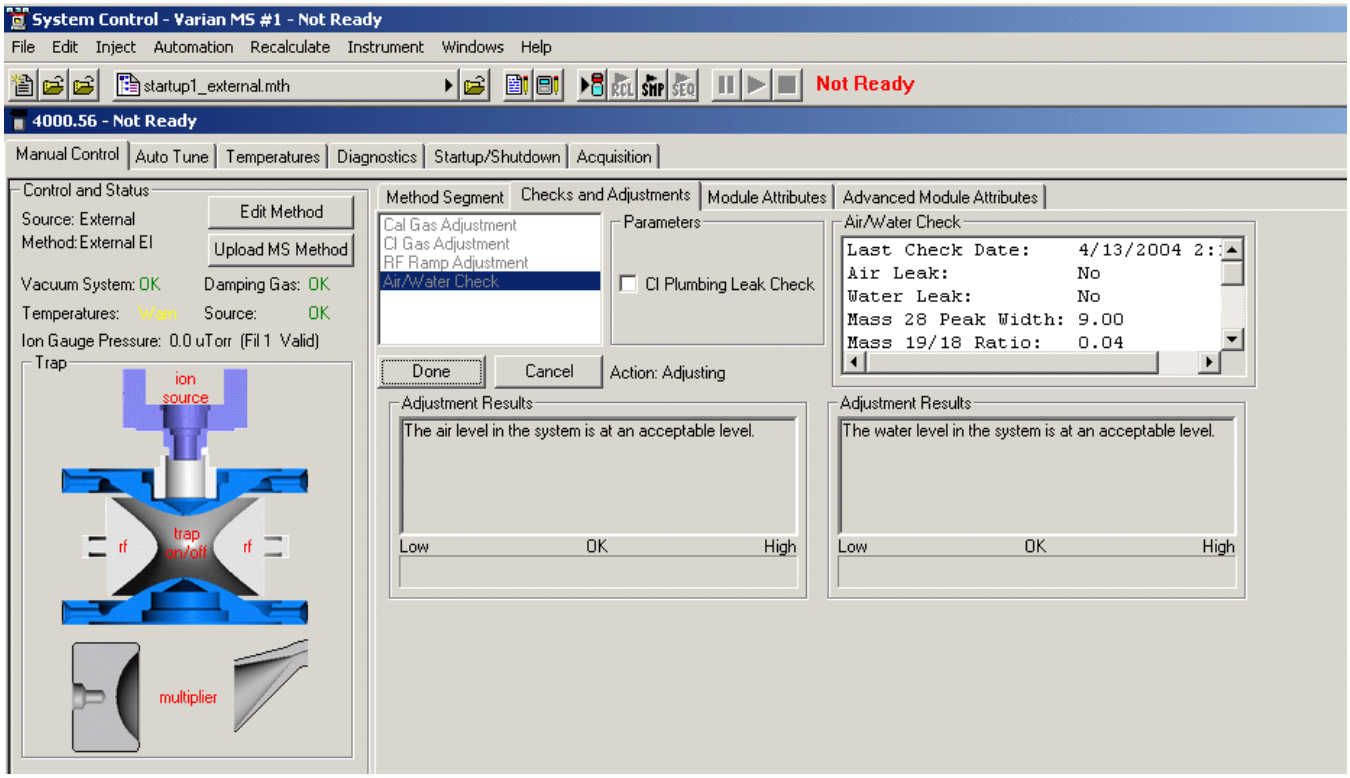

Figure 3.6

Air water Check with CI Plumbing

The air and water levels can also be checked with inclusion of the CI plumbing. From the Checks and Adjustments tab in Manual Control highlight Air/Water Check, select the CI Plumbing Leak Check box, and click **Start** (refer to Figure 3.7). Click **Done** when finished. The air and water levels can be expected to increase when the CI plumbing is included.

Air Water Check using Cal Gas Ion Ratios

Another useful monitor of the system's water level is the intensity of the m/z 197 and m/z 219 signals from the calibration gas FC-43. A high water level will cause an increase in the m/z 197 signal and a decrease in the m/z 219 signal. As the water level decreases the intensity of m/z 197 will decrease while the intensity of m/z 219 will increase.

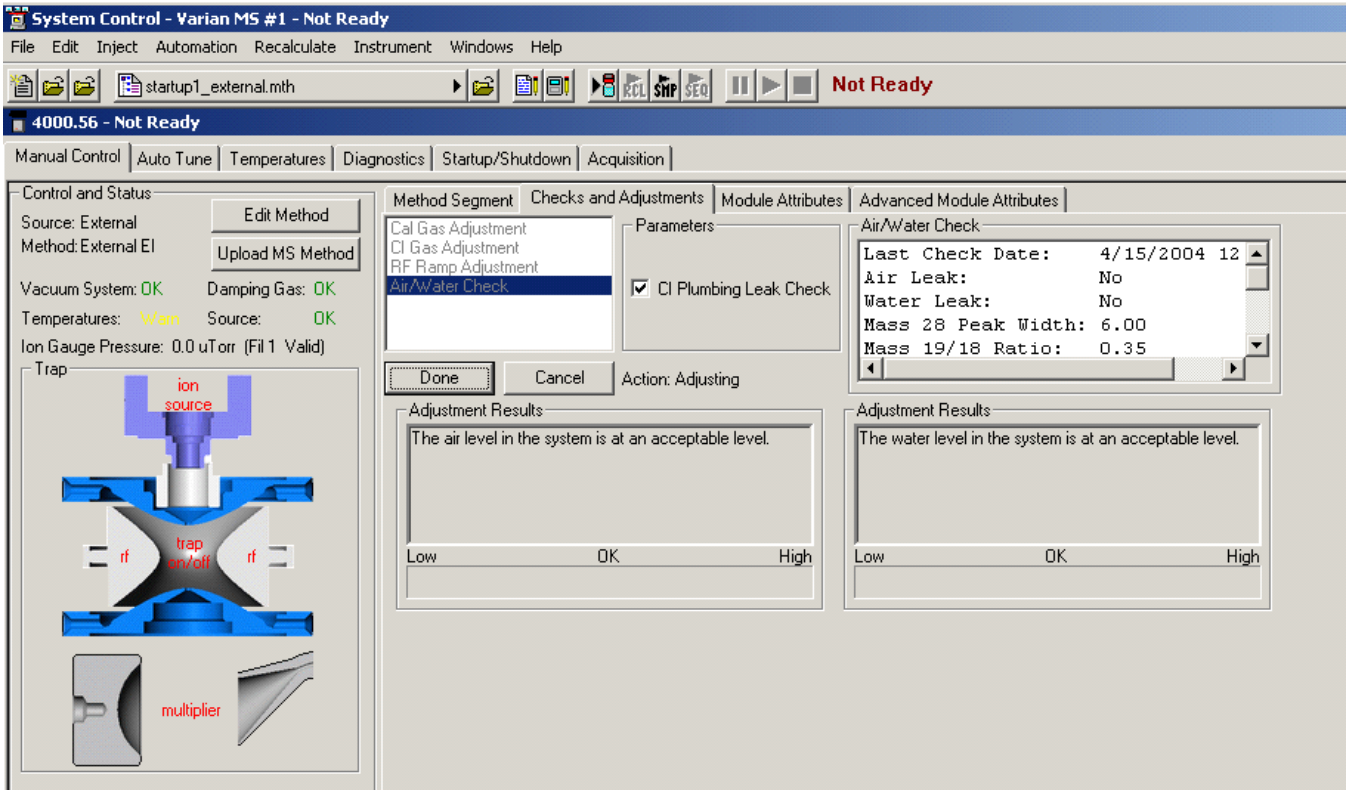

Figure 3.7

3.5 Cal Gas Adjustment

Confirm the Cal Gas vial has been filled by checking behind the MS front door. Adjust the Cal Gas needle valve so it is approximately half way open (turn the valve all the way clockwise and then turn 6-8 turns counter clockwise). Refer to Section 4 of this document and run the Auto Tunes. From the Checks and Adjustments tab in Manual Control highlight Cal Gas Adjustment and click **Start** (refer to Figure 3.8). Adjust the Cal Gas needle valve behind the MS front door until the display indicates the Cal Gas pressure is OK. Click **Done** when finished.

NOTE: The Cal Gas adjustment can vary with variations in auto tune settings, filament emission current, system sensitivity, and system background levels. Therefore, this adjustment may need to be revisited after auto tuning, editing the filament emission current, or when system sensitivity or background levels change.

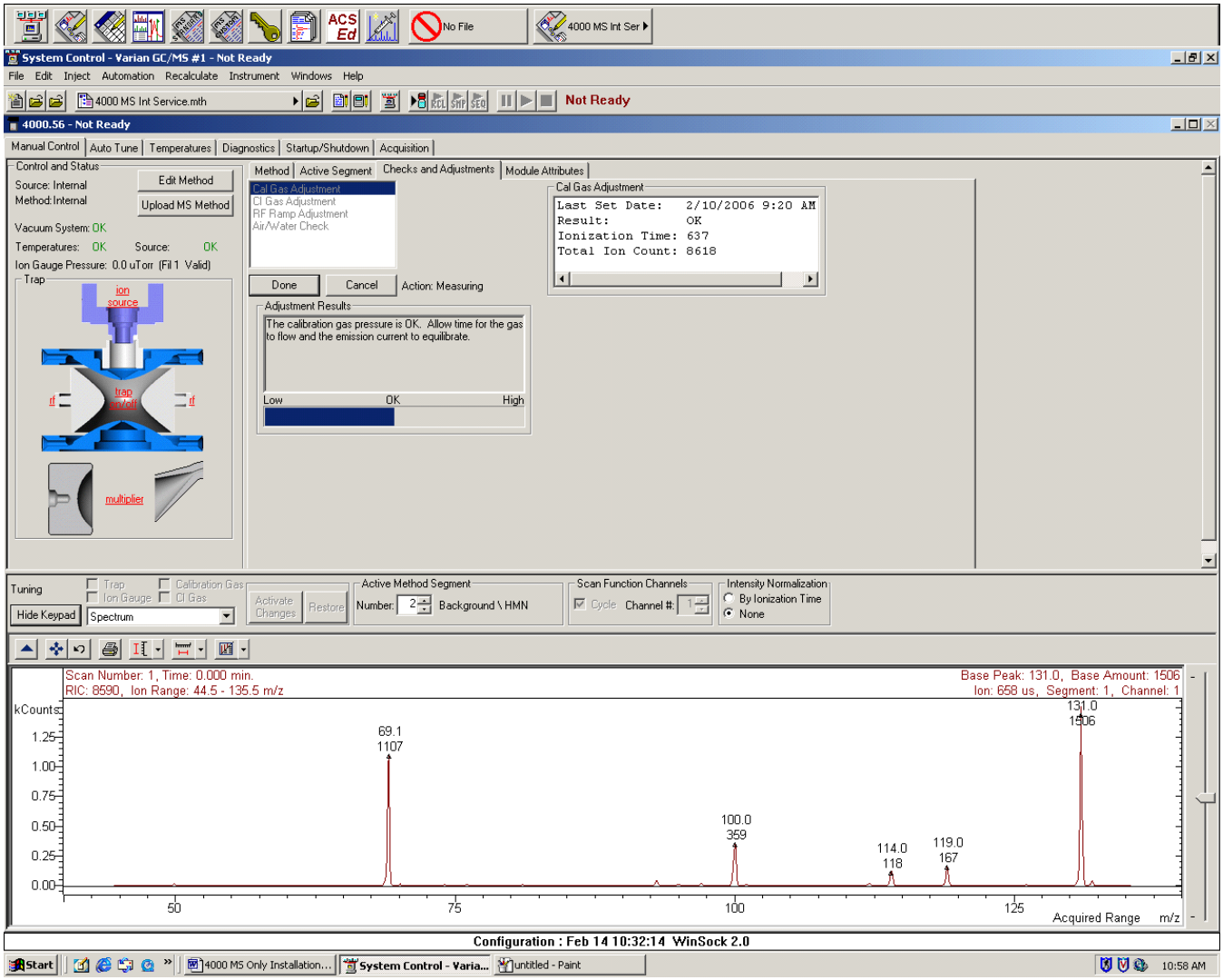
System Control - Varian GC/MS #1 - Not Ready
File Edit Inject Automation Recalculate Instrument Windows Help
4000 MS Int Service.mth
Not Ready
4000.56 - Not Ready
Manual Control | Auto Tune | Temperatures | Diagnostics | Startup/Shutdown | Acquisition
Control and Status
Source: Internal
Method: Internal
Edit Method
Upload MS Method
Vacuum System: OK
Temperatures: OK
Source: OK
Ion Gauge Pressure: 0.0 uTorr (Fil 1 Valid)
Trap
ion source
trap dipoff
rf
rf
multiplier
Method | Active Segment | Checks and Adjustments | Module Attributes
Cal Gas Adjustment
CI Gas Adjustment
RF Ramp Adjustment
Air/Water Check
Done
Cancel
Action: Measuring
Adjustment Results
The calibration gas pressure is OK. Allow time for the gas to flow and the emission current to equilibrate.
Low
OK
High
Cal Gas Adjustment
Last Set Date: 2/10/2006 9:20 AM
Result: OK
Ionization Time: 637
Total Ion Count: 8618
Tuning
Trap
Ion Gauge
Calibration Gas
CI Gas
Hide Keypad
Spectrum
Activate Changes
Restore
Active Method Segment
Number: 2
Background \ HMN
Scan Function Channels
Cycle
Channel #: 1
Intensity Normalization
By Ionization Time
None
Scan Number: 1, Time: 0.000 min.
RIC: 8590, Ion Range: 44.5 - 135.5 m/z
Base Peak: 131.0, Base Amount: 1506
Ion: 658 us, Segment: 1, Channel: 1
kCounts
1.25
1.00
0.75
0.50
0.25
0.00
69.1
1107
100.0
359
114.0
118
119.0
167
131.0
1506
50
75
100
125
Acquired Range m/z
Configuration : Feb 14 10:32:14 WinSock 2.0
Start
4000 MS Only Installation...
System Control - Varia...
untitled - Paint
10:58 AM

Figure 3.8

Section 4. Auto Tuning

Auto tuning a 4000 MS is significantly different from auto tuning a 2X00 MS. This section offers descriptions of the 4000 MS auto tune processes with the assumption that preliminary tuning such as the RF Ramp, Endcap Trapping Field Dipole, and Cal Gas adjustments were done previously. Be aware that the Cal Gas adjustment may need to be re-visited after running the auto tunes and that the auto tunes should be re-run after re-adjusting the Cal Gas needle valve because each of these two features (Cal Gas adjustment and auto tuning) can affect the other.

