

Surveyor MSQ Plus

Hardware Manual

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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 as amended by 92/31/EEC and 93/68/EEC

EMC compliance has been evaluated by UNDERWRITERS LABORATORY, INC (UL).

EN 55011	(1998)	EN 61000-4-3	(2002)
EN 61326-1	(1998)	EN 61000-4-4	(2001)
EN 61000-3-2	1995	EN 61000-4-5	(2001)
EN 61000-3-3	1995	EN 61000-4-6	(2001)
EN 61000-4-2	(2001)	EN 61000-4-11	(2001)

CFR 47 Part 15 Subpart B: 2004

Code of Federal Regulations, Part 15, Subpart B, Radio Frequency Devices Unintentional Radiators
Class A

Low Voltage Safety Compliance

This device complies with the EU directive 73/23/EEC (equivalent to IEC 1010-1, 1990 plus Amendment 1, 1991 and Amendment 2, 1995) by meeting the following standard: EN 61010-1: 2001 with Corrigendum No. 1 and 2.

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

THIS DEVICE COMPLIES WITH PART 15 OF THE FCC RULES. OPERATION IS SUBJECT TO THE FOLLOWING TWO CONDITIONS: (1) THIS DEVICE MAY NOT CAUSE HARMFUL INTERFERENCE, AND (2) THIS DEVICE MUST ACCEPT ANY INTERFERENCE RECEIVED, INCLUDING INTERFERENCE THAT MAY CAUSE UNDESIRE OPERATION.



CAUTION 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.

Notice on Lifting and Handling of Thermo Fisher 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.

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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.

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 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:







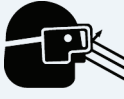

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Conformité DEEE

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:



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CAUTION Symbol	CAUTION	VORSICHT	ATTENTION	PRECAUCION	AVVERTENZA
	Electric Shock: This instrument uses high voltages that can cause personal injury. Before servicing, shut down the instrument and disconnect the instrument from line power. Keep the top cover on while operating the instrument. Do not remove protective covers from PCBs.	Elektroschock: In diesem Gerät werden Hochspannungen verwendet, die Verletzungen verursachen können. Vor Wartungsarbeiten muß das Gerät abgeschaltet und vom Netz getrennt werden. Betreiben Sie Wartungsarbeiten nicht mit abgenommenem Deckel. Nehmen Sie die Schutzabdeckung von Leiterplatten nicht ab.	Choc électrique: L'instrument utilise des tensions capables d'infliger des blessures corporelles. L'instrument doit être arrêté et débranché de la source de courant avant tout intervention. Ne pas utiliser l'instrument sans son couvercle. Ne pas enlever les étuis protecteurs des cartes de circuits imprimés.	Descarga eléctrica: Este instrumento utiliza altas tensiones, capaces de producir lesiones personales. Antes de dar servicio de mantenimiento al instrumento, éste deberá apagarse y desconectarse de la línea de alimentación eléctrica. No opere el instrumento sin sus cubiertas exteriores quitadas. No remueva las cubiertas protectoras de las tarjetas de circuito impreso.	Shock da folgorazione. L'apparecchio è alimentato da corrente ad alta tensione che può provocare lesioni fisiche. Prima di effettuare qualsiasi intervento di manutenzione occorre spegnere ed isolare l'apparecchio dalla linea elettrica. Non attivare lo strumento senza lo schermo superiore. Non togliere i coperchi a protezione dalle schede di circuito stampato (PCB).
	Chemical: This instrument might contain hazardous chemicals. Wear gloves when handling toxic, carcinogenic, mutagenic, or corrosive or irritant chemicals. Use approved containers and proper procedures to dispose waste oil.	Chemikalien: Dieses Gerät kann gefährliche Chemikalien enthalten. Tragen Sie Schutzhandschuhe beim Umgang mit toxischen, karzinogenen, mutagenen oder ätzenden/reizenden Chemikalien. Entsorgen Sie verbrauchtes Öl entsprechend den Vorschriften in den vorgeschriebenen Behältern.	Chimique: Des produits chimiques dangereux peuvent se trouver dans l'instrument. Portez des gants pour manipuler tous produits chimiques toxiques, cancérigènes, mutagènes, ou corrosifs/irritants. Utiliser des récipients et des procédures homologuées pour se débarrasser des déchets d'huile.	Química: El instrumento puede contener productos químicos peligrosos. Utilice guantes al manejar productos químicos tóxicos, carcinógenos, mutágenos o corrosivos/irritantes. Utilice recipientes y procedimientos aprobados para deshacerse del aceite usado.	Prodotti chimici. Possibile presenza di sostanze chimiche pericolose nell'apparecchio. Indossare dei guanti per maneggiare prodotti chimici tossici, cancerogeni, mutageni, o corrosivi/irritanti. Utilizzare contenitori aprovo e seguire la procedura indicata per lo smaltimento dei residui di olio.
	Heat: Before servicing the instrument, allow any heated components to cool.	Hitze: Warten Sie erhitzte Komponenten erst nachdem diese sich abgekühlt haben.	Haute Temperature: Permettre aux composants chauffés de refroidir avant tout intervention.	Altas temperaturas: Permita que los componentes se enfríen, ante de efectuar servicio de mantenimiento.	Calore. Attendere che i componenti riscaldati si raffreddino prima di effettuare l'intervento di manutenzione.
	Fire: Use care when operating the system in the presence of flammable gases.	Feuer: Beachten Sie die einschlägigen Vorsichtsmaßnahmen, wenn Sie das System in Gegenwart von entzündbaren Gasen betreiben.	Incendie: Agir avec précaution lors de l'utilisation du système en présence de gaz inflammables.	Fuego: Tenga cuidado al operar el sistema en presencia de gases inflamables.	Incendio. Adottare le dovute precauzioni quando si usa il sistema in presenza di gas infiammabili.
	Eye Hazard: Eye damage could occur from splattered chemicals or flying particles. Wear safety glasses when handling chemicals or servicing the instrument.	Verletzungsgefahr der Augen: Verspritzte Chemikalien oder kleine Partikel können Augenverletzungen verursachen. Tragen Sie beim Umgang mit Chemikalien oder bei der Wartung des Gerätes eine Schutzbrille.	Danger pour les yeux: Des projections chimiques, liquides, ou solides peuvent être dangereuses pour les yeux. Porter des lunettes de protection lors de toute manipulation de produit chimique ou pour toute intervention sur l'instrument.	Peligro par los ojos: Las salicaduras de productos químicos o partículas que saltan bruscamente pueden causar lesiones en los ojos. Utilice anteojos protectores al manipular productos químicos o al darle servicio de mantenimiento al instrumento.	Pericolo per la vista. Gli schizzi di prodotti chimici o delle particelle presenti nell'aria potrebbero causare danni alla vista. Indossare occhiali protettivi quando si maneggiano prodotti chimici o si effettuano interventi di manutenzione sull'apparecchio.
	General Hazard: A hazard is present that is not included in the above categories. Also, this symbol appears on the instrument to refer the user to instructions in this manual. When the safety of a procedure is questionable, contact your local Technical Support organization for Thermo Fisher Scientific San Jose Products.	Allgemeine Gefahr: Es besteht eine weitere Gefahr, die nicht in den vorstehenden Kategorien beschrieben ist. Dieses Symbol wird im Handbuch außerdem dazu verwendet, um den Benutzer auf Anweisungen hinzuweisen. Wenn Sie sich über die Sicherheit eines Verfahrens im unklaren sind, setzen Sie sich, bevor Sie fortfahren, mit Ihrer lokalen technischen Unterstützungsorganisation für Thermo Fisher Scientific San Jose Produkte in Verbindung.	Danger général: Indique la présence d'un risque n'appartenant pas aux catégories citées plus haut. Ce symbole figure également sur l'instrument pour renvoyer l'utilisateur aux instructions du présent manuel. Si la sûreté d'un procédé est incertaine, avant de continuer, contacter le plus proche Service Clientèle pour les produits de Thermo Fisher Scientific San Jose.	Peligro general: Significa que existe un peligro no incluido en las categorías anteriores. Este símbolo también se utiliza en el instrumento para referir al usuario a las instrucciones contenidas en este manual. Cuando la certidumbre acerca de un procedimiento sea dudosa, antes de proseguir, pongase en contacto con la Oficina de Asistencia Técnica local para los productos de Thermo Fisher Scientific San Jose.	Pericolo generico. Pericolo non compreso tra le precedenti categorie. Questo simbolo è utilizzato inoltre sull'apparecchio per segnalare all'utente di consultare le istruzioni descritte nel presente manuale. Quando è in dubbio la misura di sicurezza per una procedura, prima di continuare, si prega di mettersi in contatto con il Servizio di Assistenza Tecnica locale per i prodotti di Thermo Fisher Scientific San Jose.

CAUTION Symbol

CAUTION

危険警告

危險警告



Electric Shock: This instrument uses high voltages that can cause personal injury. Before servicing, shut down the instrument and disconnect the instrument from line power. Keep the top cover on while operating the instrument. Do not remove protective covers from PCBs.

電撃: この計測器は高電圧を使用し、人体に危害を与える可能性があります。保守・修理は、必ず操作を停止し、電源を切ってから実施して下さい。上部カバーを外したままで計測器を使用しないで下さい。プリント配線板の保護カバーは外さないで下さい。

電撃: 儀器設備使用會造成人身傷害の高伏電壓。在維修之前，必須先關儀器設備並切除電源。務必要在頂蓋蓋上的情況下操作儀器。請勿拆除PCB保護蓋。



Chemical: This instrument might contain hazardous chemicals. Wear gloves when handling toxic, carcinogenic, mutagenic, or corrosive or irritant chemicals. Use approved containers and proper procedures to dispose waste oil.

化学物質: 危険な化学物質が計測器中に存在している可能性があります。毒性、発がん性、突然変異性、腐食・刺激性などのある薬品を取り扱う際は、手袋を着用して下さい。廃油の処分には、規定の容器と手順を使用して下さい。

化学品: 儀器設備中可能存在有危險性的化學物品。接觸毒性致癌、誘變或腐蝕/刺激性化學品時，請配帶手套。處置廢油時，請使用經過許可的容器和程序。



Heat: Before servicing the instrument, allow any heated components to cool.

熱: 熱くなった部品は冷えるのを待ってから保守・修理を行って下さい。

高温: 請先等高温零件冷卻之後再進行維修。



Fire: Use care when operating the system in the presence of flammable gases.

火災: 可燃性のガスが存在する場所でシステムを操作する場合は、充分な注意を払って下さい。

火災: 在有易燃氣體の場地操作該系統時，請務必小心謹慎。



Eye Hazard: Eye damage could occur from splattered chemicals or flying particles. Wear safety glasses when handling chemicals or servicing the instrument.

眼に対する危険: 化学物質や微粒子が飛散して眼を傷つける危険性があります。化学物質の取り扱い、あるいは計測器の保守・修理に際しては防護眼鏡を着用して下さい。

眼睛傷害危険: 飛濺の化学品或顆粒可能造成眼睛傷害。處理化學品或維儀器設備時請佩戴安全眼鏡。



General Hazard: A hazard is present that is not included in the above categories. Also, this symbol appears on the instrument to refer the user to instructions in this manual.

一般的な危険: この標識は上記以外のタイプの危険が存在することを示します。また、計測器にこの標識がついている場合は、本マニュアル中の指示を参照して下さい。

一般性危険: 説明未包括在上述類別中的其他危險。此外，儀器設備上使用這個標誌，以指示用戶本使用手冊中的說明。

When the safety of a procedure is questionable, contact your local Technical Support organization for Thermo Fisher Scientific San Jose Products.

安全を確保する手順がよくわからない時は、作業を一時中止し、お近くのサーモエレクトロンサンローゼプロダクトのテクニカルサポートセンターご連絡ください。

如对安全程序有疑问，请在操作之前与当地的菲尼根技术服务中心联系。

Contents

	Preface	xiii
	About This Guide	xiii
	Related Documentation	xiii
	Safety and Special Notices	xiii
	Safety Precautions	xiv
	Solvent and Gas Purity Requirements	xv
	Contacting Us	xvi
Chapter 1	Introduction	1
	Overview	2
	Ion Polarity Modes	2
	Ionization Techniques	3
	Electrospray Ionization (ESI)	3
	Atmospheric Pressure Chemical Ionization (APCI)	6
	Scan Types	8
	Full Scan	8
	Selected Ion Monitoring (SIM)	8
	Data Types	8
	Profile Data Type	9
	Centroid Data Type	10
	MCA Data Type	10
Chapter 2	Functional Description	13
	Liquid Chromatograph	15
	Reference Inlet System	16
	MS Detector	19
	Front Panel Status Indicator	19
	Rear Panel Controls and Connections	21
	Connection Between LC and MS Detector	23
	API Sources	23
	RF/dc Prefilter	26
	Mass Analyzer	26
	Ion Detection System	28
	Vacuum System	31
	Inlet Gas Hardware	33
	Cone Wash System	36

	Data System	37
	Computer Hardware	38
	Xcalibur Software	38
	Surveyor MSQ Plus Server	39
	Tune Window	40
	Printer	44
Chapter 3	Daily Operation	45
	Things to Do Before Operating the MS Detector	45
	Checking the Nitrogen Supply	45
	Checking the Disk Space	46
	Checking the Oil Level in the Oil Mist Filter	46
	Things to Do After Operating the MS Detector	47
	Flushing the API Probes	48
	Placing the System in the Off Mode	48
	Draining the Oil Mist Filter and Purging the Pump Oil	48
	Emptying the Solvent Waste Bottles	49
Chapter 4	Switching Probes	51
	Switching From ESI to APCI	51
	Switching From APCI to ESI	53
Chapter 5	Routine and Preventive Maintenance	55
	Maintenance Schedule	55
	Maintaining the ESI Probe	56
	Removing the ESI Probe	57
	Removing the ESI Capillary	58
	Cleaning or Replacing the ESI Capillary	60
	Replacing the Ceramic Sleeve of the ESI Probe	61
	Installing the ESI Capillary	63
	Installing the ESI Probe	66
	Maintaining the APCI Probe	67
	Removing the APCI Probe	67
	Removing the APCI Capillary	68
	Installing the APCI Capillary	70
	Installing the APCI Probe	72
	Maintaining the Probe Heater	73
	Removing the Probe Heater	73
	Cleaning the Probe Heater	76
	Replacing the Detent Screw Insulator	76
	Installing the Probe Heater	78

Maintaining the Source Block Assembly	79
Preparing the LC/MS System for Maintenance	80
Clearing Access to the Source Block Assembly	81
Removing the Entrance Cone and the Cone Wash Nozzle	81
Cleaning the Entrance Cone and the Cone Wash Nozzle	83
Removing the Source Block Assembly	85
Cleaning the RF/dc Prefilter	86
Cleaning the Extraction Cone and the Source Block	88
Assembling the Source Block Assembly	90
Installing the Source Block Assembly	91
Maintaining the Forepump	93
Chapter 6 System Shutdown, Startup, and Reset	95
Shutting Down the System in an Emergency	95
Turning Off the Nitrogen Gas	96
Placing the System in the Off Mode	97
Turning the MS Detector Off from Xcalibur	98
Turning the MS Detector Off from the Tune Window	99
Shutting the System down for Non-Routine Maintenance	100
Restarting the System Following a Complete Shutdown	101
Checking the System Connections	102
Restarting the Surveyor MSQ Plus System	103
Resetting the MS Detector	104
Chapter 7 Replaceable Parts	107
Consumables	108
Spares	111
General Spares	111
Kits	111
Source Block Assembly	111
ESI probe Assembly	114
APCI Probe Assembly	115
Probe Heater Assembly	116
Vacuum Spares	117
Gas Flow Spares and Nitrogen Generator	117
Solvent Path and Calibrant Spares	118
Electronic Spares	118
Connection Kits	118
Manuals	118
Appendix A Optimizing the LC Conditions	119
Flow Rates	119
LC Solvents and Mobile Phase Additives	120
LC Solvents	120
Mobile Phase Additives	120

Contents

Cone Wash System	123
Flow Splitting	124
PEEK Tubing	124
Index	127

Preface

About This Guide

This *Surveyor MSQ Plus Hardware Manual* contains a description of the modes of operation and principal hardware components of your Surveyor MSQ Plus MS detector. In addition, this manual provides step-by-step instructions for cleaning and maintaining your Surveyor MSQ Plus MS detector.

This manual documents features of the Surveyor MSQ Plus MS detector controlled by the Surveyor MSQ 2.0 software. To view the instrument software version of the MS detector once you have configured it, choose **Help > About Home Page** from the Xcalibur Roadmap – Home Page.

Related Documentation

In addition to this guide, Thermo Fisher Scientific provides the following documents for the Surveyor MSQ Plus MS detector:

- *Surveyor MSQ Plus Preinstallation Requirements Guide*
- *Surveyor MSQ Plus Getting Connected*
- *Surveyor MSQ Plus Getting Started*

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:



CAUTION Highlights hazards to humans, property, or the environment. Each CAUTION notice is accompanied by an appropriate CAUTION symbol.

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 Precautions

Observe the following safety precautions when you operate or perform service on the MS detector:



Do not perform any servicing other than that contained in the Surveyor MSQ Plus Hardware Manual. To avoid personal injury or damage to the instrument, do not perform any servicing other than that contained in the *Surveyor MSQ Plus Hardware Manual* or related manuals unless you are qualified to do so.



Shutdown the MS detector and disconnect it from line power before you service it. High voltages capable of causing personal injury are used in the instrument. Some maintenance procedures require that the MS detector be shut down and disconnected from line power before service is performed. Do not operate the MS detector with the top or side covers off. Do not remove protective covers from PCBs.



Respect heated zones. Treat heated zones with respect. The ion transfer capillary and the APCI vaporizer might be very hot and might cause severe burns if touched. Allow heated components to cool before you service them.



Place the MS detector in Standby (or Off) before you open the atmospheric pressure ionization (API) source. The presence of atmospheric oxygen in the API source when the MS detector is On could be unsafe. The MS detector automatically goes into Standby when you open the API source; however, it is best to take this added precaution.



Take care when handling the corona pin. The corona pin is sharp and can cause personal injury. Take care when removing or installing the corona pin.



Make sure you have sufficient nitrogen for your API source. Before you begin normal operation each day, make sure that you have sufficient nitrogen for your API source. The presence of atmospheric oxygen in the API source when the MS detector is On could be unsafe. The MS detector automatically goes into Standby when you run out of nitrogen; however, it is best to take this added precaution.



Contain waste streams. Because the API source can accommodate high solvent flow rates, you must make provisions to collect the waste solvent.



Provide adequate fume exhaust systems for the API source solvent waste container and the forepump. Your laboratory must be equipped with at least two fume exhaust systems: one to vent the waste container connected to the Exhaust port (API solvent drain) on the back of the MS detector and the other to vent the forepump exhaust. As described in the *Surveyor MSQ Plus Getting Connected Guide*, route the (blue) forepump exhaust hose to a dedicated fume exhaust system. Because the exhaust hose acts as a trap for exhaust fumes that would otherwise recondense in the forepump oil, the hose should travel at floor level for a minimum of two meters (78.5 in.) before it reaches the external exhaust system. Route tubing from the waste container connected to the Exhaust port on the back of the MS detector to a second dedicated fume exhaust system. Consult local regulations for the proper method of exhausting the fumes from your system.

Do **not** vent the PVC drain tube (or any vent tubing connected to the waste container) to the same fume exhaust system to which you have connected the forepump. The forepump exhaust contains pump oil, which can seriously contaminate the analyzer optics of the mass spectrometer.

Solvent and Gas Purity Requirements

Because the Surveyor MSQ Plus MS detector is extremely sensitive to solvent impurities, use the highest purity solvents available. Use liquid chromatography grade or higher solvents and buffers. Because deionized water contains chemicals that the Surveyor MSQ Plus MS detector can detect, use distilled water.

The following table lists international sources that can supply high quality solvents.

Solvent source	Telephone number
Mallinckrodt/Baker, Inc.	Tel: (800) 582-2537 Fax: (908) 859-9370
Burdick & Jackson, Inc.	Tel: (800) 368-0050 Fax: (616) 725-6216
E. M. Science, Inc.	Tel: (800) 222-0342 Fax: (800) 336-4422

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	TechSupport.C+MS@thermofisher.com
Knowledge base	www.thermokb.com

Find software updates and utilities to download at www.mssupport.thermo.com.

❖ To contact Customer Service for ordering information

Phone	800-532-4752
Fax	561-688-8731
Web site	www.thermo.com/finnigan

❖ To suggest changes to documentation or to Help

- Fill out a reader survey online at www.thermo.com/lcms-techpubs.
- Send an e-mail message to the Technical Publications Editor at techpubs.finnigan-lcms@thermofisher.com.

Introduction

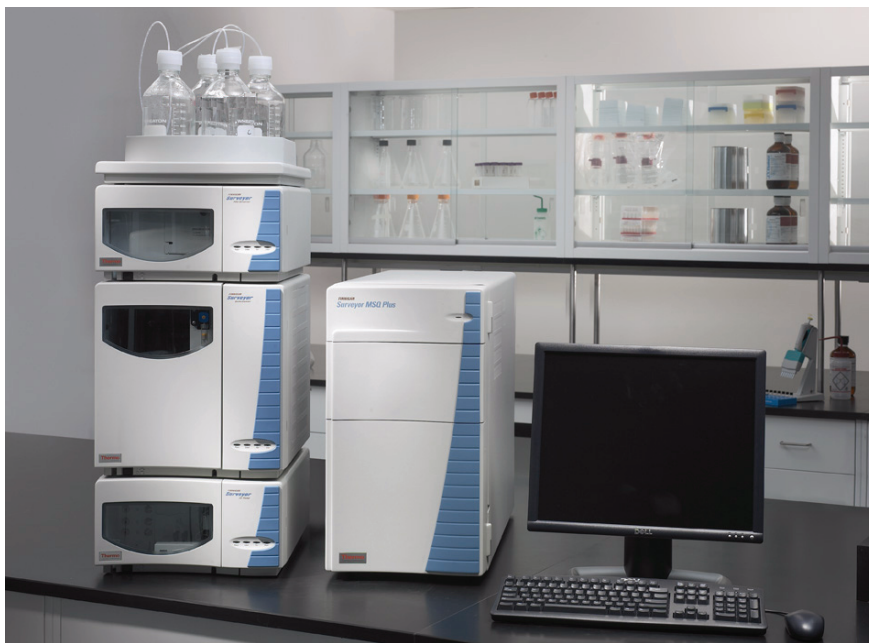
Welcome to the Surveyor MSQ™ Plus MS detector. The Surveyor MSQ Plus MS detector is a member of the Thermo Scientific family of MS detectors.

The Surveyor MSQ Plus system is an advanced analytical instrument that includes an MS detector, forepump, data system, and an optional cone wash pump. Integrated with an LC system, the Surveyor MSQ Plus MS detector provides you with the separation capability of an HPLC and the detection capability of a single-quadrupole MS detector. See [Figure 1](#).

Contents

- [Overview](#)
- [Ion Polarity Modes](#)
- [Ionization Techniques](#)
- [Scan Types](#)
- [Data Types](#)

Figure 1. View of the Surveyor MSQ Plus MS detector and the Surveyor LC system



Overview

In a typical LC/MS analysis, an analytical pump pushes solvent through an LC column under high pressure. An autosampler introduces a measured quantity of sample into this solvent stream. As the solvent stream passes through the LC column, the sample separates into its chemical components. The rate at which the components of the sample elute from the column depends on their relative affinities to the liquid mobile phase solvent and the solid particles that make up the column packing. As the separated chemical components exit the LC column they pass through a transfer line and enter the Surveyor MSQ Plus MS detector.

The Surveyor MSQ Plus MS detector consists of an atmospheric pressure ionization (API) source, a transfer lens, a mass analyzer, and an ion detection system. A vacuum manifold encloses part of the API source, the M-path, the transfer lens, the mass analyzer, and the ion detection system.

MS detectors can detect only ionized molecules. The Surveyor MSQ Plus MS detector provides two ionization techniques: atmospheric pressure chemical ionization (APCI) and electrospray ionization (ESI). In the ESI mode, molecules ionize in the liquid phase before they enter the ion source. In the APCI mode, molecules ionize in the gaseous phase as they enter the API source. For both ionization techniques, the MS detector can place either a positive or negative charge on the capillary of the API probe at any point in time. During a chromatographic run, the MS detector can switch the charge applied to the capillary. By repelling ions of like charge towards the entrance of the MS detector, the charged capillary acts as a charge filter.

The vacuum produced by the forepump draws both neutral molecules and ionized molecules through the entrance cone into the M-path region of the MS detector. The charge on the ionized molecules depends on the selected ion polarity mode. In the M-path region, the low vacuum of 1 Torr produced by the forepump draws the neutral molecules out of the MS detector, enriching the ion stream. By the time the ion stream reaches the exit cone, the solvent flow has decreased by three orders of magnitude. The charge on the exit cone focuses and propels the ionized molecules into the intermediate vacuum region of the MS detector. As the ionized molecules pass through the exit cone, the transfer lens focuses them into a fine particle stream and transmits them to the mass analyzer. The mass analyzer transmits ions of a selected mass-to-charge ratio to the ion detection system, where they produce a signal. The system electronics amplify the signal, which is then transmitted through a USB connection to the Surveyor MSQ Plus data system.

Ion Polarity Modes

You can operate the Surveyor MSQ Plus MS detector in the following ion polarity modes: positive, negative, or positive-negative switching. The MS detector controls the ion polarity by placing either a positive or negative charge on the capillary of the API probe.

The information obtained from a positive-ion mass spectrum is different from and complementary to that obtained from a negative-ion spectrum. Switching between positive and negative ionization modes in a single analytical run gives you the ability to identify more compounds in a single run.

Rapid ion polarity switching is a technique that is applied to several important areas of MS analysis, for example:

- Quantitation of different chemistries within the same run

In drug metabolism studies, certain compounds have functional groups that readily accept a proton (H^+), for example compounds containing a primary amino group ($R-NH_2 + H^+ \rightarrow R-NH_3$) and respond best in the positive ion polarity mode. Other compounds have functional groups that readily lose a proton, for example carboxylic acids ($R-CO_2H \rightarrow R-CO_2^-$) and respond best in the negative ion polarity mode.

- Rapid screening of unknown analytes

Some compounds with functional groups such as carboxylic acids respond only in the negative mode. Some compounds with functional groups, such as amines, alcohols, and ketones respond better or only in the positive mode. If you do not know the identity of your analyte, screen in both modes.

Ionization Techniques

You can operate the Surveyor MSQ Plus MS detector in the following ionization modes:

- [Electrospray Ionization \(ESI\)](#)
- [Atmospheric Pressure Chemical Ionization \(APCI\)](#)

Electrospray Ionization (ESI)

The electrospray ionization (ESI) technique transfers ions in solution into the gas phase¹.

This topic describes the following aspects of electrospray ionization:

- [Mechanism of Ion Desolvation](#)
- [Spectral Characteristics](#)

¹ Refer to the following papers for more information on the electrospray ionization process: Fenn, J. B.; Mann, M.; Meng, C. K.; Wong, S. F.; Whitehouse, C. M. *Mass Spectrometry Reviews* **1990**, 9, 37; Smith, R. D.; Loo, J. A.; Edmonds, C. G.; Barinaga, C. J.; Udseth, H. R. *Anal. Chem.* **1990**, 62, 882; Ikonomou, M. G.; Blades, A. T.; Kebarle, P. *Anal. Chem.* **1991**, 63, 1989.

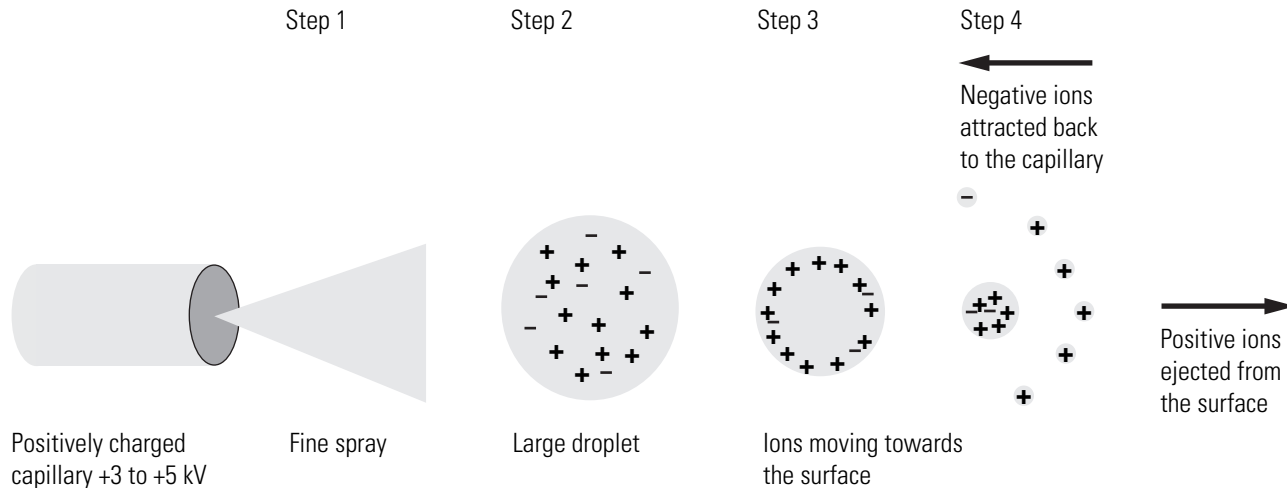
Mechanism of Ion Desolvation

To produce gas phase ions in ESI, the following sequence of events occurs:

1. The ESI capillary, to which a high voltage is applied, sprays sample solution into a fine mist of droplets that are electrically charged at their surface.
2. The electrical charge density at the surface of the droplets increases as solvent evaporates from the droplets until it reaches a critical point, known as the Rayleigh stability limit. At this critical point, the droplets divide into smaller droplets because the electrostatic repulsion is greater than the surface tension. The process repeats itself, forming smaller and smaller droplets.
3. From the very small, highly charged droplets, the force of electrostatic repulsion ejects sample ions into the gas phase.
4. The charged ESI capillary attracts gas phase ions of opposite charge and repels gas phase ions of the same charge.

The low vacuum of 1 Torr produced by the forepump draws both ionized molecules repelled by the charge on the capillary and neutral molecules in the gaseous phase into the MS detector through the entrance cone. [Figure 2](#) shows the steps in the formation of gas phase ions from highly charged droplets.

Figure 2. Positive ion electrospray mechanism



Spectral Characteristics

In the ESI mode, ionization takes place in the liquid phase. Polar compounds of low molecular weight (<1000 u) typically form singly charged ions by the loss or gain of a proton. Basic compounds (for example amines) can form a protonated molecule $[M + H]^+$, which can be analyzed in the positive ion polarity mode to give a peak at an m/z value of $M + 1$, where M equals the mass of the original molecule. Acidic compounds (for example sulphonic acids) can form a deprotonated molecule $[M - H]^-$, which can be analyzed in the negative ion polarity

mode to give a peak at an m/z value of $M - 1$. Because electrospray is a very soft ionization technique, there is usually little or no fragmentation and the spectrum contains only the protonated or deprotonated molecule.

Preformed ions can also include adducts. Adduct ions are produced by the interaction reaction between a molecule and an ionic species to form an ion that contains all the constituent atoms of the original molecule as well as one or more additional atoms. Common adducts are ammonium ions (NH_4^+), yielding an m/z value of $[M + 18]^+$, sodium ions (Na^+), yielding an m/z value of $[M + 23]^+$, and potassium ions (K^+), yielding an m/z value of $[M + 39]^+$.

Sample ions can carry a single charge or multiple charges. The number of charges carried by the sample ions depends on the structure of the analyte of interest and the carrier solvent. Because of multiple charging, you can use the ESI mode to analyze ions with molecular weights greater than 100 000 u. This makes ESI especially useful for the mass analysis of polar compounds including biological polymers and industrial polymers. The mass spectra for these compounds consist typically of a series of peaks corresponding to a distribution of multiply charged analyte ions.

You can run ESI in three ion polarity modes: positive, negative, or positive-negative switching. Because like charges repel each other, select the ion polarity mode that matches the polarity of your analytes:

- For acidic compounds, which form negative ions in solution, select the negative ion polarity mode.
- For basic compounds, which form positive ions in solution, select the positive ion polarity mode.
- For unknown mixtures, select the positive-negative switching mode.

Droplet size, surface charge, liquid surface tension, solvent volatility, and ion solvation strength affect the ESI process. Large droplets with high surface tension, low volatility, strong ion solvation, low surface charge, and high conductivity prevent good electrospray. The buffer type and buffer strength have a noticeable effect on sensitivity, making it important to choose these variables correctly.

Organic solvents such as methanol, acetonitrile, and isopropyl alcohol are superior to water for ESI. Volatile acids and bases can be used, but salt concentrations above 10 mM and strong acids and bases are extremely detrimental to the mass spectrometer.

The rules for a good electrospray are as follows:

- Keep salts out of the solvent system.
- Use organic/aqueous solvent systems and volatile acids and bases.
- Optimize the pH of the solvent system.

Atmospheric Pressure Chemical Ionization (APCI)

Atmospheric pressure chemical ionization (APCI) is a soft ionization technique that is used to analyze compounds of medium polarity that have some volatility.

This topic describes the following aspects of APCI:

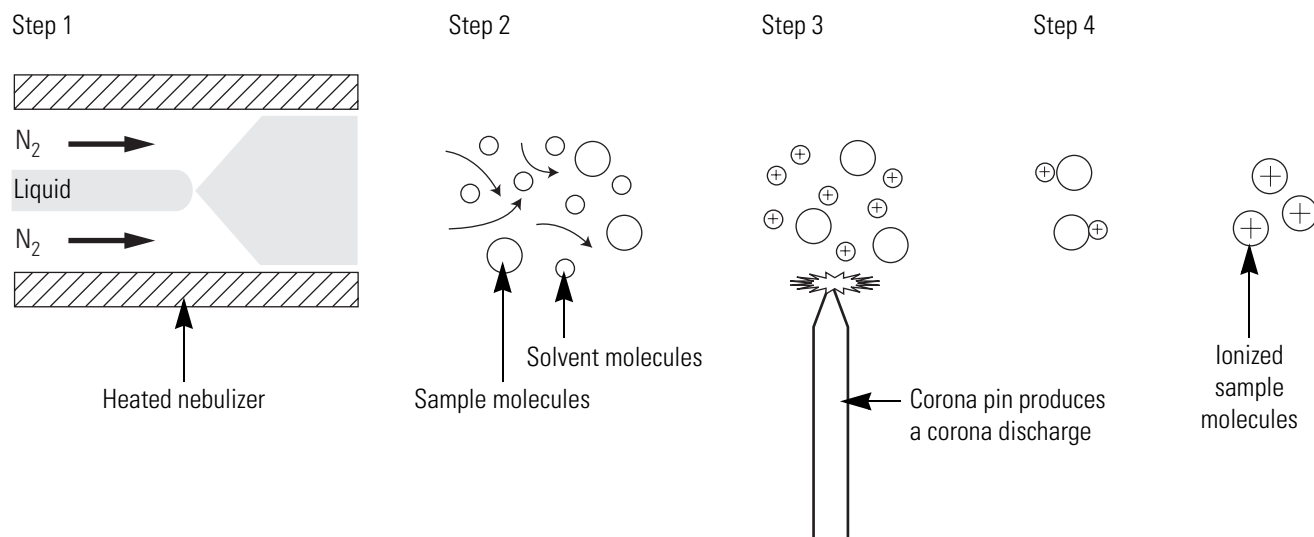
- [Mechanism of Ion Generation](#)
- [Spectral Characteristics](#)

Mechanism of Ion Generation

To produce ions in APCI, the following sequence of events occurs:

1. The APCI capillary sprays the sample solution into a fine mist of droplets.
2. A high temperature tube (the heated nebulizer) vaporizes the droplets.
3. A high voltage applied to a needle located near the exit end of the tube creates a corona discharge. Energized electrons produced by the corona discharge ionize the nitrogen nebulizing gas. The nitrogen ions react with the solvent molecules to form solvent ions.
4. The solvent ions react with sample molecules to form sample ions.

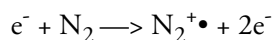
Figure 3. Positive ion APCI mechanism



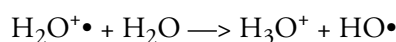
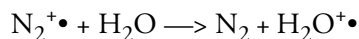
APCI is a gas phase ionization technique in which the gas phase acidities and basicities of the analyte and solvent vapor play an important role.

In the positive-ion mode, sample ionization occurs in a series of reactions that start with the electron-initiated cation formation. Typical examples of primary, secondary, and adduct ion formation are shown below:

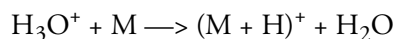
Primary ion formation



Secondary ion formation



Proton transfer



In negative-ion mode, $(M - H)^{-}$ is typically formed by the abstraction of a proton by OH^{-} .

