

**Xcalibur**

# **Ion Trap and DSQ Series**

## **Direct Probe System User's Guide**

119327-0001 Revision G

April 29, 2008

© 2009 Thermo Fisher Scientific Inc. All rights reserved. Xcalibur™, DSQ, DSQ II, TRACE DSQ, TRACE GC, TRACE GC Ultra, FOCUS GC, PolarisQ, and ITQ are trademarks and/or product names of Thermo Fisher Scientific. Microsoft® is a registered trademark of Microsoft. Adobe® is a registered trademark of Adobe Systems Incorporated in the United States and/or other countries. Septum BTO® is a registered trademark of Chromatography Research Supplies, Inc. Swagelok® is a registered trademark of Swagelok Company. ETP is an SGE product. All other trademarks are the property of Thermo Fisher Scientific and its subsidiaries.

Ion Trap series refers to ITQ and PolarisQ instruments. DSQ series refers to all DSQ and DSQ II instruments. Information about the TRACE GC and FOCUS GC instruments is included in this document.

**INSTRUMENT USAGE:** Thermo Scientific systems operate safely and reliably under carefully controlled environmental conditions. If the equipment is used in a manner not specified by the manufacturer, the protections provided by the equipment may be impaired. 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.

Thermo Fisher Scientific Inc. provides this document to its customers with a product purchase to use in the product operation. This document is copyright protected and any reproduction of the whole or any part of this document is strictly prohibited, except with the written authorization of Thermo Fisher Scientific Inc.

The contents of this document are subject to change without notice. All technical information in this document is for reference purposes only. System configurations and specifications in this document supersede all previous information received by the purchaser.

**Thermo Fisher Scientific Inc. makes no representations that this document is complete, accurate or error-free and assumes no responsibility and will not be liable for any errors, omissions, damage or loss that might result from any use of this document, even if the information in the document is followed properly.**

This document is not part of any sales contract between Thermo Fisher Scientific Inc. and a purchaser. This document shall in no way govern or modify any Terms and Conditions of Sale, which Terms and Conditions of Sale shall govern all conflicting information between the two documents.

Release history: Rev G April 2008, Rev F August 2006, Rev E May 2005. Rev D March 2004. Rev C July 1999. Rev B May 1998. Rev A May 1997.

**For Research Use Only. Not regulated for medical or veterinary diagnostic use by U.S. Federal Drug Administration or other competent authorities.**



## Reader's Survey

### Ion Trap and DSQ Series Direct Probe System User's Guide, 119327-0001, Revision G

Free gift for your returned Reader's Survey. Please help us improve the quality of our documentation by completing and returning this survey. Circle one number for each statement:

	Strongly Agree	Agree	Neutral	Disagree	Strongly Disagree
The manual is well organized.	1	2	3	4	5
The manual is clearly written.	1	2	3	4	5
The manual contains all the information I need.	1	2	3	4	5
The instructions are easy to follow.	1	2	3	4	5
The instructions are complete.	1	2	3	4	5
The technical information is easy to understand.	1	2	3	4	5
Examples of operation are clear and useful.	1	2	3	4	5
The figures are helpful.	1	2	3	4	5
I was able to operate the system using this manual.	1	2	3	4	5

If not, please comment below. Attach additional sheets if necessary.


## Customer Registration Card

Register now...and receive all the privileges associated with being a Thermo Fisher Scientific product user including customer support, application reports, and technical reports.

### 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 \_\_\_\_\_

### MY PRIMARY JOB FUNCTION IS: (Check only one)

- ☐ Administration
- ☐ Lab management
- ☐ Operator
- ☐ Other \_\_\_\_\_

Name _____	Title _____
Company _____	
Address _____	
City/State _____	Postal Code _____
Country _____	
Telephone _____	Ext. _____
Serial Number _____	Date purchased _____

**Fold and mail or email to: [techpubsaustin@thermofisher.com](mailto:techpubsaustin@thermofisher.com)**



PLACE  
STAMP  
HERE

From \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**Editor, Technical Publications**  
Thermo Fisher Scientific SID GC GC/MS  
2215 GRAND AVENUE PKWY  
AUSTIN TX 78728-3812  
UNITED STATES OF AMERICA



## Regulatory Compliance

Thermo Fisher Scientific performs complete testing and evaluation of its products to ensure full compliance with applicable domestic and international regulations. When the system is delivered to you, it meets all pertinent electromagnetic compatibility (EMC) and safety standards as described below.

### EMC Directive 89/336/EEC

EMC compliance has been evaluated by Professional Testing.

- PolarisQ, ITQ, and Ion Trap Series standards: EMC EN 55011:1998 + EN 50082-1:1998, Safety EN 61010-1:1990 + A1:1992 + A2:1995
- DSQ standards: EMC EN 61326-1:1998 + A1:1998. Safety EN 61010-1:1990 + A1:1992 + A2:1995
- DSQ II standards: EMC EN 61326-1:1997 + A1:1998 + A2:2001. Safety EN 61010-1:2001
- Direct Probe Controller (DPC) standards: EMC EN 55011:1991 + EN 50082-1:1992. Safety EN 61010-1:1994

### Low Voltage Safety Compliance

This device complies with Low Voltage Directive 73/23/EEC and harmonized standard EN 61010-1:2001. Changes that you make to your system may void compliance with one or more of these EMC and safety standards. Changes to your system include replacing a part or adding components, options, or peripherals not specifically authorized and qualified by Thermo Fisher Scientific. To ensure continued compliance with EMC and safety standards, replacement parts and additional components, options, and peripherals must be ordered from Thermo Fisher Scientific or one of its authorized representatives.

### FCC Compliance Statement

*Certifications, 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. Detailed installation requirements are in the respective instrument's preinstallation guide.



**WARNING** Read and understand the various precautionary notes, signs, and symbols contained inside this manual pertaining to the safe use and operation of this product before using the device.

## WEEE Compliance

This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2002/96/EC. It is marked with the following symbol:



Thermo Fisher Scientific has contracted with one or more recycling or disposal companies in each European Union (EU) Member State, and these companies should dispose of or recycle this product. See [www.thermo.com/WEEERoHS](http://www.thermo.com/WEEERoHS) for further information on Thermo Fisher Scientific's compliance with these Directives and the recyclers in your country.

## WEEE Konformität

Dieses Produkt muss die EU Waste Electrical & Electronic Equipment (WEEE) Richtlinie 2002/96/EC erfüllen. Das Produkt ist durch folgendes Symbol gekennzeichnet:



Thermo Fisher Scientific hat Vereinbarungen mit Verwertungs-/Entsorgungsfirmen in allen EU-Mitgliedsstaaten getroffen, damit dieses Produkt durch diese Firmen wiederverwertet oder entsorgt werden kann. Mehr Information über die Einhaltung dieser Anweisungen durch Thermo Fisher Scientific, über die Verwerter, und weitere Hinweise, die nützlich sind, um die Produkte zu identifizieren, die unter diese RoHS Anweisung fallen, finden sie unter [www.thermo.com/WEEERoHS](http://www.thermo.com/WEEERoHS).

## Conformité DEEE

Ce produit doit être conforme à la directive européenne (2002/96/EC) des Déchets d'Equipements Electriques et Electroniques (DEEE). Il est marqué par le symbole suivant:



Thermo Fisher Scientific s'est associé avec une ou plusieurs compagnies de recyclage dans chaque état membre de l'union européenne et ce produit devrait être collecté ou recyclé par celles-ci. Davantage d'informations sur la conformité de Thermo Fisher Scientific à ces directives, les recycleurs dans votre pays et les informations sur les produits Thermo Fisher Scientific qui peuvent aider la détection des substances sujettes à la directive RoHS sont disponibles sur [www.thermo.com/WEEERoHS](http://www.thermo.com/WEEERoHS).

## Notice on Lifting and Handling of Thermo Scientific Instruments

For your safety, and in compliance with international regulations, the physical handling of this Thermo Fisher Scientific instrument *requires a team effort* to lift and/or move the instrument. This instrument is too heavy and/or bulky for one person alone to handle safely.

## Notice on the Proper Use of Thermo Scientific Instruments

In compliance with international regulations: Use of this instrument in a manner not specified by Thermo Fisher Scientific could impair any protection provided by the instrument.

## **Notice on the Susceptibility to Electromagnetic Transmissions**

Your instrument is designed to work in a controlled electromagnetic environment. Do not use radio frequency transmitters, such as mobile phones, in close proximity to the instrument.

For manufacturing location, see the label on the instrument.



# Contents

Reader's Survey . . . . .	iii
Customer Registration Card . . . . .	iii
<b>Preface . . . . .</b>	<b>xiii</b>
About Your System. . . . .	xiii
Power Ratings. . . . .	xiv
Safety Alerts and Special Notices. . . . .	xiv
Safety Symbols and Signal Words . . . . .	xv
Contacting Us . . . . .	xvi
To contact Technical Support. . . . .	xvi
To contact Customer Service for ordering information. . . . .	xvii
To suggest changes to documentation or to Instrument Help. . . . .	xvii
Related Documentation . . . . .	xvii
<b>Chapter 1   Using a Direct Insertion Probe (DIP) . . . . .</b>	<b>1</b>
How the Direct Probe System Works . . . . .	2
Analyzing Cholesterol with the Ion Trap Series. . . . .	4
Step 1: Connect the Direct Probe System . . . . .	5
Step 2: Change the Ion Volume . . . . .	7
Step 3: Deselect AS and GC Devices . . . . .	9
Step 4: Tune and Calibrate the Mass Spectrometer . . . . .	10
Step 5: Program the DPC . . . . .	10
To retrieve methods . . . . .	11
Step 6: Prepare the Sample . . . . .	12
Step 7: Create a Method File. . . . .	12
Step 8: Create a Sequence File. . . . .	13
Step 9: Insert the Probe Using Xcalibur. . . . .	15
Step 10: Remove the Probe Using Xcalibur . . . . .	17
Step 11: Analyze the Data File. . . . .	18
Analyzing Cholesterol with the DSQ Series. . . . .	20
Step 1: Connect the Direct Probe System . . . . .	21
Step 2: Change the Ion Volume . . . . .	23
Step 3: Deselect AS and GC Devices . . . . .	25
Step 4: Tune and Calibrate the Mass Spectrometer . . . . .	26
Step 5: Program the DPC . . . . .	26

To retrieve methods . . . . .	28
Step 6: Prepare the Sample . . . . .	28
Step 7: Create a Method File . . . . .	28
Step 8: Create a Sequence File . . . . .	29
Step 9: Insert the Probe Using Xcalibur . . . . .	31
Step 10: Remove the Probe Using Xcalibur . . . . .	33
Step 11: Analyze the Data File . . . . .	34
Loading DIP Samples . . . . .	35
Loading Liquid Samples . . . . .	36
Loading Solid Samples . . . . .	37
Cleaning the DIP . . . . .	37
Top Terms To Know . . . . .	38
DIP Modes . . . . .	38
Probe Shaft . . . . .	39
Probe Tip . . . . .	39
Sample Cups . . . . .	39
Top Safeguards to Know . . . . .	40
Hints . . . . .	40
<b>Chapter 2 Using a Direct Exposure Probe (DEP) . . . . .</b>	<b>43</b>
How the Direct Probe System Works . . . . .	44
Analyzing Cholesterol with the Ion Trap Series . . . . .	46
Step 1: Connect the Direct Probe System . . . . .	47
Step 2: Change the Ion Volume . . . . .	48
Step 3: Deselect AS and GC Devices . . . . .	50
Step 4: Tune and Calibrate the Mass Spectrometer . . . . .	51
Step 5: Program the DPC . . . . .	51
To retrieve methods . . . . .	52
Step 6: Prepare the Sample . . . . .	53
Step 7: Create a Method File . . . . .	54
Step 8: Create a Sequence File . . . . .	55
Step 9: Insert the Probe Using Xcalibur . . . . .	57
Step 10: Remove the Probe Using Xcalibur . . . . .	59
Step 11: Analyze the Data File . . . . .	60
Analyzing Cholesterol with the DSQ Series . . . . .	62
Step 1: Connect the Direct Probe System . . . . .	63
Step 2: Change the Ion Volume . . . . .	64
Step 3: Deselect AS and GC Devices . . . . .	66
Step 4: Tune and Calibrate the Mass Spectrometer . . . . .	67
Step 5: Program the DPC . . . . .	67

	To retrieve methods . . . . .	68
	Step 6: Prepare the Sample . . . . .	69
	Step 7: Create a Method File . . . . .	70
	Step 8: Create a Sequence File . . . . .	71
	Step 9: Insert the Probe Using Xcalibur . . . . .	72
	Step 10: Remove the Probe Using Xcalibur . . . . .	74
	Step 11: Analyze the Data File . . . . .	75
	Loading DEP Samples . . . . .	76
	Loading Liquid Samples . . . . .	77
	Loading Solid Samples . . . . .	78
	Cleaning the DEP . . . . .	78
	Top Terms to Know . . . . .	81
	DEP Modes . . . . .	81
	Probe Shaft . . . . .	82
	Probe Tip . . . . .	82
	Top Safeguards to Know . . . . .	82
	Hints . . . . .	83
<b>Chapter 3</b>	<b>Getting Help . . . . .</b>	<b>85</b>
	Who to Call . . . . .	86
	Technical Support . . . . .	86
	Customer Service . . . . .	86
	Spare Parts . . . . .	86
	Technical Publications Editor . . . . .	86
	Detuning the Instrument . . . . .	87
	Getting Spare Parts . . . . .	89
	<b>Abbreviations . . . . .</b>	<b>93</b>
	<b>Index . . . . .</b>	<b>97</b>



## Preface

This guide is written for users with minimal or no prior experience with the Ion Trap and DSQ Series or mass spectrometry. It also contains instructions on getting your instrument connected to the DPC and the sample probe of choice.

The Direct Probe Controller (DPC) is used to communicate information from a sample probe (DIP or DEP) to the mass spectrometer. The DPC also allows you to enter heating and data acquisition parameters to the mass spectrometer. Direct probe systems allow rapid sample screening for any application that do not require a gas chromatograph (GC) column separation. Another advantage to using a DIP or a DEP is to have a wide range of samples to volatilize directly into the ion source.

## Safety Alerts and Special Notices

Make sure you follow the precautionary notices presented in this guide. Safety and other special notices appear in boxes and include the following:



**WARNING** This is the general warning safety symbol and safety alert word to prevent actions that *could* cause personal injury. It highlights hazards to humans or the environment. Each WARNING safety alert is preceded with this safety symbol and another appropriate safety symbol (see “[Safety Symbols and Signal Words](#)” on [page xv](#).) Then it is followed with an appropriate safety precautionary message. When you see a safety alert on your instrument or in the publications, please carefully follow the safety instructions before proceeding.

**CAUTION** This is the safety alert word to prevent actions that *may* cause personal injury or instrument damage. We use it to highlight information necessary to prevent personal injury or damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal system performance. A CAUTION safety alert is always preceded with an appropriate safety symbol (see “[Safety Symbols and Signal Words](#)” on [page xv](#).) Then it is followed with an appropriate safety precautionary message. When you see a safety alert on your instrument or in the publications, please carefully follow the safety instructions before proceeding.

**IMPORTANT** Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.

**Note** Highlights information of general interest.

**Tip** Helpful information that can make a task easier.

## Safety Symbols and Signal Words

All safety symbols are followed by a **WARNING** or **CAUTION** safety alert word, which indicate the degree of risk for personal injury and/or instrument damage. Safety alert words are followed by the safety symbol's signal word, such as **CAUTION BURN HAZARD** or **WARNING BURN HAZARD**. A **WARNING** alerts to prevent improper actions that *could* cause personal injury. Whereas, a **CAUTION** alerts to prevent improper actions that *may* cause personal injury and/or instrument damage. When you see a safety alert on your instrument or in the publications, please carefully follow the safety instructions before proceeding. Safety symbols that may be found on your instrument and/or in this manual include the following:



**BURN HAZARD.** This symbol indicates a hot surface that *could* or *may* cause burn injuries.



**ELECTRICAL SHOCK HAZARD.** This symbol indicates that an electrical shock *could* or *may* occur.



**FIRE HAZARD.** This symbol indicates a risk of fire or flammability, or that fire/flammability damage *could* or *may* occur.



**FLAMMABLE GAS HAZARD.** This symbol alerts you to gases that are compressed, liquefied or dissolved under pressure and can ignite on contact with an ignition source. This symbol indicates this risk *could* or *may* cause physical injury.



**GLOVES REQUIRED.** This symbol indicates the user must wear gloves when performing the sequence or physical injury *could* or *may* occur.



**HAND AND CHEMICAL HAZARD.** This symbol indicates that chemical damage or physical injury *could* or *may* occur.



**INSTRUMENT DAMAGE.** This symbol indicates that damage to the instrument or module *may* occur. This damage may not be covered under the standard warranty.



**LIFTING HAZARD.** This symbol indicates two or more people are required to lift the object to prevent a physical injury, which *could* or *may* occur.



**MATERIAL AND EYE HAZARD.** This symbol indicates that eye damage *could* or *may* occur.

---



**RADIOACTIVE.** This symbol indicates the presence of radioactive material *could* or *may* occur.

---



**READ MANUAL.** This symbol alerts you to carefully read your instrument's operational instructions before usage to ensure your safety and the instrument's operational ability. Failing to carefully read the instructions *could* or *may* put you at risk for a physical injury.

---



**TOXIC SUBSTANCES HAZARD.** This symbol indicates that exposure to a toxic substance *will*, *could*, or *may* cause personal injury or death.

---



This is the general warning symbol that the ISO 3864-2 standard refers to as the general warning signal to prevent personal injury. It is a triangle with an exclamation mark that precedes the **WARNING** safety alert word. In the vocabulary of ANSI Z535 signage, this symbol indicates a possible personal injury hazard exists if the instrument is improperly used or if unsafe actions occur. We use this symbol and another appropriate safety symbol to alert to an imminent or potential hazard that *could cause personal injury*.

---

## Contacting Us

There are several ways to contact Thermo Fisher Scientific.

### ❖ To contact Technical Support

Phone	800-685-9535
Fax	561-688-8736
E-mail	<a href="mailto:TechSupport.C+MS@thermofisher.com">TechSupport.C+MS@thermofisher.com</a>
Knowledge base	<a href="http://www.thermokb.com">www.thermokb.com</a>

Find software updates and utilities to download at <http://mssupport.thermo.com>.

### ❖ To contact Customer Service for ordering information

Phone	800-532-4752
Fax	561-688-8731
Web site	<a href="http://www.thermo.com/com/cda/resources/resources_detail/1,,12512,00.html">http://www.thermo.com/com/cda/resources/resources_detail/1,,12512,00.html</a>

❖ **To suggest changes to documentation or to Instrument Help**

- Fill out a reader survey online at [www.thermo.com/lcms-techpubs](http://www.thermo.com/lcms-techpubs).
- Send an e-mail message to the Technical Publications Editor at [techpubsaustin@thermofisher.com](mailto:techpubsaustin@thermofisher.com).

## Related Documentation

In addition to this guide, Thermo Scientific provides the following documents, which are also available on a “Print-By-Request” basis:

*DSQ Document Set, PN 120156*

- DSQ Preinstallation Guide, PN 120156-0001
- DSQ Hardware Manual, PN 120156-0002
- DSQ User’s Guide, PN 120156-0003

*DSQ II Document Set, PN 120299*

- DSQ II Preinstallation Guide, PN 120299-0001
- DSQ II Hardware Manual, PN 120299-0002
- DSQ II User’s Guide, PN 120299-0003

*Ion Trap Series Document Set, PN 120425-0ALL*

- ITQ Preinstallation Guide, PN 120425-00PI
- Ion Trap Series Hardware Manual, PN 120425-00HW
- Ion Trap Series User’s Guide, PN 120425-USER

*PolarisQ Document Set, PN 120000*

- PolarisQ Preinstallation Guide, PN 120011
- PolarisQ Hardware Manual, PN 120010
- PolarisQ User’s Guide, PN 120014

*Instrument Help* is available from within the software.



## About Your System

Thermo Fisher Scientific systems provide the highest caliber gas chromatography/mass spectrometry (GC/MS) instrumentation available on today's market.

GC/MS represents a combination of two powerful analytical techniques: GC, which acts as a separation technique and MS, which acts as a detection technique. Complex mixtures of individual compounds can be injected into the GC, either manually or through the use of an optional autosampler, and then separated for presentation to the MS. The MS will then generate a mass spectrum of the GC eluent and its components, which can be used for qualitative identification as well as accurate and precise quantification of the individual compounds present in the sample.

**IMPORTANT** Thermo Fisher Scientific systems are designed to optimize both the separation and detection capabilities of GC/MS techniques and combine them in a synergistic fashion to provide high performance analytical capabilities for both research and routine applications. More information on the use of this system can be found in related documentation sources and through the provided contact information.



**WARNING** Thermo Fisher Scientific systems operate safely and reliably under carefully controlled environmental conditions. If the equipment is used in manner not specified by the manufacturer, the protections provided by the equipment may be impaired. If you maintain a system outside the specifications listed in this guide, failures of many types, including personal injury or death, may occur. The repair of instrument failures caused by operation in a manner not specified by the manufacturer is specifically excluded from the Standard Warranty and service contract coverage.

## Power Ratings

### *Mass Spectrometer (MS)*

- 120 V ac +6/-10%, 50/60 Hz, 15 A max
- 230 V ac  $\pm$ 10%, 50/60 Hz, 10 A max

### *Gas Chromatograph (GC)*

- 120 V ac +6/-10%, 50/60 Hz, 16 A max
- 230 V ac  $\pm$ 10%, 50/60 Hz, 16 A max

Detailed instrument specifications are in the Product Specification or Product Brochure.



# Using a Direct Insertion Probe (DIP)

This chapter explains how to use the DIP with either the Ion Trap or the DSQ series instruments.

## Contents

- “How the Direct Probe System Works” on page 2
- “Analyzing Cholesterol with the Ion Trap Series” on page 4
- “Analyzing Cholesterol with the DSQ Series” on page 20
- “Loading DIP Samples” on page 35
- “Loading Liquid Samples” on page 36
- “Loading Solid Samples” on page 37
- “Cleaning the DIP” on page 37
- “Top Terms To Know” on page 38
- “Top Safeguards to Know” on page 40
- “Hints” on page 40

The Direct Insertion Probe (DIP) is also referred to as a solids probe. It allows you to introduce solid or liquid samples directly into the ion source, which is used to rapidly analyze solids or trace components in solid matrices.

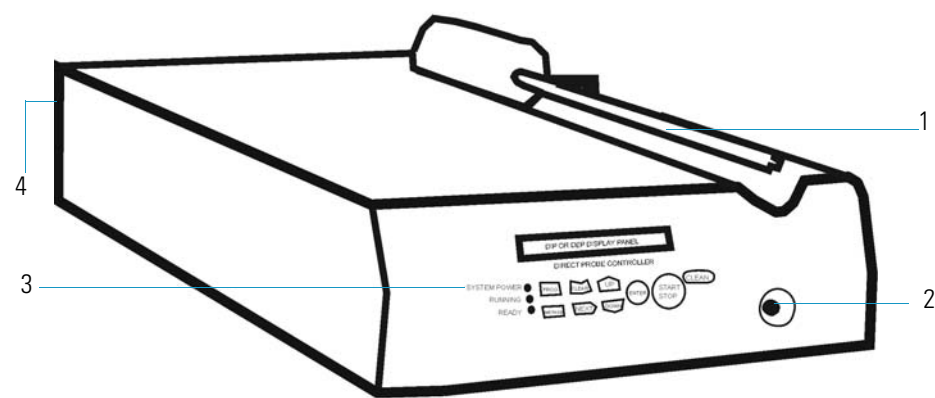
Typical samples may include:

- Forensic Samples
- Tissue Samples

## How the Direct Probe System Works

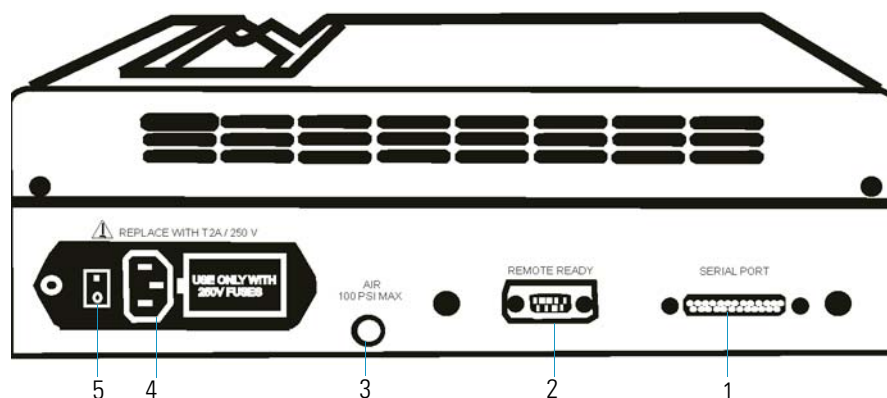
The Direct Probe System is a sample probe (Direct Insertion Probe [DIP] or Direct Exposure Probe [DEP]) connected to the Direct Probe Controller (DPC), which then connects to the mass spectrometer.

Figure 1. DPC and Sample Probe (Front View)



Item	Description	PN	Item	Description	PN
1	DIP or DEP Sample Probe	119300-ODIP or 119300-ODEP	3	DPC Keypad	
2	DPC External Cable to Probe	119303-0001	4	DPC Remote Start Cable to mass spectrometer ACCESSORY START, DB9F-DIN	119378-0032

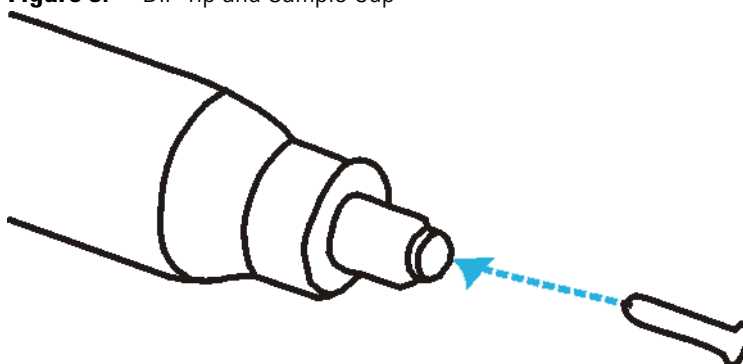
**Figure 2.** DPC (Back View)



1	Serial Port (not used)	4	Power Connection
2	REMOTE READY	5	Power Switch (0 is OFF, 1 is ON)
3	AIR (to cool Probe)		

The Direct Probe Controller (DPC) is the instrument used to communicate information from the sample probe (DIP or DEP) to the mass spectrometer. The DPC also allows you to enter heating and data acquisition parameters to the mass spectrometer. Direct probe systems allow rapid sample screening for any applications which do not require a gas chromatograph (GC) column separation. Another advantage to using a DIP or a DEP is to have a wide range of samples to volatilize directly into the ion source. [Figure 3](#) shows the DIP tip and sample cup.

**Figure 3.** DIP Tip and Sample Cup



## Analyzing Cholesterol with the Ion Trap Series

**Note** Ion Trap Series refers to both the PolarisQ and ITQ mass spectrometers.

Using an Ion Trap Series, analyzing sample involves these steps:

“Step 1: Connect the Direct Probe System” on page 5

“Step 2: Change the Ion Volume” on page 7

“Step 3: Deselect AS and GC Devices” on page 9

“Step 5: Program the DPC” on page 10

“Step 6: Prepare the Sample” on page 12

“Step 7: Create a Method File” on page 12

“Step 8: Create a Sequence File” on page 13

“Step 9: Insert the Probe Using Xcalibur” on page 15

“Step 10: Remove the Probe Using Xcalibur” on page 17

“Step 11: Analyze the Data File” on page 18

Steps 1 through 3 are considered the initial setup. After the initial setup you may skip steps 1 through 3 and proceed to steps 4 through 11.