4.1 Internal Auto Tuning

INTERNAL EI and CI

Ensure the GC column is delivering approximately 1 mL/min of helium flow into the trap cavity.

From the Auto Tune page in System Control, click **Select All**, and then click **Start** Auto Tune (refer to figure 4.1).

It is not necessary to run the auto tunes again for the alternate filament in internal mode. The values generated by this auto tune should work well for both EI and CI acquisitions in internal mode.

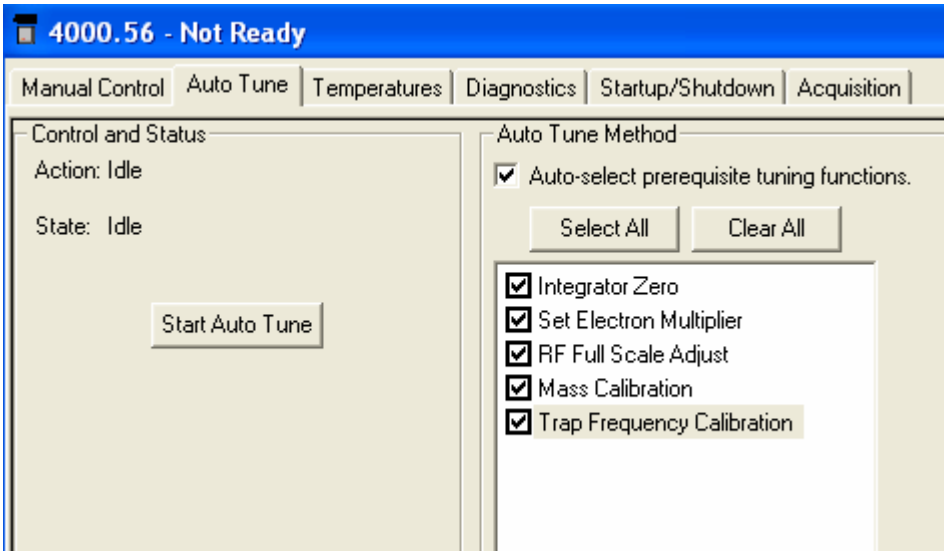

Figure 4.1

4.2 External Auto Tuning

EXTERNAL EI, PCI, and NCI

Ensure the GC column is delivering approximately 1 mL/minute of helium flow into the ion source, the Trap Damping Gas Flow is delivering approximately 1 mL/minute helium flow into the trap cavity, and the Getter temperature is near its set point.

1. From the Auto Tune page in System Control, click the Select All button, then click the Start Auto Tune button. Allow the auto tunes to run to completion.

   If the external CI option is installed continue to steps 2, 3, and 4. If the external CI option is not installed, skip steps 2 and 3 and go directly to step 4.

2. Ensure a 25 psi supply of high purity (at least 99.95%) methane is delivered through the 4 mL gaseous CI restrictor (03-930597-01) to the CI inlet port at the back of the 4000 MS (refer to the installation instructions included with the CI option for details). Turn the CI needle valve CCW until it stops. From the Manual Control page, Checks and Adjustments tab, highlight CI Gas Adjustment, select Methane as the Reagent, and click Start. Turn the CI needle valve CW to increase CI flow into the analyzer until the Ion Gauge Pressure reads approximately 50 to 60 uTorr. (The number of needle valve turns required is typically about 6 turns.) Allow time for the CI reagent flow to equilibrate while adjusting. Click Done when the adjustment is complete. Figure 8.1 shows properly adjusted methane.

3. Return to the Auto Tune page in System Control, check only the Electron Lens Tuning and Turn on CI gas flow during tune check-boxes, and click Start Auto Tune (refer to figure 4.2). Allow this CI auto tune to run to completion.

4. From the Manual Control page, Module Attributes tab, select the alternate external filament (likely Filament 2) and return to step 1. Auto tune the system using the alternate external filament including steps 2 and 3 if the CI option is installed.

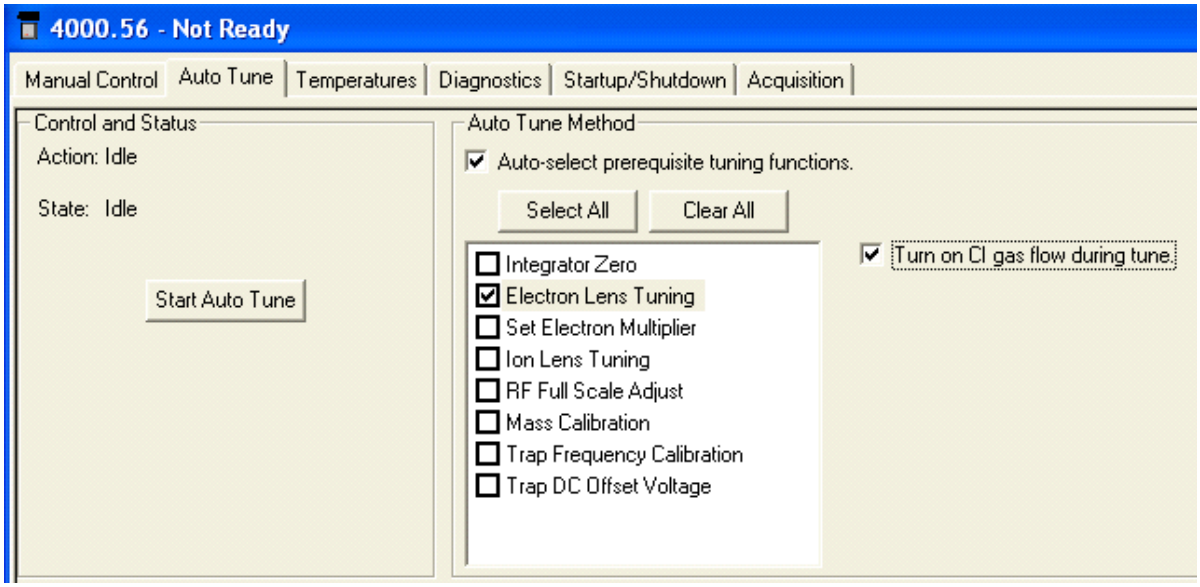

Figure 4.2

The customer should be familiar with the recommended frequencies of running auto tunes.

Run the following tunes as needed based on general system performance, perhaps once every two weeks:

- Integrator Zero, Set Electron Multiplier, Trap DC Offset Voltage, Electron Lens Tuning, and Ion Lens Tuning.

Run the following auto tunes as needed for mass assignment:

- RF Full Scale Adjust, Mass Calibration, and Trap Frequency Calibration.

NOTE: The Deviation read back on the Diagnostic page being greater than 3 µA can indicate a need for Electron Lens Tuning, and Ion Lens Tuning as mentioned in section 3.1 of this document.

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Section 5. Achieving Internal EI Specifications

NOTE: If the MS is configured in external mode please proceed to Section 7 of this document.

The EI specifications for a 4000 GCMS system in Internal mode:

20:1 RMS signal-to-noise using the m/z 272 ion from a 1 µL injection of a 2 pg/µL OFN solution (03-931126-01). A 10:1 split ratio can be used to reduce the amount of OFN from 2 pg to 200 fg. If necessary, a 10:1 dilution can be done using customer supplied glassware and 2,2,4-trimethylpentane (isooctane) to reduce the amount of OFN from 2 pg to 200fg. In addition, a background corrected spectrum should be library searchable with any match > 500. If a Quick Switch Valve is used in the GC oven the signal-to-noise spec is reduced from 20:1 to 10:1.

5.1 Methodology

NOTE: The 4000 GCMS system will arrive with a Test Files CD containing factory test method and data files. These method files can be used to pass the sensitivity tests associated with the installation. At the factory a diluted OFN standard is injected using a pressure pulse and splitless injection technique. If a diluted OFN standard is to be used at an installation the method from the Test Files CD can be used with no editing. If a split style injection is to be used at an installation, the Test Files CD method should be edited to remove the pressure pulse and introduce a 10:1 split ratio. Retain the Test Files CD for your records.

Ensure the MS temperatures are set for this acquisition:

- Trap: 150 °C
- Manifold: 40 °C
- Transfer Line: 230 °C

If necessary, edit the Test Files CD internal EI method and activate the method in System Control.

Figure 5.1 shows System Control in a Ready state. Both the GC window and the MS window must be Ready for System Control to be Ready.

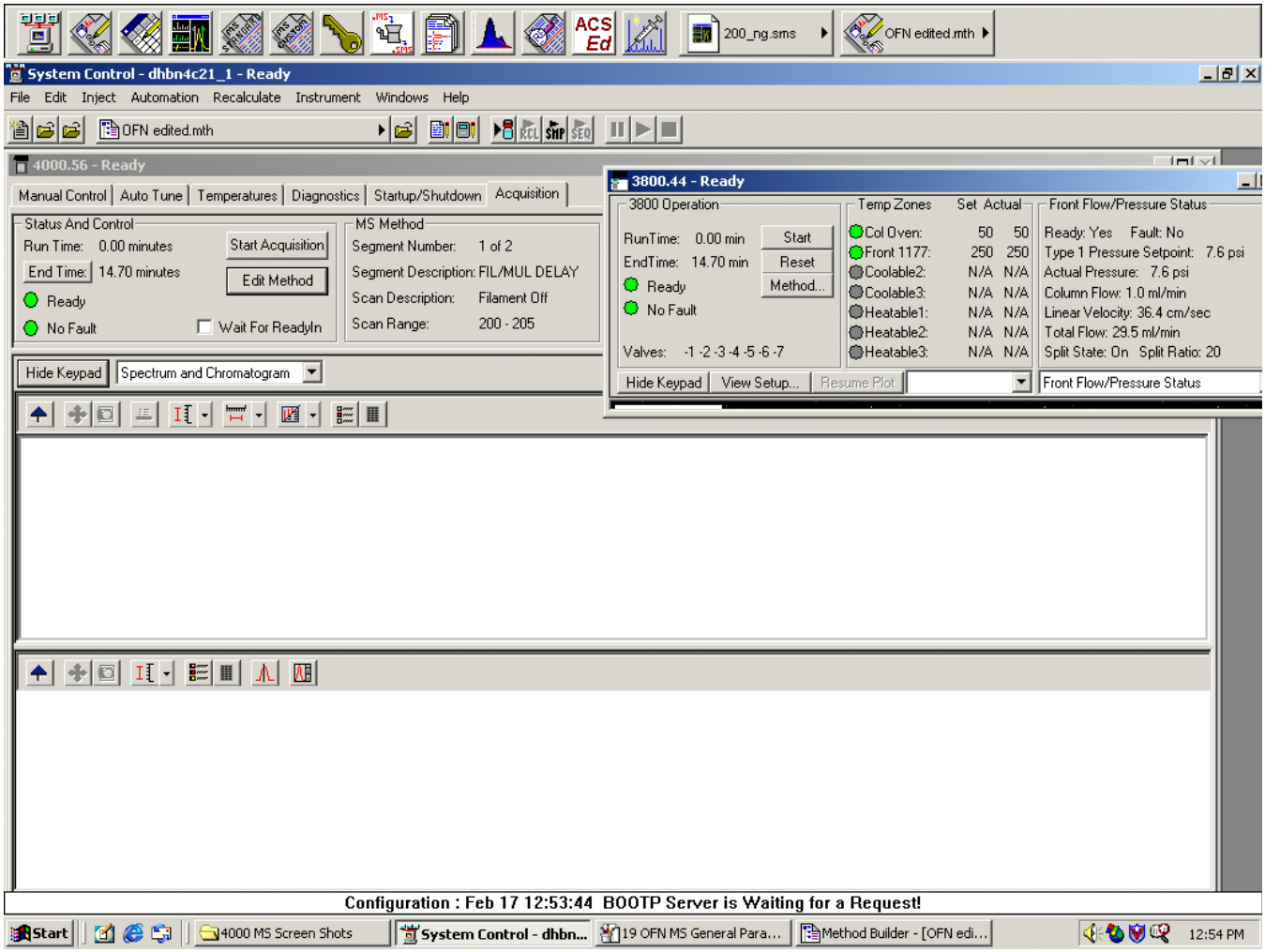

Figure 5.1

5.2 OFN Acquisition

Use the Inject Single Sample option to make a 1 µL manual injection of test standard 03-931126-01 (split style injection) or a 10:1 diluted test standard (splitless pressure pulse style injection). Use a lower air gap to avoid sample loss through the tip of the needle. Ensure the syringe plunger is not forced out of the syringe barrel if a Pressure Pulse is used in the method.

5.3 Library Search and Signal to Noise Calculation

After acquiring the data file, calculate the S/N and run a library search. This opportunity could be used to demonstrate data analysis to the customer. The .sms data file can be loaded into Mass Spectra Data Review (MSDR) using the quick load feature shown in Figure 5.2.

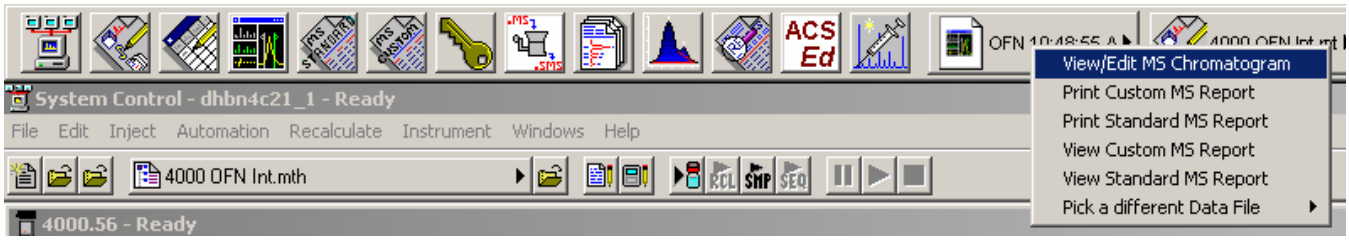

Figure 5.2

In the MSDR window type 272 into the IONS field and press Enter to display a single ion chromatogram of the m/z 272 ion signal (see Figure 5.3).