Because the APCI process produces only singly charged ions, its use is limited to small molecules with molecular weights up to about 2000 u. Because the APCI process takes place in the gas phase, minor changes in most variables such as changes in buffer or buffer strength have no effect.

You can use APCI in the positive, negative, or positive-negative switching ion polarity mode. For most molecules, the positive-ion mode produces a stronger ion current. This is especially true for molecules with one or more basic nitrogen (or other basic) atoms. Exceptions to the general rule are molecules with acidic sites such as carboxylic acids and acid alcohols, which produce more negative ions than positive ions. Although the negative ion polarity mode generates fewer ions, it also generates less chemical noise than does the positive mode, making it more selective.

Spectral Characteristics

Like electrospray, APCI is a soft ionization technique and forms singly charged ions, either the protonated, $[M + H]^{+}$ or deprotonated, $[M - H]^{-}$ molecule depending on the selected ion polarity mode. Unlike electrospray however, APCI does not produce multiply charged ions and so is unsuitable for the analysis of high molecular weight compounds such as proteins or peptides.

Because APCI uses a heated probe to aid the desolvation process, it is not suitable for thermally labile (unstable) compounds, which can fragment in the ion source.

Scan Types

The Surveyor MSQ Plus MS detector provides two scan types:

- [Full Scan](#)
- [Selected Ion Monitoring \(SIM\) \(SIM\)](#)

Full Scan

A full scan provides a mass spectrum over a defined mass range. Because the MS detector has to monitor multiple m/z values during a chromatographic run, a full scan does not provide the sensitivity that SIM provides. The faster the chromatographic peaks elute the lower the sensitivity.

Selected Ion Monitoring (SIM)

In selected ion monitoring (SIM), you specify the monitoring of a particular ion or set of ions. Because only a few ions are monitored during a chromatographic run, SIM can provide lower detection limits than a full scan analysis. If you need to detect small quantities of a target compound, use SIM if you know the mass spectrum of your target compounds and the mass spectrum of the sample matrix.

SIM can improve the detection limit for quantitative analyses, but it can also reduce specificity. In SIM, only specific ions are monitored. Therefore, any compound that produces those ions appears to be the target compound, resulting in false positives.

Data Types

The Surveyor MSQ Plus MS detector provides three data types:

- [Profile Data Type](#)
- [Centroid Data Type](#)
- [MCA Data Type](#)

From the Xcalibur data system, you can acquire and display mass spectral data (intensity versus mass-to-charge ratio) in two of these data types (peak formats): Profile or Centroid. From the Tune window, you can acquire and display mass spectral data in all three data types.

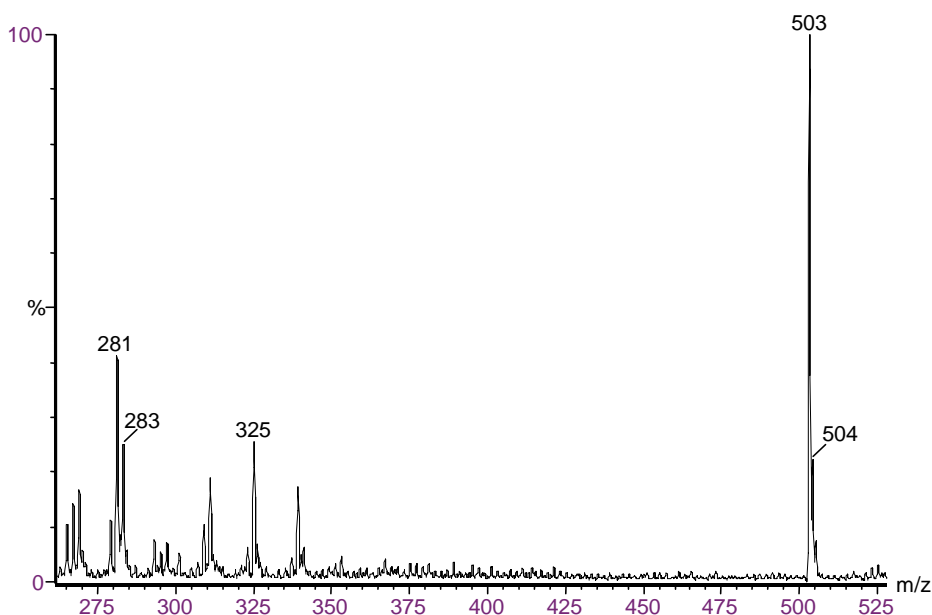
Profile Data Type

In the profile data type, you can see the shape of the spectral peaks in the mass spectrum. See [Figure 4](#).

Each atomic mass unit is divided into approximately 15 sampling intervals. The intensity of the ion current is determined at each of the sampling intervals. The intensity at each sampling interval is displayed with the intensities connected by a continuous line. In general, the profile data type is used when you tune and calibrate the MS detector so that you can easily see and measure mass resolution.

In general, the profile data type is used when you tune and calibrate the MS detector so that you can easily see and measure mass resolution.

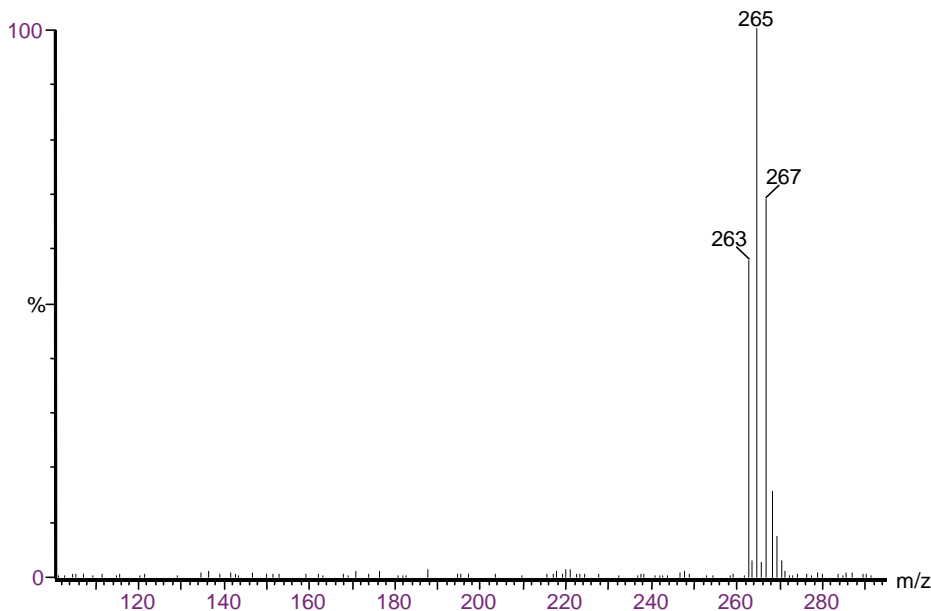
Figure 4. Full scan, profile peak format, spectrum of D-raffinose



Centroid Data Type

In the centroid data type, the mass spectrum appears as a bar graph. See [Figure 5](#). In this data type, Xcalibur sums the intensities for each 15 point sampling interval and displays the summed intensities versus the integral center of mass of the sampling interval. To increase the scan speed and reduce the disk space requirements, use the centroid data type for data acquisition. Data processing is also much faster for centroid data.

Figure 5. Full scan, centroid peak type, spectrum of pentachlorophenol



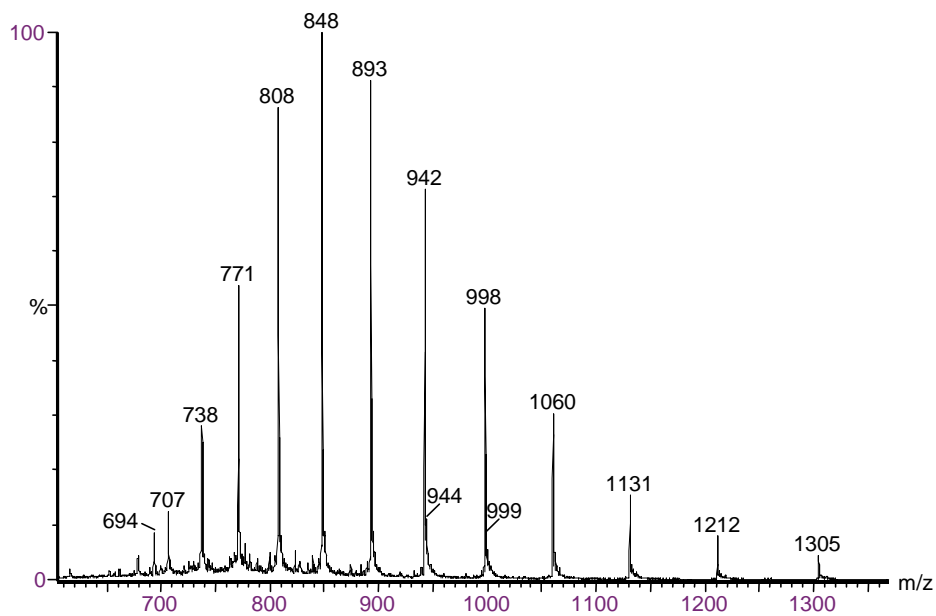
MCA Data Type

The third type of full scan acquisition is MCA. Such data can be thought of as “summed profile”, with only one intensity-accumulated scan being written to disk for a given experiment. See [Figure 6](#). As Xcalibur acquires each scan, it adds the intensity data to the accumulated summed data of previous scans.

An advantage of MCA is that although noise accumulates at the same rate as sample-related data, summing random noise over a number of scans reduces its effect, increasing the signal to noise ratio. A further advantage of MCA is that Xcalibur writes data to disk only at the end of an experiment, significantly reducing disk space requirements.

Because an MCA raw file contains only one scan, you cannot use the MCA for time-resolved data such as LC/MS analyses. Generally, you use MCA to acquire data when you perform infusion or loop injection experiments on samples of fairly weak concentration to enhance the signal. You can view the real-time spectrum and stop the data acquisition when you obtain the required results. MCA is particularly useful for the acquisition of raw data from the infusion of proteins and peptides.

Figure 6. Full scan MCA spectrum of horse heart myoglobin



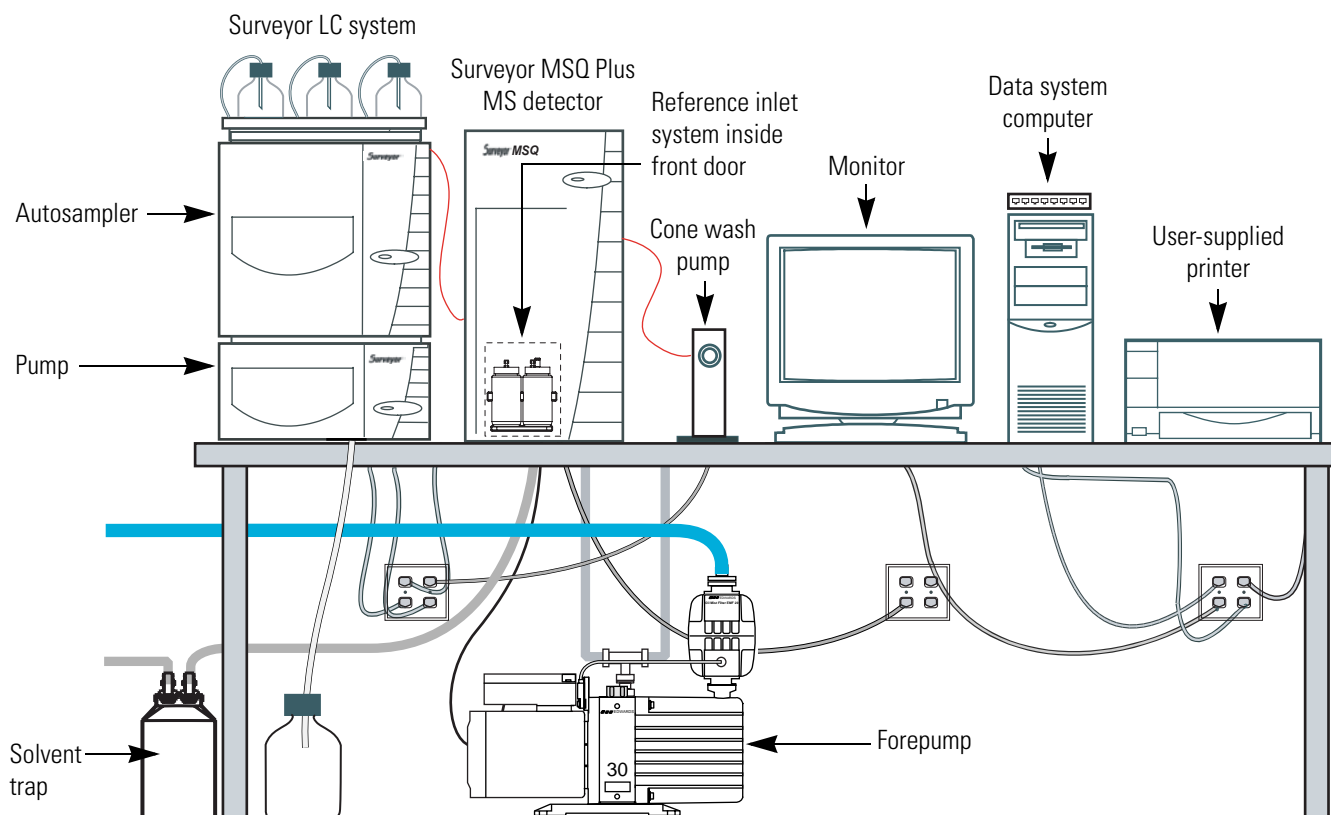
Functional Description

This chapter describes the principal components of the Surveyor MSQ Plus MS detector shown in [Figure 7](#) and their functions.

Contents

- [Liquid Chromatograph](#)
- [Reference Inlet System](#)
- [MS Detector](#)
- [Cone Wash System](#)
- [Data System](#)

Figure 7. Surveyor LC, Surveyor MSQ Plus MS detector, cone wash pump, forepump, and data system



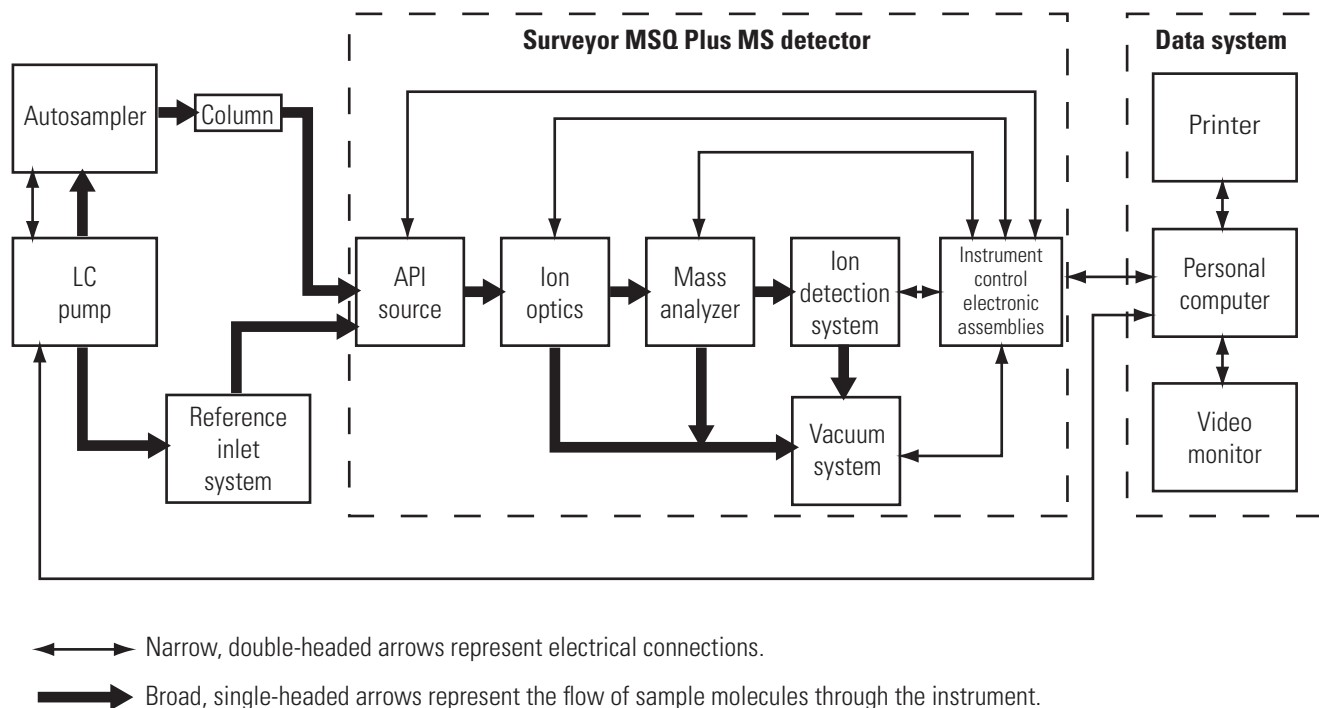
2 Functional Description

A functional block diagram of the Surveyor LC/MS integrated system with a Surveyor LC Pump, Surveyor Autosampler, and a Surveyor MSQ Plus MS detector is shown in Figure 8. A sample transfer line connects the Surveyor LC to the Surveyor MSQ Plus MS detector. The Surveyor LC system is usually installed to the left of the Surveyor MSQ Plus MS detector to minimize the length of tubing required to connect the outlet from the LC to the inlet of the mass spectrometer. Liquid chromatography systems supplied by other manufacturers can also be integrated with the Surveyor MSQ Plus MS detector.

Samples are injected by an autosampler into the mobile phase stream provided by the LC pump. As the stream passes through the LC column, the sample mixture partitions between a solid stationary phase of large surface area and the liquid mobile phase. The molecular structure of each component of the mixture determines when each component elutes from the column.

The outlet of the LC column can be directly connected to a UV detector, the MS detector, or both with a split flow tee. For instructions on connecting a split flow tee, see Appendix A, “Optimizing the LC Conditions.” Upon entering the API source of the MS detector, preformed sample ions are desolvated by electrospray ionization (ESI) or sample molecules are desolvated and ionized by atmospheric pressure chemical ionization (APCI). The vacuum system draws the vaporized molecules and ions into the ion optics. The ion optics focus and accelerate the resulting sample ions into the mass analyzer, where they are analyzed according to their mass-to-charge ratios. As the mass analyzer ejects sample ions, they are detected by an ion detection system, producing an ion current signal. The ion current signal, which is proportional to the number of ions in solution, is received and amplified by the system electronics and then passed on to the data system for further processing, storage, and display.

Figure 8. Functional block diagram of the Surveyor MSQ Plus LC/MS system



Liquid Chromatograph

The liquid chromatography devices controlled by the Xcalibur 2.0 data system includes those listed in [Table 1](#).

The LC pump is used to pump the mobile phase through the LC column and into the API source. The autosampler is used to introduce sample into the mobile phase stream.

Autosampler Start/Stop signals to the Surveyor MSQ Plus MS detector are provided by contact closure. See the *Surveyor MSQ Plus Getting Connected Guide* for information on connecting an autosampler to the Surveyor MSQ Plus MS detector by contact closure.



Configure the Xcalibur data system for your LC devices with the Xcalibur Instrument Configuration application. Access this application program by choosing **Start > All Programs > Xcalibur > Instrument Configuration** from the Windows® taskbar or by double-clicking the **Instrument Configuration** icon on the Windows desktop.

Table 1. Liquid chromatography devices controlled by the Xcalibur 2.0 data system with Quan Devices

Thermo Scientific Surveyor Autosampler	LC Pump	UV Detector
Surveyor Autosampler Plus	Surveyor LC Pump Plus	Surveyor PDA Plus Detector
Surveyor Autosampler Lite Plus	Surveyor MS Pump Plus	
Agilent Autosampler	LC Pump	UV Detector
Agilent 1100 Autosampler	Agilent 1100 isocratic	Agilent 1100 VWD
Agilent 1100 Micro	Agilent 1100 binary	Agilent 1100 MWD
Agilent 1100 Thermostatted	Agilent 1100 quaternary	Agilent 1100 DAD
Agilent 1100 WellPlate	Agilent 1100 capillary	

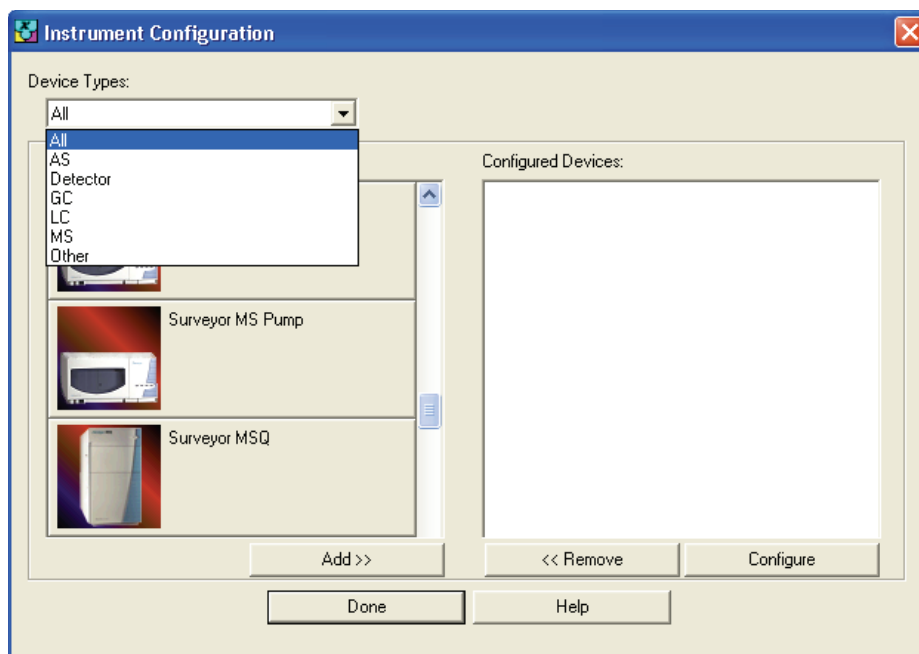
The Instrument Configuration application opens to the Instrument Configuration dialog box shown in [Figure 9](#). To minimize the number of devices displayed in the Devices list box, you can do one of the following:

- Select **All** in the Device Type box to display all the available devices controlled by Xcalibur 1.4.
- Select **LC** in the Device Type box to display only LC pumps.
- Select **AS** in the Device Type box to display only autosamplers and devices that include an autosampler.
- Select **Detector** in the Device Type box to display only detectors.
- Select **MS** in the Device Type box to display only MS detectors.

For more information on configuring the software for your LC devices, refer to the chapter in your *Surveyor MSQ Plus Getting Connected Guide* that pertains to your LC devices or the online Help available from the Xcalibur Instrument Configuration window.

For information on controlling your LC devices from the Xcalibur data system, refer to the online Help available from the Xcalibur Instrument Setup window. Front-panel (keypad) operation of the LC devices and maintenance procedures for the LC devices are described in the documentation provided with the LC.

Figure 9. Instrument Configuration dialog box



Reference Inlet System

You use the reference inlet system to introduce calibrant solution into the MS detector to perform a Full System Autotune or a Mass Scale Calibration, which is a subset procedure of the Full System Autotune procedure.

During an automated Full System Autotune procedure, the Surveyor MSQ Plus MS detector and instrument control software perform these steps:

- The MS detector infuses the calibrant solution and the software electronically adjusts the resolution of the peaks at low, mid, and high mass. The resolution of the peaks is adjusted to unity Dalton at their baselines.
- Performs a Mass Scale Calibration. During a Mass Scale Calibration, the software performs these steps:
 - Compares the empirically determined masses of the factory-supplied calibrant solution to a reference file of the same compound that contains the correct mass for each peak.
 - Adjusts the empirically determined masses in the acquired data file to match those in the reference file.

The software applies these adjustments to all subsequent acquisitions until you perform a new Full System Autotune or Mass Scale Calibration.

After installing the Surveyor MSQ Plus MS detector, a Thermo Fisher Scientific service engineer performs a Full System Autotune. You must repeat the procedure if you move the MS detector to a new location, install or update the Xcalibur data system, or the environment of your laboratory changes. If you notice a drift in the mass accuracy of your analyses, you should perform a Mass Scale Calibration.

Note Perform a mass calibration by selecting either the Full System Autotune or the Mass Scale Calibration option from the Instrument Tuning and Calibration wizard.

The reference inlet system consists of a reference inlet reservoir, a waste reservoirs nitrogen pressurization line, a PEEK delivery tube, and a Rheodyne microinjection (switching) valve. One end of the PEEK tubing is inserted into the reference reservoir and the other end of the tubing is attached to port 5 of the Rheodyne microinjection valve.

Pressuring the reference reservoir with nitrogen gas and switching the valve to the load position forces the calibrant solution through the tubing into a 500 μ L sample loop as shown in Figure 10. After the sample loop is filled, the valve switches to the inject position, allowing mobile phase to push the calibrant out of the sample loop and through the API probe as shown in Figure 11.



CAUTION The union that connects the Rheodyne microinjection (switching) valve to the API probe is a grounding union. Do **not** connect port 3 of the Rheodyne microinjection directly to the inlet of the API probe. Bypassing the grounding union could lead to instrument damage and personal injury.

2 Functional Description

Reference Inlet System

Figure 10. Microinjection (switching) valve in the Load position

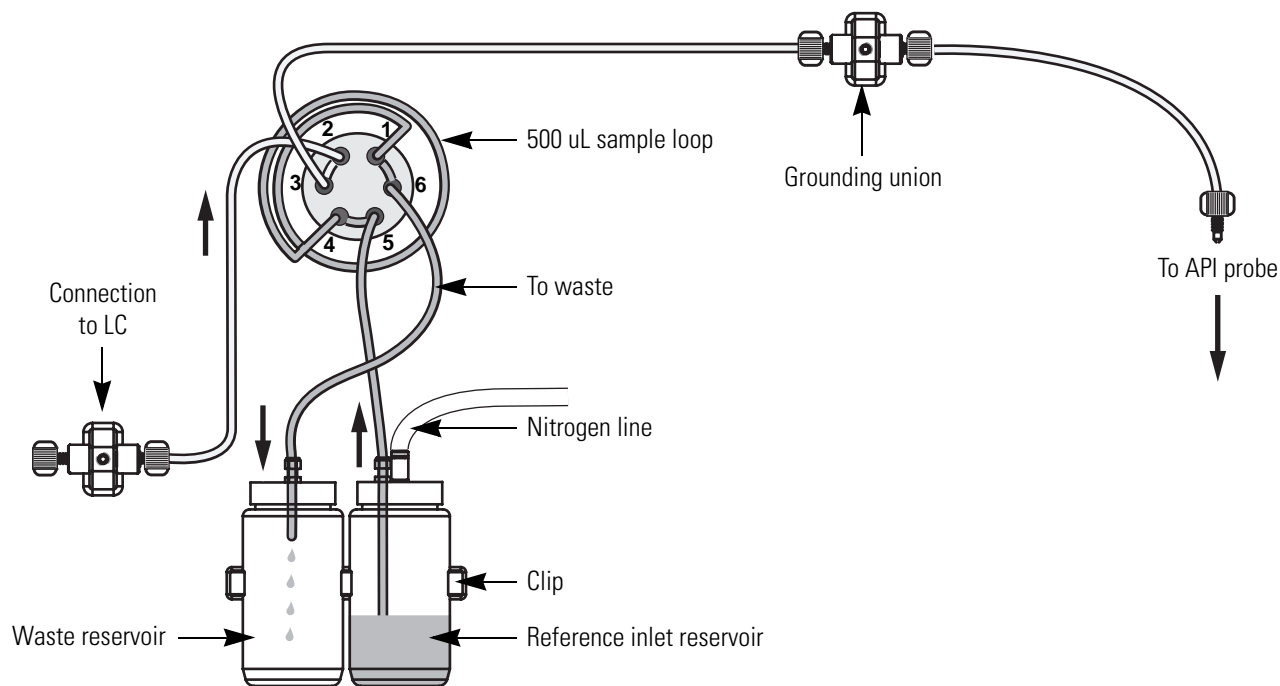
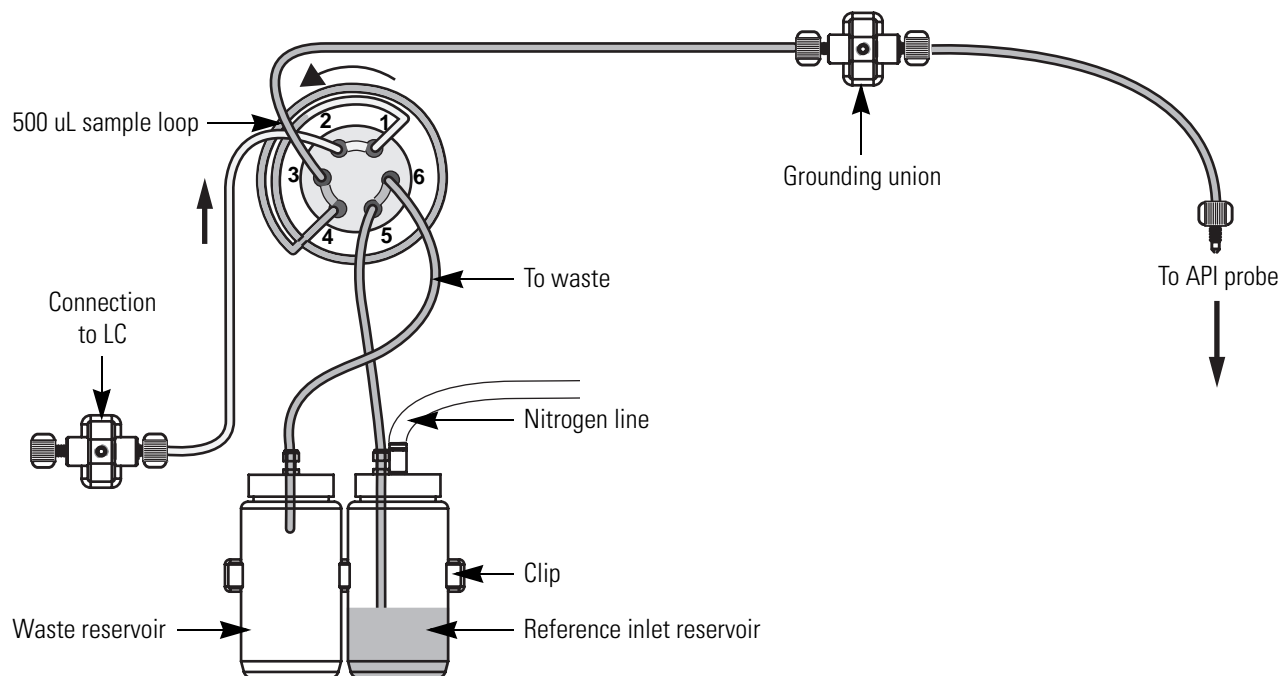


Figure 11. Microinjection (switching) valve in the Inject position



MS Detector

The MS detector provides sample ionization and mass analysis of samples injected with an autosampler or samples infused with the reference inlet system. The Surveyor MSQ Plus MS detector uses a quadrupole mass analyzer with an API source external to the mass analyzer.

The following components of the Surveyor MSQ Plus MS detector are described in this section:

- [Front Panel Status Indicator](#)
- [Rear Panel Controls and Connections](#)
- [Connection Between LC and MS Detector](#)
- [API Sources](#)
- [RF/dc Prefilter](#)
- [Mass Analyzer](#)
- [Ion Detection System](#)
- [Vacuum System](#)
- [Inlet Gas Hardware](#)

Front Panel Status Indicator

One light-emitting diode (LED) is located at the upper right corner of the front panel of the Surveyor MSQ Plus MS detector as shown in [Figure 12](#). [Table 2](#) lists the states of the Status LED.

Initially, when a Thermo Fisher Scientific service engineer installs the Surveyor MSQ Plus MS detector turns on the forepump, the status LED is red. As the system pumps down, the LED flashes yellow. After the vacuum reaches 10^{-4} Torr and the turbomolecular pump reaches its operating speed, the LED turns solid yellow.

If you vent the system for a brief period of less than 2 hour (for example to perform maintenance on the source block), wait 15 minutes after pumping down the system to place the MS detector in the Operate mode. If the system has been in a vented state for more than a brief period, pump the system down, and then wait a minimum of 8 hours before operating the system.

Figure 12. Front view of the Surveyor MSQ Plus MS detector



Table 2. States of Surveyor MSQ Plus MS Status LED

Instrument Status	Light
Vented	Red
Venting	Red
Pumping down	Flashing yellow
Under vacuum (above vacuum trip)	Red
Under vacuum (ready for use)	Yellow
Operate On (Surveyor MSQ Plus MS in use)	Green
Source enclosure open	Red

Rear Panel Controls and Connections

The rear panel of the Surveyor MSQ Plus MS detector is shown in [Figure 13](#). The rear panel controls and connections include the following:

- Mains On/Off
- Pump Out
- Mains In
- USB port
- User I/O panel
- Reset button
- Source line
- Backing line
- Exhaust line
- Gas In

The main power circuit breaker switch (labeled MAINS ON/OFF), the power source for the forepump (labeled PUMP OUT), and the connection to line power (labeled MAINS IN) are located in the lower-right corner of the rear panel of the MS Detector. In the Off (O) position, the circuit breaker removes all power to the MS detector, including the forepump (rotary vacuum pump). In the On (I) position, power is supplied to the MS detector and the forepump (rotary vacuum pump). In the standard operational mode, the circuit breaker is kept in the On (I) position.

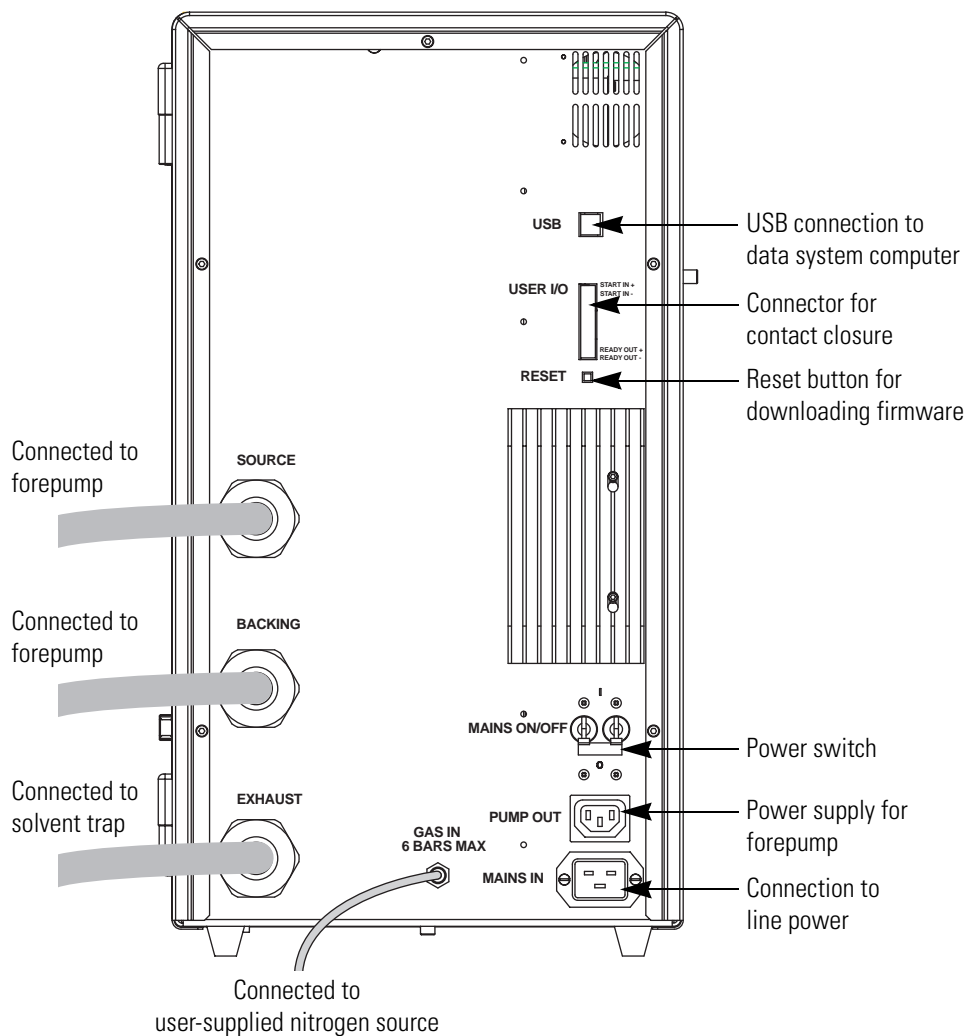
The power supply requirements for the Surveyor MSQ Plus MS detector are 230 Vac, regulated to $\pm 5\%$ at 50 or 60 Hz. The Surveyor MSQ Plus MS detector ships with power cords appropriate to its shipping destination.