#### Tools Needed

- Ion Trap Series mass spectrometer
- I/R tool and guide bar (included with mass spectrometer), PN 96000-60057
- Xcalibur Data System
- Operating System: Windows XP
- DPC Controller Assembly, PN 119300-0100
- DIP Kit (PN 119300-ODIP), includes the following:
  - (1) DIP/DEP Test Mix-10 mg/mL, PN 119343-0001
  - (1 pkg) DIP Sample Cups (25), PN 119329-0001
  - (2) Ion Volume Holders, PN 119324-0001



#### Frequency

As Needed

## Step 1: Connect the Direct Probe System

Before beginning your sample analysis, connect the DPC to the mass spectrometer and connect the sample probe to the DPC. Then make sure all connections are fully seated.

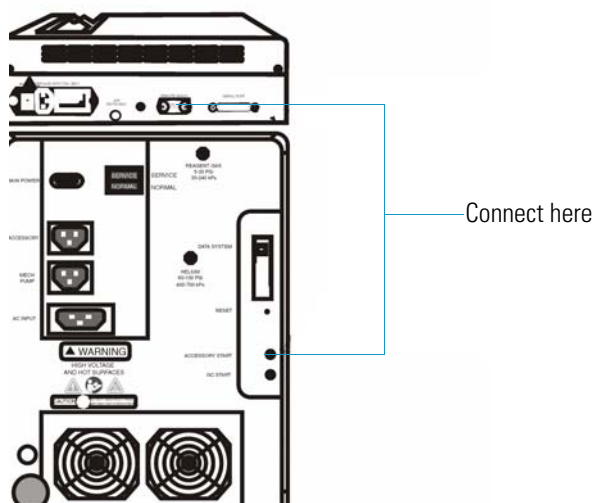
1. Place the DPC in a convenient location on top of or close to the mass spectrometer.
2. Read “[Top Safeguards to Know](#)” on [page 40](#) to be certain that you exercise all necessary safety precautions.



**WARNING ELECTRICAL SHOCK HAZARD.** Avoid exposing the DIP to electrical shock or electrical charging. Always plug the DIP cable into the DPC probe connector when performing EI or CI experiments, even if the probe is not heated. The DIP cable connection to the DPC provides electrical ground.

3. Connect the DPC to the mass spectrometer.

**Figure 4.** DPC to Mass Spectrometer (Back View)



---

Connect the REMOTE START CABLE to DPC REMOTE READY and the mass spectrometer ACCESSORY START

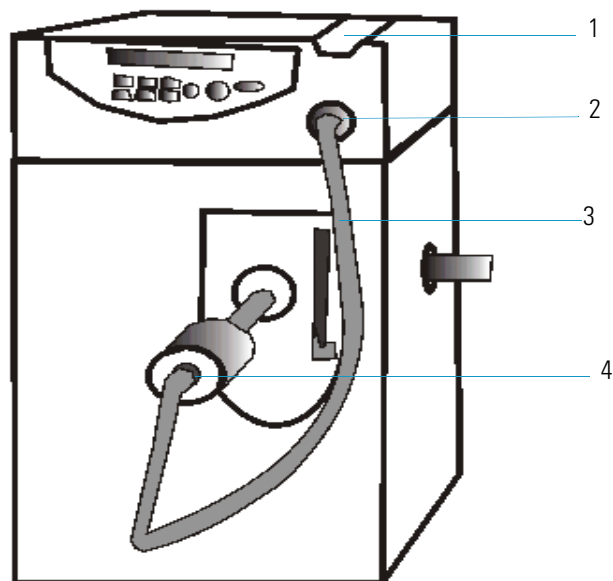
---

- a. Connect the DPC to the mass spectrometer by using the DPC REMOTE START cable to connect into the DPC REMOTE READY connection and the other end to the mass spectrometer ACCESSORY START connection.
- b. Plug the desired air source to use as a probe coolant into the DPC AIR connection.  
The air source should be dry air or nitrogen at no more than 100 psi. We recommend 80 psi for normal operation. The coolant is used with the DIP to rapidly cool the probe tip, which allows rapid sample through-put. This system is also designed for compressed air or nitrogen. However, it is not designed for subambient operation, and room temperature will affect the minimum probe tip temperature.
- c. Connect the power cord to the DPC power plug connection and the other end to the power source.
- d. Turn the DPC ON by switching the Power Switch to |. Xcalibur automatically detects the probe and activates the Insert/Probe settings on the Home page, Tune window, and Sequence Editor.



4. Connect the DPC to the Probe.

**Figure 5.** DPC to Probe Connections



Item	Description	PN	Item	Description	PN
1	DPC Cradle		3	DPC EXTERNAL Probe Cable	119303-0001
2	DPC Connection		4	Probe Connection	

- a. Plug one end of the DPC external cable to the DPC connection and the other end to the probe connection. Do this by matching the red dot located on the cable to the red dot located on the DPC and firmly pressing the cable in until it locks in position.
  - b. Place the probe in the built-in cradle located on top of the DPC. Either the DIP or DEP can safely rest in the built-in cradle when not in use.
5. Turn on the MS, DPC, and the Xcalibur Data System software.
  6. Confirm that the Insert/Remove Probe button displays on the Xcalibur Data System Home-Page.

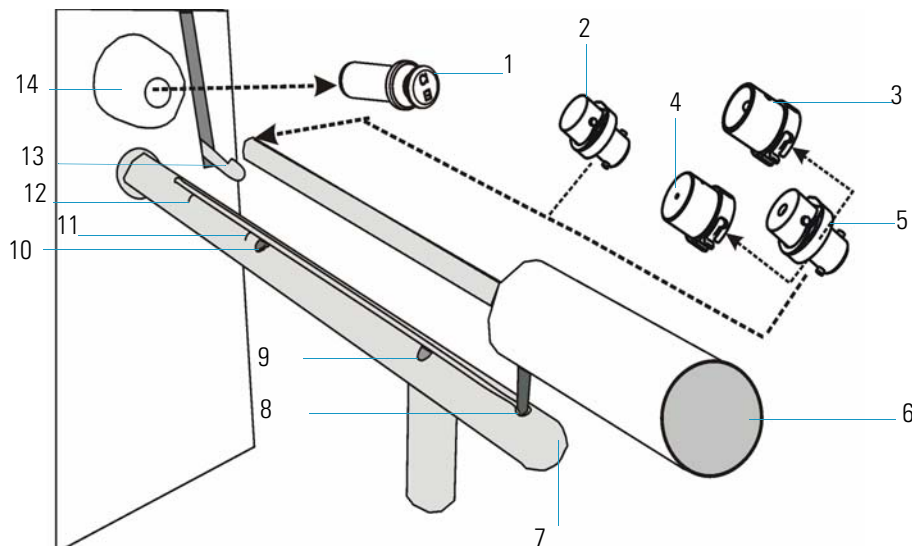
Typically, the software automatically detects if the probe is connected.

## Step 2: Change the Ion Volume

Samples can be analyzed in either EI or CI mode. Standard ion volumes are used using the probe ion volume holder included with your probe. The probe ion volume holder has a hole

that allows the sample from the probe tip to enter the ion volume as compared to the GC ion volume holder, which allows sample to enter through the mass spectrometer transfer line.

**Figure 6.** I/R Tools



Item	Description	PN	Item	Description	PN
1	Inlet Valve Plug	119273-0001	8	Guide Ball start position	
2	GC Ion Volume holder (standard)	70001-20532	9	1st Stop	
3	EI Ion Volume	119650-0220	10	2nd Stop	
4	CI Ion Volume	119650-0230	11	1st etching	
5	Probe Ion Volume Holder	119324-0001	12	2nd etching	
6	I/R Tool	96000-60057	13	Inlet Valve lever	
7	Guide Bar	119687-0010	14	Inlet Valve (vacuum interlock chamber)	

1. Confirm that you are at the Xcalibur Home-Page.

**Note** Carefully follow each step on the Insert Remove Probe screen.

2. Remove the current ion volume.
  - a. From the Xcalibur Home Page select the Insert/Remove Probe button (hint: it's located on the Status tabs) to display the Insert Remove Probe screen.
  - b. Select the Insert Probe tab.
  - c. Attach the probe ion volume holder on the I/R tool.

**Note** When prompted on the Insert Remove Probe screen, insert the I/R tool into the guide bar start position using the guide bar to enter the inlet valve chamber and remove the current ion volume.

- d. Insert the guide bar into the mass spectrometer as illustrated in [Figure 6](#).
  - e. Follow each step listed on the Insert Probe tab to insert the probe and remove the current ion volume.
  - f. Select the Remove Probe tab and carefully follow each step to remove the probe.
3. Install an EI or CI ion volume.
    - a. Attach either an EI ion volume or CI ion volume to the probe ion volume holder while making sure it is tightly secured.
    - b. From the Xcalibur Home Page select the Insert/Remove Probe button to display the Insert Remove Probe screen.
    - c. Use the Insert Probe tab instructions to insert the probe install a CI or EI ion volume.
    - d. Use the Remove Probe tab instructions to remove the probe.
    - e. Close the Insert Remove Probe screen and return to the Xcalibur Data System Home-Page.

### Step 3: Deselect AS and GC Devices

If you are running several probe runs, deselect the AS and GC instruments in the Xcalibur Instrument Configuration software. Configure for the mass spectrometer only when analyzing samples with the probe. Deselecting the AS and GC simplifies the methods and avoids waiting on GC equilibration time before every run. However, it is not necessary.

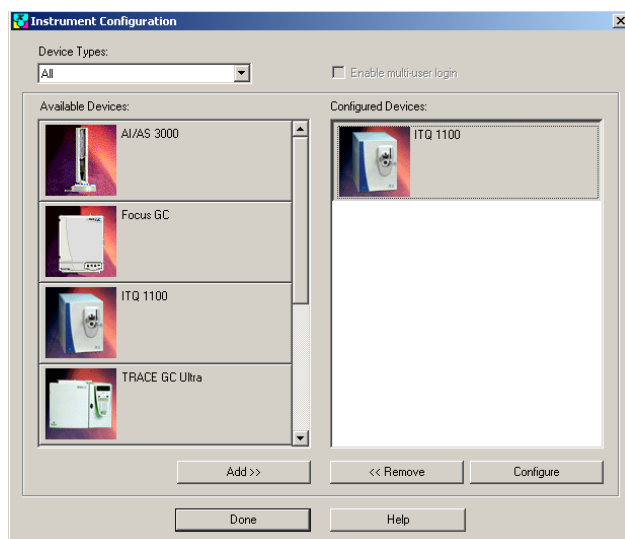
1. Start Xcalibur Instrument Configuration.



From the Windows desktop, select the Instrument Configuration shortcut to display the Instrument Configuration screen.

2. Remove the AS and GC devices from Configured Devices.

**Figure 7.** Instrument Configuration: Ion Trap Series



Select the item to be removed from the Configured Devices section and click the Remove button. We recommend using the Ion Trap and DSQ Series configuration only when analyzing samples with the probe.

3. Click Done to return to the Windows desktop.

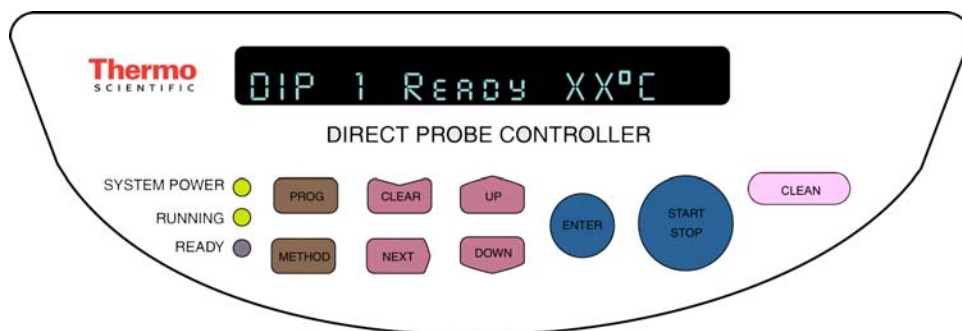
## Step 4: Tune and Calibrate the Mass Spectrometer

Refer to the Tune Online Help for instructions in determining if the instrument has been recently tuned and calibrated.

## Step 5: Program the DPC

1. Turn the DPC ON to display DIP 1 Ready.

**Figure 8.** DIP DPC Display Panel and Keypad



The DPC automatically detects if a DIP or DEP is connected, and will display the DIP or DEP series of menu controls for setting the temperature and for heating the probe tip.

2. Enter the initial temperature.
  - a. Press METHOD to display Initial Temp.
  - b. Press UP or DOWN until you see 50 °C.
3. Enter the initial time.
  - a. Press NEXT to display Initial Time.
  - b. Press UP or DOWN to display initial time of 60 seconds.
4. Enter the ramp 1 rate.
  - a. Press NEXT to display Ramp 1 Rate.
  - b. Press UP or DOWN to set the Ramp Rate to MAX. This ramps the probe tip ballistically at its maximum rate of approximately 150 °C/min.
5. Enter the ramp 1 temperature.
  - a. Press NEXT to display Ramp 1 Temp.
  - b. Press UP or DOWN until the temperature is 350 °C.
6. Enter the ramp 1 hold time.
  - a. Press NEXT to display Ramp 1 Hold.
  - b. Press UP or DOWN until the Ramp 1 Hold time displays 40 seconds.
7. Enter the ramp 2 rate.
  - a. Press NEXT to display Ramp 2 Rate.
  - b. Press UP or DOWN until the Ramp 2 Rate is OFF.
8. Return to DIP 1 Ready.

Press NEXT to display DIP 1 Ready.
9. Load the method.

Press PROG to display Load Method: X.
10. Save the method.
  - a. Press NEXT to display Save Method: X<sup>1</sup>.
  - b. Press ENTER to return to DIP 1 Ready.
  - c. Listen for the DPC to beep three times to indicate that your current method configuration is saved and stored in the DPC as method 1, and may be recalled by loading method 1.

❖ **To retrieve methods**

1. Press the Prog key to display Load Method: X<sup>1</sup>.
2. Press Up or Down to locate the desired method number.

3. Press Enter when you see the desired number. This loads the method.

## Step 6: Prepare the Sample

1. Prepare the sample cup holder and sample cup.
  - a. Place the sample cup holder on a level surface.
  - b. Insert a clean sample cup into the sample cup holder.
2. Place sample in the sample cup using a 10  $\mu$ L syringe to place 1  $\mu$ L of the 10  $\mu$ g/mL cholesterol solution on the bottom of the sample cup.
3. Wait several minutes for all of the solvent to evaporate. It takes several minutes to evaporate the solvent, giving you ample time to set up a method and a sequence file (“[Step 7: Create a Method File](#)” on [page 12](#) and “[Step 8: Create a Sequence File](#)” on [page 13](#)).
4. Place the sample cup onto the DIP tip using forceps to insert the sample cup onto the DIP tip.

## Step 7: Create a Method File



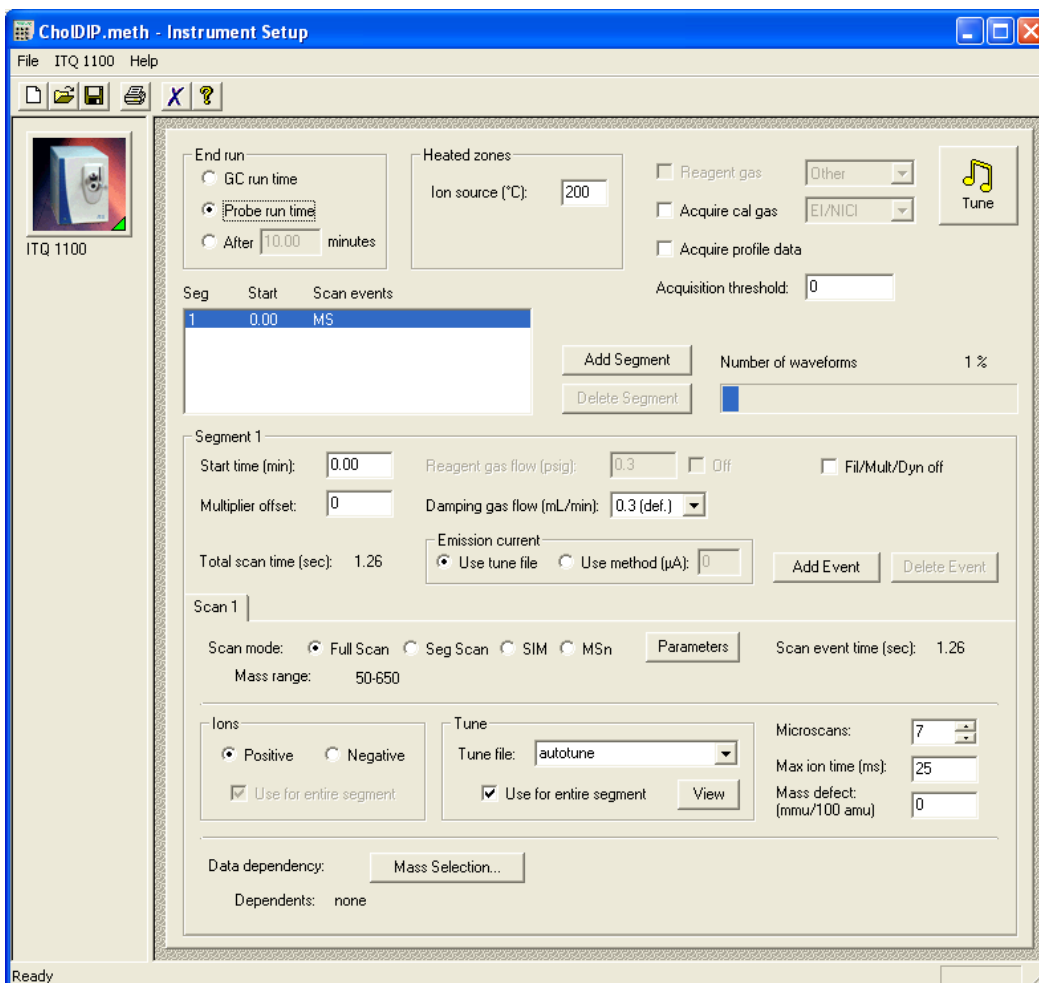
1. Create a method using Instrument Setup.
  - a. From the Windows desktop, click the Xcalibur icon to display the Xcalibur Home Page.
  - b. Select Instrument Setup to display the Instrument Setup window.
2. Enter the instrument method parameters.
  - a. Select File | New to create a new method.

---

<sup>1</sup>X refers to methods numbered 1 - 9.

- b. Enter the method settings shown in Figure 9.

**Figure 9.** Instrument Setup: Ion Trap Series CholdIP Method



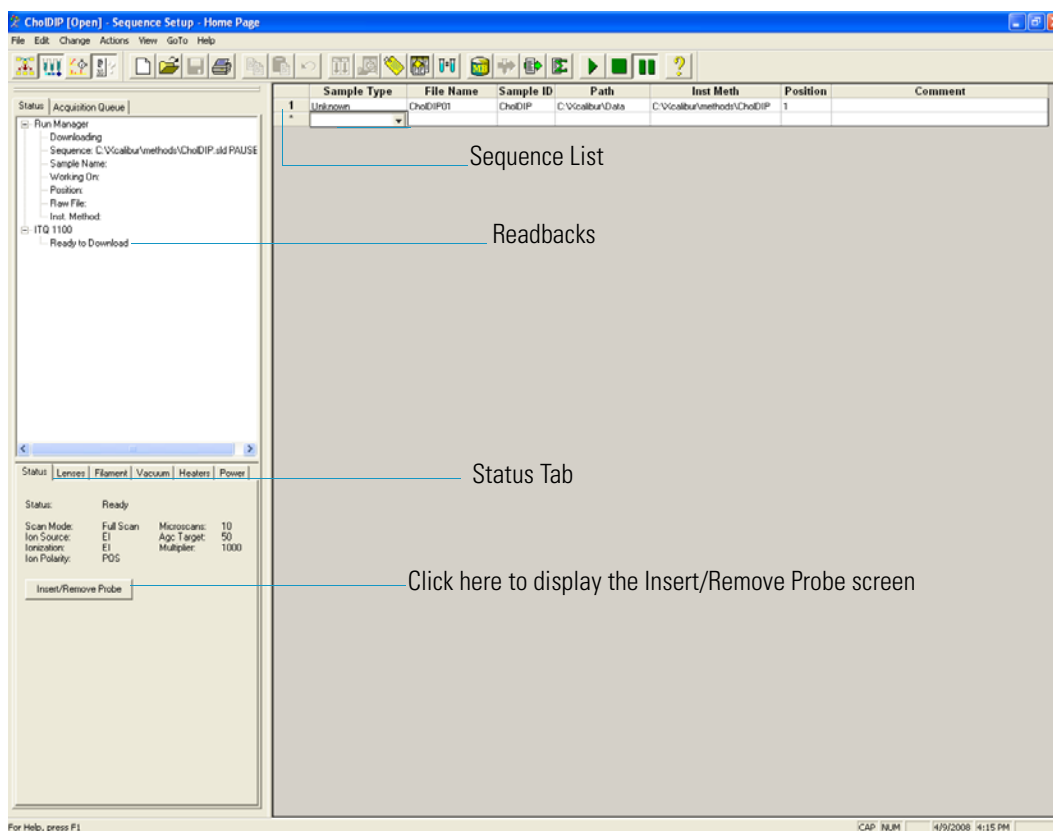
3. Save the method.
  - a. Select File | Save to display the Save As screen.
  - b. Enter CholdIP for the cholesterol method and click Save. The software automatically adds the \*.meth extension to any method file.
  - c. Return to the Xcalibur Home Page.

## Step 8: Create a Sequence File

1. From the Xcalibur Home Page select Sequence Setup to display the Sequence Setup window.
2. Set up a sequence file.
  - a. Select File | New to display a blank sequence file.

- b. Enter the settings displayed in [Figure 10](#).

**Figure 10.** Sequence Setup: Ion Trap Series CholDIP Sequence



**Note** You can type filenames in all uppercase or lowercase. Ion Trap and DSQ Series filenames are not case-sensitive.

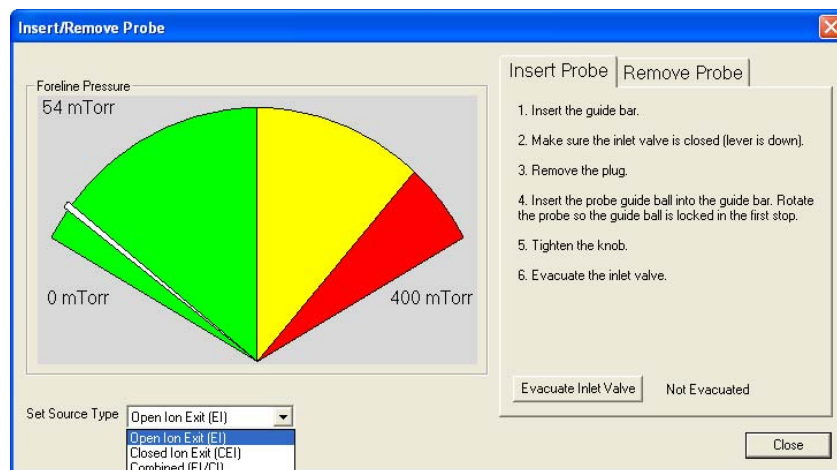
3. Save the sequence file.
- Select File | Save to display the Save As screen.
  - Enter CholDIP as the new sequence file and click Save to return to the Sequence Setup. Xcalibur automatically adds the \*.sld extension to the sequence filename.



## Step 9: Insert the Probe Using Xcalibur

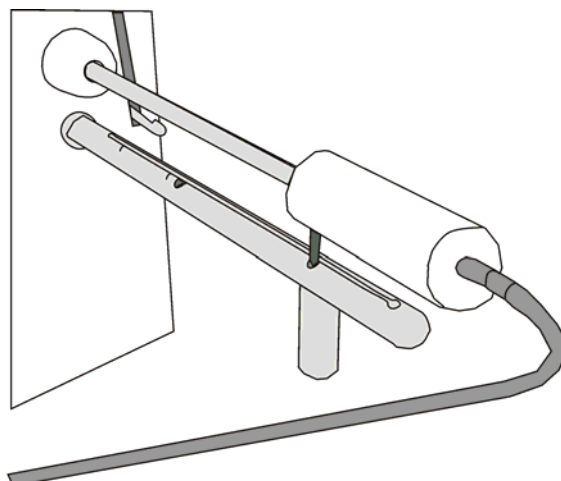
1. Select the Insert / Remove Probe button from the Sequence Setup Status tab to display the Insert/Remove Probe screen.

**Figure 11.** Insert/Remove Probe Screen: Ion Trap Series



- a. Follow the screen instructions on the Insert Probe tab.
- b. At step 4 on the screen instructions, slide the probe into the inlet valve chamber until the stem of the probe shaft reaches the 1st stop on the guide bar (Figure 12).

**Figure 12.** Sample Probe at 1st Stop

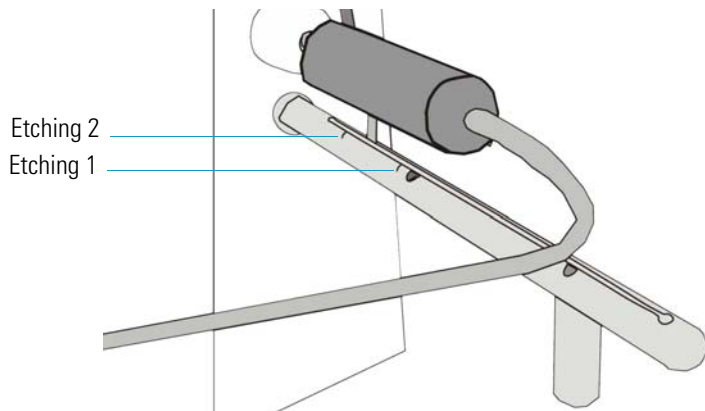


- c. At step 6 on the screen instructions, click the Evacuate Inlet Valve button that is located on the Xcalibur Insert Remove Probe screen.
- d. Wait to see the Inlet Valve Message prompt you to insert the probe further into the chamber.
- e. Open the inlet valve by lifting the inlet valve lever up.



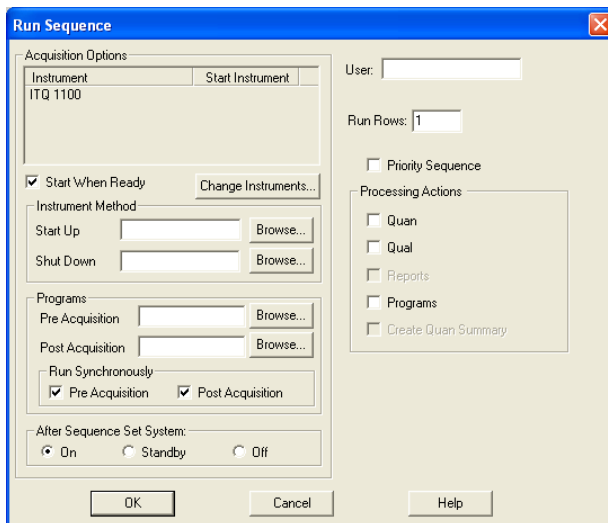
- f. Slide the probe into the chamber until the probe is all the way in the ion volume holder. The stem of the probe shaft is about 15 mm from the 2nd etching on the guide bar (Figure 13).

**Figure 13.** Sample Probe before the 2nd Etching



- g. Click the Close button located on the Insert/Remove Probe screen.
2. Run the saved sequence.
    - a. Highlight the line and sequence you wish to run.
    - b. Select Actions | Run This Sample to display the Run Sequence screen.