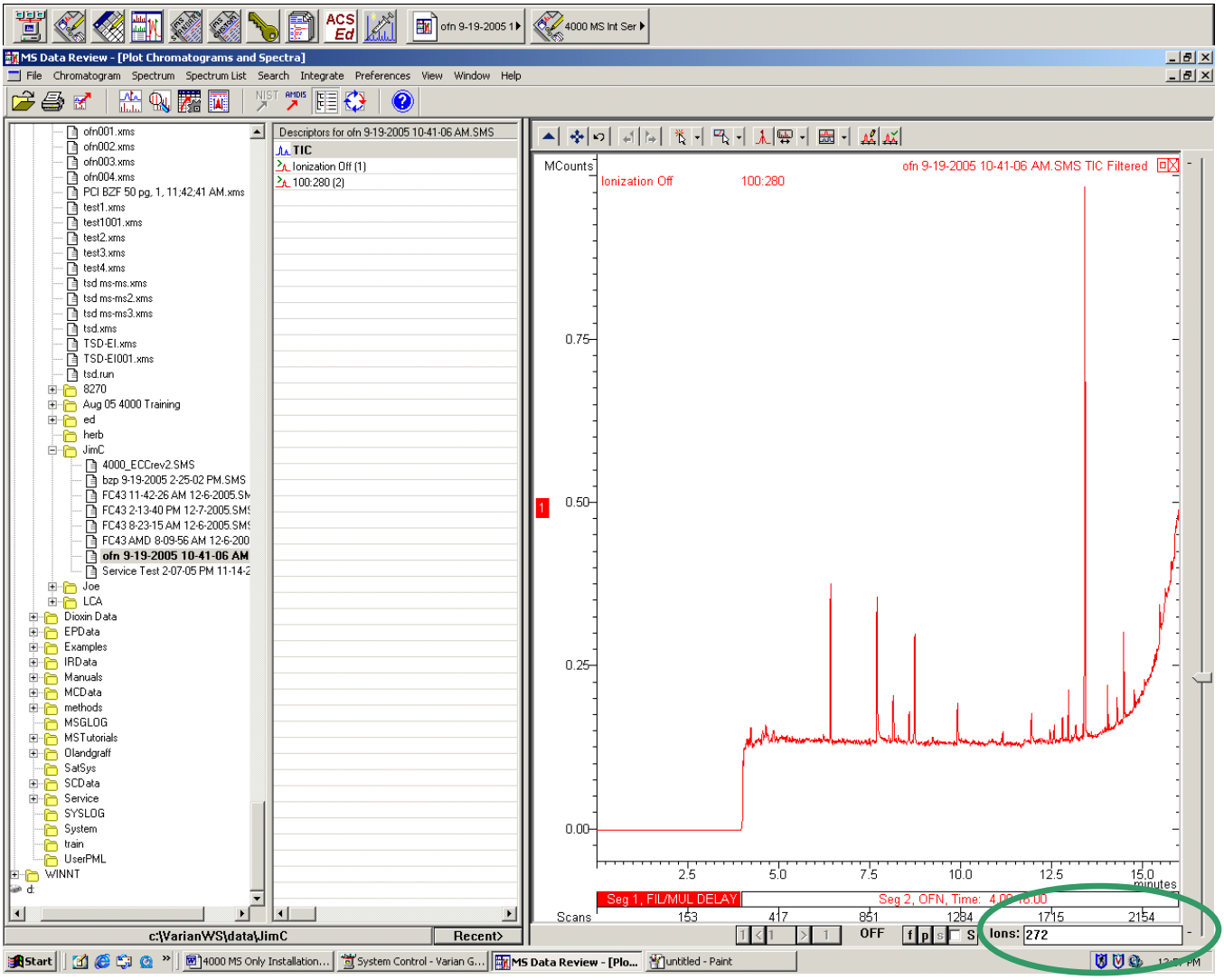

Figure 5.3

Click-and-drag a zoom highlight box around the m/z 272 ion from OFN (see Figure 5.4)

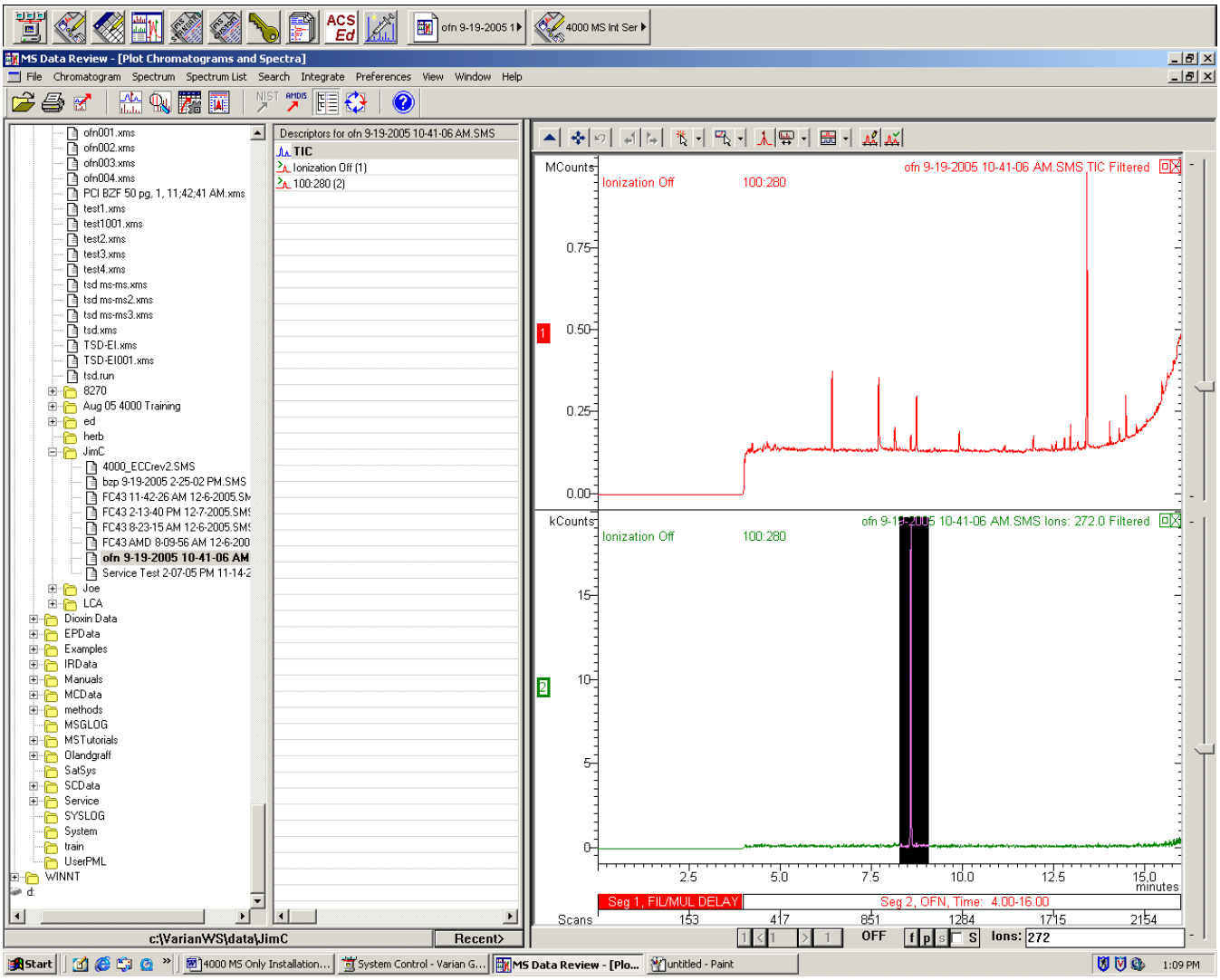

Figure 5.4

Right mouse click on the apex scan of the 272 single ion chromatogram and select Calculate Noise to generate the RMS signal-to-noise ratio (see Figure 5.5).

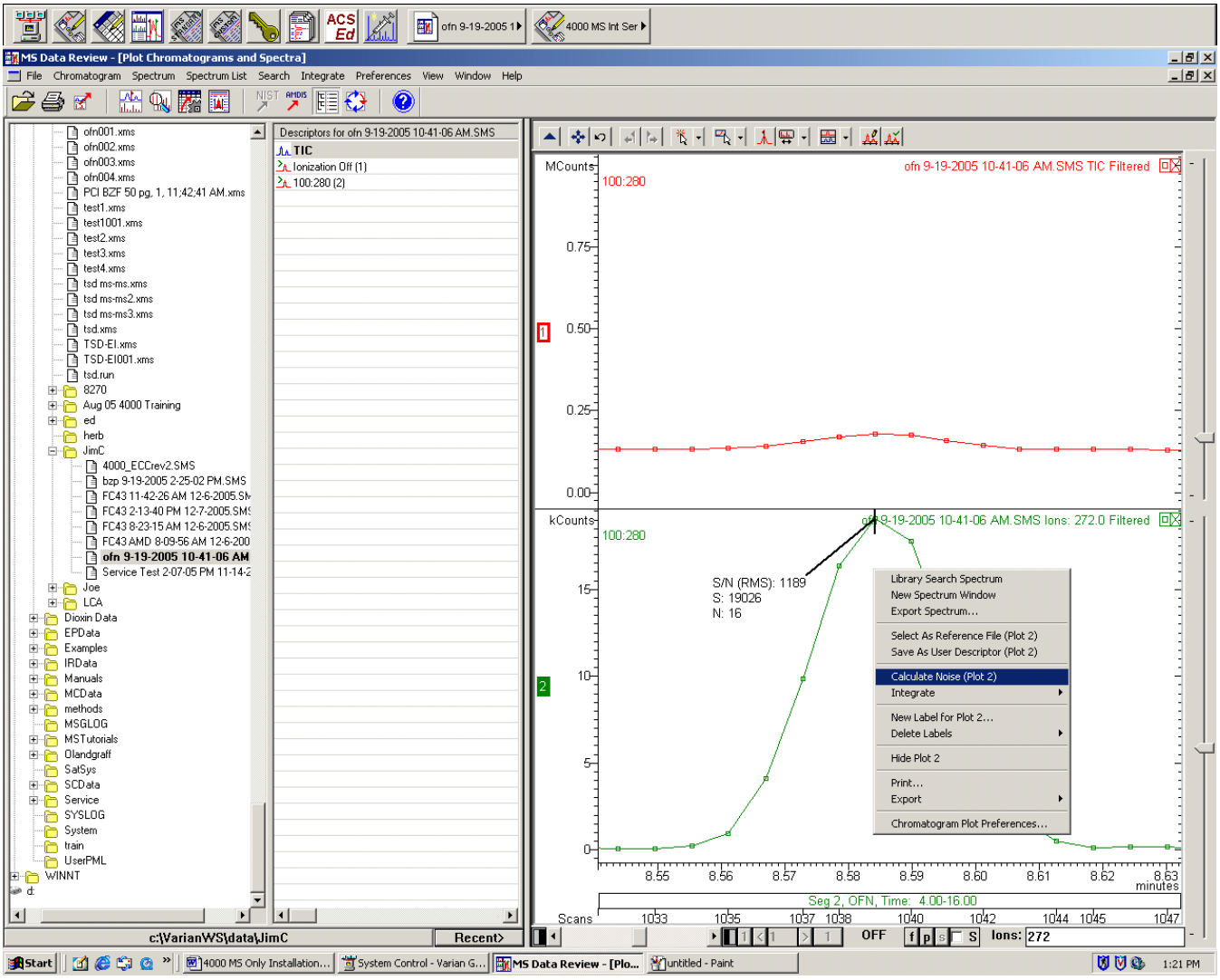

Figure 5.5

Note that the RMS signal-to-noise is calculated using 5-point Savitsky-Golay smoothing. How to manually set these Chromatogram Plot Preferences from the Chromatogram Plot and Noise tabs is shown in Figures 5.6, 5.7, and 5.8. Right mouse click the chromatogram and select Chromatogram Plot Preferences… (refer to Figure 5.6 and 5.7 and 5.8).

