TRACE DSQ™
and
FOCUS DSQ

User's Guide

PN 120156-0003
Revision B

Analyze • Detect • Measure • Control™

Technical Support

Thermo Electron Corporation offers Technical Support via phone Monday through Friday 9:00 AM to 6:00 PM Eastern US time. Our staff of certified professionals will answer your technical questions regarding all Thermo Electron Corporation product lines. Please contact us by phone/fax or mail at:

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Technical Publications: K. Johns

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Important Information

Copyright

FCC Compliance

FCC part 15, Class A

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy. If it is not installed and used in accordance with the instruction manual, it may cause harmful interference to radio communication. Operation of this equipment in a residential area is likely to cause harmful interference. In this case, users will be required to correct the interference at their own expense.

NOTE: *TRACE DSQ or FOCUS DSQ* systems operate reliably under carefully controlled environmental conditions. If you maintain a system outside the specifications listed in this guide, failures of many types may occur. The repair of such failures is specifically excluded from the Standard Warranty and service contract coverage.

Regulatory Compliance

Thermo Electron thoroughly tests and evaluates its products to ensure full regulatory compliance with applicable domestic and international regulations. Your system (instruments and software) is CE compliant and meets Electromagnetic Compatibility (EMC) and safety standards.

- EN 55011 (1991) Group 1 Class A
- EN 50082-1 (1992) IEC 801.2 (1991) Level 3
- EN 50082-1 (1992) IEC 801-3 (1984) Level 2
- EN 50082-1 (1992) IEC 801-4 (1988) Level 2
- EN 50082-1 (1992) IEC 801-5 (1993) Class 3

CAUTION
Instrument Damage

Please be aware that any changes you make to your system may void compliance with one of more of these EMC or safety standards, or warranty.

To ensure compliance with EMC and safety standards, order replacement parts from only Thermo Electron factory-trained representatives. If you make any changes to your system, you may void compliance with one or more of these EMC or safety standards, or warranty. Changes to your system include replacing a part.

Safety Compliance

- EN 61010-1 1993 plus Amendment 2, 1995

Standard Warranty

This standard warranty covers all products manufactured by Thermo Electron hereinafter referred to as the "Seller".

WARRANTY AND LIMITATIONS. Seller warrants that the products to be delivered hereunder "products" are free from defects in material and workmanship and meet Seller's field performance specifications as published by the Seller, provided, however, that

1. Seller's liability under this warranty is, at the discretion of the Seller, limited to repairing or replacing or issuing a credit for any product delivered hereunder not conforming to this warranty.

2. The alternative remedies provided for herein are Buyer's sole and exclusive remedies.

3. Except as otherwise provided in this document, Buyer's warranty on all products is limited to a period of ninety (90) days, commencing with the date of acceptance of any such product or 120 days after shipment, whichever occurs first.

4. Minor deviations from specifications that do not affect performance of the products covered hereby are excluded from this warranty.

5. Warranty on repaired or replaced parts is extended only to the expiration date of an issued standard warranty, and only after receipt of written authorization from Seller.

6. Seller makes no warranty as to expendable items including but not limited to electron multipliers, filaments, O-rings, vacuum gauge tubes, fuses, septa, lamps, sources, reaction tubes, and solvents.

7. Moreover, after initial installation, any readjustment, recleaning or recalibration is expressly excluded from this warranty.

8. The warranty will be null and void and Seller will not be liable under this warranty upon the occurrence of the following events unless Seller's prior written consent to the continuation of this warranty is granted:

   a. In the event Buyer installs products or physically moves products from their position of installation at time of acceptance by Buyer, or

   b. In the event Buyer transfers ownership of products to third parties other than Buyer.

9. Seller will not be liable under this warranty unless:

   (i) Seller is promptly notified in writing by Buyer upon discovery of the failure of any products to conform to this warranty,

   (ii) when possible, such, product is returned to Seller with Seller's written approval, transportation charges prepaid by Buyer,

   (iii) such product is received by Seller not more than ten (10) days after the last day of the applicable warranty period,

   (iv) Seller's examination of such product shall disclose to Seller's reasonable satisfaction that such defects or failures have not been caused by misuse, neglect, improper installation by Buyer, repair, alteration or accident, including but not limited to failure of electric power, excessive or erratic power, failure of environmental control equipment as defined in Seller's published specifications, failure of Buyer's self-maintenance program, or failure of Buyer to maintain product in accordance with Seller's manual or instructions.

10. All claims under warranty must be made promptly after occurrence of circumstances giving rise thereto and must be received within the applicable warranty period by Seller or its factory-trained representative.

    Such claims should include the product type and serial numbers and a full description of the circumstances giving rise to the claim. At it discretion, Seller may authorize the prepaid return to Seller's plant or designated repair center of products or parts that prove to be defective, or Seller may repair or replace at Buyer's premises. Before any products are returned for repair and / or adjustment, written authorization from Seller for the return and instructions as to how and where the product should be shipped must be obtained.

11. When any product is returned for examination and inspection or for any other reason, Buyer shall be responsible for all damage resulting from improper packing or handling and for loss in transit, notwithstanding any defect or nonconformity in the product.

If it is found that the defective product returned to Seller has been caused by the fault of the Buyer, or has been returned without cause and is still serviceable, Seller shall notify Buyer and shall return such product to Buyer at Buyer's expense; in addition, a charge for testing and examination, may at Seller's discretion, be made on products so returned.

12. Seller will not authorize return or repair or replacement at Buyer's lab of defective products that have been used for the analysis of toxic, carcinogenic, mutagenic or corrosive / irritant chemicals until said products are certified to have been decontaminated by Buyer.

Seller shall not warranty products that have not been decontaminated, and Seller shall have no obligation to repair or replace such products. Any products returned to Seller for examination shall be sent prepaid via the means of transportation indicated as acceptable to Seller.

13. Seller reserves the right to reject any warranty claim not made in accordance with the foregoing procedures or any warranty claim on any item that has been altered or has been shipped by non-acceptable means of transportation.

14. Except as above expressly provided, no warranty is made as to the merchant ability of the goods to be delivered, nor is except as expressly provided above, no warranty is made as to the fitness of such goods for any particular purpose.

15. Statements, expressed or implied, by any person, including employees or factory-trained representatives of Seller, that are inconsistent or in conflict with this warranty shall not be binding upon Seller unless said statement is in writing and signed by an officer of Seller.

a. Liability: In no event shall Seller be liable to buyer or any other party for incidental, consequential, or special damages.

b. Damages: Seller's liability (whether by reason of breach of warranty, breach of contract, tort, or otherwise and irrespective of Seller's negligence) for damages shall in no event exceed the payment, if any, received by seller for the unit or productor service furnished or to be furnished, as the case may be, which is the subject of claim or dispute.

August 1998
This warranty shall be governed by the laws of the State of California
P / N QSF-PR-0012 Rev A

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Reader Survey...

Trace DSQ & Focus DSQ User's Guide, PN 120156-0003, Revision B. Help us improve the quality of our manuals by circling one number for each statement:

	Strongly Agree	Agree	Disagree	Strongly Disagree
The manual is well organized.	1	2	4	5
The manual is clearly written.	1	2	4	5
The manual contains all the information I need.	1	2	4	5
The instructions are easy to follow.	1	2	4	5
The instructions are complete.	1	2	4	5
The technical information is easy to understand.	1	2	4	5
Examples of operation are clear and useful.	1	2	4	5
The figures are helpful.	1	2	4	5
I was able to operate the system using this manual. (If not please comment below.)	1	2	4	5

Additional Comments. Attach additional sheets if necessary.

fold

_____ _____
_____ _____
_____ _____
_____ _____

Customer Registration...

MY ORGANIZATION IS: (Check only one)

- ☐ Commercial (for profit) lab
- ☐ Government lab
- ☐ Hospital/Clinic
- ☐ Industrial lab
- ☐ Research Institute
- ☐ University/College
- ☐ Veterinary
- ☐ Other_____

MY PRIMARY APPLICATION IS: (Check only one)

- ☐ Analytical
- ☐ Biomedical
- ☐ Clinical/Toxicology
- ☐ Energy
- ☐ Environmental
- ☐ Food/Agricultural
- ☐ Forensic/Toxicology
- ☐ Pharmaceutical
- ☐ Research/Education
- ☐ Other_____

Job Function is: (Check one only)

- ☐ Administration
- ☐ Lab management
- ☐ Operator
- ☐ Other_____

Name _____ Title_____ _____
Company _____ _____
Address _____ _____
City/State/Postal Code_____ _____
Country _____ _____
Telephone _____ Ext._____ _____
System Type _____ Serial Number _____ Date purchased _____

From _____

_____

_____

_____

PLACE
STAMP
HERE

Editor, Technical Publications
THERMO ELECTRON Corporation
2215 GRAND AVENUE PKWY
AUSTIN TX 78728-3812
UNITED STATES OF AMERICA

Contents

Read Me

In This Chapter

Analyze • Detect • Measure • Control™

This chapter explains: how this book is organized and designed, specifications about your system, and where to find more information about your system.

Be sure to tear out the Contact Us... card located at the front of this book and return it to us after filling out the Reader Survey and the Customer Registration forms. The Reader Survey allows you to give us feedback, which helps us to improve the quality of our documentation. The Customer Registration Card allows you to register your product to receive all the privileges associated with being a Thermo Electron Corporation product user. After we receive your Contact Us... card, we'll send you a FREE Thermo Electron Corporation gift and complimentary application and technical reports.

Before shipping your system, Thermo Electron Corporation installed the *Xcalibur Data System*, Ver. 1.4 on your computer's hard drive.

Where to Find More Information

Look at your Xcalibur Installation CD for a complete set of hardware and software documentation.

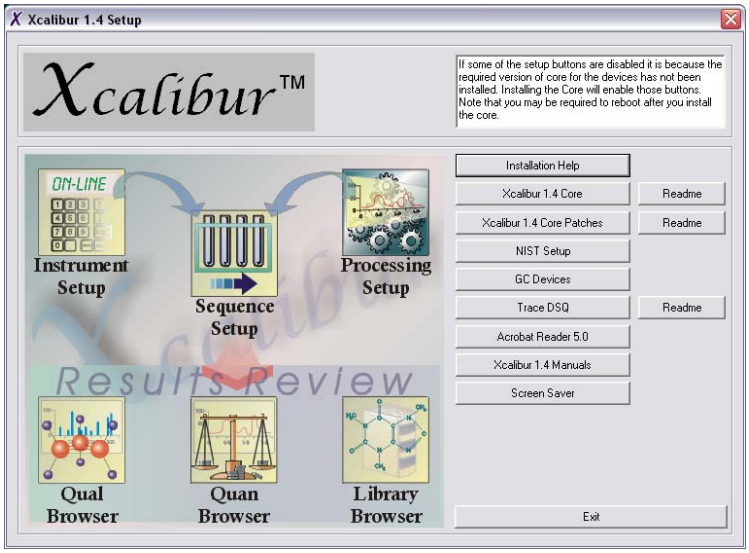


Figure i-1. Xcalibur 1.4 *TRACE DSQ* Installation CD Screen

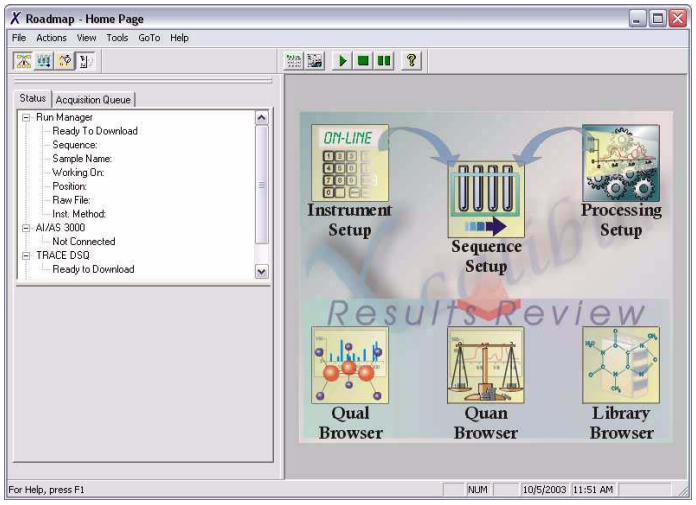


Figure i-2. Xcalibur 1.4 Home Page

Xcalibur software provides you with:

Instrument Configuration—after installation, access to this module is outside the Xcalibur home page and located as a Windows desktop icon. Use this module to configure your instruments for use with Xcalibur.

Instrument Setup—use this module to set up instrument methods and as quick access to the ***Tune*** program for calibrating your instrument for sensitivity and mass assignments.

The remaining Xcalibur modules fulfill your data acquisition and data processing needs. ***Data Acquisition***—store mass spectra peaks as they evolve and monitor the total ion chromatogram (TIC) or the intensity of a selected ion. ***Data Processing***—integrate chromatograms, view mass spectral data, compare spectra to reference libraries, quantify and qualify data, and customize and automate reports.

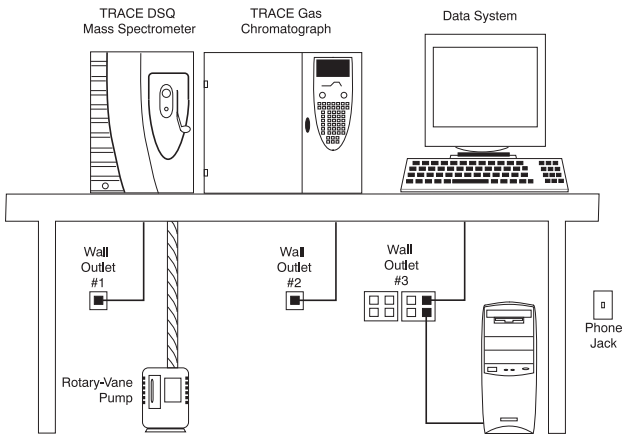
About Your TRACE DSQ or FOCUS DSQ System

The *TRACE DSQ or FOCUS DSQ* is a powerful ***quadrupole*** mass spectrometer (MS mass spectrometer), with features benefiting those in the chemical science industry—analysts, technicians, and chemists.

The *TRACE DSQ* system includes the *TRACE DSQ* mass spectrometer, the ***TRACE GC***, and the Xcalibur Ver. 1.4 or higher software on a computer.

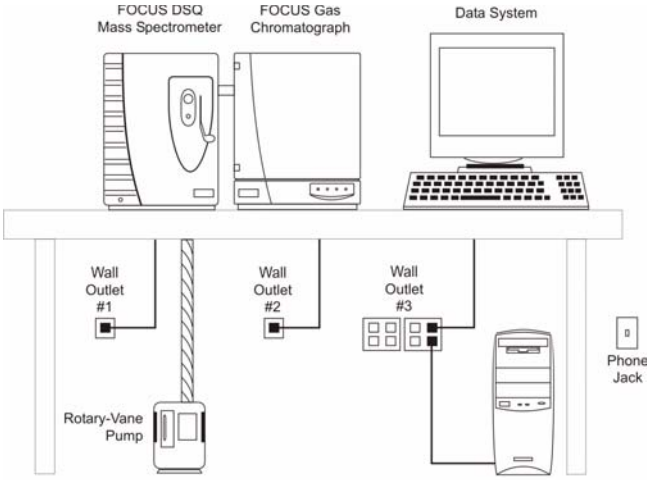
The *FOCUS DSQ* system includes the *FOCUS DSQ* mass spectrometer, the *Focus GC*, the Xcalibur Ver. 1.4 or higher software on a computer.

Table i.1 *TRACE DSQ* System Specifications



Specifications	Features	Options
Certification: Electromagnetic Compatibility (EMC) and FCC Class A	Electron Ionization (EI) with Exchangeable Ion Volumes	Autosamplers: Liquid or Headspace
Dimensions: l 68 cm x w 33 cm x h 44 cm	70 L/s Turbomolecular Pump	Chemical Ionization (CI)
Space Requirements: 2 m (6 ft)	RF-Only Curved Quadrupole Prefilter	Data System Software Options: NIST, Wiley, or Pfleger-Maurer-Weber Library, EnviroLab Forms, ToxLab 2.0
Weight: 45 kg (98 lbs)	Ion Source Temperature up to 300 °C and GC Interface Temperature up to 350 °C	Direct Probes System (DIP or DEP)
Power: 120 V ac or 230 V ac, 50/60 Hz	Mass Range 1 – 1050 u (Unit Mass Resolution)	Inlet valve
Temperature: 15-31 °C (59-88 °F)	Scan Rate up to 10,000 U/s	PPINICI (Pulsed Positive Ion, Negative Ion CI)
Humidity: 40-80%	Full Scan, SIM, and Simultaneous Full Scan/SIM	Programmable Temperature Vaporizer or Cold On-Column Injector
	Post-Acceleration ±10 kV Conversion Dynode	Turbomolecular Pumps: 250 L/s or 200/200 L/s Split-Flow
	Xcalibur Ver. 1.4 Running Under Microsoft® Windows® XP Professional with Office XP	Ion Gauge with internal vacuum protection safety

Table i.2 Focus DSQ System Specifications



Specifications	Features	Options
Certification: Electromagnetic Compatibility (EMC) and FCC Class A	Electron Ionization (EI) with exchangeable Ion Volumes	Autosamplers: Liquid or Headspace
Dimensions: l 68 cm x w 33 cm x h 44 cm	70 L/s Turbomolecular Pump	Data System Software Options: NIST, Wiley, or Pfleger-Maurer-Weber Library, EnviroLab Forms, ToxLab 2.0
Space Requirements: 2 m (6 ft)	RF-Only Curved Quadrupole Prefilter	Direct Probes System (DEP or DIP)
Weight: 45 kg (98 lbs)	Ion Source Temperature up to 300 °C and GC Interface Temperature up to 350 °C	Inlet Valve
Power: 120 V ac or 230 V ac, 50/60 Hz	Mass Range 1– 1050 u (Unit Mass Resolution)	Ion Gauge with internal vacuum protection safety
Temperature: 15-31 °C (59-88 °F)	Scan Rate up to 10,000 u/s	
Humidity: 40-80%	Full Scan, SIM, and Simultaneous Full Scan/SIM	
	Post-Acceleration ±10 kV Conversion Dynode	
	Xcalibur Ver. 1.4 Running Under Microsoft® Windows® XP Professional with Office XP	

Safety Information

- Read each instruction carefully before using the procedure.
- Follow all instructions marked on the product and in the manuals.
- Contact Tech Support to enroll in ***Instrument Training*** to prevent accidents and to get maximum use of the instrument.
- Look for safety alerts (see the following) placed in this manual and on the instrument to protect you from injury and the instrument from damage.

CAUTION **Instrument Damage**	
	This safety alert contains information to prevent instrument damage or alert against practices that could possibly void the manufacturer's warranty.
CAUTION **Personal Injury**	
	A **CAUTION** safety alert indicates a potential hazard exists that **MAY** result in minor or moderate personal injury. These safety alerts also warn against **unsafe** practices.
WARNING **Personal Injury**	
	A **WARNING** safety alert indicates a potential hazard exists that **COULD** result in death or serious personal injury.
DANGER **Personal Injury**	
	A **DANGER** safety alert indicates a potential hazard exists that **WILL** result in death or serious personal injury.

Safety Alerts Found on Instruments	
	**Read Manual** Please **READ** the manual before using to **PREVENT** an imminent or potential hazard.
	**Personal Injury** Indicates imminent or a potential personal injury hazard exists. Please use caution and consult the manual for details.
	**Burn Hazard** Indicates a hot surface. Make sure the part is room temperature before touching.
	**Explosion Hazard** Indicates risk of explosion from flammable gas or liquid. Use proper ventilation.
	**Shock Hazard** Indicates risk of electrical shock. Make sure the instrument is unplugged.
	**Material Hazard** Wear impermeable laboratory gloves.
	**Eye Hazard** Wear safety glasses.
	**Lifting Hazard** Use proper lifting technique.

Typographical Conventions

Typographical conventions describe how information is presented to our readers.

For example, some text is specially formatted to help you quickly find information contained in the document. For example,

- **Bold** is used for information required by or for the reader.
- References are emphasized in *italics*.
- ***Special terms*** are emphasized in bold and italic.

Notes provide important or helpful information about the current topic. In addition, notes can contain troubleshooting information.

NOTE: **Notes** are contained in margin boxes like this. They provide important or helpful information about the current topic.

Changes to the Manual

Your comments and suggestions help us to correct errors and improve the documentation. Please send your suggestions and comments to:

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1 Getting Connected

In This Chapter

Analyze • Detect • Measure • Control™

This chapter contains step-by step instructions for factory trained Field Engineers installing the *TRACE DSQ* or *FOCUS DSQ* system and for experienced users finding it necessary to re-setup the instrument.

NOTE: This chapter is primarily written for factory trained **Field Engineers.**

1.1 Getting the Right Tools

Use this table to determine the tools and parts you will need to take with you to the installation. However, keep in mind some items are included with the system.

Table 1-1 Required Tools

	**Tools Needed**
	• Brass back ferrules, Swagelok (PN A0101-02500)
	• Brass front ferrules, Swagelok (PN A0101-08500)
	• Brass nuts, Swagelok (PN A0101-15500)
	• Cable, PC to mass spectrometer, 15 ft Ethernet crossover (PN 76396-0052)
	• Cable, PC to GC (PN 230 980 00)
	• Cable, GC to mass spectrometer remote start (PN 76396-0050)
	• Capillary column, 5MS, 15 m, 0.25 mm i.d., 0.25 µm (PN 76317-3015)
	• Column Measuring Tool (PN 119640-0550)
	• Copper tubing, clean (PN 76381-0041 or Mueller Industries refrigeration tubing, 1/8-in. o.d., 0.030-in. wall)
	• CI ion volume (PN 119650-0230), if applicable
	• Digital voltmeter (Fluke model 77 or equivalent)
	• Dusting spray containing tetrafluoroethane (Falcon® Dust-Off®, MicroCare® MicroBlast™, or equivalent)
	• Flashlight, small hand-held
	• Flow meter (ADM 1000 or equivalent)
	• Forceps (PN 76360-0400)
	• Gas filter, helium (PN A0950-01600)
	• Gloves, clean, lint and powder free (medium PN 23827-0008, large PN 23827-0009)
	• Hex nut driver, 5.5 mm

Table 1-1 Required Tools (Continued)

	• Injector ferrule, for 0.25 mm column (PN 290 134 88)
	• Ion volume tool (PN 119270-0001)
	• I/R tool and guide bar, if applicable
	• Leak detector (GL Sciences, Inc. Model LD-228 or equivalent)
	• Magnifying glass
	• Methanol or other suitable solvent
	• Potentiometer adjustment tool
	• Power cords with appropriate plugs
	• Preinstallation Guide (PN 120156-0001)
	• Scoring wafer (or sapphire scribe) to cut capillary column
	• Screwdriver, flat blade
	• Screwdriver, phillips #1
	• Screwdriver, phillips #2
	• Syringe, 10 µL, 70 mm needle (PN 365 001 03)
	• Tape measure
	• Teflon® thread tape
	• Tissue, lint free
	• Tubing cutter
	• Test mix, octafluoronaphthalene and benzophenone (PN 120150-TEST)
	• Transfer line ferrule, 0.4 mm i.d. (PN A0101-18100)
	• Wrench set, (Allen) 1.5, 2, 2.5, 3, 4, 5 mm (PN 3812-0100)
	• Wrench set, (Open ended) 5–17 mm
	• Wrench, (Adjustable) 12-in.
	• Wrench, (Allen) 1/16-in.
	• Wrench set, (Open ended) 1/4-in., 5/16-in., 7/16-in. (2), 9/16-in., 1/2-in.