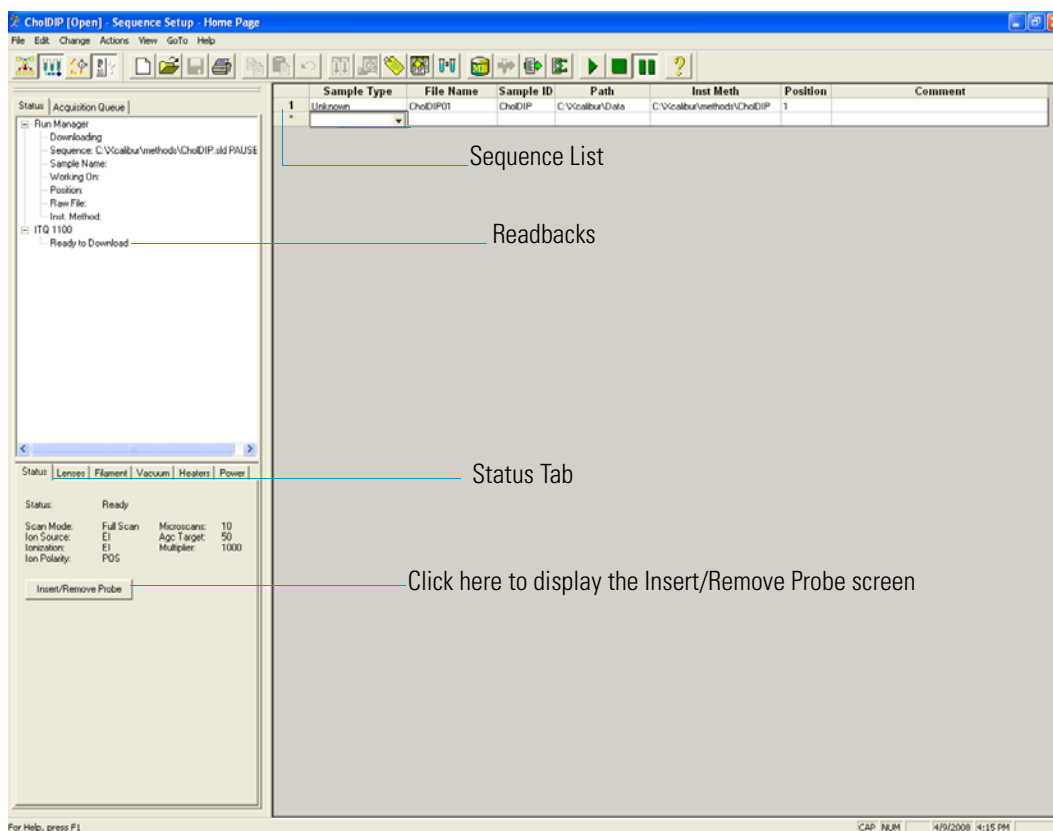
**Figure 14.** Run Sequence Screen: Ion Trap Series



- c. Click OK to return to the Sequence Setup window.

3. Look at the Status tab to read the Readbacks.

**Figure 15.** Sequence Setup: Ion Trap Series CholDIP



- When you see the readback Waiting for Contact Closure, press the Start button on the DPC panel to run the sample.
- Then look for the readback to display Running. If it doesn't, verify that the DPC plug is firmly connected to the back of the mass spectrometer.
- Select View | Real Time Plot View from the Sequence Setup window to monitor the run. The run is completed when the readback displays Ready to Download and the view displays No DataFile.
- Go to ["Step 10: Remove the Probe Using Xcalibur"](#) on [page 17](#).

## Step 10: Remove the Probe Using Xcalibur

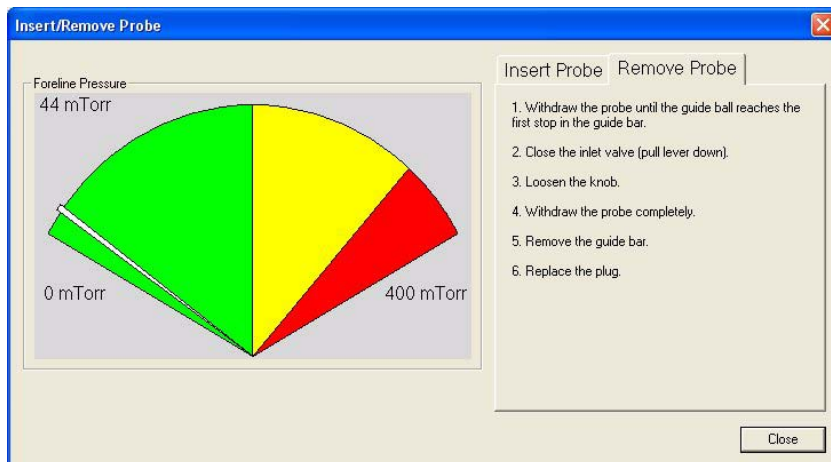
You should still be at the Sequence Setup window. In this step you remove the probe to prepare for the next analysis.



**CAUTION INSTRUMENT DAMAGE.** Avoid accidentally venting the system. Do not remove the probe beyond the point at which the pin in the probe handle reaches the 1st stop in the guide bar. Do not completely withdraw the probe until the ball valve has been closed. Otherwise, the system vents to the atmosphere.

1. Wait until the DPC display panel indicates that the probe has cooled down below 100 °C.
2. Select the Insert/Remove Probe button again from the Status tab.
  - a. Select the Remove Probe tab and carefully follow steps 1–6 shown on the Insert/Remove Probe screen.

**Figure 16.** Remove Probe Tab: Ion Trap Series



- b. Click the Close button to return to the Sequence Setup window.
- c. Begin the next step.

## Step 11: Analyze the Data File

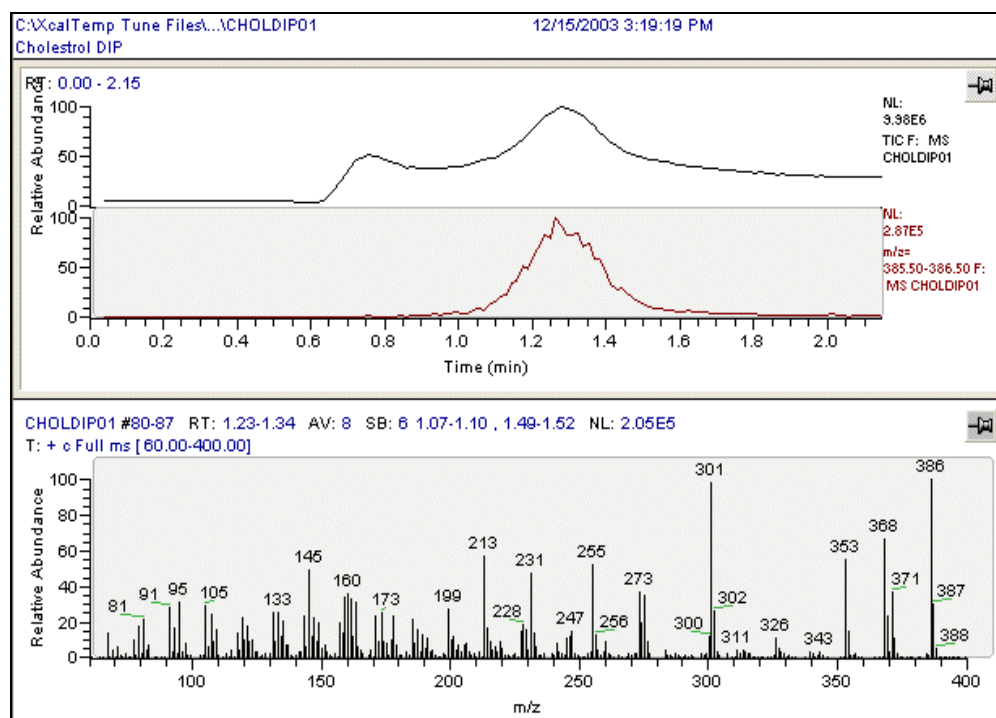
Use these steps to process the data file you just acquired. If you want to analyze another cholesterol sample, repeat the steps listed under “[Step 6: Prepare the Sample](#)” on [page 12](#), making the appropriate addition to the sequence table.

1. At the Sequence Setup window, click the Roadmap icon to return to the Xcalibur Home Page.

Select Qual Browser to open the Qual Browser window. Refer to the Qual Browser manual to learn how to use Qual Browser techniques.
2. Select File | Open and select the CholDIP.raw data file you just ran.

Compare your results with [Figure 17](#).

**Figure 17.** Qual Browser: Ion Trap Series DIP



For information on how to load samples see [“Loading DIP Samples”](#) on [page 35](#), clean the probe tip see [“Cleaning the DIP”](#) on [page 37](#), and terms to know see [“Top Terms To Know”](#) on [page 38](#).

## Analyzing Cholesterol with the DSQ Series

**Note** DSQ Series refers to both the DSQ II and DSQ mass spectrometers.

Using a DSQ, analyzing sample involves these steps:

“Step 1: Connect the Direct Probe System” on page 21

“Step 2: Change the Ion Volume” on page 23

“Step 3: Deselect AS and GC Devices” on page 25

“Step 4: Tune and Calibrate the Mass Spectrometer” on page 26

“Step 5: Program the DPC” on page 26

“Step 6: Prepare the Sample” on page 28

“Step 7: Create a Method File” on page 28

“Step 8: Create a Sequence File” on page 29

“Step 9: Insert the Probe Using Xcalibur” on page 31

“Step 10: Remove the Probe Using Xcalibur” on page 33

“Step 11: Analyze the Data File” on page 34

Steps 1 through 3 are considered the initial setup. After the initial setup you may skip steps 1 through 3 and proceed to steps 4 through 11.



#### Tools Needed

- DSQ Series mass spectrometer
- I/R tool and guide bar (included with mass spectrometer), PN 96000-60057
- Xcalibur Data System
- Operating System: Windows XP
- DPC Controller Assembly, PN 119300-0100
- DIP Kit (PN 119300-ODIP), includes the following:
  - (1) DIP/DEP Test Mix-10 mg/mL, PN 119343-0001
  - (1 pkg) DIP Sample Cups (25), PN 119329-0001
  - (2) Ion Volume Holders, PN 119324-0001



#### Frequency

As Needed

## Step 1: Connect the Direct Probe System

Before beginning your sample analysis, connect the DPC to the mass spectrometer and connect the sample probe to the DPC. Then make sure all connections are fully seated.

1. Place the DPC in a convenient location on top of or close to the mass spectrometer.

Read “[Top Safeguards to Know](#)” on [page 40](#) to be certain that you exercise all necessary safety precautions.



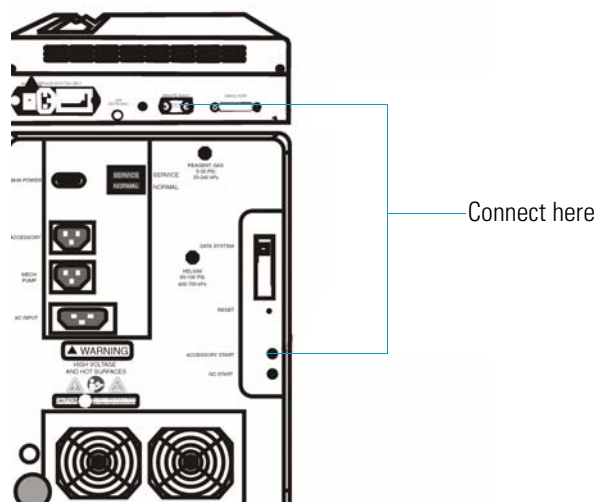
**WARNING ELECTRICAL SHOCK HAZARD.** Avoid exposing the DIP to electrical shock or electrical charging. Always plug the DIP cable into the DPC probe connector when performing EI or CI experiments, even if the probe is not heated. The DIP cable connection to the DPC provides electrical ground.

## 1 Using a Direct Insertion Probe (DIP)

Analyzing Cholesterol with the DSQ Series

2. Connect the DPC to the mass spectrometer (Figure 18).

**Figure 18.** DPC to Mass Spectrometer (Back View)



---

Connect the REMOTE START CABLE to DPC REMOTE READY and the mass spectrometer ACCESSORY START

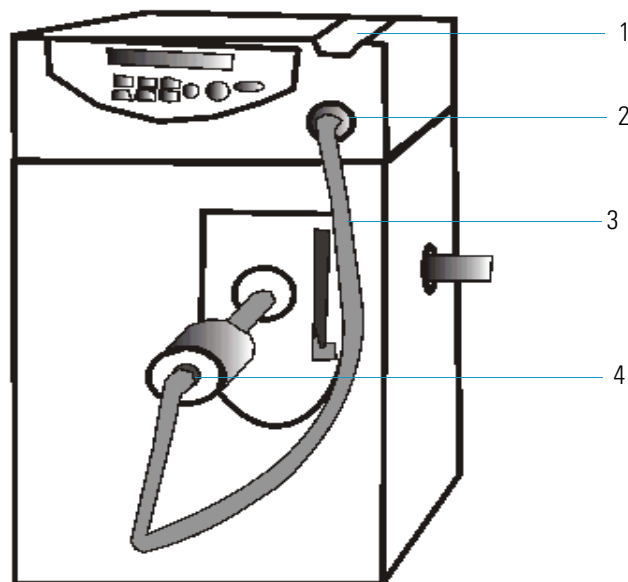
---

- a. Connect the DPC to the mass spectrometer by using the DPC REMOTE START cable to connect into the DPC REMOTE READY connection and the other end to the mass spectrometer ACCESSORY START connection.
- b. Plug the desired air source to use as a probe coolant into the DPC AIR connection.
- c. The air source should be dry air or nitrogen at no more than 100 psi. We recommend 80 psi for normal operation. The coolant is used with the DIP to rapidly cool the probe tip, which allows rapid sample through-put. This system is also designed for compressed air or nitrogen. However, it is not designed for subambient operation, and room temperature will affect the minimum probe tip temperature.
- d. Connect the power cord to the DPC power plug connection and the other end to the power source.
- e. Turn the DPC on by switching the Power Switch to |. Xcalibur automatically detect the probe and activates the Insert/Probe settings on the Home page, Tune window, and Sequence Editor.



3. Connect the DPC to the probe.

**Figure 19.** DPC to Probe Connections



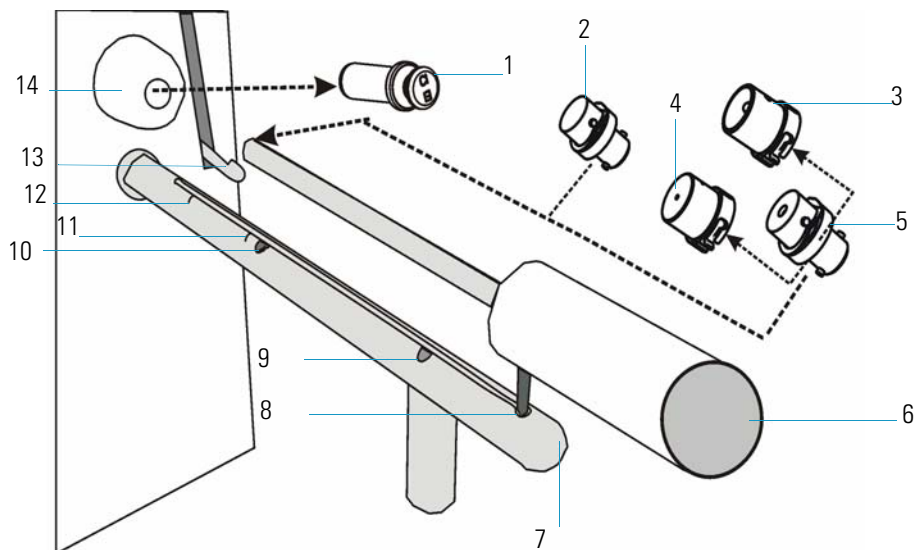
Item	Description	PN	Item	Description	PN
1	DPC Cradle		3	DPC EXTERNAL Probe Cable	119303-0001
2	DPC Connection		4	Probe Connection	

- a. Plug one end of the DPC external cable to the DPC connection and the other end to the probe connection. Do this by matching the red dot located on the cable to the red dot located on the DPC and firmly pressing the cable in until it locks in position.
  - b. Place the probe in the built-in cradle located on top of the DPC. Either the DIP or DEP can safely rest in the built-in cradle when not in use.
4. Turn on the MS, DPC, and the Xcalibur Data System software.
  5. Confirm that the Insert/Remove Probe button displays on the Xcalibur Data System Home-Page. Typically, the software automatically detects if the probe is connected.

## Step 2: Change the Ion Volume

Samples can be analyzed in either EI or CI mode. Standard ion volumes are used using the probe ion volume holder included with your probe. The probe ion volume holder has a hole that allows the sample from the probe tip to enter the ion volume as compared to the GC ion volume holder, which allows sample to enter through the mass spectrometer transfer line (see [Figure 20](#)).

**Figure 20.** I/R Tools



Item	Description	PN	Item	Description	PN
1	Inlet Valve Plug	119273-0001	8	Guide Ball start position	
2	GC Ion Volume holder (standard)	70001-20532	9	1st stop	
3	EI Ion Volume	119650-0220	10	2nd stop	
4	CI Ion Volume	119650-0230	11	1st etching	
5	Probe Ion Volume holder	119324-0001	12	2nd etching	
6	I/R Tool	96000-60057	13	Inlet Valve lever	
7	Guide Bar	119687-0010	14	Inlet Valve (vacuum interlock chamber)	

1. Confirm that you are at the Xcalibur Home-Page.

**Note** Carefully follow each step on the Insert Remove Probe screen.

2. Remove the current ion volume.
  - a. From the Xcalibur Home Page select the Insert/Remove Probe button (Hint: it's located on the Status tabs) to display the Insert Remove Probe screen.
  - b. Select the Insert Probe tab.
  - c. Attach the probe ion volume holder on the I/R tool.

**Note** When prompted on the Insert Remove Probe screen, insert the I/R tool into the guide bar start position using the guide bar to enter the inlet valve chamber and remove the current ion volume.

- d. Insert the guide bar into the mass spectrometer as illustrated in [Figure 20](#).
  - e. Follow each step listed on the Insert Probe tab to insert the probe and remove the current ion volume.
  - f. Select the Remove Probe tab and carefully follow each step to remove the probe.
3. Install an EI or CI ion volume.
    - a. Attach either an EI ion volume or CI ion volume to the probe ion volume holder while making sure it is tightly secured.
    - b. From the Xcalibur Home Page select the Insert/Remove Probe button to display the Insert Remove Probe screen.
    - c. Use the Insert Probe tab instructions to insert the probe install a CI or EI ion volume.
    - d. Use the Remove Probe tab instructions to remove the probe.
    - e. Close the Insert Remove Probe screen and return to the Xcalibur Data System Home-Page.

### Step 3: Deselect AS and GC Devices

If you are running several probe runs, deselect the AS and GC instruments in the Xcalibur Instrument Configuration software. Configure for the mass spectrometer only when analyzing samples with the probe. Deselecting the AS and GC simplifies the methods and avoids waiting on GC equilibration time before every run. However, it is not necessary.

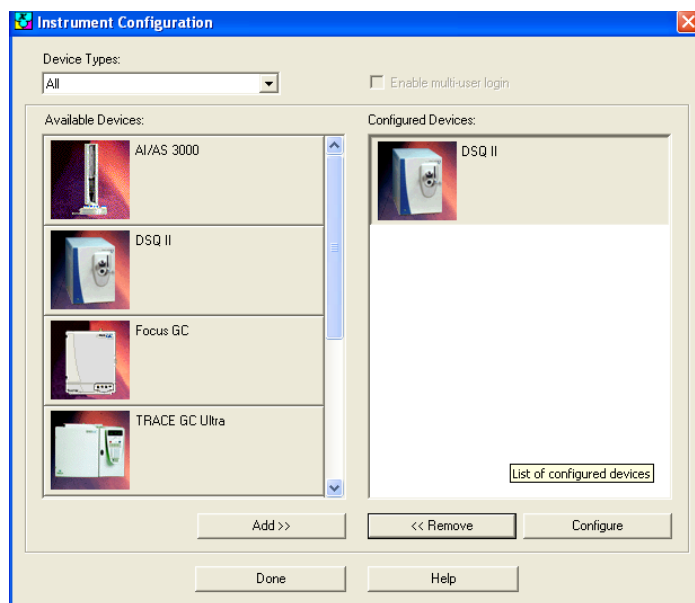


1. Start Xcalibur Instrument Configuration.

From the Windows desktop, select the Instrument Configuration shortcut to display the Instrument Configuration screen.

2. Remove the AS and GC devices from Configured Devices.

**Figure 21.** Instrument Configuration: DSQ Series



Select the item to be removed from the Configured Devices section and click the Remove button. We recommend using the Ion Trap and DSQ Series configuration only when analyzing samples with the probe.

3. Click Done to return to the windows desktop.

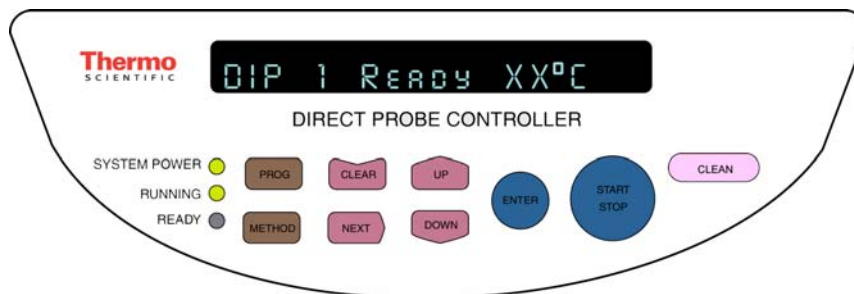
## Step 4: Tune and Calibrate the Mass Spectrometer

Refer to the Tune online help for instructions in determining if the instrument has been recently tuned and calibrated.

## Step 5: Program the DPC

1. Turn the DPC ON to display DIP 1 Ready.

**Figure 22.** DIP DPC Display Panel and Keypad



The DPC automatically detects if a DIP or DEP is connected, and will display the DIP or DEP series of menu controls for setting the temperature and for heating the probe tip.

2. Enter the initial temperature.
  - a. Press METHOD to display Initial Temp.
  - b. Press UP or DOWN until you see 50 °C.
3. Enter the initial time.
  - a. Press NEXT to display Initial Time.
  - b. Press UP or DOWN to display the initial time of 60 seconds.
4. Enter the ramp 1 rate.
  - a. Press NEXT to display Ramp 1 Rate.
  - b. Press UP or DOWN to set the Ramp Rate to MAX. This ramps the probe tip ballistically at its maximum rate of approximately 150 °C min.
5. Enter the ramp 1 temperature.
  - a. Press NEXT to display Ramp 1 Temp.
  - b. Press UP or DOWN until the temperature is 350 °C.
6. Enter the ramp 1 hold time.
  - a. Press NEXT to display Ramp 1 Hold.
  - b. Press UP or DOWN until the Ramp 1 Hold time displays 40 seconds.
7. Enter the ramp 2 rate.
  - a. Press NEXT to display Ramp 2 Rate.
  - b. Press UP or DOWN until the Ramp 2 Rate is OFF.
8. Return to DIP 1 Ready.

Press NEXT to display DIP 1 Ready.
9. Load the method.

Press PROG to display Load Method: X.
10. Save the method.
  - a. Press NEXT to display Save Method: X<sup>1</sup>.
  - b. Press ENTER to return to DIP 1 Ready.
  - c. Listen for the DPC to beep three times to indicate that your current method configuration is saved and stored in the DPC as method 1, and may be recalled by loading method 1.

## 1 Using a Direct Insertion Probe (DIP)

Analyzing Cholesterol with the DSQ Series

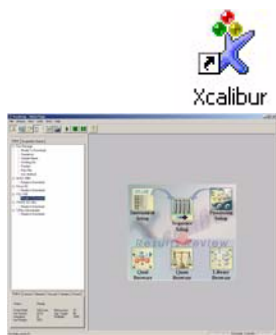
### ❖ To retrieve methods

1. Press the Prog key to display Load Method: X<sup>1</sup>.
2. Press Up or Down to locate the desired method number.
3. Press Enter when you see the desired number. This loads the method.

## Step 6: Prepare the Sample

1. Prepare the sample cup holder and sample cup.
  - a. Place the sample cup holder on a level surface.
  - b. Insert a clean sample cup into the sample cup holder.
2. Place sample in the sample cup using a 10 µL syringe to place 1 µL of the 10 µg/mL cholesterol solution on the bottom of the sample cup.
3. Wait several minutes for all of the solvent to evaporate. It takes several minutes to evaporate the solvent, giving you ample time to create a method (“[Step 7: Create a Method File](#)” on [page 28](#)) and create a sequence file (“[Step 8: Create a Sequence File](#)” on [page 29](#)).
4. Place the sample cup onto the DIP tip using forceps to insert the sample cup onto the DIP tip.

## Step 7: Create a Method File



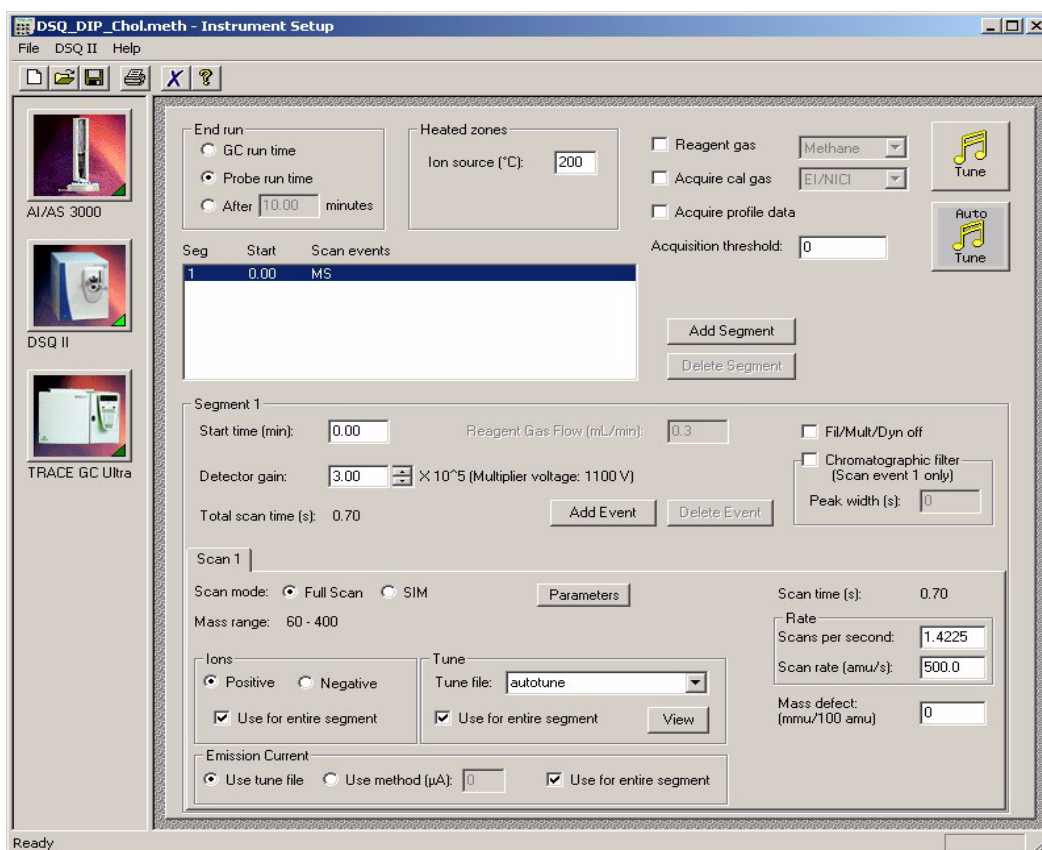
1. Create a method using Xcalibur Instrument Setup.
  - a. From the Windows desktop, select the Xcalibur shortcut to display the Xcalibur Home Page.
  - b. Select the instrument setup icon to display the Instrument Setup window.
2. Enter the instrument method parameters.
  - a. Select File | New to create a new method.

---

<sup>1</sup>X refers to methods numbered 1-9.

- b. Enter the method settings shown in [Figure 23](#).

**Figure 23.** Instrument Setup: DSQ Series CholDIP Method



3. Save the method.
  - a. Select File | Save to display the Save As screen.
  - b. Enter CholDIP for the cholesterol method and click Save. The software automatically adds the \*.meth extension to any method file.
  - c. Return to the Xcalibur Home Page.