A USB port, an eight-terminal contact closure connector (labeled USER I/O), and a reset button are located in the upper-right corner of the rear panel. A USB cable connects the Surveyor MSQ Plus MS detector to the data system computer. Hardwiring terminals 1 and 2 on the User I/O panel to the autosampler provides contact closure. Pressing the reset button downloads the software (firmware) for the Surveyor MSQ Plus MS detector from the data system.

Three manifolds are located on the left-side of the rear panel. The lines that exit the source and backing manifolds are connected to the forepump (also referred to as a backing pump, rotary vacuum pump, or roughing pump). The line that exits the exhaust manifold is connected to a solvent trap, which is connected to a user-supplied fume hood or industrial vent.

The connection to the user-supplied nitrogen source (labeled GAS IN) is located in the bottom-middle of the rear panel. The Surveyor MSQ Plus MS detector is connected to the user-supplied nitrogen source with 6 mm OD PTFE tubing.

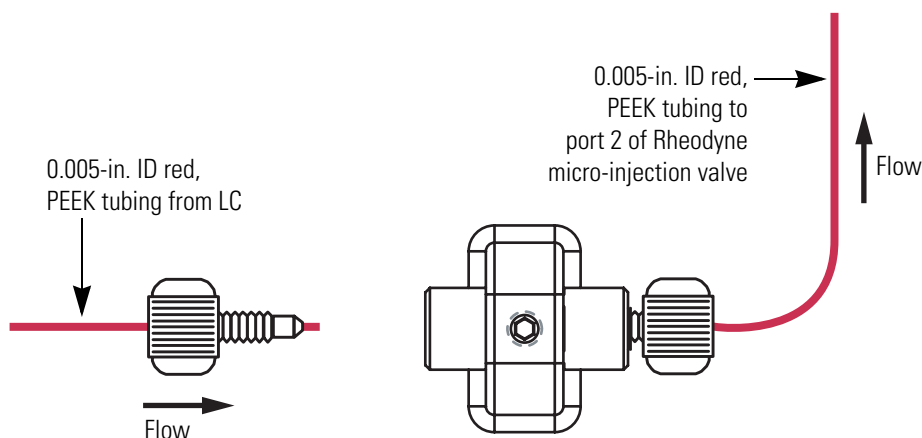
Figure 13. Rear panel of Surveyor MSQ Plus MS detector



Connection Between LC and MS Detector

The connection between the liquid chromatograph and the Surveyor MSQ Plus MS detector is a PEEK union. This union is located behind the front door of the MS detector to the left of the source block cover. See [Figure 14](#).

Figure 14. Connection between LC and MS detector



API Sources

The atmospheric pressure ionization (API) source forms gas phase sample ions from sample molecules that are contained in solution. The API source also serves as the sample interface between the LC and the MS detector. A catch on the left side of the source enclosure door shuts off the high voltage supply if the door is opened.

You can operate the API source using the following techniques:

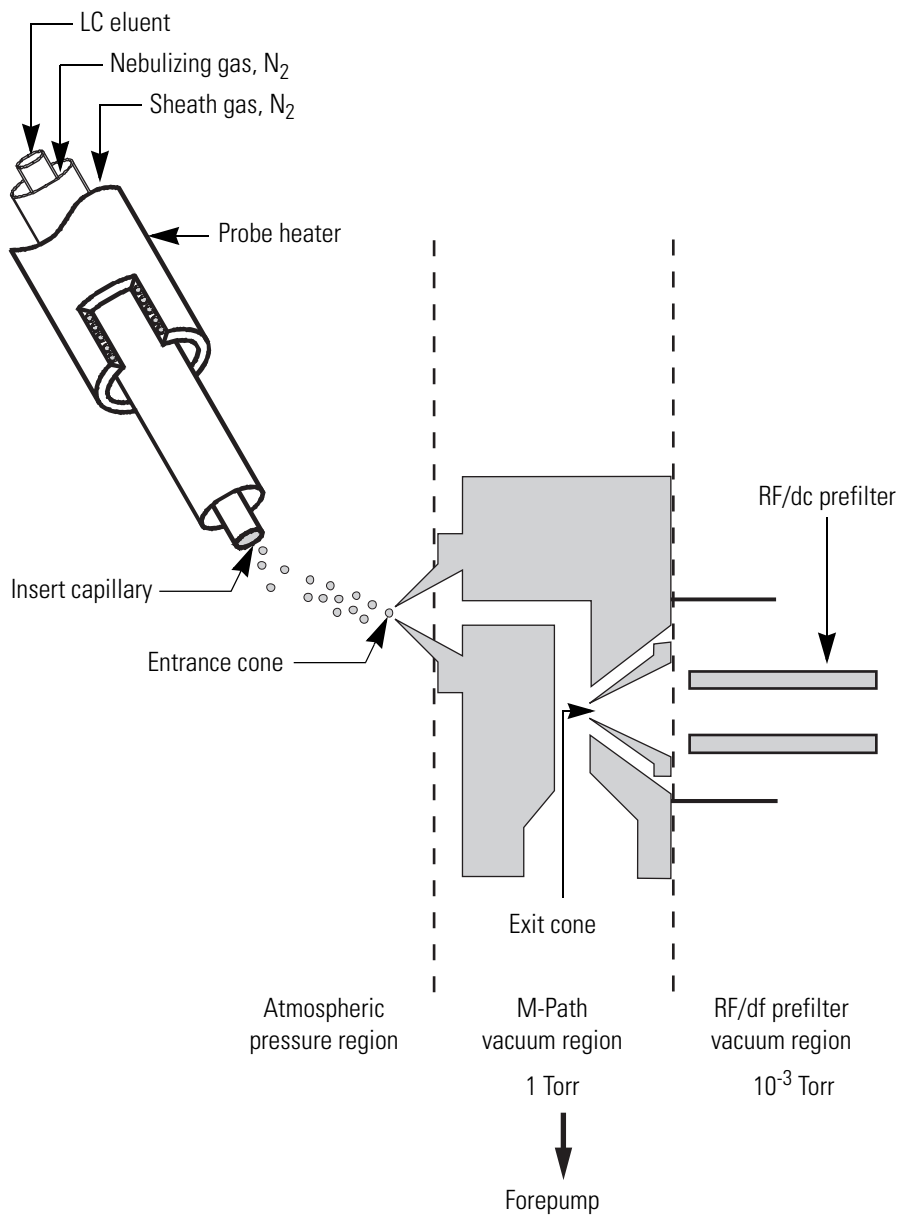
- [Electrospray Ionization \(ESI\)](#)
- [Atmospheric Pressure Chemical Ionization \(APCI\)](#)

Electrospray Ionization (ESI)

The sample, consisting of preformed ions in solution, enters the source through a stainless steel insert capillary held at a voltage of ± 1 to 5 kV. The insert capillary is surrounded by a tube that directs a concentric flow of nitrogen nebulizing gas past the droplets of liquid forming at the tip of the probe. The action of the nebulizing gas and the high voltage produces an aerosol of liquid droplets containing sample ions and solvent ions. A second concentric flow of nitrogen gas, referred to as the sheath gas, assists the ion evaporation process. This highly efficient desolvation process close to the entrance cone enables the routine use of high LC flow rates (up to 2 mL/min) with the ESI technique.

Drawn in by the low vacuum produced by the forepump, the desolvated ions enter the M-Path region through the entrance cone. As the ions exit the focusing region, they pass into the RF/dc prefilter. See [Figure 15](#).

Figure 15. View of the ESI probe, showing the principal components and the pressure regions



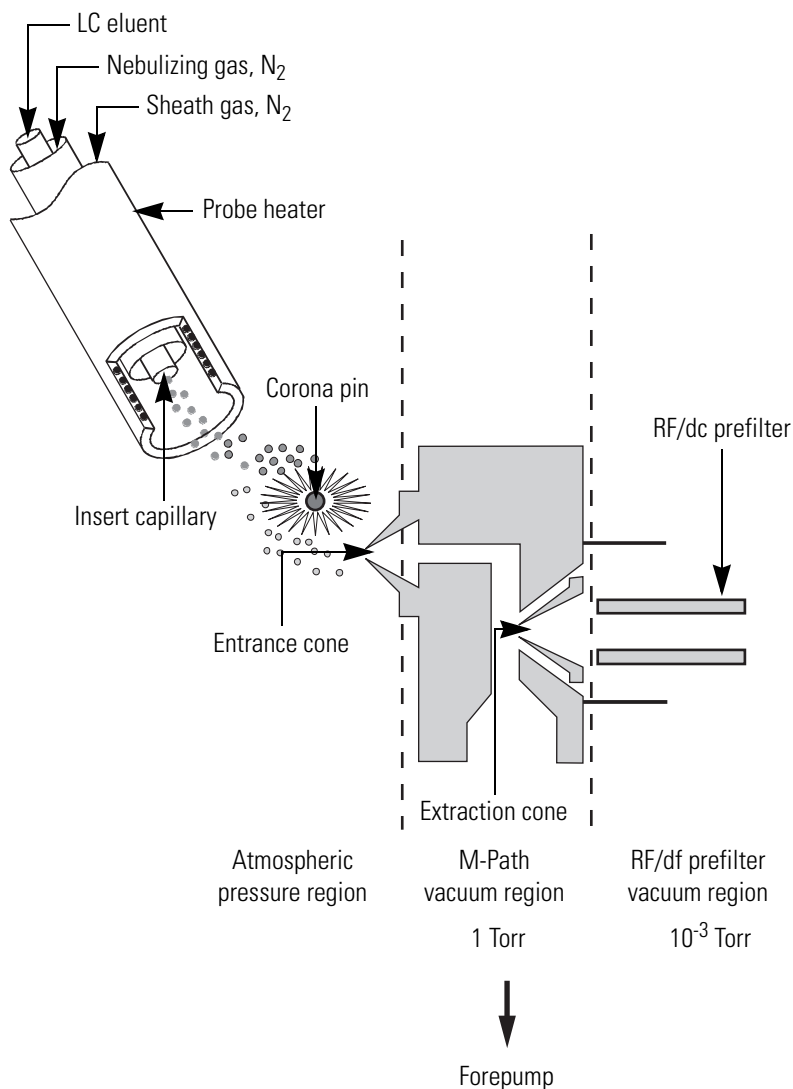
Atmospheric Pressure Chemical Ionization (APCI)

In contrast to electrospray, APCI is a gas phase ionization technique. The sample is carried to a spray region through a stainless steel insert capillary. The action of both the nebulizing gas and the heated probe lead to the formation of an aerosol. The desolvation process is assisted by a second concentric flow of nitrogen gas called the sheath gas.

Ionization occurs as the aerosol leaves the heated nebulizer region. A corona pin, which is mounted between the heated region and the entrance cone, ionizes the sample molecules with a discharge needle operating at a constant current of 2 to 10 μA in the positive polarity mode or 5 to 30 μA in the negative polarity mode.

The newly formed ions then enter the focusing region through the entrance cone and pass into the RF/dc prefilter region. See [Figure 16](#).

Figure 16. View of the APCI probe, showing the principal components and the pressure regions

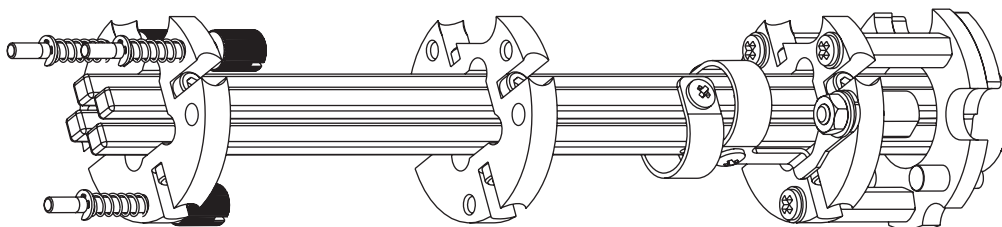


RF/dc Prefilter

The RF/dc prefilter focuses the ions produced in the API source and transmits them to the mass analyzer. The RF/dc prefilter is a square array of square-profile rods that acts as an ion transmission device and as a wide band-pass mass filter. See [Figure 17](#).

During ion transmission, the offset voltage is positive for the positive ion polarity mode and negative for the negative ion polarity mode. Increasing the offset voltage increases the kinetic energy of the ions along the axis of the quadrupole through the differential aperture. Allowable values for the RF lens bias are -10 V to $+10\text{ V}$. In the default Tune file (*default.tune*), the RF lens bias is set to 1.0 V . The default RF lens bias for the Autotune procedure is 0.5 V .

Figure 17. RF/dc prefilter, square array of square-profile rods



Mass Analyzer

The mass analyzer separates ions according to their mass-to-charge ratio and then passes them to the ion detection system. In the Surveyor MSQ Plus MS detector, the mass analyzer is a single quadrupole rod assembly.

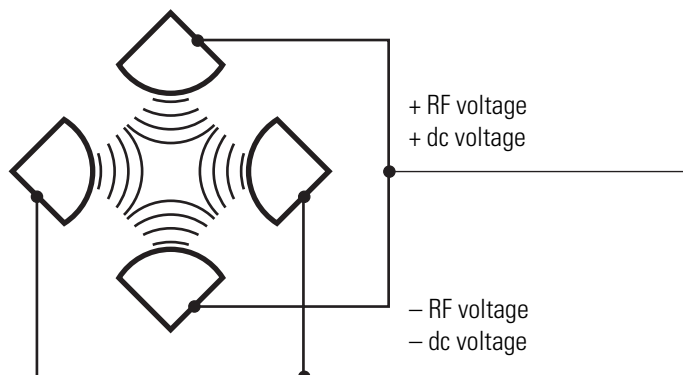
The mass analyzer is described in detail in the following topics:

- [RF and DC Fields Applied to the Quadrupoles](#)
- [Mass Analysis](#)

RF and DC Fields Applied to the Quadrupoles

In a quadrupole rod assembly, rods diagonally opposite each other in the array are connected electrically. Thus, the four rods can be considered to be two pairs of two rods each. Ac and dc voltages are applied to the rods and these voltages are ramped during the scan. Voltages of the same magnitude and sign are applied within the rods of each pair. Voltages equal in magnitude but opposite in sign (dc) and phase (RF) are applied to the different rod pairs. See [Figure 18](#).

The ac voltage applied to the quadrupole rods is of constant frequency. Because the frequency of this ac voltage is in the radio frequency range, it is referred to as **RF voltage**. The ratio of RF voltage to dc voltage determines the resolving power of the quadrupole and the ability of the MS detector to distinguish between ions of different mass-to-charge ratios.

Figure 18. Polarity of the RF and dc voltages applied to the rods of the mass analyzer

Mass Analysis

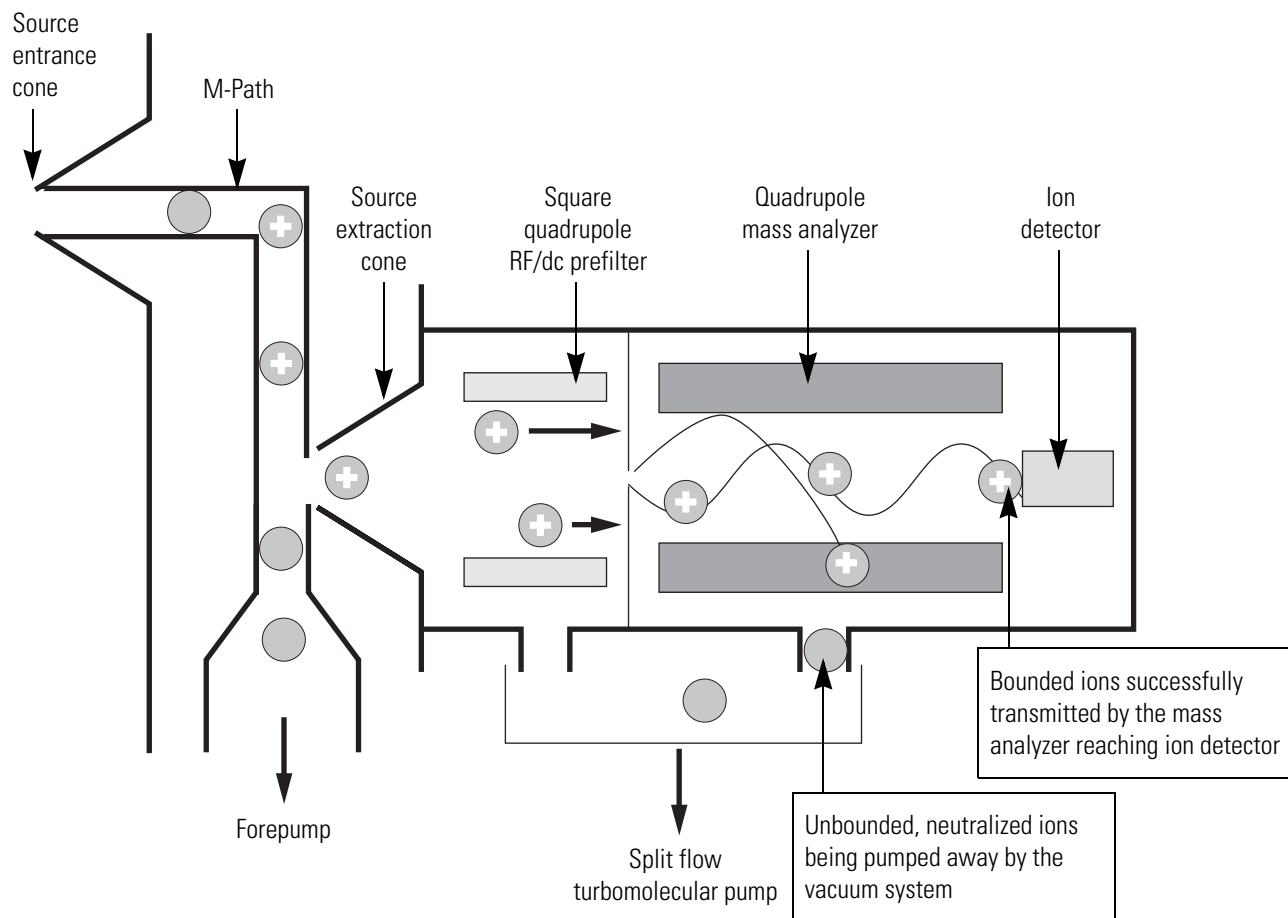
The mass analyzer in the Surveyor MSQ Plus MS detector is a square array of round rods. The rods are charged with a variable ratio of RF voltage and dc voltage. These potentials give rise to an electrostatic field that gives stable oscillations to ions with a specific mass-to-charge ratio and unstable oscillations to all others. The mass range for the Surveyor MSQ Plus MS detector is 17 to 2000 u at unit resolution.

At any given instant, one particular set of RF and dc voltage values is being applied to the mass analyzer rods. Under these conditions, only ions of one mass-to-charge ratio (for example, m/z 180) are maintained within bounded oscillations as their velocity carries them through the mass analyzer. During this same time, all other ions undergo unbounded oscillations. These ions strike one of the rod surfaces, become neutralized, and are pumped away by the vacuum system. See [Figure 19](#).

Then, at a later time, as the mass analyzer scans to a higher mass, both RF and dc voltages change, and ions of the next mass-to-charge ratio (for example, m/z 181) are allowed to pass, while all other ions (including m/z 180) become unstable and undergo unbounded oscillations. As the mass analyzer scans over the designated mass range, this process continues with ions of one mass-to-charge ratio after another being transmitted, as the RF and dc voltages change in value. At the end of the scan, the RF and dc voltages are discharged to zero, and the process is repeated.

The Surveyor MSQ Plus MS detector can scan the RF and dc voltages over the full mass range of the system (for example, m/z 17 to 2000) in as short a time as 0.2 s. However, under the conditions usually employed in mass analysis, such a scan should normally be done in about 2 or more seconds to fall within the upper limit of the calibrated scan rate of 1000 u/s.

Figure 19. Schematic of the RF/dc prefilter, mass analyzer, and ion detector



Ion Detection System

The Surveyor MSQ Plus MS detector is equipped with a high sensitivity, off-axis ion detection system that produces a high signal-to-noise ratio and allows for voltage polarity switching between positive ion and negative ion modes of operation. The ion detection system includes a 10 kV conversion dynode and a channel electron multiplier. The ion detection system is located at the rear of the vacuum manifold behind the mass analyzer. See [Figure 20](#).

The conversion dynode is a concave metal surface that is located at a right angle to the ion beam. A potential of +10 kV for negative ion detection or -10 kV for positive ion detection is applied to the conversion dynode. When an ion strikes the surface of the conversion dynode, one or more secondary particles are produced. These secondary particles can include positive ions, negative ions, electrons, and neutrals. When positive ions strike a negatively charged conversion dynode, the secondary particles of interest are negative ions and electrons. When negative ions strike a positively charged conversion dynode, the secondary particles of interest are positive ions. These secondary particles are focused by the curved surface of the conversion

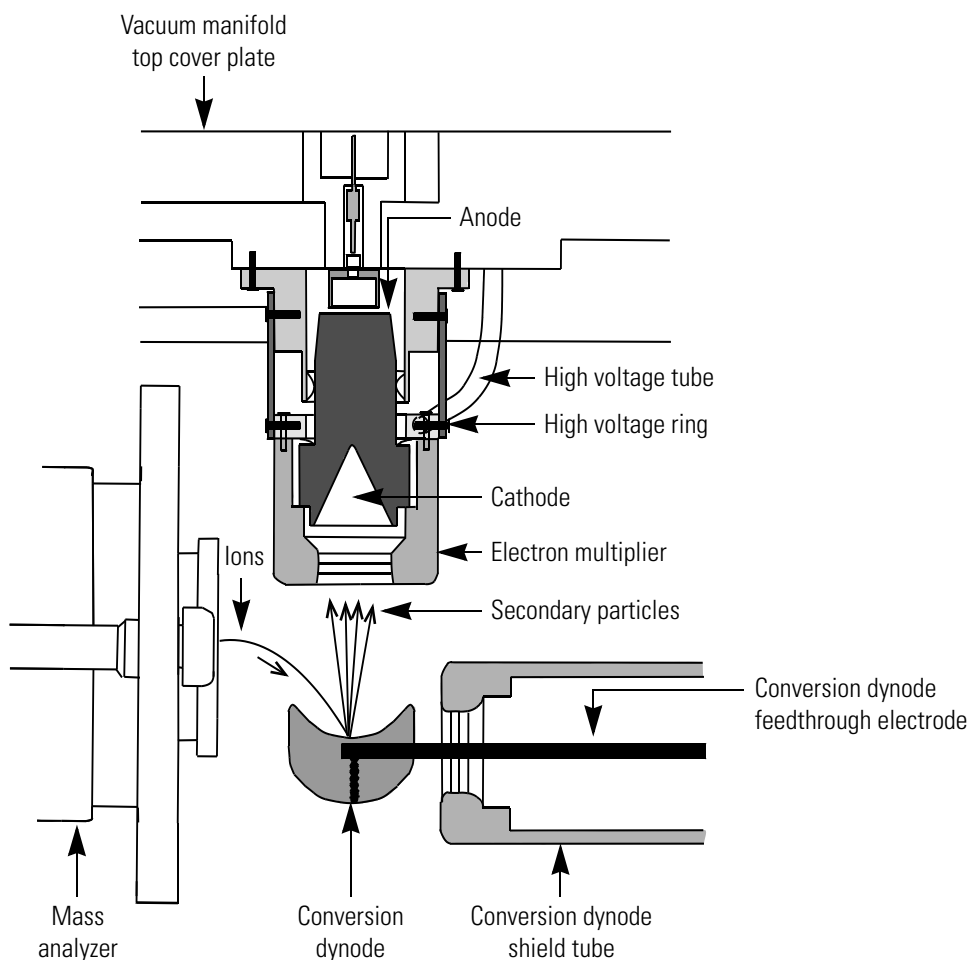
dynode and are accelerated by a voltage gradient into the electron multiplier. The conversion dynode shield, tube, and disk shield the vacuum manifold from the electric field produced by the conversion dynode.

The electron multiplier is mounted on the top cover plate of the vacuum manifold next to the mass analyzer. The electron multiplier includes a cathode and an anode. The cathode of the electron multiplier is a lead-oxide, funnel-like resistor. A potential of up to -2.5 kV is applied to the entrance of the cathode by the high voltage ring. The exit end of the cathode (at the anode) is near ground potential.

The anode of the electron multiplier is a small cup located at the exit end of the cathode. The anode collects the electrons produced by the cathode. The anode screws into the anode feedthrough in the top cover plate.

Secondary particles from the conversion dynode strike the inner walls of the electron multiplier cathode with sufficient energy to eject electrons. The ejected electrons are accelerated farther into the cathode, drawn by the increasingly positive potential gradient. Due to the funnel shape of the cathode, the ejected electrons do not travel far before they again strike the inner surface of the cathode, thereby causing the emission of more electrons. Thus, a cascade of electrons is created that finally results in a measurable current at the end of the cathode where the electrons are collected by the anode. The current collected by the anode is proportional to the number of secondary particles striking the cathode.

Figure 20. Cross sectional view of the ion detection system, showing the electron multiplier and the conversion diode



Typically, the electron multiplier is set to a gain of about 3×10^5 (which means that for each ion or electron that enters, 3×10^5 electrons exit). The current that leaves the electron multiplier by way of the anode is converted to a voltage by the electrometer circuit and recorded by the data system.

The ion detection system of the Surveyor MSQ Plus MS detector increases signal while maintaining a low level of noise. The high voltage applied to the conversion dynode results in a high conversion efficiency and increased signal. That is, for each ion striking the conversion dynode, many secondary particles are produced. The increase in conversion efficiency is more pronounced for more massive ions than for less massive ions.

Because of the off-axis orientation of the ion detection system relative to the mass analyzer, neutral molecules from the mass analyzer tend not to strike the conversion dynode or electron multiplier. As a result, the noise from neutral molecules is greatly reduced.

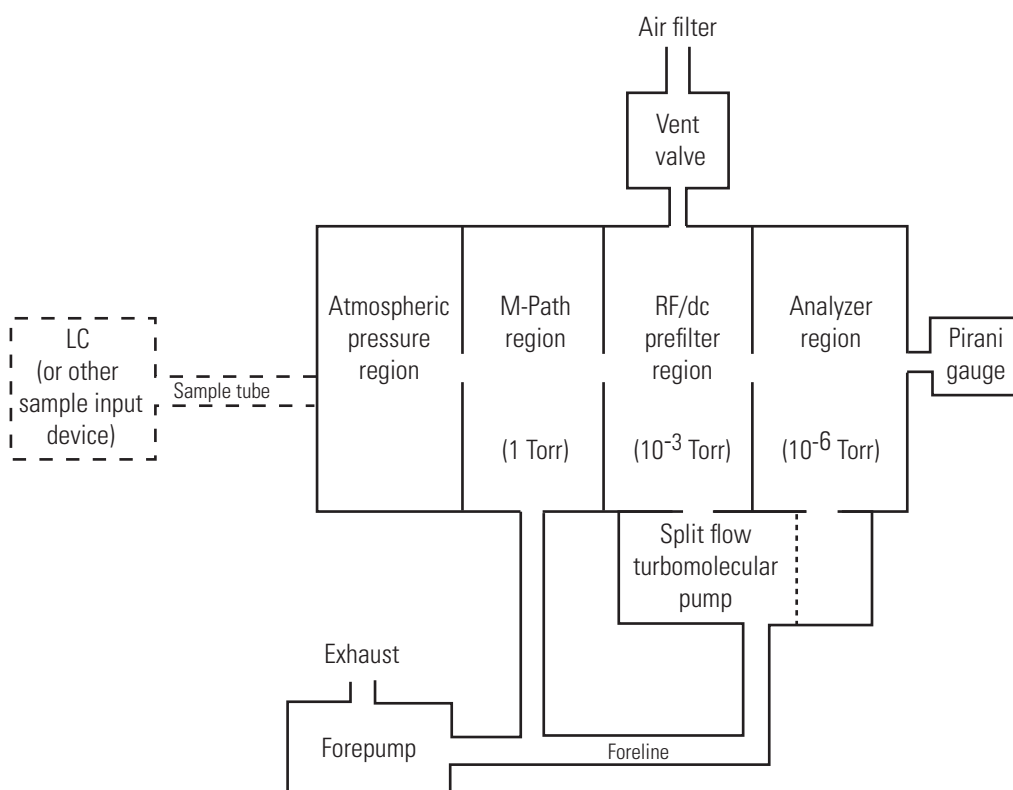
Vacuum System

The vacuum system evacuates the region around the ion optics, mass analyzer, and ion detection system. The principal components of the vacuum system include the following:

- Vacuum Manifold
- Turbomolecular Pump
- Forepump
- Pirani Gauge
- Vent Valve

A functional block diagram of the vacuum system is shown in Figure 21.

Figure 21. Functional block diagram of the vacuum system



Vacuum Manifold

The vacuum manifold encloses the ion optics, mass analyzer, and ion detection system assemblies. The vacuum manifold is a thick-walled, aluminum chamber with a removable top cover plate, machined flanges on the front, sides, and bottom, and various electrical feedthroughs and gas inlets.

The vacuum manifold is divided into three chambers by two baffles. The region inside the first chamber, called the M-path region, is evacuated to 1 Torr by the forepump. The region inside the second chamber, called the transfer lens region, is evacuated to 10^{-3} Torr by the interstage port of the split-flow turbomolecular vacuum pump. The region inside the third chamber, called the mass analyzer region, is evacuated to 10^{-6} Torr by the high vacuum port of the split-flow turbomolecular pump.

Turbomolecular Pump

A Balzers-Pfeiffer TMH 260-130 split-flow turbomolecular pump provides the vacuum for the transfer lens and mass analyzer regions of the vacuum manifold. The turbomolecular pump mounts onto the underside of the vacuum manifold with two 4 mm socket screws. The interstage port of the turbomolecular pump, which evacuates the transfer lens region, is rated at 125 L/s. The high vacuum port of the turbomolecular pump, which evacuates the mass analyzer region, is rated at 200 L/s. Under normal operating conditions the pump provides a vacuum of approximately 10^{-3} Torr in the transfer lens region, and 10^{-6} Torr in the mass analyzer region.

The main power circuit breaker switch turns the power to the turbomolecular pump On or Off. The turbomolecular pump controller regulates the power provided to the turbomolecular pump. A fan that draws air in from the underside of the instrument cools the turbomolecular pump.

Forepump

An Edwards forepump (also known as a roughing pump, backing pump, or rotary pump) establishes the vacuum necessary for the proper operation of the turbomolecular pump. The forepump also evacuates the M-path region of the vacuum manifold. The pump has a maximum displacement of 30 m³/h and maintains a minimum pressure of approximately 100 Pa (0.75 Torr).

The forepump is connected to the turbomolecular pump by a section of 2.54 cm (1-in.) ID reinforced PVC tubing. The power cord of the forepump is plugged into the outlet labeled PUMP OUT on the rear panel of the Surveyor MSQ Plus MS detector. This outlet supplies power to the forepump and is controlled by the main power circuit breaker switch. The Edwards forepump has an On/Off switch that must be turned to the On position to operate the forepump.



CAUTION Always plug the forepump power cord into the outlet labeled PUMP OUT on the rear panel of the Surveyor MSQ Plus MS detector. **Never** plug it into a wall outlet. Failure to follow these instructions could lead to instrument damage and personal injury.

Pirani Gauge

A Pirani gauge measures the pressure in the analyzer region of the vacuum manifold.

Vent Valve

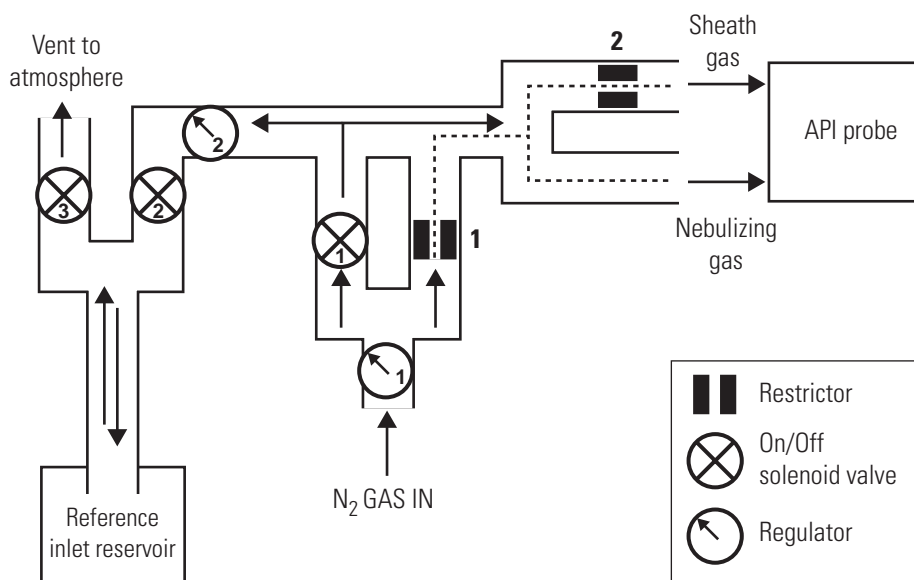
The vent valve allows the vacuum manifold to be vented to air that has been filtered through a metal mesh. The vent valve is a solenoid-operated valve. The vent valve is closed when the solenoid is energized.

Inlet Gas Hardware

Nitrogen gas is used as both the nebulizing gas and the sheath gas for the API probe. Nitrogen gas is also used to pressurize the reference inlet reservoir that contains the calibration solution for the MS detector. Dry nitrogen [nominally set to 520 kPa (75 psi) or 450 kPa (45 psi), 99% purity] enters the Surveyor MSQ Plus MS detector through a 6 mm port labeled GAS IN located on the rear panel of the MS detector. See [Figure 13](#) on [page 22](#). The inlet gas hardware then controls the flow of nitrogen gas to the API probes and the reference inlet reservoir.

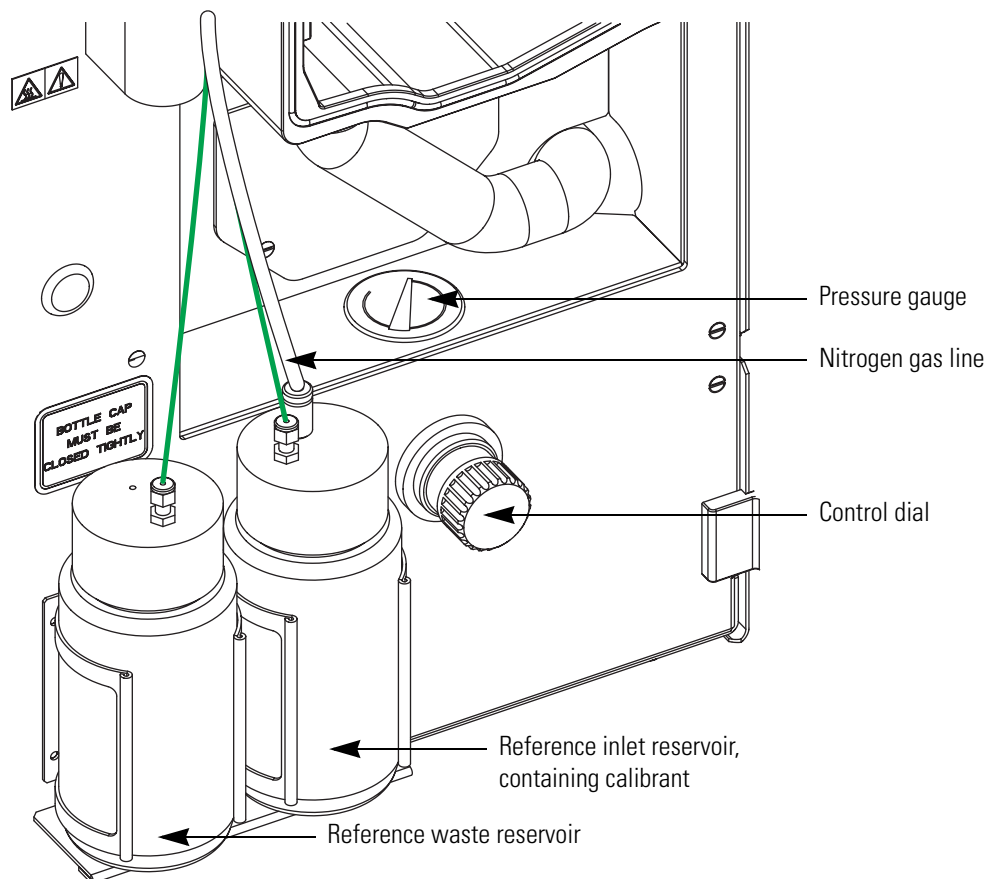
As [Figure 22](#) shows, the inlet gas hardware consists of two regulators, three solenoid valves, and two restrictors.