1.2 Verifying Site Preparation

	**Tools Needed** • Digital voltmeter (Fluke model 77 or equivalent) • Preinstallation checklist from customer • Preinstallation Guide (PN 120156-0001) • Tape measure
	**Frequency** Day 1

Review the customer's Preinstallation Checklist, keeping these items in mind:

- The customer is responsible for providing a completed *Preinstallation Checklist* prior to the installation. If the checklist is lost or unreadable, you can make a copy of *A.2 Installation Checklist, pp. 131* and determine if the installation can still be performed.
- All preinstallation requirements are listed in the *TRACE DSQ Preinstallation Guide (PN 120156-0001)*, so you only need to double-check that the proper workbench, power outlets, power cords, temperature, and gases are available.

1.3 Unpacking the Instruments

	**Tools Needed** • Original purchase order • Packing slip • Installation report (supplied by service organization)
	**Frequency** Day 1

1 Unpack the instruments to confirm that each item the customer ordered arrived without incident.

WARNING **Personal Injury**	
	**Lifting Hazard** Use proper lifting techniques, as several items are heavy.

2 Check for damage.

a. Check carefully for obvious damage—tip indicators displaying a tipped condition or evidence of rough handling.

b. If external damage is apparent, note this fact on the installation report, and describe briefly the extent of the damage. Have the customer sign or initial next to your comments.

3 Confirm that the shipment is complete.

a. Open and carefully remove all items from all shipping containers. The packing list is located in the installation packet.

b. Verify that each item listed on the customer's purchase order and packing list is included with the shipment. If any items are missing, make a note on the installation report and immediately contact the factory Quality Representative at 512-251-1400.

4 Set up the instruments.

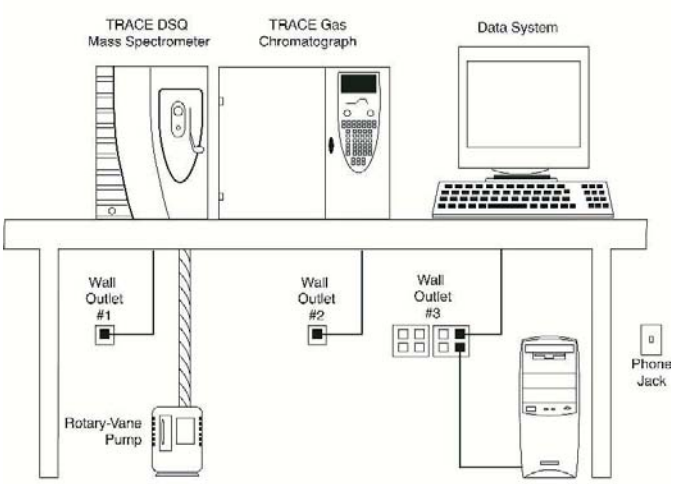


Figure 1-1 *TRACE DSQ* System Layout (Front)

a. Place the instruments from left to right, in this order: Mass Spectrometer, GC, and the computer monitor, with the mid-tower CPU beside the monitor or under the bench.

b. Place the rotary-vane pump on the floor. If placed on the bench, excess vibration could affect instrument performance.

5 Inspect and store the shipping containers.

a. Carefully inspect ALL boxes and packing materials for missed items.

b. Put packing materials back into shipping crates, and store the empty boxes.

NOTE: Install optional accessories, for example, the autosamplers *after* the installation and qualification tests are complete.

1.4 Connecting the Gas Lines

Tools Needed

- Brass back ferrules, Swagelok (PN A0101-02500)
- Brass front ferrules, Swagelok (PN A0101-08500)
- Brass nuts, Swagelok (PN A0101-15500)
- Copper tubing, clean (PN 76381-0041 or Mueller Industries refrigeration tubing, 1/8-in. o.d., 0.030-in. wall)
- Gas filter, helium (PN A0950-01600)
- Leak detector (GL Sciences, Inc. Model LD-228 or equivalent)
- Teflon® thread tape
- Tubing cutter
- Wrench, (Adjustable) 12-in.
- Wrench, (Open ended) 7/16-in. (2)
- Wrench, (Open ended) 1/2-in.
- Wrench, (Open ended) 9/16-in.

Frequency

Day 1

Assembly Tips:

- To insure safe, leak-tight connections, follow these instructions: *B.1 Assembling Swagelok Fittings, pp. 134* before assembling the ***swagelok fittings***.
- Leave enough slack in the tubing so the GC and mass spectrometer may be moved at least 40 cm (16-in.) away from each other. This ensures that column connections and system maintenance are easily performed.
- Forming ***Shock Loops*** with the tubing helps to dampen vibrations that can loosen the fittings.

1 Connect the GC Carrier Gas.

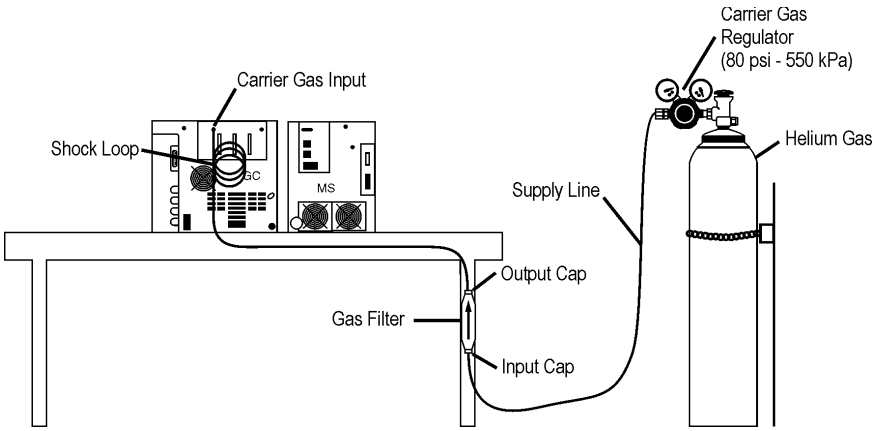


Figure 1-2 GC Carrier Gas Connections (Back)

2 Install the **Carrier Gas Regulator** and **Gas Filter**.

a. Connect the **Carrier Gas Regulator**.

b. Set the **Gas Filter** as close as possible to the GC. It should be easy to access since it must be changed regularly. Do not put it behind the GC where hot air from the exhaust vents will hit the gas filter. Mount it to the side of the GC or MS or on top.

CAUTION **Instrument Damage**	
	Do not loosen or remove caps from the Gas Filter until PURGED lines are connected. Loosening or removing caps early contaminates the filter.

3 Prepare the **Supply Line**.

a. Run a **Supply Line** from the **Carrier Gas Regulator** to the **Gas Filter** but do not remove the **Caps** on the **Gas Filter**.

b. Run a **Supply Line** from the **Gas Filter** to the **Carrier Gas Input** on the GC. Do not connect it to the **Gas Filter** or GC yet.

c. Include a **Shock Loop** to isolate vibration and allow enough tubing to move the GC at least 40 cm (16-in.)

4 Purge the **Supply Line**.

a. Turn on the **Carrier Gas** supply and set the **Regulator** to 5 psi (35 kPa).

b. Purge the line for 2-min to remove air and debris.

c. Without turning off the **Carrier Gas**, quickly remove the **Inlet Cap** from the **Gas Filter** and attach the **Supply Line**.

d. Remove the **Outlet Cap** from the **Gas Filter** and attach the **Supply Line**.

e. Before attaching the **Supply Line** to the GC, purge it for 10-min.

f. Attach the **Supply Line** to the **Carrier Gas Input** on the GC. If the GC is equipped with an additional inlet, you can use a Swagelok tee union to connect the **Supply Line** to the second **Carrier Gas Input**.

5 Set the **Carrier Gas Regulator** to 80 psi (550 kPa).

6 **(Upgrade Option)** Connect the **CI Reagent Gas**, otherwise proceed to *Step 10 Leak check all the gas lines., pp. 30*

If the system is equipped with the **chemical ionization** upgrade option, you must connect CI Reagent Gas. **Methane** is the most common CI Reagent Gas.

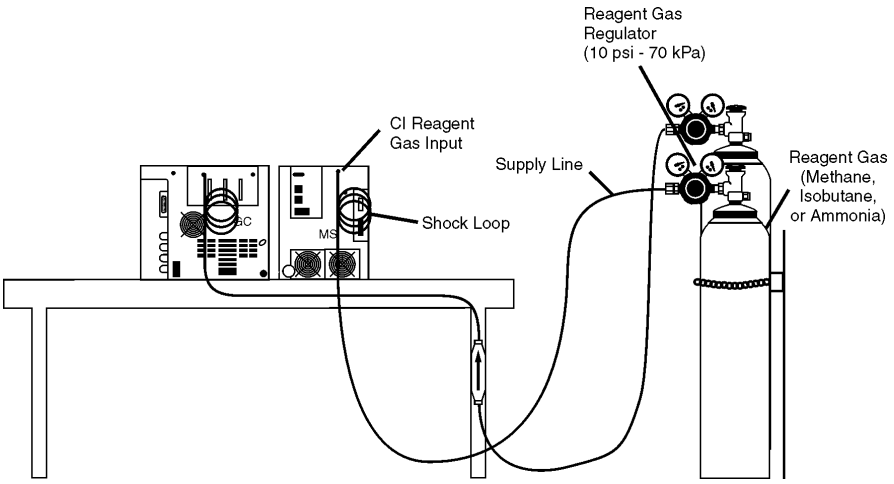


Figure 1-3 CI Reagent Gas Connections (Back)

7 Install the **Reagent Gas Regulator** to the **Reagent Gas Cylinder**.

WARNING Personal Injury	
	**Explosive or Corrosive Gas Hazard** CI reagent gases are often flammable or corrosive. You must vent the gas to a fume hood or other suitable exhaust. The CI reagent gas supply line must be leak tight.

8 Prepare the **Reagent Gas Supply Line**.

a. Run a **Supply Line** from the **Reagent Gas Regulator** to the **CI Reagent Gas Input** located on the Mass Spectrometer.

b. Do not connect it yet.

c. Include a **Shock Loop** to isolate vibration and allow enough tubing to move the Mass Spectrometer at least 40 cm (16-in.)

9 Purge the **Supply Line**.

a. Route the **Supply Line** to a vent hood or other suitable exhaust.

b. Turn on the **Reagent Gas** supply and set the **Regulator** to 30 psi (200 kPa).

c. Purge the line for about 15 seconds to remove air and debris.

d. Set the **Reagent Gas Regulator** to 5-10 psi (35-70 kPa). Lower supply pressures produce more stable CI reagent gas flows.

e. Turn off the **Reagent Gas** supply.

f. Attach the **Supply Line** to the **CI Reagent Gas Input** on the Mass Spectrometer.

g. Turn on the **Reagent Gas** supply.

CAUTION Instrument Damage	
	Do not exceed 35 psi (240 kPa) or damage to the CI Reagent Gas Flow Module results.

10 Leak check all the gas lines.

a. Use an electronic hand-held leak detector (sensitivity: helium 0.01 mL/min) to check all gas fittings for leaks.

CAUTION **Instrument Damage**	
	Do not use liquid soap leak detectors (such as Snoop) to check for leaks. Liquid soap leak detectors may contaminate your system.

b. Leak check the **Carrier Gas Supply Line**.

c. Leak check the **Reagent Gas Supply Line**.

1.5 Installing the Data System

The data system is the computer and Xcalibur software.

	**Tools Needed** • Cable, PC to Mass Spectrometer, 15 ft Ethernet crossover (PN 76396-0052) • Cable, serial, PC to GC (PN 230 980 00)
	**Frequency** Day 1

1 Setup the **Data System** as demonstrated in Figure 1-4.

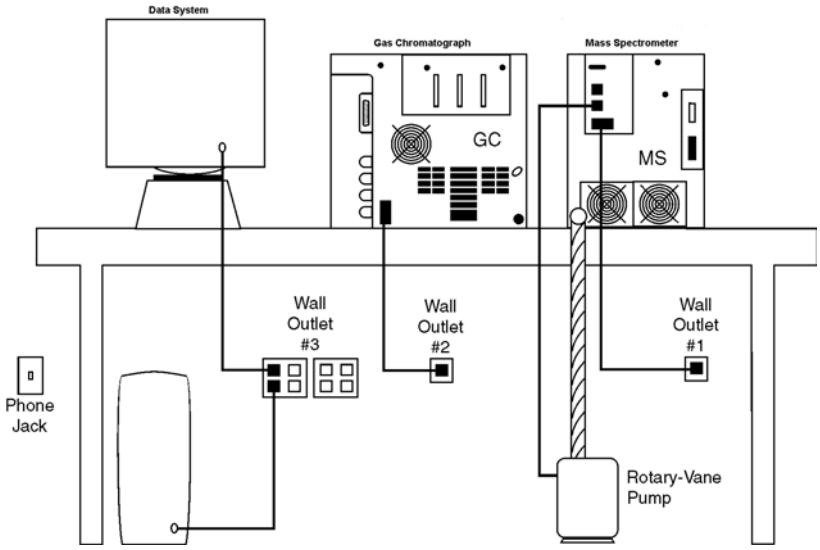


Figure 1-4 Data System Power Connections (Back View)

a. Plug the power cords for the computer, monitor, and printer (upgrade option) into **Wall Outlet #3**.

b. Set up and connect the keyboard, mouse, monitor cable, and printer cable if applicable.

2 Connect the **GC serial cable** and the mass spectrometer's **Ethernet cables**.

a. Connect the **GC Serial Cable** to the computer's **COM1** port.

b. Connect the **Ethernet Cable**, which is supplied with the Mass Spectrometer to the **computer's Ethernet port**. The computer has two Ethernet ports. The Ethernet port on a card is for the instrument, whereas, the Ethernet port built into the motherboard may be used for a local area network.

NOTE: Be sure you use the supplied shielded, crossover cable to connect to the instrument. If you need a longer cable, you can connect a standard **Category 5 Ethernet cable** between the computer and the supplied cable.

3 Turn on the computer, monitor, and printer.

a. Set the time and date.

b. Set up passwords, if desired.

c. Test the printer and install drivers[1] as necessary.

Computers purchased from the Thermo Electron Corporation factory have been fully setup and tested with the **Xcalibur 1.4** data system software. However, if you experience the rare situation where you need to reinstall software on a new computer or new hard drive, follow these instructions: *B.6 Reinstalling the Data System, pp. 143*.

1.Tune reports will not display if a printer driver is not installed.

1.6 Installing the Gas Chromatograph

	**Tools Needed** • Capillary column, 5MS, 15 m, 0.25 mm i.d., 0.25 µm (PN 76317-3015) • Gloves, clean, lint and powder free • Injector ferrule, for 0.25 mm column (PN 290 134 88) • Leak detector (GL Sciences, Inc., model LD-228, or equivalent) • Magnifying glass • Methanol or other suitable solvent • Scoring wafer (or sapphire scribe) to cut capillary column • Tissue, lint free • Wrench, (Open ended) 6 mm
	**Frequency** Day 1

1 Check the **GC** configuration against the sales order.

2 Connect the **Transfer Line Cable** to the left of the **GC**.

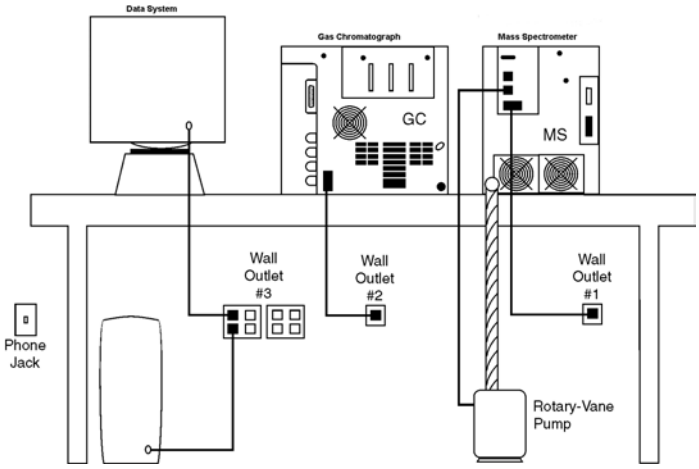


Figure 1-5 Power Connections (Back View)

3 Plug the power cord into the back of the GC and then into **Wall Outlet #2**.

4 Connect the **Column** to the **Injector**.

The test column is conditioned at the factory and requires no additional conditioning.

Focus GC users must set up instrument parameters using the virtual **Keypad button** located on the **Xcalibur Home Page**.

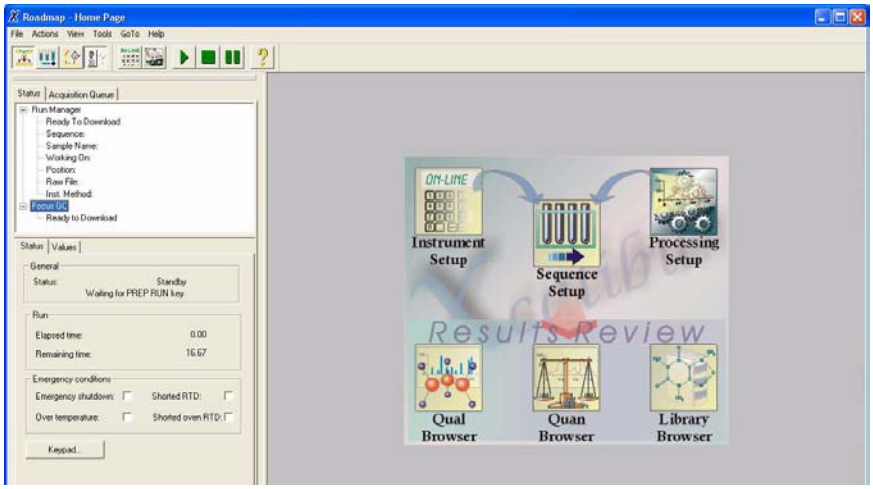
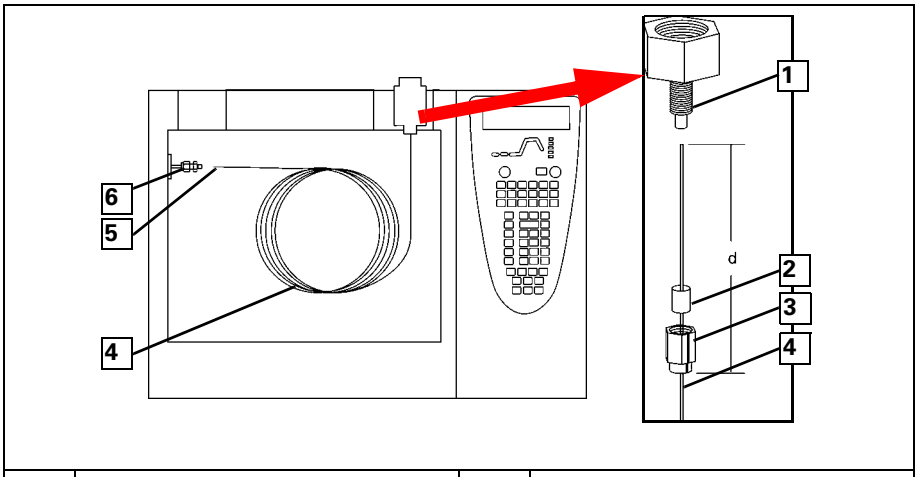


Figure 1-6 *Focus GC* Instrument Status View

Refer to the ***Focus GC*** **User's Manual** for instructions on using the keypad.

The arrow in Figure 1-7, points to an exploded view of the injector.



1	Injector	**4**	Column
2	Injector Ferrule	**5**	Column Outlet
3	Injector Nut	**6**	Transfer Line

Figure 1-7 GC Injector (Front View)

NOTE: Wear clean, lint and powder free gloves when you handle the Column and Injector Ferrule.

a. Unwind the **Column (4)** about a half-turn.

b. Wipe about 100 mm (4-in.) of the **Column (4)** with a tissue soaked in methanol.

c. Insert the **Column (4)** through the **Injector Nut (3)** and **Ferrule (2)** open-end-up.

d. Wipe the **Column (4)** again with a tissue soaked in methanol.

e. Score and then break the **Column (4)** about 2.5 cm (1-in.) from the end with a **Scoring Wafer**. With the magnifying glass, check for an even, flat cut. Repeat if necessary.

f. Insert the **Column (4)** into the **Injector (1)** so that the end of the **Column (4)** is the proper distance from the back of the **Injector Nut (3)**. Proper distances are as follows: splitless = 64 mm, split = 40 mm, PTV = 30 mm.

NOTE: Sliding a Septum on the Column before the Injector Nut will help you measure the proper distance between the Nut and the end of the Column. If you want to remove it later, cut the Septum from the center to the edge.

g. Finger-tighten the **Injector Nut (3)** and then give it an additional 1/4-turn with the wrench.

h. Score and then break the **Column Outlet (5)** about 2.5 cm (1-in.) from the end with a **Scoring Wafer**.

i. Turn on the GC.

5 Setup the **GC**.

a. Set the **Oven, Injector, and Transfer Line** temperatures to 30 °C.

b. Set the **Injector flow** to 1.0 mL/min.

c. Turn the **Vacuum Compensation Off** (under the Right, or Left Carrier menu).

d. Dip the **Column Outlet (5)** in a small vial of methanol. Bubbles indicate there is flow through the **Column (4)**.

e. Allow the **Column (4)** to purge for at least 10-min.

6 Perform a **Column Characterization**.

a. Raise the **Oven** and **Injector temperatures** to 50 °C and allow them to stabilize.

b. Run **Column Eval** key to characterize the column. This takes several minutes.

c. Expect a **K-factor** of about 0.7 – 0.9 for a 15 m, 0.25 mm i.d. column. If the column does not report a K-factor within this range or within 0.1 units of the previous stored value, check for a leak or broken column using the leak detector. The K-factor is a measured resistance for the **Column**. A K-factor that is too low may indicate a leak in the system, while a K-factor that is too high may indicate a blockage.

CAUTION Instrument Damage	
	Do not raise the oven temperature until you are sure the system is leak free. At temperatures above 100 °C, the column will be destroyed if exposed to oxygen.

d. Raise the **Oven temperature** to 150 °C and allow it to stabilize.

7 Perform a column **Leak Check**.

a. Run the automated **Leak Check** on the GC.

b. If the report indicates a leak, look for and fix leaks at all the fittings in the GC using the leak detector.

c. Repeat the **Column Evaluation** and **Leak Check** until no leaks are indicated.

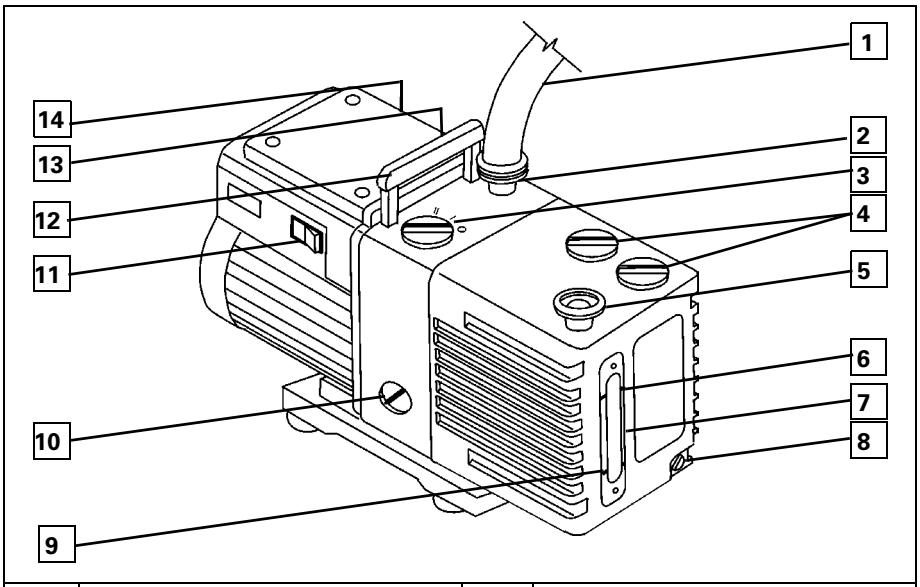
d. Raise the **Injector temperature** to 220 °C.