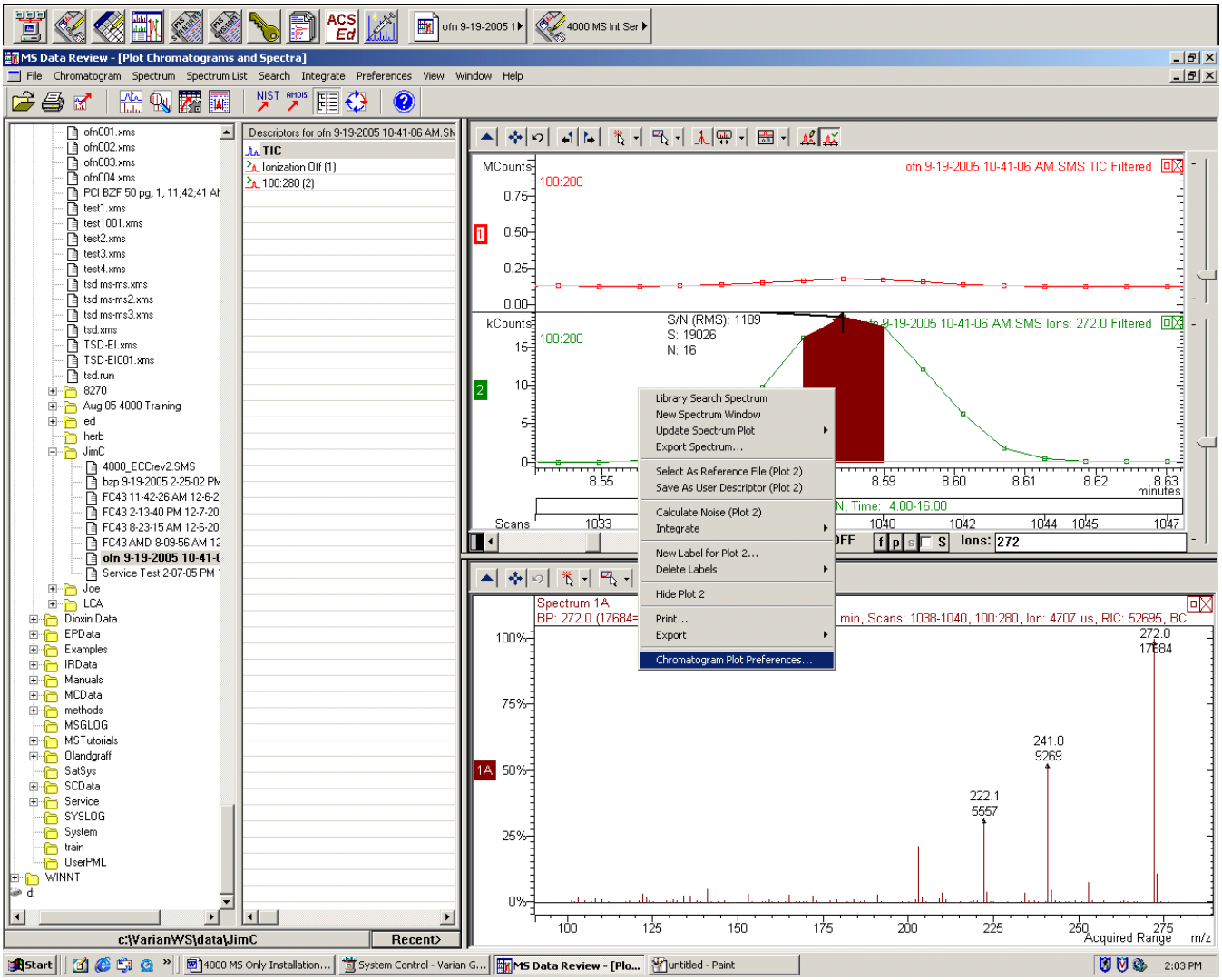
MS Data Review - [Plot Chromatograms and Spectra]
File Chromatogram Spectrum Spectrum List Search Integrate Preferences View Window Help
Descriptors for ofn 9-19-2005 10-41-06 AM.SM
TIC
Ionization Off (1)
100:280 (2)
MCounts
100:280
ofn 9-19-2005 10-41-06 AM.SMS TIC Filtered
kCounts
S/N (RMS): 1189
S: 19026
N: 16
ofn 9-19-2005 10-41-06 AM.SMS Ions: 272.0 Filtered
Library Search Spectrum
New Spectrum Window
Update Spectrum Plot
Export Spectrum...
Select As Reference File (Plot 2)
Save As User Descriptor (Plot 2)
Calculate Noise (Plot 2)
Integrate
New Label for Plot 2...
Delete Labels
Hide Plot 2
Print...
Export
Chromatogram Plot Preferences...
minutes
Scans
Ions: 272
Spectrum 1A
BP: 272.0 (17684
min, Scans: 1038-1040, 100:280, Ion: 4707 us, RIC: 52695, BC
272.0
17684
241.0
9269
222.1
5557
Acquired Range
m/z
c:\VarianWS\data\JimC
Recent>
Start
4000 MS Only Installation...
System Control - Varian G...
MS Data Review - [Plo...
untitled - Paint
2:03 PM

Figure 5.6

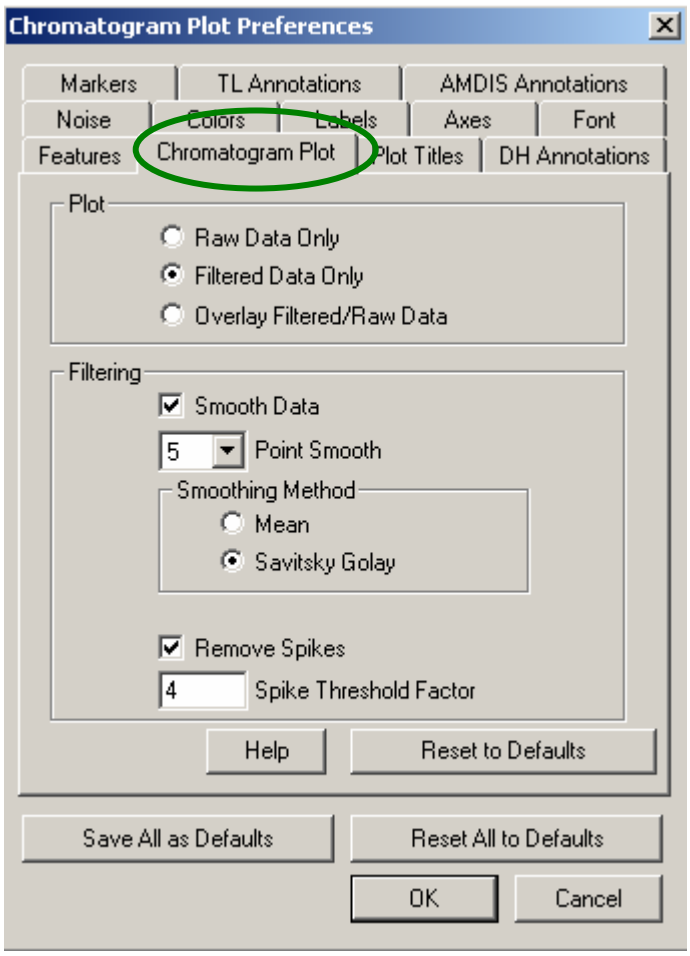
Chromatogram Plot Preferences
Markers
TL Annotations
AMDIS Annotations
Noise
Colors
Labels
Axes
Font
Features
Chromatogram Plot
Plot Titles
DH Annotations
Plot
Raw Data Only
Filtered Data Only
Overlay Filtered/Raw Data
Filtering
Smooth Data
5
Point Smooth
Smoothing Method
Mean
Savitsky Golay
Remove Spikes
4
Spike Threshold Factor
Help
Reset to Defaults
Save All as Defaults
Reset All to Defaults
OK
Cancel

Figure 5.7

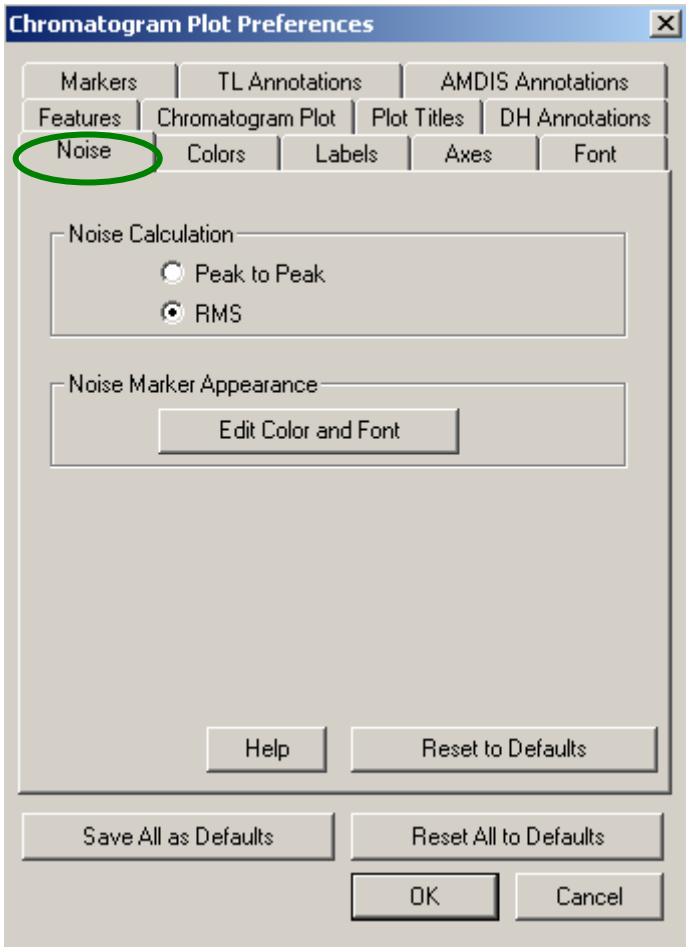
Chromatogram Plot Preferences
Markers
TL Annotations
AMDIS Annotations
Features
Chromatogram Plot
Plot Titles
DH Annotations
Noise
Colors
Labels
Axes
Font
Noise Calculation
Peak to Peak
RMS
Noise Marker Appearance
Edit Color and Font
Help
Reset to Defaults
Save All as Defaults
Reset All to Defaults
OK
Cancel

Figure 5.8

From the Chromatogram pull down menu, set Select Spectra to Average to 3 (see Figure 5.9).

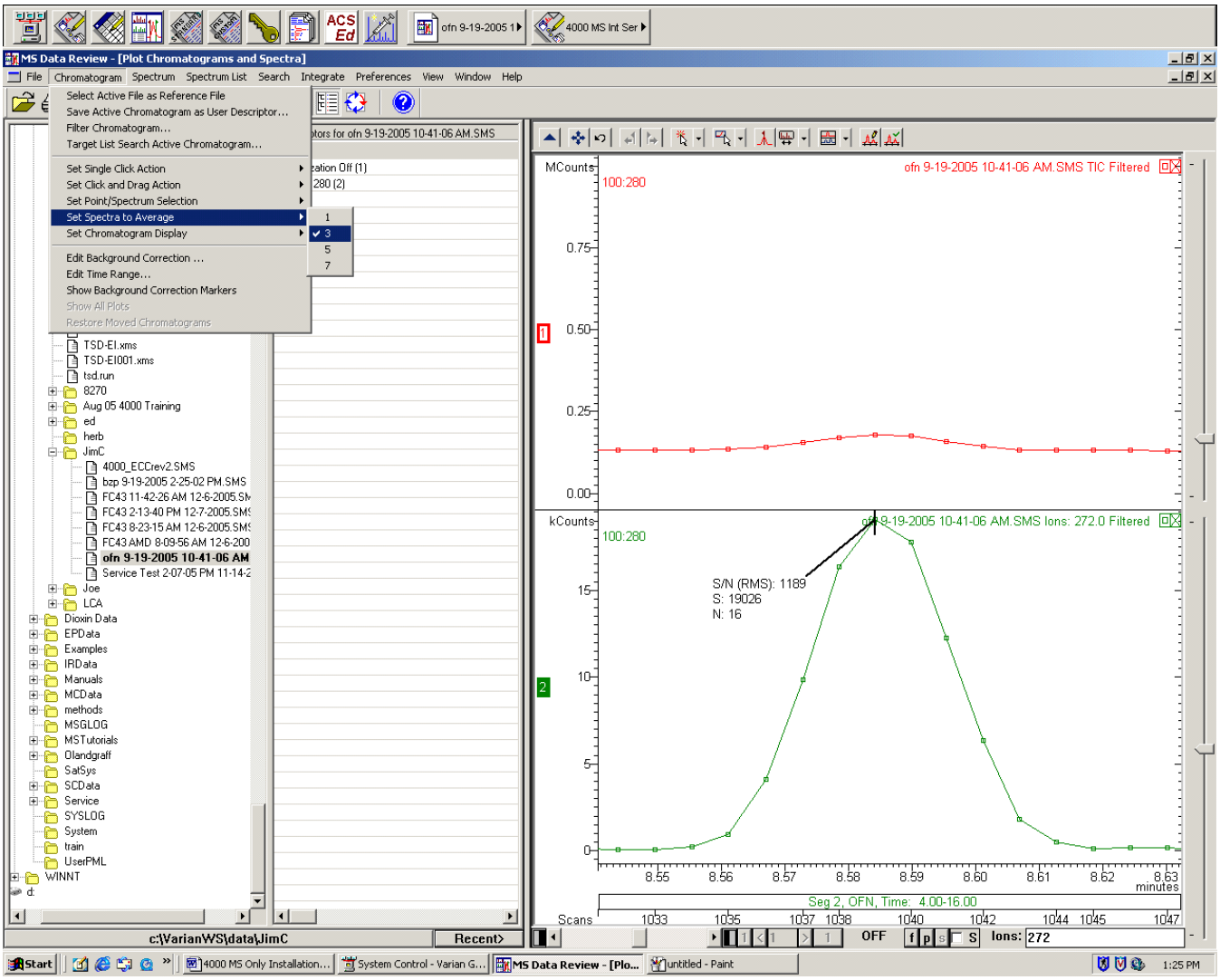

Figure 5.9

From the Chromatogram pull down menu select Edit Background Correction… (see Figure 5.10).

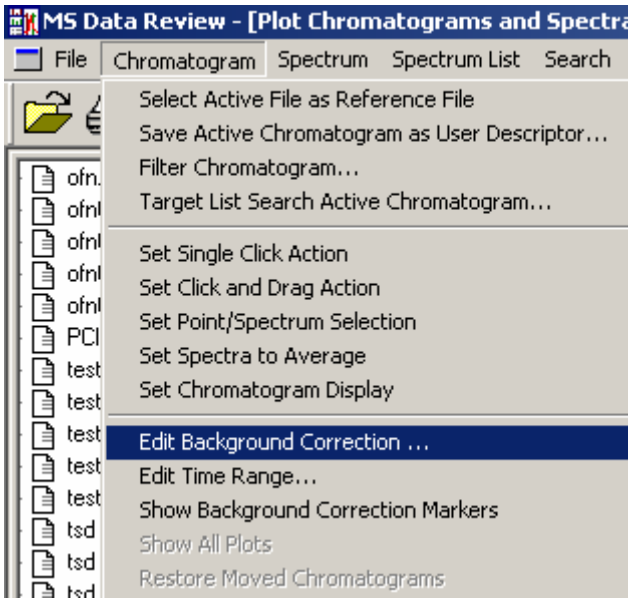

Figure 5.10

Click Auto Background Correction and Done in the Background Correction dialog box (see Figure 5.11).