## Step 8: Create a Sequence File

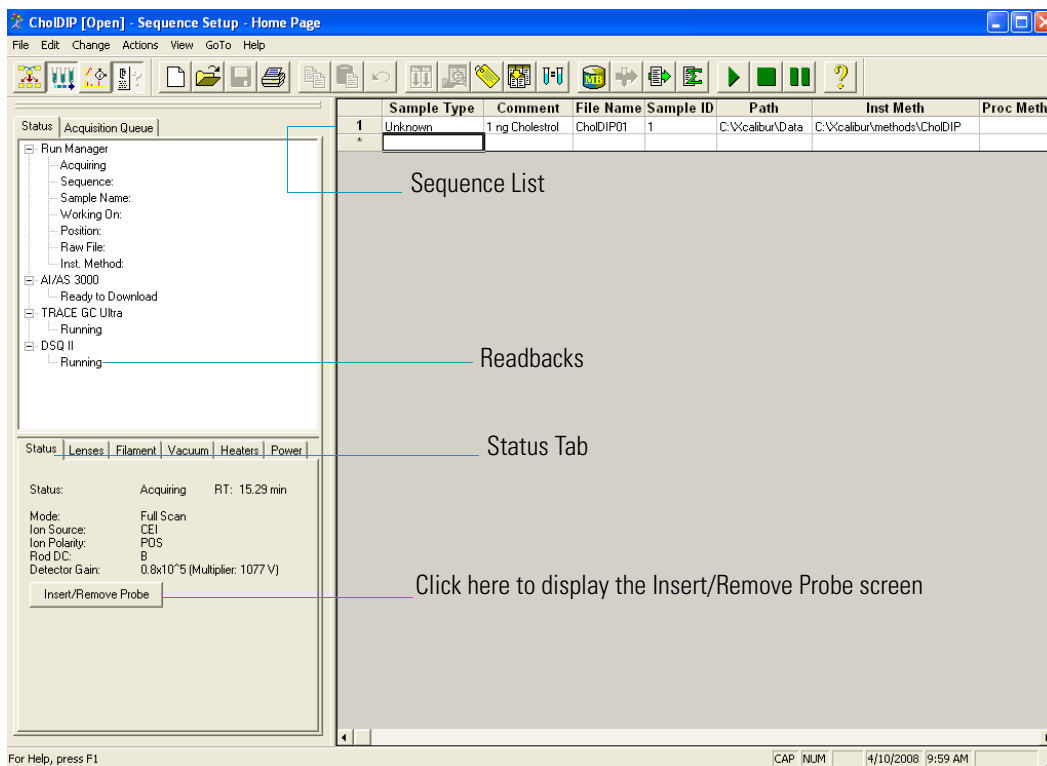
1. From the Xcalibur Home Page select Sequence Setup to display the Sequence Setup window.
2. Set up a sequence file.
  - a. Select File | New to display a blank sequence file.

## 1 Using a Direct Insertion Probe (DIP)

Analyzing Cholesterol with the DSQ Series

- b. Enter the settings displayed in Figure 24.

**Figure 24.** Sequence Setup: DSQ Series CholdIP Sequence



**Note** You can type filenames in all uppercase or lowercase. Ion Trap and DSQ Series filenames are not case-sensitive.

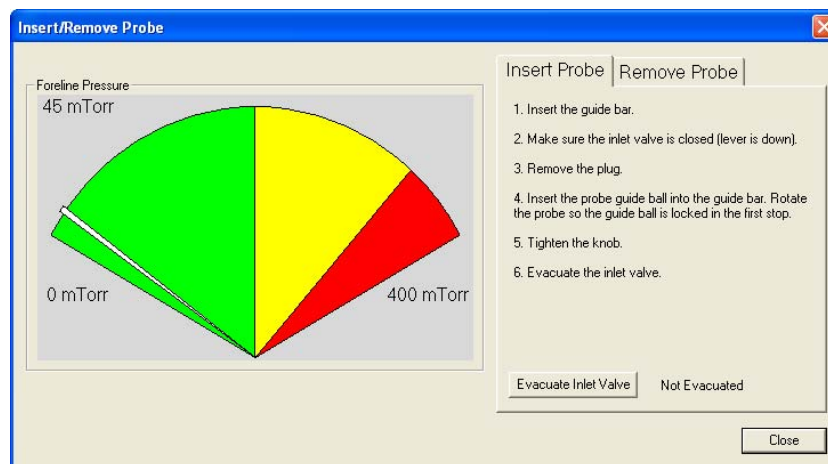
3. Save the sequence file.
  - a. Select File | Save to display the Save As screen.
  - b. Enter CholdIP as the new sequence file and click Save to return to the Sequence Setup. Xcalibur automatically adds the \*.sld extension to the sequence filename.



## Step 9: Insert the Probe Using Xcalibur

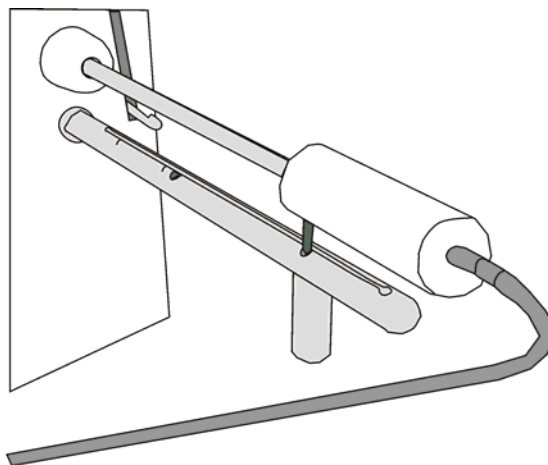
1. Select the Insert / Remove Probe button from the Sequence Setup Status tab to display the Insert/Remove Probe screen.

**Figure 25.** Insert/Remove Probe Screen: DSQ Series



- a. Follow the screen instructions on the Insert Probe tab.
- b. At step 4 on the screen instructions, slide the probe into the inlet valve chamber until the stem of the probe shaft reaches the 1st stop on the guide bar (Figure 26).

**Figure 26.** Sample Probe at 1st Stop

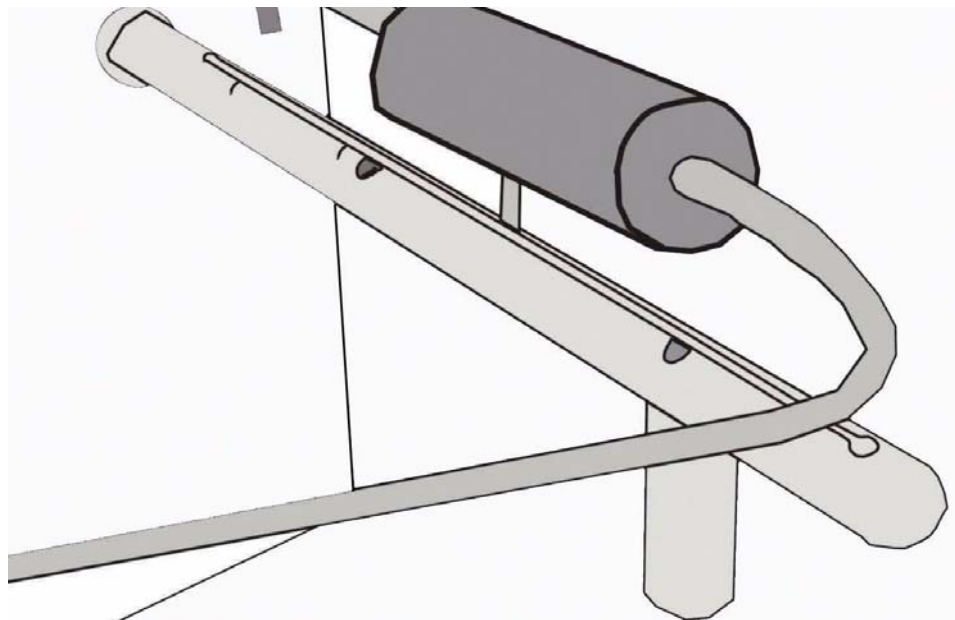


- c. At step 6, click the Evacuate Inlet Valve button that is located on the Xcalibur Insert Remove Probe screen.
- d. Wait to see the Inlet Valve Message prompt you to insert the probe further into the chamber.
- e. Open the inlet valve by lifting the inlet valve lever up.



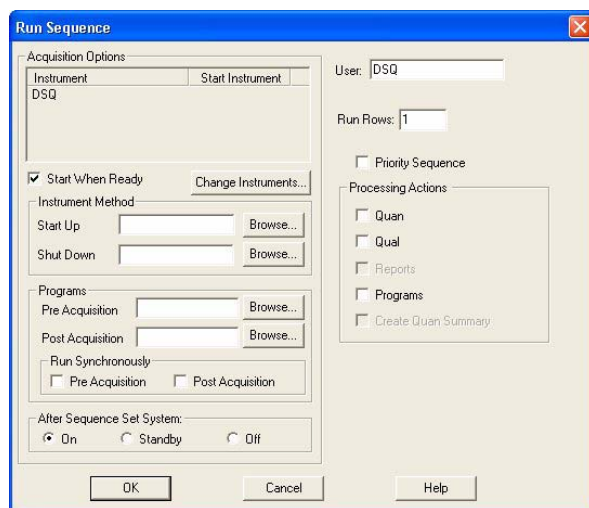
- f. Slide the probe into the chamber until the probe is all the way into the ion volume holder and the stem of the probe shaft *ALMOST* reaches the 2nd stop on the guide bar (Figure 27).

**Figure 27.** Sample Probe before the 2nd Stop



- g. Click the Close button located on the Insert/Remove Probe screen.
2. Run the saved sequence.
    - a. Highlight the line and sequence you wish to run.
    - b. Select Actions | Run This Sample to display the Run Sequence screen.

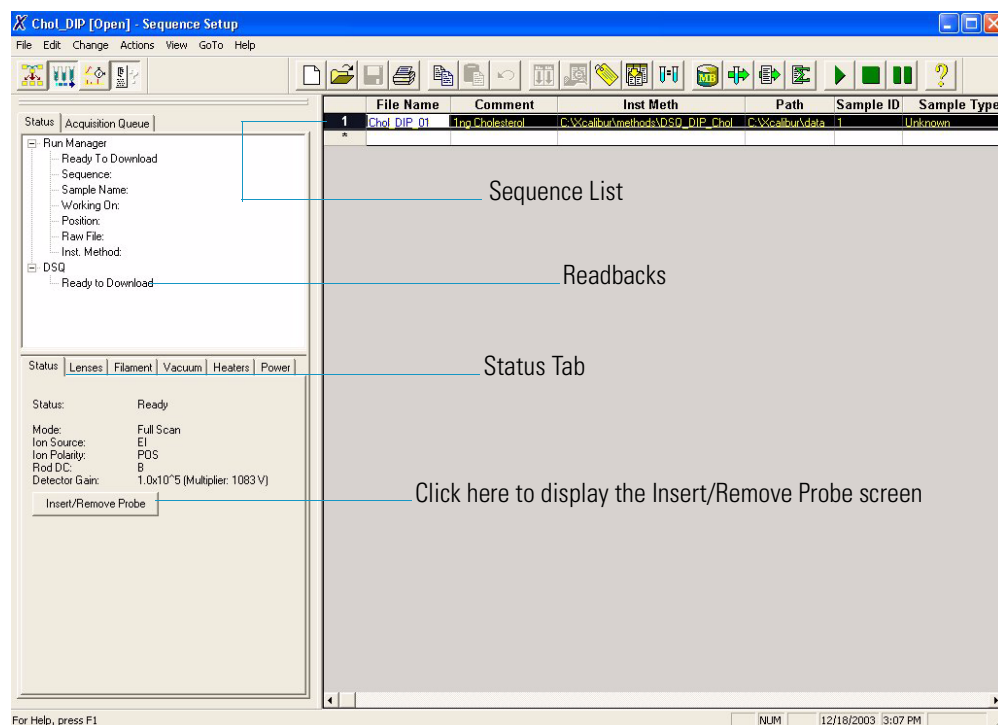
**Figure 28.** Run Sequence Screen: DSQ Series



- c. Click OK to return to the Sequence Setup window.

3. Look at the Status tab to read the DSQ readbacks.

**Figure 29.** Sequence Setup: DSQ Series CholdIP



- a. When you see the readback Waiting for Contact Closure, press the Start button on the DPC panel to run the sample.
- b. Look for the DSQ readback to display Running. If it doesn't, verify that the DPC cable is firmly connected to the back of the DSQ.
- c. Select View | Real Time Plot View from the Sequence Setup window to monitor the run.
- d. The run is completed when the readback displays Ready to Download and the view displays No DataFile.
- e. Go to the next step, ["Step 10: Remove the Probe Using Xcalibur"](#) on [page 33](#).

## Step 10: Remove the Probe Using Xcalibur

You should still be at the Sequence Setup window. In this step you remove the probe to prepare for the next analysis.

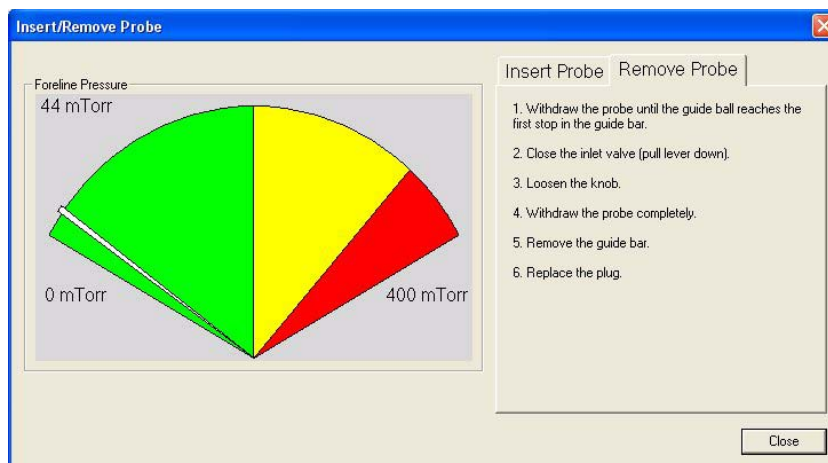


**CAUTION INSTRUMENT DAMAGE.** Avoid accidentally venting the system. Do not remove the probe beyond the point at which the pin in the probe handle reaches the stop in the guide bar. Do not completely withdraw the probe until the ball valve has been closed. Otherwise, the system vents to the atmosphere.

1. Wait until the DPC display panel indicates that the probe has cooled down below 100 °C.

2. Select the Insert/Remove Probe button again from the Status tab.
  - a. Select the Remove Probe tab and *carefully* follow steps 1–6.

**Figure 30.** Remove Probe Tab: DSQ Series



- b. Click the Close button to return to the Sequence Setup window.
    - c. Begin the next step.

## Step 11: Analyze the Data File

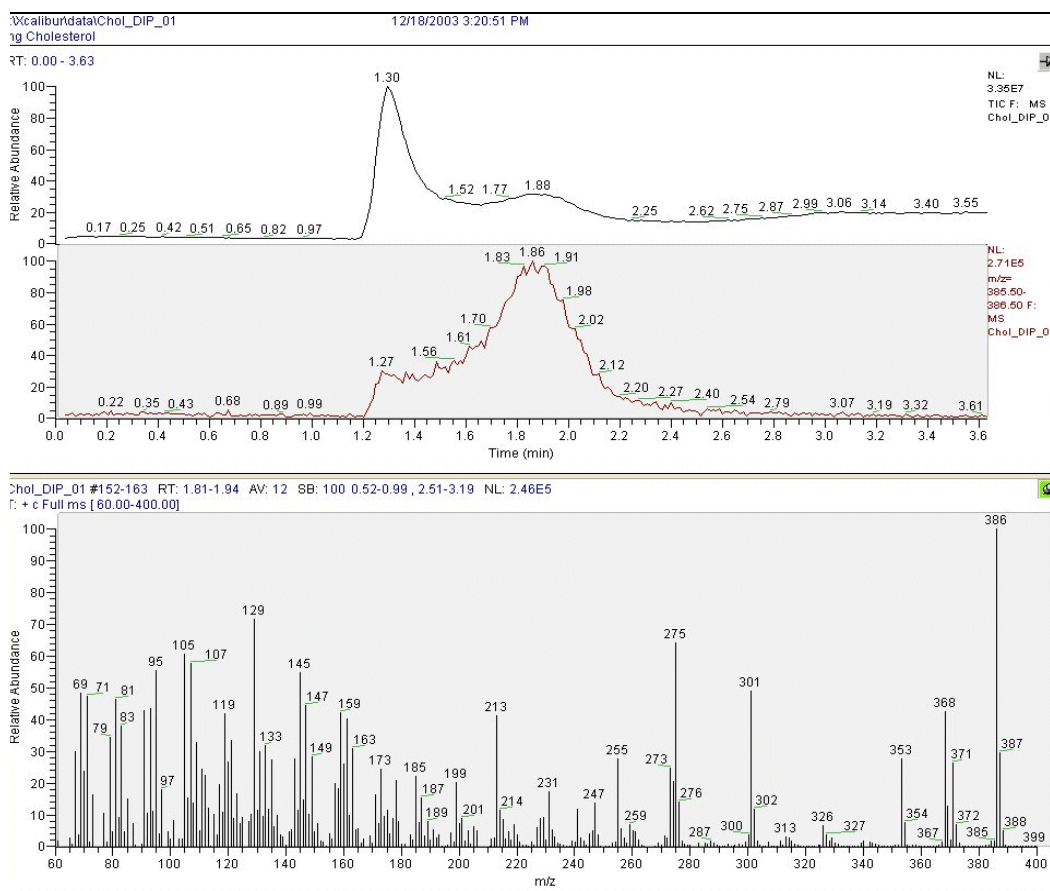
Use these steps to process the data file you just acquired. If you want to analyze another cholesterol sample, repeat the steps listed under “[Step 6: Prepare the Sample](#)” on [page 28](#), making the appropriate addition to the sequence table.

1. At the Sequence Setup window, click the Roadmap icon to return to the Xcalibur Home Page.

Select Qual Browser to open the Qual Browser window. Refer to the Qual Browser manual to learn how to use Qual Browser techniques.
2. Select File | Open and select the CholDIP.raw data file you just ran.

Compare your results with [Figure 31](#).

**Figure 31.** Qual Browser: DSQ Series DIP



For information on how to load samples see [“Loading DIP Samples”](#) on [page 35](#), clean the probe tip see [“Cleaning the DIP”](#) on [page 37](#), and terms to know see [“Top Terms To Know”](#) on [page 38](#).

## Loading DIP Samples

You can load either liquid or solid samples into your DIP. However, before loading samples use these precautions to minimize contaminating the ion source.

1. Inspect the probe tip and the sample cup holder.

Clean the probe tip prior to loading the first sample. A given probe tip may be contaminated by handling or from previous sample runs. For more detailed instructions, see [“Cleaning the DIP”](#) on [page 37](#).

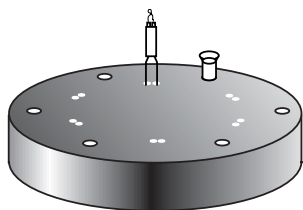
2. Handle sample cups with clean forceps.
  - a. Use one sample cup for each sample to avoid cross-contamination.

- b. Make sure the sample cup is clean. For more details, see “Cleaning the DIP” on page 37.
3. Place the sample cup holder on a level surface.

**Note** The sample cup holder can be used to hold several samples at one time and can be carefully warmed on a laboratory hot plate to evaporate the solvent.

Use clean forceps to place a sample cup into the sample cup holder (Figure 32).

**Figure 32.** Sample Cup Holder, 119342-0001



4. Put a liquid or solid sample into the sample cup.
  - a. Liquid samples may be injected directly into the sample cup. However, you may choose to dilute your liquid sample in a solvent.
  - b. Put solid samples directly into the sample cup. Or, you may choose to dissolve solid samples in a solvent.
  - c. Do not overload sample cups.
  - d. Use small samples to help keep the system clean, thereby reducing the amount of maintenance required. Small samples also keep the background low. In general, the sample size should be 1  $\mu\text{L}$  or less to minimize contaminating the ion source.
  - e. Sample along the walls may evaporate at a different temperature than sample at the bottom, and could therefore distort the evaporation profile.
  - f. Avoid putting sample on the outer wall of the sample cup. This contaminates the probe tip.

## Loading Liquid Samples

1. [Optional] Dilute the sample.



**CAUTION INSTRUMENT DAMAGE.** Allow sample solvent to evaporate completely before inserting probe into the mass spectrometer. If solvent is not allowed to evaporate, then sample loss may occur. This may cause the pump to stall, because the solvent rapidly evaporates within the vacuum.

If you are using the sample in its original form, proceed to the next step otherwise, dilute the sample in a small quantity of solvent.

2. Use a 10  $\mu\text{L}$  syringe to draw 1 or 2  $\mu\text{L}$  of sample.

- a. Inject the liquid into the bottom of the sample cup.
  - b. Wait for all of the solvent to evaporate.
3. Use a pair of clean forceps to insert the sample cup into the DIP tip.

You can use an infrared or incandescent bulb as a heat source to increase the evaporation rate of your solvent. However, be careful not to warm the sample droplet too quickly, or the solvent could sputter and cause sample loss.

## Loading Solid Samples

1. [Optional] Dilute the solid sample.
  - a. Dissolve the sample in a small quantity of solvent. If you are using the sample in its original form, proceed to the next step otherwise, dilute the sample in a small quantity of solvent.
  - b. Use a 10  $\mu$ L syringe to draw 1 or 2  $\mu$ L of sample.
  - c. Inject the liquid into the bottom of the sample cup.
  - d. Wait for all of the solvent to evaporate.



**CAUTION INSTRUMENT DAMAGE.** Allow sample solvent to evaporate completely before inserting probe into the mass spectrometer. If solvent is not allowed to evaporate, then sample loss may occur. This may cause the pump to stall, because the solvent rapidly evaporates within the vacuum.

You can use an infrared or incandescent bulb as a heat source to increase the evaporation rate of your solvent. However, be careful not to warm the sample droplet too quickly, or the solvent could sputter and cause sample loss.

2. Load solid samples with a dry syringe needle or use a piece of clean wire or capillary column to collect some of the sample.

Put the solid sample into the sample cup and compress it into the bottom.

3. Use a pair of clean forceps to insert the sample cup into the DIP tip.

## Cleaning the DIP

1. After your DIP has thoroughly cooled, check it for obvious residue or abrasions.

Examine and clean your DIP and sample cups often to ensure the integrity of your experiments.
2. Clean the probe.
  - a. Use only a soft cloth to wipe the probe handle and probe shaft.



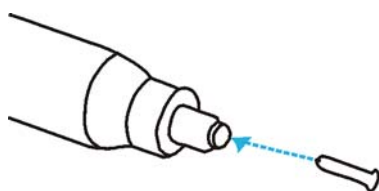
- b. Use a soft cloth dampened in a mild detergent and water solution to remove dirt. Never use abrasive cleaners.
    - c. Wipe the probe with water to remove detergent residue.
    - d. Allow the probe to dry completely before resuming operation.
  3. Clean the sample cups.

If you do not want to clean your sample cups, then use them only once and discard them appropriately. Otherwise, carefully clean the sample cups before you use them. Sample cup contaminants contribute to the ion current during evaporation.

    - a. Clean the sample cups in an ultrasonic solvent bath.
    - b. Dry the sample cups in a drying oven at 250 °C.
    - c. Store clean sample cups in a clean glass container.
    - d. Use clean forceps to handle the clean sample cups and to avoid contamination.

## Top Terms To Know

**Figure 33.** DIP Sample Probe and Sample Cup



## DIP Modes

The DIP can be operated in either ballistic mode or temperature-programmed mode. To program the DIP for the ballistic and temperature-programmed modes, use the DPC keypad after the DIP, DPC, and mass spectrometer are connected.

- Ballistic Mode

When using the DIP in ballistic mode, the probe heats at the maximum possible rate (approximately 150 °C/min) until the final setpoint is achieved.

Use the ballistic mode for:

- Checking the evaporation behavior of unknown samples
- Looking for the spectrum or the main peaks of an unknown sample
- Determining the evaporation parameters for the temperature-programmed mode of operation
- Temperature-Programmed Mode



In the temperature-programmed mode, the DIP is programmed to heat at a specified rate (or gradient). The probe ramps at this rate until the final temperature is reached. The DIP can be programmed to heat in 10 °C/min increments up to a maximum rate of 100 °C/min.

Use the temperature-programmed mode for:

- Observing the evaporation of different components of the sample as a function of temperature
- Creating a longer lasting signal from the sample to allow more complete characterization

## Probe Shaft

The probe shaft is a long metal rod attached to a cylindrical handle. It measures approximately 17-in. long and 0.531-in. in diameter. Electrical cabling and coolant gas lines pass through the probe shaft from the cable connection in the handle to the probe tip.

## Probe Tip

At the end of the probe shaft is the probe tip, which has a spring-loaded holder into which the sample cup is inserted. The probe tip seals against the outer surface of the ion volume holder. This positions the sample cup in front of the ion volume. As the probe tip is heated by a coiled heater, its temperature is measured by an RTD (resistive temperature device). The DIP temperature is controlled through circuitry located in the DPC, which precisely regulates the probe tip temperature during the heating cycle. After a heating cycle, a solenoid is automatically activated, and compressed air or nitrogen flows through the probe tip. This rapidly cools the probe tip. The coolant is not designed for subambient operations or for use with condensable cooling gases. The operating range of the DIP is from ambient to 450 °C. However, once inserted into the ion source, the DIP tip begins heating from the radiant heat of the ion source. For volatile samples, it may be necessary to start the mass spectral analysis with the DIP inserted just slightly away from the ion source, then gently insert into the ion source and begin temperature programming.

## Sample Cups

Sample cups are small glass vials. A sample is introduced into a sample cup. Then, the sample cup is inserted into a spring-loaded holder in the probe tip. The DIP is inserted through the inlet valve and into the ion source. The DIP uses 9-mm (0.35-in.) one-piece Pyrex sample cups for standard DIP operations.

# Top Safeguards to Know

When using the DIP, please use these safety precautions.



**WARNING ELECTRICAL SHOCK HAZARD.** Avoid exposing the DIP to electrical shock or electrical charging. Always plug the DIP cable into the DPC probe connector when performing EI or CI experiments, even if the probe is not heated. This is because the DIP cable provides electrical ground.

1. When not using the probe, place it in the DPC cradle, which is on top of the DPC.
2. Avoid damaging the tip when inserting sample cups or cleaning.
3. Avoid scratching or otherwise abrading the surface of the probe shaft. Damage to the probe shaft could result in leaks in the inlet valve vacuum seal.
4. Avoid contaminating the sample cup or the probe shaft with the sample, solvent, or finger oils. Contamination produces unwanted background or memory effects.
5. Be careful to not push the sample probe into the inlet valve.
6. Do not heat the DIP beyond the point necessary to obtain the desired spectra, or needless ion source contamination will occur.
7. Do not heat the DIP over 350 °C for extended periods. The maximum temperature is 450 °C for 1 min.
8. Do not withdraw the DIP until it has cooled to below 100 °C.



**CAUTION INSTRUMENT DAMAGE.** Removing the DIP while it is hot (above 100 °C) damages the inlet valve vacuum seals.

9. Avoid accidentally venting the system. Make sure the inlet valve is closed (the valve handle is in the down position) before removing the DIP. For instructions when using the Ion Trap Series see [“Step 10: Remove the Probe Using Xcalibur” on page 17](#) or for the DSQ Series see [“Step 10: Remove the Probe Using Xcalibur” on page 33](#).

## Hints

- To keep sample cool for volatile samples, keep the probe outside the ion source until ready to press the DPC Go button.

- [DSQ only] High Mass may require a higher ion offset and resolution for high scan rates.
- [DSQ only] Get the best resolution by scanning slowly over a narrow mass range.

## **1 Using a Direct Insertion Probe (DIP)**

Hints

## Using a Direct Exposure Probe (DEP)

This chapter explains how to use the DEP with either the Ion Trap or the DSQ series instruments.