1.7 Installing the Mass Spectrometer

	**Tools Needed** • Cable, GC to Mass Spectrometer remote start (PN 76396-0050) • Column Measuring Tool (PN 119640-0550) • Gloves, clean, lint and powder free • Leak detector, hand-held electronic (GL Sciences Inc., model LD-228, or equivalent) • Magnifying glass • Methanol or other suitable solvent • Potentiometer adjustment tool • Scoring wafer (or sapphire scribe) to cut capillary column • Tissue, lint free • Transfer line ferrule, 0.4 mm i.d. (PN A0101-18100) • Wrench, Open ended 5/16-in. • Wrench, 2, Open ended 7/16-in.
	**Frequency** Day 1

1 Install the **Rotary-Vane Pump**.

a. Set the **Rotary-Vane Pump** on the floor behind the system. The pump should not be placed on the workbench as it produces excessive vibration that may affect the performance of the system.



1	Foreline Vacuum Hose	**8**	Drain Plug
2	Inlet Port	**9**	MIN (minimum level mark)
3	Gas-Ballast Control	**10**	Mode Selector
4	Oil Filler Plugs	**11**	On/Off Switch
5	Outlet Port	**12**	Handle
6	MAX (maximum level mark)	**13**	Voltage Indicator
7	Oil-Level Sight-glass	**14**	Electrical Inlet Connector

Figure 1-8 Rotary-Vane Pump

b. Check the **Voltage Indicator (13)** to verify the Rotary-Vane Pump is configured for the same voltage as the Mass Spectrometer.

c. Remove one of the **Oil Filler Plugs (4)**.

d. Add oil to the **Oil Filler Plug (4)** opening half way between the **MIN (9)** and **MAX (6)** level marks.

e. If the oil level goes above the **MAX (6)** level mark, remove the **Drain Plug (8)** and drain the excess oil from the pump.

f. Replace the **Oil Filler Plug (4)**.

g. Connect the **Foreline Vacuum Hose (1)**.

h. Connect the **Outlet Port (5)** to a suitable exhaust.

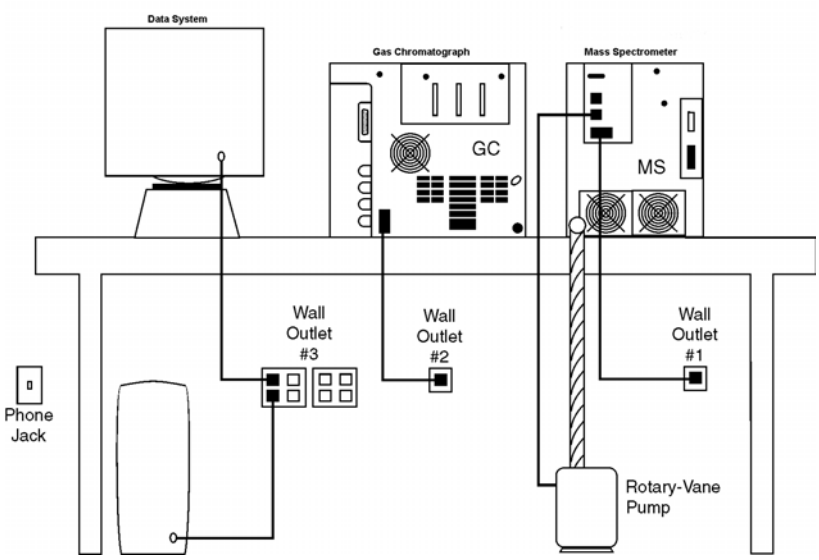


Figure 1-9 Rotary-Vane Pump Power Connection (Back View)

i. Plug the power cord into the **Mech Pump plug** located on the back of the **Mass Spectrometer**.

2 **[Upgrade Option]** Repeat this procedure for a **second Rotary-Vane Pump**, which is included with the Sample Probe upgrade option.

a. Plug the **second pump** into the **Accessory plug** on the back of the **Mass Spectrometer**.

b. Make sure the **Inlet Valve Vacuum Hose** exits the **Mass Spectrometer** just above the **Foreline Vacuum Hose**.

3 Connect the **transfer line**, **Ethernet**, and **remote start** cables.

a. Confirm the Transfer Line Cable is plugged into the left side of the GC.

b. Carefully push the GC next to the Mass Spectrometer, making sure the **Transfer Line** extends into the GC.

c. Connect the **Ethernet cable** leading from the computer to the back of the Mass Spectrometer.

d. Connect the **Remote Start Cable** to the **GENERIC H/S** port located on the GC to the **GC START** port located on the Mass Spectrometer.

4 Inspect the Mass Spectrometer.

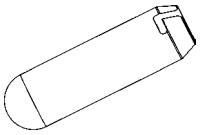
a. Remove the covers (front, top, and left side) from the Mass Spectrometer. Detailed instructions for removing covers are in the *TRACE DSQ Hardware Manual (PN 120156-0002).*

b. Remove the shipping foam that secures the **Vacuum Manifold Cover**. Store the foam with the Mass Spectrometer accessories in case the instrument needs to be moved in the future.

c. Check the Mass Spectrometer configuration against the sales order.

d. Look for anything that might have come loose during shipping.

e. Verify that the **Ion Volume** is fully inserted. You can use the **Ion Volume Tool** to push on the Ion Volume. The Ion Volume is held loosely in the Ion Source at room temperature so that it does not seize when the source heats to 300 °C.



Ion Volume

5 Connect the **GC Column** to the **MS Transfer Line**.

a. Lower the **oven temperature** to 30 °C and allow it to cool before continuing.

CAUTION Personal Injury	
	**Burn Hazard** The oven and transfer line may be hot. Allow them to cool to room temperature before touching them. Do not touch the hot injector.

b. Unwind about one-turn of the **Column** from the end of the **Column Outlet**.

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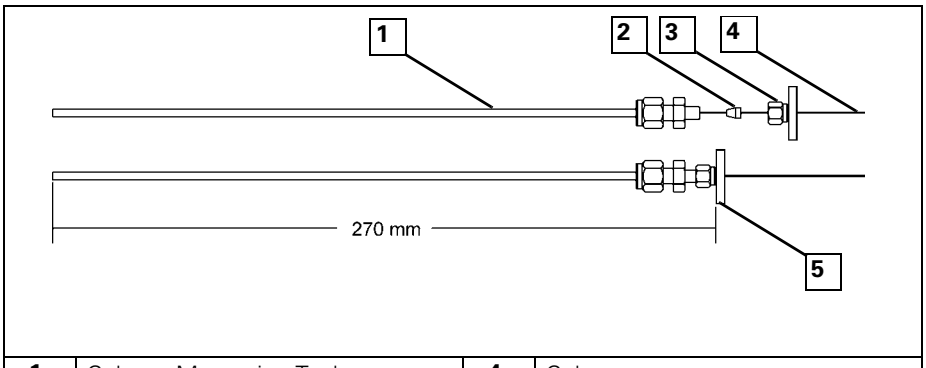


1	Column Measuring Tool	**4**	Column
2	Transfer Line Ferrule	**5**	Septum
3	Transfer Line Nut		

Figure 1-10 Column Measuring Tool and Transfer Line

c. Wipe about 300 mm (12-in.) of the **Column (4)** with a tissue soaked in methanol.

d. Insert the **Column (4)** through the **Septum**[1] **(5)**, **Transfer Line Nut (3)**, and **Ferrule (2)**. Wipe the **Column (4)** again with a tissue soaked in methanol.

e. Screw the **Transfer Line Nut (3)** onto the **Column Measuring Tool (1)**.

f. Push the **Column (4)** passed the end of the **Column Measuring Tool (1)**, then score and break the end of the **Column (4)** with a **Scoring Wafer**. Using a magnifying glass, check for an even, flat cut. Repeat if necessary.

g. Pull the **Column (4)** back so that it is flush with the end of the **Column Measuring Tool (1)**.

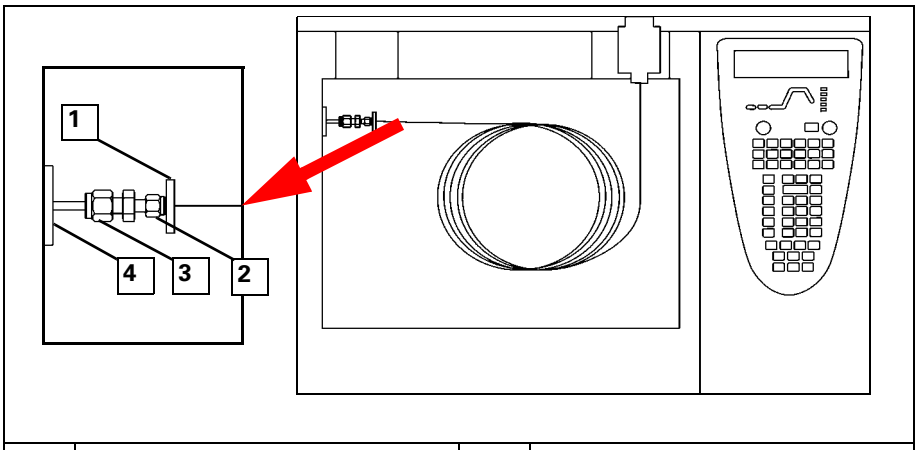
h. Tighten the **Transfer Line Nut (3)**.

i. Slide the **Septum (5)** up to the back of the **Transfer Line Nut (3)**.

j. Remove the **Column (4)**, **Transfer Line Nut (3)** and **Ferrule (2)** from the **Column Measuring Tool (1)**.

NOTE: Wear clean, lint and powder free gloves when you handle the Column and Transfer Line Ferrule.

1.Sliding a Septum on the Column before the Transfer Line Nut will help you measure the proper distance between the nut and the end of the column. If you want to remove it later, cut the Septum from the center to the edge.



1	Septum	3	Transfer Line Union
2	Transfer Line Nut	4	Transfer Line

Figure 1-11 Transfer Line (Front)

k. Insert the **Column** into the **Transfer Line (4)**. Be careful not to move the **Septum (1)**. For proper operation, the **Column** must extend approximately 1 mm passed the end of the **Transfer Line**.

l. Tighten the **Transfer Line Nut (2)** and the **Transfer Line Union (3)**.

6 Condition the **Transfer Line Ferrule**.

Graphite/Vespel ferrules like the **Transfer Line Ferrule** require conditioning to insure a leak tight seal.

a. Raise the oven temperature to 300 °C.

b. Wait for 10-min.

c. Lower the oven temperature to 30 °C and allow it to cool before continuing.

CAUTION
Personal Injury

Burn Hazard
The oven may be hot. Allow it to cool to room temperature before opening it. The injector will still be hot, so don't touch it!

d. Re-tighten the **Transfer Line Nut (2)** and the **Transfer Line Union (3)**.

7 Setup the **GC**.

a. Make sure the **Column** does not have any sharp bends and that it does not touch any metal objects or walls inside the **Oven**.

b. Raise the **Oven temperature** to 40 °C.

c. Turn the **Vacuum Compensation On** (under the Right, or Left Carrier menu).

8 Replace the covers to the MS, in this order: left, top, and front.

9 Plug the power cord for the Mass Spectrometer into **Wall Outlet #1**.

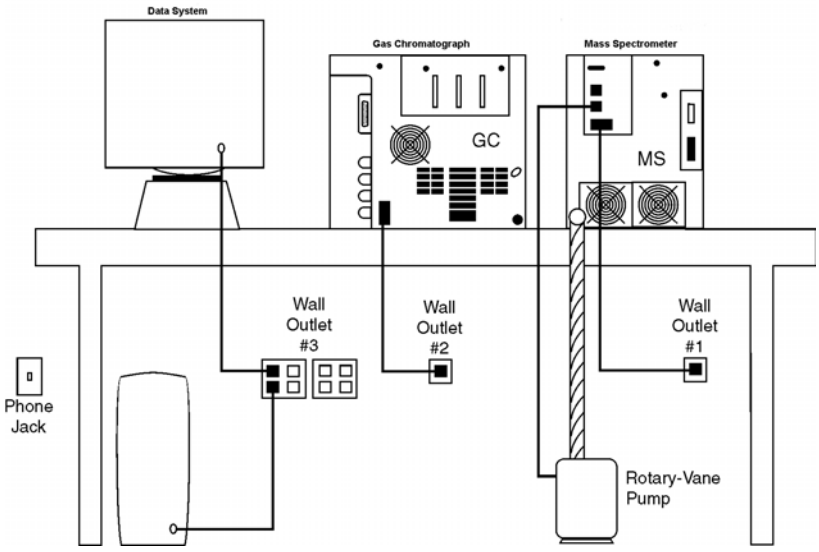


Figure 1-12 Mass Spectrometer Power Connection (Back)

10 Double-check all vacuum, gas, and electrical connections.

1.8 Starting Up the System

	**Tools Needed** None
	**Frequency** Day 1

1 Turn the **Mass Spectrometer** on by setting the **Main Circuit Breaker** to **ON (I)** to:

- Turn on the Rotary-Vane Pump
- Allow the Fore pressure to reach the proper operating pressure
- Turn on the Turbomolecular Pump

CAUTION Instrument Damage	
	Damage occurs if you turn on the mass spectrometer without column flow. This forces air to be drawn through the column, damaging it. This large air leak into the mass spectrometer will also cause the ion source to require cleaning.

2 Set the **Transfer Line** to 250 °C.

3 Start **Xcalibur** to establish communication and initialize each instrument configured for use.

a. Wait about 60-seconds for the Mass Spectrometer to initialize.

b. Then, look for Xcalibur to display a screen "**There is not sufficient vacuum**."

c. Choose **Ignore** so you can continue to monitor the instrument status.

d. If you see any other error messages, check that all instruments are connected and turned on. Also, consult the Troubleshooting section of the *TRACE DSQ or FOCUS DSQ Hardware Manual (PN 120156-0002)*.

4 Start **Tune**.

Click the **Instrument Tune desktop icon** 

from the Windows desktop.

1.9 Confirming Readbacks

	**Tools Needed** None
	**Frequency** Day 1

Once the instruments have been initialized, check the Mass Spectrometer **Instrument Status Tabs** to confirm the **vacuum** and **heater readbacks** are OK before running diagnostics. Otherwise, improper vacuum will cause damage to the filament during the Ionization/Lens Test.

Information contained in the Instrument status tabs let you watch the Mass Spectrometer heat up and pump down.

1 In the **Tune** window, select the **Vacuum** tab to validate the **Fore Pressure**, **Turbo Pump** status, and **Ion Gauge** status.

a. Verify that the **Fore Pressure** is less than 300 mTorr.

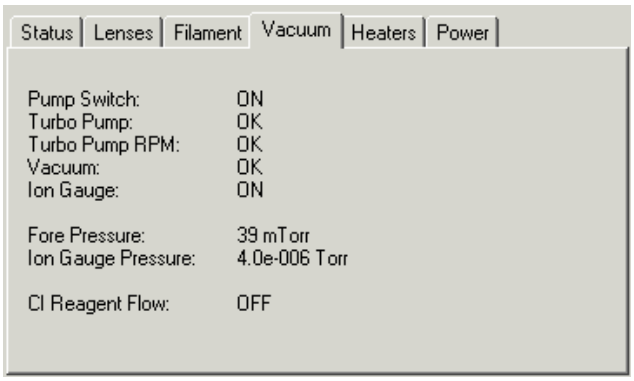


Figure 1-13 TRACE DSQ Vacuum Status

NOTE: If the fore pressure is not less than 300 mTorr within 5-min, there is a large leak which must be fixed immediately.

It should reach this level within 5-min after turning on the system. If it doesn't, there is a large leak which must be fixed immediately. Consult the *TRACE DSQ or FOCUS DSQ Hardware Manual (PN 120156-0002)* for troubleshooting a large leak.

b. Make sure the turbomolecular **Pump Switch** is **ON**. Within 5-min after turning the turbomolecular pump, the readback for the **Turbo Pump** should be OK indicating it has reached operating speed.

c. Wait until the **Ion Gauge Pressure** is less than 1 x 10^{-4} Torr. If the system is not equipped with an ion gauge, wait at least 15-min.

2 Select the **Heaters** tab to confirm the **Ion Source Temp** to 200 °C.

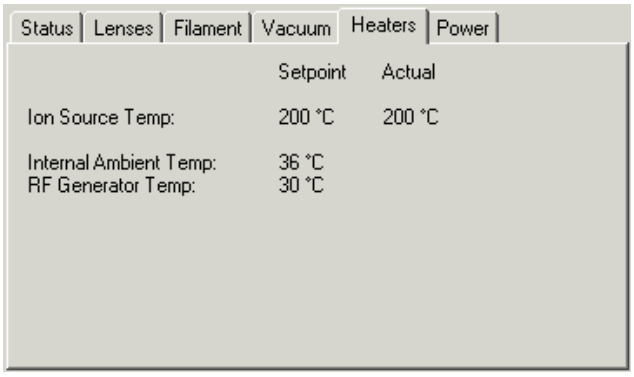


Figure 1-14 Mass Spectrometer Heaters Readbacks

Use this tab to view the temperature. To set the temperature go to **Tune | Instrument | Set Temperatures** to display the **Temperature Settings screen**. Then Set the Ion to 200 and select OK to return to the Tune window.

1.10 Running Diagnostics

	**Tools Needed** None
	**Frequency** Day 1

After confirming that the heaters and vacuum readbacks are OK, run Xcalibur diagnostics to test other internal components for functionality.

CAUTION
Instrument Damage

Make sure the vacuum readbacks are OK before running diagnostics. Otherwise, improper vacuum will cause damage to the filament during the Ionization/Lens Test.

1 Open the Tune window Diagnostic screen.

Select **Tune | Diagnostics | Run Tests** to display the **Diagnostics screen**.

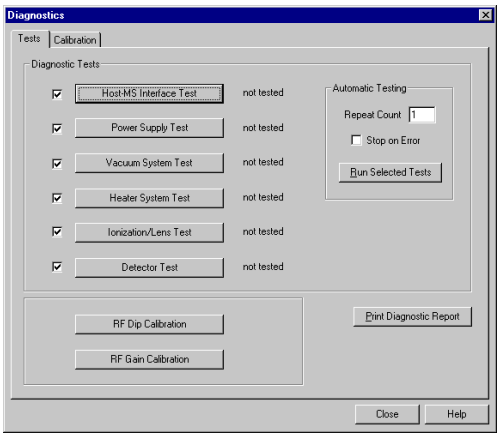


Figure 1-15 Tune Window Diagnostics Screen

2 Run each test listed in the **Diagnostic Tests** group box individually so that you can closely monitor each test's readbacks and note any failures.

If a diagnostic fails, then view the help topic for that diagnostic. However, don't run the **RF Dip** and **RF Gain Calibrations** tests until the next procedure.

1.11 Checking the RF Dip Calibration

	Tools Needed None
	Frequency Day 1

NOTE: Run this test only after the heaters and vacuum have stabilized.

After you have finished running the Xcalibur diagnostics from the Diagnostics Tests group box and they have all passed, then it's time to check the RF Dip Calibration. However, run this test only after the heaters and vacuum have stabilized.

1 Open the Tune window **Diagnostic screen**.

Select **Tune | Diagnostics | Run Tests** to display the Diagnostics screen.

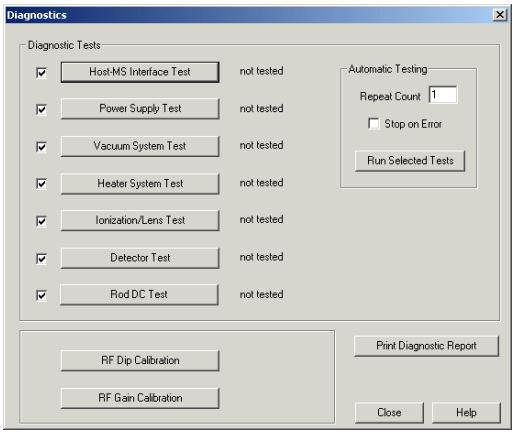


Figure 1-16 Tune Diagnostic Screen

2 Select the **RF Dip Calibration** button to display the RF Dip Calibration screen.

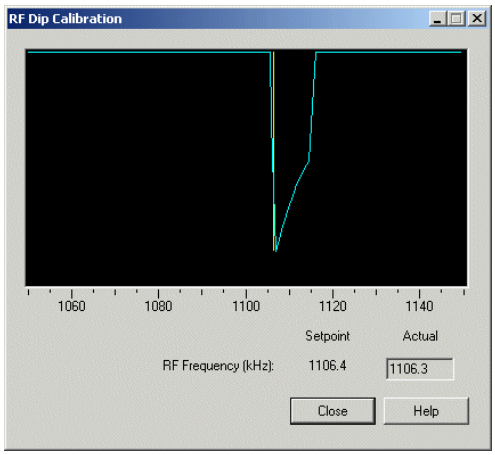


Figure 1-17 RF Dip Calibration Screen

a. Allow the display to stabilize for approximately 15 seconds.

b. Verify that the **Setpoint** and **Actual** are within 0.2 kHz.

c. If they are not, follow these instructions: *B.5 Adjusting the RF Dip Calibration, pp. 142*.

NOTE: The RF Dip Calibration looks different than the Polaris *Q* RF Dip calibration. This is normal as the TRACE DSQ use a more comprehensive algorithm. The RF frequency is set correctly if the Setpoint and Actual are within 0.2 kHz.

3 Press **Close** to return to the **Diagnostics screen**.

NOTE: If the **Rod DC Test Fails** after the RF Dip Calibration and RF Gain Calibration pass, follow the instructions in *B.3 Adjusting the RF Null, pp. 138*.

1.12 Checking the RF Gain Calibration

	**Tools Needed** • Potentiometer adjustment tool • Wrench, (Allen) 3 mm
	**Frequency** Day 1

After you see a successful RF Dip Calibration, check the RF Gain Calibration.

1 Open the Tune window **Diagnostic screen**.

Select **Tune | Diagnostics | Run Tests** to display the Diagnostics screen.

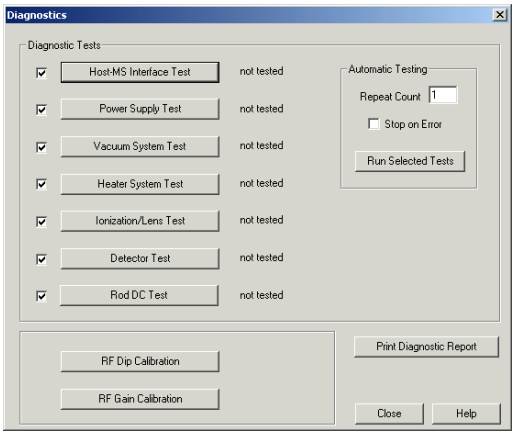
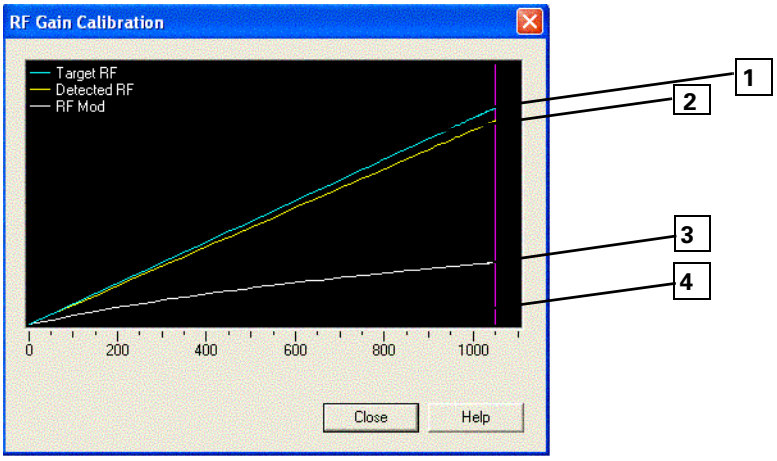


Figure 1-18 Tune Diagnostic Screen

2 Select the **RF Gain Calibration button** to display the **RF Gain Calibration screen** and run the **RF Gain Calibration test**.