Figure 22. Functional block diagram of the inlet gases hardware



The first regulator, which is located on the front of the MS detector below the source compartment, as shown in [Figure 23](#), limits the flow of nitrogen gas to the API probe. You nominally set this regulator to 5.2 bar (75 psi) for the ESI mode or 3.1 bar (45 psi) for the APCI mode.

Figure 23. User controlled auxiliary nitrogen regulator



The second regulator limits the flow of nitrogen gas to the reference inlet reservoir. This regulator, which is inside the MS detector, is preset at the factory.

Note Call a Thermo Fisher Scientific service representative if you suspect that the nitrogen regulator inside the MS detector is malfunctioning.

The first solenoid valve is the On/Off control for the flow of nitrogen gas into the Surveyor MSQ Plus MS detector. To turn this valve on or off, you click the Nitrogen Gas On/Off toggle button in the Per Method Parameters table of the Tune window (see [Figure 24](#)). After you turn on the nitrogen gas, you can hear the jet of nitrogen gas flowing into the API probe.

The second solenoid valve is the On/Off control for the flow of nitrogen gas into the reference inlet reservoir. The third solenoid valve is the On/Off control for venting the reference inlet reservoir to the atmosphere. When the nitrogen gas flow is On, selecting either a Full System Autotune or a Mass Scale Calibration causes the system to alternate between pressurizing and depressurizing the reference inlet reservoir.

Clicking the Inject From Ref. Inlet button (in the Per Method Parameters table) has a similar effect. When you click this toggle button, nitrogen gas pressurizes the reference inlet reservoir. Pressurizing the reference inlet reservoir pushes the calibrant out of the reference inlet reservoir bottle and into the 500 μ L sample loop attached to the microinjection valve of the MS detector. After the sample loop fills with calibrant, the microinjection valve switches to the inject position. With the valve in the inject position, the sample loop is open to the mobile phase stream from the LC pump. The stream pushes the calibrant out of the sample loop and through the API probe. After the microinjection valve switches to the inject position, the third solenoid valve switches to the On position, allowing the nitrogen gas to vent to atmosphere and depressurizing the reference inlet reservoir.

The first restrictor allows a constant low flow of nitrogen gas to the API probe when the first solenoid valve is turned off. See dashed line shown in [Figure 22](#). The second restrictor shown in [Figure 22](#) limits the flow of sheath gas and acts as a split flow regulator by forcing the majority of the gas flow to the API probe through the tubing for the nebulizing gas.

Note There is a constant low flow of nitrogen gas through the MS detector when it is not in use. This low flow of nitrogen gas maintains a positive pressure in the source compartment, preventing fumes from the solvent trap connected to the detector's exhaust port from being drawn into the detector. Depending on the API mode and whether the instrument is in use, the nitrogen consumption is as follows:

- The ESI mode consumes approximately 720L/hr.
- The APCI mode consumes approximately 480 L/hr.
- The Standby mode consumes approximately 20 to 50 L/hr.

Figure 24. View of the Per Method Parameters table

Description	Readback	Setpoint
Tune Control		
Probe Temperature (°C)	0	550
Needle (kV)	0.0	3.0
RF Lens Bias (V)	0.0	0.5
Ion Energy (V)	n/a	0.5
LM Res	n/a	12.7
HM Res	n/a	12.5
Acquisition Control		
Profile Resolution	32 points/da	32 points/da
Retention Time (mins)	0.00	n/a
General Control		
Operate	n/a	Off <input type="checkbox"/>
Nitrogen Gas	n/a	On <input checked="" type="checkbox"/>
Ionization Mode	n/a	ESI
Sequence Control		
Inject from Ref. Inlet	n/a	Started <input checked="" type="checkbox"/>

Toggle buttons

Cone Wash System

The API source on the Surveyor MSQ Plus MS detector includes a self-cleaning solvent delivery system (the cone wash). This makes the source robust and productive, greatly increasing the number of samples that can be analyzed before maintenance is required.

The self-cleaning API source delivers a constant, low flow of solvent to the edge of the inlet orifice. See Figure 25. This low flow of wash solvent prevents the build-up of non-volatile components during LC/MS analysis that occurs with typical chromatographic buffers (for example phosphates and ion pairing agents) and dramatically extends the length of time possible for analyses.

The cone wash system consists of a cone wash pump, PEEK tubing, and a cone wash nozzle. Red, 0.005-in. ID, PEEK tubing connects the cone wash pump to the Surveyor MSQ Plus MS detector. Inside the Surveyor MSQ Plus MS detector, the red, PEEK tubing is connected to one end of a union. Green 0.030-in. ID, PEEK tubing is connected to the other end of the union. The green, PEEK tubing is connected to the back of the source block behind the cone wash nozzle.

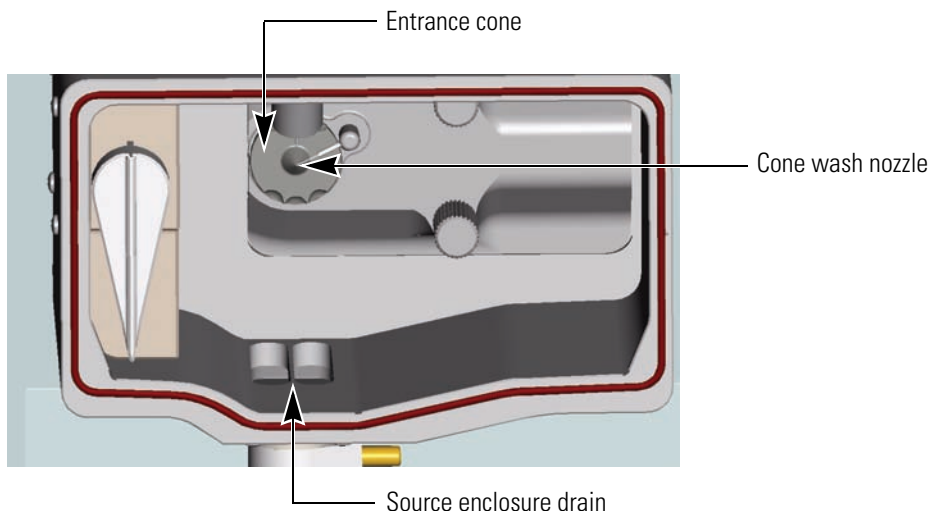
When you are using the cone wash system, adjust the cone wash nozzle so that solvent flowing out its tip just touches the orifice of the entrance cone as it falls towards the drain. See Figure 25. When you are not using the cone wash system, adjust the cone wash nozzle so that it faces away from the entrance cone. Storing the cone wash nozzle in the 12 o'clock to 2 o'clock position helps to prevent its blockage.



CAUTION The corona needle is very sharp. Do **not** attempt to adjust the position of the cone wash nozzle before you turn the corona pin knob to its vertical position.

Instructions for connecting the cone wash pump to the MS detector are included in the *Surveyor MSQ Plus Getting Connected Guide*.

Figure 25. View of the source enclosure



Data System

Xcalibur is the data system that controls the modules of your LC/MS system and Server is the software that handles the communication between the Surveyor MSQ Plus MS detector and the data system PC. The Xcalibur data system also processes data that is acquired by the Surveyor MSQ Plus MS detector. Information about the status of the Surveyor MSQ Plus MS detector is available from the Xcalibur HomePage - Information view and from the Server "LED" icon.

The data system for the Surveyor MSQ Plus MS detector includes the following:

- [Computer Hardware](#)
- [Xcalibur Software](#)
- [Surveyor MSQ Plus Server](#)
- [Tune Window](#)
- [Printer](#)

Computer Hardware

The data system computer satisfies the following minimum system requirements:





- Intel® Pentium® 4 at 2.4 GHz processor
- 256 MB of random access memory (RAM)
- 40 GB HDD
- CD-R drive
- USB adapter (data system to MS detector)
- Ethernet adapter (data system to local area network)
- 1.44-MB, 3.5-in. diskette drive
- Video Graphics card and monitor capable of 1024 × 768 resolution and 65536 colors (16-bit color quality).

For more information about the computer, refer to the manuals that come with it.

Xcalibur Software

Xcalibur™ software controls the Surveyor MSQ Plus MS detector and a variety of liquid chromatography devices. The Xcalibur software package for the Surveyor MSQ Plus MS detector includes the application programs listed in Table 3. Xcalibur Home Page version 2.0 is required for the MSQ 2.0 Plus version of the Surveyor MSQ Plus detector software. The firmware versions of the LC devices controlled by Xcalibur 2.0 with Quan Devices are listed in your *Surveyor MSQ Plus Getting Connected Guide*.

Table 3. Xcalibur software application programs

Desktop Icon	Program Name	Filename
	Xcalibur	Homepage.exe
	Instrument Configuration	Xconfig.exe
	Tune	MSinst.exe
	Signal to Noise Calculator	SigNoise.exe

When you launch Xcalibur from the Windows desktop by clicking its application icon, the Home Page opens to show a ‘road map’ view of the data system. The icons shown on the road map provide an easy way to access all the major modules of the data system. See Figure 26. In addition, Xcalibur runs the Surveyor MSQ Plus Server.

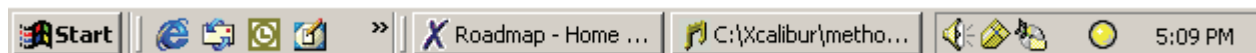
Figure 26. Xcalibur Home Page – Road Map view



Surveyor MSQ Plus Server

The Server is the software that handles all communication between the Surveyor MSQ Plus MS detector and its controlling PC. When you activate the Server (by running either Xcalibur or Tune), the Server “LED” icon is displayed in the System Tray of the Windows Taskbar, just to the left of the time display.

Figure 27. View of taskbar, showing the Server icon



The Server icon provides you with information about the status of the Surveyor MSQ Plus MS detector and mimics a tri-state LED display (red, green, yellow) showing:

- Steady red when the mains are on and the instrument is vented or an error has occurred within the system
- Flashing yellow when the instrument is pumping down
- Steady yellow when the instrument is pumped down but is not in the Operate mode
- Steady green when the instrument is pumped down and is in the Operate mode

Right-clicking the Server icon opens the shortcut menu shown in [Figure 28](#). This menu allows you to do the following:

- Choose **Manual Tune** to open the Tune window, where you can optimize the performance of your Surveyor MSQ Plus MS detector for a specific application.
- Choose **Instrument Tune and Calibration** to display the Instrument Tuning and Calibration wizard.
- Choose **Vent** to turn off the turbomolecular pump and vent the vacuum. If the system is not under vacuum, the menu contains the Pump command. Choose **Pump** to pump down the system. The Edwards forepump is not turned off by venting the system. To turn off the Edwards forepump without turning off the MS detector, set the On/Off switch on the forepump to the Off position.
- Choose **Exit** to close the server. If Xcalibur is running, exiting the server is not allowed and the error message shown in [Figure 29](#) appears.

Figure 28. Server shortcut menu

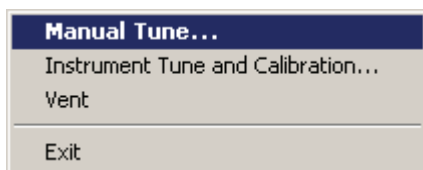
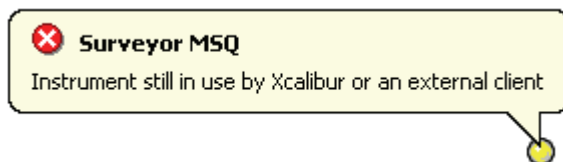


Figure 29. Error message displayed when Xcalibur is running and you attempt to exit the Server application



Tune Window

The Surveyor MSQ Plus Tune window allows you to optimize the MS detector parameters (manually tune) for your analytes and acquire data to a raw file (.raw). When you finish tuning your MS detector for a compound of interest, you save the current values of the tuning parameters in a tune file (.tune). In ESI, you empirically determine the optimal probe temperature, needle voltage, and cone voltage for each application. In APCI, you empirically determine the optimal probe temperature, corona current, and cone voltage for each application.

Tip There are three ways to open the Tune window:

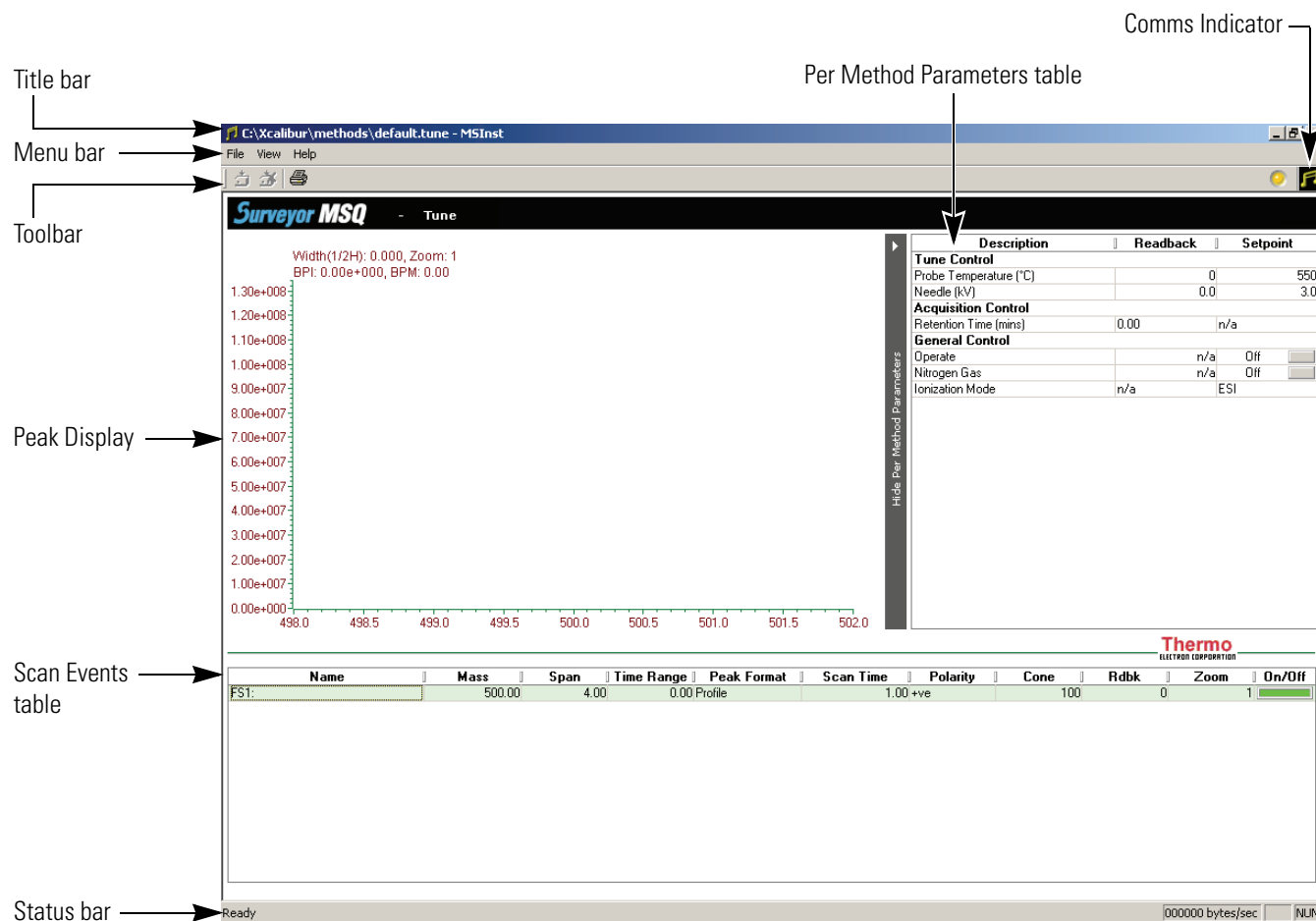
- Launch Xcalibur. Then, double-click the Server icon that appears in the System Tray of the Windows taskbar.
- Double-click the Tune icon on the Windows desktop. Then, double-click the Server icon that appears in the System Tray of the Windows taskbar.
- From the Windows XP taskbar, choose **Start > All Programs > Xcalibur > Tune**. Then, double-click the Server icon that appears in the System Tray of the Windows taskbar.

Note When you create an instrument method to control your LC/MS instrument during a sequence run, you import the tune file that contains the empirically determined optimal needle voltage or corona current for your analyte. You then manually enter all of the other MS parameters and the chromatography conditions for your analyte. You create instrument methods in the Instrument Setup window in Xcalibur.

The Tune window shown in [Figure 30](#) includes the following:

- Title Bar
- Menu Bar
- Comms Indicator
- Scan Events Table
- Per Method Parameters Table
- Peak Display
- Status Bar

Figure 30. View of Tune window



Title Bar

The title bar is the horizontal band at the very top of the window. It contains the file name of the currently active tune file. For example: *default.tune* is the name of the Surveyor MSQ Plus default tuning file.

Menu Bar

The Tune window menu bar contains the following menus: File, View, and Help.

Toolbar

You can use the toolbar buttons to start and stop acquiring data, and to print a tune report.

Comms Indicator

The Tune Comms Indicator, positioned to the right of the Tune toolbar, indicates whether or not there is proper communication between the Tune software and the Surveyor MSQ Plus MS detector. The Comms Indicator is represented by an icon, depending on whether you are in Tune mode or Acquisition mode. The icon can be either stationary or spinning, depending on whether there is working communication occurring.



The icon to the left appears when you are in Tune mode (that is, when you are in the Tune window and not acquiring data to a file). The icon spins continuously to show that communications between the Surveyor MSQ Plus MS detector and the PC are established. For example, whenever the Tune software uploads or downloads a command, the icon spins to indicate that communication is working correctly.



The icon to the left appears when you are in Acquisition mode (that is, when you are acquiring data to file from the Tune window). A spinning icon indicates that communication is working correctly.

Scan Events Table

Use the Tune Scan Events table (or Peak Display controls) to define all the scan events that you want to acquire when performing a manual tune.

Per Method Parameters Table

Use the Tune Per Method Parameters table to enter values and define the settings for the 'per method' tuning parameters. The table also provides individual tuning parameter readbacks. You can adjust the tuning of the Surveyor MSQ Plus MS detector by altering the values of these tuning parameters. In ESI, you optimize the probe temperature and the needle voltage for your application. In APCI, you optimize the probe temperature and the corona current for your application.

Peak Display

The Tune Peak Display displays a real-time view of each tuning peak as defined by an enabled scan event (row) in the Tune Scan Events table. The Peak Display is used to monitor the tuning peaks for both peak form and intensity, particularly when optimizing the Surveyor MSQ Plus MS detector during a manual tune. You can change the number of peaks displayed in the Peak Display, and their appearance, using the parameters available in the Scan Events table.

Status Bar

The Status Bar at the bottom of the Tune window displays information on the current status of the Surveyor MSQ Plus MS detector.

Printer

Thermo Fisher Scientific does not ship a printer with the Surveyor MSQ Plus MS detector. If you choose to connect a printer that communicates through a USB cable, connect the cable to one of the USB ports on the front of the data system computer.

You set up the printer from the Print Setup dialog box. To open the Print Setup dialog box, choose **File > Print Setup** in any window.

Daily Operation

To optimize the performance of your Surveyor MSQ Plus MS detector, you need to perform various routine operations both before and after you operate the system.

Contents

- [Things to Do Before Operating the MS Detector](#)
- [Things to Do After Operating the MS Detector](#)

Things to Do Before Operating the MS Detector

This section contains the following procedures that you might want to perform on a daily basis before you begin your analyses:

- [Checking the Nitrogen Supply](#)
- [Checking the Disk Space](#)
- [Checking the Oil Level in the Oil Mist Filter](#)

Checking the Nitrogen Supply

Check the nitrogen supply on the regulator of the nitrogen gas tank or liquid nitrogen boil-off tank. Make sure that you have sufficient gas for your analysis. Typical nitrogen consumption is 15 800 liters per day (based on a 24 hr day). If necessary, replace the tank. Verify that the pressure of nitrogen reaching the mass spectrometer is between 520 ± 35 kPa (75 ± 5 psi). If necessary, adjust the pressure with the tank pressure regulator.

Checking the Disk Space

Periodically, verify that your hard disk drive has enough free space for data acquisition. The amount of available disk space is shown in the Disk Space dialog box.

❖ To determine the amount of available disk space

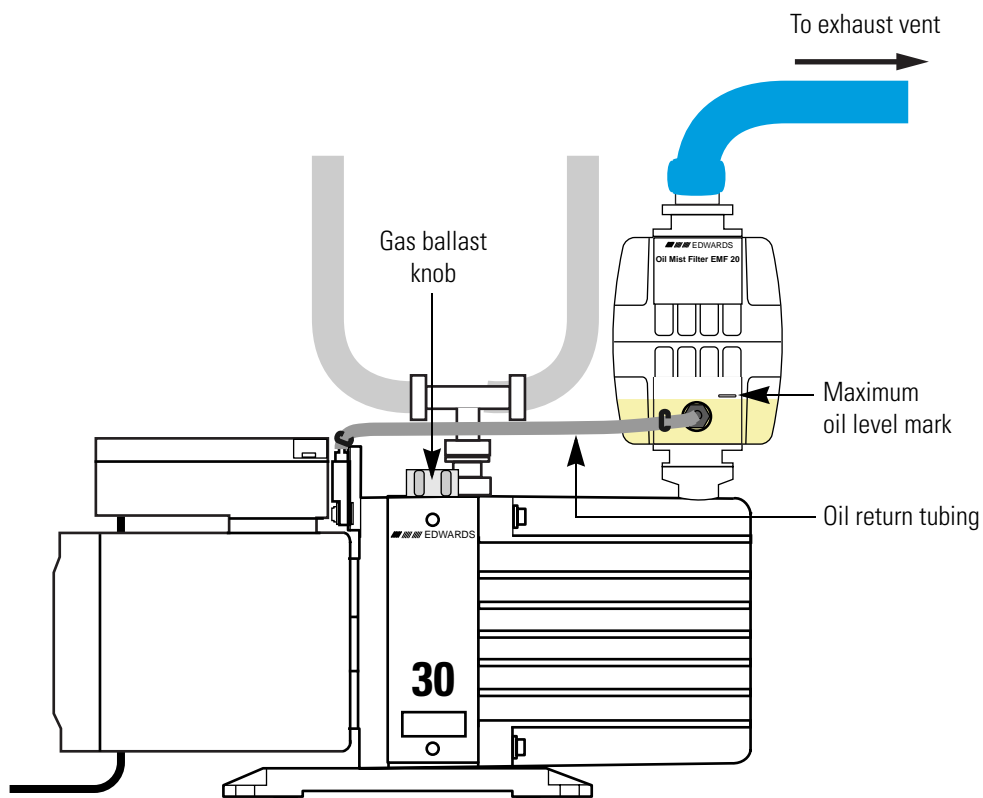
1. From the Home Page window (which is available by choosing **Start > All Programs > Xcalibur > Xcalibur** from the Windows Taskbar), choose **Actions > Check Disk Space** to open the Disk Space dialog box. The Disk Space dialog box lists the following:
 - Current drive and directory (for example, *C:\Xcalibur\system\programs*)
 - Number of Mbytes that are available (free) on the current drive
 - Percentage of the current drive that is available
 - Total capacity of the current drive
2. To select another disk drive so that you can determine its disk space, click the Directory button.
3. When you have completed this procedure, click **OK** to close the dialog box.

If necessary, free space on the hard disk by deleting obsolete files and by moving files from the hard disk drive to a backup medium. First, copy files to the backup medium. Then, delete them from the hard disk.

Checking the Oil Level in the Oil Mist Filter

Once the oil level in the oil mist filter that is attached to the Edwards forepump rises above the maximum oil level mark (see [Figure 31](#)), the oil mist filter becomes ineffective in trapping exhaust fumes. Therefore, it is important that you check the oil level in the oil mist filter on a daily basis. Refer to “[Draining the Oil Mist Filter and Purging the Pump Oil](#)” on [page 48](#) for instructions on draining the oil mist filter.

Figure 31. View of Edwards forepump with oil mist filter, showing the maximum oil level mark



Things to Do After Operating the MS Detector

This section contains the following procedures that you might want to perform after you complete your analyses:

- [Flushing the API Probes](#)
- [Placing the System in the Off Mode](#)
- [Draining the Oil Mist Filter and Purging the Pump Oil](#)
- [Emptying the Solvent Waste Bottles](#)

3 Daily Operation

Things to Do After Operating the MS Detector

Flushing the API Probes

After running phosphate salts, ion pairing agents, acids, or other additives through the system, flush the probe with [50:50] acetonitrile / water or methanol / water to prevent blockage.

Note To prevent blockage, always flush the probes after using buffered solvents.

❖ To flush the capillary of the API probe

1. Attach the analytical pump outlet directly to the Surveyor MSQ Plus MS detector. Because they might contain contaminants, bypass the injection valve of the autosampler and the LC column.
2. Pump a non-buffered solvent that is miscible with the buffered mobile phase through the probe at a flow rate of 2 mL/min for a few minutes.
3. Then, pump [50:50] acetonitrile / water or [50:50] methanol / water through the probe at a flow rate of 2 mL/min for 30 minutes.

Placing the System in the Off Mode

Place the Surveyor MSQ Plus MS detector in the Off mode if you are not going to use it for a short period of time, such as overnight or over weekends. In the Off mode, the system is left under vacuum, but the nitrogen flow is reduced to a bleed through the API probe, the electron multiplier and conversion dynode are turned off, the power to the ion optics is turned off, and the power to the probe heater is turned off.

Refer to “[Placing the System in the Off Mode](#)” on [page 97](#) for instructions on turning the system to the Off mode.

Draining the Oil Mist Filter and Purging the Pump Oil

During normal operation the oil in the forepump becomes contaminated with dissolved chemicals and water vapor. In addition, the oil mist filter fills with condensed oil. Over time, the rising water content of the oil can cause corrosion and decrease the lifetime of the forepump. And once the oil level in the oil mist filter rises above the maximum oil level mark (see [Figure 31](#) on [page 47](#)), the oil mist filter becomes ineffective in trapping exhaust fumes. Therefore, it is important that you drain the oil back into the forepump and purge the oil on a routine basis.



CAUTION Do not operate the forepump with the oil level in the oil mist filter above the maximum level mark.

Operating the Edwards forepump with the gas ballast valve open allows the oil in the oil mist filter to drain back into the forepump by way of the oil return tubing. Operating the forepump with the gas ballast valve open also allows the removal of water and other volatile contaminants from the forepump oil.

A good time to drain the oil from the oil mist filter and to remove volatile contaminants from the oil in the forepump is at the end of the working day or after the LC/MS system completes a sequence run.

❖ **To drain the oil mist filter and purge volatile contaminants from the oil in the forepump**

1. Turn off the LC pump flow.
2. Turn the nitrogen gas, ion optics, and probe heater off by doing one of the following:
 - From the Info view - Status page in Xcalibur, right-click the Surveyor MSQ listing and choose **Turn Device Off** from the shortcut menu.
 - Open the Per Method Parameters table in the Tune window. Take the system out of the Operate mode by clicking the Operate On/Off toggle button, and then turn off the nitrogen gas by clicking the Nitrogen Gas On/Off toggle button.
3. Open the gas ballast valve on the Edwards forepump by turning it six rotations counter-clockwise.
4. Operate the Edwards forepump for approximately 15 minutes with the gas ballast valve open.

The oil in the oil mist filter returns to the forepump relatively quickly. The prescribed time period of 15 minutes for ballasting is for the removal of volatile contaminants, such as water.

5. After ballasting the forepump for a period of approximately 15 minutes, close the gas ballast valve by turning the gas ballast knob clockwise until you feel resistance.

Note Operating the forepump with the gas-ballast valve open increases the rate of oil loss from the pump. During normal operations, run the forepump with the gas ballast valve closed.

Emptying the Solvent Waste Bottles

Waste solvents are produced by both the Surveyor MSQ Plus MS detector and the LC system. In the Surveyor MSQ Plus MS detector, waste solvent flows from the drain port at the bottom of the source enclosure, out the back of the detector through the Exhaust manifold, and into a solvent trap. Autosamplers such as the Surveyor Autosampler Plus perform a flush operation after each injection. The flush solution drains to a solvent waste bottle. See [Figure 13](#). Dispose of the solvent waste in accordance with local and federal regulations.

Switching Probes

This chapter describes how to connect an API (atmospheric pressure ionization) probe to the Surveyor MSQ Plus MS detector.

Contents

- [Switching From ESI to APCI](#)
- [Switching From APCI to ESI](#)

Switching From ESI to APCI

❖ To switch from the ESI mode to the APCI mode



CAUTION Allow the probe heater to cool before you remove the ESI probe.

1. Turn off the LC pump flow. If you are using the cone wash pump, turn it off.
2. Turn the nitrogen gas, ion optics, and probe heater off by doing one of the following:
 - From the Info view – Status page in Xcalibur, right-click the Surveyor MSQ listing to display a shortcut menu. Then choose **Turn Device Off** from the menu.
 - Open the Per Method Parameters table in the Tune window. Take the system out of the Operate mode by clicking the Operate On/Off toggle button, and then turn off the nitrogen gas by clicking the Nitrogen Gas On/Off toggle button.
3. Allow the probe heater to cool.
4. Unscrew and remove the PEEK fingertight fitting from the ESI probe.
5. Turn the locking plate on the ESI probe clockwise to the open position. Then, pull the ESI probe out of the probe heater.
6. Remove the APCI probe from the holder located in the door of the Surveyor MSQ Plus MS detector and replace it with the ESI probe.



CAUTION Take care not to damage the capillary of the probe as you insert the APCI probe into the probe heater.

7. Turn the locking plate on the APCI probe clockwise to the open position. Insert the APCI probe into the probe heater as shown in [Figure 32](#). Then, turn the locking plate counterclockwise to the closed position.

[Figure 32](#) shows the Surveyor MSQ Plus MS detector setup for the APCI mode. [Figure 33](#) shows the corona pin in the operational position for the APCI mode.

8. Turn the corona pin knob 90 degrees to its horizontal position.
9. Insert the PEEK fingertight fitting into the APCI probe and screw in.
10. Adjust the nitrogen gas pressure to 310 kPa (45 psi).

Figure 32. Surveyor MSQ Plus MS detector setup for the APCI mode

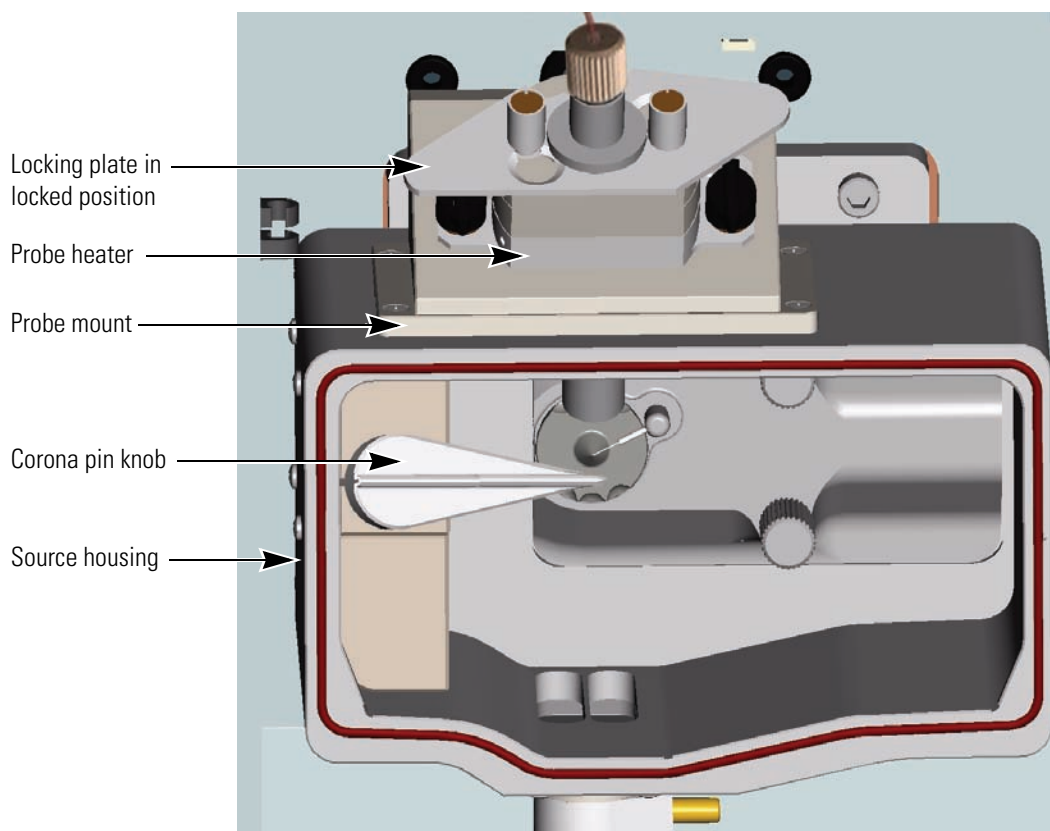


Figure 33. View of the corona pin in the operational position for the APCI mode



Switching From APCI to ESI

❖ To switch from the APCI mode to the ESI mode



CAUTION Allow the probe heater to cool before you remove the APCI probe.

1. Turn off the LC pump flow. If you are using the cone wash pump, turn it off.
2. Turn the nitrogen gas, ion optics, and probe heater off by doing one of the following:
 - From the Info view - Status page in Xcalibur, right-click the Surveyor MSQ listing to display a shortcut menu. Then, choose **Turn Device Off** from the menu.
 - Open the Per Method Parameters table in the Tune window. Take the system out of the Operate mode by clicking the Operate On/Off toggle button, and then turn off the nitrogen gas by clicking the Nitrogen Gas On/Off toggle button.
3. Allow the probe heater to cool.
4. Unscrew and remove the PEEK fingertight fitting from the APCI probe.
5. Turn the corona pin knob 90 degrees to its vertical position.
6. Turn the locking plate of the APCI probe clockwise to the open position and remove the APCI probe from the MS detector.
7. Remove the ESI probe from the holder located in the door of the Surveyor MSQ Plus MS detector and replace it with the APCI probe.



CAUTION Take care not to damage the capillary of the probe as you insert the ESI probe into the probe heater.

4 Switching Probes

Switching From APCI to ESI

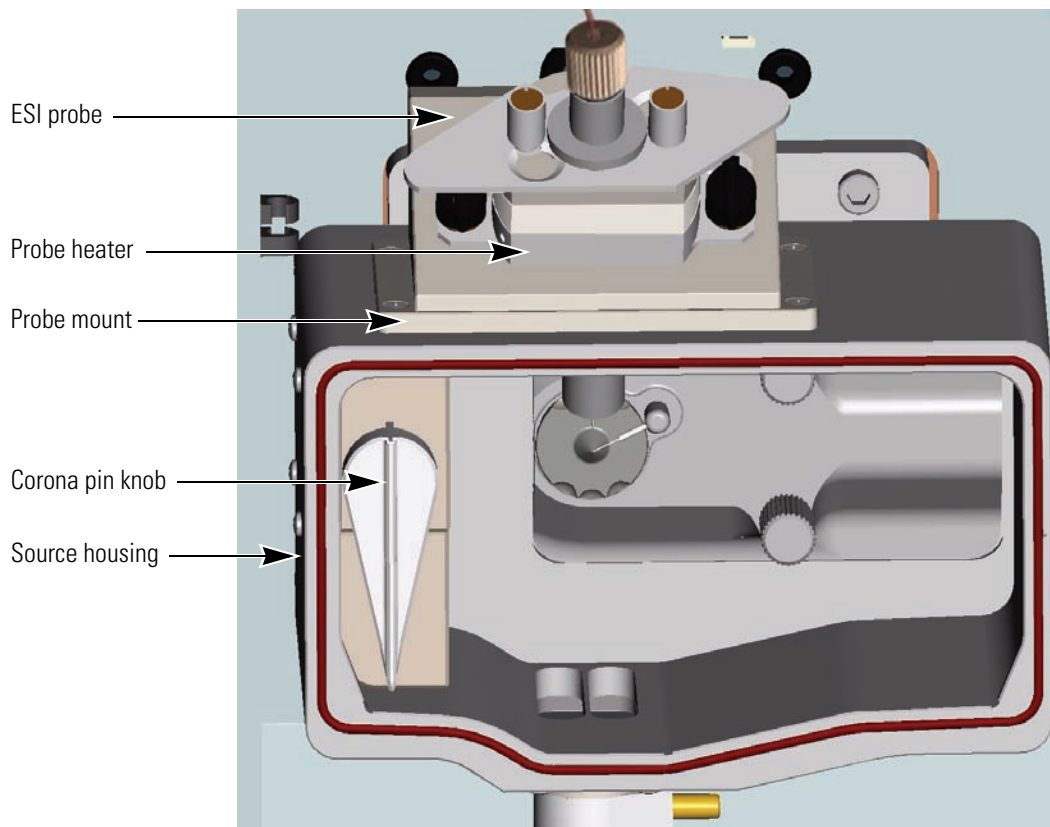
8. Turn the locking plate on the ESI probe clockwise to the open position. Insert the ESI probe into the probe heater as shown in [Figure 34](#). Then, turn the locking plate counterclockwise to the closed position.