### Contents

- “How the Direct Probe System Works” on page 44
- “Analyzing Cholesterol with the Ion Trap Series” on page 46
- “Analyzing Cholesterol with the DSQ Series” on page 62
- “Loading DEP Samples” on page 76
- “Loading Liquid Samples” on page 77
- “Loading Solid Samples” on page 78
- “Cleaning the DEP” on page 78
- “Top Terms to Know” on page 81
- “Top Safeguards to Know” on page 82
- “Hints” on page 83

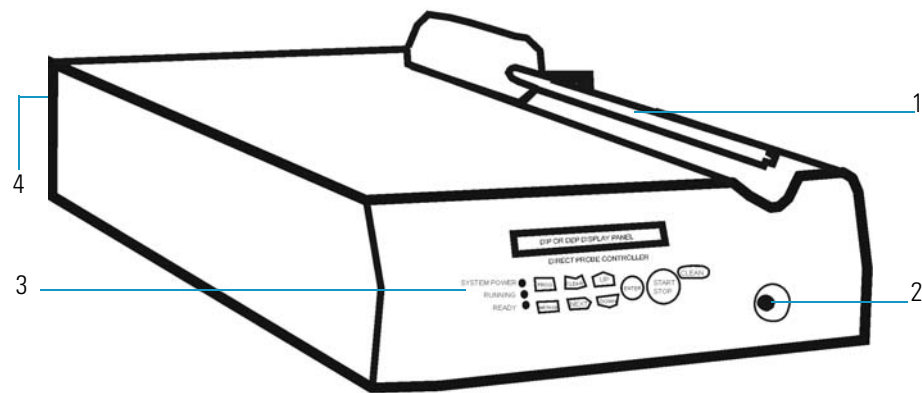
Use a DEP to rapidly introduce and analyze thermally labile or polar compounds that are otherwise not introduced using a DIP or a gas chromatograph (GC). A DEP is ideal for confirming the rapid molecular weight of solids dissolved in a suitable solvent. Typical samples may include:

- antibiotics
- complex lipids
- nucleosides
- saccharides
- small peptides
- surfactants
- unstable polymers

## How the Direct Probe System Works

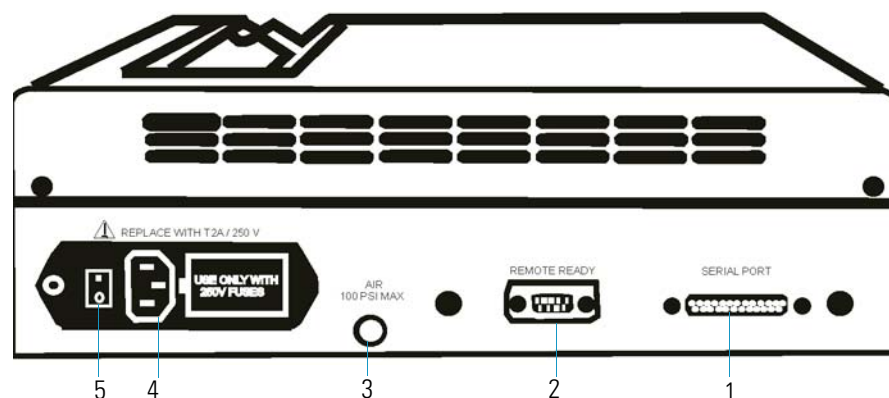
The Direct Probe System is a sample probe (Direct Insertion Probe [DIP] or Direct Exposure Probe [DEP]) that is connected to the Direct Probe Controller (DPC), which then connects to the mass spectrometer.

**Figure 34.** DPC and Sample Probe (Front View)



Item	Description	Part Number	Item	Description	Part Number
1	DIP or DEP Sample Probe	119300-ODIP or 119300-ODEP	3	DPC Keypad	
2	DPC External Cable to Probe	119303-0001	4	DPC Remote Start Cable to mass spectrometer ACCESSORY START, DB9F-DIN	119378-0032

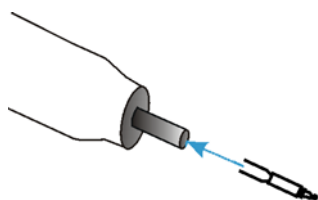
**Figure 35.** DPC (Back View)



1	Serial Port (not used)	4	Power Connection
2	REMOTE READY	5	Power Switch (0 is OFF,   is ON)
3	AIR (to cool Probe)		

The Direct Probe Controller (DPC) is the instrument used to communicate information from the sample probe (DIP or DEP) to the mass spectrometer. The DPC also allows you to enter heating and data acquisition parameters to the mass spectrometer. Direct probe systems allow rapid sample screening for any application that does not require a gas chromatograph (GC) column separation. Another advantage to using a DIP or a DEP is to have a wide range of samples to volatilize directly into the ion source.

**Figure 36.** DEP Tip and Filament



Using a DEP in CI mode is sometimes referred to as DCI (direct chemical ionization). Many thermally labile compounds may be vaporized or ionized before thermal decomposition occurs. Also, a higher evaporation temperature can be obtained than with other sample introduction techniques. Hence, one major benefit of using a DEP is to get molecular ion information not easily obtained from other techniques. The DEP is most often used with CI, although it works very well with EI. Depending upon the compound, the heat of the ion source alone may be sufficient to volatilize the sample from the filament. Alternatively, the probe filament may be heated very rapidly. The low thermal inertia of the filament allows rapid heating rates and high surface temperatures. Volatilization of the sample from the DEP filament occurs very rapidly in comparison to the heated sample cup of the DIP. As a result, less thermal degradation of the sample occurs.

## Analyzing Cholesterol with the Ion Trap Series

**Note** Ion Trap Series refers to both the PolarisQ and ITQ mass spectrometers.

Using an Ion Trap Series, analyzing sample involves these steps:

“Step 1: Connect the Direct Probe System” on page 47

“Step 2: Change the Ion Volume” on page 48

“Step 3: Deselect AS and GC Devices” on page 50

“Step 4: Tune and Calibrate the Mass Spectrometer” on page 51

“Step 5: Program the DPC” on page 51

“Step 6: Prepare the Sample” on page 53

“Step 7: Create a Method File” on page 54

“Step 8: Create a Sequence File” on page 55

“Step 9: Insert the Probe Using Xcalibur” on page 57

“Step 10: Remove the Probe Using Xcalibur” on page 59

“Step 11: Analyze the Data File” on page 60

Steps 1 through 3 are considered the initial setup. After the initial setup you may skip steps 1 through 3 and proceed to steps 4 through 11.



### Tools Needed

- ITQ mass spectrometer
- I/R tool and guide bar (included with mass spectrometer), PN 96000-60057
- Xcalibur Data System
- Operating System: Windows XP
- DPC Controller Assembly, PN 119300-0100
- DEP Kit (PN 119300-ODEP), includes the following:
  - (1 pkg) DEP Filaments (5), PN 119322-0001
  - (1) DIP/DEP Test Mix-10 mg/mL, PN 119343-0001
  - (2) Probe Ion Volume Holders, PN 119324-0001



### Frequency

As Needed



## Step 1: Connect the Direct Probe System

Before beginning your sample analysis, make sure the DPC is connected to the mass spectrometer and the sample probe is connected to the DPC. Then make sure all connections are fully seated.

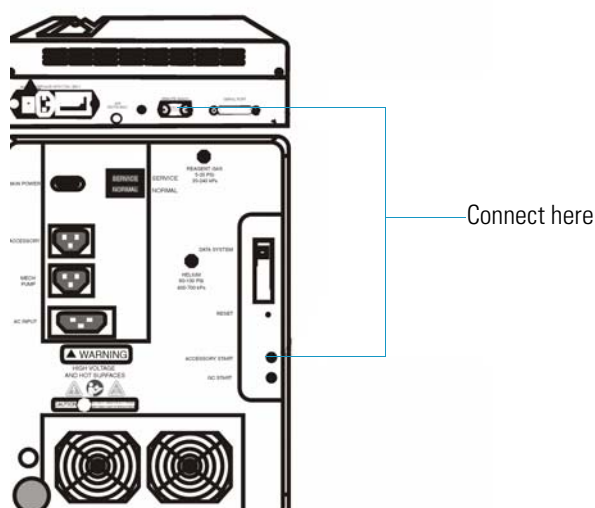
1. Place the DPC in a convenient location on top of or close to the mass spectrometer.
2. Read Section “[Top Safeguards to Know](#)” on [page 82](#) to be certain that you exercise all necessary safety precautions.



**WARNING ELECTRICAL SHOCK HAZARD.** Avoid exposing the DIP to electrical shock or electrical charging. Always plug the DIP cable into the DPC probe connector when performing EI or CI experiments, even if the probe is not heated. The DIP cable connection to the DPC provides electrical ground.

3. Connect the DPC to the mass spectrometer.

**Figure 37.** DPC to Mass Spectrometer (Back View)

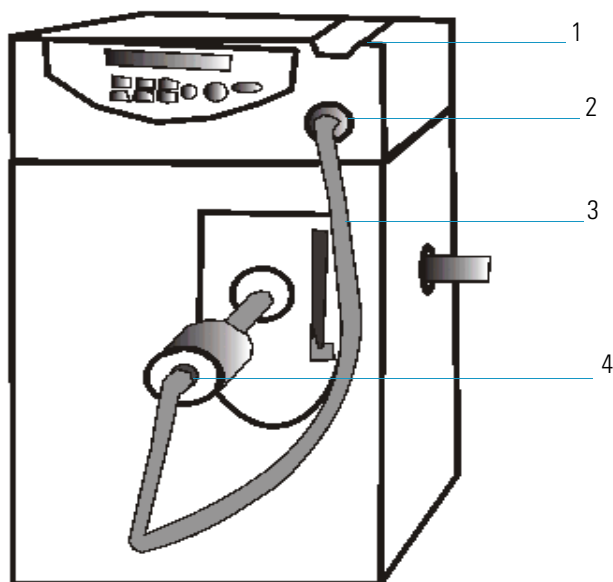


Connect the REMOTE START CABLE to DPC REMOTE READY and the mass spectrometer ACCESSORY START

- a. Connect the DPC to the mass spectrometer by using the DPC REMOTE START cable to connect into the DPC REMOTE READY connection and the other end to the mass spectrometer ACCESSORY START connection.
- b. Connect the power cord to the DPC power plug connection and the other end to the power source.

- c. Turn the DPC ON by switching the Power Switch |. Xcalibur automatically detect the probe and activates the Insert/Probe settings on the Home page, Tune window, and Sequence Editor.
4. Connect the DPC to the Probe.

**Figure 38.** DPC to Probe Connections



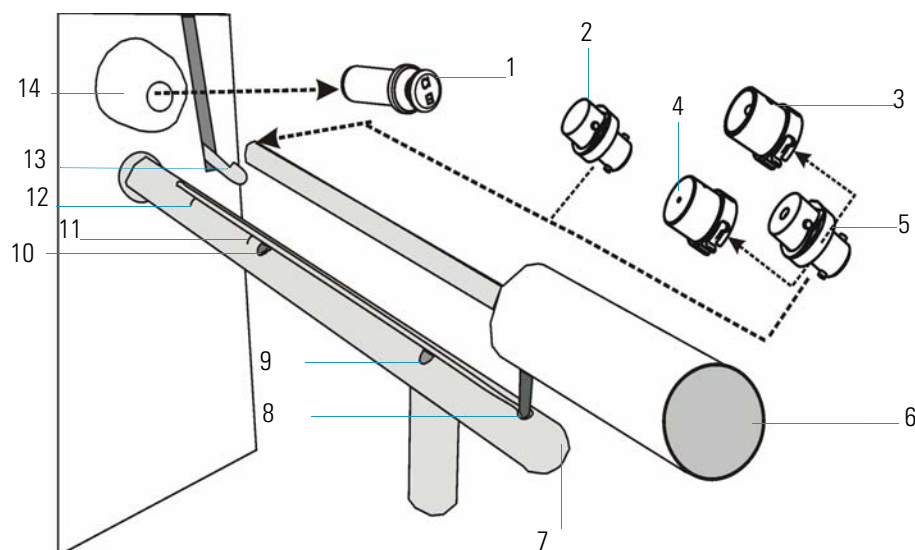
Item	Description	Item	Description	PN
1	DPC Cradle	3	DPC EXTERNAL Probe Cable	119303-0001
2	DPC Connection	4	Probe Connection	

- a. Plug one end of the DPC external cable to the DPC connection and the other end to the probe connection. Do this by matching the red dot located on the cable to the red dot located on the DPC and firmly pressing the cable in until it locks in position.
- b. Place the probe in the built-in cradle located on top of the DPC. Either the DIP or DEP can safely rest in the built-in cradle when not in use.
5. Turn on the MS, DPC, and the Xcalibur Data System software.
6. Confirm that the Insert/Remove Probe button displays on the Xcalibur Data System Home-Page. Typically, the software automatically detects if the probe is connected.

## Step 2: Change the Ion Volume

Samples can be analyzed in either EI or CI mode. Standard ion volumes are used using the probe ion volume holder included with your probe. The probe ion volume holder has a hole that allows the sample from the probe tip to enter the ion volume as compared to the GC ion volume holder, which allows sample to enter through the mass spectrometer transfer line.

**Figure 39.** I/R Tools



Item	Description	PN	Item	Description	PN
1	Inlet Valve Plug	119273-0001	8	Guide Ball Start Position	
2	GC Ion Volume Holder (standard)	70001-20532	9	1st Stop	
3	EI Ion Volume	119650-0220	10	2nd Stop	
4	CI Ion Volume	119650-0230	11	1st Etching	
5	Probe Ion Volume Holder	119324-0001	12	2nd Etching	
6	I/R Tool	96000-60057	13	Inlet Valve Lever	
7	Guide Bar	119687-0010	14	Inlet Valve (vacuum interlock chamber)	

1. Confirm that you are at the Xcalibur Home-Page.

**Note** Carefully follow each step on the Insert Remove Probe screen.

2. Remove the current ion volume.
  - a. From the Xcalibur Home Page select the Insert/Remove Probe button (hint: it's located on the Status tabs) to display the Insert Remove Probe screen.
  - b. Select the Insert Probe tab.
  - c. Attach the probe ion volume holder on the I/R tool.

## 2 Using a Direct Exposure Probe (DEP)

### Analyzing Cholesterol with the Ion Trap Series

**Note** When prompted on the Insert Remove Probe screen, insert the I/R tool into the guide bar start position using the guide bar to enter the inlet valve chamber and remove the current ion volume.

- d. Insert the guide bar into the mass spectrometer as illustrated in [Figure 39](#).
  - e. Follow each step listed on the Insert Probe tab to insert the probe and remove the current ion volume.
  - f. Select the Remove Probe tab and carefully follow each step to remove the probe.
3. Install an EI or CI ion volume.
    - a. Attach either an EI ion volume or CI ion volume to the probe ion volume holder while making sure it is tightly secured.
    - b. From the Xcalibur Home Page select the Insert/Remove Probe button to display the Insert Remove Probe screen.
    - c. Use the Insert Probe tab instructions to insert the probe install a CI or EI ion volume.
    - d. Use the Remove Probe tab instructions to remove the probe.
    - e. Close the Insert Remove Probe screen and return to the Xcalibur Data System Home-Page.

## Step 3: Deselect AS and GC Devices

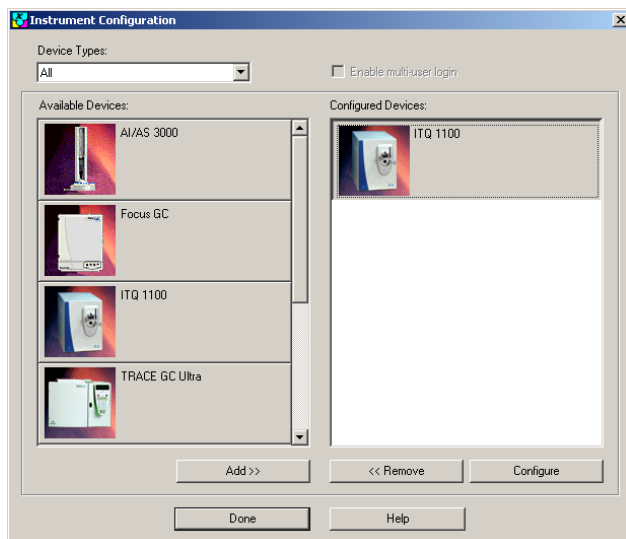
If you are running several probe runs, deselect the AS and GC instruments in the Xcalibur Instrument Configuration software. Configure for the mass spectrometer only when analyzing samples with the probe. Deselecting the AS and GC simplifies the methods and avoids waiting on GC equilibration time before every run. However, it is not necessary.

1. Start Xcalibur Instrument Configuration.



From the Windows desktop, select the Instrument Configuration shortcut to display the Instrument Configuration screen.

**Figure 40.** Instrument Configuration: Ion Trap Series



2. Remove the AS and GC devices from Configured Devices.

Select the item to be removed from the Configured Devices section and click the Remove button. We recommend using the Ion Trap and DSQ Series configuration only when analyzing samples with the probe.

3. Click Done to return to the Windows desktop.

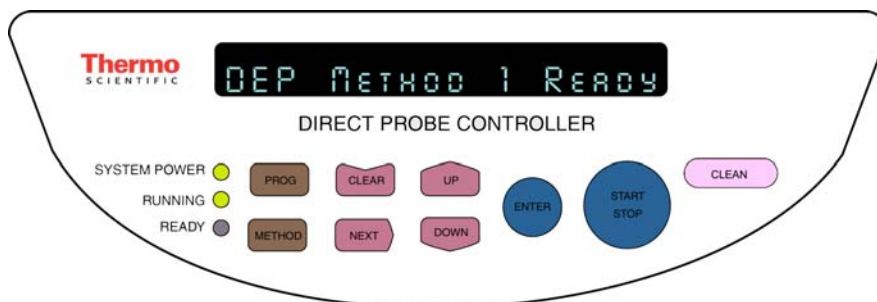
## Step 4: Tune and Calibrate the Mass Spectrometer

Refer to the Tune online help for instructions in determining if the instrument has been recently tuned and calibrated.

## Step 5: Program the DPC

1. Turn the DPC ON using the switch located on the back of the DPC for the DPC panel to display DEP Method 1 Ready.

**Figure 41.** DEP DPC Display Pane



The DPC automatically detects if a DIP or DEP is connected, and will display the DIP or DEP series of menu controls for setting the temperature and for heating the probe tip.

2. Enter the initial current.
  - a. Press METHOD to display Initial Curr.
  - b. Press UP or DOWN until you see Curr: 0 mA.
3. Enter the initial time.
  - a. Press NEXT to display Initial Time.
  - b. Press UP or DOWN to display Initial Time of 30 seconds.
4. Enter the ramp 1 rate.
  - a. Press NEXT to display Ramp1 Rate.
  - b. Press UP or DOWN to set the Ramp Rate to 20 mAs.
5. Enter the ramp 1 current.
  - a. Press NEXT to display Ramp 1 Curr.
  - b. Press UP or DOWN until the Ramp 1 Current is 1000 mA. This sets the ramp final current to 1000 mA.
6. Enter the ramp 1 hold time.
  - a. Press NEXT to display Ramp1 Hold.
  - b. Press UP or DOWN until the Ramp 1 Hold time displays 30 seconds.
  - c. Press NEXT to display DEP Method 1 Ready.
7. Load the method.

Press PROG to display Load Method: X<sup>1</sup>.
8. Save the method.
  - a. Press NEXT to display Save Method: X<sup>1</sup>.
  - b. Press Up or Down to enter the desired method number.
  - c. Press ENTER to return to DEP Method.
  - d. Listen for the DPC to beep three times to indicate that your current method configuration is saved and stored in the DPC as Method 1, and may be recalled by loading Method 1.

❖ **To retrieve methods**

1. Press the Prog key to display Load Method: X<sup>1</sup>.

---

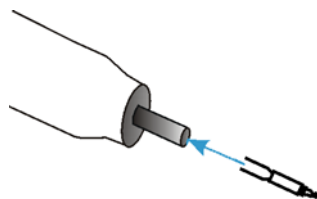
<sup>1</sup>X refers to methods numbered 1 - 9.

2. Press Up or Down to locate the desired method number.
3. Press Enter when you see the desired number. This loads the method.

## Step 6: Prepare the Sample

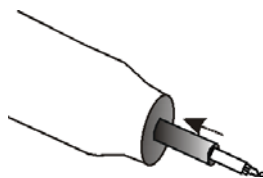
1. Insert the filament on the DEP tip.

**Figure 42.** Before Inserting the Filament



- a. Using a pair of clean small needle nose pliers or a pair of tweezers, carefully grip the two filament mounting leads just below the ceramic portion of the filament.
- b. Align the filament mounting leads with the two holes located at the DEP tip.
- c. Push the filament mounting leads into the holes. Be careful not to allow the mounting leads to turn. This causes the filament to mount to one side of the probe center.
- d. Once the filament mounting leads are inside the holes, release the grip on the filament leads and grip the ceramic with the pliers or tweezers.
- e. Push the ceramic inside the probe tip until the filament is recessed into the probe end.

**Figure 43.** After Inserting the Filament



- f. Inspect the filament to be sure it is centered into the end of the probe.

Any misalignment prevents the filament from entering the hole in the ion volume holder. If the filament is not centered, you will have to remove the filament, correct the alignment, and reinsert the filament.

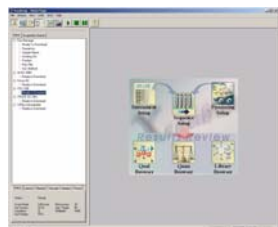
The filament ceramic should be recessed into the probe end as illustrated in [Figure 43](#). If it is not, the filament may extend too far beyond the probe end. This may cause the filament to come in contact with the ball valve in the vacuum interlock. If the filament does not recess into the end of the probe, check to be sure the two pins inside the end of the probe are not extending beyond the end of the white insulator that holds the pins in place.

## 2 Using a Direct Exposure Probe (DEP)

### Analyzing Cholesterol with the Ion Trap Series

2. Use a 10  $\mu\text{L}$  syringe to place 1  $\mu\text{L}$  of the 10  $\mu\text{L}/\text{mL}$  cholesterol solution on the filament assembly.
3. Wait several minutes for all of the solvent to evaporate. It takes several minutes to evaporate the sample, giving you ample time to set up a method (“[Step 7: Create a Method File](#)” on [page 54](#)) and a sequence file (“[Step 8: Create a Sequence File](#)” on [page 55](#)).

## Step 7: Create a Method File

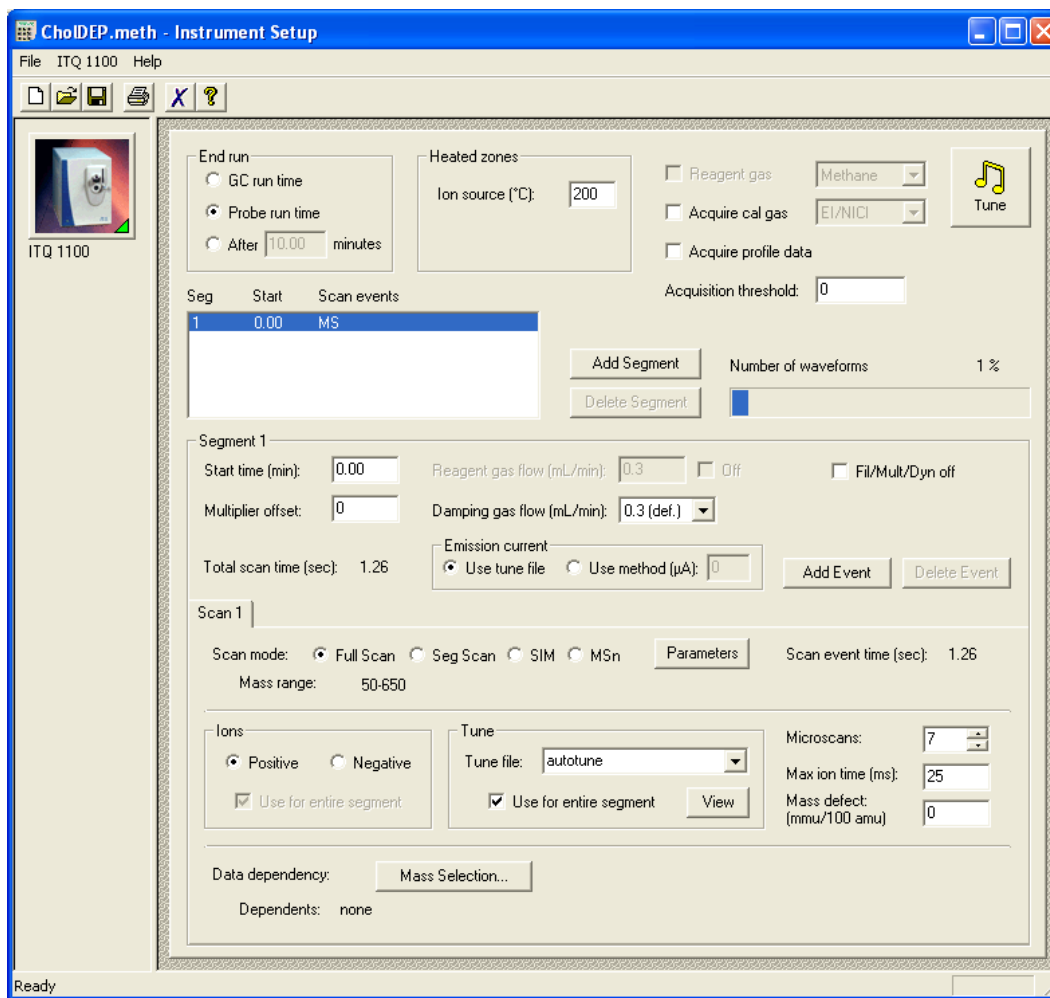


1. Create a method using Xcalibur Instrument Setup.
  - a. From the Windows desktop, click the Xcalibur icon to display the Xcalibur Home Page.
  - b. Select Instrument Setup to display the Instrument Setup window.
2. Enter the instrument method parameters.
  - a. Select File | New to create a new method.



- b. Enter the method settings shown in Figure 44.

**Figure 44.** Instrument Setup: Ion Trap Series CholDEP Method



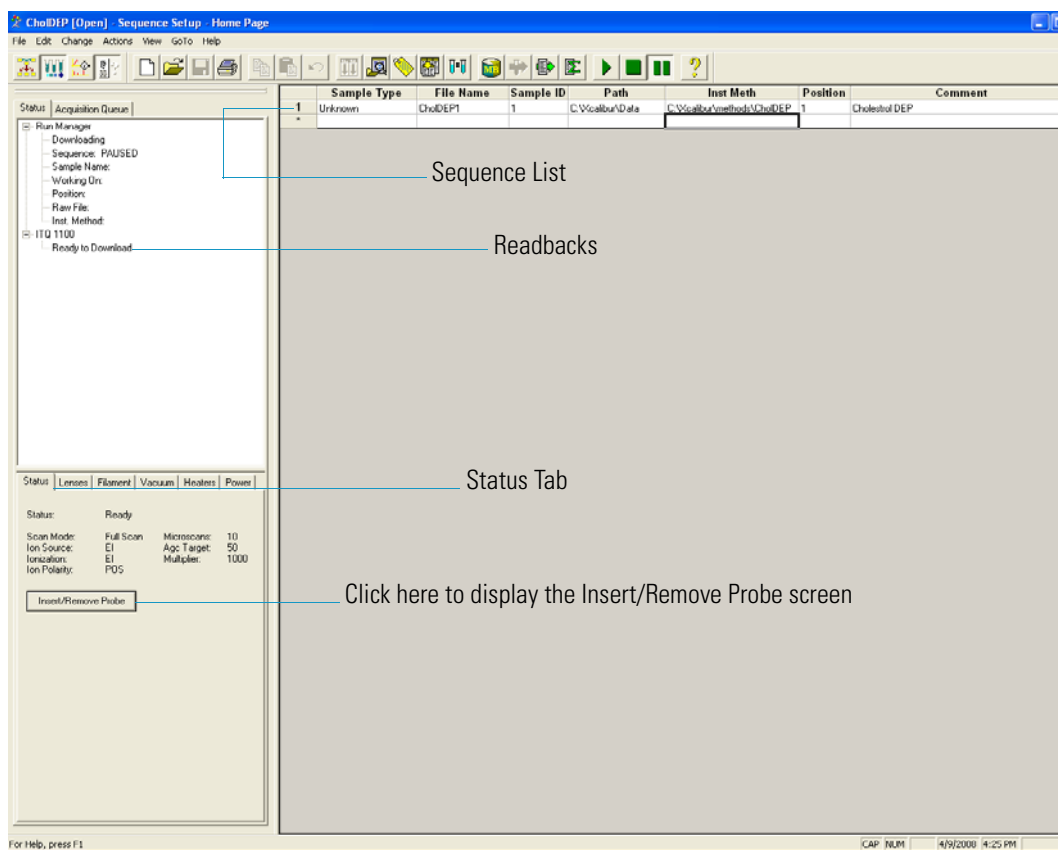
3. Save the method.
  - a. Select File | Save to display the Save As screen.
  - b. Enter CholDEP for the cholesterol method and click Save. The software automatically adds the \*.meth extension to any method file.