1	Target RF (blue line)	**3**	RF Modulation (white line)
2	Detected RF (yellow line)	**4**	*m/z* 1050 (red/purple vertical line)

Figure 1-19 RF Gain Calibration Screen

This screen plots these values versus *m/z*:

- **Target RF** (**1**) ramp is linear and corresponds to the ramp used to scan the quadrupole to produce a mass spectrum.
- **Detected RF** (**2**) measures the actual RF applied to the quadrupole. It must be a linear ramp all the way to the *m/z* 1050 line (**4**).
- **RF Modulation** (**3**) measures the power output of the RF generator. It is usually slightly curved.

a. Allow the display to stabilize for approximately 15 seconds.

b. Verify that the Detected RF is linear to the *m/z* 1050.

If the Detected RF is not linear to the *m/z* 1050, the mass calibration may be incorrect. At this point, check the mass calibration by viewing the calibration gas spectrum (detailed instructions are in *Step 4 Check the calibration gas spectrum., pp. 61*.)

If the masses are not within 2 *m/z* of the theoretical *m/z*, wait until the automatic tune calibration is finished to pass the RF Gain Calibration Diagnostic.

If the Detected RF shows noise or spikes, shut down the instrument and check for dust or fibers on the quadrupole and prefilter, or check for loose connections between the RF Coil, Low Pass Filter, and Quadrupoles.

3 Select the **Close button** to return to the **Diagnostics screen**.

4 Press **Close** to return to the Tune window.

5 Proceed to step *1.13 Checking the Air/Water Spectrum, pp. 56*.

1.13 Checking the Air/Water Spectrum

	**Tools Needed** None
	**Frequency** Day 1

After successfully completing the RF Gain Calibration test, it's a good time to check the air/water spectrum to look for leaks.

1 In the **Tune window**, select the **Vacuum** tab from the Status display.

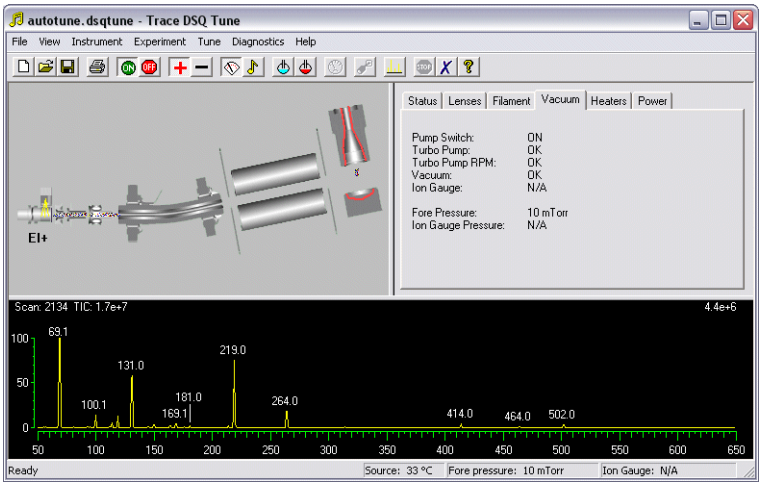


Figure 1-20 Tune Window, Vacuum Tab

a. Verify that the **Fore Pressure** is less than 50 mTorr. For some instruments that have been exposed to high humidity while vented for an extended period of time, this may require several hours. This is common for an instrument shipped with the 200/200 L/s pumping option, because the large surface of the turbomolecular pump requires time to outgas.

b. If the system has an ion gauge, verify that the **Ion Gauge Pressure** is less than 7×10^{-5} Torr

c. If the pressure is too high, there is a leak that must be fixed immediately. Refer to the *TRACE DSQ or FOCUS DSQ Hardware Manual (PN 120156-0002)* for troubleshooting instructions.

2 Make sure the **calibration gas** has been off for at least 5-min. Because, the calibration gas flow module introduces air into the instrument.

3 Check the **Air/Water Spectrum**.

a. Open the **autotune.dsqtune** tune file by selecting **File | Open | autotune.dsqtune**.

b. Select **Experiment | Full Scan** and scan from *m/z* 10 to 90.

c. Select **Instrument | Fil/Mult/Dyn On** to turn the instrument on.

d. Compare the **Air/Water Spectrum** with those in Figure 1-21 through Figure 1-23to determine whether or not you have a leak.

Usually, if *m/z* 28 is larger than *m/z* 18 there is a leak. refer to procedure *1.14 Leak Checking, pp. 59*.

NOTE: Figure 1-21 through Figure 1-23 were acquired with a 250 L/s turbomolecular pump system. Relative abundance of air/water ions can vary depending upon the pump option and the amount of air in the helium supply. Nitrogen is typically the most abundant contaminant in any helium supply.

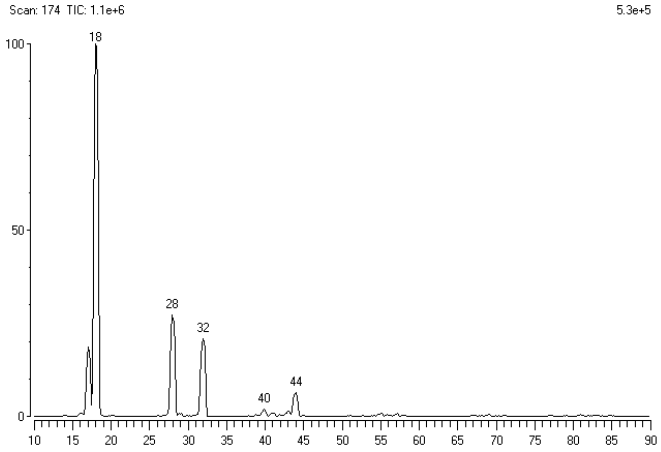


Figure 1-21 Air/Water Spectrum (No Leak)

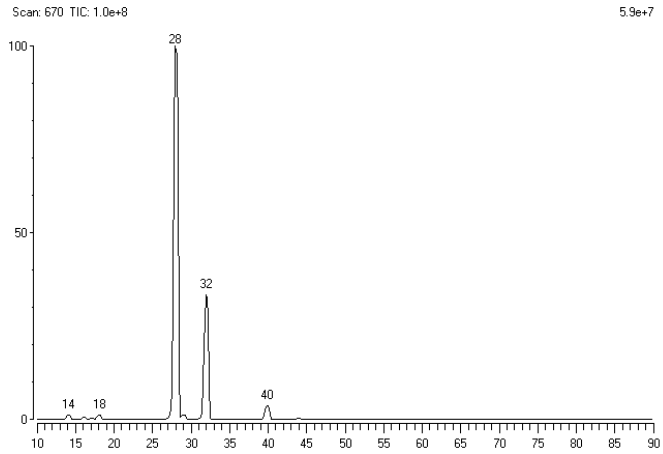


Figure 1-22 Air/Water Spectrum (Small Leak)

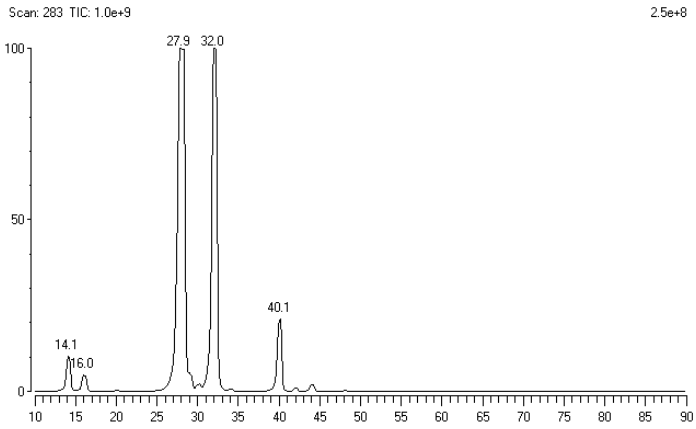


Figure 1-23 Air/Water Spectrum (Large Leak)

1.14 Leak Checking

	**Tools Needed** • Dusting spray containing tetrafluoroethane (Falcon® Dust-Off®, MicroCare® MicroBlast™, or equivalent) • Leak detector (GL Sciences, Inc. Model LD-228 or equivalent)
	**Frequency** When the Air/Water spectrum indicates *m/z* 28 ion is larger than *m/z* 18

1 Check for external leaks.

a. Use the leak detector to check all gas fittings for leaks. These include the carrier gas and CI reagent gas lines. Also check the capillary column connection to the injector. Even a small leak in a gas line can allow air to enter.

b. Use the leak detector to check the septum for a leak. Replace if necessary.

2 Check for vacuum leaks.

a. Scan from *m/z* 50 to 100 by selecting **Experiment | Full Scan**.

b. Spray dusting spray near the suspected leak while monitoring for an increase in *m/z* 69 and 83. Common leaks occur at the transfer line capillary column connection and the vacuum manifold cover.

1.15 Checking the Initial Tune

	**Tools Needed** None
	**Frequency** Day 1

After a successful Air/Water check, check the initial tune of the system.

1 Make sure the instrument is on, select **Instrument | System On**.

2 Make sure the **filament** is on, select **Instrument | Fil/Mult/Dyn On**.

3 Check the **background spectrum**.

a. Select **Experiment | Full Scan** and scan from *m/z* 10 to 650.

b. Verify that the **background spectrum** is similar to Figure 1-24. The background may be higher the first day after being pumped down.

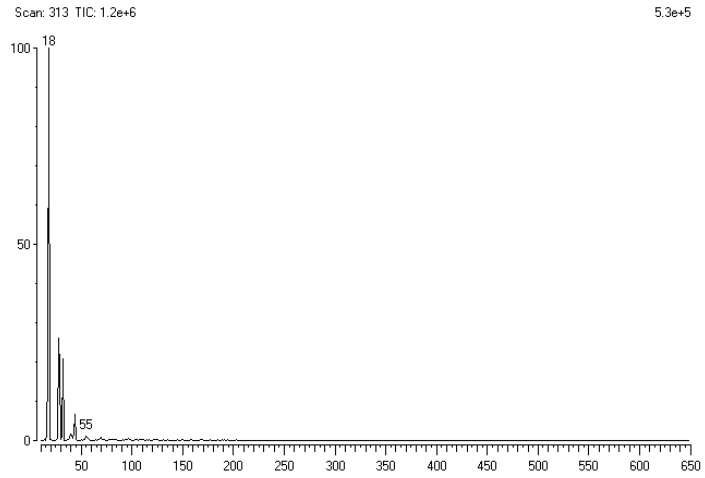


Figure 1-24 Background Spectrum (*m/z* 10 - 650)

4 Check the **calibration gas spectrum**.

a. Select **Experiment | Full Scan** and scan from *m/z* 50 to 650.

b. Select **Instrument | Calibration Gas | EI/NICI** to turn the **EI Calibration Gas** on.

c. Verify that all peaks are present (*m/z* 69, 131, 264, 414, and 502) as in Figure 1-25.

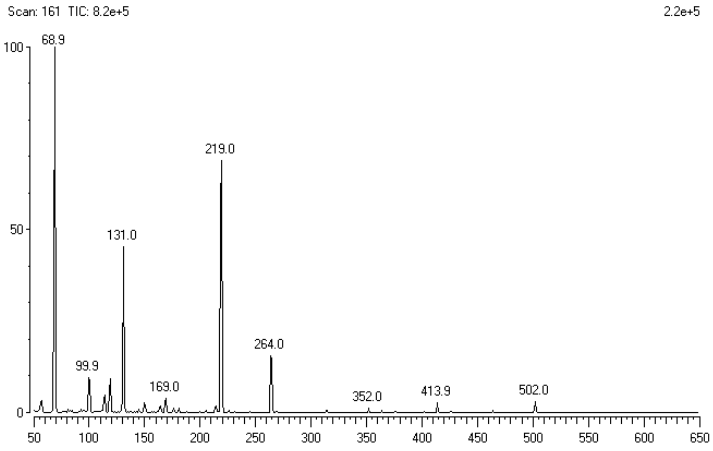


Figure 1-25 Calibration Gas Spectrum (*m/z* 50 - 650)

5 Run a **Full Automatic Tune** if ions are within ±12.5% or ±20 *m/z* units, whichever is smaller, then run a full automatic tune to calibrate the instrument.

a. Run an **Automatic Tune** by selecting **Tune | Automatic Tune** to display the **Automatic Tune screen**. Select these options:

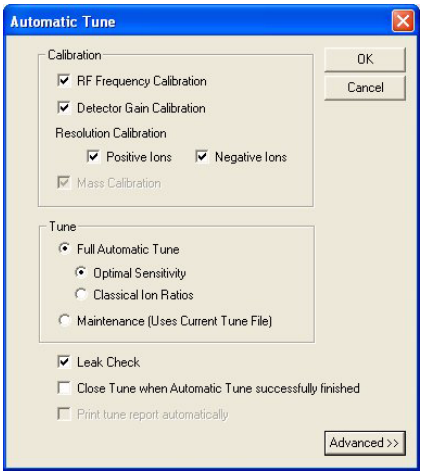


Figure 1-26 Automatic Tune - Full

b. Select the options indicated in Figure 1-26 Automatic Tune - Full. Except, select the **Resolution Calibration** option for **Negative Ions** only if the instrument will be used for negative chemical ionization. This calibration may require that the mass spectrometer gain be increased from 1 x 10^5 to 3 x 10^5 in order to pass. Only increase the mass spectrometer gain if the calibration fails on the first attempt. The tune report will display *m/z* -452 when this calibration has been run. It is normal for this ion to display poor resolution. The resolution for negative ions can be tuned once the instrument is switched to Negative Chemical Ionization mode.

c. Press the **Advanced** button to access the **Advanced Calibration screen**. Using the **Advanced Calibration Screen** lets you compensate for a dirty instrument by adjusting the lens values, while still allowing you to use full tuning options. For example, if you adjust the lenses by resetting the **Prefilter Offset** and **Ion Offset** values, you can save these values to the **Cal_tune.dsqtune** file. And then after getting a successful tune, return to the **Advanced Calibration screen** to press the **Restore button**, which restores the factory default tuning factors which are optimal for a clean instrument.

6 Run a **Manual Calibration** if ions are further away by selecting **Tune | Manual Calibration** to display the **Manual Calibration screen**.

However, this option is only available if you selected **Enable manual calibration** (located on the **Service** tab) during **Instrument**

Configuration. See the *Tune Help Topic: Manual Calibration Screen* for detailed information.

7 Press **OK** to return to the **Tune window**.

8 Turn the **filament** off by selecting **Instrument | Fil/Mult/Dyn Off**.

1.16 Stabilizing the Instrument

	**Tools Needed** None
	**Frequency** End of Day 1

Once the initial tune looks good, allow the system to bake-out the vacuum to stabilize, and allow the gas lines to purge before performing the installation qualification tests tomorrow.

1 Replace any covers that were removed.

2 Set the **CI Reagent Gas Flow** to 2.5 mL/min.

3 Perform a column **leak check**.

a. Run the automated Leak check on the GC.

b. If the report indicates a leak, then look for and fix leaks at all the fittings in the GC using the leak detector.

c. Repeat this step, until no leaks are indicated.

CAUTION Instrument Damage	
	Do not raise the oven temperature until you are sure the system is leak free. At temperatures above 100 °C, the column will be destroyed if exposed to oxygen.

4 Set the GC **Oven Temperature** to 250 °C.

5 Set the **Ion Source Temperature** to 250 °C.

6 Allow the **CI Reagent Gas Flow** to purge.

7 Allow the **Ion Source** and **Oven** to bake-out overnight.

1.17 Running EI Qualification Tests

	**Tools Needed** • Syringe, 10 µL, 70 mm needle (PN 365 001 03) • Test mix, octafluoronaphthalene and benzophenone (PN 120150-TEST)
	**Frequency** Beginning of Day 2

Instrument Methods are already loaded inside the computer for use in running the qualification tests. These methods were used during the factory qualification tests. They can be found in \Xcalibur\Examples\Methods and are organized by GC configuration. Choose the *EI.meth for EI.

1 Set the **Ion Source Temperature** to 200 °C.

2 Set the **GC Oven Temperature** to 40 °C.

3 Turn off the **CI Reagent Gas**.

4 Wait at least 30-min for the system to stabilize.

5 Check **Air/Water Spectrum**.

a. Scan from *m/z* 10 to 90 by selecting **Experiment | Full Scan**.

b. Turn instrument on by selecting **Instrument | Fil/Mult/Dyn On**.

c. Compare the **Air/Water Spectrum** with those in Figure 1-21 through Figure 1-23 to determine whether or not you have a

leak. Usually, if *m/z* 28 is larger than *m/z* 18 there is a leak, go to procedure *1.14 Leak Checking, pp. 59*.

NOTE: When archiving data, include the Mass Spectrometer serial number and sales order number.

d. Archive the **Air/Water Spectrum**.

6 Check **Background Spectrum**.

a. Scan from *m/z* 10 to 650 by selecting **Experiment | Full Scan**.

b. Verify that the **Background Spectrum** is similar to Figure 1-24 Background Spectrum (m/z 10 - 650).

c. Archive the **Background Spectrum**.

7 Check the **Calibration Gas Spectrum**.

a. Scan from *m/z* 50 to 650 by selecting **Experiment | Full Scan**.

b. Turn the **EI Calibration Gas** On.

c. Verify that all peaks are present (*m/z* 69, 131, 264, 414, and 502) as in Figure 1-25 Calibration Gas Spectrum (m/z 50 - 650).

8 Run a **Full Automatic Tune**.

a. Run a full automatic tune by selecting **Tune | Automatic Tune** to display the **Automatic Tune Screen**.

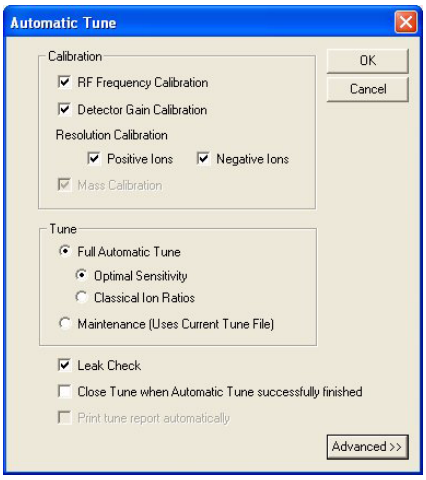


Figure 1-27 Automatic Tune - Full

b. Check the tests indicated in Figure 1-27 Automatic Tune - Full and select **OK**.

c. When finished, archive the **Tune Report.**

d. Turn the instrument off by selecting **Instrument | Fil/Mult/ Dyn Off**.

e. Save the tune file for the **EI Qualification Test** by changing the emission current to 350 µA in the AutoTune tune file and save it as **EI.dsqtune**.

f. Select **OK** to return to the **Tune window**.

9 Inject 1 µL of a 1 pg/µL solution of **octafluoronaphthalene** using *EI.meth as the instrument method.

10 Review the results.

a. Look at the background subtracted spectrum in **Qual Browser**.

b. Archive the chromatogram and spectrum.

c. Use the **Signal-to-noise Calculator** (accessible from a shortcut on the desktop) to display a mass chromatogram for the range 271.5-272.5.The minimum **Signal-to-Noise Ratio** should be:

- 25:1– 70 L/s pump
- 50:1– 250 L/s pump
- 50:1– 200/200 L/s pump

d. Archive the signal-to-noise result.

NOTE: To add Tools to the Home Page select Tools | Add Tools. The location is \Xcalibur\System\Programs \SigNoise.exe.

11 Repeat the octafluoronaphthalene injection.

If the instrument does not pass after several injections, check for leaks especially at the **transfer line union**.

Troubleshooting Tips

Comparing the factory test RAW files to your files. Look for any discrepancies in your methods, or instrument readbacks by selecting **View | Report | Status log, Tune Method, and Instrument Method** in **Qual Browser**.

1.18 Running CI Qualification Tests

	**Tools Needed** • CI ion volume (PN 119650-0230) • I/R tool and guide bar • Syringe, 10 µL, 70 mm needle (PN 365 001 03) • Test mix, octafluoronaphthalene and benzophenone (PN 120150-TEST)
	**Frequency** Day 2 (If Applicable)

If the Mass Spectrometer has the CI (chemical ionization) upgrade option, you must run CI Qualification Tests.

1 Load the appropriate CI method.

a. Select **Instrument Setup** from the Xcalibur home page.

b. Select **File | Open** to load the appropriate factory instrument method.

c. Navigate to **\Xcalibur\Examples\Methods**. Method files are organized by several GC configurations, so you can choose either the ***PCI.meth** for **positive ion CI** or ***NCI.meth** for **negative ion CI**.

2 Prepare for the instrument for **CI** mode.

a. Install a clean **CI Ion Volume** using the **I/R Tool** and the **Inlet Valve** found on the Mass Spectrometer.

b. Open the **Tune window** and select **Instrument | CI Reagent Gas** to display the **CI Reagent Gas Flow screen**.

c. Check the **Reagent Gas On** option.

d. Enter 2.5 mL/min in the **Reagent Gas Flow box** or use the slider to set the flow in the **Reagent Gas Flow box**.

e. Click the **Apply button** and then the **OK button** to return to the **Tune window**. Now, you'll see the Automatic Tune window changed from EI mode to CI mode.

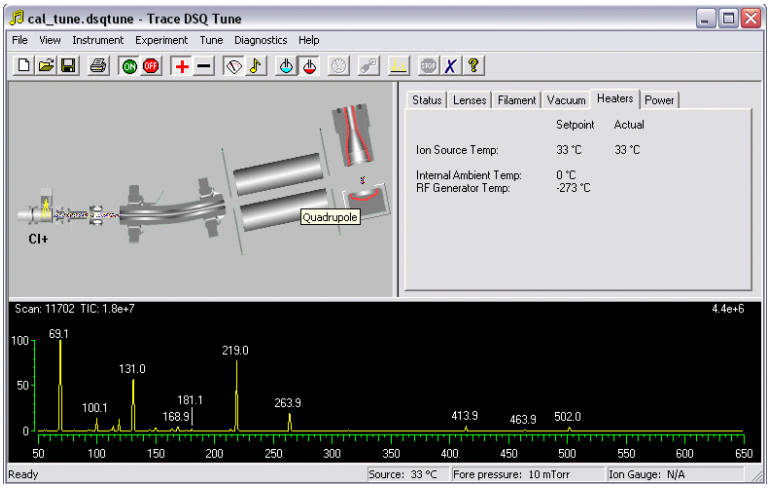


Figure 1-28 Tune Window - CI Mode

f. Wait at least 30-min for the **Ion Volume** to heat up and bake-out.

3 Check the **Reagent Gas spectrum**.

a. Select **Experiment | Full Scan** and scan from *m/z* 10 to 90.

b. Verify that the **Reagent Gas spectrum** is similar to Figure 1-29 Positive Ion CI Spectrum (2.5 mL/min Methane).

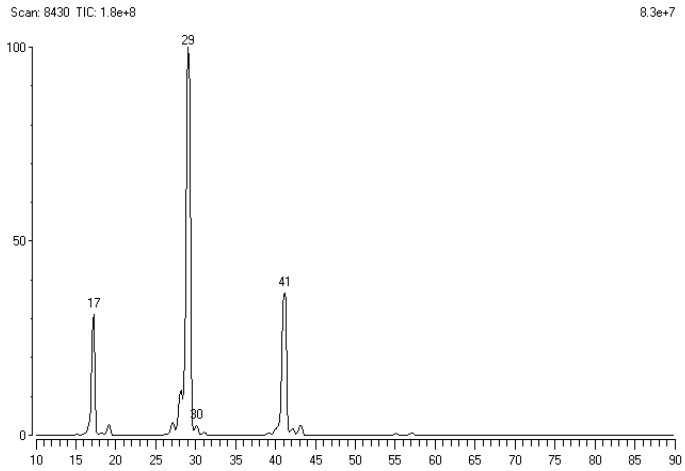


Figure 1-29 Positive Ion CI Spectrum (2.5 mL/min Methane)

c. If it doesn't, see if the **Ion Volume** is inserted correctly (adjust it with the **I/R Tool**) or if the **filament** is properly aligned with the **electron lens hole** (shut down the instrument and use a needle to bend the filament wire to the center of the electron lens hole.)

d. Archive the **Reagent Gas spectrum**.