9. Insert the PEEK fingertight fitting into the ESI probe and screw in.

See [Figure 34](#), which shows the Surveyor MSQ Plus MS detector setup for the ESI mode.

10. Adjust the nitrogen gas pressure to 520 kPa (75 psi).

Figure 34. Surveyor MSQ Plus MS detector setup for the ESI mode



Routine and Preventive Maintenance

The Surveyor MSQ Plus MS detector is a low maintenance instrument. Apart from fairly light periodic preventive maintenance, it requires only simple source cleaning and inspection on a “loss of performance” basis.

This chapter contains a maintenance schedule and instructions for the maintenance tasks that you must perform to keep your Surveyor MSQ Plus MS detector in optimal working condition.

Contents

- [Maintenance Schedule](#)
- [Maintaining the ESI Probe](#)
- [Maintaining the APCI Probe](#)
- [Maintaining the Probe Heater](#)
- [Maintaining the Source Block Assembly](#)
- [Maintaining the Forepump](#)

Maintenance Schedule

[Table 4](#) contains a list of routine maintenance procedures that you must perform at the intervals specified.

The maintenance schedule provides only a rough guide to the maintenance tasks you are responsible for. The appropriate frequency for these tasks depends on instrument usage and the level of system-induced contamination from samples and mobile phase solvents.

Table 4. Maintenance Schedule

Frequency	Action
As needed	Clean the source if a drop in sensitivity is seen during analyses.
	Flush the capillaries after running an analysis that required buffered solvents. Refer to “Flushing the API Probes” on page 48.
	Replace the capillary if it becomes clogged. Refer to “Maintaining the ESI Probe” on page 56 or “Maintaining the APCI Probe” on page 67.
	Clean the probe if it becomes contaminated. Refer to “Maintaining the ESI Probe” on page 56 or “Maintaining the APCI Probe” on page 67.
	Drain the oil from the oil mist filter and purge the oil in the forepump as described in “Draining the Oil Mist Filter and Purging the Pump Oil” on page 48.
Monthly	Clean the probe heater. Refer to “Maintaining the Probe Heater” on page 73.
	Check the oil level and color in the rotary pump and add oil if necessary. Refer to “Maintaining the Forepump” on page 93. Refer to the manual that ships with the rotary pump for instructions on changing the oil.
3 to 6 months	Replace the rotary pump oil after 3000 hours of operation. Refer to the manual that ships with the pump.
> 6 months	Clean the RF/dc prefilter. Refer to “Cleaning the RF/dc Prefilter” on page 86.

Maintaining the ESI Probe

Flushing the probe on a regular basis helps to prevent contamination and blockage of its capillary. But even with the best preventive maintenance, occasionally, the capillary can become blocked and the internal components can become contaminated.

A significant increase in LC pump backpressure (that is, up to 300 psi at a flow rate of 1 mL/min added to the total LC system backpressure) can be symptomatic of a blocked capillary. Instability in the MS signal can be symptomatic of a partially blocked capillary. Replace the capillary if it becomes blocked or partially blocked.



CAUTION Wait for the source block and the probe heater assembly to cool before you remove the ESI probe.

Follow these procedures to perform maintenance on the ESI probe:

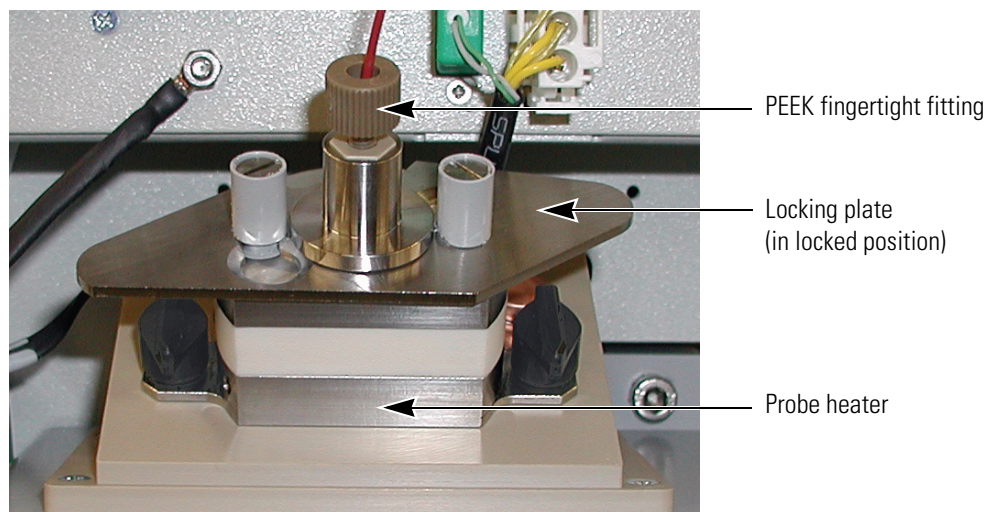
- Removing the ESI Probe
- Removing the ESI Capillary
- Cleaning or Replacing the ESI Capillary
- Replacing the Ceramic Sleeve of the ESI Probe
- Installing the ESI Capillary
- Installing the ESI Probe

Removing the ESI Probe

❖ To remove the ESI probe from probe heater

1. Ensure that the solvent flow from LC pump is turned off. If you are using the optional cone wash pump, ensure that it is turned off.
2. Turn off the nitrogen gas, the probe heater, and the ion optics by doing one of the following:
 - From the Info view - Status page in the Xcalibur data system, right-click the Surveyor MSQ listing and choose **Turn Device Off** from the shortcut menu.
 - Open the Per Method Parameters table in the Tune window. Take the system out of the Operate mode by clicking the Operate toggle button, and then turn off the nitrogen gas by clicking the Nitrogen Gas toggle button.
3. Wait for the source block and the probe heater of the MS detector to cool.
4. Unscrew and remove the PEEK fingertight fitting from the ESI probe. See [Figure 35](#).

Figure 35. ESI probe installed in the probe heater

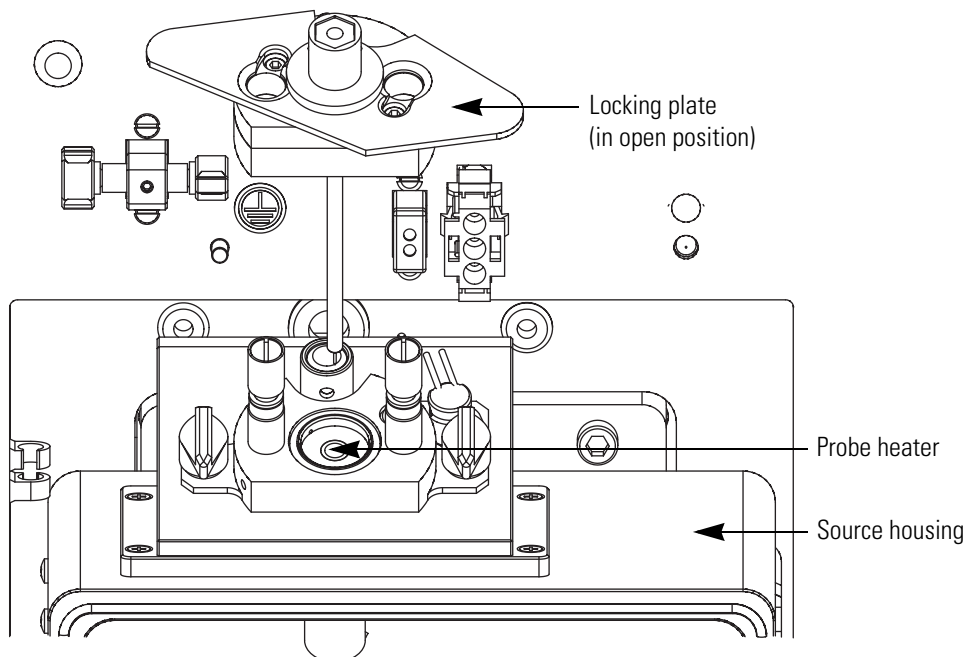




CAUTION Because its capillary tip and ceramic sleeve are fragile, exercise care when you remove the ESI probe from the probe heater.

5. Turn the locking plate clockwise to the open position, and then carefully remove the ESI probe from the probe heater. See [Figure 36](#).

Figure 36. Removing the ESI probe from the probe heater



Removing the ESI Capillary

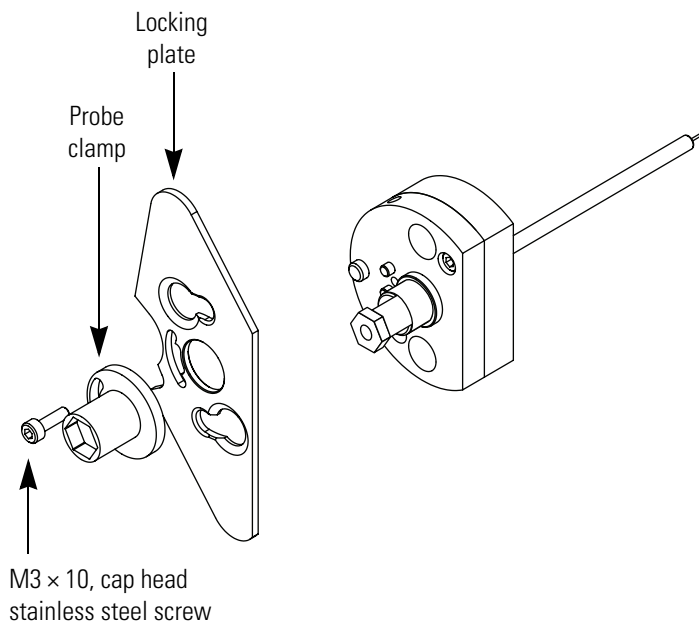
To clean or replace the ESI capillary or to replace the ceramic sleeve, you must remove the ESI capillary from the probe. See [Figure 83](#) on [page 114](#) for the part numbers of the ESI probe components.

❖ To remove the capillary from the ESI probe

1. Remove the ESI probe from the MS detector as described in [“Removing the ESI Probe”](#) on [page 57](#).

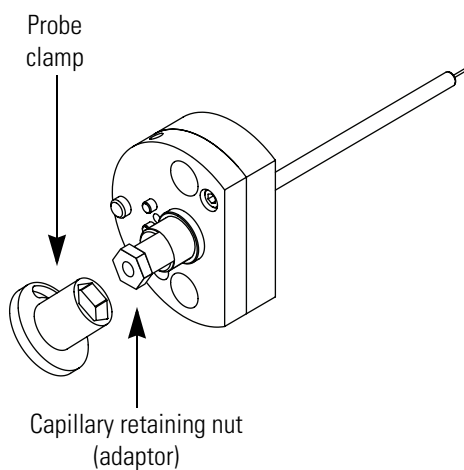
- Using the 2.5 mm Allen key, unscrew the M3 × 10 cap head stainless steel screw from the probe clamp, and then remove the probe clamp and the locking plate. See [Figure 37](#).

Figure 37. Removing the probe clamp and locking plate



- Using the front end of the probe clamp, unscrew the capillary retaining nut (adaptor) and remove it from the ESI probe. See [Figure 38](#).

Figure 38. Removing the capillary retaining nut (adaptor)

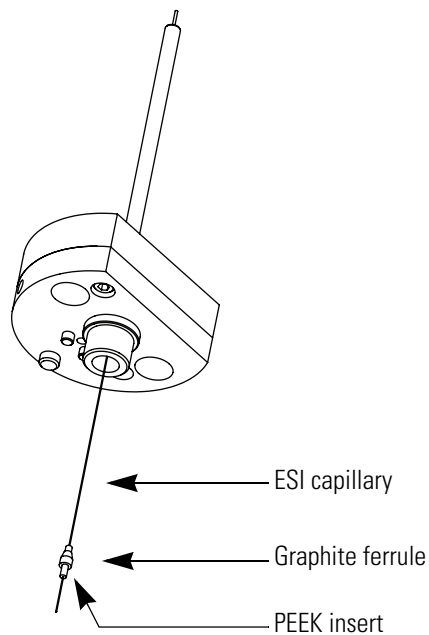


- Place a lint-free cloth on the workbench, and then gently shake the ESI capillary (P/N FM102598), graphite ferrule (P/N 6070119), and PEEK tube insert (P/N FM102591) assembly out of the ESI probe onto the cloth. See [Figure 39](#).

5 Routine and Preventive Maintenance

Maintaining the ESI Probe

Figure 39. Removing the capillary, graphite ferrule, and PEEK insert assembly from the probe



CAUTION Because the ESI capillary is fragile and can be damaged easily, exercise care when handling it.

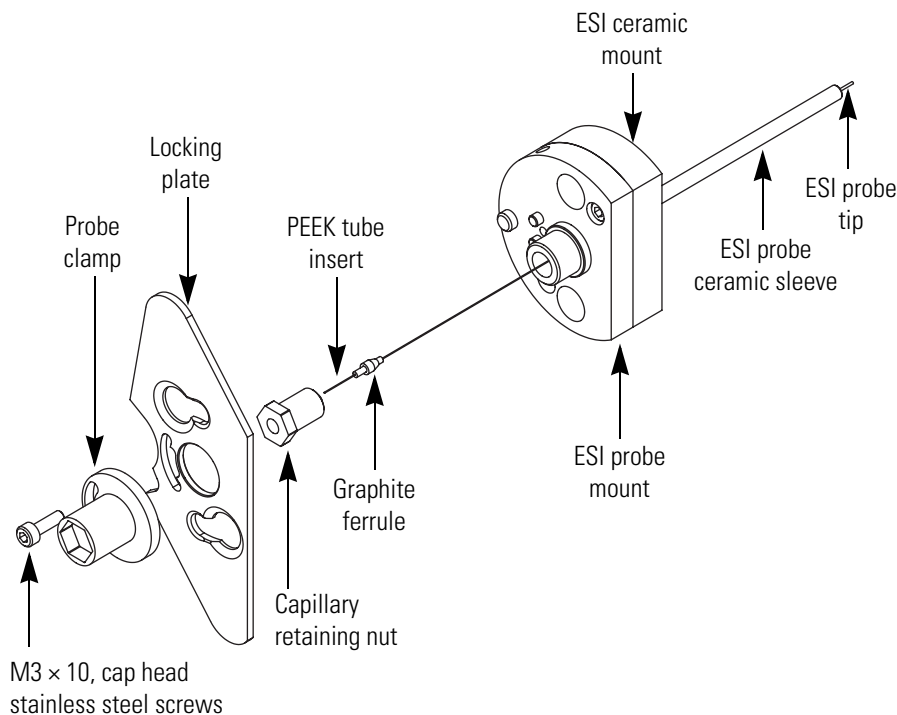
5. Pull the ESI capillary out of the PEEK tube insert.

Cleaning or Replacing the ESI Capillary

❖ To clean or replace an ESI capillary

1. Remove the ESI probe from the probe heater as described in “[Removing the ESI Probe](#)” on [page 57](#).
2. Remove the ESI capillary from the probe as described in “[Removing the ESI Capillary](#)” on [page 58](#).
3. If the capillary is reusable, clean its surface with [50:50] methanol / water.
4. Reinstall the clean capillary or a new capillary as described in “[Installing the ESI Capillary](#)” on [page 63](#).

Figure 40. View of partially disassembled ESI probe with capillary removed



Replacing the Ceramic Sleeve of the ESI Probe

If you break the ceramic sleeve of the ESI probe, replace it. See [Figure 83](#) on [page 114](#) for the part numbers of the ESI probe components.

❖ To replace the ceramic sleeve of the ESI probe

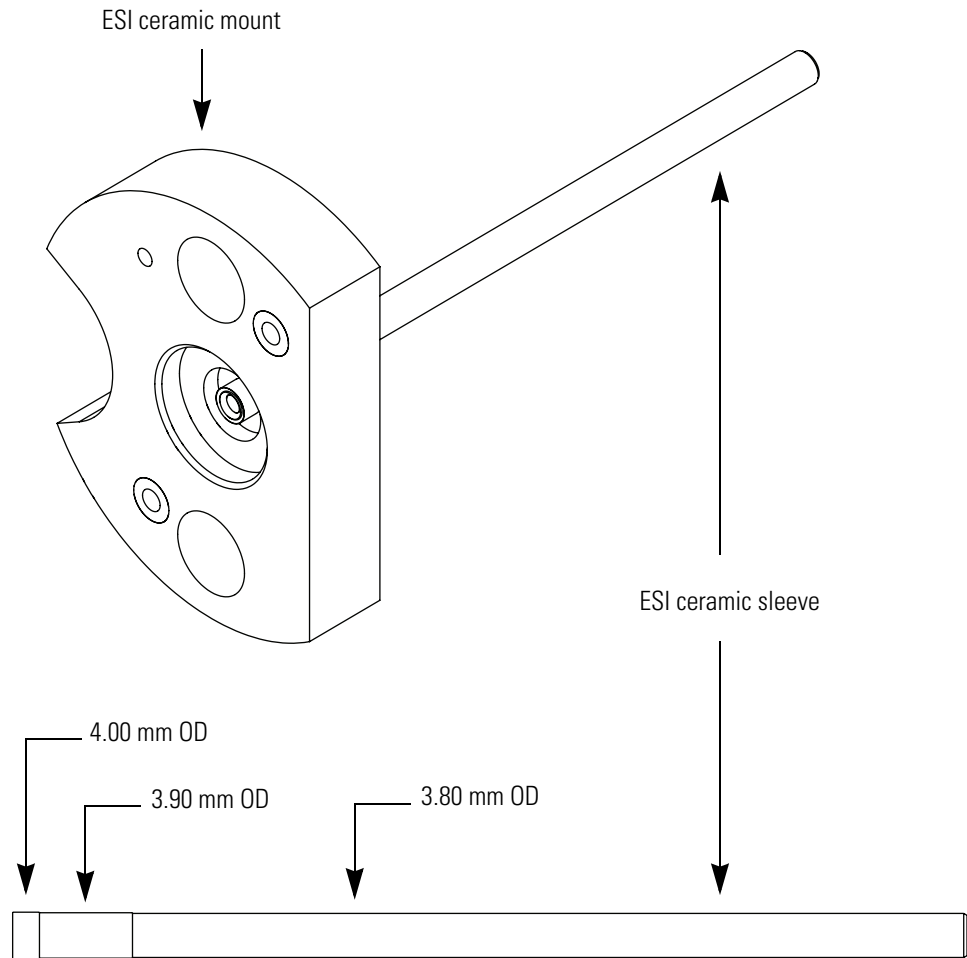
1. Remove the ESI probe from the MS detector as described in [“Removing the ESI Probe”](#) on [page 57](#).
2. Disassemble the ESI probe and remove the capillary as described in [“Removing the ESI Capillary”](#) on [page 58](#).
3. Use the 2.5 mm Allen key to unscrew the two M3 x 8 cap head stainless steel screws from the ESI probe mount. See [Figure 42](#).
4. Pull the ESI probe mount out of the ESI ceramic sleeve and ceramic mount assembly.
5. Remove the ESI ceramic sleeve from the ESI ceramic mount by pulling the sleeve forward through the top of the mount.

As [Figure 41](#) shows, the ESI ceramic sleeve is slightly flared at one end. This flare holds the ESI ceramic sleeve in place when you insert it into the ESI ceramic mount.

5 Routine and Preventive Maintenance

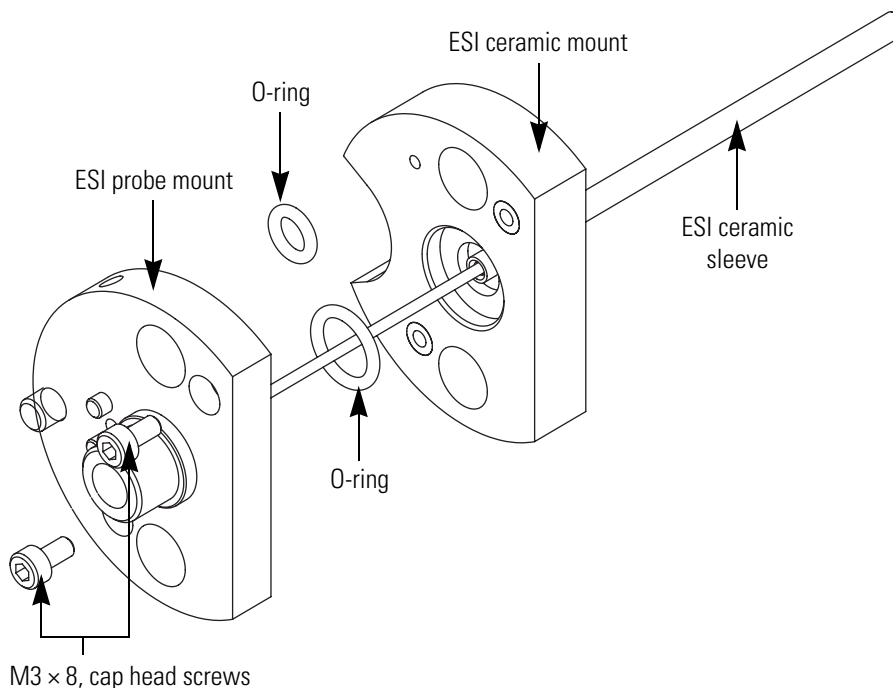
Maintaining the ESI Probe

Figure 41. ESI ceramic mount and ceramic sleeve



6. Insert a new ESI ceramic sleeve (P/N FM103394) into the ESI ceramic mount. See [Figure 42](#).
7. Place the O-rings in position in the ESI ceramic mount, and then insert the ESI probe mount into the ESI ceramic mount and ceramic sleeve assembly.
8. Insert the two M3 × 8 cap head stainless steel screws into the ESI probe mount and tighten with the 2.5 mm Allen key.
9. Reinstall the ESI capillary and reassemble the probe as described in [“Installing the ESI Capillary”](#) on [page 63](#).

Figure 42. Pulling the ESI probe mount out of the ESI ceramic mount



Installing the ESI Capillary

The ESI capillary is pre-sized for the probe, but its installation requires careful alignment.

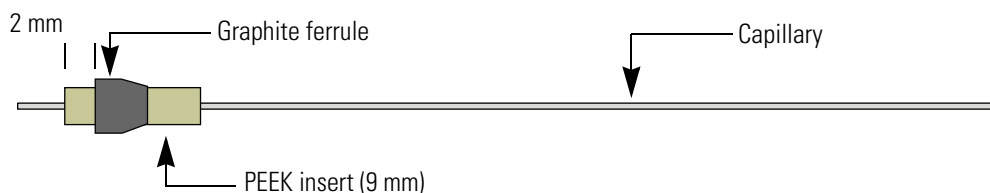
❖ To install the ESI capillary



CAUTION Take care when handling the ESI capillary. It bends easily.

1. Using tweezers, insert the ESI capillary into the PEEK tube insert and graphite ferrule. Adjust the graphite ferrule such that 2 mm of the PEEK tube insert is visible on the back end of the assembly. See [Figure 43](#).

Figure 43. Capillary, PEEK tube, and graphite ferrule assembly

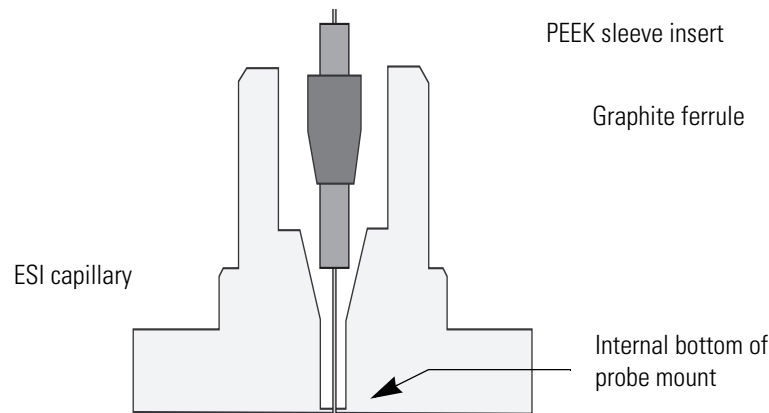


2. While holding the ESI probe in the vertical position, use tweezers to insert the capillary, PEEK tube insert, and graphite ferrule assembly into the ESI probe. See [Figure 44](#).

5 Routine and Preventive Maintenance

Maintaining the ESI Probe

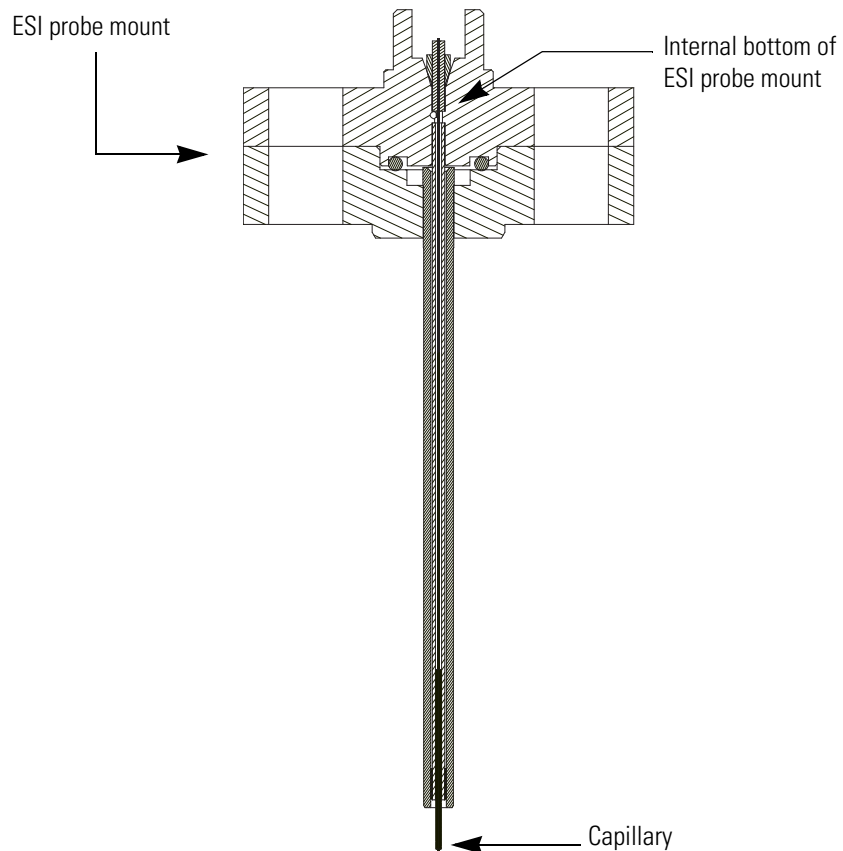
Figure 44. View of ESI probe mount, showing the insertion of the capillary



3. Adjust the position of the ESI capillary:

- Holding the ESI probe in the vertical position, carefully shake the PEEK tube insert into the probe until it meets resistance.
- Using tweezers, push the capillary into the probe until it is flush with the PEEK sleeve.
- Visually check that the capillary is protruding from the tip of the probe. As [Figure 45](#) shows, the capillary should protrude from the tip of the probe.

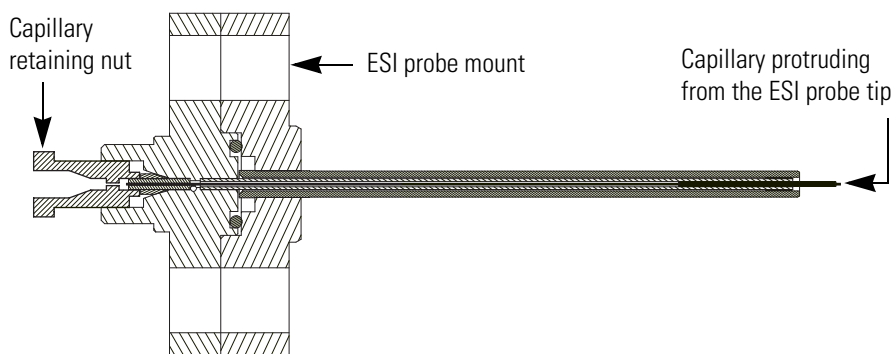
Figure 45. View of capillary inserted into the ESI probe mount



4. Depending on whether the capillary protrudes from the tip of the probe, do one of the following:
 - If the capillary protrudes from the probe tip, go to [step 5](#).
 - If the capillary does not protrude from the tip of the probe, turn the probe upside-down and gently shake the capillary assembly out of the probe. Adjust the position of the graphite ferrule so that it is closer to the end of the PEEK insert, and repeat [step 2](#) and [step 3](#) of this procedure. Continue to adjust the position of the graphite ferrule until the capillary protrudes from the tip of the probe when you insert the capillary assembly into the probe.
5. Screw the capillary retaining nut (adaptor) into the ESI probe until finger tight, and then visually check that the capillary protrudes from the probe tip.

[Figure 46](#) shows the capillary protruding from the probe tip.

Figure 46. View of the ESI probe with the capillary retaining nut screwed in finger tight

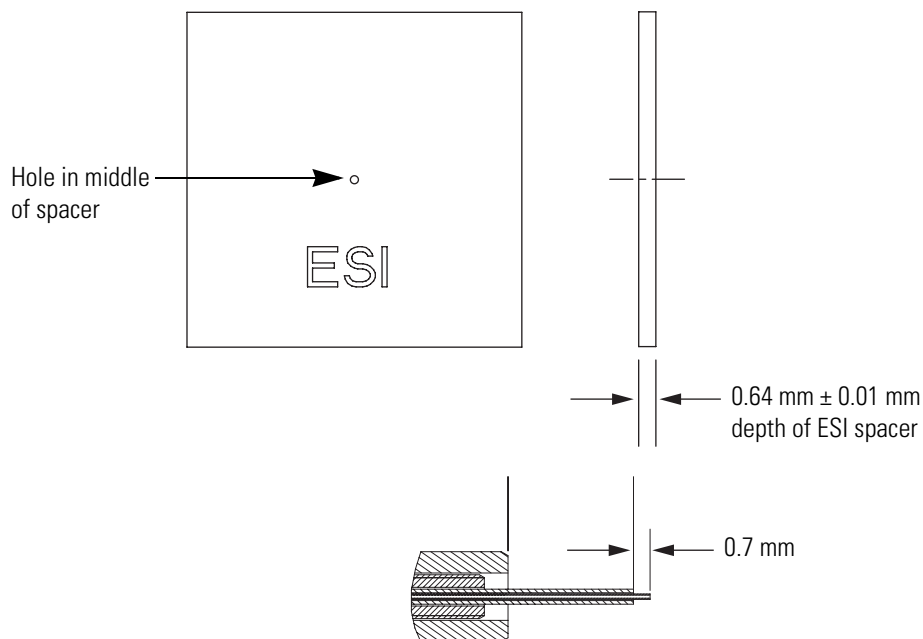


6. Using the probe clamp, tighten the capillary retaining nut by one-half turn. Do not overtighten the nut. Overtightening the nut can cause solvent leakage.
7. Finish reassembling the ESI probe:
 - a. Place the locking plate and probe clamp in position.
 - b. Insert the M3 × 10 cap head stainless steel screw (P/N FM103046) and tighten with the 2.5 mm Allen key.

The capillary must protrude from the probe tip. In addition to visually checking the protrusion depth of the capillary, you can use the ESI spacer plate. The ESI spacer plate is 0.64 mm thick. If you can see or feel the capillary protruding through the hole in the center of the plate, the capillary protrudes from the probe tip by more than 0.64 mm.
8. (Optional) Using the ESI spacer plate, confirm that the capillary protrudes from the tip of the probe.

[Figure 47](#) shows the ESI spacer being used to check the protrusion depth of the capillary.

Figure 47. View of the ESI probe tip with the capillary protruding by 0.7 mm



Installing the ESI Probe

❖ To install the ESI probe into the MS detector



CAUTION Take care when inserting the ESI probe into the probe heater. The capillary protruding from the end of the ceramic sleeve and the ceramic sleeve itself are easily damaged. In addition, you can contaminate the capillary if you let it touch the inner surface of the probe heater as you insert the probe into the probe heater.

1. If you have not already done so, turn the probe heater off.
2. Turn the locking plate of the probe clockwise to the open position, and then carefully slide the ESI probe into the probe heater.
3. Turn the locking plate counterclockwise to the closed position to lock the probe in place. See [Figure 35](#) on [page 57](#).
4. Connect the PEEK fingertight fitting to the ESI probe. See [Figure 35](#).

Maintaining the APCI Probe

Flushing the probe on a regular basis helps to prevent contamination and blockage of the capillary. But even with the best preventive maintenance, occasionally, the capillary can become blocked.

A significant increase in LC pump backpressure (that is, up to 300 psi at a flow rate of 1 mL/min added to the total LC system backpressure) or instability in the signal can be symptomatic of a blocked or partially blocked capillary. Replace the capillary if it has become blocked or partially blocked during operation. The APCI capillary (P/N FM102594) is pre-sized for the probe, but its installation requires careful alignment.



CAUTION Wait for the source block and probe heater assembly to cool before changing ionization modes.

Follow these maintenance procedures to replace a capillary or clean the APCI probe:

1. [Removing the APCI Probe](#)
2. [Removing the APCI Capillary](#)
3. [Installing the APCI Capillary](#)
4. [Installing the APCI Probe](#)

Removing the APCI Probe

❖ To remove the APCI probe from the probe heater

1. Ensure that the flow from the LC pump is turned off.
2. Turn off the nitrogen gas, the probe heater, and the ion optics by doing one of the following:
 - In the Status page of the Xcalibur data system, right-click the Surveyor MSQ listing and choose **Turn Device Off** from the shortcut menu.
 - Or open the Per Method Parameters table in the Tune window. Take the system out of the Operate mode by clicking the Operate toggle button and turn off the nitrogen gas by clicking the Nitrogen Gas toggle button. See [Figure 30](#).
3. Wait for the source block and the probe heater to cool.
4. Unscrew and remove the PEEK fingertight fitting from the APCI probe. See [Figure 48](#).



CAUTION Because it is fragile and can be damaged easily, exercise care when handling the APCI capillary.

5 Routine and Preventive Maintenance

Maintaining the APCI Probe

5. Turn the locking plate clockwise to the open position and remove the APCI probe from the probe heater. See [Figure 49](#).