## Step 8: Create a Sequence File

1. Got to the Sequence Setup window, by selecting Sequence Setup from the Xcalibur Home Page.
2. Set up a sequence file.
  - a. Select File | New to display a blank sequence file.

- b. Enter the settings displayed in Figure 45.

**Figure 45.** Sequence Setup: Ion Trap Series CholDEP Sequence



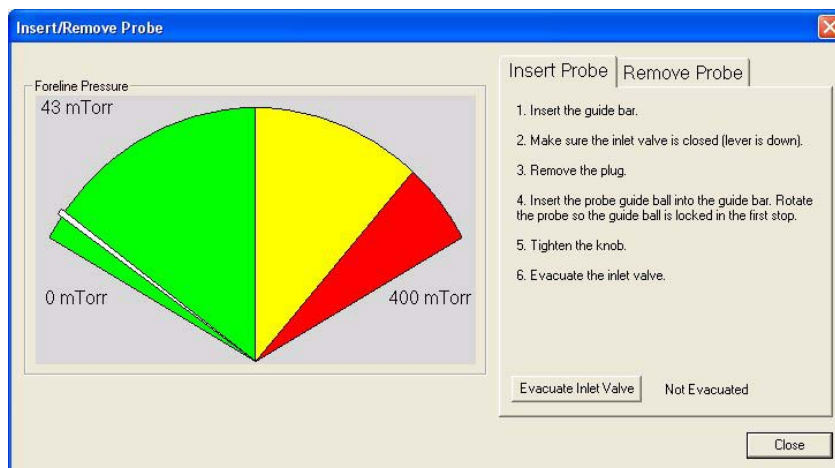
**Note** You can type filenames in all uppercase or lowercase. Ion Trap and DSQ Series filenames are not case-sensitive.

3. Save the sequence file.
  - a. Select File | Save to display the Save As screen.
  - b. Enter CholDEP as the new sequence file and click Save to return to Sequence Setup. Xcalibur automatically adds the \*.sld extension to the sequence filename.

## Step 9: Insert the Probe Using Xcalibur

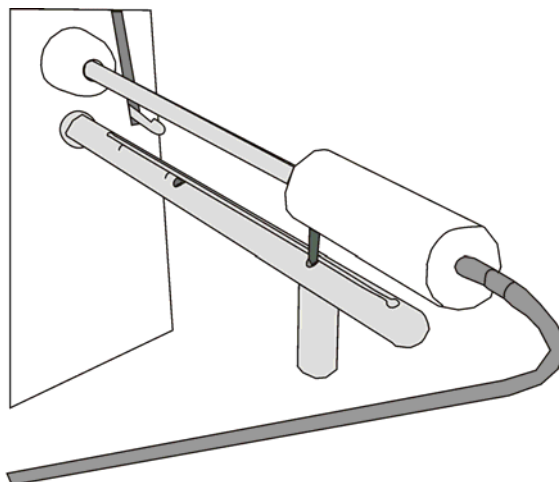
1. Select the Insert / Remove Probe button from the Sequence Setup Status tab to display the Insert/Remove Probe screen.

**Figure 46.** Insert/Remove Probe Screen: Ion Trap Series



- a. Follow the screen instructions on the Insert Probe tab.
- b. At step 4 on the screen instructions, slide the probe into the inlet valve chamber until the stem of the probe shaft reaches the 1st stop on the guide bar.

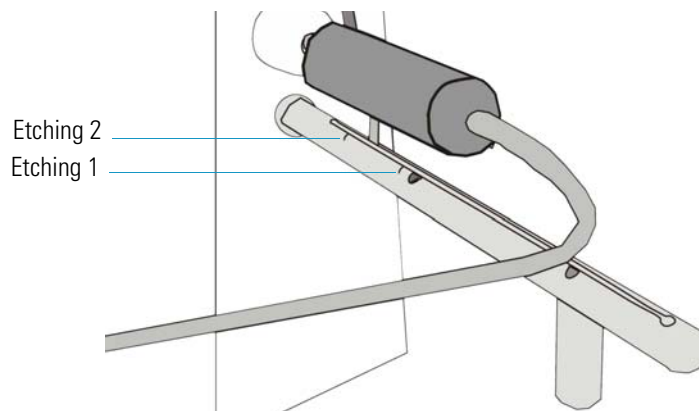
**Figure 47.** Sample Probe at 1st Stop



- c. At step 6 on the screen instructions, click the Evacuate Inlet Valve button that is located on the Insert Remove Probe screen.
- d. Wait to see the Inlet Valve Message prompt you to insert the probe further into the chamber.
- e. Open the inlet valve by lifting the inlet valve lever up.

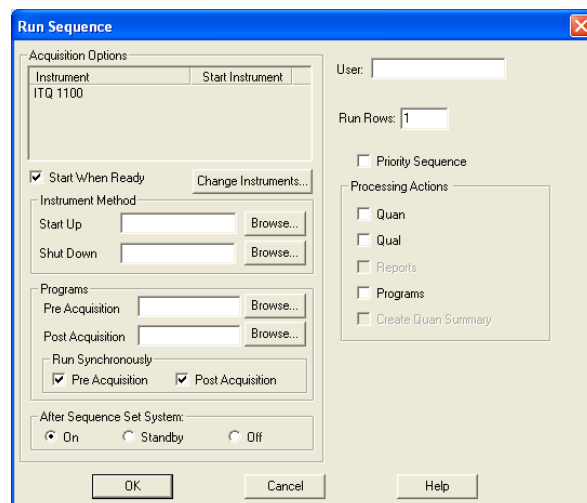
- f. Slide the probe into the chamber until the probe is all the way in the ion volume holder. The stem of the probe shaft is about 15 mm from the 2nd etching on the guide bar.

**Figure 48.** Sample Probe before the 2nd Etching



- g. Click the Close button located on the Insert/Remove Probe screen.
2. Run the saved sequence.
    - a. Highlight the line and sequence you wish to run.
    - b. Select Actions | Run This Sample to display the Run Sequence screen.

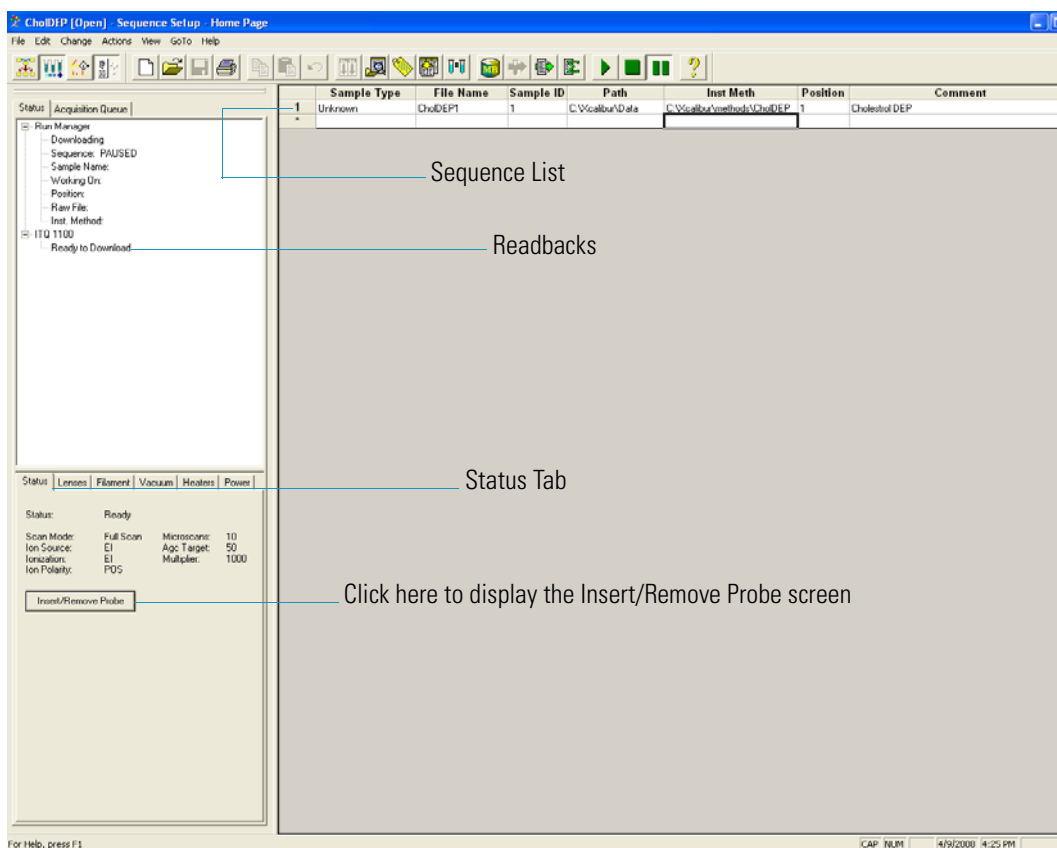
**Figure 49.** Run Sequence Screen: Ion Trap Series



- c. Click OK to return to the Sequence Setup window.

3. Look at the Status tab to read the readbacks (Figure 16).

**Figure 50.** Sequence Setup: Ion Trap Series ChOI DEP Sequence



- When you see the readback Waiting for Contact Closure, press the Start button on the DPC panel to run the sample.
- Look for the readback to display Running. If it doesn't verify that the DPC plug is firmly connected to the back of the mass spectrometer.
- Select View | Real Time Plot View from the Sequence Setup window to monitor the run.
- The run is completed when the readback displays Ready to Download and the view displays No DataFile.
- Go to ["Step 10: Remove the Probe Using Xcalibur"](#) on [page 59](#).

## Step 10: Remove the Probe Using Xcalibur

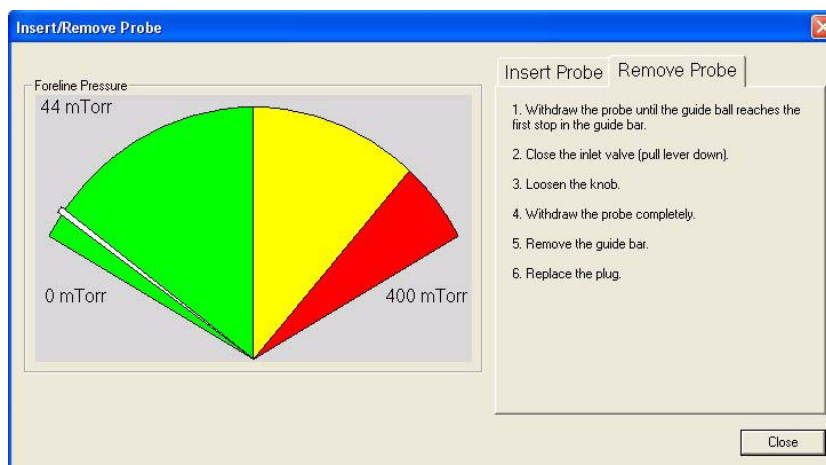
You should still be at the Sequence Setup window. In this step you remove the probe to prepare for the next analysis.



**CAUTION INSTRUMENT DAMAGE.** Avoid accidentally venting the system. Do not remove the probe beyond the point at which the pin in the probe handle reaches the stop in the guide bar. Do not completely withdraw the probe until the ball valve has been closed. Otherwise, the system vents to the atmosphere.

1. Select the Insert/Remove Probe button again from the Status tab.
  - a. Select the Remove Probe tab and *carefully* follow Steps 1–6.

**Figure 51.** Remove Probe Tab: Ion Trap Series



- b. Click the Close button to return to the Sequence Setup window.
    - c. Begin the next step.

## Step 11: Analyze the Data File

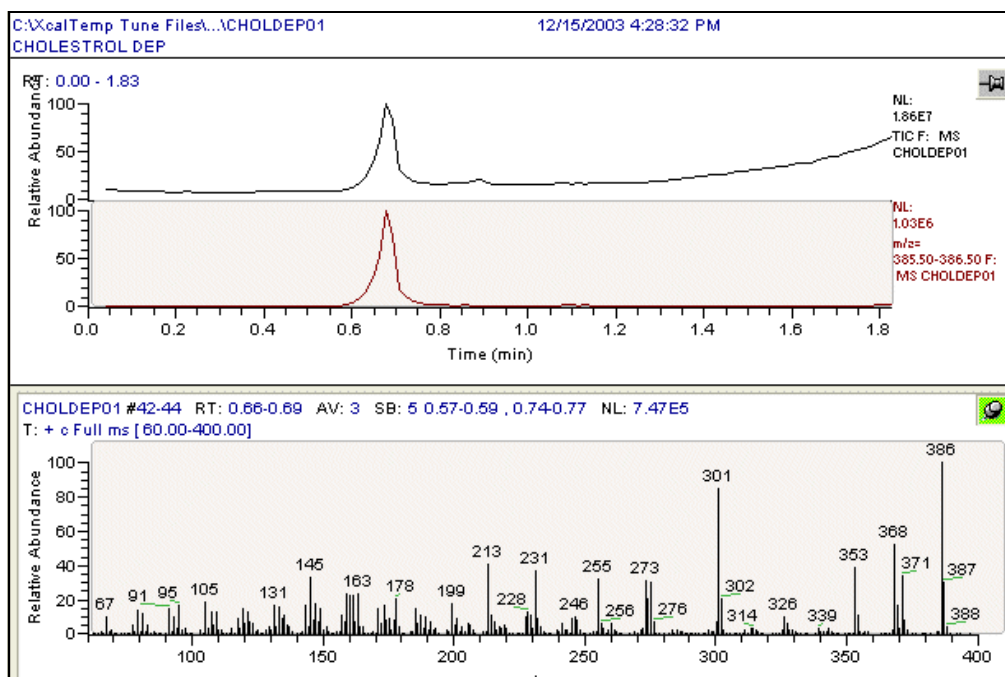
Use these steps to process the data file you just acquired. If you want to analyze another cholesterol sample, repeat the steps listed under “[Step 6: Prepare the Sample](#)” on [page 53](#), making the appropriate addition to the sequence table.

1. At the Sequence Setup window, click the Roadmap icon to return to the Xcalibur Home Page.

Select Qual Browser to open the Qual Browser window. Refer to the Qual Browser manual to learn how to use Qual Browser techniques.
2. Select File | Open and select the CholDEP.raw data file you just ran.

Compare your results with this Qual Browser example.

**Figure 52.** Qual Browser: Ion Trap Series DEP



For information on how to load samples see “[Loading DEP Samples](#)” on [page 76](#), clean the probe tip see “[Cleaning the DEP](#)” on [page 78](#), and terms to know see “[Top Terms to Know](#)” on [page 81](#).

# Analyzing Cholesterol with the DSQ Series

**Note** DSQ Series refers to both the DSQ II and DSQ mass spectrometers.

Using a DSQ, analyzing sample involves these steps:

“Step 1: Connect the Direct Probe System” on page 63

“Step 2: Change the Ion Volume” on page 64

“Step 3: Deselect AS and GC Devices” on page 66

“Step 4: Tune and Calibrate the Mass Spectrometer” on page 67

“Step 5: Program the DPC” on page 67

“Step 6: Prepare the Sample” on page 69

“Step 7: Create a Method File” on page 70

“Step 8: Create a Sequence File” on page 71

“Step 9: Insert the Probe Using Xcalibur” on page 72

“Step 10: Remove the Probe Using Xcalibur” on page 74

“Step 11: Analyze the Data File” on page 75

Steps 1 through 3 are considered the initial setup. After the initial setup you may skip steps 1 through 3 and proceed to steps 4 through 11.



### Tools Needed

- Ion Trap Series or DSQ mass spectrometer
- I/R tool and guide bar (included with mass spectrometer), PN 96000-60057
- Xcalibur Data System
- Operating System: Windows XP
- DPC Controller Assembly, PN 119300-0100

DEP Kit (PN 119300-ODEP), includes the following:

- (1 pkg) DEP Filaments (5), PN 119322-0001
- (1) DIP/DEP Test Mix-10 mg/mL, PN 119343-0001
- (2) Probe Ion Volume Holders, PN 119324-0001



### Frequency

As Needed



## Step 1: Connect the Direct Probe System

Before beginning your sample analysis, verify that the DPC is connected to the mass spectrometer and the sample probe is connected to the DPC. Then make sure all connections are fully seated.

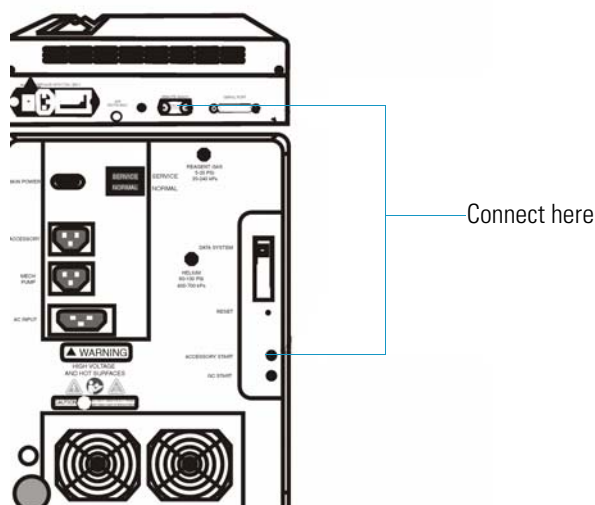
1. Place the DPC in a convenient location on top of or close to the mass spectrometer.
2. Read Section “[Top Safeguards to Know](#)” on [page 82](#) to be certain that you exercise all necessary safety precautions.



**CAUTION INSTRUMENT DAMAGE.** Avoid electrical shock or electrical charging of the DIP. Always plug the DIP cable into the DPC probe connector when performing EI or CI experiments, even if the probe is not heated. This is because the DIP cable connection to the DPC provides electrical ground.

3. Connect the DPC to the mass spectrometer.

**Figure 53.** DPC to Mass Spectrometer (Back View)

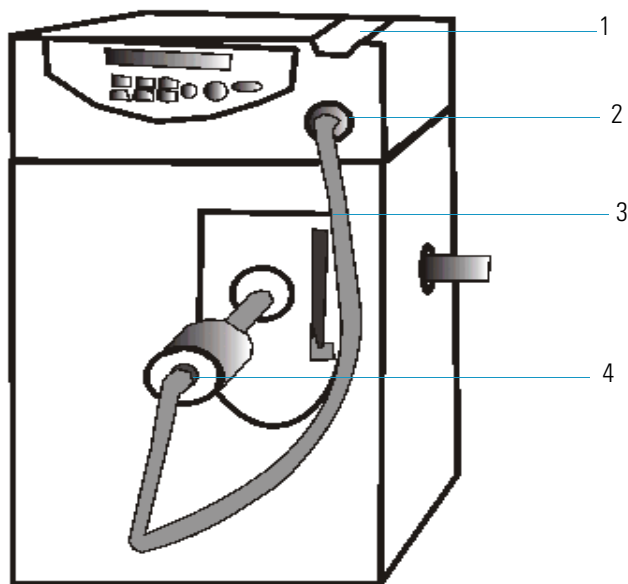


Connect the REMOTE START CABLE to DPC REMOTE READY and the mass spectrometer ACCESSORY START

- a. Connect the DPC to the mass spectrometer by using the DPC REMOTE START cable to connect into the DPC REMOTE READY connection and the other end to the mass spectrometer ACCESSORY START connection.
- b. Connect the power cord to the DPC power plug connection and the other end to the power source.
- c. Turn the DPC ON by switching the Power Switch |. Xcalibur automatically detect the probe and activates the Insert/Probe settings on the Home page, Tune window, and Sequence Editor.

4. Connect the DPC to the Probe (Figure 54).

**Figure 54.** DPC to Probe Connections



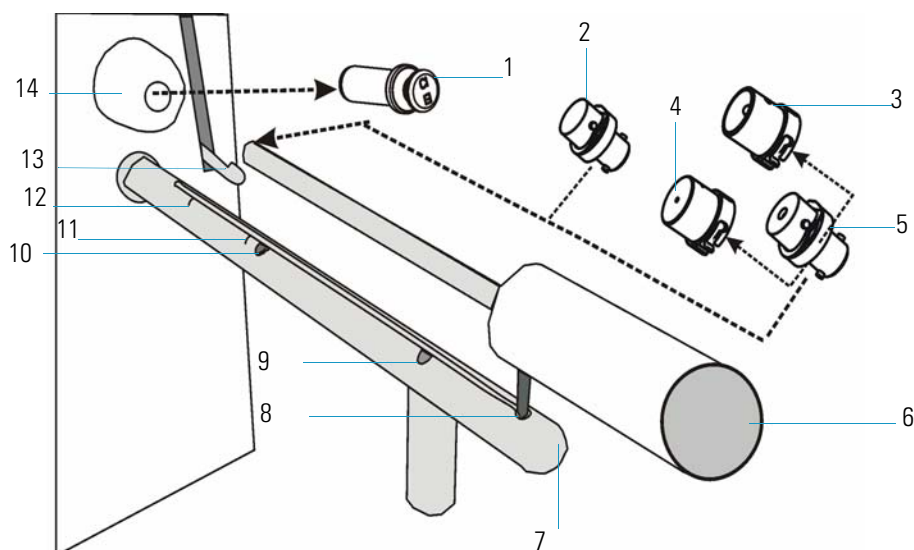
Item	Description	PN	Item	Description	PN
1	DPC cradle		3	DPC EXTERNAL probe cable	119303-0001
2	DPC connection		4	Probe connection	

- a. Plug one end of the DPC external cable to the DPC connection and the other end to the probe connection. Do this by matching the red dot located on the cable to the red dot located on the DPC and firmly pressing the cable in until it locks in position.
  - b. Place the probe in the built-in cradle located on top of the DPC. Either the DIP or DEP can safely rest in the built-in cradle when not in use.
5. Turn on the MS, DPC, and the Xcalibur Data System software.
  6. Confirm that the Insert/Remove Probe button displays on the Xcalibur Data System Home-Page. Typically, the software automatically detects if the probe is connected.

## Step 2: Change the Ion Volume

Samples can be analyzed in either EI or CI mode. Standard ion volumes are used using the probe ion volume holder included with your probe. The probe ion volume holder has a hole that allows the sample from the probe tip to enter the ion volume as compared to the GC ion volume holder, which allows sample to enter through the mass spectrometer transfer line.

**Figure 55.** I/R Tools



Item	Description	PN	Item	Description	PN
1	Inlet Valve plug	119273-0001	8	Guide Ball start position	
2	GC Ion Volume holder (standard)	70001-20532	9	1st stop	
3	EI Ion Volume	119650-0220	10	2nd stop	
4	CI Ion Volume	119650-0230	11	1st etching	
5	Probe Ion Volume holder	119324-0001	12	2nd etching	
6	I/R tool	96000-60057	13	Inlet Valve lever	
7	Guide Bar	119687-0010	14	Inlet Valve (vacuum interlock chamber)	

1. Confirm that you are at the Xcalibur Home-Page.

**Note** Carefully follow each step on the Insert Remove Probe screen.

2. Remove the current ion volume.

- From the Xcalibur Home Page select the Insert/Remove Probe button (Hint: it's located on the Status tabs) to display the Insert Remove Probe screen.
- Select the Insert Probe tab.
- Attach the Probe ion volume holder on the I/R tool.

## 2 Using a Direct Exposure Probe (DEP)

Analyzing Cholesterol with the DSQ Series

**Note** When prompted on the Insert Remove Probe screen, insert the I/R tool into the guide bar start position using the guide bar to enter the inlet valve chamber and remove the current ion volume.

- d. Insert the guide bar into the mass spectrometer as illustrated in [Figure 55](#).
  - e. Follow each step listed on the Insert Probe tab to insert the probe and remove the current ion volume.
  - f. Select the Remove Probe tab and carefully follow each step to remove the probe.
3. Install an EI or CI ion volume.
    - a. Attach either an EI ion volume or CI ion volume to the probe ion volume holder while making sure it is tightly secured.
    - b. From the Xcalibur Home Page select the Insert/Remove Probe button to display the Insert Remove Probe screen.
    - c. Use the Insert Probe tab instructions to insert the probe install a CI or EI ion volume.
    - d. Use the Remove Probe tab instructions to remove the probe.
    - e. Close the Insert Remove Probe screen and return to the Xcalibur Data System Home-Page.

### Step 3: Deselect AS and GC Devices

If you are running several probe runs, deselect the AS and GC instruments in the Xcalibur Instrument Configuration software. Configure for the mass spectrometer only when analyzing samples with the probe. Deselecting the AS and GC simplifies the methods and avoids waiting on GC equilibration time before every run. However, it is not necessary.

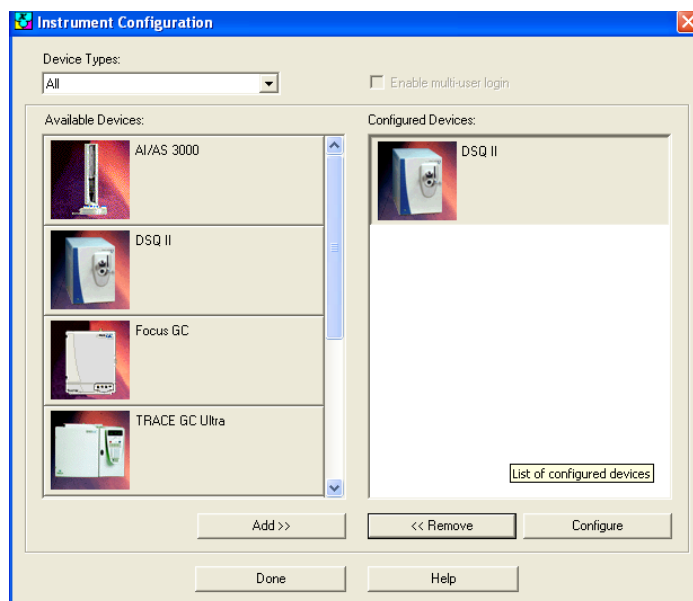
1. Start Xcalibur Instrument Configuration.



From the Windows desktop, select the Instrument Configuration shortcut to display the Instrument Configuration screen.

2. Remove the AS and GC devices from Configured Devices.

**Figure 56.** Instrument Configuration: DSQ Series



Select the item to be removed from the Configured Devices section and click the Remove button. We recommend using the Ion Trap and DSQ Series configuration only when analyzing samples with the probe.

3. Click Done to return to the Windows desktop.

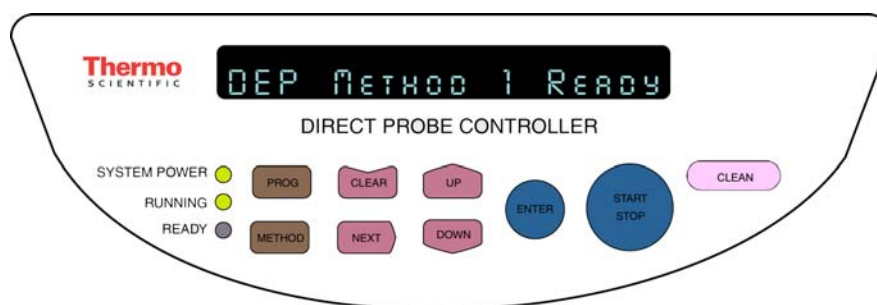
## Step 4: Tune and Calibrate the Mass Spectrometer

Refer to the Tune online help for instructions in determining if the instrument has been recently tuned and calibrated.

## Step 5: Program the DPC

1. Turn the DPC ON using the switch located on the back of the DPC for the DPC panel to display DEP 1 Ready.

**Figure 57.** DEP DPC Display Panel



## 2 Using a Direct Exposure Probe (DEP)

### Analyzing Cholesterol with the DSQ Series

The DPC automatically detects if a DIP or DEP is connected, and will display the DIP or DEP series of menu controls for setting the temperature and for heating the probe tip.