4 Check the **Calibration Gas Spectrum**.

a. Select **Experiment | Full Scan** and scan from *m/z* 60 to 700.

b. Select **Instrument | Calibration | PICI** to turn the **PICI Calibration Gas** on.

c. Verify that the **Calibration Gas Spectrum** is similar to Figure 1-30 Positive Ion Calibration Gas CI Spectrum (2.5 mL/min Methane).

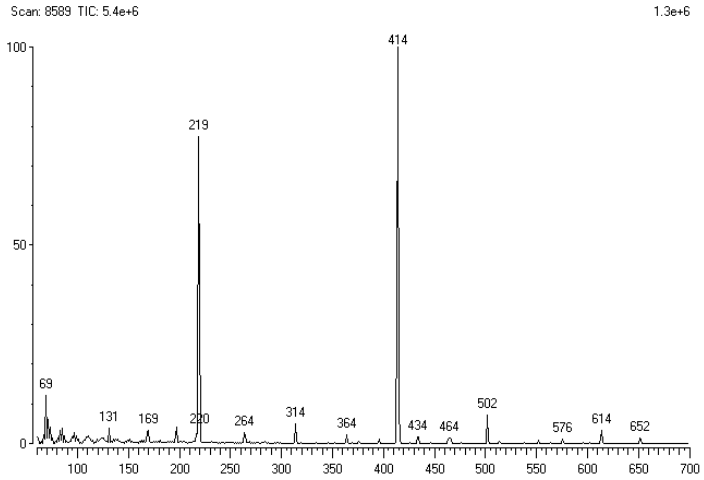


Figure 1-30 Positive Ion Calibration Gas CI Spectrum (2.5 mL/min Methane)

d. If it doesn't, see if the **Ion Volume** is inserted correctly (adjust it with the **I/R Tool**) or if the **filament** is properly aligned with the **electron lens hole** (shut down the instrument and use a needle to bend the filament wire to the center of the electron lens hole.)

5 Select **File | Open | Tune** and select the **autotune.dsqtune** file.

Change the:

- Electron energy to 120 eV
- Emission current to 50 µA
- Lens 2 to approximately 2 V closer to 0 V

6 Begin **PICI tests**.

a. Select **Tune | Automatic Tune** to display the **Automatic Tune CI screen**.

b. Make the selections illustrated in Figure 1-31 CI Automatic Tune - PICI Tests.

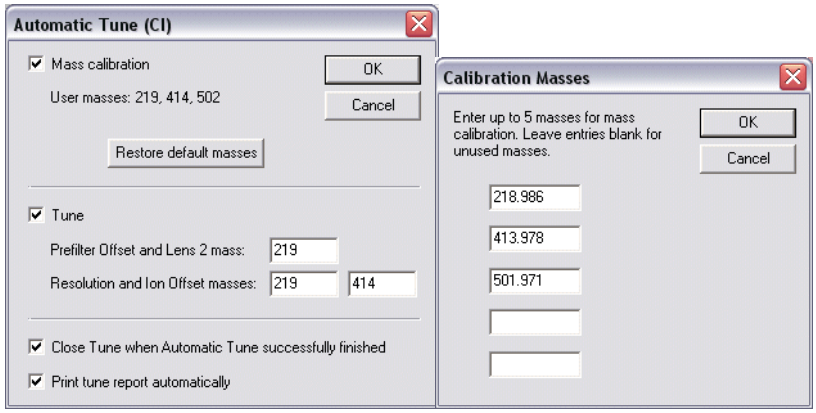


Figure 1-31 CI Automatic Tune - PICI Tests

c. Select the **Calibration Masses button** to display the **Calibration Masses screen.**

d. Enter these masses **218.986**, **413.978**, and **501.971** for the mass calibration.

e. Click **OK** to return to the **Tune window**.

f. Save the PICI auto-tune file as **PICI.dsqtune** for the **Installation Qualification test**.

g. Archive the **Calibration Gas Spectrum**.

h. Close the **Tune window**.

7 Perform the **PICI Installation Qualification test**.

a. Go to Xcalibur | Sequence Setup and open the preset method ***PCI.meth**.

b. Inject 1 µL of a 10 pg/µL solution of **benzophenone** using the preset instrument method ***PCI.meth**.

c. Go to **Qual Browser** to review the results, by looking at the **background subtracted spectrum.**

d. Archive the **chromatogram** and **spectrum**.

e. Select the **Signal-to-noise Calculator** (accessible from a shortcut on the desktop) to display a mass chromatogram for the range **182.5—183.5.** The minimum **Signal-to-Noise Ratio** should be **10:1**.

f. Archive the signal-to-noise ratio.

g. Repeat the **benzophenone injection**.

NOTE: To add tools to the Home Page select Tools | Add Tools. The location is \Xcalibur\System \Programs\SigNoise.exe.

8 Begin **Negative CI Installation Qualification tests**.

a. Go to the **Tune window** and open the **autotune.dsqtune file**.

b. Select the  **negative-ion button** to change to **negative ion mode**. You can confirm this by viewing the Tune window.

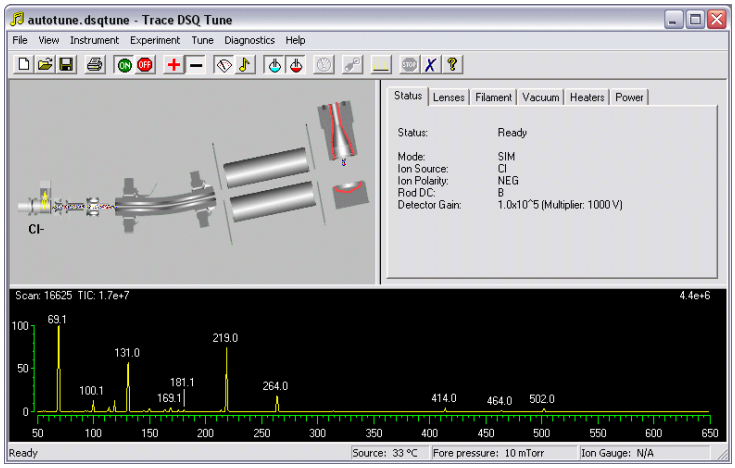


Figure 1-32 Tune Window - Negative CI Mode

c. Select **Instrument | CI Reagent Gas** to display the **CI Reagent Gas screen**.

d. Select the option **CI Reagent Gas On** to turn the CI reagent gas on.

e. Select **Tune | Automatic Tune** to display the **Automatic Tune (CI) screen** and enter the Prefilter settings displayed in Figure 1-33 Automatic Tuning - Negative CI Tests.

f. Select the **Change Masses button** to enter these *m/z* values **282.9855**, **311.9871**, **413.9775**, **451.9743** into the **Calibration Masses screen**:

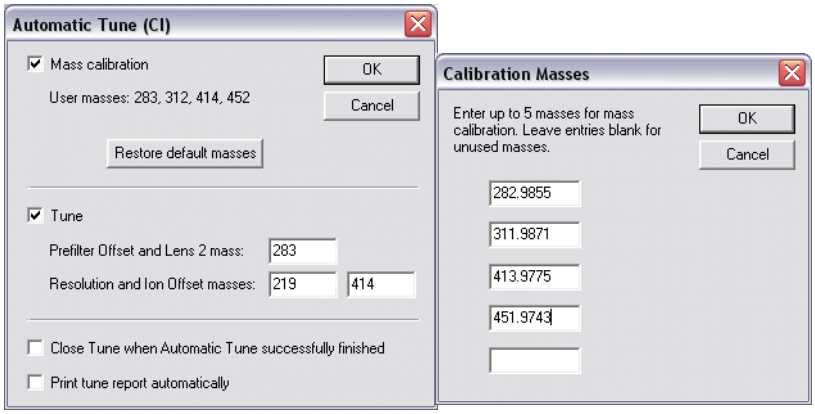


Figure 1-33 Automatic Tuning - Negative CI Tests

g. Save the NICI auto-tune file as **NCI.dsqtune** for the **Installation Qualification test**.

h. Close the **Tune window**.

9 Perform the **NCI Installation Qualification test**.

a. Inject 1 µL of a 1 pg/µL solution of **octafluoronaphthalene** using the preset ***NCI.meth** instrument method.

b. **Go to Qual Browser** to review the results by looking at background subtracted spectrum.

c. Archive the **chromatogram** and **spectrum**.

d. Use the **Signal-to-noise Calculator** (accessible from a shortcut on the desktop) to display a mass chromatogram for the range **271.5—272.5**. The minimum **S/N** should be **500:1**.

e. Archive the signal-to-noise ratio.

f. Repeat the **octafluoronaphthalene injection**.

1.19 Installing Upgrade Options

	**Tools Needed** Option dependent
	**Frequency** Installation Day 2

Once you have finished installing and testing the basic TRACE DSQ or FOCUS DSQ system, install any additional options. Upgrade options may include:

- Autosampler(s): With a TRACE GC, the serial connection to the AI/AS 3000 or AS 2000 should be connected to the GC.

  With a *Focus GC* or other type of autosampler, connect the serial connection to COM 2 on the computer.

- Direct Sample Probe (DIP or DEP)

1.20 Providing Basic Training

	**Tools Needed** None
	**Frequency Day 2**

Now that the system is running to factory specifications, show the customer how to use the Xcalibur software.

1 Demonstrate **Instrument Configuration**.

a. Start **Instrument Configuration** from the desktop.

b. Show how to add, configure, and delete components.

2 Demonstrate the **Roadmap View** in the **Xcalibur Home Page**.

a. Start the **Xcalibur Home Page** from the desktop.

b. Show how to use the **Roadmap View**.

c. Show how to view the instrument status.

3 Demonstrate **Instrument Setup**.

a. Start **Instrument Setup** from the **Home Page**.

b. Show how to setup a **GC** method.

c. Show how to setup a Mass Spectrometer method.

d. Show how to setup an autosampler method (if applicable).

4 Demonstrate the TRACE DSQ Tune window.

a. Start **Tune** from **Instrument Setup**.

b. Show how to open Tune files.

c. Show how to look at an air/water spectrum.

d. Show how to look at a calibration gas spectrum.

e. Show how to run an Automatic Tune.

f. Show how to run Diagnostics.

5 Demonstrate the **Sequence View** in the **Xcalibur Home Page**.

a. Set up a new sequence.

b. Run a sample.

6 Demonstrate the **Qual Browser**.

a. Open a *.raw file.

b. Add a cell to view a spectrum.

c. Explain how to use push-pins.

d. Explain background subtraction.

e. Demonstrate how to change the chromatogram range and mass.

7 Remind the customer to use *Chapter 2, Getting Started, pp. 81* to reinforce all that you have demonstrated today.

2 Getting Started

This chapter provides GC/MS instructions using **Xcalibur 1.4** software.

In This Chapter

Analyze • Detect • Measure • Control™

Upgrades

Periodically visit our website, http://www.thermo.com, for the latest releases on application notes, new manuals, software and firmware updates, and information on new features.

2.1 Using Desktop Icons

NOTE: You might find it helpful to copy this page and keep it out to use as a quick reference template while you're getting used to Xcalibur.

After the instrument is connected, it's a good idea to get acquainted with how your software is organized on the desktop. Xcalibur desktop icons can help you quickly get to where you want to go.

Desktop Icon	What it Does...
 Instrument Configuration	Launches the Xcalibur Instrument Configuration program. Use this program to set up and inform Xcalibur which devices are connected to the data system.
 Xcalibur	Launches the Xcalibur Home Page program.
 DSQTune	Launches the DSQ Tune Window. HINT: When running Xcalibur, you can also use the Q icon located in your Windows task bar.
 tuneedit	Launches the DSQ Autotune Tune Edit program. This program is a replicate of the Show Manual Tune View command, which is located in the DSQ Tune Window. Use this to create and edit tune files.
 Signal to Noise Calculator	Launches the Xcalibur Signal-to-noise Calculator. This program allows the Signal-to-Noise to be calculated for specific ranges within *.raw files.
 Signal to Noise Instructions	Launches the MS Word based signal-to-noise Instructions. Use this for help in using the Signal-to-noise Calculator.
 Merlin	Launches a MS Word based document. Merlin is a Microsoft Word Template, designed to help you create reports. It automatically loads the Merlin.dot file and automatically applies the styles to your text.

2.2 Configuring Instruments for Use

1 Select the **Xcalibur Instrument Configuration icon** 

from the Windows desktop.

2 Configure the mass spectrometer (MS).

a. Double-click the **TRACE DSQ** icon located in the **Available Devices column** to move it to the **Configured Devices panel**. Select the *TRACE DSQ* even if you have a FOCUS DSQ.

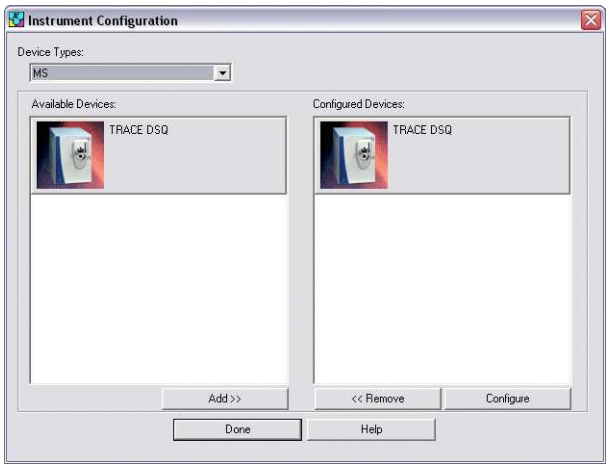


Figure 2-1 MS Instrument Configuration

b. Double click the **TRACE DSQ icon** from in the **Configured Devices panel** or click the **Configure button** to display the **Instrument Configuration screen**.

c. Use the tabs on the **Instrument Configuration screen** to select options to display on the **Instrument Setup** and **Tune windows**.

NOTE: Select the **Help button** to use the online help for more detailed instructions.

d. Configure **General tab** options:

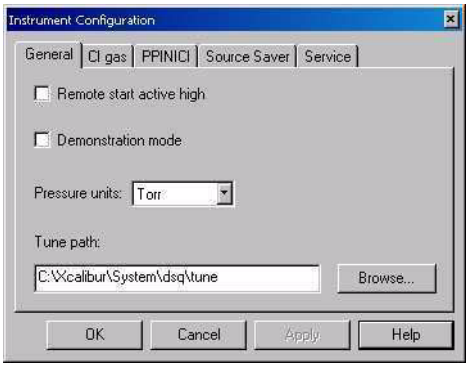


Figure 2-2 Instrument Configuration: General

- The default setting for **Remote Start Active High** is unchecked. This starts the GC with an active high signal instead of active low.
- **Demonstration Mode** is used to start the instrument in demonstration mode. This mode is useful if no instrument is connected, but you would like to demonstrate the software. But don't select this to run your first sample.
- Specify the **Pressure Units** to display in the **Tune window** and the **Status tab**.
- Specify the **Tune Path** for Xcalibur to automatically retrieve stored tune files. Use the **Browse button** to quickly navigate to the folder storing your tune files. The factory default is **Xcalibur | System | DSQ | Tune**.

e. **[Optional]** Configure **CI Gas tab** options, if you have the CI option installed.

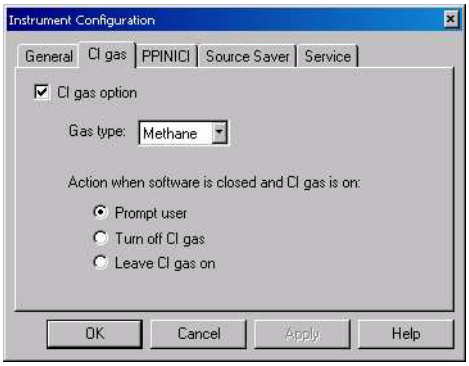


Figure 2-3 Instrument Configuration: CI Gas

- **CI Gas option**: check to activate CI gas options.
- **CI Gas Type**: select a **gas type**. Select this option to activate chemical ionization gas controls. Options include: Ammonia, CO_2, Isobutane, Methane, and Other. **Other** refers to any gas type not listed in this list. When **Other** is selected, the flow module is set to a pressure of 0.3–7.4 psig against a fixed flow restrictor. If a specific gas is selected, the CI flow module outputs a calibrated flow for that gas in mL/min.
- **Action when Software is closed and CI Gas is on**: When Xcalibur software is closed, these options determine what happens if CI gas is on. You can choose to turn it off, leave it on, or receive a prompt at the time the software is closed asking if you want to turn the CI gas off. If you choose to leave the CI gas on, it remains on until you restart the software. However, as soon as you restart the software, the CI gas is turned off.

f. **[Optional]** Configure **PPINICI tab** options.

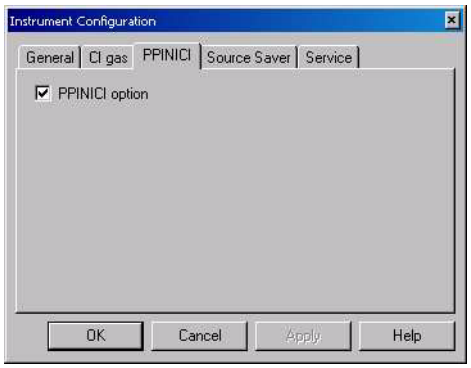


Figure 2-4 Instrument Configuration: PPINICI

- Check the **PPINICI** option, if you have the PPINICI option installed on your mass spectrometer.

g. Configure **Source Saver tab** options.

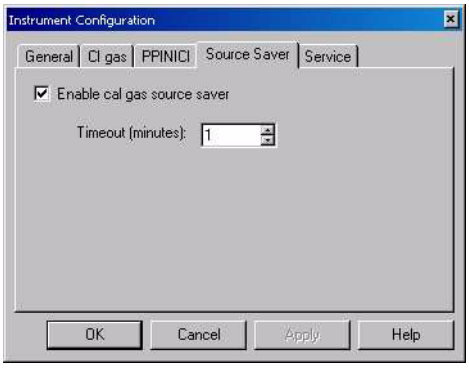


Figure 2-5 Instrument Configuration: Source Saver

- **Enable Cal Gas Source Saver**: Check this option to enable the instrument to turn off the filament and cal gas if left unattended with the cal gas on. If the cal gas is not on, source saver will not turn off the filament.

- **Timeout (minutes)**: Enter the time in minutes that the instrument must wait after the last user action in tune before turning off the filament and cal gas.

h. **[Field Engineers Only]** Configure **Service tab** options.

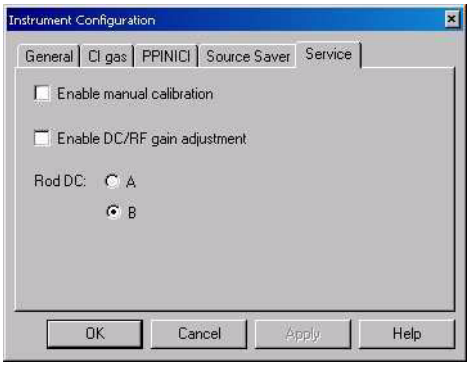


Figure 2-6 Instrument Configuration: Service

NOTE: Please do not adjust these settings. Only factory-trained Field Engineers are qualified to set the options in the Instrument Configuration Service tab.

- **Enable Manual Calibration**: Select this option to display the **Manual Calibration menu item** in Tune. This allows the field engineer to manually calibrate the coarse mass calibration and resolution for troubleshooting. We do not recommend using manual calibration values for normal operation because the calibration will not be accurate for changing scan rates and the manual resolution calibration makes resolution values in tune files meaningless.

- **Enable DC/RF Gain Adjustment:** Select this option to display the DC/RF Gain menu item in Tune.
- **Rod DC**: Select A or B. This should be set in the factory and never change unless the DC Driver PCB, RF subsystem, or quadrupole mass filter is changed. If changed, calibrations for both positive and negative ions must be performed. The terms A and B refer to the selection of which DC polarity is applied to each quadrupole rod pair for each ion polarity.

3 Configure the gas chromatograph (GC).

a. Move the **GC** from the **Available Devices panel** to the **Configured Devices panel**.

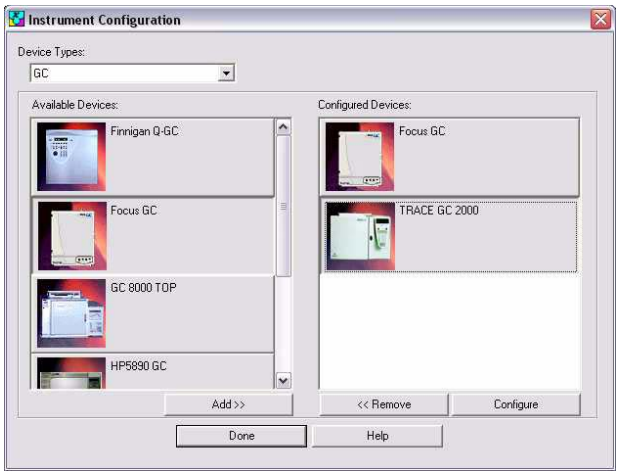


Figure 2-7 GC Instrument Configuration

b. Select the **GC icon** in the **Configured Devices panel**, then click the **Configure button** to display the **GC Configuration screen**.

c. Make these selections on the **General tab**:

- **Serial Port** = COM 1.
- Set the **preferred pressure units**.
- Click the **Get button** to retrieve the GC configuration through the serial port to Xcalibur.

d. Verify the settings on the **Inlet tab** match the GC configuration.

e. Verify the settings on the **Detectors and Data tab** match the GC configuration.

f. Verify the settings on the **Auxiliary and Oven Options tab** match the GC configuration and **Aux 1 Present** has the **MS Transfer Line** selected.

g. Click **OK**, if no problems are detected and return to the **Instrument Configuration screen**.

4 Configure the autosampler (AS).

a. Move the **AS icon** from the **Available Devices panel** to the **Configured Devices panel**.

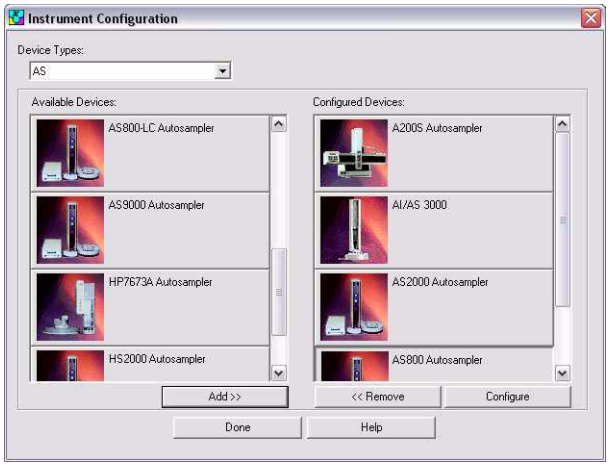


Figure 2-8 AS Instrument Configuration

b. Double-click the **AS icon** in the **Configured Devices panel**, then click the **Configure button** to display the **autosampler Configuration screen**.

c. Enter autosampler parameters in the **AS Configuration screen**. The autosampler is usually connected to a COM port on the computer (with a Focus GC) or through the GC (with a TRACE GC and an AI/AS 3000 or AS 2000.) A 10 µL syringe is standard.

5 Set up the GC handshaking parameters by referring to the GC manuals for configuration instructions.

Table 2-1 GC Handshaking Parameters

Remote Start In	High to Low
Inhibit Ready In	When High
End of Run Out	High to Low
Start of Run Out	High to Low
GC Ready Out	When Low
Prep Run Out	When Low

6 Click the 

windows desktop short-cut to display the Xcalibur home page.