Figure 48. Surveyor MSQ Plus MS detector set up for the APCI mode

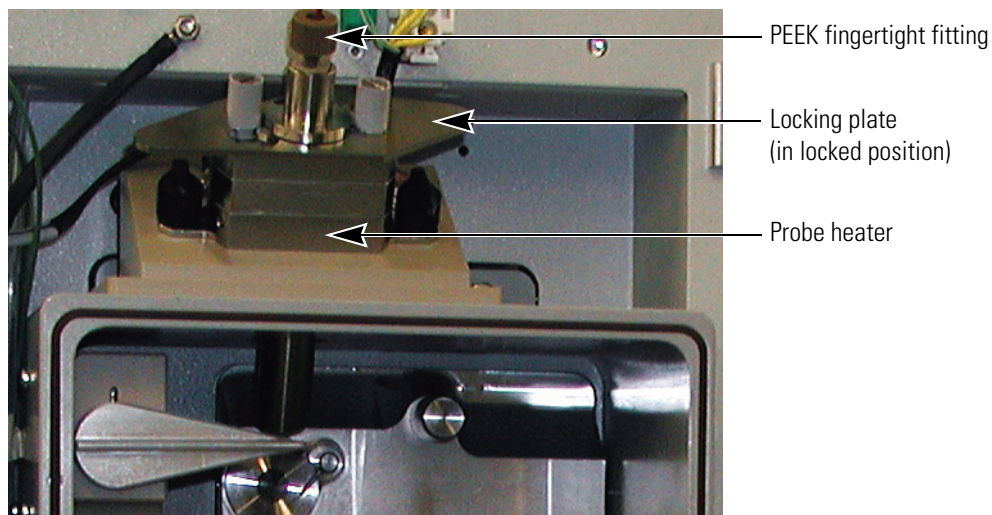
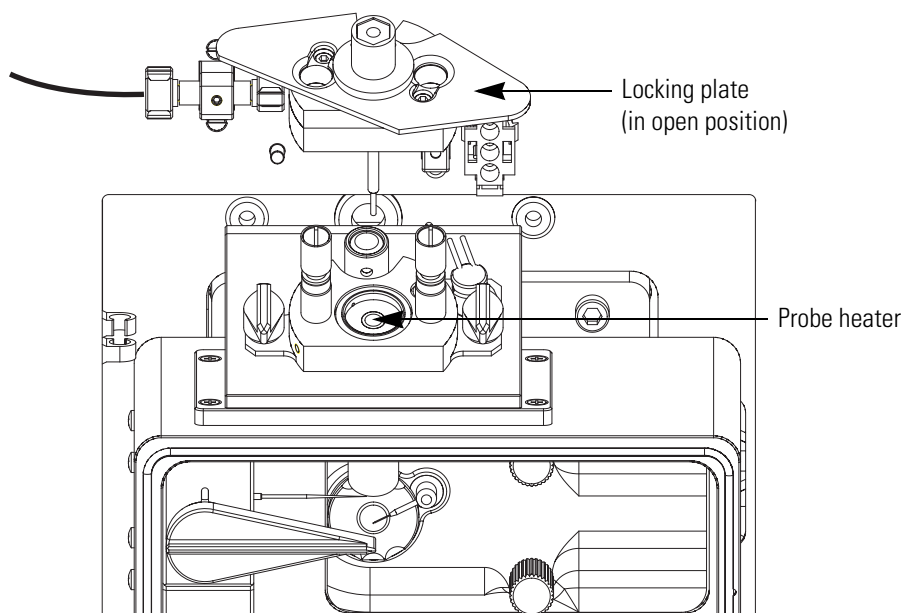


Figure 49. Remove the APCI probe from the probe heater



Removing the APCI Capillary

❖ To remove the capillary from the APCI probe

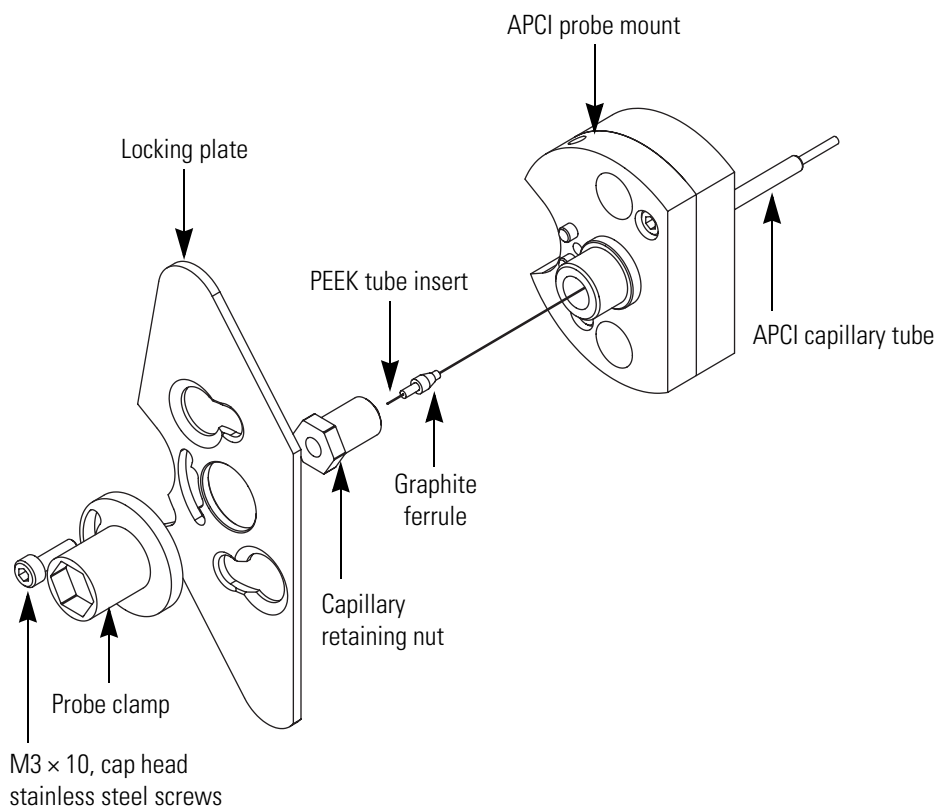
1. Using a 2.5 mm Allen key, unscrew the M3 × 10 cap head stainless steel screw (P/N FM103046) from the probe clamp. See [Figure 50](#).
2. Remove the probe clamp and the locking plate.

- Using the probe clamp, unscrew the capillary retaining nut.
- Carefully shake the graphite vespel ferrule (P/N 6070119), PEEK tube insert (P/N FM102591) and APCI probe capillary (P/N FM102594) out of the probe.



CAUTION Exercise care when handling the APCI capillary because it is fragile and can be damaged easily.

Figure 50. View of partially disassembled APCI probe with capillary removed



- Pull the APCI capillary out of the PEEK tube insert, and then do one of the following:
 - If the capillary is partially blocked or blocked, dispose of it.
 - If the capillary is reusable, clean its surface with [50:50] methanol / water.

Installing the APCI Capillary

❖ **To install the APCI capillary into the APCI probe**

1. Using tweezers, insert the APCI capillary into the PEEK tube insert and graphite Vespel ferrule assembly.
2. Using tweezers, insert the APCI capillary, PEEK tube insert, and graphite ferrule assembly into the probe. See [Figure 51](#) and [Figure 52](#).
3. Adjust the position of the APCI capillary:
 - a. Holding the APCI probe in the vertical position, carefully shake the PEEK tube insert into the probe until it meets resistance.
 - b. Using tweezers, push the capillary into the probe until it is flush with the PEEK sleeve.
 - c. Visually check that the capillary is protruding from the tip of the probe.

Figure 51. Inserting the capillary, PEEK sleeve, and graphite ferrule into the APCI probe

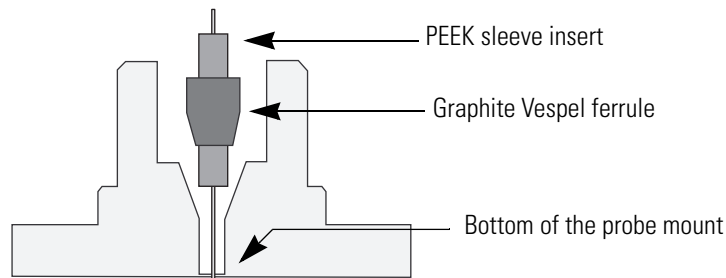
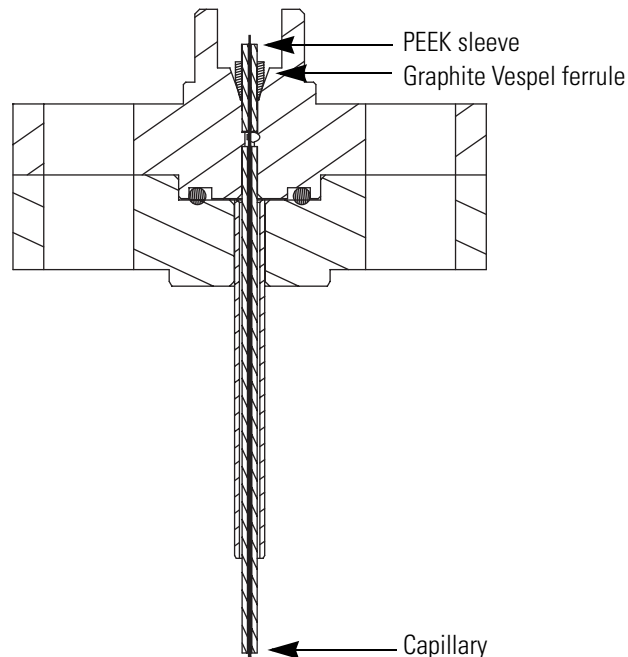
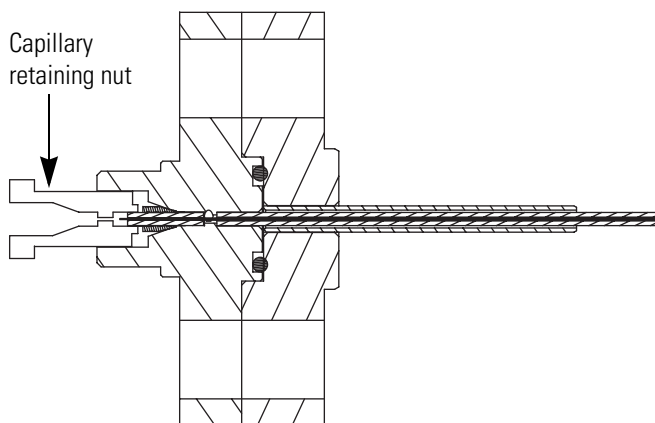


Figure 52. APCI capillary, PEEK sleeve, and graphite ferrule inserted into probe



4. Depending on whether the capillary protrudes from the tip of the probe, do one of the following:
 - If the capillary protrudes from the probe tip, go to [step 5](#).
 - If the capillary does not protrude from the tip of the probe, turn the probe upside-down and gently shake the capillary assembly out of the probe. Adjust the position of the graphite ferrule so that it is closer to the end of the PEEK insert, and repeat [step 2](#) and [step 3](#) of this procedure. Continue to adjust the position of the graphite ferrule until the capillary protrudes from the tip of the probe when you insert the capillary assembly into the probe.
5. Screw the capillary retaining nut (adaptor) into the probe until fingertight and recheck that the capillary protrudes from the tip of the probe. See [Figure 53](#).

Figure 53. View of APCI probe with the capillary retaining nut screwed in finger tight



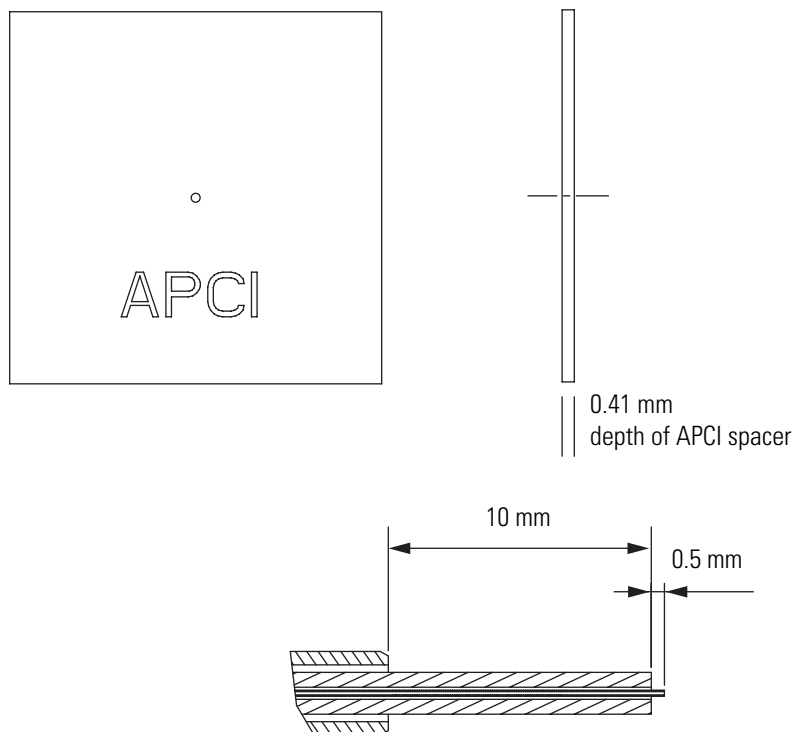
6. Using the probe clamp, tighten the capillary retaining nut by one-half turn. Do not overtighten the screw.
7. Finish reassembling the probe:
 - a. Place the locking plate and probe clamp in position.
 - b. Insert the M3 × 10 cap head stainless steel screw (P/N FM103046) and tighten with the 2.5 mm Allen key.

Typically, a visual confirmation that the capillary protrudes from the probe tip is sufficient, but you can also use the APCI spacer plate provided in the Surveyor MSQ Plus tool kit to check the capillary protrusion depth.

8. (Optional) Align the hole in the center of the APCI spacer plate over the tip of the probe and check that you can feel the capillary protruding through the spacer.

The APCI spacer is 0.41 mm thick. If you can feel the capillary protruding through the hole in the center of the spacer plate, the capillary protrudes from the APCI probe tip by more than 0.41 mm. See [Figure 54](#).

Figure 54. Enlarged view of the tip of the APCI probe



Installing the APCI Probe

❖ To install the APCI probe into the probe heater



CAUTION Take care when inserting the APCI probe into the probe heater. The capillary protruding from the end of the probe is fragile and easily damaged. In addition, you can contaminate the capillary if you let it touch the inner surface of the probe heater as you insert the probe into the probe heater.

1. Turn the locking plate of the probe clockwise to the open position, and then carefully slide the APCI probe into the probe heater. See [Figure 49](#) on [page 68](#).
2. Turn the locking plate counterclockwise to the closed position to lock the probe in place. See [Figure 48](#) on [page 68](#).
3. Connect the PEEK fingertight fitting to the APCI probe. See [Figure 48](#).

Maintaining the Probe Heater

If you use the instrument primarily in the APCI mode, clean the probe heater on a monthly basis. If you use the instrument primarily in the ESI mode, you have to clean the probe heater less frequently.

Occasionally, tension or sharp edges on the probe latching plate can cause breakage of the PEEK mounting pins. Use the parts supplied in the Probe Heater Repair Kit (P/N 60111-62010) to repair the probe.

To clean or repair the probe heater, follow these procedures:

1. [Removing the Probe Heater](#)
2. [Cleaning the Probe Heater or Replacing the Detent Screw Insulator](#)
3. [Installing the Probe Heater](#)

Removing the Probe Heater

❖ To remove the probe heater from the probe mount

1. Remove the ESI or APCI probe. Refer to “[Removing the ESI Probe](#)” on [page 57](#) or “[Removing the APCI Probe](#)” on [page 67](#).



CAUTION Wait for the probe heater to cool before you remove it.

2. Rotate the black knobs of the probe heater outwards 90 degrees, so that they face away from each other. See [Figure 55](#) and [Figure 56](#).

The black knobs are connected to locking cams. When the black knobs are facing away from each other, the cams are in the unlocked position and you can pull the probe heater out of the probe mount.

5 Routine and Preventive Maintenance

Maintaining the Probe Heater

Figure 55. View of the probe heater and its connections to the front panel of the MS detector

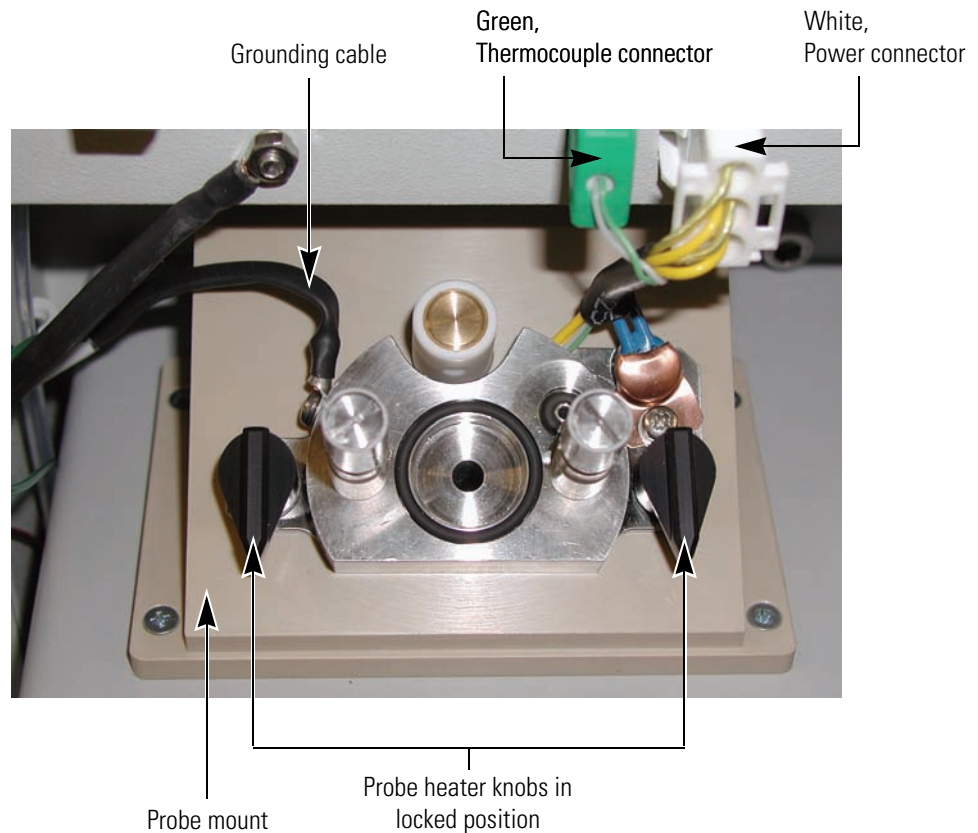
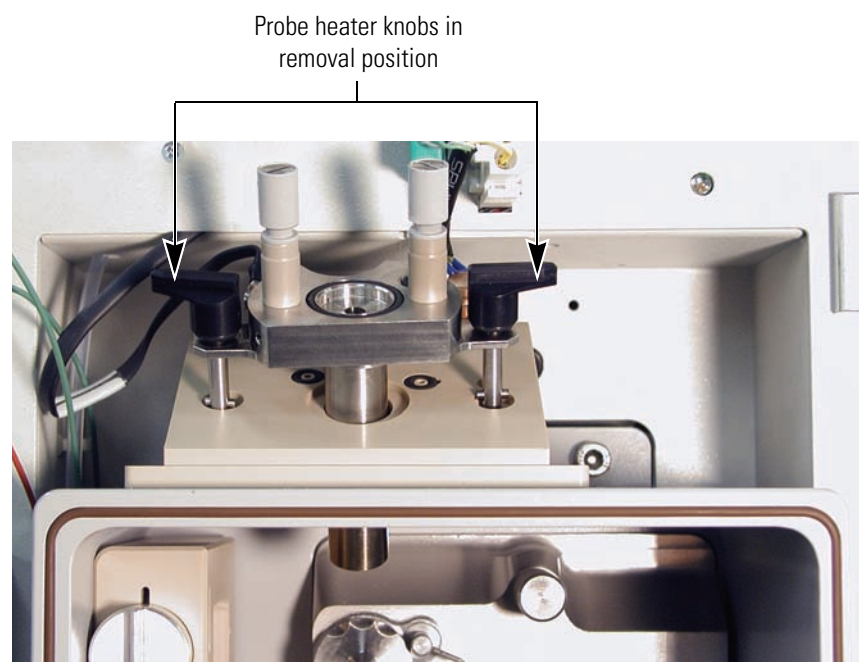


Figure 56. View of the probe heater knobs in the removal position



3. Pull the probe heater out of the probe mount, and then disconnect the green connector and the white connector from the MS detector. See [Figure 57](#).

The green connector connects the probe heater to the temperature sensor. The white connector connects the probe heater to its power source.

4. Store the probe heater in the holder to the left of the source compartment until you are ready to clean it. See [Figure 58](#).
5. To remove the probe heater from the MS detector, use a #1 Phillips head screwdriver to detach the grounding strap from the probe heater. See [Figure 58](#).

Figure 57. View of power connector and thermocouple connector

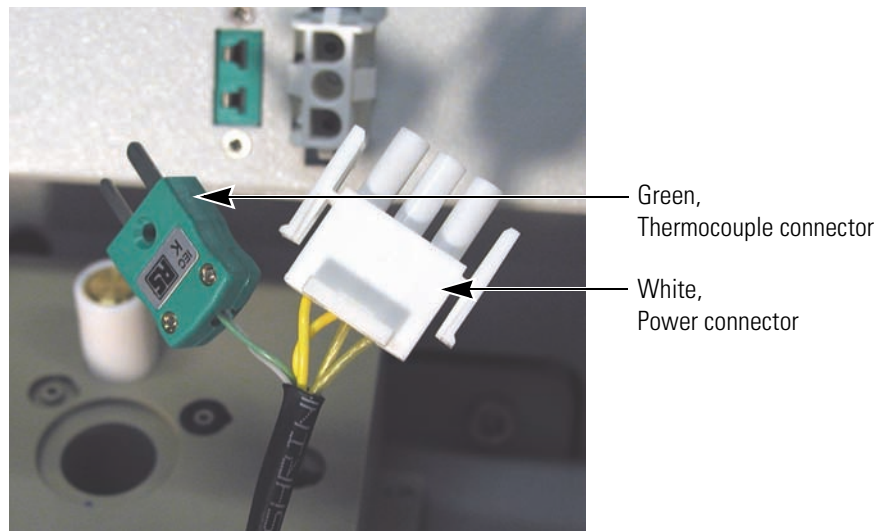
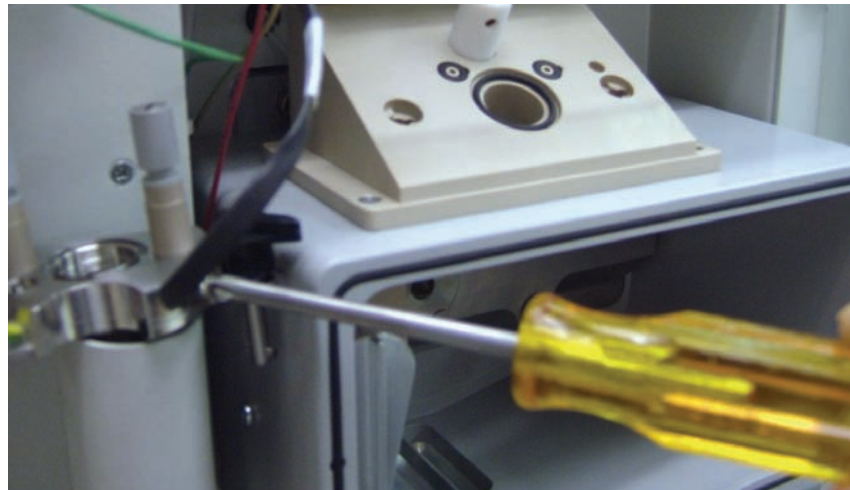


Figure 58. View of probe heater with the operator detaching the grounding strap



Cleaning the Probe Heater

❖ **To clean the probe heater**

Clean the inside of the heater tube with a cotton swab soaked in [50:50] methanol/water. See [Figure 61](#).

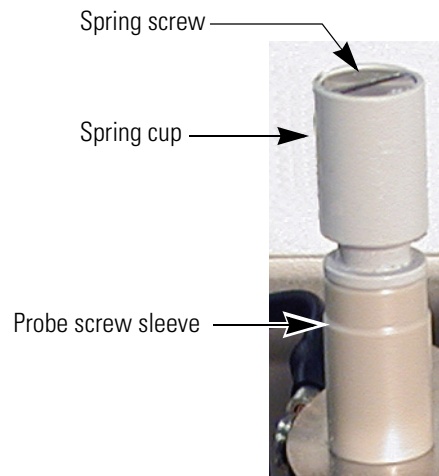
Replacing the Detent Screw Insulator

Use the parts supplied in the Probe Heater Repair Kit (P/N 60111-62010) to repair the probe. See [Figure 86](#) on [page 117](#).

❖ **To replace a broken detent screw insulator**

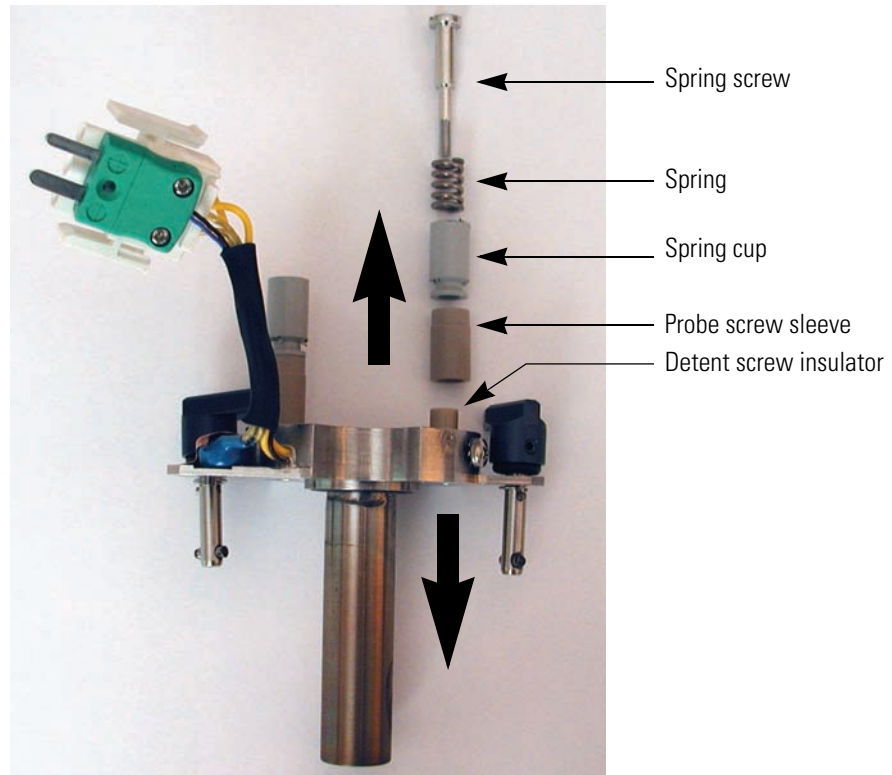
1. Disassemble the detent screw assembly:
 - a. Using a flat-blade screwdriver, loosen and remove the spring screw.

Figure 59. View of the slot in the top of the spring screw



- b. Remove the spring from the spring cup and set the spring aside.
Because the spring is reusable, it is not provided in the repair kit.
 - c. Pull the probe screw sleeve off the detent screw insulator.
 - d. Pull the detent screw insulator out the underside of the probe heater body. See [Figure 60](#).

Figure 60. Partially disassembled probe heater



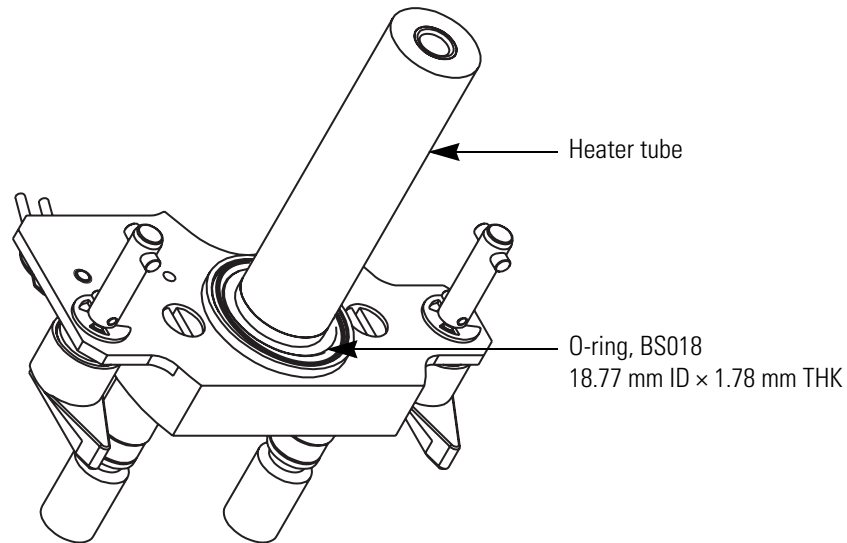
2. Using the parts supplied in the repair kit, rebuild the assembly:
 - a. Insert a new detent screw insulator into the underside of the probe heater body.
 - b. Slide the probe screw sleeve over the top of the detent screw insulator.
 - c. Insert the spring that you removed from the broken assembly into the spring cup.
 - d. Align the bottom of the spring cup with the probe screw sleeve.
 - e. Insert the spring screw into the spring cup and tighten the assembly with a flat-blade screwdriver.

Installing the Probe Heater

❖ **To install the probe heater**

1. If you removed the O-ring from the underside of the probe heater base, reinstall it. See [Figure 61](#).

Figure 61. View of O-ring installed in the underside of the probe heater base



2. If you disconnected the grounding strap from the probe heater, reconnect it to the probe heater. See [Figure 58](#) on [page 75](#).



CAUTION Make sure that the grounding strap is connected to the probe heater before you insert the probe heater into the probe mount.

3. Rotate the black knobs of the probe heater, so that they face away from each other. See [Figure 56](#) on [page 74](#).
4. Insert the tube portion of the probe heater into the probe mount.
5. Rotate the black knobs of the probe heater forward to their locking position. See [Figure 55](#) on [page 74](#).
6. Plug the green connector and the white connector into their respective receptacles located above the probe mount. See [Figure 55](#).

Maintaining the Source Block Assembly

The entrance cone is the only component of the source block assembly that requires frequent cleaning. The cone wash nozzle rarely requires cleaning because it is used to spray the entrance cone with cleaning solvent.

Note When it is not in use, ensure that the cone wash nozzle faces away from the entrance cone. See [Figure 62](#) on [page 82](#).

You do not need to disassemble the source block assembly to clean the entrance cone and the cone wash nozzle. To clean the RF/dc prefilter, the extraction cone, or the source block itself, you must remove the source block assembly from the MS detector and disassemble it.

To clean any or all of the components of the source block assembly, follow these procedures:

1. [Preparing the LC/MS System for Maintenance](#)
2. [Clearing Access to the Source Block Assembly](#)
3. [Removing the Entrance Cone and the Cone Wash Nozzle](#)
4. [Cleaning the Entrance Cone and the Cone Wash Nozzle](#)
5. [Removing the Source Block Assembly](#)
6. [Cleaning the RF/dc Prefilter](#)
7. [Cleaning the Extraction Cone and the Source Block](#)
8. [Assembling the Source Block Assembly](#)
9. [Installing the Source Block Assembly](#)



CAUTION Wait for the source block and probe heater assembly to cool before carrying out any maintenance.

Preparing the LC/MS System for Maintenance

❖ To prepare your LC/MS system for maintenance

1. Turn off the flow from the LC pump.
2. If the cone wash is in use, turn it off.
3. Turn off the nitrogen gas, the ion optics, and the probe heater by doing one of the following:
 - In the Per Method Parameters table of the Tune window, click the Nitrogen Gas On/Off toggle button to Off.
 - Or open the Information View - Status page of Xcalibur. Right-click the Surveyor MSQ listing to open a shortcut menu and choose **Turn Device Off** from the shortcut menu.
4. If the Surveyor MSQ Plus MS detector is under vacuum, vent it. Right-click the **Server** icon and choose **Vent** from the shortcut menu.

Venting the Surveyor MSQ Plus MS detector turns off the turbomolecular pump. It does not turn off the Edwards forepump.
5. Wait 2 or more minutes before proceeding so that the system has time to vent.



CAUTION It takes approximately 2 minutes for the vacuum rotor to decelerate after you choose Vent. To avoid damaging the vacuum pump, allow time for this deceleration process before you turn off the power to the vacuum pump.

6. Turn off the Edwards forepump by doing one of the following:
 - Turn off the power to the Surveyor MSQ Plus MS detector by placing its MAINS ON/OFF switch to the Off position.
 - Flip the power switch on the Edwards forepump to the Off position.



CAUTION Wait for the source block and probe heater to cool before performing any maintenance.

Clearing Access to the Source Block Assembly

❖ To clear access to the source block assembly

1. To open the front door of the MS detector, depress the door latch on the left-side of the detector as you pull the door forward.
2. Remove the API probe from the probe heater:
 - a. Remove the PEEK fingertight fitting from the probe.
 - b. Turn the locking plate clockwise to the open position, and then carefully pull the API probe out of the probe heater.



CAUTION Exercise care when handling the API probe. The ceramic sleeve of the ESI probe and the capillaries of both probes are fragile and can be damaged easily.

3. Rotate the black knobs of the probe heater, so that they face away from each other, and then pull the probe heater out of the source mount. See [Figure 56](#) on [page 74](#).
4. Store the probe heater in the holder to the left of the source compartment.
5. If your Surveyor MSQ Plus MS detector is set up in the APCI mode, rotate the corona pin knob downward to its vertical position.

Removing the Entrance Cone and the Cone Wash Nozzle

❖ To remove the entrance cone and the cone wash nozzle

1. If you have not already done so, vent the MS detector as described in “[Preparing the LC/MS System for Maintenance](#)” on [page 80](#) and clear the access to the source block as described in “[Clearing Access to the Source Block Assembly](#)” on [page 81](#).



CAUTION The corona needle is very sharp. Do **not** attempt to remove the entrance cone or the cone wash nozzle before you turn the corona pin knob to its vertical position. See [Figure 62](#).

2. Being careful to handle the cone wash nozzle by its base, rotate the cone wash nozzle away from the entrance cone. See [Figure 62](#).

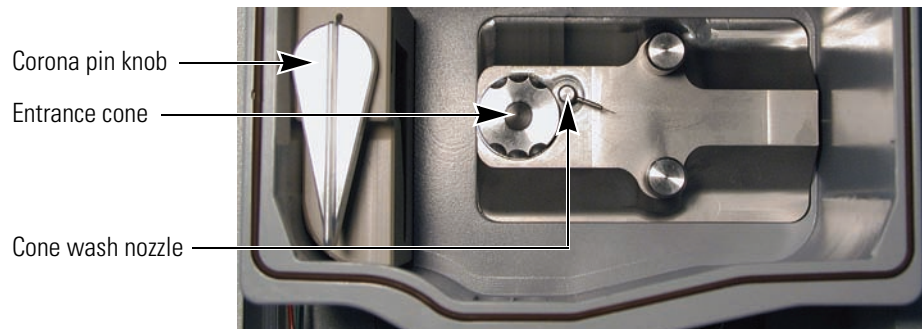


CAUTION The tip of the cone wash nozzle is very fragile, take care to handle the cone wash nozzle by its base. See [Figure 66](#) on [page 84](#).

5 Routine and Preventive Maintenance

Maintaining the Source Block Assembly

Figure 62. View of the front side of the source block with the cone wash nozzle turned away from the entrance cone



3. Place a lint-free cloth over the drainage holes in the bottom of the source enclosure.

Tip It is easy to drop small objects such as the entrance cone and the cone wash nozzle into the drain at the bottom of the source enclosure. Temporarily blocking the drainage holes in the bottom of the source enclosure will prevent small objects from falling into the drainage tubing.

4. Turn the entrance cone clockwise and pull forward to remove it.

Note The entrance cone assembly is reverse-threaded. Therefore, to remove it, you turn it clockwise. To install it, you turn it counter-clockwise.



CAUTION Take care when handling the entrance cone. Always store the entrance cone with its cone facing upwards. See [Figure 63](#).

5. Taking care to handle it by its base, carefully remove the cone wash nozzle from the source block.

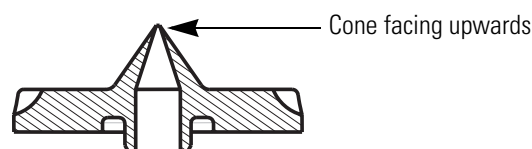


CAUTION Because its tip is fragile, take care when handling the cone wash nozzle.

6. Proceed to one of the following:

- For instructions on cleaning the entrance cone, go to the next procedure.
- For instructions on cleaning the internal components of the source block assembly, go to [“Removing the Source Block Assembly”](#) on [page 85](#).

Figure 63. Entrance cone with cone facing upwards



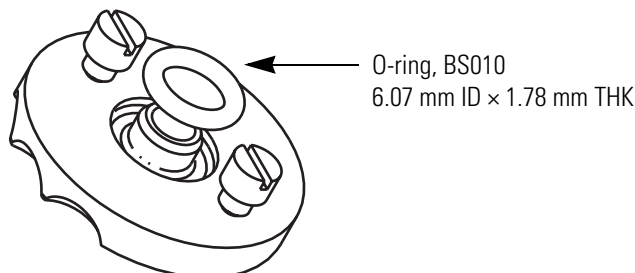
Cleaning the Entrance Cone and the Cone Wash Nozzle

The cone wash nozzle requires cleaning only if it becomes blocked.

❖ To clean the entrance cone and the cone wash nozzle

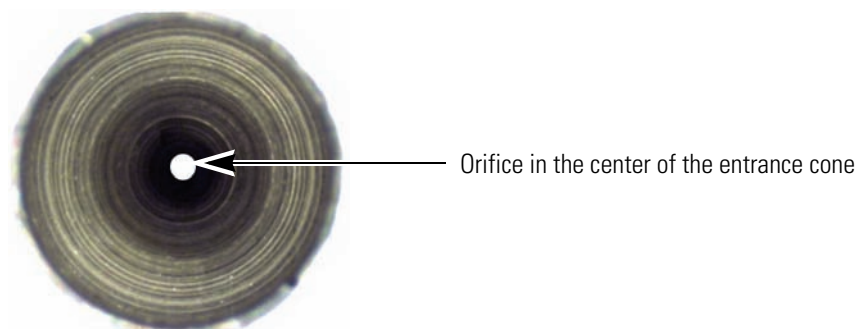
1. Use a 2.5 mm flat blade screwdriver to remove the O-ring on the back of the entrance cone. See [Figure 64](#). For O-ring part numbers, see [Figure 81](#) on [page 110](#).

Figure 64. Backside of the entrance cone, showing its O-ring (BS010)



2. Sonicate the entrance cone in 100% methanol. If methanol does not remove the contamination, sonicate the cone in a 10% v/v aqueous solution of formic acid, rinse with distilled water, and then rinse with 100% methanol.
3. Using a microscope set to 30x magnification, inspect the inside of the cone to ensure cleanliness. See [Figure 65](#).