2. Enter the initial current.
  - a. Press METHOD to display Initial Curr.
  - b. Press UP or DOWN until you see Curr: 0 mA.
3. Enter the initial time.
  - a. Press NEXT to display Initial Time.
  - b. Press UP or DOWN to display the Initial Time of 30 seconds.
4. Enter the ramp 1 rate.
  - a. Press NEXT to display Ramp1 Rate.
  - b. Press UP or DOWN to set the Ramp Rate to 20 mAs.
5. Enter the ramp 1 current.
  - a. Press NEXT to display Ramp 1 Curr.
  - b. Press UP or DOWN until the Ramp 1 Current is 1000 mA. This sets the ramp final current to 1000 mA.
6. Enter the ramp 1 hold time.
  - a. Press NEXT to display Ramp1 Hold.
  - b. Press UP or DOWN until the Ramp 1 Hold Time displays 30 seconds.
  - c. Press NEXT to display DEP Method 1 Ready.
7. Load the method.

Press PROG to display Load Method: X.
8. Save the method.
  - a. Press NEXT to display Save Method: X<sup>1</sup>.
  - b. Press Up or Down to enter the desired method number.
  - c. Press ENTER to return to DEP Method.
  - d. Listen for the DPC to beep three times to indicate that your current method configuration is saved and stored in the DPC as Method 1, and may be recalled by loading Method 1.

#### ❖ To retrieve methods

1. Press the Prog key to display Load Method: X<sup>1</sup>.

---

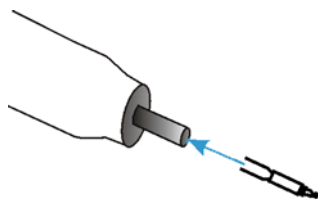
<sup>1</sup>X refers to the methods numbered 1 - 9.

2. Press Up or Down to locate the desired method number.
3. Press Enter when you see the desired number. This loads the method.

## Step 6: Prepare the Sample

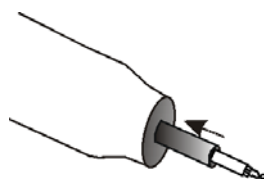
1. Insert the filament on the DEP tip.

**Figure 58.** Before Inserting Filament



- a. Using a pair of clean small needle nose pliers or a pair of tweezers, carefully grip the two filament mounting leads just below the ceramic portion of the filament.
- b. Align the filament mounting leads with the two holes located at the DEP tip.
- c. Push the filament mounting leads into the holes. Be careful not to allow the mounting leads to turn. This causes the filament to mount to one side of the probe center.
- d. Once the filament mounting leads are inside the holes, release the grip on the filament leads and grip the ceramic with the pliers or tweezers.
- e. Push the ceramic inside the probe tip until the filament is recessed into the probe end.

**Figure 59.** After Inserting Filament



- f. Inspect the filament to be sure it is centered into the end of the probe.

Any misalignment prevents the filament from entering the hole in the ion volume holder. If the filament is not centered, you will have to remove the filament, correct the alignment, and reinsert the filament.

The filament ceramic should be recessed into the probe end as illustrated in [Figure 59](#). If it is not, the filament may extend too far beyond the probe end. This may cause the filament to come in contact with the ball valve in the vacuum interlock. If the filament does not recess into the end of the probe, check to be sure the two pins inside the end of the probe are not extending beyond the end of the white insulator that holds the pins in place.

## 2 Using a Direct Exposure Probe (DEP)

### Analyzing Cholesterol with the DSQ Series

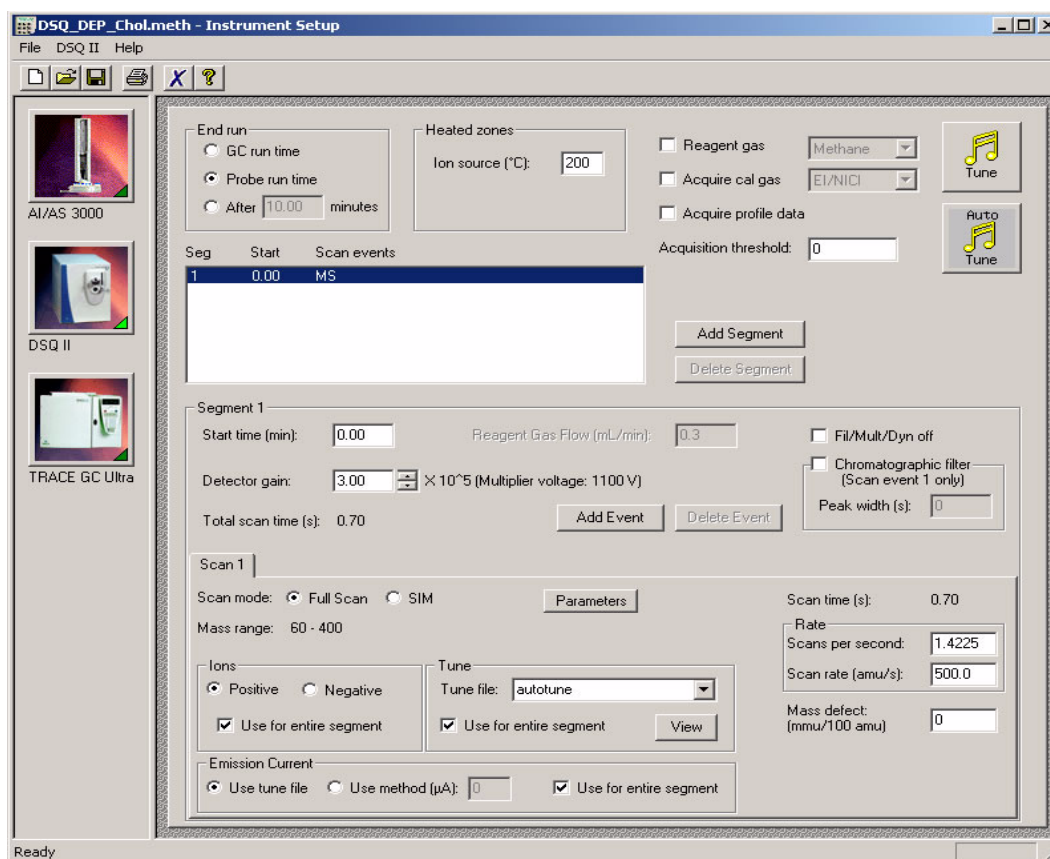
2. Use a 10  $\mu\text{L}$  syringe to place 1  $\mu\text{L}$  of the 10  $\mu\text{L}/\text{mL}$  cholesterol solution on the filament assembly.
3. Wait several minutes for all of the solvent to evaporate. It takes several minutes to evaporate the sample, giving you ample time to set up a method and a sequence file (“[Step 7: Create a Method File](#)” on [page 70](#) and “[Step 8: Create a Sequence File](#)” on [page 71](#)).

## Step 7: Create a Method File



1. Create a method using Xcalibur Instrument Setup.
  - a. From the Windows desktop, click the Xcalibur icon to display the Xcalibur Home Page.
  - b. Select Instrument Setup to display the Instrument Setup window.
2. Enter the instrument method parameters.
  - a. Select File | New to create a new method.
  - b. Enter the method settings shown in [Figure 60](#).

**Figure 60.** Instrument Setup: DSQ Series ChIDEP Method



3. Save the method.
  - a. Select File | Save to display the Save As screen.

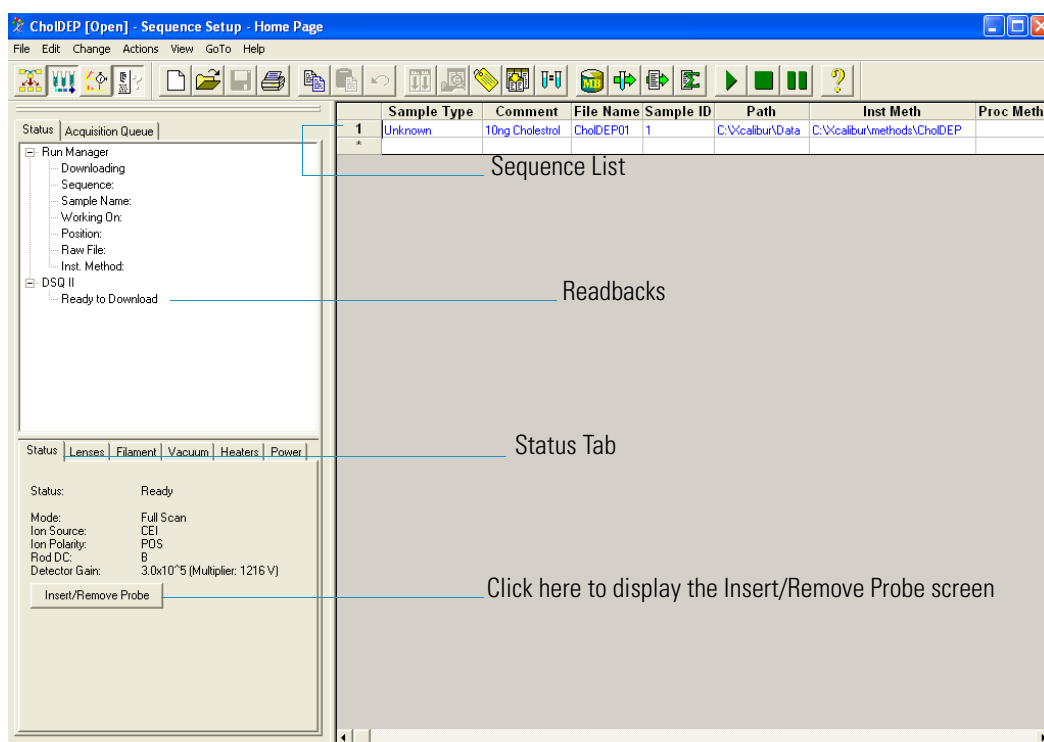


- b. Enter CholDEP for the cholesterol method and click Save. The software automatically adds the \*.meth extension to any method file.

## Step 8: Create a Sequence File

1. Got to the Sequence Setup window, by selecting Sequence Setup from the Xcalibur Home Page.
2. Set up a sequence file.
  - a. Select File | New to display a blank sequence file.
  - b. Enter the settings displayed in Figure 61.

**Figure 61.** Sequence Setup: DSQ Series CholDEP Sequence



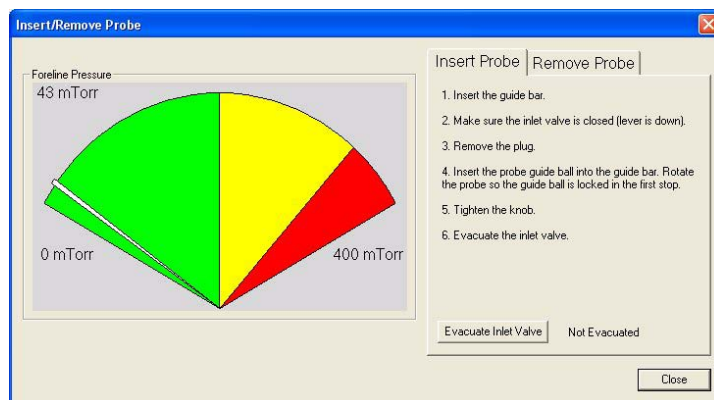
**Note** You can type filenames in all uppercase or lowercase. Ion Trap and DSQ Series filenames are not case-sensitive.

3. Save the sequence file.
  - a. Select File | Save to display the Save As screen.
  - b. Enter CholDEP as the new sequence file and click Save to return to Sequence Setup. Xcalibur automatically adds the \*.sld extension to the sequence filename.

## Step 9: Insert the Probe Using Xcalibur

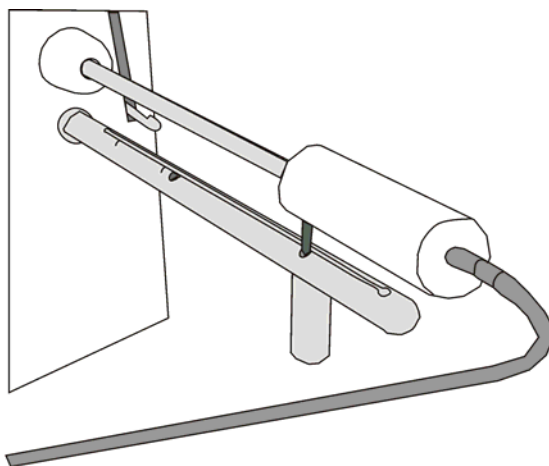
1. Select the Insert / Remove Probe button from the Sequence Setup Status tab to display the Insert/Remove Probe screen.

**Figure 62.** Insert/Remove Probe Screen: DSQ Series



- a. Follow the screen instructions on the Insert Probe tab.
- b. At step 4 on the screen instructions, slide the probe into the inlet valve chamber until the stem of the probe shaft reaches the 1st stop on the guide bar.

**Figure 63.** Sample Probe at 1st Stop

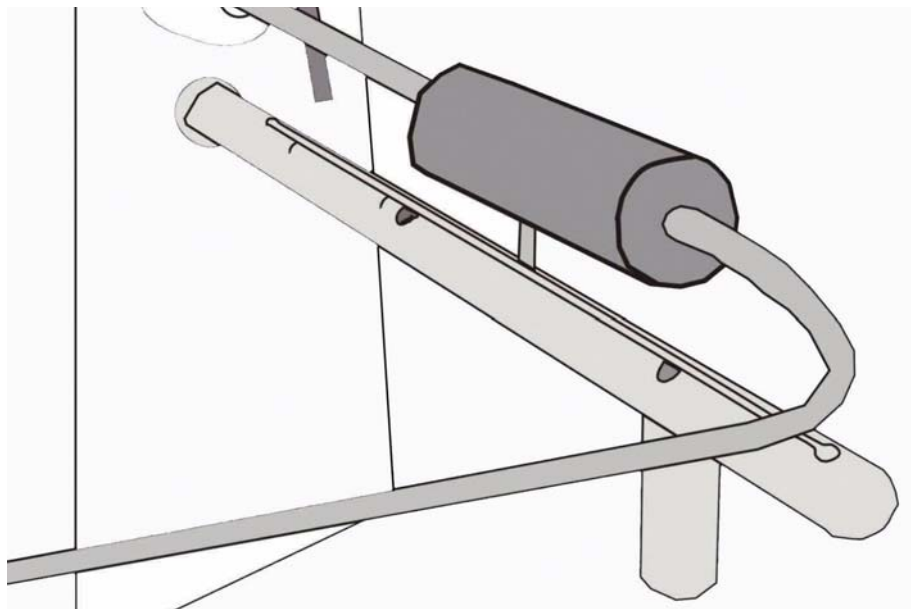


- c. At step 6, click the Evacuate Inlet Valve button that is located on the Xcalibur Insert Remove Probe screen.
- d. Wait to see the Inlet Valve Message prompt you to insert the probe further into the chamber.
- e. Open the inlet valve by lifting the inlet valve lever up.



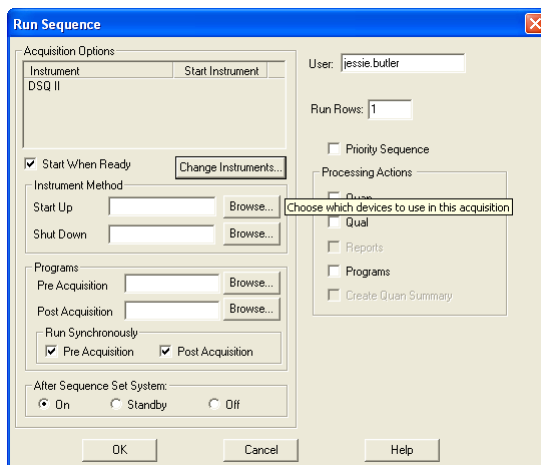
- f. Slide the probe into the chamber until the probe is all the way into the ion volume holder and the stem of the probe shaft *ALMOST* reaches the 2nd Stop on the guide bar.

**Figure 64.** Sample Probe before the 2nd Stop



- g. Click the Close button located on the Insert/Remove Probe screen.
2. Run the saved sequence.
    - a. Highlight the line and sequence you wish to run.
    - b. Select Actions | Run This Sample to display the Run Sequence screen.

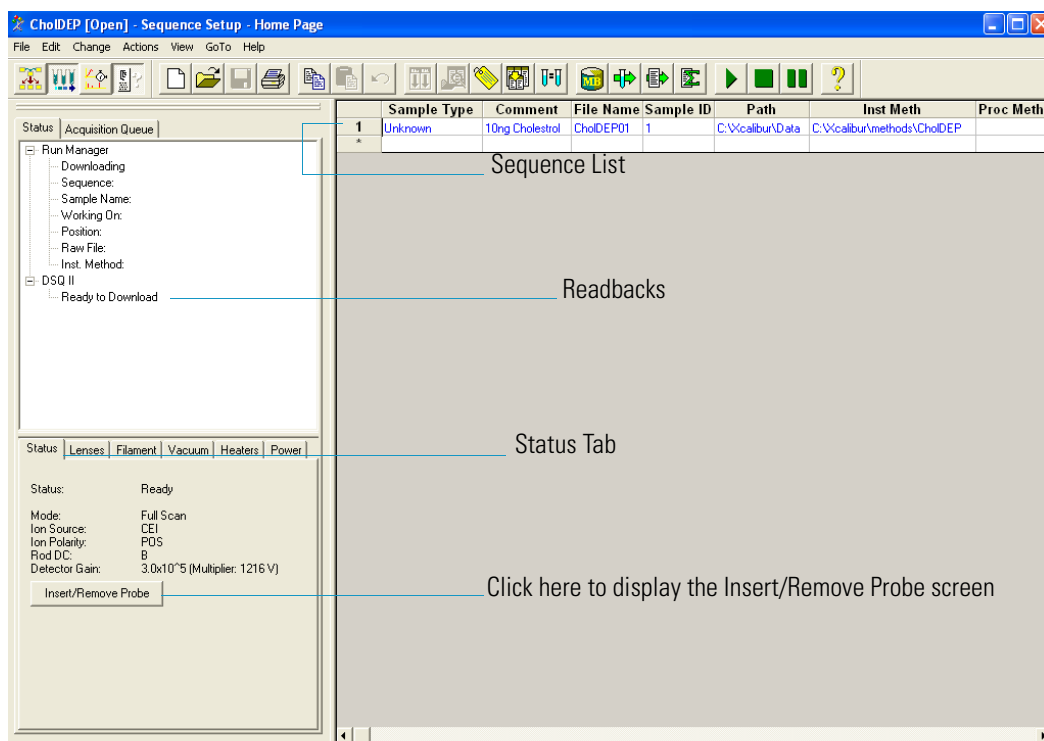
**Figure 65.** Run Sequence Screen: DSQ Series



- c. Click OK to return to the Sequence Setup window.

3. Look at the Status tab to read the DSQ Readbacks.

**Figure 66.** Sequence Setup: DSQ Series CholDEP Sequence



- When you see the readback Waiting for Contact Closure, press the Start button on the DPC panel to run the sample.
- Then look for the DSQ readback to display Running. If it doesn't verify that the DPC cable is firmly connected to the back of the DSQ.
- Select View | Real Time Plot View from the Sequence Setup window to monitor the run.
- The run is completed when the readback displays Ready to Download and the view displays No DataFile.
- Go to ["Step 10: Remove the Probe Using Xcalibur"](#) on page 74.

## Step 10: Remove the Probe Using Xcalibur

You should still be at the Sequence Setup window. In this step you remove the probe to prepare for the next analysis.

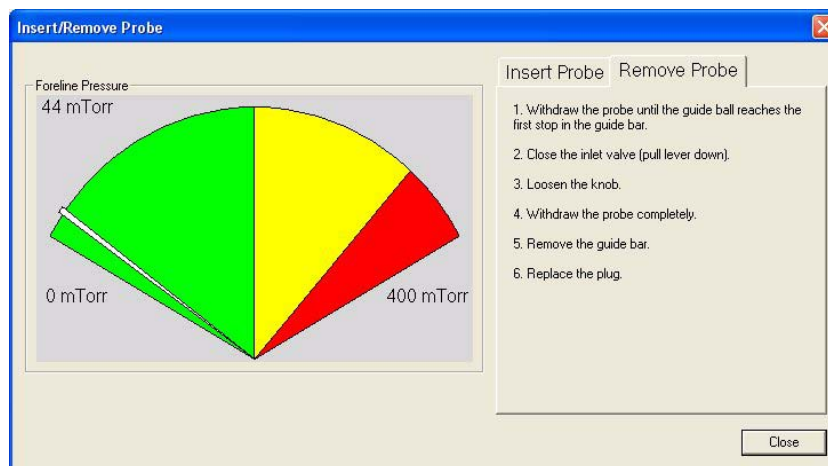


**CAUTION INSTRUMENT DAMAGE.** Avoid accidentally venting the system. Do not remove the probe beyond the point at which the pin in the probe handle reaches the stop in the guide bar. Do not completely withdraw the probe until the ball valve has been closed. Otherwise, the system vents to the atmosphere.

- Select the Insert/Remove Probe button again from the Status tab.

- a. Select the Remove Probe tab and *carefully* follow steps 1–6.

**Figure 67.** Remove Probe Tab: DSQ Series



- b. Click the Close button to return to the Sequence Setup window.
- c. Begin the next step.

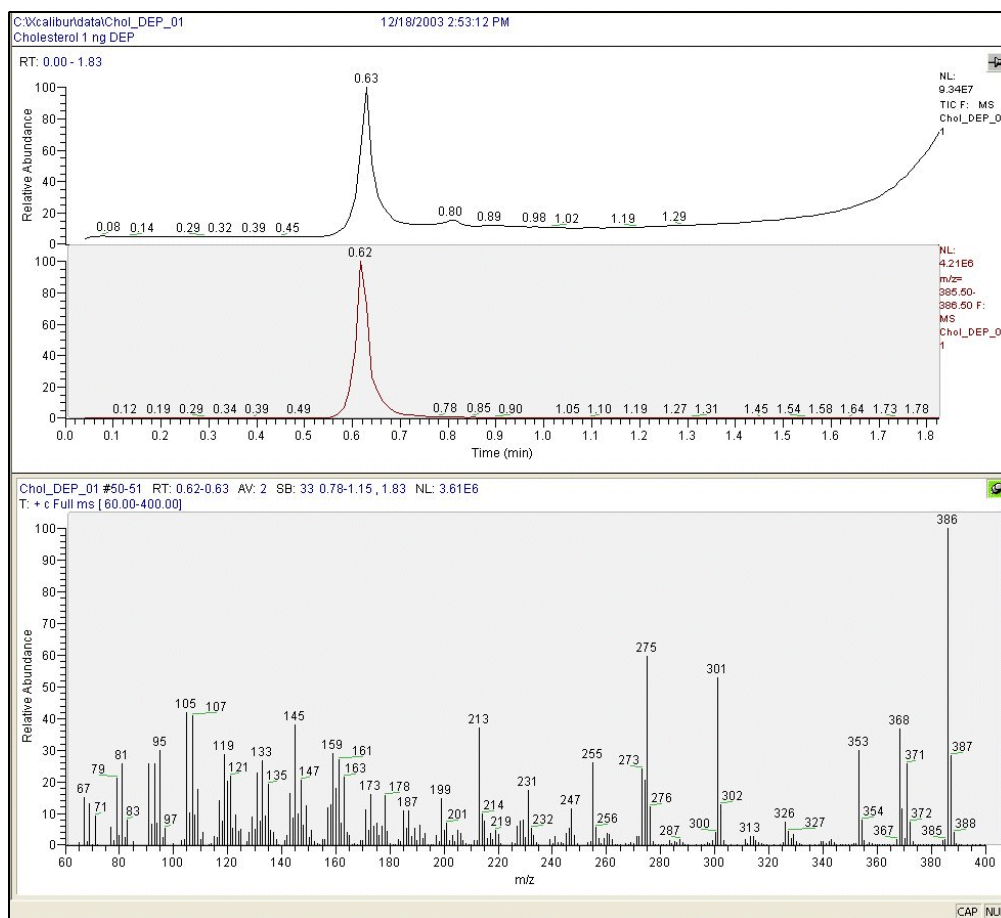
## Step 11: Analyze the Data File

Use these steps to process the data file you just acquired. If you want to analyze another cholesterol sample, repeat the steps listed under “[Step 6: Prepare the Sample](#)” on [page 69](#), making the appropriate addition to the sequence table.

1. At the Sequence Setup window, click the Roadmap icon to return to the Xcalibur Home Page.
2. Select Qual Browser to open the Qual Browser window. Refer to the Qual Browser manual to learn how to use Qual Browser techniques.
3. Select File | Open and select the CholDEP.raw data file you just ran.

Compare your results with the Qual Browser example (Figure 68).

**Figure 68.** Qual Browser: DSQ Series DEP



For information on how to load samples see “Loading DEP Samples” on page 76, clean the probe tip see “Cleaning the DEP” on page 78, and terms to know see “Top Terms to Know” on page 81.

## Loading DEP Samples

You can load either liquid or solid samples onto your DEP. However, before loading a sample onto the filament wire, follow these instructions to avoid contaminating the ion source.

1. Clean the probe tip prior to loading the first sample, because probe tips may be contaminated by handling or from previous sample runs. See “Cleaning the DEP” on page 78.
2. Inspect the filament to make sure the filament wire is not short-circuited across the support posts.
3. Check the filament wire to be sure that it is straight with a smooth uniform loop.

4. [Optional] If necessary, replace bent filaments using a pair of clean forceps to insert a clean filament assembly into the socket on the end of the probe tip.
5. Ensure that the filament assembly is fully inserted into the probe tip, and that the filament legs make good electrical contact in the probe tip sockets. If you are unable to make good contact with this method, you can gently separate the filament legs.
6. Use small samples to help keep the system clean, thereby reducing the amount of maintenance required.

Small samples also keep the background low. In general, the sample size should be 1  $\mu\text{L}$  or less to minimize contaminating the ion source.

Now you are ready to apply liquid or solid samples.

## Loading Liquid Samples

Apply liquid samples<sup>1</sup> directly to the sample loop or the end of the filament.

1. Use a 10  $\mu\text{L}$  syringe to draw 1 or 2  $\mu\text{L}$  of sample.

Apply the liquid onto the filament loop as shown in [Figure 69](#). The loop will support the resulting bead of liquid.

**Figure 69.** Loading a Liquid Sample onto the DEP Filament



**CAUTION INSTRUMENT DAMAGE.** Allow sample solvent to evaporate completely before inserting probe into the mass spectrometer. If solvent is not allowed to evaporate, then sample loss may occur, which may cause the pump to stall, because the solvent rapidly evaporates within the vacuum.

2. Evaporate the solvent carefully, so that the sample remains deposited uniformly on the tip of the filament wire.

You can use an infrared or incandescent bulb as a heat source to increase the evaporation rate of your solvent. However, be careful not to warm the sample droplet too quickly, or the solvent could sputter and cause sample loss.

<sup>1</sup> [[Optional] Dilute the sample in a small quantity of solvent.

# Loading Solid Samples

Apply solid samples<sup>1</sup> directly to the filament.

1. Load solid samples with a dry syringe needle, or use a piece of clean wire or capillary column to collect some of the sample.
2. Lightly touch the sample to the filament being careful not to bend or damage the filament.

# Cleaning the DEP

1. Examine and clean your probe and filament wire often to ensure the integrity of your experiments.
2. Clean the probe.



**CAUTION INSTRUMENT DAMAGE.** Supplying current greater than 100 mA to the filament in the presence of oxygen can burn out the filament.

- a. Use only a soft cloth to wipe the probe handle and probe shaft.
- b. Use a soft cloth dampened in a mild detergent and water solution to remove dirt. Never use abrasive cleaners.

**Note** Don't use solvent to clean a probe tip. Instead, insert the probe and filament into the inlet vacuum chamber.

3. Allow the probe to dry completely before continuing.
4. Clean the probe filament by inserting the probe with the filament tip into the inlet vacuum chamber, and pressing the DPC clean button once or twice to burn off any contaminants.
  - a. Select the Insert Remove Probe button from the Xcalibur Home Page to display the Insert Remove Probe screen.