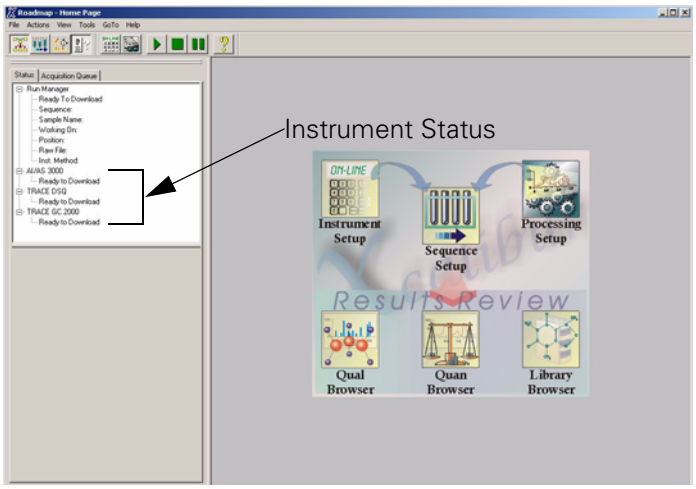


Figure 2-9 Xcalibur Home Page

7 Test the AS, GC, and MS communication to Xcalibur.

a. Check the Instrument Status in the Status tab of the Info View. All instruments should report Ready to Download. If not,

consult the troubleshooting section of the instrument's appropriate manual.

NOTE: The MS may take up to 1-min to initialize.

b. Okay, if you're ready to run your first sample, go to *Chapter 3, Running Your 1st Sample, pp. 93*.

3 Running Your 1st Sample

In This Chapter

Analyze • Detect • Measure • Control™

After your instruments are configured for use with Xcalibur you're ready to run your first sample. In just three easy steps you'll be running your first sample using *octafluoronaphthalene*, which comes with your instrument, as your test compound.

NOTE: These instructions are written with Trace GC, and AI/AS 3000 assumptions. However, making the necessary modifications and/or omissions will still help you accomplish each step.

3.1 Calibrating and Tuning the Instrument

Before you run a sample, it's wise to calibrate and tune the instrument using the **Tune window**.

NOTE: Select online help for **Tune Window** information.

1 Open the **Tune program** using one of these methods:

- Click the Tune from the **MS method editor** or,
- Select the DSQTune shortcut from the **Windows desktop**.

2 Check the instrument's basic operation by selecting **Diagnostics | Run Test** to display the **Diagnostics screen**.

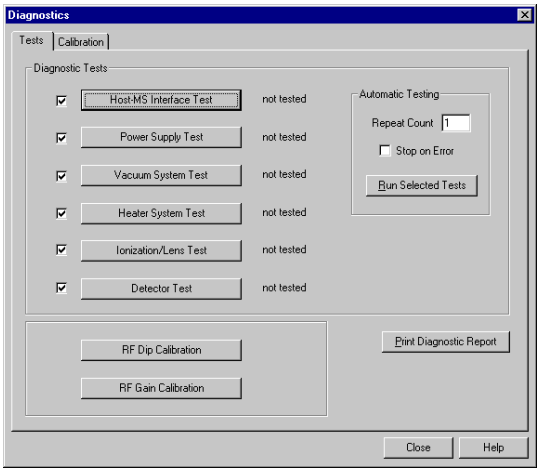


Figure 3-1 Tune Window Diagnostic Screen

a. Click on the **Test buttons** to run each test.

b. Select the **Print Diagnostics Report button** to print the results from all the tests you ran.

c. Close the Diagnostics.

3 Check the last **Tune Report**.

a. Select **Tune | View Tune Report** to view the last tune report and determine when the instrument was last calibrated. Select the **Print button** if you want to have hard-copy handy.

b. Close the **Tune Report**.

4 **[Optional]** Perform a Full **Automatic Tune** if you have just performed maintenance or your standard operating procedure requires it.

It's not necessary to calibrate and tune the instrument more than once every week. However, the number and type of samples you run determine how often you must tune your instrument.

a. Select **Tune | Automatic Tune** to display the **Automatic Tune screen**.

NOTE: How often you tune your instrument is determined by the number and type of samples you run. Please refer to *B.4 Automatic Tune Flow Chart, pp. 141*.

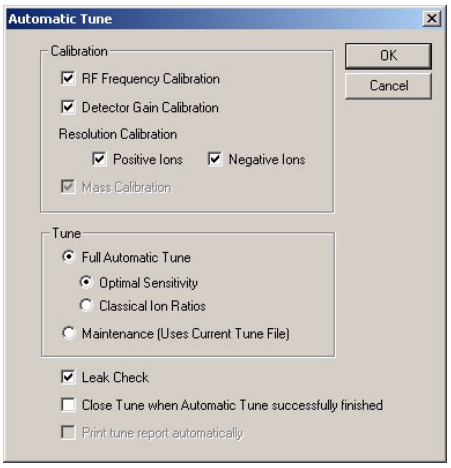


Figure 3-2 Automatic Tune Screen - Full

5 **[Optional]** Perform a manual Tune if needed for other applications.

The tuning parameters generated during automatic tune work well for most applications, but you may find the **Manual Tune** options suitable for other applications.

a. Select **Tune | Show Manual Tune View** to manually set the tune parameters in the tabs indicated in Figure 3-3.

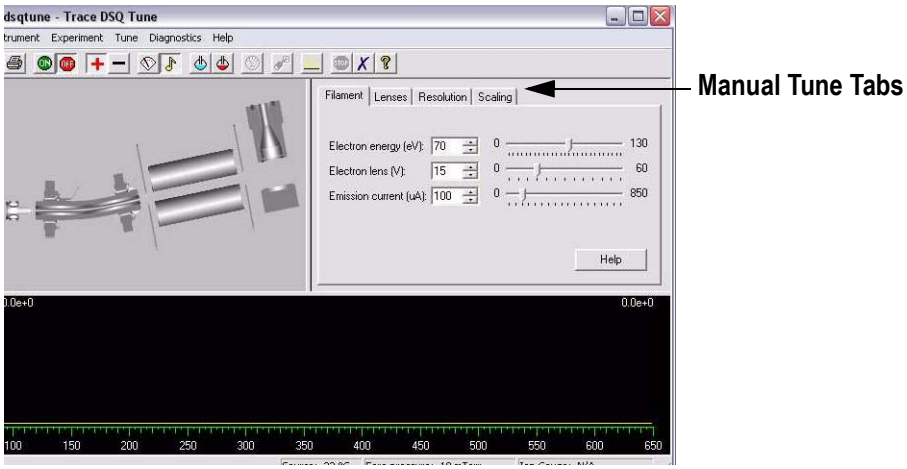


Figure 3-3 Tune | Show Manual Tune Tabs

b. Select **File | Exit** to close the **Tune window**.

3.2 Setting Up Methods

After your instrument is calibrated and tuned, you're ready to set up the instrument methods using **Instrument Setup**. Instrument methods are created and modified in the **Instrument Setup window** and saved as ***meth** files. Method files are stored in the \Xcalibur\Methods\. folder. In this example, you will enter the method parameters for the Mass Spectrometer, GC, and the AS.

1 Load an instrument method.

a. Select **Instrument Setup** from the **Xcalibur Home Page** to display **Instrument Setup window**.

b. Select **File | Open** and navigate to **Xcalibur\Methods**. Alternatively, navigate to **Examples\Methods** and then the folder matching your GC instrument configuration.

c. Choose[1] an ***_EI_FullScan.meth**. This is the full scan method file that was used to validate that your instrument met factory specifications and saved for you to acquire a test sample.

2 Review MS method parameters by selecting the **MS instrument icon** from the **Instrument Setup window**.

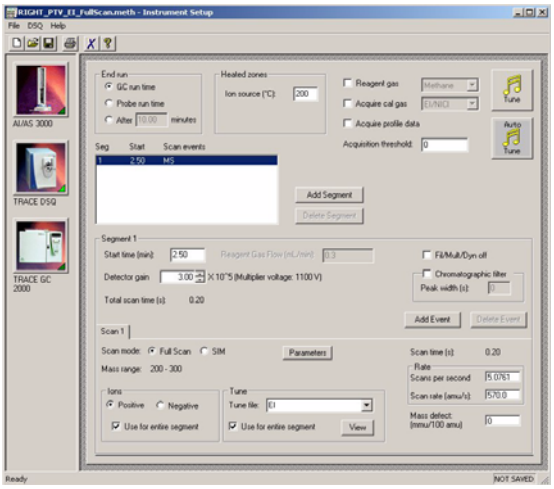


Figure 3-4 Trace DSQ Right_PTV_EI_FullScan.meth Parameters

1.In this example, we went to \Xcalibur\Examples\Methods\Trace Right PTV Methods and selected RIGHT_PTV_EI_Fullscan.meth.

This method acquires *m/z* 200–300. It starts 2.5-min after the GC injection to allow the solvent to elute from the GC column.

3 Review the GC method parameters by selecting the **GC instrument icon** from the **Instrument Setup window**.

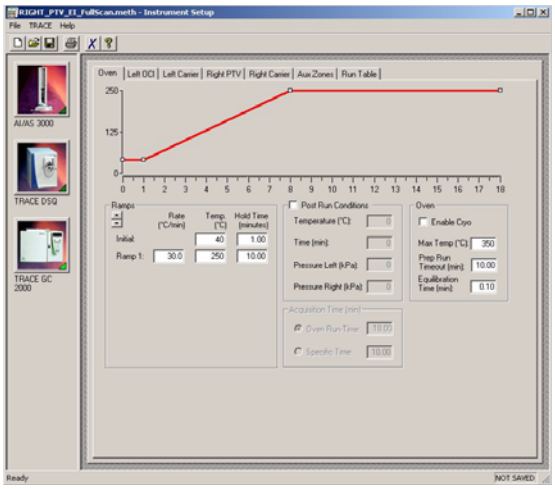


Figure 3-5 Trace GC Method Parameters

This method holds the GC at 40 °C for 1-min, ramps it at 30 °C/min to 250 °C, and holds it there for 10-min. The injector is 220 °C operated in splitless mode. The column flow is 1.0 mL/min. The transfer line temperature is 250 °C.

4 **[Optional]** Review AS method parameters by selecting the **AS instrument icon**.

If you're using an AI/AS 3000 you can enter the parameters shown in Figure 3-6. For other autosamplers, there are several basic settings for all autosamplers for sampling/injection and washes. All other parameters can be left at their default values.

Sampling/Injection:

- Sample Volume = 1 µL (This is the sample volume that will be injected.)
- Air Volume = 1 µL (Draw air after the sample volume to make sure no solvent is in the needle. Having no solvent in the needle and using a pre-injection dwell time of 1-second will give you a hot needle injection. AI/AS 3000 is always 1 µL.)
- Plunger strokes = 10 (Also called pull-ups or bubble elimination strokes. This is the number of times the plunger is pulled up and down while in the sample vial. The plunger volume should be set larger than the sample volume to say 4 µL.)
- Pre-injection dwell time = 1 s (Also called Injection Delay. This gives time for the needle to heat up in the injector before the sample is injected. This hot needle injection helps all analytes to vaporize at the same time.)
- Post-injection dwell time = 1 s (Also called the Pull-Out Delay. This time allows residual solvent in and on the needle to evaporate. If set too long, chromatographic peak tailing can result.)

Washes:

- Pre Injection Washes = A, 2 cycles (This will wash the syringe with solvent vial A before drawing the sample for the injection. This makes sure the syringe is wet and it is clean prior to drawing up the sample. If required, the volume should be set to the size of the syringe, 10 µL.)
- Sample Rinses = 1 (Also called Sample Cleans. This is how many times your sample is drawn into the syringe and discarded before the injection is made. Using at least one means the syringe will be rinsed once with your sample.)
- Post Injection Washes = B, 6 cycles (This will wash the syringe with solvent vial B after the injection is made. This will remove any residual analytes or solvent from the syringe. It is a good idea to use a different vial for pre and post injection washes. This way, the pre injection solvent vial A will stay cleaner.)

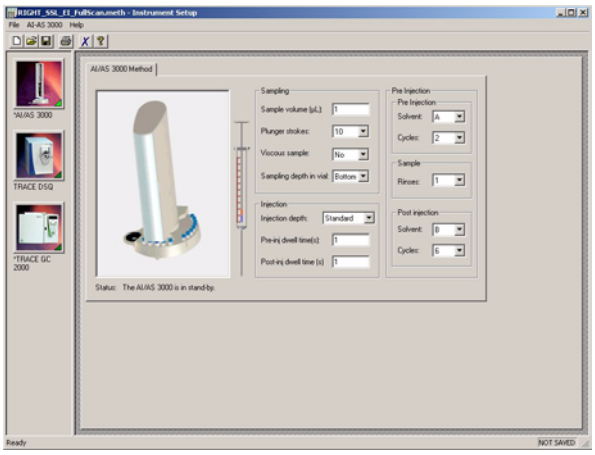


Figure 3-6 AI/AS 3000 Method Parameters

This method injects 1 µL of Iso-octane.

d. Fill the AS solvent vials A and B with a suitable **rinse solvent** such as Iso-octane.

5 Save the **instrument method** for use in the next step.

Method files have the ***.meth** extension and are by default saved to the **Xcalibur | Methods** folder. But, you can save method files to any folder you prefer.

3.3 Running Methods

After setting up the methods in Instrument Setup, you're ready to run the method in Sequence Setup.

1 Create a sequence to run in the ***Sequence Setup window*** to record sample details and determine how you want to acquire the data.

a. Select  or  from the home page to display the **Sequence Setup window**.

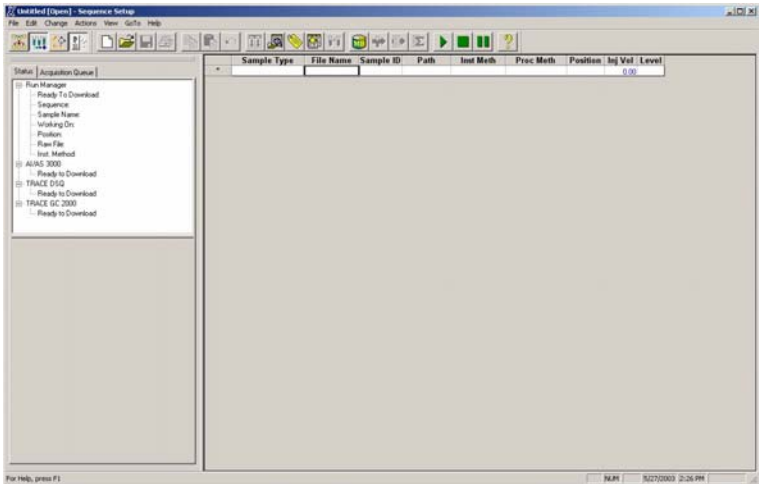


Figure 3-7 Sequence Setup Window - Untitled

Sequence Setup works just like a spreadsheet, with each row representing a single sample and each column an item of information required for a sample.

Column descriptions are in Table 3-1, Sequence Setup Columns, pp.103. Additional columns are available by choosing **Change | Column Arrangement**, such as **Comment**.

Table 3-1 Sequence Setup[1] Columns

Item	Description
Sample Type	Type of sample, selected from the following: • Unknown (the normal choice for qualitative analysis; all other types are only normally used for quantitative analysis) • Blank • QC (quality control) • Standard Clear • Standard Update • Start Bracket • End Bracket • Standard Bracket
File Name	Name of the file where to save the sample data.
Sample ID	An identifier unique to the sample. This field can also be used to import a barcode identifier.
Path	The path to the raw file that Xcalibur creates for the sample data. Xcalibur creates this file with extension.raw. Double-click on this field to select a directory.
Inst Meth	The path and file name of the Instrument Method to be used for acquisition.
Proc Meth	The path and file name of the processing method used to process the data after acquisition.
Position	The sample's vial number. The format of the entry depends on the configured autosampler. AI/AS 3000 and AS 2000 use simple integers.
Inj Vol	Not used in this editor, but used instead in the AS method editor.
Level	Used with Standard and QC samples to enter the sample's concentration level.

1.More information about **recording samples** are in the Xcalibur Online Help or the Xcalibur Getting Productive: Qualitative Analysis manual or the Getting Productive: Quantitative Analysis manual.

b. Start entering information in the blank row at the bottom of the list to add a new sequence.

c. Begin entering the sample information for the file name and path (right-click in the field to display the drop-down menu, choose **Browse**, to select the file.)

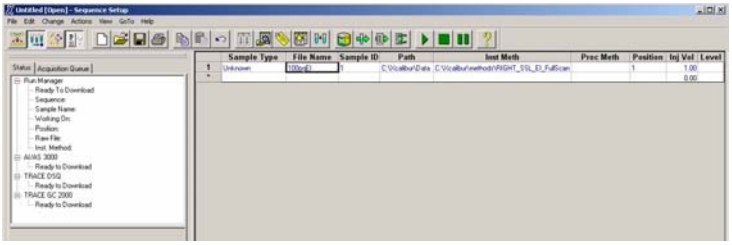
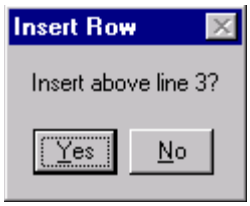


Figure 3-8 Sequence Setup - Sample Information

- To insert a row within a list of samples you have already entered, right-click anywhere in the row below which the new sample is to be inserted and choose **Edit | Insert Row** to display the **Insert Row screen**.



NOTE: Press F2 on your computer to edit text already in a cell. Press F2 two times to see an edit dialog box.

2 Save the sequence, by selecting **File | Save**. Enter an appropriate name such as OFN.sld.

3 Run the sequence by clicking on the row number you wish to run (this highlights the row).

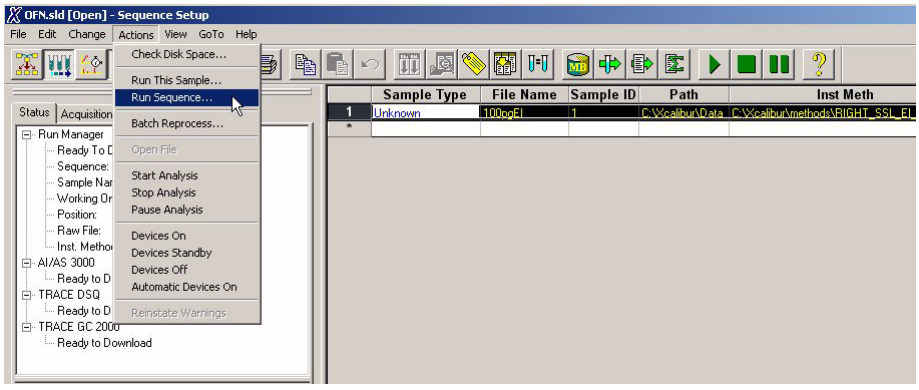


Figure 3-9 Sequence Setup - Running A Sequence

a. Select **Action | Run Sequence** to display the **Run Sequence screen**.

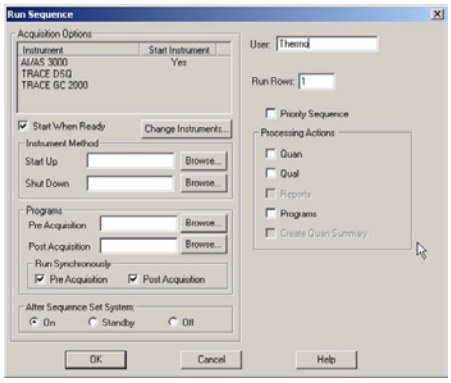


Figure 3-10 Sequence Setup - Run Sequence Screen

b. Select **Start When Ready**.

c. Press the **Change Instruments button** to display the **Change Instruments screen** and set up your instruments for an **automatic injection** or a **manual injection**. See the topic *If You're Doing Automatic Injections..., pp. 3-106* or topic *If You're Doing Manual Injections..., pp. 3-107* for instructions.

If You're Doing Automatic Injections...

An autosampler performs automatic injections and you instruct Xcalibur when to make the injection.

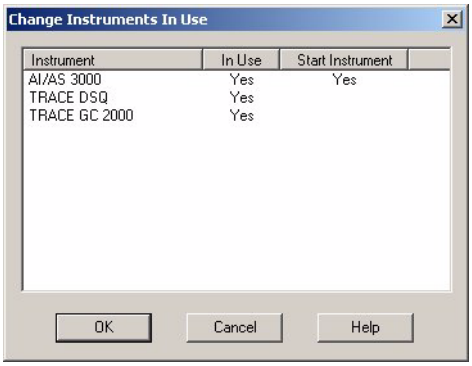


Figure 3-11 Sequence Setup—Change Instruments In Use Screen (automatic injections)

1 Configure the **Change Instruments In Use screen** for automatic injections:

a. Make **Yes** display for each instrument (AS, GC, MS) listed in the **In Use** fields *(Fig. 3-11)*.

However, if **Yes** is not listed, then click on the blank field to toggle **Yes** on.

b. Make **Yes** display in the **Start Instrument field** for the AS. This makes the autosampler start as soon as all devices are ready.

c. Select **OK** to return to the **Run Sequence screen**.

2 Prepare the injection.

a. Open and transfer the **100 pg octafluoronaphthalene** sample— that is in the **GC/MS Test Kit** (PN 120150-TEST)— into an autosampler vial (vials are supplied with the autosampler.)

b. Load the sample vial into position 1 of the autosampler.

3 Start the Run, by selecting **OK** from the **Run Sequence screen** *(Fig. 3-10)*.

If You're Doing Manual Injections...

Manual injections are necessary for those not having an autosampler or not wishing to use it to run this method.

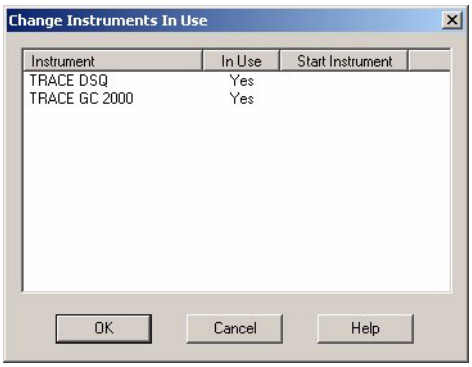


Figure 3-12 Sequence Setup-Change Instruments In Use Screen (manual injections)

1 Configure the **Change Instruments In Use** screen for manual injections:

a. Make **Yes** display for the **GC and MS instruments** listed in the **In Use** fields.However, if Yes is not listed, then click on the blank field to toggle Yes on.

b. Make sure the GC and MS instruments **Start Instrument** fields are empty *(Fig. 3-12)*.

c. Make sure the AS (if listed) **does not say Yes** in both the **In Use** or **Start Instrument** fields.

d. Select **OK** to return to the **Run Sequence screen**.

2 Open the **100 pg octafluoronaphthalene** sample—that is in the **GC/MS Test Kit** (PN 120150-TEST).

3 Select **OK** from the **Run Sequence screen** *(Fig. 3-10)* to start the run.

4 Monitor the Status View until the instrument is **"Waiting for Contact Closure."**

5 Prepare the injection.

a. Draw 1 µL of the **100 pg octafluoronaphthalene** sample into the supplied syringe. Be sure there are no bubbles in the syringe.

b. Draw in 1 µL of air.

6 Make the injection and start the GC.

a. Carefully, insert the needle into the injector port.

b. Wait one-second and push the plunger.

c. Quickly, press the Start button located on the GC.

d. Remove the syringe.

7 Watch the data, while the sequence is running.

This is a good time to watch the data by selecting from one of the two views:

a. Click the **Real Time Plot icon** to view the Chromatogram and Spectrum acquisition in real time. When the run is finished this screen goes blank.

b. Select the **Acquisition Queue tab** to view the list of the samples you have selected to run. Green items mean the sequence is running.

3.4 Analyzing the Data: Compounds With a Known Retention Time

When you're ready to analyze your data, use the Xcalibur browsers (Qual, Quan, and Library) to view the data file results.

NOTE: See the online Help for additional instruction or see: Getting Productive: Qualitative Analysis, Getting Productive: Quantitative Analysis, Getting Productive: Processing Setup and Analysis of Quantitation Data, Getting Productive: Creating and Searching Libraries, and Getting Productive: Designing and Generating Custom Reports with Merlin.