Figure 65. View of the center of the entrance cone



CAUTION Because solvent and acid can damage them, do not sonicate O-rings.



CAUTION Take care when handling the cone wash nozzle. Its tip is extremely fragile.

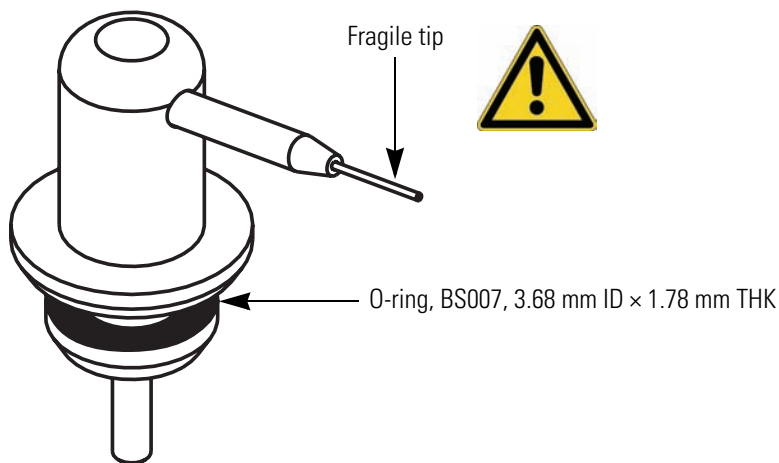
5 Routine and Preventive Maintenance

Maintaining the Source Block Assembly

4. If the cone wash nozzle requires cleaning, remove its O-ring, and then sonicate the nozzle in 100% methanol. If methanol does not remove the contamination, sonicate the cone in a 10% v/v aqueous solution of formic acid, rinse with distilled water, and then rinse with 100% methanol.

For O-ring part numbers, see [Figure 81](#) on [page 110](#).

Figure 66. Cone wash nozzle assembly



5. If you removed the O-rings for the entrance cone and the cone wash nozzle, re-install the O-rings.

Tip Wetting an O-ring with 100% methanol makes it easier to install.

6. Depending on whether the internal components of the source block need cleaning, do one of the following:
 - If the internal components of the source block assembly do not need cleaning, go to [step 7](#).
 - If the internal components of the source block assembly need cleaning, go to the next procedure, [Removing the Source Block Assembly](#).
7. Holding the cone wash nozzle by its base, insert it into the source block, and then turn the nozzle to the right. See [Figure 62](#) on [page 82](#).
8. Insert the entrance cone into the source block, and then turn the entrance cone counter-clockwise until it locks in place. See [Figure 62](#).
9. Remove the lint-free cloth from the bottom of the source enclosure.
10. Take the probe heater out of its holder. Ensure that the knobs of the probe heater are facing away from each other, and then insert the probe heater into the probe mount. See [Figure 56](#) on [page 74](#).

11. Lock the probe heater in place by turning its black knobs forward 90 degrees. See [Figure 55](#) on [page 74](#).
12. Reinsert the API probe into the probe heater and reattach the PEEK fingertight fitting to the probe. See [Figure 35](#) on [page 57](#).

Removing the Source Block Assembly

You must clean the entire source block assembly on a regular basis if you inject complex sample matrices or use highly buffered mobile phases. To clean the source block assembly, you must remove it from the instrument and disassemble it.

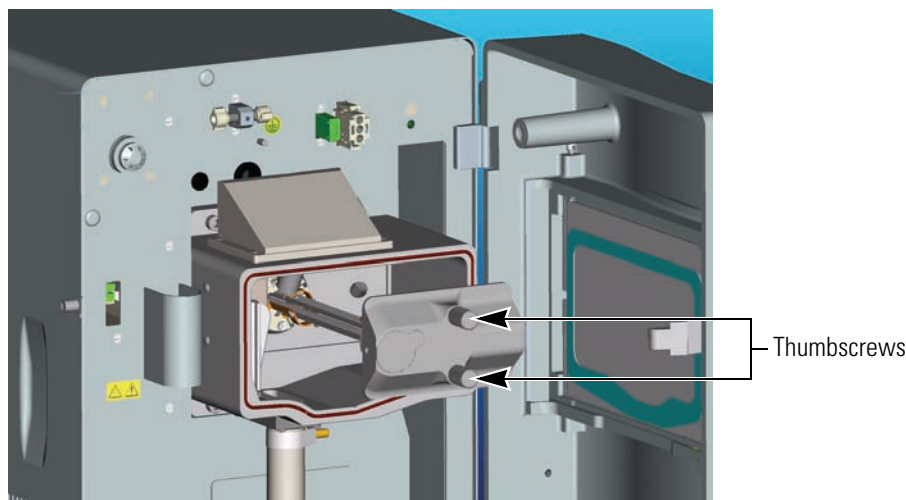
❖ To remove the source block assembly from the MS detector



CAUTION Wait for the source block and probe heater assembly to cool before carrying out any maintenance.

1. If you have not already done so, do the following:
 - Prepare your LC/MS system for maintenance as described in [“Preparing the LC/MS System for Maintenance”](#) on [page 80](#).
 - Remove the probe and the probe heater as described in [“Clearing Access to the Source Block Assembly”](#) on [page 81](#).
 - Remove the entrance cone and the cone wash nozzle as described in [“Removing the Entrance Cone and the Cone Wash Nozzle”](#) on [page 81](#).
2. Loosen the thumbscrews on the source block and pull the source block assembly out of the MS detector. See [Figure 67](#).

Figure 67. View of the source block assembly being removed from the MS detector



Cleaning the RF/dc Prefilter

Refer to [Table 5](#) to determine the frequency at which you need to clean the RF/dc prefilter.

Table 5. Cleaning schedule for RF/dc prefilter

Sample/Solvent Type	Cleaning Frequency
Pure samples and solvents	6 months
Standard samples	3 to 6 months
Complex matrices (for example, crude plasma and urine)	1 to 4 weeks with operation of cone wash
Non-volatile buffer	Weekly with operation of cone wash

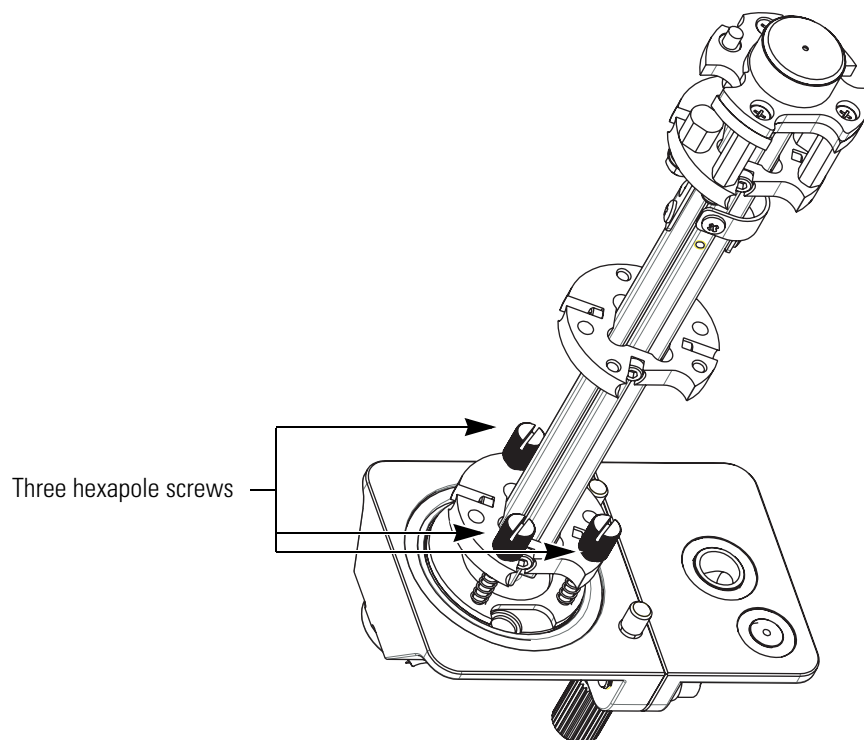
❖ To clean the RF/dc prefilter

IMPORTANT You must wear non-powdered gloves to handle the quadrupole.

1. Support the quadrupole as you unscrew the hexapole screws at the base of the RF/dc prefilter to disconnect it from the source block. See [Figure 68](#).

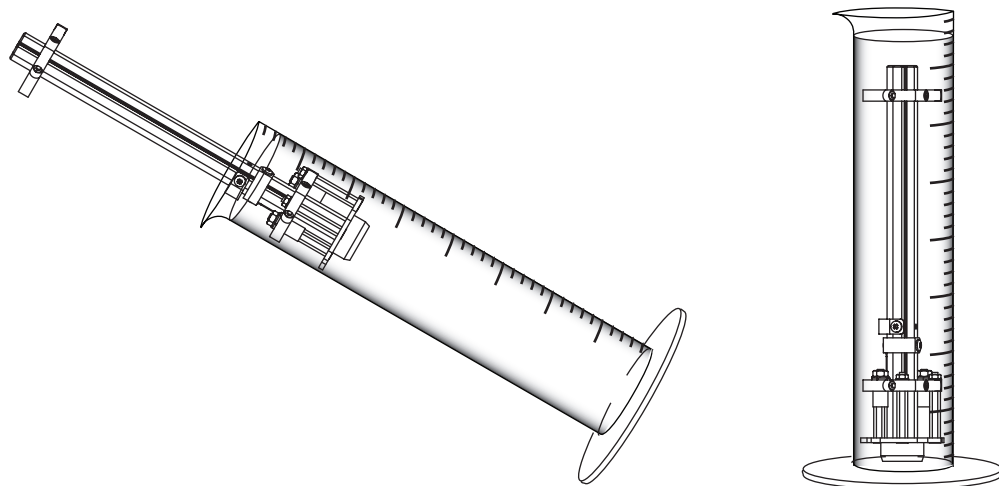
IMPORTANT The hexapole screws shown in [Figure 68](#) have slotted heads. Because these screws should be hand-tightened only, the slots have been removed from the latest version.

Figure 68. Source block assembly with the three screws connecting the RF/dc prefilter to the source block



- Carefully slide the RF/dc prefilter in a 500 mL graduated glass cylinder containing 100% methanol or a mixture of methanol and water. See [Figure 69](#).

Figure 69. Cleaning the RF/dc prefilter



- Sonicate the cylinder for 15 minutes.
- Carefully remove the RF/dc prefilter from the graduated cylinder. Then dry the prefilter with a gentle stream of nitrogen gas.
- Reassemble the source block assembly and reinstall it into the MS detector.
- Pump down the system and determine if the RF/dc prefilter is still dirty.

Note Typically, sonicating the RF/dc prefilter in methanol is adequate to remove contamination. However, contamination from some compounds and some sample matrices can be more difficult to remove. If your RF/dc prefilter is still dirty after sonicating it in methanol, deep clean it as described in step 5 of this procedure.

- If the RF/dc prefilter is still dirty, perform a deep cleaning as described in the following procedure.

❖ **To perform a deep cleaning of the RF/dc prefilter**

- Immerse the RF/dc prefilter in a graduated cylinder containing 100% distilled water. Sonicate for 15 min.
- Decant the water and fill the graduated cylinder with 100% methanol. Sonicate for 15 min.
- Decant the methanol and fill the graduated cylinder with 100% acetone. Sonicate for 15 min.
- Carefully remove the RF/dc prefilter from the cylinder. Rinse the RF/dc prefilter with methanol.
- Dry the prefilter with a gentle stream of nitrogen gas.

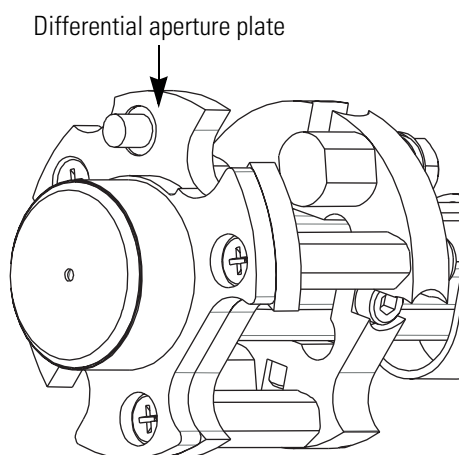
5 Routine and Preventive Maintenance

Maintaining the Source Block Assembly

6. If the differential aperture plate is contaminated with ion burn, clean it:
 - a. Unscrew the three screws that connect the differential aperture plate to the quadrupole. See [Figure 70](#).
 - b. Wipe the inside of the differential aperture plate with a cotton swab soaked in [50:50] methanol / water.
 - c. Reconnect the differential aperture plate to the quadrupole.
 - d. Examine the screws of the differential aperture plate to ensure that they are burr-free and flush or below the plane of the aperture plate.

If you do not need to clean the remaining components of the source block assembly, proceed to [“Assembling the Source Block Assembly”](#) on [page 90](#).

Figure 70. View of differential aperture plate



Cleaning the Extraction Cone and the Source Block

If you use buffered mobile phases or inject samples with complex matrices or both, you might need to clean the source block and the extraction cone on a weekly basis.

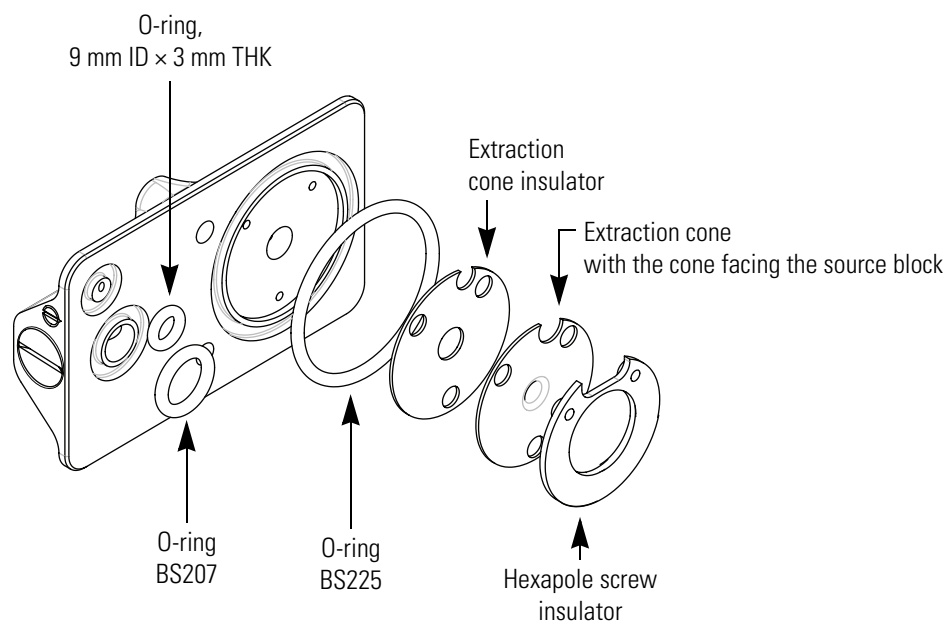


CAUTION Wait for the source block and probe heater assembly to cool before carrying out any maintenance.

❖ **To clean the extraction cone and the source block**

1. Disassemble the remaining components of the source block assembly:
 - a. Remove the hexapole screw insulator, extraction cone, and extraction cone insulator. See [Figure 71](#).

Figure 71. Exploded view of the source block and its components



CAUTION When the extraction cone is not installed in the source block, store it with its cone side facing upwards.

- b. Remove the three O-rings:
 - Top-left O-ring, 9.12 mm ID x 3.53 mm THK, viton black (P/N 00107-01-00047)
 - Bottom-left O-ring, BS207, viton black (P/N FM101417)
 - Right O-ring, BS225, viton black (P/N FM103048)



CAUTION Because solvent and acid can damage the O-rings, do not sonicate them.

5 Routine and Preventive Maintenance

Maintaining the Source Block Assembly

2. To clean the source block, sequentially sonicate it in the following solvents:
 - a. 1% v/v solution of formic acid / distilled water
 - b. 100% distilled water
 - c. 100% methanol
3. To clean the extraction cone:
 - a. Place the extraction cone, with the cone side facing upwards, into a beaker.



CAUTION Exercise great care when handling the extraction cone. Use tweezers to handle the extraction cone and ensure that the cone side faces upwards when you place the extraction cone on a solid surface.

- b. Fill the beaker with 10% v/v solution of formic acid, and then sonicate for approximately 15 min.
- c. Decant the formic acid, fill the beaker with methanol, and then sonicate again for approximately 15 min.

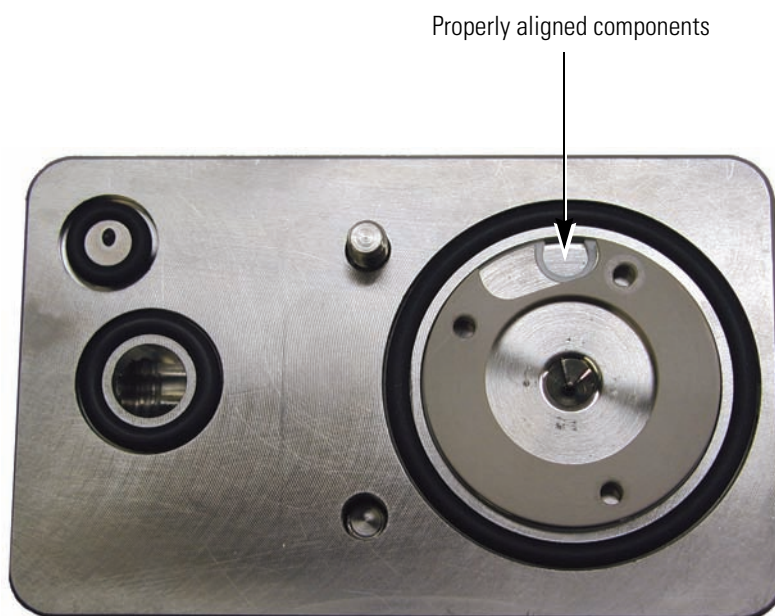
Assembling the Source Block Assembly

After you clean and dry the components of the source block assembly, reassemble it.

❖ To reassemble the source block assembly

1. Install the three o-rings (see [Figure 71](#)):
 - Top-left O-ring, 9.12 mm ID × 3.53 mm THK, viton black (P/N 00107-01-00047)
 - Bottom-left O-ring, BS207, viton black (P/N FM101417)
 - Right O-ring, BS225, viton black (P/N FM103048)
2. First install the extraction cone insulator (P/N FM102264). Then install the extraction cone (P/N FM102263) with the cone facing the source block. And finally install the hexapole screw insulator (P/N FM102248). See [Figure 71](#).
3. Ensure that the semicircular cutouts in these three components line up as shown in [Figure 72](#).
4. Align the three spring screws at the base of the RF/dc prefilter to the three holes on the backside of the source block. Support the quadrupole as you alternately screw the three spring screws at the base of the RF/dc prefilter into the source block. See [Figure 68](#) on [page 86](#).

Figure 72. View of the source block with the proper alignment of the extraction cone insulator, extraction cone, and hexapole insulator



Installing the Source Block Assembly

You must reinstall the source block assembly before you prepare the system for operation.



CAUTION The corona pin is very sharp. Do **not** attempt to install the entrance cone or the cone wash nozzle before you turn the corona pin knob to its vertical position. See [Figure 73](#).

❖ To install the source block assembly into the MS detector

1. Insert the source block assembly (P/N FM102573) into the source block enclosure, and then tighten the thumbscrews on the source block. See [Figure 67](#) on [page 85](#).



CAUTION The tip of the cone wash nozzle is very fragile.

2. Handling the cone wash nozzle by its base because the tip is fragile, insert the cone wash nozzle (P/N FM102521) into the source block, and then carefully rotate the nozzle tip to the right. See [Figure 73](#).

Figure 73. View of the source block, showing the cone wash nozzle turned to the right



3. Remove the lint-free cloth from the bottom of the source enclosure.
4. Insert the entrance cone into the source block, and then turn it counter-clockwise. [Figure 63 on page 82.](#)

Note The entrance cone assembly is reverse-threaded. Therefore, to remove it, you turn it clockwise. To install it, you turn it counter-clockwise.

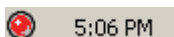
5. Install the probe heater:
 - a. Remove the probe heater from its holder, and then rotate the black knobs of the probe heater, so that they face away from each other. See [Figure 56 on page 74.](#)
 - b. Insert the tube portion of the probe heater into the probe mount.
 - c. Rotate the black knobs of the probe heater forward to their locking position. See [Figure 55 on page 74.](#)
6. Install the API probe:
 - a. Turn the locking plate clockwise to the open position and carefully insert the ESI probe (P/N FM102595) (see [Figure 36 on page 58](#)) or the APCI probe (P/N FM102587) (see [Figure 49 on page 68](#)) into the probe heater.



CAUTION Take care not to damage the tip of the API probe capillary. If you are installing the ESI probe, take care not to damage its ceramic sleeve.

- b. Turn the locking plate counterclockwise into the closed position.
- c. Screw the PEEK fingertight fitting into the ESI probe assembly (P/N FM102595) or APCI probe assembly (P/N FM102587).

7. Pump down the MS detector:



5:06 PM

- a. Right-click the **Surveyor MSQ Plus Server** icon in the System Tray portion of the Windows Taskbar to open the shortcut menu.

The Server icon is red because the system is vented.

- b. Choose **Pump** from the shortcut menu.
- c. Wait for the Surveyor MSQ Plus MS detector to reach high vacuum. The server light will change from flashing amber to solid amber. Reaching high vacuum takes approximately 10 minutes.

Maintaining the Forepump

The vacuum system consists of two types of vacuum pumps, a turbomolecular pump and a forepump. The turbomolecular pump is housed within the Surveyor MSQ Plus MS detector and requires a trained service engineer for servicing. The forepump (also referred to as a backing pump, a rotary pump, or a roughing pump) is external to the Surveyor MSQ Plus MS detector and requires routine maintenance for optimal performance.

Note You can find more information on operating and maintaining the rotary pump in the manual that is shipped with the pump.

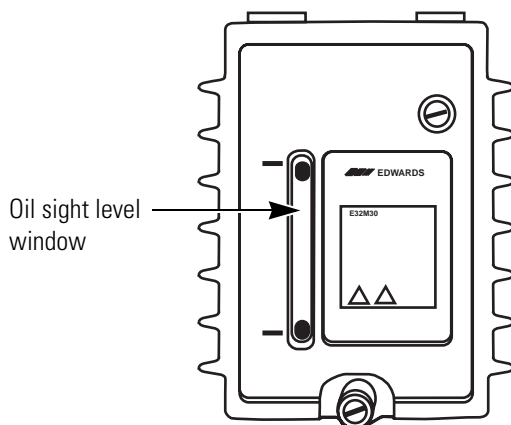
Check both the level and the color of the oil in the forepump at least once a month.

Check the oil by looking through the oil sight level window of the forepump. See [Figure 74](#). The oil level should be between the upper and lower marks positioned next to the window. The oil color should be a clear straw color.

- If the oil level is near or below the lower mark, add more oil as described in the manual that comes with the Edwards forepump.
- If the oil has turned red in color or if the pump has been in operation for more than 3000 hours since the oil was replaced, replace the oil as described in the manual that comes with the Edwards forepump.

Purge the oil on a regular basis as described in “[Draining the Oil Mist Filter and Purging the Pump Oil](#)” on [page 48](#).

Figure 74. View of Edwards forepump that shows the oil sight level window



System Shutdown, Startup, and Reset

When you are not performing analyses, you can temporarily turn off the nitrogen gas or set the Surveyor MSQ Plus MS detector to the Off mode. Turning off the nitrogen gas between intermittent analyses conserves the nitrogen supply. Placing the system in the Off mode conserves the laboratory nitrogen supply and increases the life of the ion detection system.

Some of the maintenance procedures contained in the Maintenance chapter require that the system be completely shut down. To shut down the system, you must turn the vacuum system off.

Restarting the system after a complete shutdown requires building up the vacuum to a working level. If you are restarting the system after moving it to a new location, we recommend that you perform a Full System Autotune.

Contents

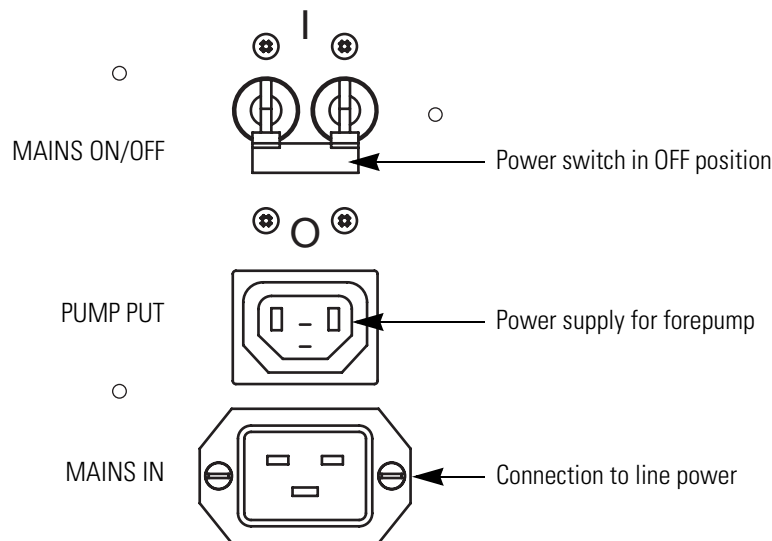
- [Shutting Down the System in an Emergency](#)
- [Turning Off the Nitrogen Gas](#)
- [Placing the System in the Off Mode](#)
- [Shutting the System down for Non-Routine Maintenance](#)
- [Restarting the System Following a Complete Shutdown](#)
- [Resetting the MS Detector](#)

Shutting Down the System in an Emergency

If you need to turn off the MS detector in an emergency, place the main power circuit breaker switch in the Off (O) position. See [Figure 75](#). The main power switch, which is labeled MAINS ON/OFF is located on the rear panel of the MS detector in the lower-right quadrant. Turning the main power switch to the Off position turns off all power to the MS detector, including the forepump. Although removing power abruptly will not harm any component within the system, this is not the recommended shutdown procedure to follow. Refer to [“Shutting the System down for Non-Routine Maintenance”](#) on [page 100](#) for the recommended procedure.

To turn off your LC devices and your data system computer in an emergency, use their ON/OFF switches.

Figure 75. View of MAINS ON/OFF circuit breaker switch



Turning Off the Nitrogen Gas

If you are performing intermittent analyses throughout the day and you want to conserve nitrogen, you can turn off the nitrogen gas between analyses.

❖ To turn off the nitrogen gas

1. If you are using the optional cone wash pump, turn it off by turning its power switch to the Off position.
2. Turn off the flow from your LC pump:
 - a. If your LC pump is controlled from the Xcalibur data system, turn it off from its respective Direct Control dialog box or place it in the Standby mode from the Info View - Status page.
 - b. If your LC pump is not controlled from the Xcalibur data system, turn it off from its keypad control panel.
3. If the Tune window is not open, open it:



- a. Double-click the **Tune** icon on the Windows desktop.

After you double-click the Tune icon, the Server icon appears in the System tray (to the left of the clock) of the Windows taskbar.

- b. Double-click the Server icon to open the Tune window.

4. If the Per Method Parameters table is not open, open it by clicking the Show Per Method Parameters slide bar on the right side of the Tune window.
5. Turn off the nitrogen gas by clicking the Nitrogen Gas toggle On/Off button in the General Control group of the Per Method Parameters table.

The On/Off button turns from green to gray and the text to the left of the button changes from On to Off. Within a few seconds, you hear the nitrogen supply to the API source shut off. In the Nitrogen Gas Off mode, the system maintains a bleed of nitrogen gas to the probe to prevent a rise in humidity within the source compartment.

Placing the System in the Off Mode

Place the Surveyor MSQ Plus MS detector in the Off mode if you are not going to use it for a short period of time, such as overnight or over weekends. In the Off mode, the system is left under vacuum, but the nitrogen flow is reduced to a bleed through the API probe, the electron multiplier and conversion dynode are turned off, the power to the ion optics is turned off, and the probe heater is turned off.

Therefore, placing the instrument in the Off mode allows you to conserve your laboratory nitrogen supply and increase the lifespan of the electron multiplier. In addition, you can restart and operate a Surveyor MSQ Plus MS detector that has been left in the Off mode without waiting for the vacuum system to pump down to a working level.

Note

1. Leave the Surveyor MSQ Plus MS detector under vacuum when you are switching the API probes. **Do not vent** the instrument unless you are performing a maintenance procedure that requires you to break the integrity of the vacuum.
2. Before you place the Surveyor MSQ Plus MS detector in the Off mode, turn off the flow from the LC pump and the flow from the optional cone wash pump.

You turn the MS detector off from the Status view in Xcalibur or from the Tune window. This section contains the following topics:

- [Turning the MS Detector Off from Xcalibur](#)
- [Turning the MS Detector Off from the Tune Window](#)

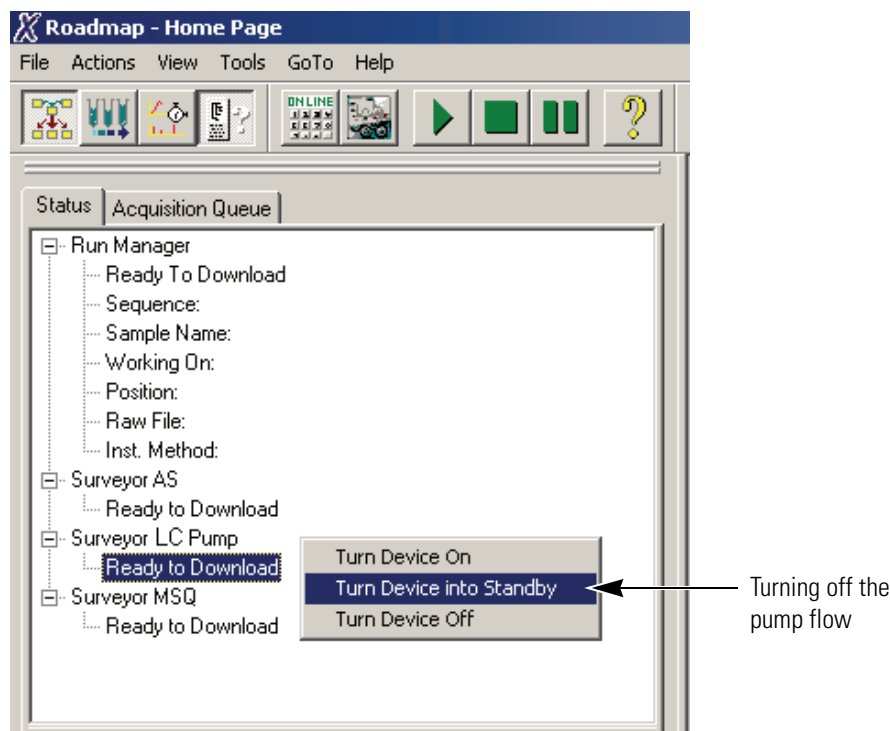
Turning the MS Detector Off from Xcalibur

❖ **To turn off the MS detector from the Info View - Status page of Xcalibur**



1. If you are using the optional cone wash pump, turn it off by turning its power switch to the Off position.
2. If Xcalibur is not open, open it by double-clicking the **Xcalibur** icon on the Windows desktop.
3. If the Info view is not displayed, choose **View > Info View** to display it. Then, click the Status tab to open the Status page.
4. To turn off the flow from the LC pump:
 - If your LC pump is controlled from the Xcalibur data system, right-click the pump listing on the Status page. Then, choose **Turn Device Into Standby** from the shortcut menu. See [Figure 76](#).
 - If your LC pump is not controlled from the Xcalibur data system, turn it off from its control keypad.

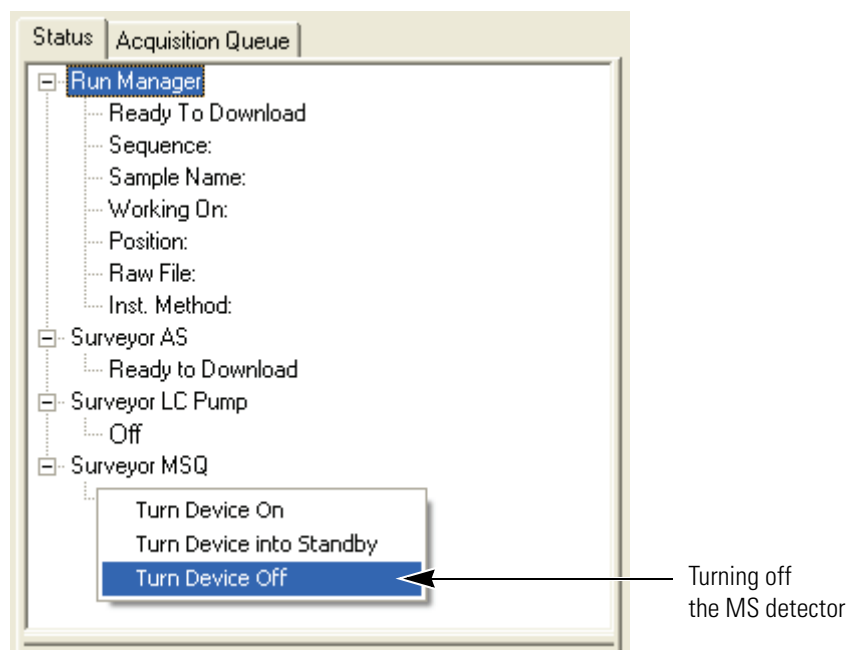
Figure 76. Info View- Status page, showing the status of the Surveyor MS Pump and its shortcut menu



5. Right-click the Surveyor MSQ listing on the Status page. Then, choose **Turn Device Off** from the shortcut menu to place the MS detector in the Off mode. See [Figure 77](#).

The status LED on the front of the Surveyor MSQ Plus MS detector turns yellow.

Figure 77. Info view - Status page, showing the shortcut menu for the Surveyor MSQ Plus MS detector



Turning the MS Detector Off from the Tune Window

❖ To turn off the Surveyor MSQ Plus MS detector from the Tune window

1. If you are using the optional cone wash pump, turn it off by turning its power switch to the Off position.
2. Turn off the flow from your LC pump:
 - a. If your LC pump is controlled from the Xcalibur data system, turn it off from its respective Direct Control dialog box or place it in the Standby mode from the Info View - Status page.
 - b. If your LC pump is not controlled from the Xcalibur data system, turn it off from its keypad control panel.
3. If the Tune window is not open, open it:
 - a. Double-click the **Tune** icon on the Windows desktop.
After you double-click the Tune icon, the Server icon appears in the Windows taskbar.
 - b. Double-click the Server icon to open the Tune window.
4. If the Per Method Parameters table is not open, open it by clicking the Per Method Parameters slide bar on the right side of the Tune window.



5. Turn off the power to the ion optics by clicking the Operate toggle On/Off button.

The On/Off button turns from green to gray and the text to the left of the button changes from On to Off.

6. Turn off the nitrogen gas by clicking the Nitrogen Gas toggle On/Off button in the General Control group of the Per Method Parameters table.

The On/Off button turns from green to gray and the text to the left of the button changes from On to Off. Within a few seconds, you hear the nitrogen supply to the API source shut off. In the Nitrogen Gas Off mode, the system maintains a bleed of nitrogen gas to the probe to prevent a rise in humidity within the source compartment.

7. When you plan to leave the Surveyor MSQ Plus MS detector in the Off mode for a significant period of time, turn off the nitrogen supply to the system at the main regulator.

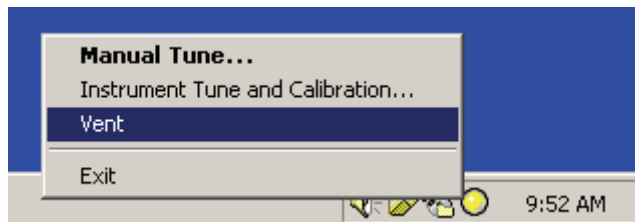
Shutting the System down for Non-Routine Maintenance

❖ To shutdown the Surveyor MSQ system for a non-routine maintenance procedure or to relocate the instrument

1. If you are using the optional cone wash pump, turn it off by turning its power switch to the Off position.
2. Open the Xcalibur data system by choosing **Start > All Programs > Xcalibur > Xcalibur** from the Windows desktop.
3. If the Info View is not displayed, choose **View > Info View** to display it.
4. Turn off the flow from the LC pump:
 - a. If your LC pump is controlled from the Xcalibur data system, turn it off from its respective Direct Control dialog box or place it in the Standby mode from the Info View - Status page.
 - b. If your LC pump is not controlled from the Xcalibur data system, turn it off from its keypad control panel.
5. Right-click the Surveyor MSQ listing on the Status page. Then, choose **Turn Device Off** from the shortcut menu.
6. Right-click the Surveyor MSQ Plus Server in the System Tray portion of the Windows Taskbar. Then, choose **Vent** from the shortcut menu to vent the system. Venting the system turns off the turbomolecular pump.