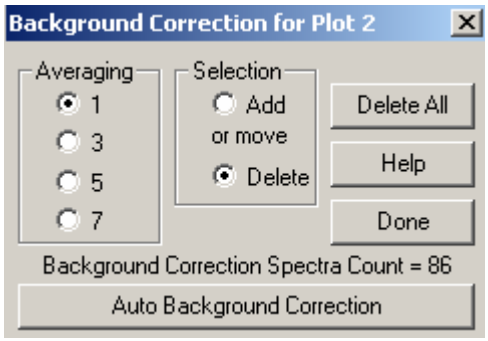

Figure 5.11

Click the apex scan of the m/z 272 single ion chromatogram to generate the spectrum. Note the BC in the upper right corner of the spectral window which indicates a background corrected spectrum (see Figure 5.12).

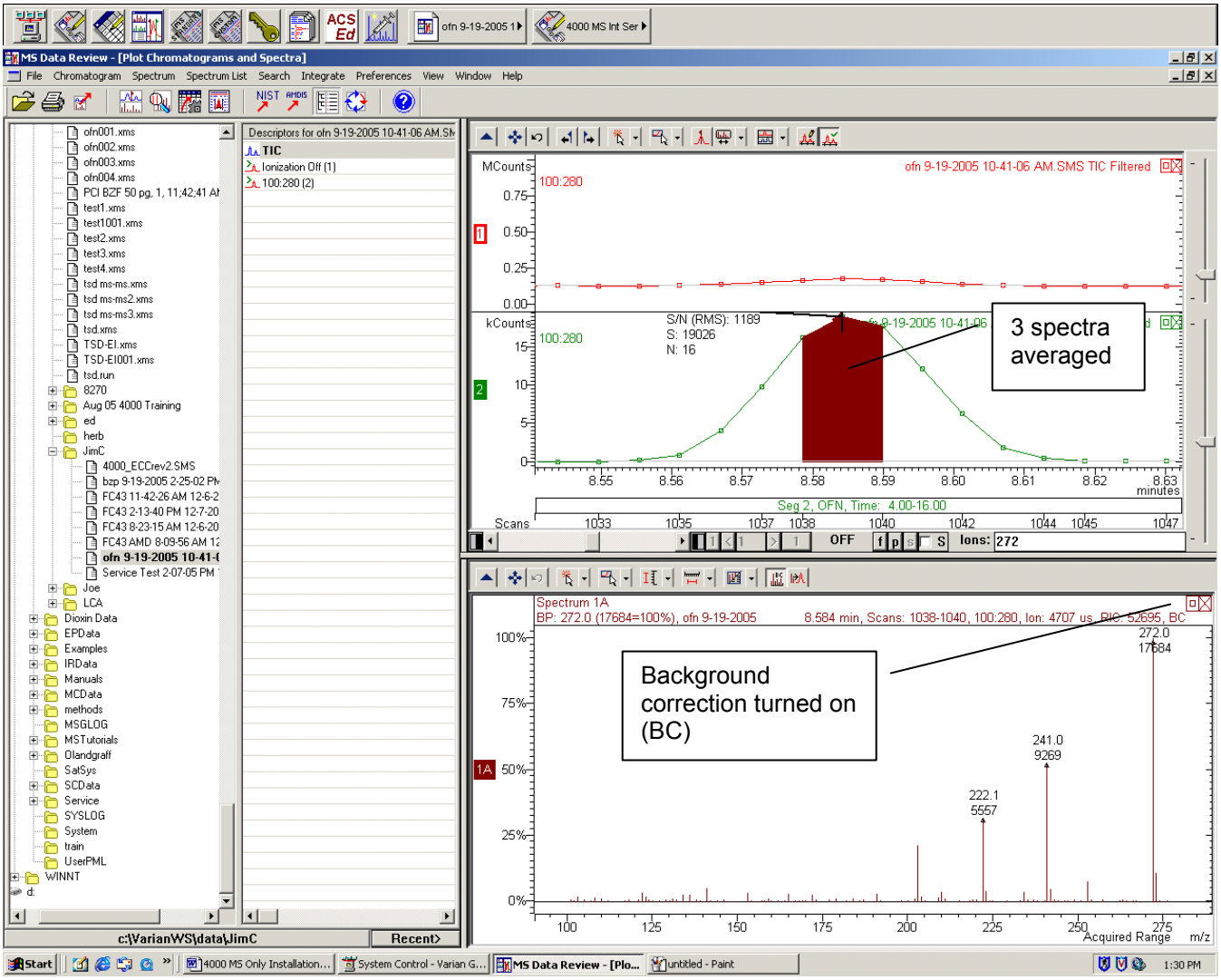

Figure 5.12

From the Search pull down menu select Library Search a Spectrum / 1A (see Figure 5.13).

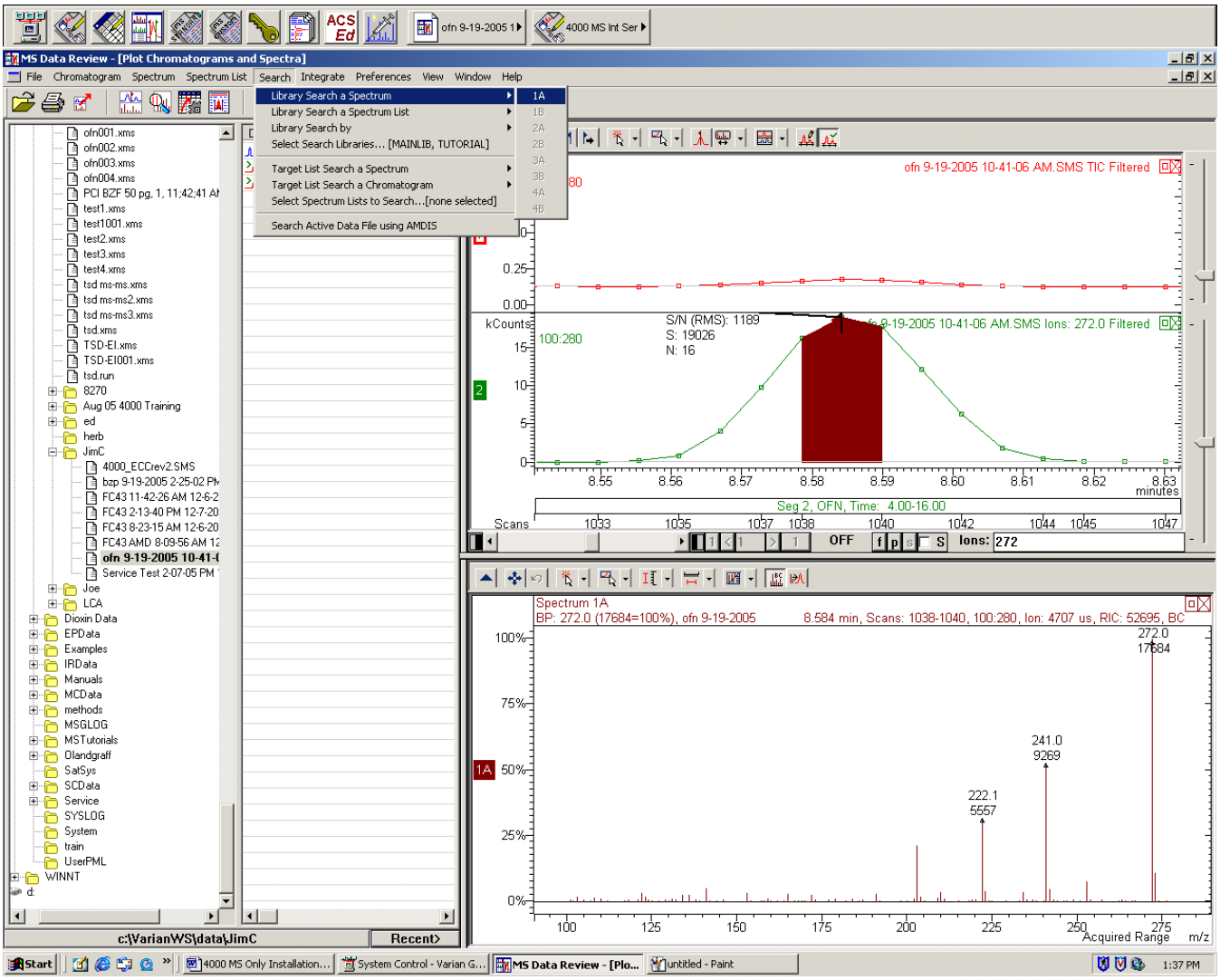

Figure 5.13

Figure 5.14 shows Octafluoronaphthalene with a match score of > 500. Note that OFN is in the Tutorial library.

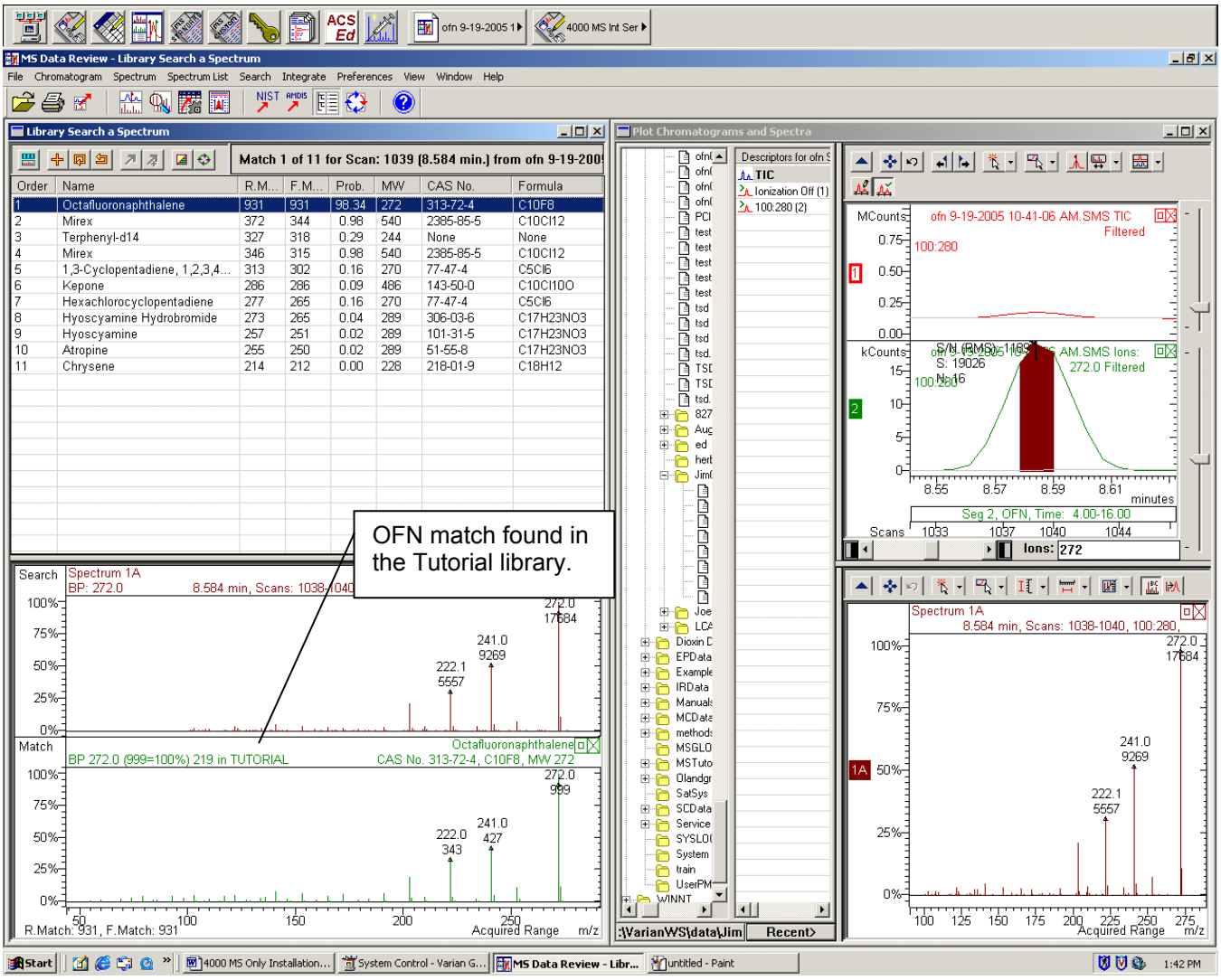

Figure 5.14

Click the Do NIST Search button  and select Spectrum Search… to edit the library search parameters and re-search if necessary. Suggested library search parameters are shown in Figure 5.15

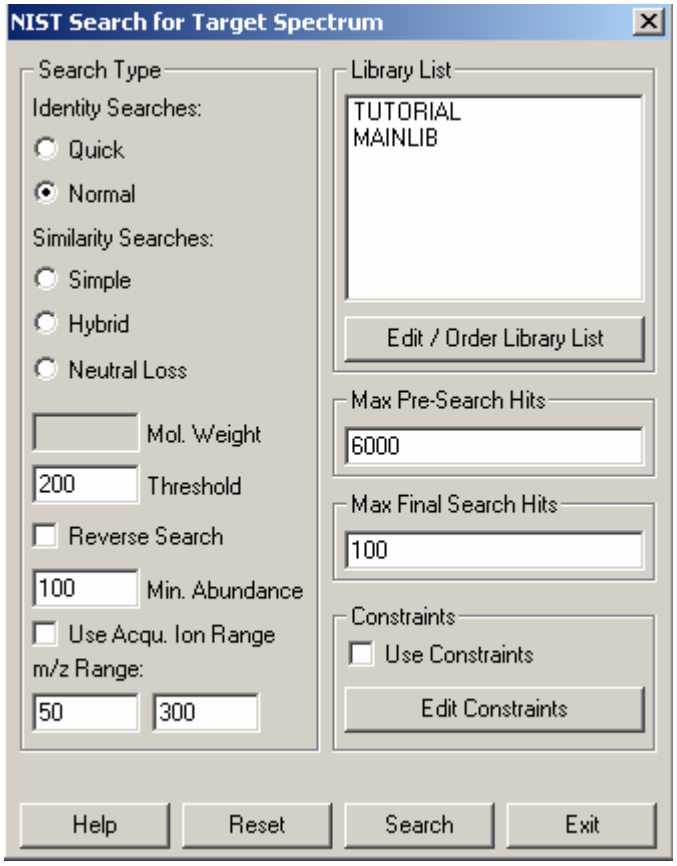

Figure 5.15

5.4 Customer Familiarization

An important part of each installation is familiarizing the user with the following items:

- Software features including: Diagnostics, Manual Control, Auto Tune, Acquisition, and MS Data Review with library searching. Emphasize that there are critical (affecting system performance) and non-critical fault messages.
- Library searching using the PurgeB standard (00-996882-03) The column test mix is NOT recommended for demonstrating library searching.
- Hardware features with emphasis on user accessible items.
- Daily and routine maintenance including: Manual Control, the Maintenance section of the Hardware Manual (03-914998-00), and parts ordering.
- Troubleshooting including: Manual Control, Diagnostics, the Troubleshooting section of the Hardware Manual, and calling Technical Support.
- Warranty issues such as the system includes a 1 year warranty and extended contract coverage is available.
- Safety issues including proper venting of the roughing pump fumes, discussion of heated zones, and Varian's policy of not allowing customers to perform maintenance that exposes them to high voltage areas of the system.