Reviewing Cursor Actions in Browsers

Before you begin using the Browsers, let's review some of the cursor actions and effects within the chromatogram and spectrum cells:

- A click picks a point on the cell
- A line dragged parallel to any axis picks a range
- A line dragged in any diagonal direction selects an area

The effect of these actions depends on one of these cell states:

- **Inactive**
- **Active and unpinned—** each cell has a pin icon in its top right corner. Only one of the cells can be active at any one time. The active cell is highlighted with a gray border.
- **Active and pinned** — pinning fixes the active status of a cell.

1 Unpin an active cell, by clicking once on the pin icon.

2 Make a cell active, by clicking anywhere within the cell.

Xcalibur highlights the cell with a gray border. Click on its pin icon if you want to fix it as the active cell. Cursor actions in an active cell cause the cell to be scaled according to the dimensions of the dragged line or area (see Table 3-2, Cursor Actions In Active and Unpinned Cells, pp.110).

Table 3-2 Cursor Actions In Active and Unpinned Cells

Cursor Action	Effect
Drag parallel to X-axis	Rescale graph showing selected X range only, same Y range
Drag parallel to Y-axis	Rescale graph showing selected Y range only, same X range
Dragged area	Rescale graph showing both the selected X and Y ranges

The same actions in the unpinned or inactive cell have a very different effect. In this case, the cursor actions affect the active, pinned cell (see Table 3-3, Cursor Actions In An Inactive Or Unpinned Cell, pp.110).

Table 3-3 Cursor Actions In An Inactive Or Unpinned Cell

Pinned cell	Cursor action	Effect
spectrum	Click in a chromatogram cell	The spectrum cell displays the mass spectrum at that retention time.
spectrum	Drag across a time range in a chromatogram cell	The spectrum cell displays the averaged mass spectrum from that retention time range.
chromatogram	Click in a spectrum cell	The chromatogram cell displays the mass chromatogram of the selected mass. If the Plot Type is TIC or Base Peak, it is changed to Mass Range.
chromatogram	Drag across an *m/z* range in a spectrum cell	The chromatogram cell displays the mass range chromatogram of the selected mass range. If the Plot Type is TIC or Base Peak, it is changed to Mass Range.

Important points to note are:

- The cursor action is always applied to the pinned cell
- Within an active cell, cursor actions rescale the plot

Using Qual Browser you can answer the question, *"Does this sample contain compound 'X'? X* being the octafluoronaphthalene *(OFN)* you used in the previous topic. Then we will determine the peak height, area, and signal-to-noise ratio.

In this example, we used the **\Xcalibur\Data\100pgEI.raw** file you just acquired (*Step 6, Make the injection and start the GC., pp. 3-108*), which has a retention time of approximately 3.7-min. If you did not acquire this sample, you can use the sample file located in **Xcalibur\Data\Factory Test Data\ 100pgEI.raw**.

Let's assume that you have previously carried out a GC run with a pure sample of the compound you are interested in, compound '*X*', and consequently you know the retention time.

1 Open Qual Browser and select the *.raw file that contains the data for the sample you want to work on.

a. Select **Qual Browser** from the Home page.

b. Choose **File | Open** and navigate to **Xcalibur\Data** and open the sample file **100pgEI.raw** or a ***.raw** file containing the data you're interested in.

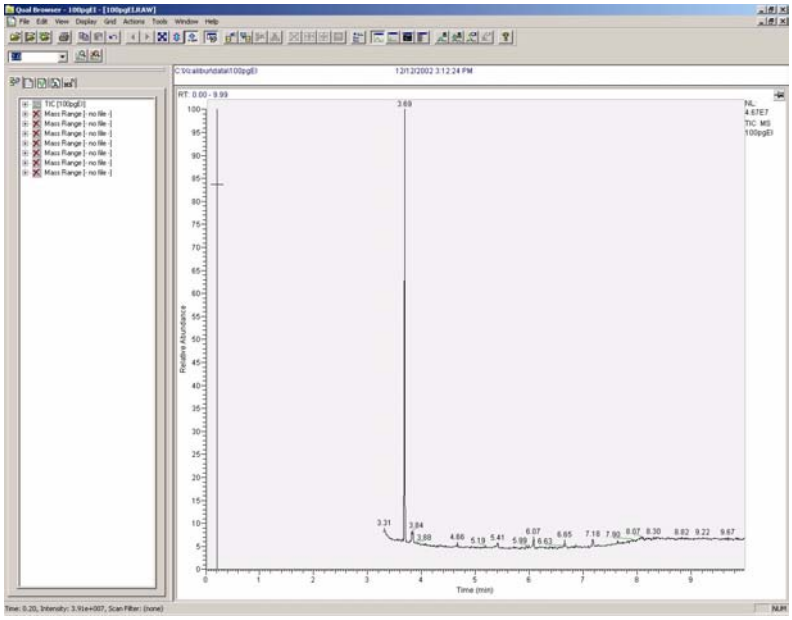


Figure 3-13 100pgEI.raw

Don't be surprised if the Qual Browser window looks different than Figure 3-13. The default layout can be changed and saved.

2 Display a **Chromatogram** and a **Mass Spectrum** in the same window.

If you want to identify the compound that is eluting at approximately 3.69-min, look at the mass spectrum recorded at that time in the chromatogram.

a. Click the **Insert Cell** toolbar button to display a new cell containing the same information as the existing cell.

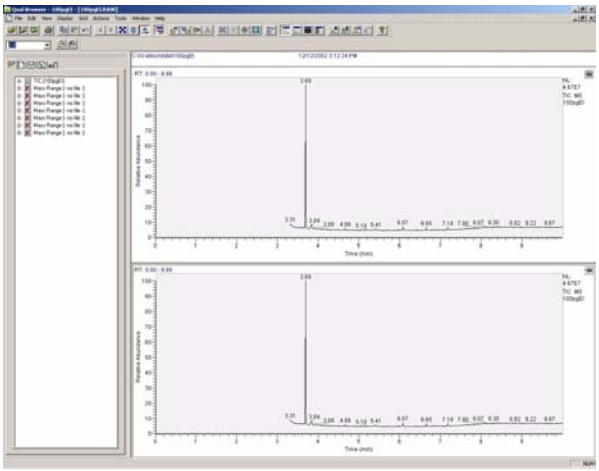


Figure 3-14 Displaying Two Chromatograms

b. Select the lower cell and then right-click your mouse to display the context sensitive menu.

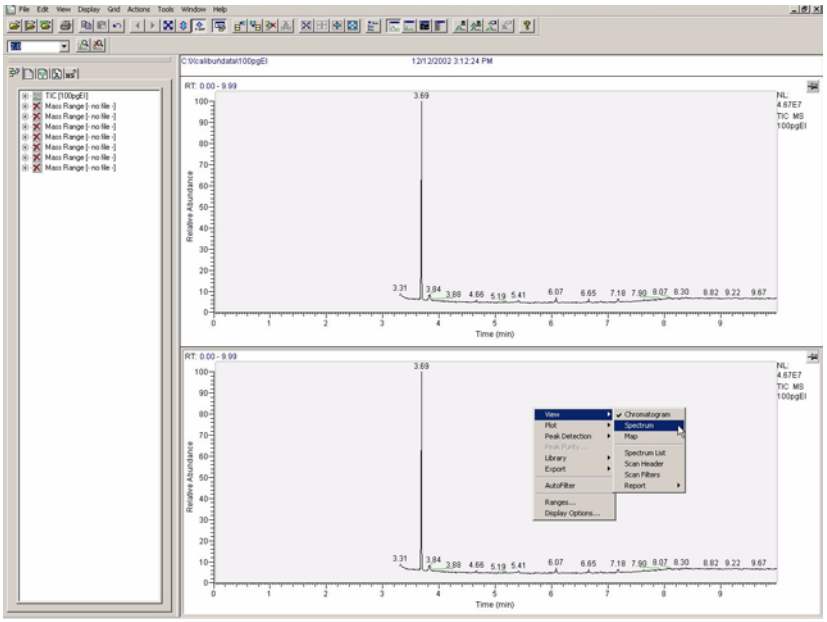


Figure 3-15 Qual Browser Chromatogram Context Menu

c. Choose **View | Spectrum** from this menu to change the view from chromatogram to spectrum. The lower cell now displays spectral data.

Notice that the lower cell now displays a graph of **Relative Abundance vs.** *m/z*; that is, a mass spectrum. Initially, you'll see the first mass spectrum.

3 Zoom in on the **Time Period of Interest**.

Although the full TIC chromatogram is currently displayed in the upper cell, only the retention time around three or four minutes is of particular interest. Therefore, zoom in on that part of the chromatogram and view it in more detail.

There are two ways of doing this; using **Mouse** functions or using **Range** functions.

Using Mouse Functions

- **Click the pin icon** that is located in the top right corner of the chromatogram cell so it is green (pinned).
- **Position the cursor at the starting time** you are interested in, at a position close to or on the baseline.
- **Click and drag** the cursor parallel to the time axis to the end of the period required. Notice that a line is drawn on the screen as you do this. As the cell redisplays, notice only that part of the chromatogram within the line you have just drawn.

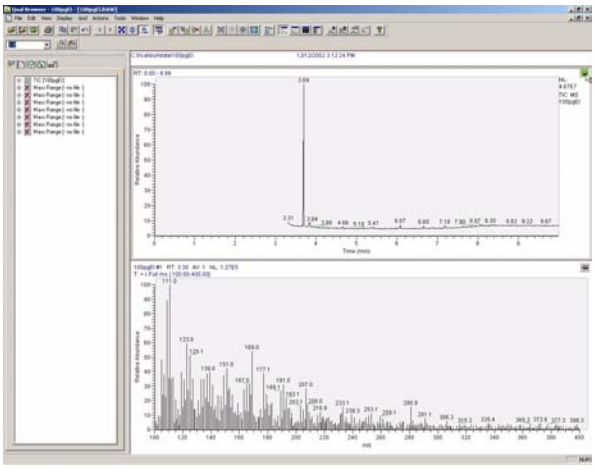


Figure 3-16 Chromatogram Using Mouse Functions

Using Range Functions

- Right-click on the chromatogram to display the context-sensitive menu and choose **Ranges** to display the **Chromatogram Ranges screen**.
- Enter the time range of interested in the **Time Range field** such 3.4 to 4.0-min.

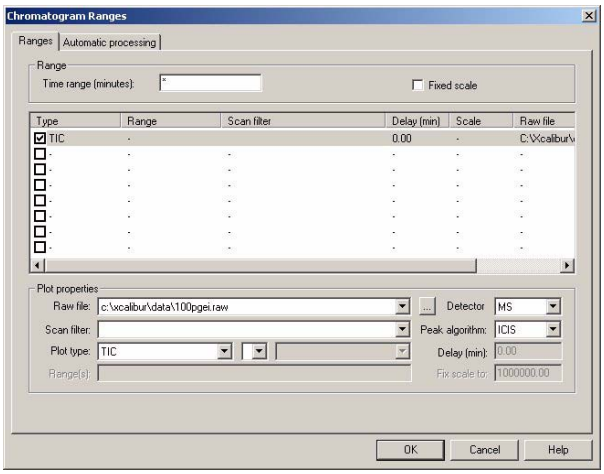


Figure 3-17 Chromatogram Ranges Screen Using Range Functions

- Click **OK** to return to **Qual Browser**.

4

View the mass spectrum for a specific chromatogram retention time.

Currently, a selected chromatogram time period is displayed in one cell and a mass spectrum in another cell. This step describes how to select a particular retention time on the chromatogram and view the corresponding mass spectrum.

a. Locate the pin icon shown in the top right corner of the mass spectrum cell and click on it. The icon changes from **unpinned**  to **pinned**  .

b. **Move the cursor over the chromatogram** until it is positioned at the retention time you are interested in and **click on it** to view the mass spectrum for the desired retention time.

5 Subtract the **Background Contamination** from the spectrum.

Peaks eluting from a GC column can contain low levels of contamination due to material bleeding from the matrix of the column. This material will have its own mass spectrum that will be superimposed on that of the sample compound. The mass spectrum of a chromatogram peak can therefore be made up of mass peaks resulting from the sample compound plus mass peaks resulting from background contamination.Therefore, to obtain a mass spectrum for the compound alone (that can be used in a library search), subtract the mass spectrum of the background contamination.

a. Make sure that the mass spectrum cell is pinned.

b. Choose **Actions | Subtract Spectra | 2 Ranges**.

You can choose between subtracting the background from either one side of the peak you are interested in or from both sides. To get the best results you will usually want to subtract from both sides, so choose two Ranges.

c. **Move the cursor** into the chromatogram cell and position it slightly to one side of the peak you are interested in.

d. **Click and drag the cursor away from the peak**, making sure to stop before reaching the next discernible peak on the chromatogram. Notice a line drawn on the screen as you do this.

e. Repeat **to mark the opposite side of the peak**.

In general, after background subtraction, most of the larger mass peaks in the mass spectrum will be unchanged; these are "true" peaks, due to the sample compound. Common contamination peaks are at *m/z* 207, and 281. The removal of such contamination peaks will help you to obtain a more accurate result from a library search.

6 Use the spectrum to perform a **Library Search.**

When you have a clean mass spectrum, you can use it to search the NIST library, to determine the nature of the compound.

a. Right-click in the mass spectrum cell and choose **Library | Search** from the context-menu.

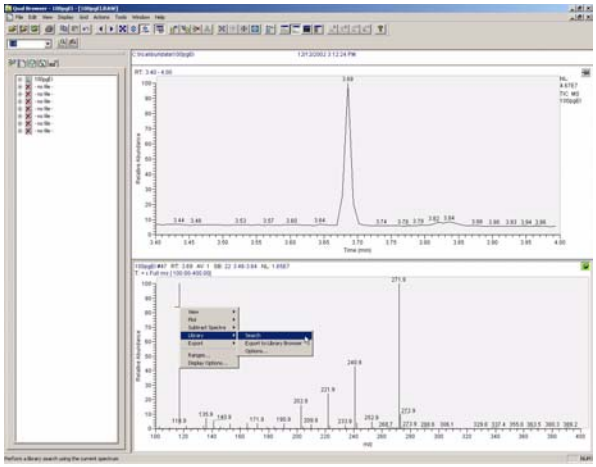
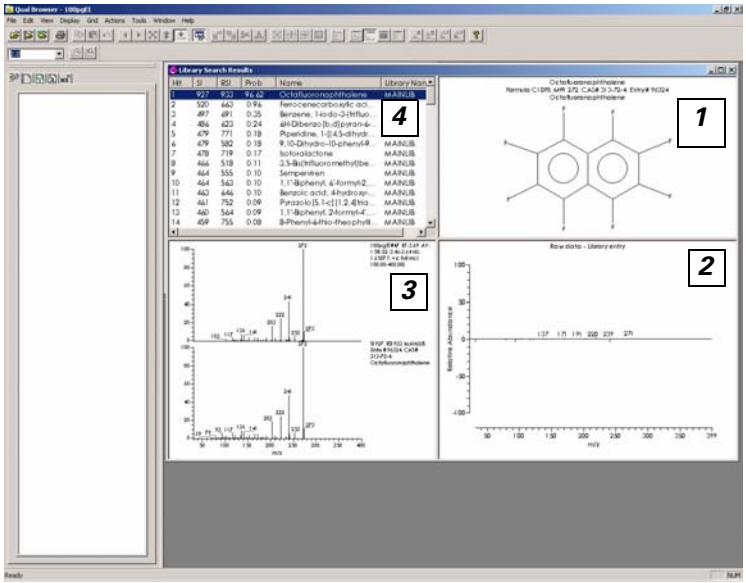


Figure 3-18 Library Search Pull-down Menu

b. If you have the optional NIST library installed look for the results:



1	Chemical Structure Cell	**3**	Library Spectrum Cell
2	Difference Spectrum Cell	**4**	Hit List Cell

Figure 3-19 Library Search Results window

c. Look in the hit list cell (**4**) for the most likely compounds, in decreasing order of match probability. If octafluoronaphthalene is not one of the top hits, you may not have the optional NIST

library. Check which libraries are available for search by selecting the **Library Options** pull-down menu.

For each compound in the hit list, Xcalibur displays a chemical structure (**1**), a library spectrum (**3**) to compare with your spectrum, and a difference spectrum (**2**); that is, the library spectrum subtracted from your spectrum.

3.5 Analyzing the Data: Compounds With a Known Ion Pattern

NOTE: See the online Help for additional instruction or see: Getting Productive: Qualitative Analysis, Getting Productive: Quantitative Analysis, Getting Productive: Processing Setup and Analysis of Quantitation Data, Getting Productive: Creating and Searching Libraries, and Getting Productive: Designing and Generating Custom Reports with Merlin.

Using Qual Browser you can answer the question, *"Does this sample contain compound "x"? X* being the octafluoronaphthalene *(OFN)* you used in the *3.3 Running Methods, pp. 102*. Then we will determine the peak height, area, and signal-to-noise ratio.

In this example, we used the **Xcalibur\Data\100pgEI.raw** file you just acquired (*Step 6, Make the injection and start the GC., pp. 3-108*), which has a retention time of approximately 3.7-min.

In this example, let's assume that you already have some knowledge of the mass spectrum of the compound of interest, compound 'X', and consequently you know the characteristic ions present in its mass spectrum. For the purposes of this example, *m/z* 272 represents an ion, perhaps the molecular ion of the compound, and therefore this ion represents a "marker" for the compound.

1 Open Qual Browser.

a. Set up the cells so you have only one cell, a chromatogram. You can delete other cells by pinning them and selecting **Delete Grid Row**  from the toolbar.

b. Reset the time range to the full range by selecting **Zoom Reset**  from the toolbar.

2 Add a mass range chromatogram.

a. Select the chromatogram cell and then right-click your mouse to display the context sensitive menu.

b. Choose **Ranges** from this menu to display the **Chromatogram Ranges screen**.

c. Add another plot by clicking the check box under **Type**.

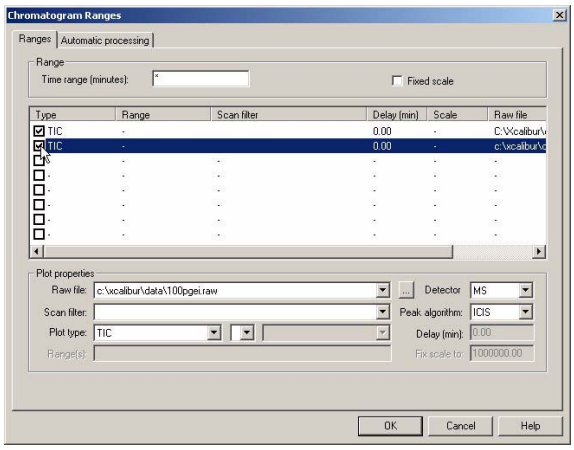


Figure 3-20 Qual Browser—Chromatogram 1

d. Select **Mass Range** from the **Plot Type** field.

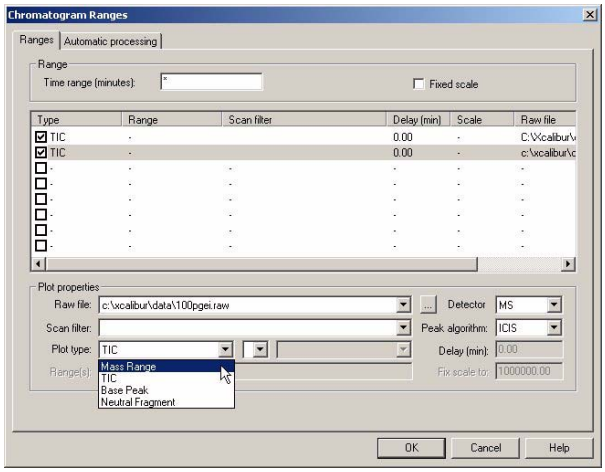


Figure 3-21 Qual Browser—Chromatogram 2

e. Enter the mass range in the **Range(s) field**. In this example, enter **272**.

f. Click **OK** to return to **Qual Browser**, where the chromatogram cell now shows a mass TIC and a chromatogram for the selected mass, *m/z* 272.

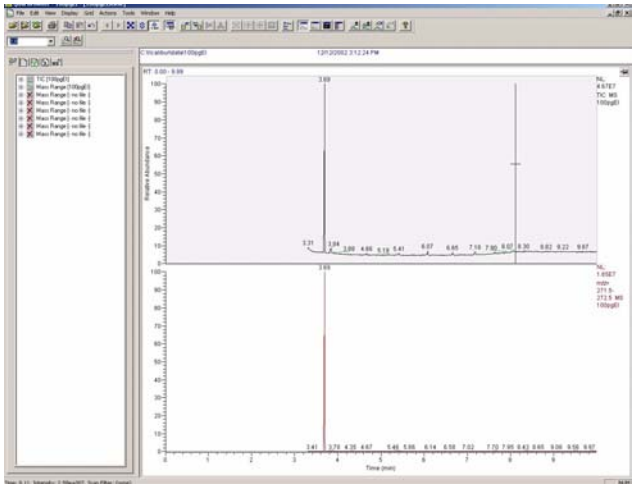


Figure 3-22 Displaying a TIC And Mass Range Chromatogram

The result in this example shows that the peak at retention time 3.69-min is of particular interest, because it includes *m/z* 272, the molecular ion from octafluoronaphthalene.

3 View the mass spectrum.

Having identified a particular retention time of interest, you can investigate the peak further by looking at the mass spectrum. Follow the same procedure you did in the previous section for adding a spectrum cell.

3.6 Analyzing the Data: Peak Height and Area

In a chromatogram, the area of an isolated peak is directly proportional to the amount of material present; for peaks that overlap, height may give a more accurate determination. Determining the area and/or height of a peak is therefore an important feature of quantitation.

1 **Zoom in** on the part of the chromatogram that is of particular interest (3.4–4.0-min.)

a. **Right-click on the chromatogram** and choose **Peak Detection | Toggle Detection in All Plots** from the context menu. Individual peaks are shown shaded and the extent of each peak shown by blocks on the baseline.

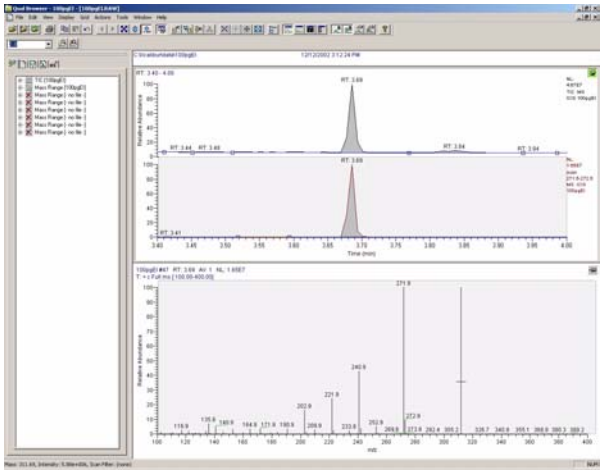


Figure 3-23 Qual Browser—Peak Detection

b. **Right-click on the chromatogram** and choose **Display Options** to display the **Display Options screen**.

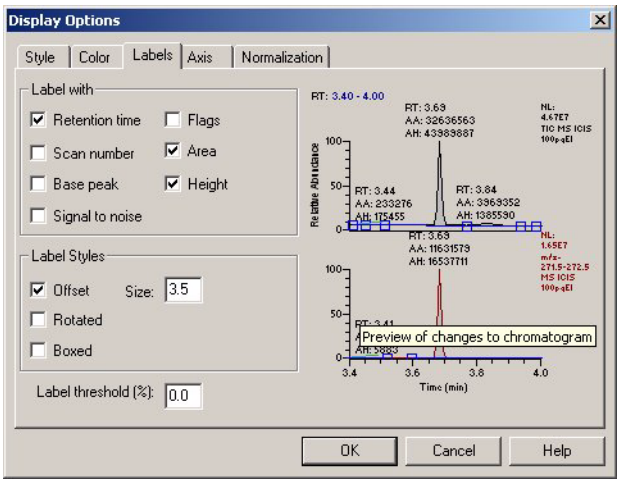


Figure 3-24 Qual Browser—Labels

c. Select the **Labels tab**.

d. Select both the **Area** and **Height check boxes**.

e. Click on **OK** to return to the chromatogram.