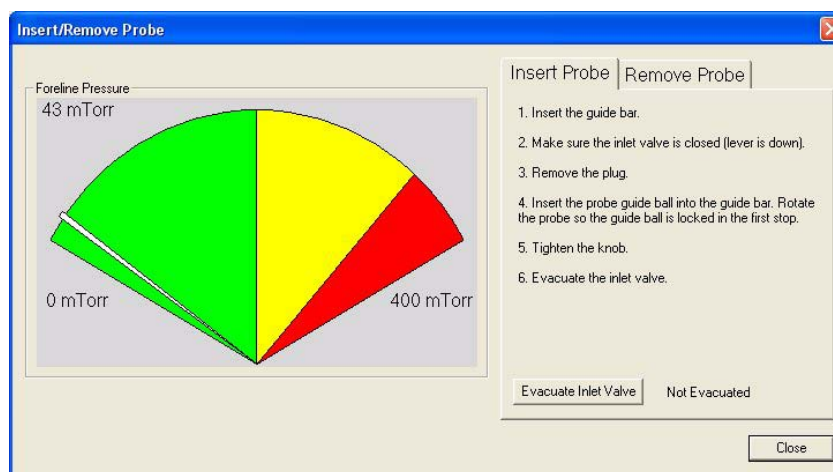
---

<sup>1</sup> [[Optional]] Dilute the sample in a small quantity of solvent to convert it to liquid.



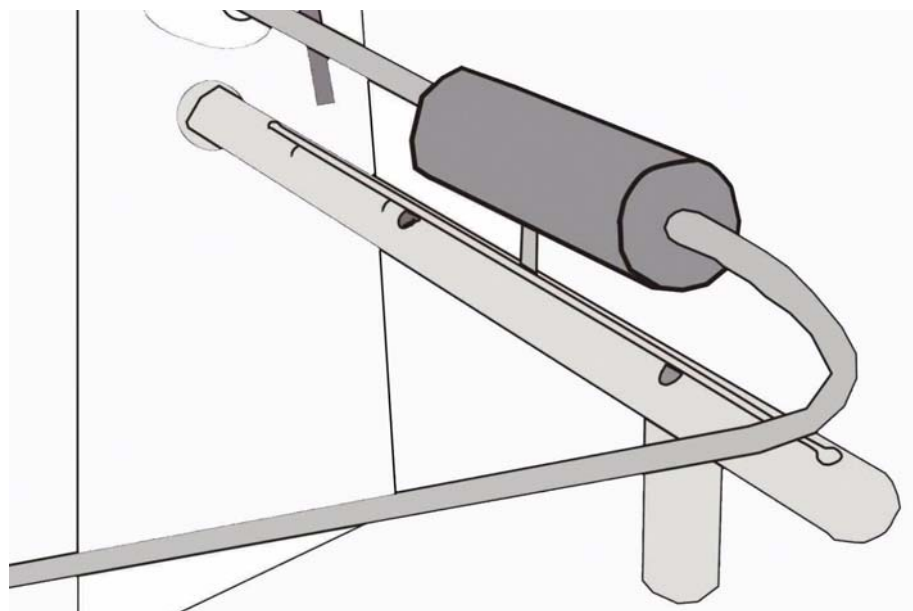
- b. Select the Insert Probe tab and carefully follow each instruction.

**Figure 70.** Insert/Remove Probe Screen: DSQ Series



- c. At step 4 on the screen instructions, slide the probe into the inlet valve chamber until the stem of the probe shaft reaches the 1st Stop on the guide bar.

**Figure 71.** Sample Probe before the 2nd Stop



- d. At step 6, click the Evacuate Inlet Valve button that is located on the Insert Remove Probe screen.
- e. Wait to see the Inlet Valve Message prompt you to insert the probe further into the chamber.
- f. Open the inlet valve by lifting the lever up.

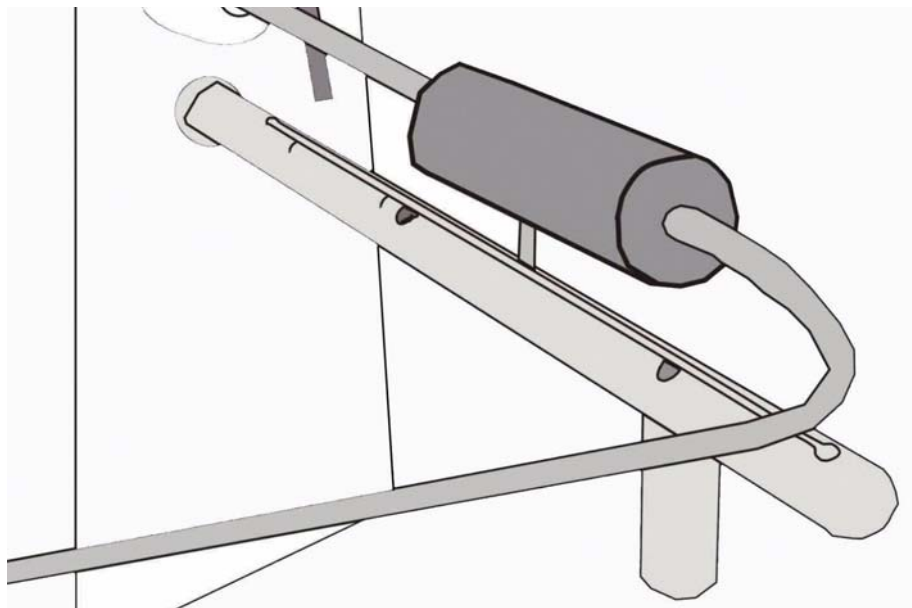


## 2 Using a Direct Exposure Probe (DEP)

### Cleaning the DEP

- g. Slide the probe into the chamber until the stem of the probe is between the 1st and 2nd stop on the guide bar.

**Figure 72.** Probe before the 2nd Stop



- h. Press the DPC Clean button once or twice to burn off any contaminants.
  - i. Click the Close button located on the Insert/Remove Probe screen.
5. Remove the probe.

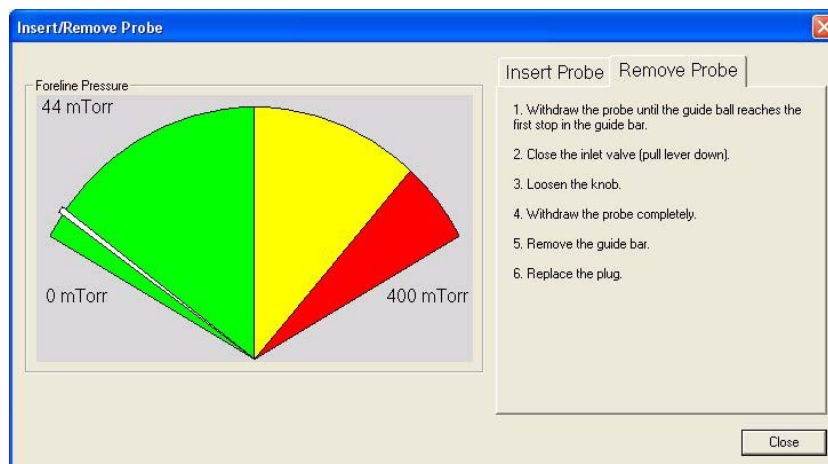


**CAUTION INSTRUMENT DAMAGE.** Avoid accidentally venting the system. Do not remove the probe beyond the point at which the pin in the probe handle reaches the 1st stop in the guide bar while exiting. Do not completely withdraw the probe until the ball valve has been closed. Otherwise, the system vents to the atmosphere.

- a. Select the Insert / Remove Probe button from the Tune window to display the Insert/Remove Probe screen.

- b. Select the Remove Probe tab (Figure 73) and *carefully* follow steps 1–6.

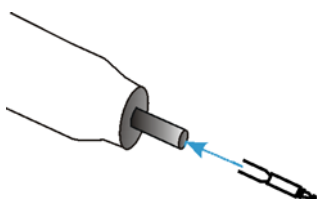
**Figure 73.** Remove Probe Tab: DSQ Series



- c. When instructed, remove the probe from the instrument.

## Top Terms to Know

**Figure 74.** DEP Sample Probe and Filament



## DEP Modes

- Current-Programmed Mode

The DEP operates in a current-programmed mode, which means you program the filament current to ramp at a given rate. The maximum filament current is 1000 mA, and it can be ramped up to 1000 mA/s. Fast ramp rates are useful for quickly vaporizing thermally labile samples, and minimizing thermal decomposition. However, fast ramp rates mean the sample is completely consumed in a fraction of a second. Typical ramp rates are 20 to 500 mA/s.

**Note** Optical pyrometer measurements indicate that the actual filament temperature in degrees celsius closely approximates the filament current in milliamperes. For example, an indicated current of 1000 mA corresponds to an approximate temperature of 1000 °C.

- Filament

The filament is a wire made of rhenium with a loop at the end. The filament wire is set in a ceramic base, which is inserted into the probe tip. Samples are placed on the filament wire and

## 2 Using a Direct Exposure Probe (DEP)

### Top Safeguards to Know

allowed to dry. Once in the ion source, a current passes through the filament to heat it, rapidly vaporizing the sample. The vaporized sample is exposed directly to the electron beam in the ion source for EI or the reagent gas plasma for CI.

## Probe Shaft

The probe shaft is a long metal rod attached to a cylindrical handle. It measures approximately 17-in. long and 0.531-in. in diameter. Electrical cabling and coolant gas lines pass through the probe shaft from the cable connection in the handle to the probe tip.

## Probe Tip

At the end of the probe shaft, the probe tip contains a plug into which you insert the DEP filament assembly. The probe tip seals against the outer surface of the ion volume holder. This positions the DEP filament inside the ion volume.

## Top Safeguards to Know

When using the DEP, use these safety precautions.



**WARNING ELECTRICAL SHOCK HAZARD.** Avoid exposing the DEP to electrical shock or electrical charging. Always plug the DEP cable into the DPC probe connector when performing EI or CI experiments, even if the probe is not heated. This is because the DEP cable provides electrical ground.

1. When the probe is not in use, be sure to place it on top of the DPC.
2. Handle the DEP filament with care to avoid breaking or distorting the wire.
3. Avoid bending or damaging the filament wire. It should look straight, with a smooth uniform loop. A stress or break in this wire creates an open circuit. Consequently, the probe tip does not receive any current.
4. Avoid scratching or otherwise abrading the surface of the probe shaft. Damage to the surface of the probe shaft could result in leaks in the inlet valve vacuum seal.
5. Avoid contaminating the DEP filament and probe shaft. Remember that both are inserted into the Ion Trap and DSQ Series. Handle them with care.



**CAUTION INSTRUMENT DAMAGE.** Supplying a current greater than 100 mA to the filament in the presence of oxygen can burn out the filament.

6. Avoid contaminating the filament's ceramic base or the probe shaft with the sample, solvent, or finger oils. Contamination produces unwanted background or memory effects.
7. Always start and clean the probe when it is in a vacuum, and the vacuum manifold is sufficiently pumped down.
8. Avoid accidentally venting the system. Make sure the inlet valve is closed (the valve handle is in the down position) before removing the DEP. Ion Trap Series users see [“Step 10: Remove the Probe Using Xcalibur”](#) on [page 59](#). DSQ users see [“Step 10: Remove the Probe Using Xcalibur”](#) on [page 74](#).

## Hints

- To keep sample cool for volatile samples, keep the probe outside the ion source until ready to press the DPC Go button.
- [DSQ only] High Mass may require a higher ion offset and resolution for high scan rates.
- [DSQ only] Get the best resolution by scanning slow over a narrow mass range.

## 2 Using a Direct Exposure Probe (DEP)

Hints

## Getting Help

This chapter contains additional information for getting help.

### Contents

- “Who to Call” on page 86
- “Detuning the Instrument” on page 87
- “Getting Spare Parts” on page 89

## Who to Call

We value opportunities to hear from you or to help you. Here are a list of frequently used contacts.

### Technical Support

Thermo Fisher Scientific 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 Fisher Scientific product lines. Please contact us by phone/fax or mail at:

Thermo Fisher Scientific Technical Support  
1400 Northpoint Parkway, Suite 10  
West Palm Beach, FL 33407  
Phone: 1 (800) 685-9535  
Fax: 1 (561) 688-8736

### Customer Service

For customer service, contact your local Thermo Fisher Scientific office or dealer.

### Spare Parts

To order spare parts, contact:

Customer Service & O/P  
1400 North Pointe Parkway, Suite 10  
West Palm Beach, FL 33407

Phone: 1 (800) 532-4752

### Technical Publications Editor

Thermo Fisher Scientific Corporation  
2215 Grand Ave. Pkwy  
Austin, TX 78728 USA  
(512)-251-1400

Email: [techpubsaustin@thermo.com](mailto:techpubsaustin@thermo.com)



## Detuning the Instrument

Detuning is sometimes necessary to correct saturation in the instrument. Detuning the instrument means you adjust the tuning parameters from the instrument's default to make the instrument less efficient in making and transmitting ions. On the Ion Trap Series, you adjust the emission current and lens 2 as compared to adjusting the emission current and resolution on the DSQ Series.

1. Go to the Tune window and open the default tune file or whichever tune file you typically use.

2. Select Experiment | Full Scan to set the scan range on the Full Scan screen.

Enter the first mass and the last mass of your scan range. For example, a full scan range could be 50 for the first mass and 650 for the last mass, thus giving you a scan range of 50 to 650.

3. Select Instrument | Calibration Gas | EI/NICI to turn ON the calibration gas.

4. Select Instrument | Fil/Mult/Dyn/On to turn ON the ion source filament.

5. Monitor the TIC and lower the emission current until the TIC drops by the amount you want to detune the instrument.

For both the DSQ and Ion Trap Series, adjust the tuning parameters as needed to detune the instrument to the desired amount. For example, if the TIC is  $4 \times 10^6$  and you want to detune the instrument by 10 times, adjust the tuning parameters until the TIC is approximately  $4 \times 10^5$ .

- **Ion Trap Series Users**

- a. Select Tune | Manual to display the Manual Tune screen.
- b. Select the Filament tab to adjust the emission current from the default 250  $\mu\text{A}$  to as low as 50  $\mu\text{A}$ . Lower emission current means fewer ions are made in the source.
- c. Select the Lenses tab to adjust lens 2 from the default value of 100 V to as low as 50 V. Changing lens 2 reduces the efficiency by which ions are transmitted from the ion source to the ion trap.

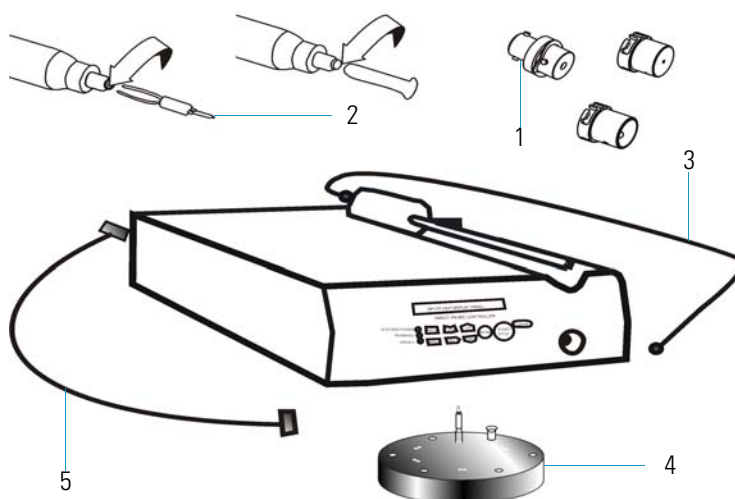
- **DSQ Series Users**

- a. Select Tune | Show Manual Tune View to display the Tune Window Status tabs.
  - b. Select the Filament tab to adjust the emission current from the default 100  $\mu\text{A}$  to as low as 20  $\mu\text{A}$ . Lower emission current means fewer ions are made in the source.
  - c. Select the Resolution tab and increase the resolution (for mass 1.0 and mass 1050.0) slowly in increments of 0.1 as needed to get the TIC as low as you want.
6. Select File | Save As to save the tune file with a different name other than the one you just adjusted.
    - a. Name the file Probe.tune or Probe.dsqtune for your current tune configuration and future probe experiments.

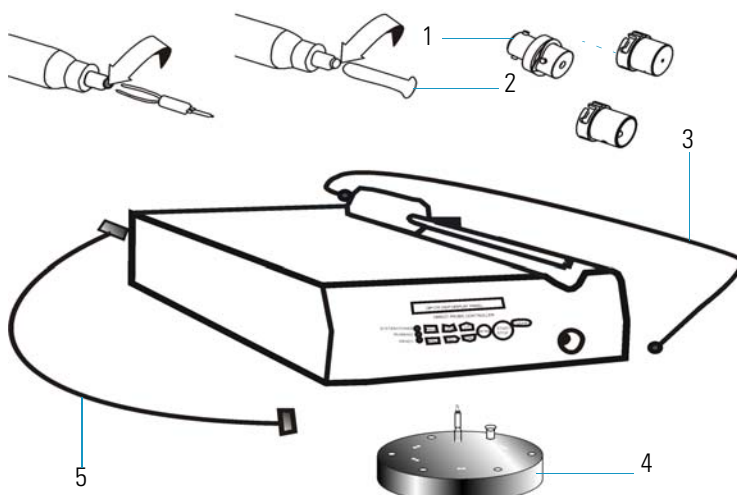
- b. Choose this tune file when setting up your method.
7. Begin your probe experiment.

## Getting Spare Parts

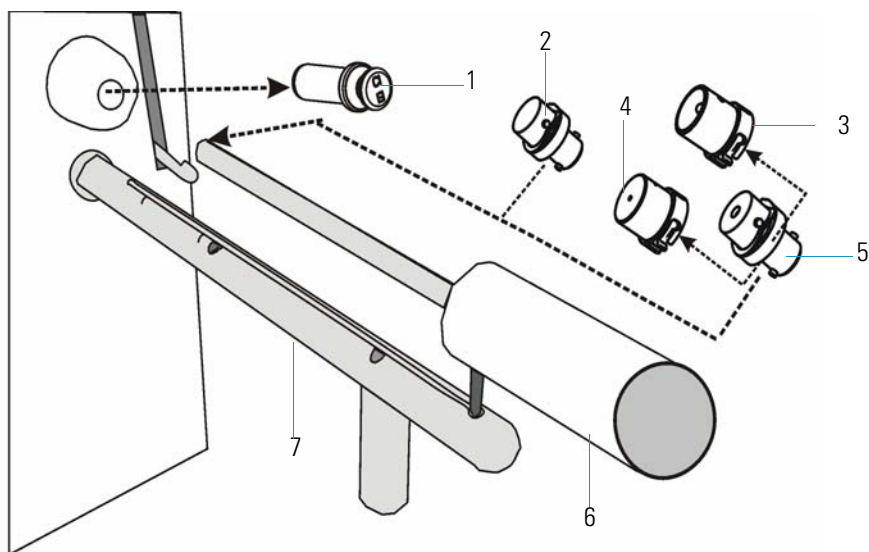
To order spare parts call Customer Service. Some technical issues can be resolved by replacing the DPC and/or sample probe rather than replacing parts.



Item	Description	PN	Qty
<b>DEP &amp; DPC for the DSQ and Ion Trap Series</b>		<b>119300-1577</b>	
	DEP Sample Probe Kit (6 items)	119300-ODEP	
	Probe Plastic Case	119321-0001	1
1	Probe Ion Volume Holders	119324-0001	2
	Test Sample, DEP/DIP	119343-0001	1
2	Filaments Pkg/5	119322-0001	1
	DPC Assembly Parts (10 items)	119300-0100	
	3-prong Power Cord, 110 V	3467-0034	1
	3-prong Power Cord International NT, 220-240 VAC	76357-0018	1
	Fuses, 5 x 20, 250 V	76706-0200	2
	Hose Barb, Tee Fitting	76256-0039	1
	Tubing Beswick MUT 1332, 427 cm	76433-0107	1
3	DPC External Cable to Probe	119303-0001	1
	Direct Probe System's User's Guide	119327-0001	1
4	Sample Cup/Filament Holder	119342-0001	1
5	DPC Remote Start Cable to mass spectrometer ACCESSORY START, DB9F-DIN	119378-0032	1



Item	Description	PN	Qty
<b>DIP &amp; DPC for the DSQ and Ion Trap Series</b>		<b>119300-1576</b>	
	DIP Sample Probe Kit (6 items)	119300-ODIP	
	Probe Plastic Case	119321-0001	1
1	Probe Ion Volume Holders	119324-0001	2
	Test Sample, DEP/DIP	119343-0001	1
2	Sample Cups, Pkg/25	119329-0001	1
	DPC Assembly Parts (10 items)	119300-0100	
	3-prong Power Cord, 110 V	3467-0034	1
	3-prong Power Cord International NT, 220-240 VAC	76357-0018	1
	Fuses, 5 x 20, 250 V	76706-0200	2
	Fitting Hose Barb	76256-0039	1
	Tubing Beswick MUT 1332, 427 cm	76433-0107	1
3	DPC External Cable to Probe	119303-0001	1
	Direct Probe System's User's Guide	119327-0001	1
4	Sample Cup/Filament Holder	119342-0001	1
5	DPC Remote Start Cable to mass spectrometer ACCESSORY START, DB9F-DIN	119378-0032	1
<b>3CFM Pump Assembly Kit (7 items)</b>		<b>119317-0001</b>	
	Hose Adapter Fitting	119178-0001	1
	Hose Barb 1/8 Fitting	76256-0040	1
	Vacuum Pump	76505-3007	1



Item	Description	PN	Qty
1	Inlet Valve Plug	119273-0001	1
2	GC Ion Volume Holders	70001-20532	2
3	EI Ion Volumes	119650-0220	2
4	CI Ion Volumes	119650-0230	2
5	Probe Ion Volume Holders	119324-0001	2
6	Ion Volume I/R Tool	96000-60057	1
7	Guide Bar, Ion Trap Series/DSQ Series	119687-0010	1



# Abbreviations

## Numerics

**μ** micro ( $10^{-6}$ )

**Ω** ohm

## A

**A** ampere

**ACE** automatic collision energy

**ac** alternating current

**amu** atomic mass unit

**ADC** analog-to-digital converter

**ASCII** american standard code for information interchange

## B

**baud rate** data transmission speed in events per second.

## C

**°C** degrees Celsius

**CD-ROM** compact disc read-only memory

**CE** (F. Conformité Européenne) European conformity. Mandatory european marking for certain product groups to indicate conformity with essential health and safety requirements set out in european directives.

**cfm** cubic feet per minute

**CI** chemical ionization

**CID** collision induced dissociation

**CIP** carriage and insurance paid to

**cm** centimeter

**cc or cm<sup>3</sup>** cubic centimeter

**CPU** central processing unit (in a computer)

## D

**Da** Dalton

**DAC** digital-to-analog converter

**dc** direct current

**DDS** data dependency scanning

**DEP™** direct exposure probe

**DIP** direct insertion probe

**DS** data system

**DSP** digital signal processor

## E

**EI** electron ionization

**ESD** electrostatic discharge

**eV** electron volt

**EN** european conformity

## F

**f** femto ( $10^{-15}$ )

**°F** degrees Fahrenheit

**FCC** federal communication commission

**FOB** free on board

**ft.** foot

**FSE** field service engineer

**FTP** file transfer protocol

## G

**g** gram

**G** giga ( $10^9$ )

**GB** gigabytes 1024 MB (billion)

**GC** gas chromatograph

**GC/MS** gas chromatograph / mass spectrometer

**GND** electrical ground

**GPB** general-purpose interface bus

**GUI** graphical user interface

## H

**HV** high voltage

**Hz** hertz (cycles per second)

## I

**ICIS™** interactive chemical information system

**IEC** international electrotechnical commission

**IEEE** institute of electrical and electronics engineers

**in.** inch

**I/O** input/output

## K

**k** kilo ( $10^3$ , 1000)

**K** Kelvin

**kb** kilobyte 1024 bytes (thousand)

**kg** kilogram

**kPa** kilopascal

**kVA** kilo volt amperes (1000 VA)

## L

**L** liter

**LAN** local area network

**lb** pound

**LED** light-emitting diode

## M

**m** meter; milli ( $10^{-3}$ )

**M** mega ( $10^6$ )

**MB** megabyte or 1,024 kb (million)

**M<sup>+</sup>** molecular ion

**min** minute

**mL** milliliter

**mm** millimeter

**MS** scan power: MS<sup>1</sup>, mass spectrometer

**m/z** mass-to-charge ratio

## N

**n** nano ( $10^{-9}$ )

## P

**p** pico ( $10^{-12}$ )



**Pa** Pascal

**PCB** printed circuit board

**PID** proportional / integral / differential

**PN** part number

**P/P** peak-to-peak voltage

**PPINICI** pulsed positive ion negative ion chemical ionization

**ppm** parts per million

**PQD** pulsed  $q$  dissociation

**psig** pounds per square inch, gauge

## Q

$q$  maximum excitation energy

## R

**RAM** random access memory

**RF** radio frequency

**RMS** root mean square

**ROM** read-only memory

**RS-232** industry standard for serial communications

## S

**s** second

**SCSI** small computer system interface

**SIM** selected ion monitoring

**SI** international system of units

**S/N** signal to noise ratio

## T

**TIC** total ion current

**TCP/IP** transmission control protocol / Internet protocol

**Torr** torr

## U

**URL** uniform resource locator

**USB** universal serial bus

## V

**V** volt

**V ac** volts alternating current

**V dc** volts direct current

**Abbreviation:**

# Index

## A

air source 6, 22

## C

Calibration Gas 87

CholDEP.raw data file 60, 75

CholDIP.raw data file 18, 34

Cholesterol example

DEP 46

DIP 4

Cleaning

DEP 78

DIP 37

coolant 6, 22

Customer Service 86

## D

Data File

DEP 60, 75

DIP 18, 34

Data file

DEP 60, 75

DIP 18, 34

DCI 45

DEP

Loading samples 76

Liquid 77

Solid 78

DEP Kit 46, 62

detune

detuning 87

DIP 1

Loading Samples 35, 36, 37

Modes 40

DIP Kit 5, 21

direct chemical ionization 45

Direct Probe Controller (DPC) 2, 44

## E

Emission Current 87

Evacuate Inlet Valve button 15, 31, 57, 72, 79

Examples

DEP 46

DIP 4

## F

feedback iii

Filament 81

Full Scan 87

## G

gas chromatograph xiii, 3, 45

Getting spare parts 89

## I

Inlet Message screen 15, 31, 57, 72, 79

Insert probe

DEP 57, 72

DIP 15, 31

Insert Remove Probe screen 15, 31, 57, 72, 79

Insert/Remove Probe button 7, 18, 23, 34, 48, 60, 64, 74

ion source filament 87

## L

Lens 2 87

Liquid 36

Liquid samples

DEP 77

DIP 1, 36

Loading samples

DEP 76

## M

Maximum rate

- DEP 81
- DIP 39
- Maximum temperature
  - DEP 81
  - DIP 39, 40
- Method setup
  - DEP 52
  - DEP DSQ Series 68
  - DEP Ion Trap Series 55
  - DIP 12, 28
  - DIP DSQ 27
  - DIP Ion Trap Series 11
  - Ion Trap Series 52, 54
- Modes
  - DEP
    - Current-programmed 81
  - DIP 40
    - Ballistic 38
    - Temperature-programmed 38

## P

- Parts
  - DEP Kit 46, 62
  - DIP Kit 5, 21
- probe shaft 39, 82
- probe tip
  - DEP 82
  - DIP 39

## R

- reader survey iii
- registration card iii
- Remove Probe tab 34, 60, 75, 81
- Resolution 87
- RTD 39

## S

- Safeguards 40
  - DEP 62, 82
  - DIP 5, 21
- Sample cups 39
- Samples 76, 77
  - DEP 43, 46, 76, 81
  - DIP 1, 4, 7, 23, 35, 37, 48, 64
  - Forensic 1
  - Tissue 1
- Sequence list
  - DEP 71
  - DEP Ion Trap Series 55
- Solid 37

- Solid samples
  - DEP 76, 78
  - DIP 35, 36
- Spare parts
  - List 89
  - Ordering 86

## T

- Technical Support 86