Customer familiarization at installation should be done throughout the installation visit as opportunities arise. This installation task is only meant to familiarize the customer with the basic features of the system. If more thorough training is required the customer should be advised to contact a sales representative to purchase one of Varian's training offerings.

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Section 6. Achieving Internal CI Specifications

The PCI specification for a 4000 GCMS system in Internal mode:

- 50:1 RMS signal-to-noise using the m/z 183 ion from a 1 µL injection of a 5 pg/µL BZP solution (03-931126-01). The required CI reagent is high purity (> 99.95%) methanol.

6.1 Install Liquid CI

Before introducing methanol into the CI plumbing it may be necessary to condition the CI plumbing to remove air and water by opening the CI solenoids in Manual Control. Install the 4000 MS Liquid CI Option according to the instructions included with the option. A picture of the installed liquid option is shown in Figure 6.1. Note that the 4000 MS inlet port for the CI restrictor is different from the inlet port on a 2000 MS.

Figure 6.1

6.2 Adjust Liquid CI Reagent Flow

Go to the Checks and Adjustments tab in Manual Control, select CI Gas Adjustment and the Reagent Methanol as shown in Figure 6.2, and click **Start**.

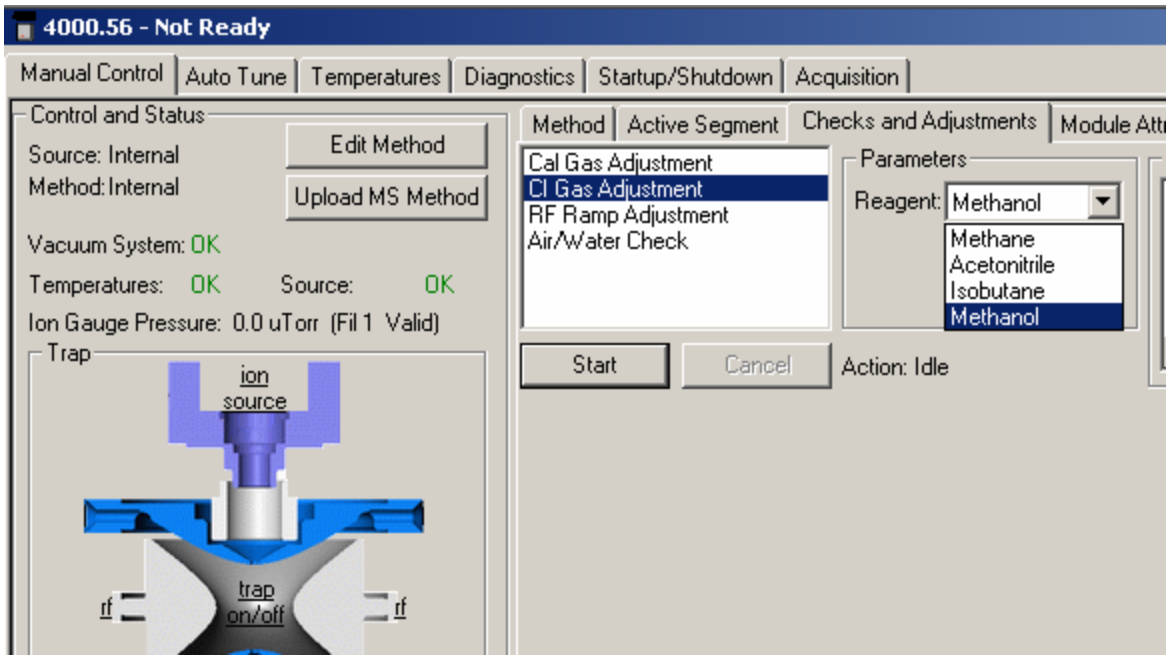

Figure 6.2

Open the 4000 MS front door and adjust the CI needle until the ratio between the m/z 33 and m/z 31 ions is approximately 10:1 (33:31 ≈ 10:1). Click **Done** when finished. To minimize contamination do not open the CI needle valve more than necessary. Figure 6.3 shows an example of properly adjusted methanol.

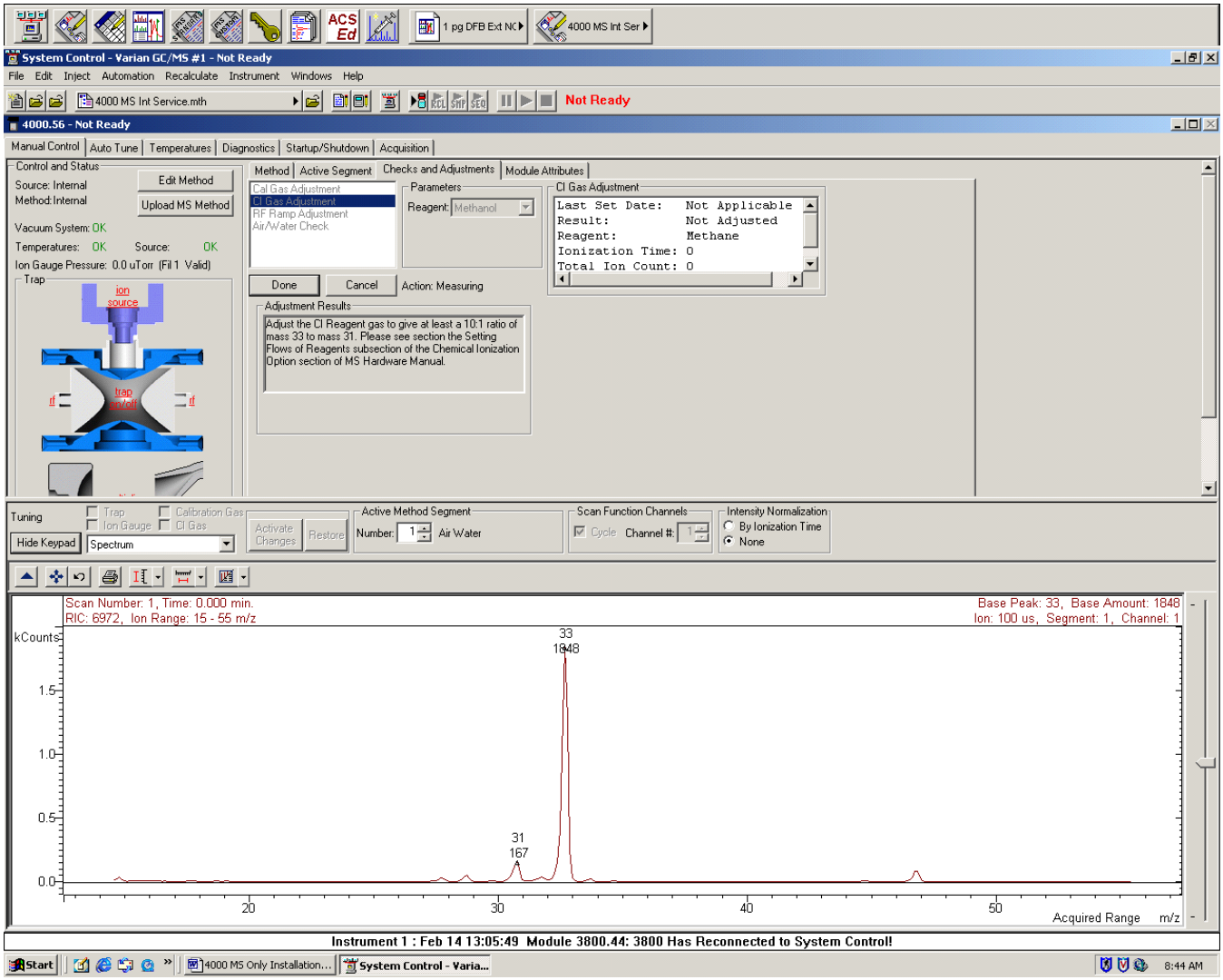

Figure 6.3

6.3 Methodology

NOTE: The 4000 GCMS system will arrive with a Test Files CD containing factory test method and data files. These method files can be used to pass the sensitivity tests associated with the installation. Retain the test files CD for your records.

Ensure the MS temperatures are set for this acquisition:

- Trap: 150 °C
- Manifold: 40 °C
- Transfer Line: 230 °C

In System Control, activate the internal CI method from the Test Files CD.

6.4 BZP Acquisition

Use the Inject Single Sample option to make a 1µL manual injection of test mix 03-931126-01. Use a lower air gap to avoid sample loss through the tip of the needle. Ensure the syringe plunger is not forced out of the syringe barrel if a Pressure Pulse is used in the method. Inject slowly (1µL/sec.) to avoid sample loss through explosive vaporization. Ensure the split valve has changed from opened to closed before injecting by striking the injector auto-start switch with the syringe barrel before depressing the syringe plunger.

6.5 Signal-to-noise Calculation

Use the quick load option shown in Figure 5.2 to load the BZP data file into MSDR. Plot a single ion chromatogram of the m/z 183 ion. Refer to Figures 5.3 and 5.4 but be sure to plot ion m/z 183. Click the apex scan to display the spectrum. Refer to Figures 5.10 and 5.11 and background correct the spectrum. Right mouse click the apex scan and select Calculate Noise (refer to Figure 5.5). The RMS signal-to-noise calculation will be performed for the scan closest to where the cursor was placed for the right mouse click. Use 5 Point Savisky Golay smoothing. A passing signal-to-noise calculation is shown in Figure 6.4.

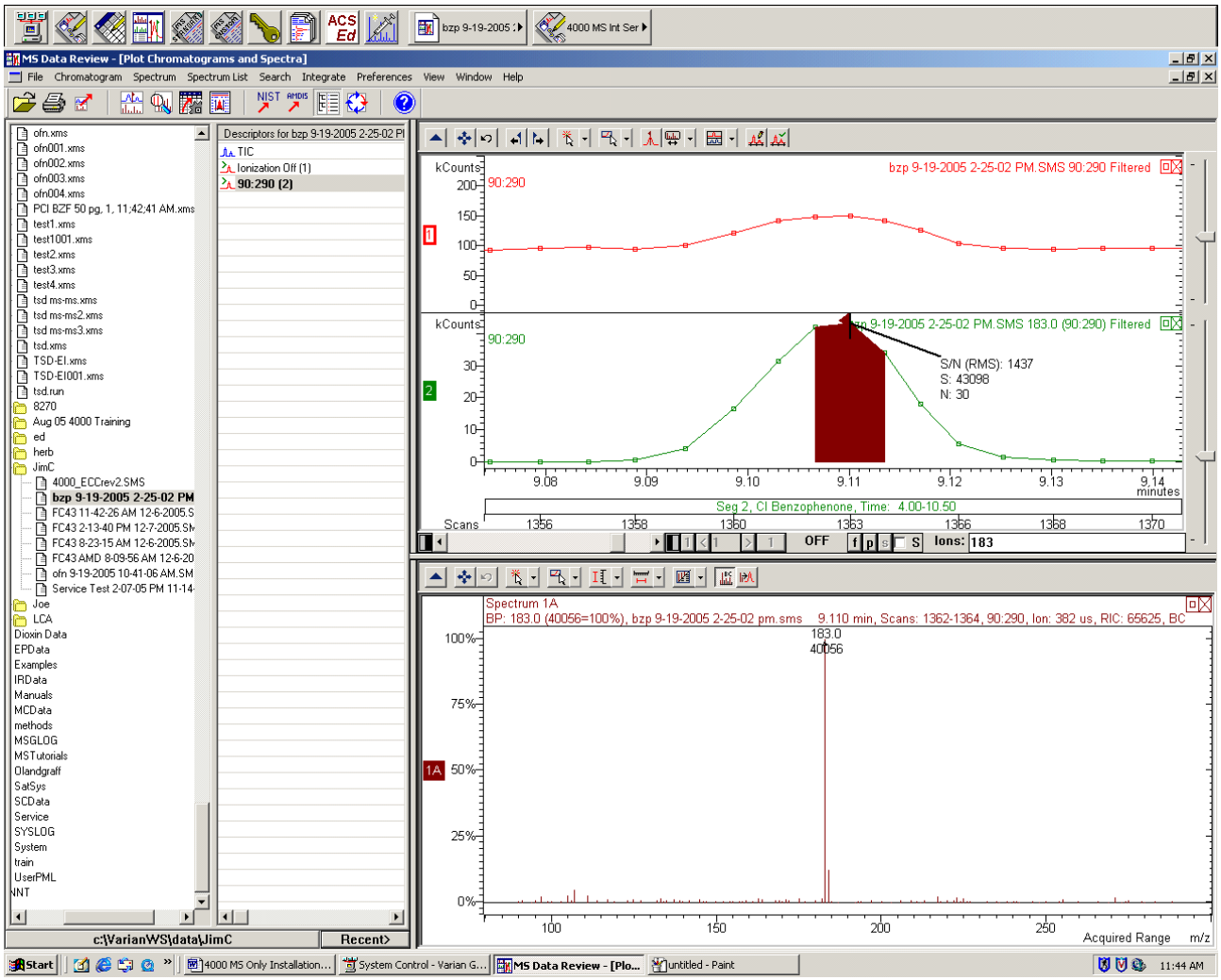

Figure 6.4

Section 7. Achieving External EI Specifications

If necessary, convert the MS to external mode as described in the Hardware Manual. Purge the MS pneumatics as described in Section 2.1. Auto tune the system as described in Section 4.2. Ensure the MS damping gas flow is set appropriately and the Getter is at least 300 °C. The appropriate Damping Gas flow rates can be found embedded in the names of the test data files from the Test Files CD included with the system, and also in the Module Attributes section of the Data File Information dialog of each external data file. Set the MS damping gas flow rate to this value to ensure optimal system performance. Refer to section 2.1 for help in programming the MS damping gas flow rate

The EI specifications for a 4000 GCMS system in External mode:

- 30:1 RMS signal-to-noise using the m/z 272 ion from a 1 µL injection of a 5 pg/µL OFN solution (03-931127-02). A 10:1 split ratio can be used to reduce the amount of OFN from 5 pg to 500 fg. If necessary, a 10:1 dilution can be done using customer supplied glassware and 2,2,4-trimethylpentane (isooctane) to reduce the amount of OFN from 5 pg to 500 fg. In addition, a background corrected spectrum should be library searchable with any match > 500. If a Quick Switch Valve is used in the GC oven the signal-to-noise spec is reduced from 30:1 to 15:1.