3.7 Analyzing the Data: Signal-to-Noise Ratio

The ***signal-to-noise ratio*** is a good measure of the performance of the system, because it defines the smallest amounts of compound that can be detected and quantified.

If the signal-to-noise ratio is low, true peaks (signals) resulting from the presence of very small amounts of material can be lost in the background noise.

Low signal-to-noise ratios can result from:

- Poor column quality
- A dirty ion source
- Poor quality carrier gas
- Inadequate sample cleanup (extraction)
- Poor chromatography techniques

There are two ways to determine the Signal-to-noise ratio of a peak. The first uses Qual Browser and the second uses the Signal-to-Noise Calculator. Note that in Xcalibur version 1.3 and earlier, the Qual Browser method is not always accurate. This is because you cannot select the region of the chromatogram from which to calculate noise.

Using Qual Browser...

1 Display the Signal-to-noise label on the chromatogram.

a. Using Xcalibur, right-click on the chromatogram and choose **Display Options** from the context menu to display the **Display Options screen**.

b. Select the **Labels tab**.

c. Select the **Signal-to-noise** checkbox.

d. Click on **OK** to return to the chromatogram.

2 Choose the **baseline noise range**.

a. Click on the **Peak Detection Settings** tab  in the **Info bar**.

b. Pin the chromatogram cell by clicking in the top right corner.

c. Click on the chromatogram plot on which you wish to measure the Signal-to-noise. It will turn grey.

d. Check **Manual Noise Region** in the **Info bar**.

e. Enter a **noise range** near the peak such 4.0–5.0-min. Avoid choosing a region with a peak in it.

f. Choose **Apply**. The **RMS** (**root mean square**) Signal-to-noise is calculated and displayed.The noise range you chose is shown by a red line in the chromatogram.

Using the Signal-to-Noise Calculator...

1 Use the Signal-to-noise Calculator.

Alternatively, you can use the Signal-to-noise calculator, which is provided with all versions of Xcalibur.

a. Run the **Signal-to-noise Calculator** by double-clicking the icon the desktop. You can also run it from **\Xcalibur\System\Programs\Signoise.exe** if the icon is not available.

b. Choose **File | Open** and select the **100pgEI.raw** file.

c. Select **Chromatogram | Ranges**.

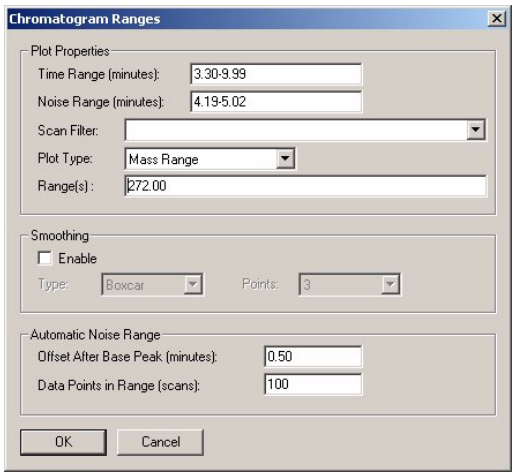


Figure 3-25 Signal-to-noise Calculator

d. Select **Mass Range** for **Plot Type**.

e. Enter **272** for **Range(s)**.

f. Select **OK** to display the Signal-to-noise ratio.

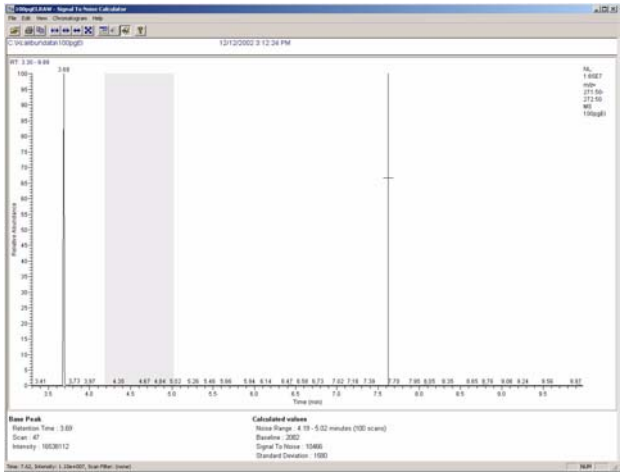


Figure 3-26 Signal-to-noise Calculator

g. Select different regions of the baseline for the noise range if necessary. Choose **Chromatogram | Automatic Noise Range** to disable the automatic range made. Choose **Chromatogram | Specify Noise Range** and click and drag the noise range you want (for example, 4.0–5.0 min). Avoid choosing a region with a peak in it.

3.8 Automating Processing

When you're ready to automate your processing please read the following Xcalibur books for instructions:

- *Getting Productive: Quantitative Analysis*
- *Getting Productive: Processing Setup and the Analysis Of Quantitation Data*
- *Getting Productive: Creating and Generating Custom Reports with Merlin*

Appendix A Preinstallation Checklists

In This Chapter

This appendix provides checklists for both the *TRACE DSQ or FOCUS DSQ* preinstallation and the field service installation. Do not write on the copies presented in this chapter. Rather, make duplicate copies whenever you need a checklist or report.

Analyze • Detect • Measure • Control™

A.1 Preinstallation Checklist

The customer should have met all the requirements listed on this checklist prior to your arrival. Ask the customer to give you their copy of the Preinstallation Checklist and check each item. Detailed information matching the items on this checklist are in your *Preinstallation Guide*. *Preinstallation Guides* are given to the customer by the sales person taking the order.

- ❑ **1. Entrance Requirements:** delivery doors are at least 102 cm (40 in.) wide?
- ❑ **2. Workbench Space, Weight and Stability Requirements:** workbench is at least 2 m (6 ft) across, supports a 119 kg (262 lb) load, and is sturdy and free from vibration?
- ❑ **3. Lighting Requirements:** work area is properly lit?
- ❑ **4. Power Requirements:** acceptable power source available and nearby?
- ❑ **5. Wall Outlets & Voltage Requirements**: wall outlets meet system voltage current specifications?
- ❑ **6. Environment Requirements:**
  - ❑ **a. Temperature:** room temperature is 15-31 °C (59-88 °F)?
  - ❑ **b. Humidity**: relative humidity is between 40 and 80%, with no condensation?
  - ❑ **c. Install** a temperature and humidity monitor
  - ❑ **d. Particulate Matter:** air quality is free of excess particulate matter?
  - ❑ **e. Electrostatic Discharge:** work area is free of electrostatic discharge?
  - ❑ **f. Exhaust System:** suitable exhaust system available?
- ❑ **7. Gas Equipment Requirements:** is the necessary gas equipment available?
- ❑ **8. Solvent Requirements:** are the recommended solvents available?
- ❑ **9. Telephone Requirements:** a telephone is located near the workbench?

A.2 Installation Checklist

Two-Day Schedule At A Glance	
	*30 Days before* ❑ Call customer: ❑ Is Preinstallation checklist complete? ❑ Are there any questions? ❑ Is there anything additional I need to bring? ❑ Step 1.1 Get the right tools
	*Day 1* **Morning** ❑ Step 1.2 Verify site conforms to specifications ❑ Step 1.3 Unpack the instruments **Afternoon** ❑ Step 1.4 Connect the Gas lines ❑ Step 1.5 Install the Data System ❑ Step 1.6 Install the GC ❑ Step 1.7 Install the Mass Spectrometer ❑ Step 1.8 Start the Instrument ❑ Step 1.9 Confirm Readbacks ❑ Step 1.10 Run Diagnostics ❑ Step 1.11 Check RF DIP Calibration ❑ Step 1.12 Check RF Gain Calibration ❑ Step 1.13 Check Air/Water Spectrum ❑ Step 1.14 Leak check ❑ Step 1.15 Check the Initial Tune ❑ Step 1.16 Stabilize the instruments
	*Day 2* **Morning** ❑ Repeat steps 1-8 to 1.14 **Afternoon** ❑ Step 1.17 Run an EI Qualification Test ❑ Step 1.18 Run a CI Qualification Test ❑ Step 1.19 Install Upgrade Options ❑ Step 1.20 Provide basic training

Appendix B Additional Helps

In This Chapter

B.1 Assembling Swagelok Fittings

	**Tools Needed:** • Swagelok Fitting • Wrench set (open end), 5/16-in., 7/16-in., 1/2-in., 9/16-in.
	**Frequency:** Fixing leaks

Building Swagelok compression fittings and connecting tees and crossings are essential to a leak-free system.

A Swagelok Fitting is a compression fitting that has four components—a Swagelok Nut, a Back Ferrule, a Front Ferrule, and an Inlet.

The completed assembly is comprised of five-pieces when the Swagelok Ferrule and Nut assembly is affixed to Tubing. Two ferrules merge when the nut is tightened, forming a safe and leak-free seal between the tubing and the inlet body.

1 Place a Swagelok nut on the end of the tubing.

The threads on the nut should face the open end of the tubing as shown in *Figure B-1 Swagelok Ferrule and Nut Assembly*.

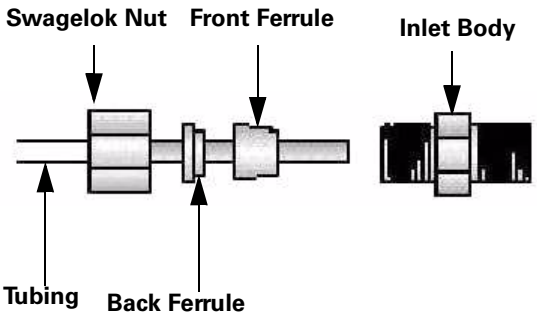


Figure B-1 Swagelok Ferrule and Nut Assembly

2 Place a Swagelok back ferrule over the tubing with the smaller side facing the open end of the tubing.

NOTE: Do **not** put tape on the threads of a Swagelok fitting.

3 Place a Swagelok front ferrule over the tubing with the smaller side facing the open end of the tubing.

4 Push the ferrules down into the Swagelok nut.

5 Insert the tubing into the Swagelok inlet as far as it will go as shown in *Figure B-2 Swagelok and Inlet Connection*.

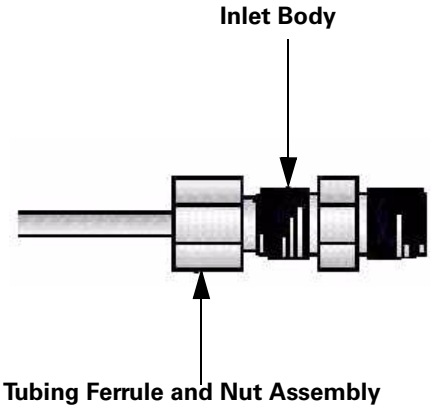


Figure B-2 Swagelok and Inlet Connection

6 Slide the nut over the inlet and tighten until finger-tight.

7 Pull the tubing out from the nut very slightly (1/16-in.)

8 While holding the inlet tight with a backup wrench, tighten the nut 3/4-turn past finger-tight as shown in *Figure B-3 Tightening Swagelok Fittings*.

You can make a mark on the nut before you tighten it. This helps ensure that you have turned the nut a 3/4-turn.

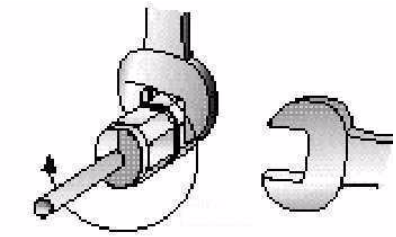


Figure B-3 Tightening Swagelok Fittings

B.2 Using a Swagelok Tee or Cross

	**Tools Needed:** • Ruler or measuring tape • Swagelok Fittings • Swagelok Plugs • Tubing Cutter
	**Frequency:** Fixing leaks

To use a single gas source for more than one inlet or detector module, you must use a Swagelok tee or cross to properly split the gas flow.

1 Cut the tubing.

With a tubing cutter, cut the tubing where the tee or cross will go.

2 Connect the tubing to the tee or cross with a Swagelok fitting.

Refer to instruction *B.1 Assembling Swagelok Fittings, pp. 134*.

3 Measure and cut.

Measure the distance from the tee or cross to the inlets or detectors, and cut the tubing in the appropriate lengths.

4 Connect the tubing to the tee or cross ends with Swagelok fittings.

5 Install **Swagelok Plugs**.

Install Swagelok Plugs instead of an Inlet Body on any open ends located on a tee or cross that are not connected with tubing.

B.3 Adjusting the RF Null

Use this procedure only if an RF Null adjustment is necessary. To adjust the RF Null you adjust the **R5** potentiometer located behind the **heatsink** of the **RF generator PCB**. This procedure should only be performed by factory trained Field Engineers.

WARNING
Personal Injury

Electrical Shock Hazard
This procedure requires the safety covers to be removed and an adjustment made inside while the instrument is running. This procedure should only be performed by factory trained Field Engineers.

CAUTION
Instrument Damage

Improperly adjusting the RF Null will permanently damage the RF system. This procedure should only be performed by factory trained Field Engineers.

1 Run the **Rod DC Test** in continuous mode.

a. Open the Tune window **Diagnostic screen** by selecting **Tune | Diagnostics | Run Tests** to display the **Diagnostics screen**.

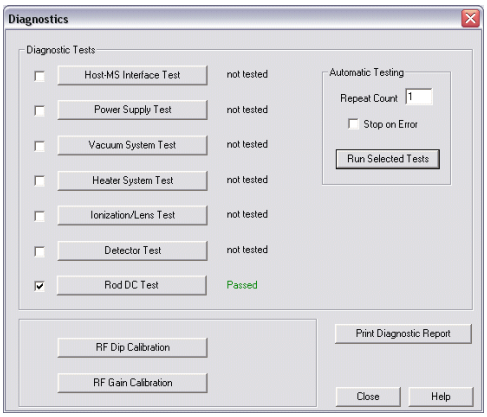


Figure B-4 Tune Diagnostic Screen

b. Uncheck all options except the **Rod DC Test**.

c. Select the **Rod DC Test button** to display the **Rod DC Test screen**.

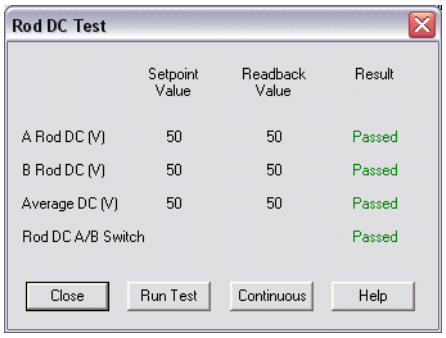


Figure B-5 Rod DC Test

d. Select the **Continuous button,** to run the test continuously.

2 Remove the Mass Spectrometer covers in this order: front, top, and left.

3 Adjust the **RF Null**, which is located behind the **heatsink** of the **RF generator PCB**.

a. Insert the potentiometer adjustment tool into the slotted screw of the **RF Null Adjustment**.

b. *Slowly* turn until the **A** and **B Rod DC values** are closest to the average **Rod DC readback** or 50 V.

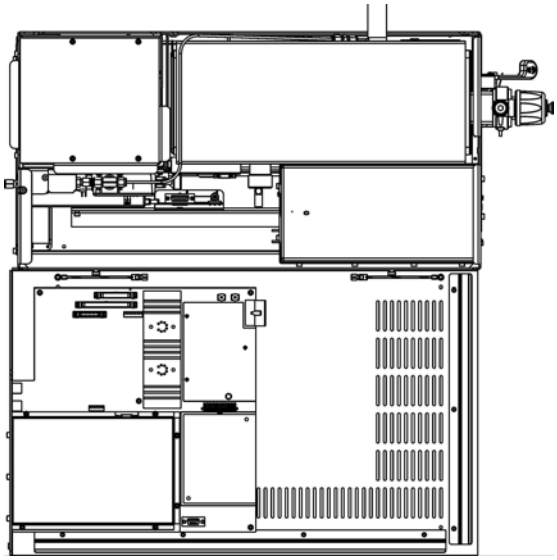
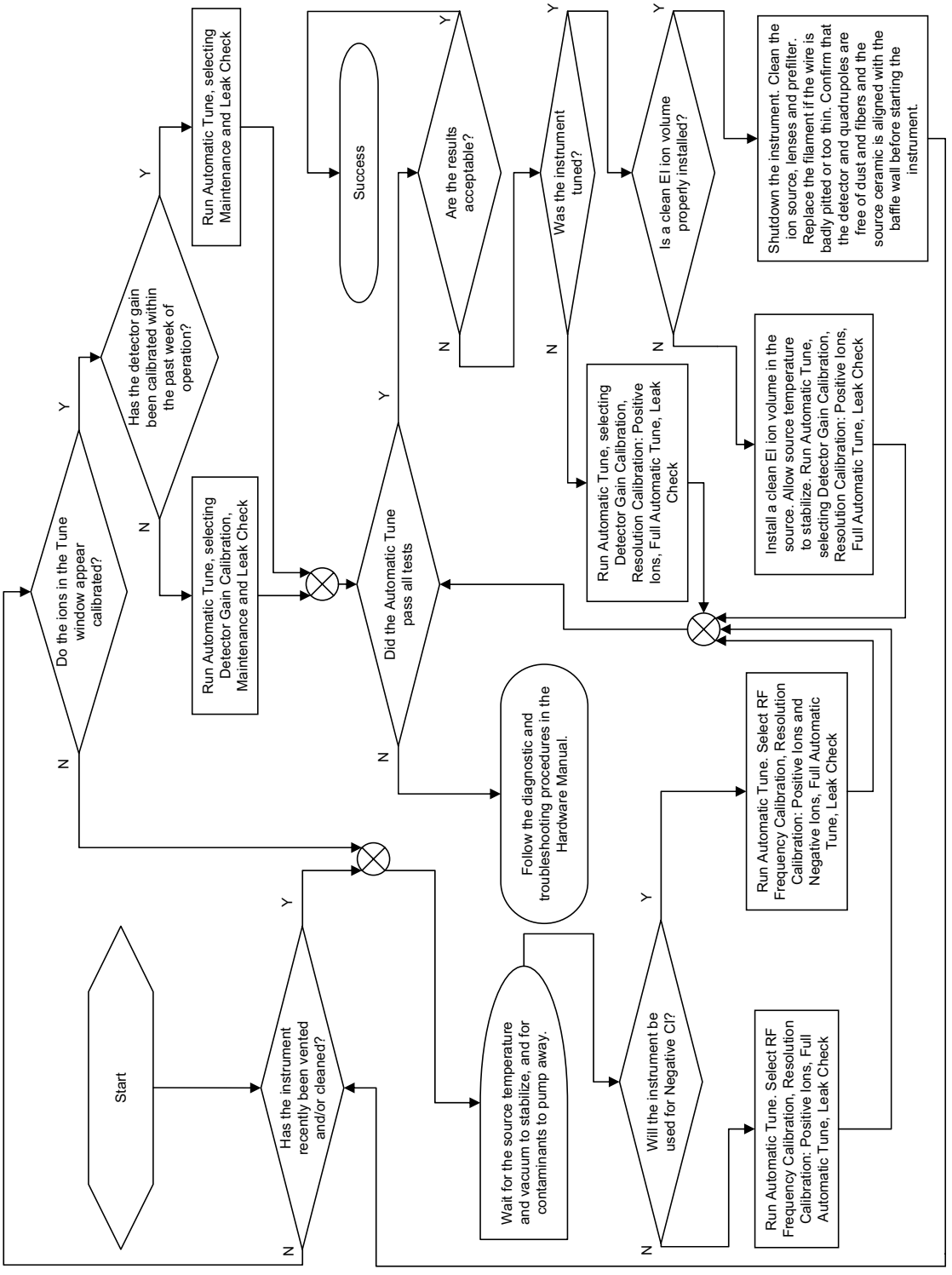


Figure B-6 RF Null Position

4 Replace the Mass Spectrometer covers in this order: left, top, and front.

B.4 Automatic Tune Flow Chart

Refer to this chart to determine when to use various tuning features.



B.5 Adjusting the RF Dip Calibration

Use this instruction to adjust the RF Dip Calibration readback, however, make sure you run this calibration **after** the heaters and vacuum have stabilized.

1 Make sure the **heaters** and **vacuum status readbacks** have stabilized.

2 Open the **Automatic Tune screen** from the Tune window.

a. Select **Tune | Automatic Tune** to display the **Automatic Tune screen**.

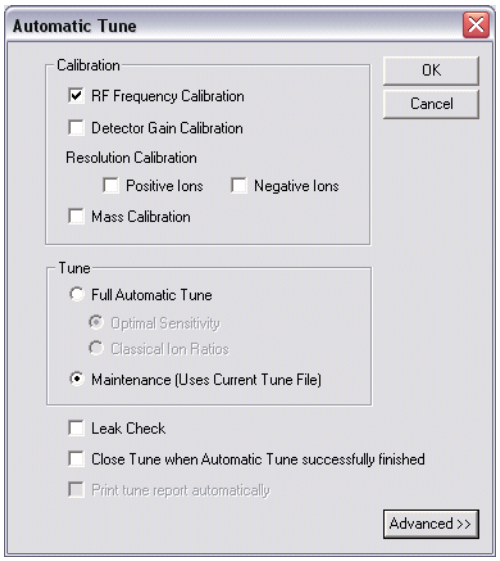


Figure B-7 Xcalibur 1.4 Automatic Tune Screen

b. Select the **RF Frequency Calibration** option.

c. Select the **Maintenance** option.

d. Uncheck all other options.

e. Click **OK** to adjust the **RF Dip Calibration** automatically and return to the **Tune window**.

B.6 Reinstalling the Data System

Use this procedure only if installing a new computer or replacing a hard drive.

1 Follow the instructions on the Xcalibur CD.

a. Insert the **Xcalibur CD** into your computer.This should automatically display the **Xcalibur Setup screen** (Figure B-8). If it doesn't, use windows to run the **XInstall.exe** from the CD.

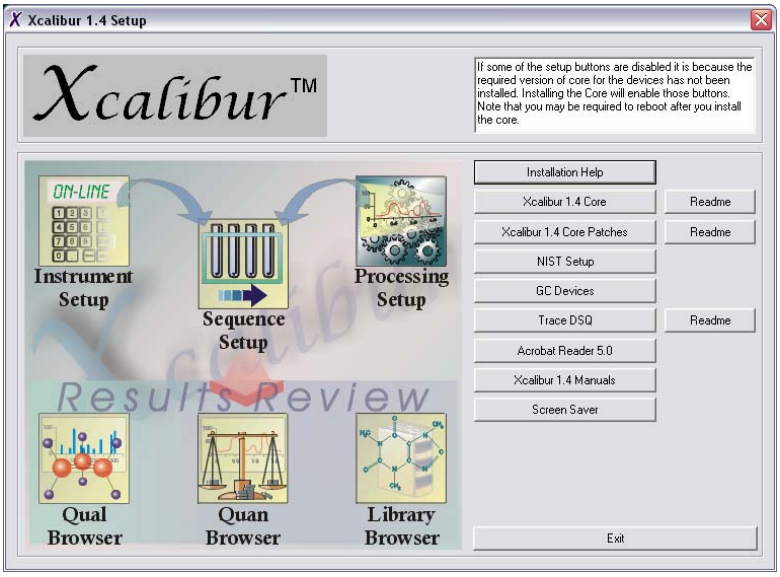


Figure B-8 *Xcalibur Data System* **Setup Screen**

b. Select the **Installation Help button** file to display the **Instructions.doc** file and then send it to your printer. Follow the instructions to install the data system software.

NOTE: Click the **Readme button** located on the Setup Screen to read important new information about this option.

2 Install any **optional upgrade software**.You should now install any optional software like libraries. Follow the instructions provided with these options.

3 Configure the **instruments**.

Follow the instructions listed in *2.2 Configuring Instruments for Use, pp. 84*.

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