7.1 Methodology

NOTE: The 4000 GCMS system will arrive with a Test Files CD containing factory test method and data files. These method files can be used to pass the sensitivity tests associated with the installation. At the factory a diluted OFN standard is injected using a pressure pulse and splitless injection technique. If a diluted OFN standard is to be used at an installation the method from the Test Files CD can be used with no editing. If a split style injection is to be used at an installation, the Test Files CD method should be edited to remove the pressure pulse and introduce a 10:1 split ratio. Retain the Test Files CD for your records.

Ensure the MS temperatures are set for this acquisition:

- Trap: 150 °C
- Manifold: 40 °C
- Transfer Line: 230 °C
- Source: 150 °C

If necessary, edit the Test Files CD external EI method and activate the method in System Control.

7.2 OFN Acquisition

Use the Inject Single Sample option to make a 1 µL manual injection of test standard 03-931127-02 (split style injection) or a 10:1 diluted test standard (splitless pressure pulse style injection). Use a lower air gap to avoid sample loss through the tip of the needle. Ensure the syringe plunger is not forced out of the syringe barrel if a Pressure Pulse is used in the method.

7.3 Library Search and Signal-to-noise Calculation

After acquiring the data file, calculate the S/N and run a library search. Use this opportunity to demonstrate data analysis to the customer. The .sms data file can be loaded into Mass Spectra Data Review (MSDR) using the quick load feature shown in Figure 5.2. Refer to Section 5 of this document for assistance in plotting the m/z 272 ion from the OFN acquisition, calculating the RMS signal-to-noise, and achieving a passing library search. Be sure to use 5 point Savitsky Golay smoothing on the chromatogram and background correction on the spectrum. A passing signal-to-noise calculation and library search are shown in Figure 7.1. Note that OFN is found in the Tutorial Library.

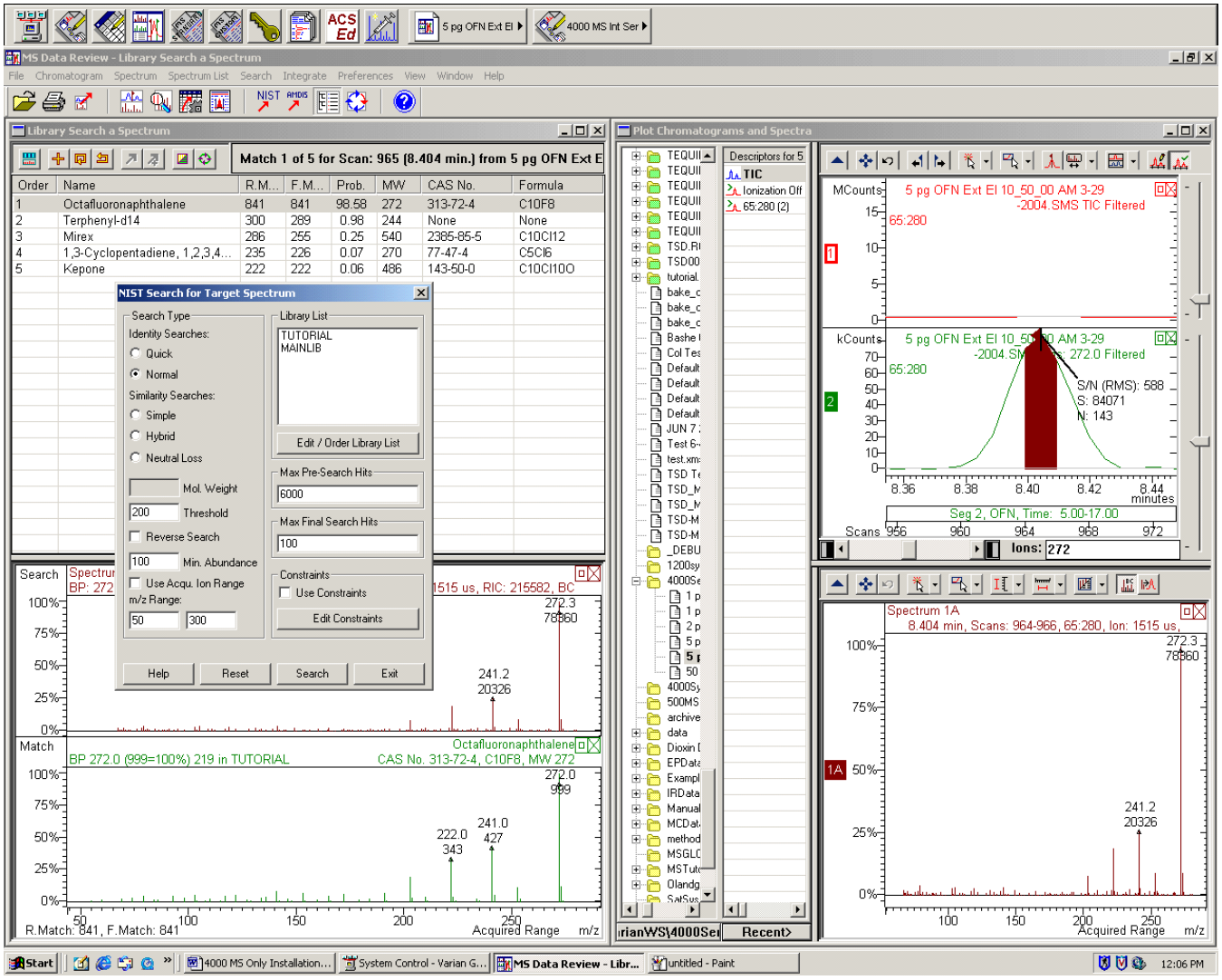

Figure 7.1

Section 8. Achieving External PCI Specifications

The PCI specification for a 4000 GCMS system in External mode:

- 20:1 RMS signal-to-noise using the m/z 183 ion from a 1 µL injection of a 50 pg/µL BZP solution (03-920305-00). The required CI reagent is high purity (> 99.95%) methane.

8.1 Install Gaseous CI

Before introducing methane into the CI plumbing it may be necessary to condition the CI plumbing to remove air and water by opening the CI solenoids in Manual Control. Install or convert to gaseous CI according to the instructions in the CI Kit. The external PCI specification test run should be performed using 25 psi line pressure of high purity methane as the CI reagent gas. High purity (at least 99.95%) methane gas is recommended to minimize source contamination.

8.2 Adjust Gaseous CI Reagent Flow

Turn the CI needle valve behind the MS front door counter clockwise (CCW) until it stops. Go to the Checks and Adjustments tab in Manual Control and select CI Gas Adjustment as shown in Figure 6.2. Be sure to select **Methane** as the Reagent and click **Start**. Slowly turn the CI needle valve behind the MS front door clockwise (CW) until ion signal begins to show in the spectral window. Continue turning the CI needle valve CW only as long as the m/z 29 signal intensity increases. Once the signal intensity of m/z 29 has maximized further CW turning of the CI needle valve will degrade system performance by introducing hydrocarbon background. Typically, an Ion Gauge Pressure of 30 to 70 µTorr will be displayed when a proper adjustment is achieved as shown in Figure 8.1. The current factory recommendation is 50 uTorr. Click **Done** when finished.

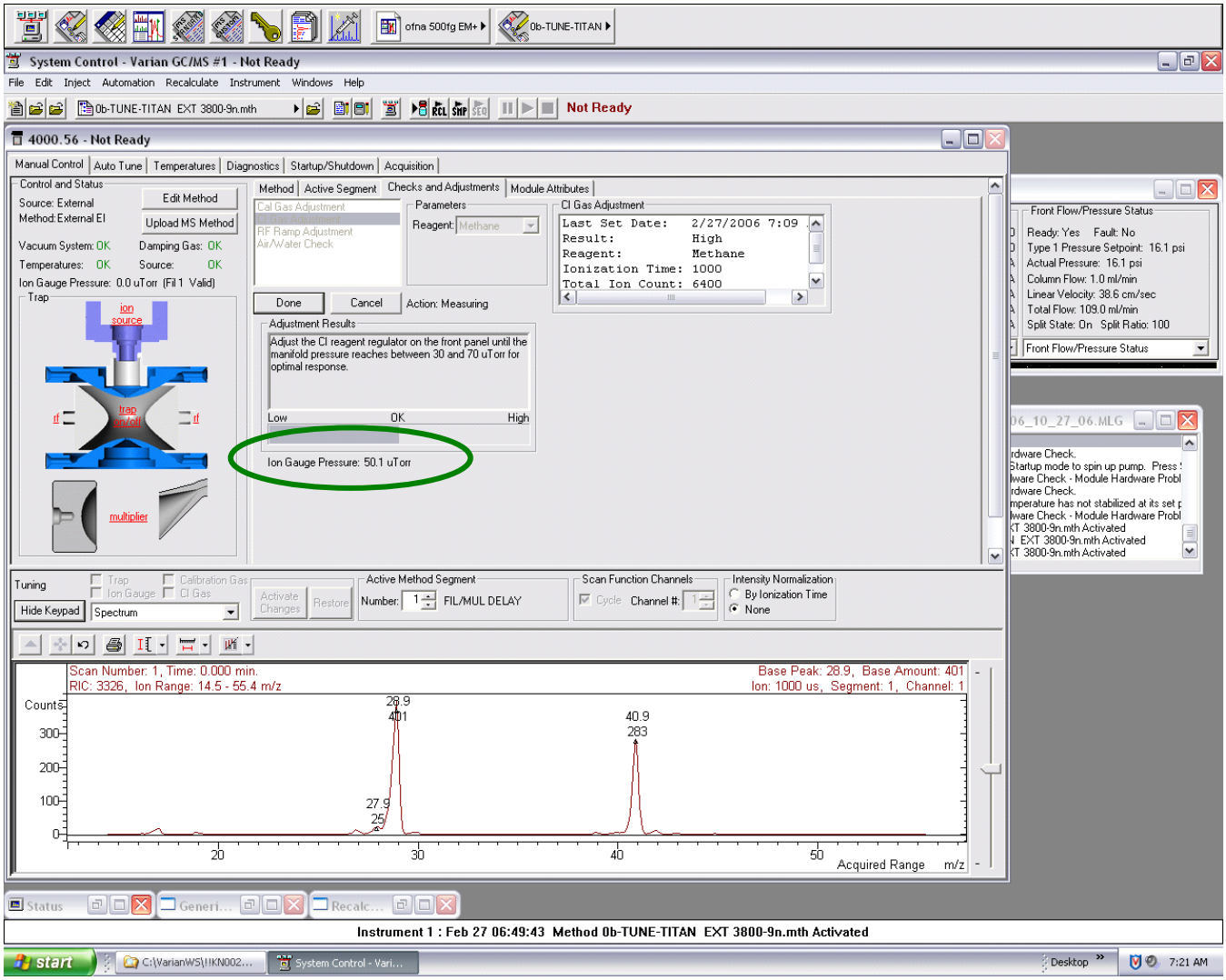
System Control - Varian GC/MS #1 - Not Ready
File Edit Inject Automation Recalculate Instrument Windows Help
0b-TUNE-TITAN EXT 3800-9n.mth
Not Ready
4000.56 - Not Ready
Manual Control | Auto Tune | Temperatures | Diagnostics | Startup/Shutdown | Acquisition
Control and Status
Source: External
Method: External EI
Edit Method
Upload MS Method
Vacuum System: OK
Damping Gas: OK
Temperatures: OK
Source: OK
Ion Gauge Pressure: 0.0 uTorr (Fil 1 Valid)
Trap
ion source
trap
multiplier
Method | Active Segment | Checks and Adjustments | Module Attributes
Cal Gas Adjustment
CI Gas Adjustment
RF Ramp Adjustment
Air/Water Check
Parameters
Reagent: Methane
CI Gas Adjustment
Last Set Date: 2/27/2006 7:09
Result: High
Reagent: Methane
Ionization Time: 1000
Total Ion Count: 6400
Done
Cancel
Action: Measuring
Adjustment Results
Adjust the CI reagent regulator on the front panel until the manifold pressure reaches between 30 and 70 uTorr for optimal response.
Low OK High
Ion Gauge Pressure: 50.1 uTorr
Front Flow/Pressure Status
Ready: Yes Fault: No
Type 1 Pressure Setpoint: 16.1 psi
Actual Pressure: 16.1 psi
Column Flow: 1.0 ml/min
Linear Velocity: 38.6 cm/sec
Total Flow: 109.0 ml/min
Split State: On Split Ratio: 100
Tuning
Hide Keypad
Spectrum
Activate Changes
Restore
Active Method Segment
Number: 1 FIL/MUL DELAY
Scan Function Channels
Cycle Channel #: 1
Intensity Normalization
By Ionization Time
None
Scan Number: 1, Time: 0.000 min.
RIC: 3326, Ion Range: 14.5 - 55.4 m/z
Base Peak: 28.9, Base Amount: 401
Ion: 1000 us, Segment: 1, Channel: 1
Counts
28.9
401
40.9
283
27.9
25
Acquired Range m/z
Instrument 1 : Feb 27 06:49:43 Method 0b-TUNE-TITAN EXT 3800-9n.mth Activated
start
System Control - Vari...
Desktop
7:21 AM

Figure 8.1

8.3 Methodology

NOTE: The 4000 GCMS system will arrive with a Test Files CD containing factory test method and data files. These method files can be used to pass the sensitivity tests associated with the installation. Retain the test files CD for your records.

Ensure the MS temperatures are set for this acquisition:

- Trap: 150 °C
- Manifold: 40 °C
- Transfer Line: 230 °C
- Source: 150 °C

In System Control, activate the external PCI method from the Test Files CD.

The appropriate MS damping gas flow rate is embedded in the name of the corresponding data file found on the Test Files CD included with the system. The Damping Gas flow rate can also be found in the Data File Information dialog in the Module Attributes section. Set the MS damping gas flow rate to this value to ensure optimal system performance. Refer to section 2.1 for help in programming the MS damping gas flow rate.

8.4 BZP Acquisition

Use the Inject Single Sample option to make a 1 µL manual injection of test mix 03-920305-00. Use a lower air gap to avoid sample loss through the tip of the needle. Inject slowly (1 µL/sec.) to avoid sample loss through explosive vaporization. Ensure the split valve has changed from opened to closed before injecting by striking the injector auto-start switch with the syringe barrel before depressing the syringe plunger.

8.5 Signal-to-noise Calculation

Use the quick load option shown in Figure 5.2 to load the BZP data file into MSDR. Plot a single ion chromatogram of the m/z 183 ion. Refer to Figures 5.3 and 5.4 but be sure to plot ion m/z 183. Click the apex scan to display the spectrum. Refer to Figures 5.10 and 5.11 and background correct the spectrum. Right mouse click the apex scan and select Calculate Noise (refer to Figure 5.5). The RMS signal-to-noise calculation will be performed for the scan closest to where the cursor was placed for the right mouse click. Use 5 Point Savisky Golay smoothing. A passing signal-to-noise calculation is shown in Figure 8.2.

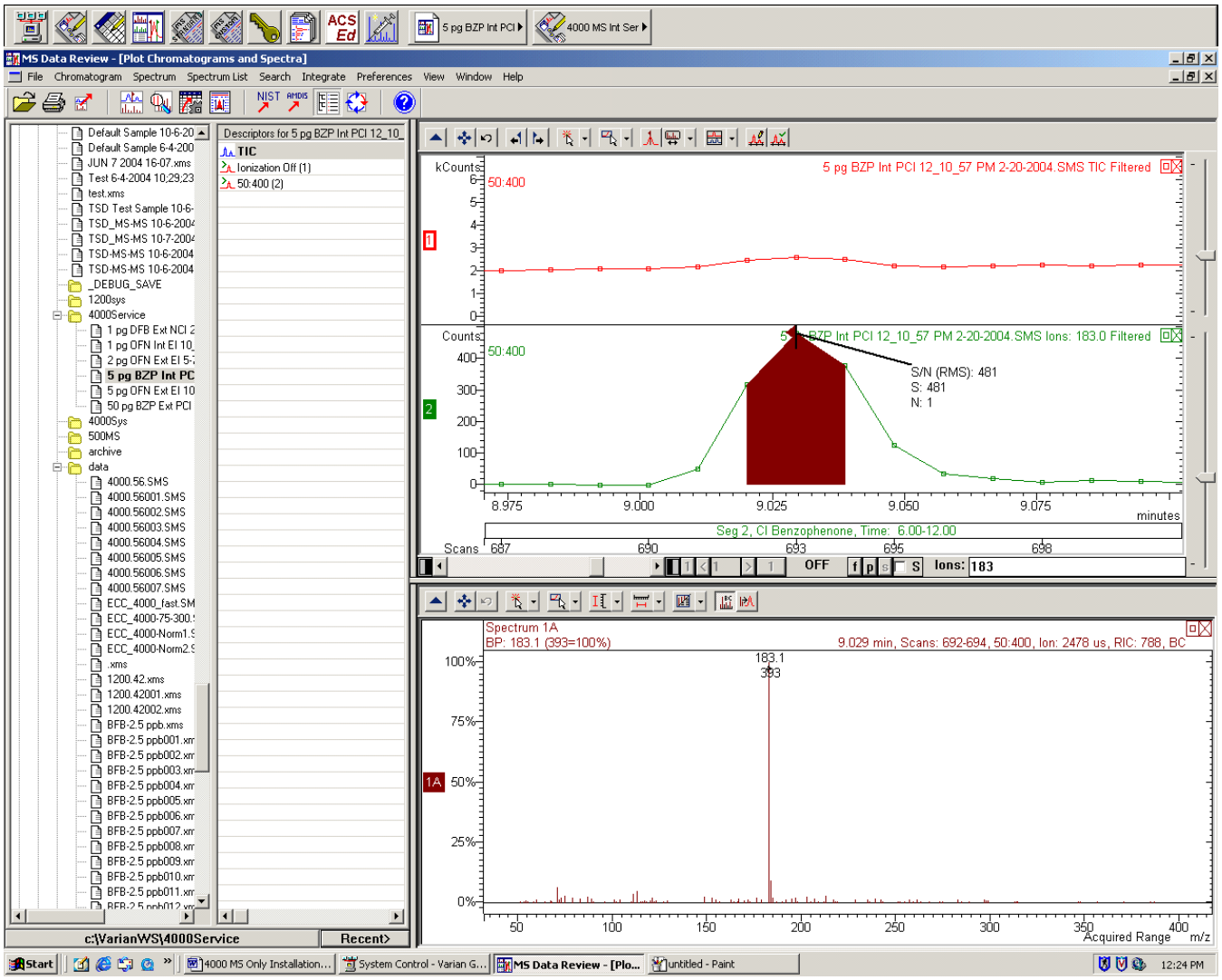

Figure 8.2

Section 9. Achieving External NCI Specifications

If necessary, install or convert to gaseous CI according to the instructions in the CI Kit. This external NCI acquisition should be performed using 25 psi line pressure of high purity methane as the CI reagent gas. High purity (at least 99.95%) methane gas is recommended to minimize source contamination. Set the gas flow rate according to the instructions in Section 8.2 of this document.

The NCI specifications for a 4000 MS upgrade in External mode:

- 50:1 RMS signal-to-noise using the m/z 362 ion from a 1 µL injection of a 1 pg/µL DFB solution (03-931130-01).

9.1 Methodology

NOTE: The 4000 GCMS system will arrive with a CD containing factory test method and data files. These method files can be used to pass the sensitivity tests associated with the installation. Retain the test files CD for your records.

Ensure the MS temperatures are set for this acquisition:

- Trap: 150 °C
- Manifold: 40 °C
- Transfer Line: 230 °C
- Source: 150 °C

In System Control, activate the external NCI method from the Test Files CD.

The appropriate MS damping gas flow rate is embedded in the name of the corresponding data file found on the Test Files CD included with the system. The Damping Gas flow rate can also be found in the Data File Information dialog in the Module Attributes section. Set the MS damping gas flow rate to this value to ensure optimal system performance. Refer to section 2.1 for help in programming the MS damping gas flow rate.

9.2 DFB Acquisition

Use the Inject Single Sample option to make a 1 µL manual injection of test mix 03-931130-01. Use a lower air gap to avoid sample loss through the tip of the needle. Inject slowly (1 µL/sec) to avoid sample loss through explosive vaporization. Ensure the split valve has changed from opened to closed before injecting by striking the injector auto-start switch with the syringe barrel before depressing the syringe plunger.

9.3 Signal-to-noise Calculation

Use the quick load option shown in Figure 5.2 to load the BZP data file into MSDR. Plot a single ion chromatogram of the m/z 362 ion. Refer to Figures 5.3 and 5.4 but be sure to plot ion m/z 362. Click the apex scan to display the spectrum. Refer to Figures 5.10 and 5.11 and background correct the spectrum. Right mouse click the apex scan and select Calculate Noise (refer to Figure 5.5). The RMS signal-to-noise calculation will be performed for the scan closest to where the cursor was placed for the right mouse click. Use 5 Point Savisky Golay smoothing. A passing signal-to-noise calculation is shown in Figure 9.1.

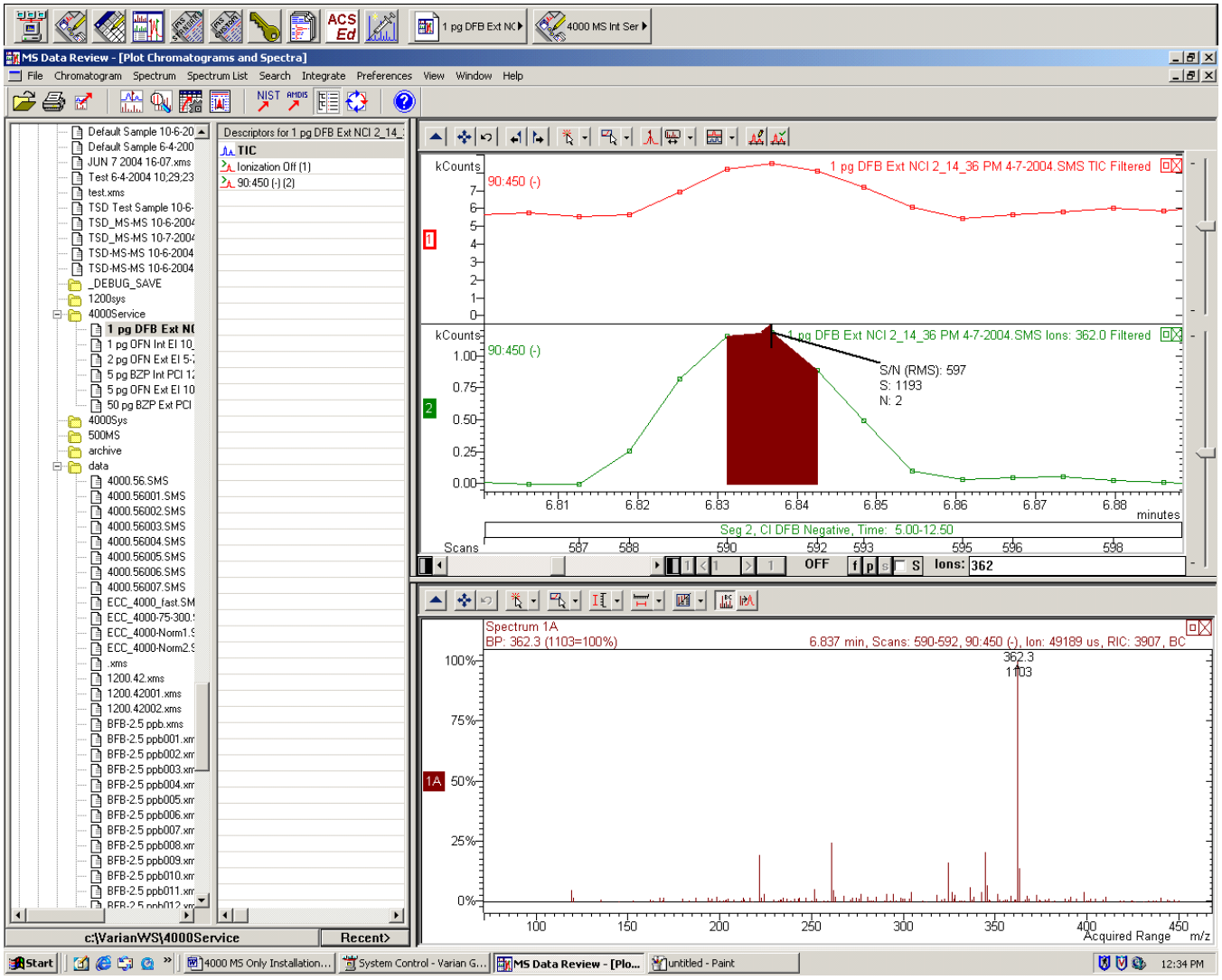

Figure 9.1

Appendix A. 4000 MS Installation Specifications and Test Samples

Ionization Mode	Test Sample	Amount of Analyte Measured	m/z	RMS S/N Specification	Library Match (any search type)
Internal EI	10:1 split or dilution of 03-931126-01 (2 pg/µL OFN, 5 pg/µL BZP)	200 fg OFN	272	≥ 20:1	≥ 500
Internal CI	03-931126-01 (2 pg/µL OFN, 5 pg/uL BZP)	5 pg BZP	183	≥ 50:1	N/A
Internal EI with a Quick Switch Valve in the GC	10:1 split or dilution of 03-931126-01 (2 pg/µL OFN, 5 pg/uL BZP)	200 fg OFN	272	≥ 10:1	≥ 500
External EI	10:1 split or dilution of 03-931127-02 (5 pg/µL OFN)	500 fg OFN	272	≥ 30:1	≥ 500
External PCI	03-920305-00 (50 pg/µL BZP)	50 pg BZP	183	≥ 20:1	N/A
External NCI	03-931130-01 (1 pg/µL DFB)	1 pg DFB	362	≥ 50:1	N/A
External EI with a Quick Switch Valve in the GC	10:1 split or dilution of 03-931127-02 (5 pg/µL OFN)	500 fg OFN	272	≥ 15:1	≥ 500