The Server is displayed as an icon in the Windows Taskbar just to the left of the time display. See [Figure 78](#).

Figure 78. View of taskbar showing the Server icon and the shortcut menu



7. Exit Xcalibur. Then, close the Server.
8. Wait for approximately 2 minutes. Then, turn off the Surveyor MSQ Plus MS detector by setting the MAINS IN switch to the Off position.

Turning off the power to the Surveyor MSQ Plus MS detector also turns off the power to the Edwards forepump, which gets its line power from the Pump Out receptacle on the rear panel of the MS detector.
9. Close the nitrogen gas cylinder at the main regulator.

Restarting the System Following a Complete Shutdown

Following a long-term shutdown, carry out the visual checks listed in the Pre-switch on checklist shown in [Table 6](#), and then follow the system start-up procedure.

This section contains the following topics:

- [Checking the System Connections](#)
- [Restarting the Surveyor MSQ Plus System](#)

After you complete these procedures, the system is ready for a Full System Autotune. Refer to the *Surveyor MSQ Getting Started* manual for details.

Checking the System Connections

Before you switch on the system after an extended shutdown period, a major overhaul, or instrument relocation, perform a number of visual checks on the system.

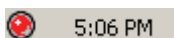
Table 6. Pre-switch on checklist

Items	Check
Power Connections	
The Surveyor MSQ Plus MS detector and your LC devices are connected to line power.	
The Edwards forepump is connected to the Pump Out receptacle on the rear panel of the Surveyor MSQ Plus MS detector.	
Communication Connections	
Surveyor MSQ Plus MS detector is connected to the data system computer with a USB cable	
The communication cables for the LC devices are appropriately connected to the data system computer.	
Gas Connections	
<ul style="list-style-type: none">The GAS IN port on the rear panel of the Surveyor MSQ Plus MS detector is connected to a nitrogen supply and the auxiliary regulator is set to 75 psi (5.2 bar).Any required gas connections required for the LC system have been made.	
Vacuum Connections	
<ul style="list-style-type: none">The Source manifold on the rear panel of the Surveyor MSQ Plus MS detector is connected to the forepump.The Backing manifold on the rear panel of the Surveyor MSQ Plus MS detector is connected to the forepump.	
Exhaust Connections	
<ul style="list-style-type: none">The Exhaust manifold on the rear panel of the Surveyor MSQ Plus MS detector is connected to the solvent trap. The solvent trap is connected to a fume hood or an industrial vent.The Oil Mist Filter is connected to the exhaust port of the Edwards forepump. The blue hosing is used to connect the Oil Mist Filter to a fume hood or an industrial vent.	
LC Plumbing, Hardwire Connections, and Solvent Supply	
<ul style="list-style-type: none">The appropriate plumbing connections have been made for the LC system.The appropriate contact closure connections have been made between the modules of the LC system and between the LC system and the Surveyor MSQ Plus MS detector.For the Surveyor LC, check the connections for the system synchronization harness.The solvent reservoirs for the LC system are filled with the appropriate solvents.The waste bottle for the LC system waste solvents is empty.The solvent lines for the LC system are free of air.	

Restarting the Surveyor MSQ Plus System

❖ To start up the Surveyor MSQ Plus system

1. Turn on the power for your system:
 - a. Turn on the power to the Surveyor MSQ system by toggling the MAINS IN switch to the On position.
 - b. Turn on the power to your LC devices. Wait for the LC devices to complete their initialization before proceeding.
 - c. Turn on the Edwards forepump by setting its power switch to the On position.
2. Turn on the data system computer. Wait until Windows is running. From the Windows desktop, double-click the Xcalibur icon.
3. Pump down the instrument:



- a. Right-click the **Surveyor MSQ Plus Server** icon in the System Tray portion of the Windows Taskbar and choose **Pump** from the shortcut menu.
 - b. Wait for the Surveyor MSQ Plus MS detector to reach high vacuum.
When system reaches the appropriate vacuum pressure, the server light changes from flashing amber to solid amber. Reaching high vacuum takes approximately 10 minutes.
 - c. If the Surveyor MSQ Plus MS detector has not reached vacuum after 30 minutes (The server light might still be red or flashing amber. Refer to [Table 2](#) on [page 20.](#)), check for leaks in the system.
4. Open the Tune window by double-clicking the Server icon in the System Tray of the Windows Taskbar.
 5. If the Per Method Parameters table is not open, open it by clicking the Per Method Parameters slide bar on the right side of the Tune window. See [Figure 79](#).
 6. Turn on the nitrogen gas by clicking the Nitrogen Gas toggle On/Off button in the General Control group of the Per Method Parameters table ([Figure 79](#)).
The On/Off toggle button turns from gray to green and the text to the left of the button changes from Off to On. Within a few seconds, you hear the nitrogen supply to the API source turn on.
 7. Put the instrument into the Operate mode by clicking the toggle Operate On/Off button ([Figure 79](#)).
The button turns from gray to green and the text changes from Off to On.

- To set the probe temperature, click the Probe Temperature Setpoint box, and then type an appropriate value for your application (Figure 79).

The Surveyor MSQ Plus MS detector is ready to use as soon as the Probe Temperature readback value approaches that in the setpoint box, although for most stable operation Thermo Fisher Scientific recommends that you wait approximately 10 min for the source to equilibrate.

- Turn on the flow from the LC pump.

Figure 79. Per Method Parameters table

Per Method Parameters slide bar

Description	Readback	Setpoint
Tune Control		
Probe Temperature (°C)	0	334
Needle (kV)	0.0	3.0
Acquisition Control		
Retention Time (mins)	0.00	n/a
General Control		
Operate	n/a	Off <input type="checkbox"/>
Nitrogen Gas	n/a	Off <input type="checkbox"/>
Ionization Mode	n/a	ESI

Hide Per Method Parameters

Probe Temperature Setpoint box

Operate toggle button

Nitrogen Gas toggle button

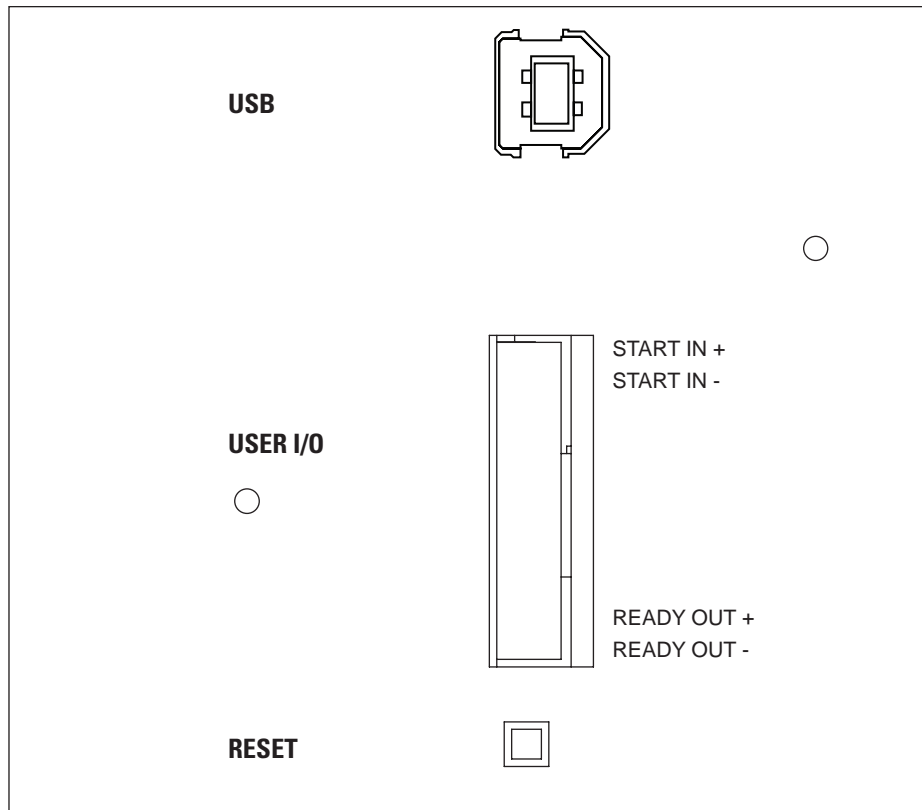
Resetting the MS Detector

If communication between the MS detector and data system computer is lost, it might be necessary to reset the MS detector using the Reset button on the power panel. Pressing the Reset button creates an interrupt on the CPU PCB of the embedded computer. This causes the embedded computer to restart into a known (default) state.

The procedure given here assumes that the MS detector and data system computer are both powered on and operational. If the MS detector, data system computer, or both are off, refer to “Restarting the Surveyor MSQ Plus System” on page 103.

To reset the MS detector, press the Reset button located in the upper right-hand corner of the MS detector’s rear panel. See Figure 80.

Figure 80. View of the upper right-hand corner of the MS detector's rear panel



Replaceable Parts

This chapter lists the parts most commonly used in the course of working with and maintaining your Surveyor MSQ Plus MS detector.

The parts are categorized as follows:

- **Consumables.** Keep a stock of each of these parts, as you might need to replace them frequently.
- **Spares.** You can order these parts as required.
- **Connection Kits.** This is the interface kit that you use to connect an Agilent LC system to your MS detector. You do not need an interface kit to connect a Surveyor LC to your Surveyor MSQ Plus MS detector.

The manuals for the Surveyor MSQ Plus MS detector are provided on the software CD.

Consumables

The MSQ Plus Annual Maintenance kit (P/N 60111-62014) contains all the consumables required for the upkeep of your Surveyor MSQ Plus MS detector. The parts contained in this kit are listed below.

MSQ Plus Annual Maintenance kit	60111-62014
Adaptor (capillary retainer nut)	FM102590
APCI probe capillary (3 each)	FM102594
Aqua nozzle assembly (cone wash nozzle) (2 each)	FM102521
Entrance skimmer assembly, titanium (1 each)	60111-60049
ESI probe capillary (3 each)	FM102598
ESI ceramic sleeve (1 each)	FM103394
Exit cone (extraction cone) (1 each)	FM102263
Ferrule, SGE 1/16 graphite vespel (10 per package)	6070119
Kit, hardware and O-ring, MSQ Plus	60111-62018
Kit, heater repair	60111-62010
Oil reservoir 190/240/260 for turbomolecular pump	00950-01116
Tool, turbomolecular pump oil change	FM104442
Tube insert for API probe, PEEK (package of 12)	FM102591
Heater Repair kit	60111-62010
Detent screw insulator	FM102585
Screw sleeve	FM102582
Spring screw	FM102583
Spring cup	FM102584
MSQ Plus Hardware and O-ring kit	60111-62018
Sealing plug (2 each)	FM102277
Ferrule, SGE 1/16 graphite vespel (10 each)	6070119
Spring, E-type clip, stainless steel, MSQ only (3 each)	FM102574
Spring, compression, 4.6 mm OD, 0.45 N/mm, 30 mm length	00111-01-00013
O-ring, 13.87 mm ID × 3.53 mm THK, BS207 ¹ , BLK VITON (1 each) ..	FM101417
O-ring, 3.68 mm ID × 1.78 mm THK, BS007, BLK VITON (2 each) ...	FM101464
O-ring, 3.0 mm ID × 1.0 mm THK, BLK VITON (2 each)	5711933
O-ring, 9.25 mm ID × 1.78 mm THK, BS012, VITON, for entrance cone(2 each)	FM100231
O-ring, 12.42 mm ID × 1.78 mm THK, BS014, BLK VITON (1 each) ...	FM101522
O-ring, 9.0 mm ID × 3.0 mm THK, BLK VITON (1 each)	00107-01-00026
O-ring, 3.30 mm ID × 2.4 mm THK, BLK VITON (2 each)	FM103016
O-ring, 47.22 mm ID × 3.53 mm THK, BS225, BLK VITON (1 each) ...	FM103048
O-ring, 6.07 mm ID × 1.78 mm THK, BS010, BLK VITON (2 each)	5711000
O-ring, 18.77 mm ID × 1.78 mm THK, BS018, BLK VITON (2 each)	5711035
O-ring, 2.90 mm ID × 1.78 mm THK, BS5006, BLK VITON (2 each) ...	TORN003
O-ring, 5.28 mm ID × 1.78 mm THK, BS009, BLK VITON (2 each)	5711020

¹British Standard

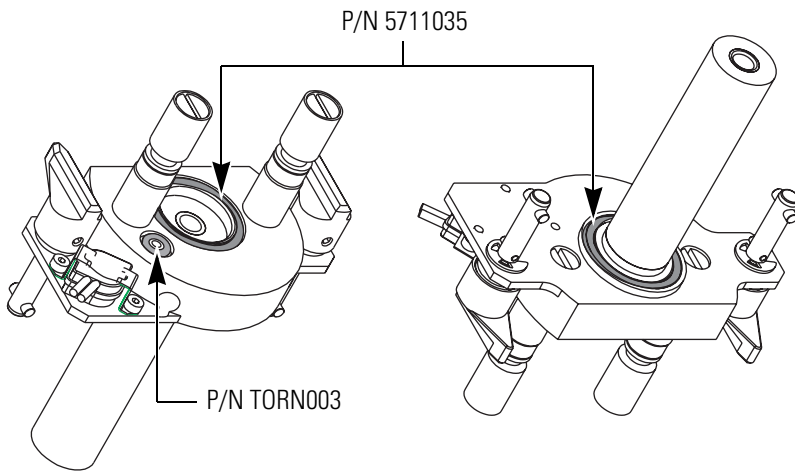
Table 7 lists the location of the O-rings and Figure 81 shows the location of the O-rings.

Table 7. Description of where the O-rings (listed in order of size) are used

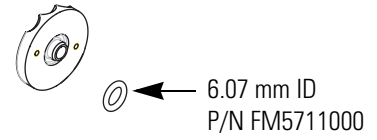
Part Number	Size			Where Used
	ID (mm)	THK (mm)		
TORN003	2.9	1.78	BS006	Probe heater assembly
5711933	3.0	1.0		Source block assembly, small sealing plug
FM103016	3.3	2.40		Probe mount assembly
FM101464	3.68	1.78	BS007	Cone wash nozzle
5711020	5.28	1.78	BS009	API probe
5711000	6.07	1.78	BS010	Entrance cone
00107-01-00026	9.12	3.53		Source block assembly
FM100231	9.25	1.78	BS012	API probe
FM101522	12.42	1.78	BS014	Source block assembly, large sealing plug
FM101417	13.87	3.53	BS207	Source block assembly
5711035	18.77	18.77	BS018	Probe heater assembly
FM103048	47.22	3.53	BS225	Source block assembly

Figure 81. Location of O-rings

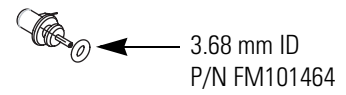
Probe heater



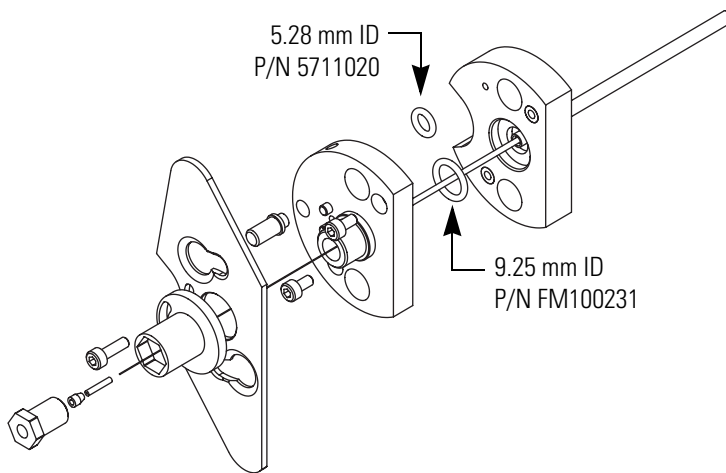
Entrance cone



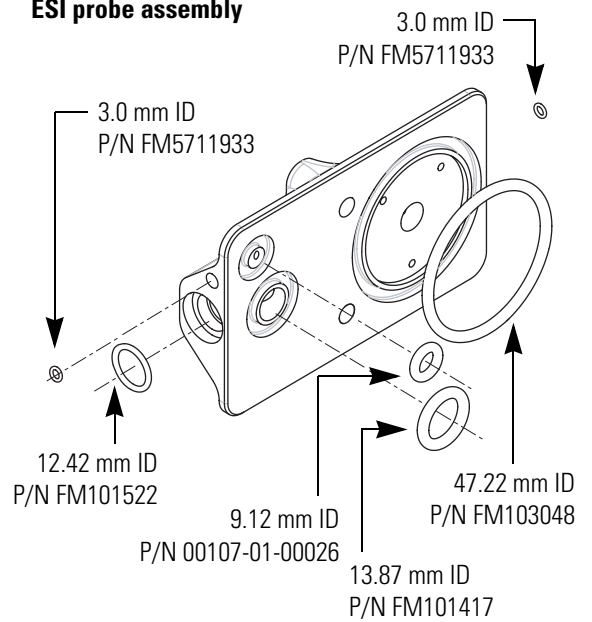
Cone wash nozzle



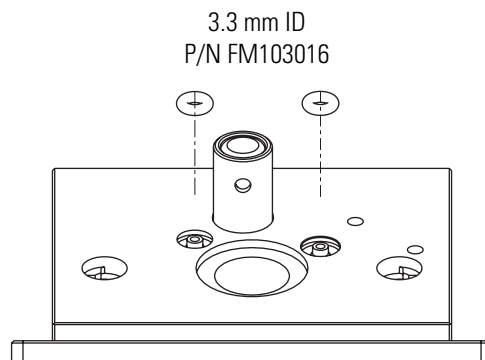
ESI probe assembly



ESI probe assembly



Probe mount assembly



Spares

Order the following spare parts and kits as required.

General Spares

Assembly, probe heater	60111-60023
Cable, USB, A to B (2 m)	00302-99-00008
Conversion dynode	FM102888
Digital board	60111-61050S
Exchange board	EXFM102818
Door, main assembly	60111-60009
Entrance, cone, titanium (with O-ring and bayonet pins)	60111-60049
Fitting, Swagelok tube fitting, stainless steel female ISO tapered thread connector for 1/4-in. OD tubing	00101-02-00006
Fitting, pipe, 6 mm × 1/4-in. NPT	00103-02-00001
Hexapole screw insulator	FM102248
Needle, corona (for APCI mode)	70005-98033
Source board, sub-assembly standard with bracket	60111-61030
Thumbnuts, source block	FM101528
Hexapole screws	60111-20055
Tubing, 6 mm OD, PTFE, for nitrogen line, (order by the foot length)	00109-99-00004

Kits

Kit, tool, Surveyor MSQ	60111-62100
Kit, Surveyor MSQ Plus installation	60111-62006
Kit, sensitivity test, Surveyor MSQ system	FM104284
Qualification	OPTON-00915
Software CD kit, MSQ Plus	OPTON-20432

Source Block Assembly

Figure 82 contains an exploded view of the source block assembly with component descriptions and part numbers.

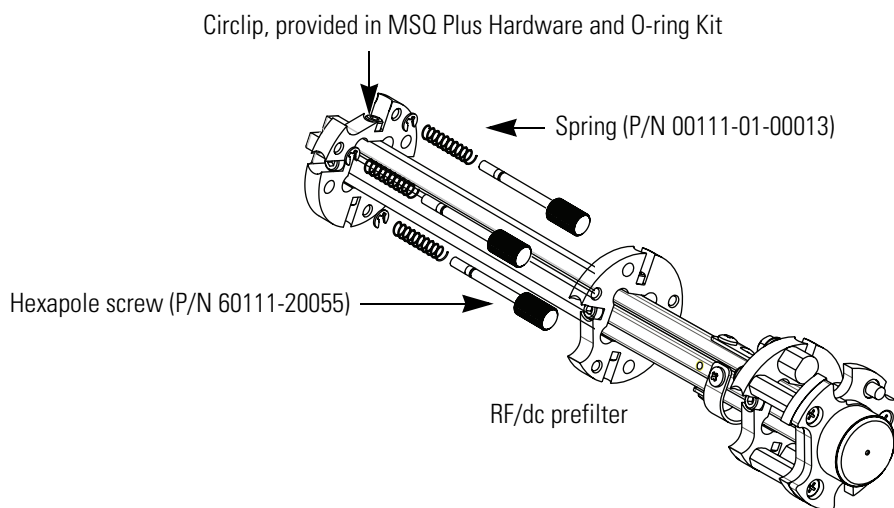
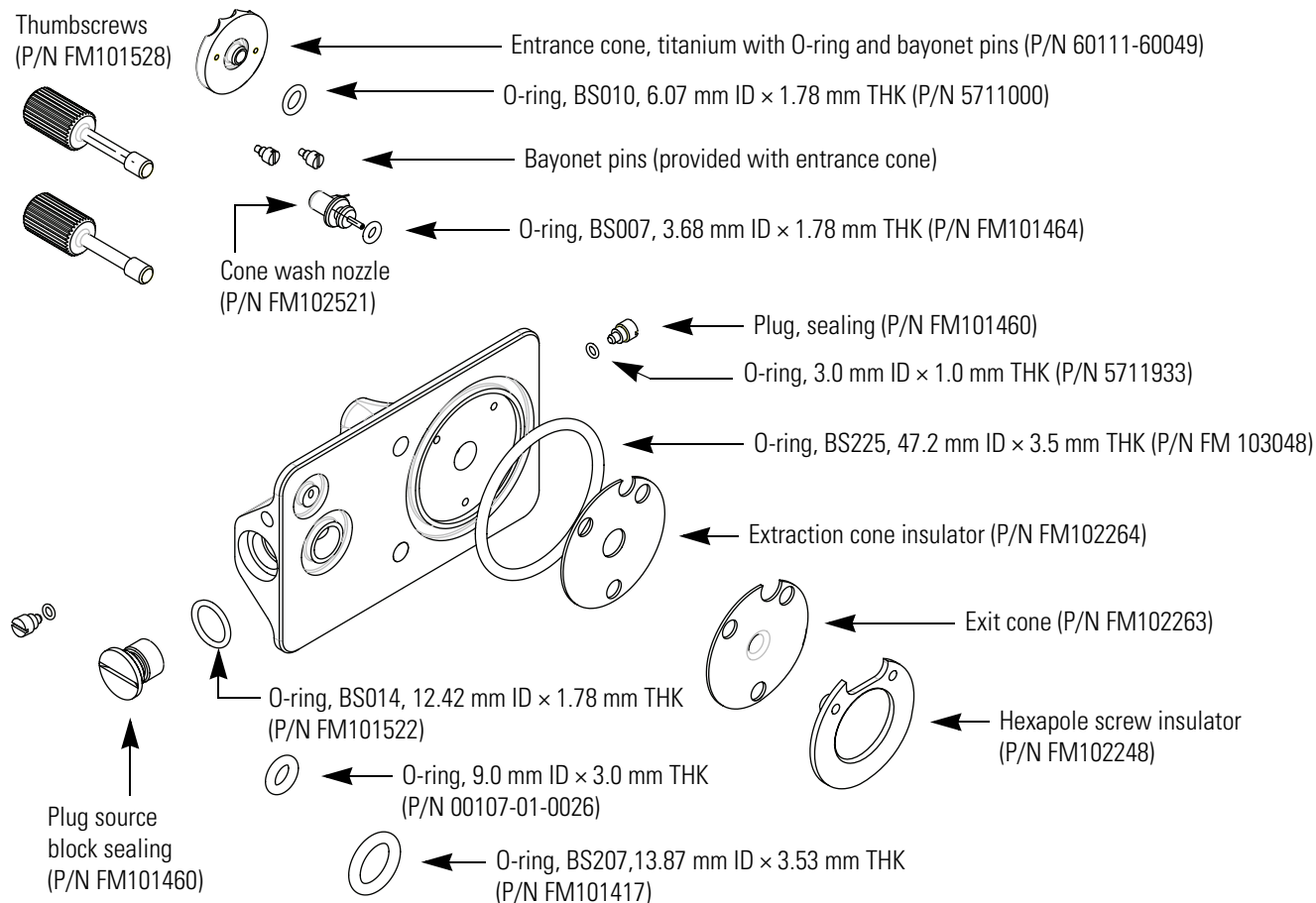
Assembly, source block and transfer lens	60111-60051
Assembly, titanium entrance cone (with O-ring and bayonet pins)	60111-60049
Assembly, nozzle	FM102521
CAP kit	60111-62012
CAP repair kit	60111-62017
Circlip, 2.5 mm, E-type	FM102574
Exit cone (extraction skimmer)	FM102263
Exit cone insulator (extraction cone insulator)	FM102264
Hexapole screw insulator	FM102248
O-ring, 6.07 mm ID × 1.78 mm THK, BS010, BLK VITON (for entrance cone)	5711000

7 Replaceable Parts

Spares

O-ring, 3.0 mm ID × 1.0 mm THK, BLK VITON (for sealing plug)	5711933
O-ring, 13.87 mm ID × 3.53 mm THK, BS207, BLK VITON.	FM101417
O-ring, 12.42 mm ID × 1.78 mm THK, BS014, BLK VITON.	FM101522
O-ring, 9.0 mm ID × 3.0 mm THK, BLK VITON	00107-01-00026
O-ring, 47.22 mm ID × 3.53 mm THK, BS225, BLK VITON.	FM103048
O-ring, 3.68 mm ID × 1.78 mm THK, BS007, BLK VITON (for cone wash nozzle)	FM101464
Plug, source block sealing	FM101460
Screw, M3 × 10, cap head, stainless steel.	FM103046
Screw, hexapole.	60111-20055
Source block	FM102279
Spring.	00111-01-00013

Figure 82. Source Block Assembly, exploded

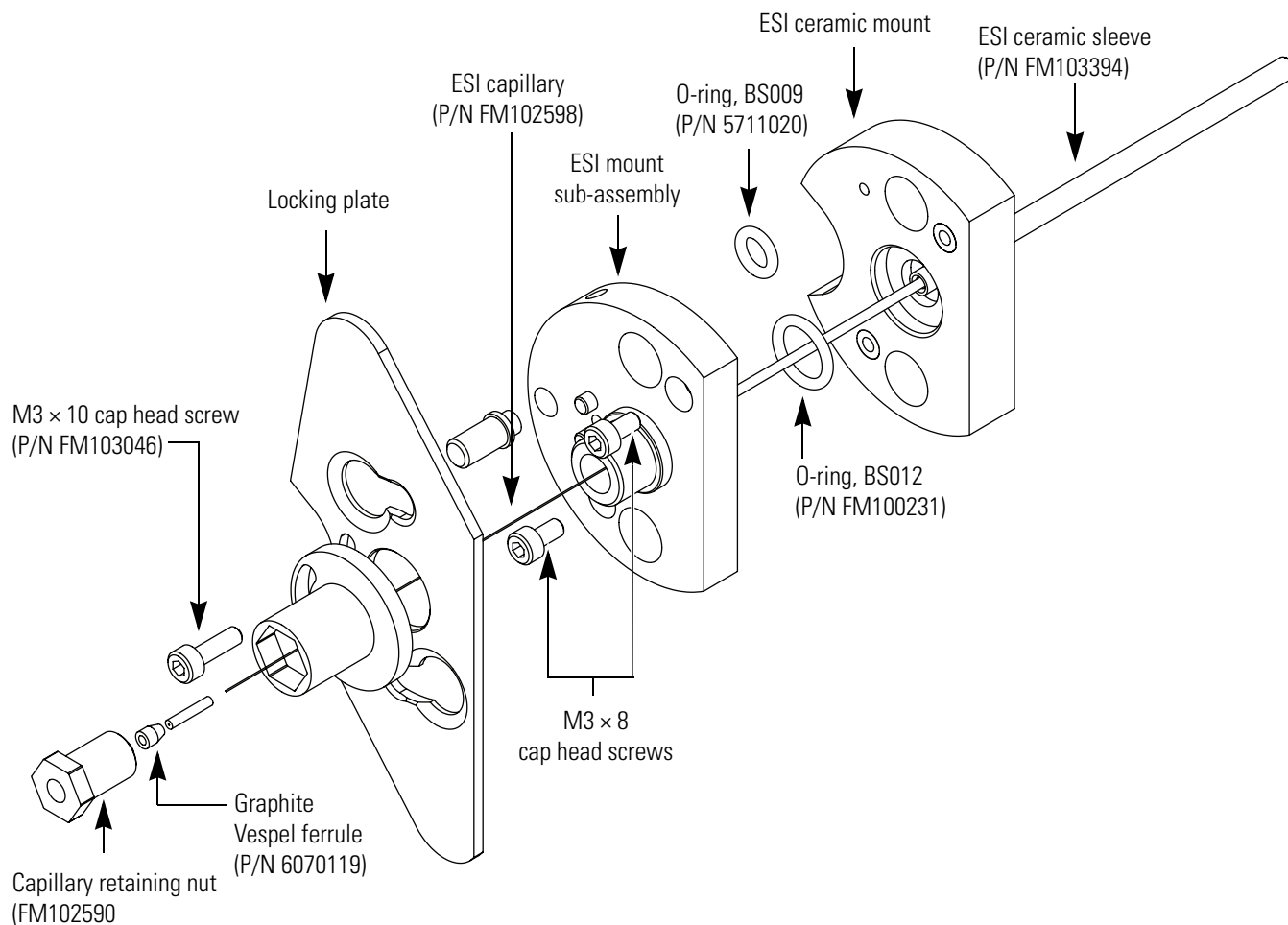


ESI probe Assembly

An exploded view of the ESI probe assembly is shown in [Figure 83](#).

Assembly, ESI probe	FM102595
ESI probe capillary	FM102598
ESI ceramic sleeve	FM103394
Ferrule, GVF/16, graphite vespel	6070119
O-ring, 9.25 mm ID × 1.78 mm THK, BS012, BLK VITON	FM100231
O-ring, 5.28 mm ID × 1.78 mm THK, BS009, BLK VITON	5711020
PEEK tube insert	FM102591
Screw, M3 × 8, cap head, stainless steel (10 each)	N/A
Screw, M3 × 10, cap head, stainless steel	FM103046
Capillary retaining nut	FM102590

Figure 83. ESI probe Assembly, exploded view

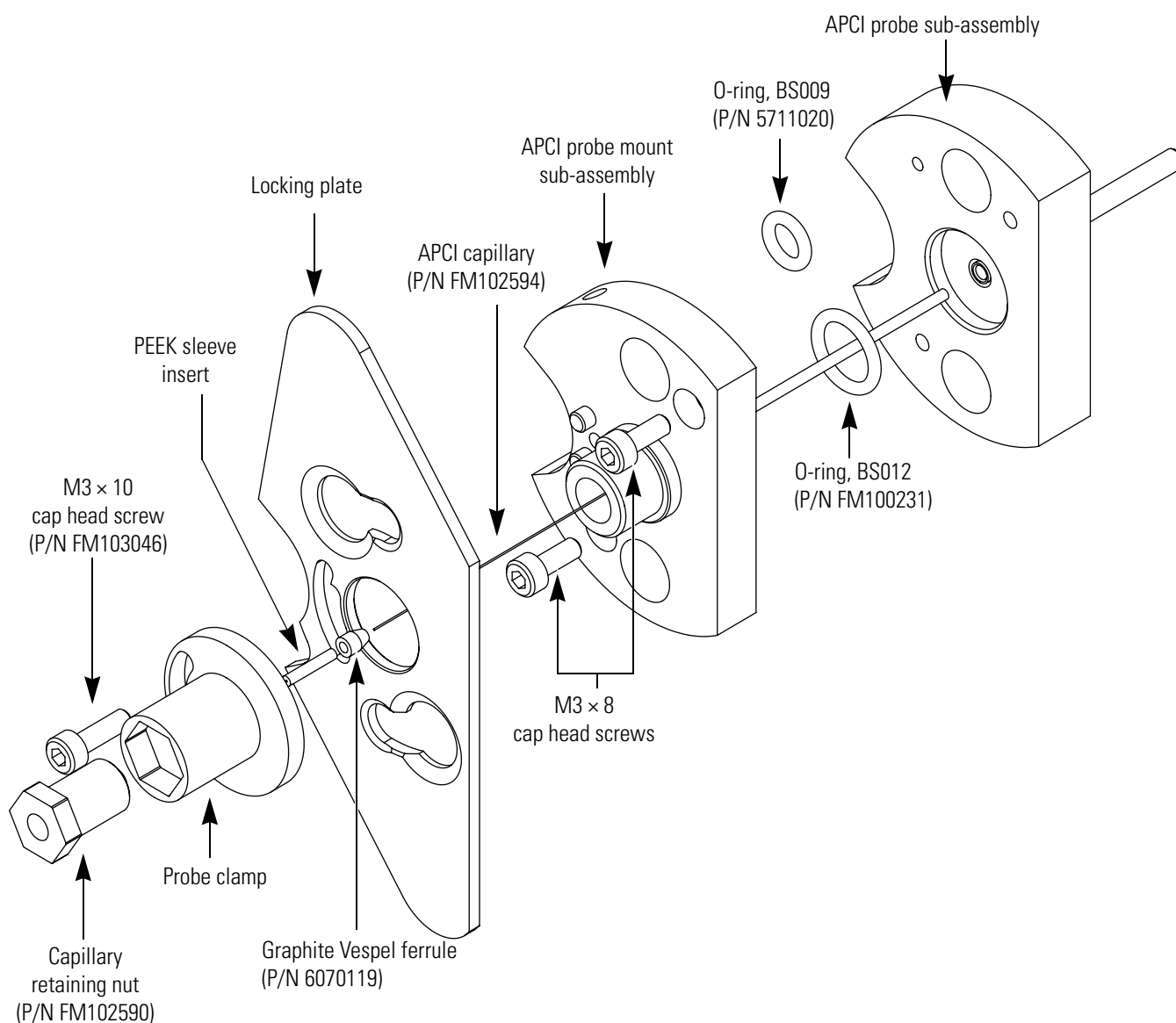


APCI Probe Assembly

An exploded view of the APCI probe assembly is shown in [Figure 84](#).

Assembly, APCI probe	FM102587
APCI capillary tube (6 each).....	FM102594
Ferrule, GVF/16, graphite vespel.....	6070119
O-ring, 9.25 mm ID × 1.78 mm THK, BS012, BLK VITON.....	FM100231
O-ring, 5.28 mm ID × 1.78 mm THK, BS009, BLK VITON.....	5711020
PEEK tube insert (12 each).....	FM102591
Screw, M3 × 8, cap head, stainless steel (10 each).....	N/A
Screw, M3 × 10, cap head, stainless steel.....	FM103046

Figure 84. APCI probe assembly, exploded view



Probe Heater Assembly

Figure 85 shows the probe heater assembly. Figure 86 shows the components of the Probe Heater Repair Kit (P/N 60111-62010).

Probe heater assembly	60111-60023
Detent screw insulator	FM102585
Spring screw	FM102583
Spring cup	FM102584
Probe screw sleeve	FM102582
O-ring, 18.77 mm ID × 1.78 mm THK, BS018, BLK VITON	5711035

Figure 85. Probe heater assembly with a view of the O-rings

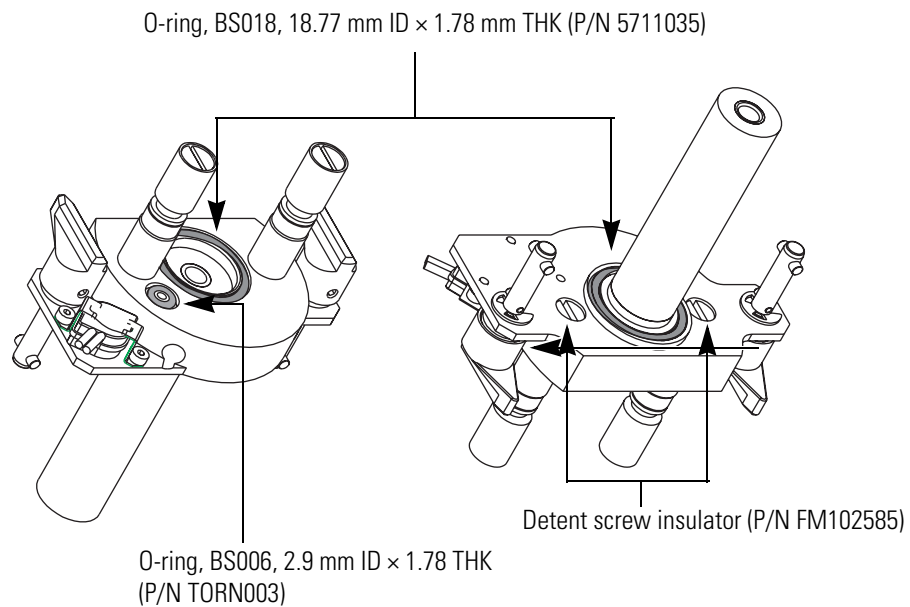
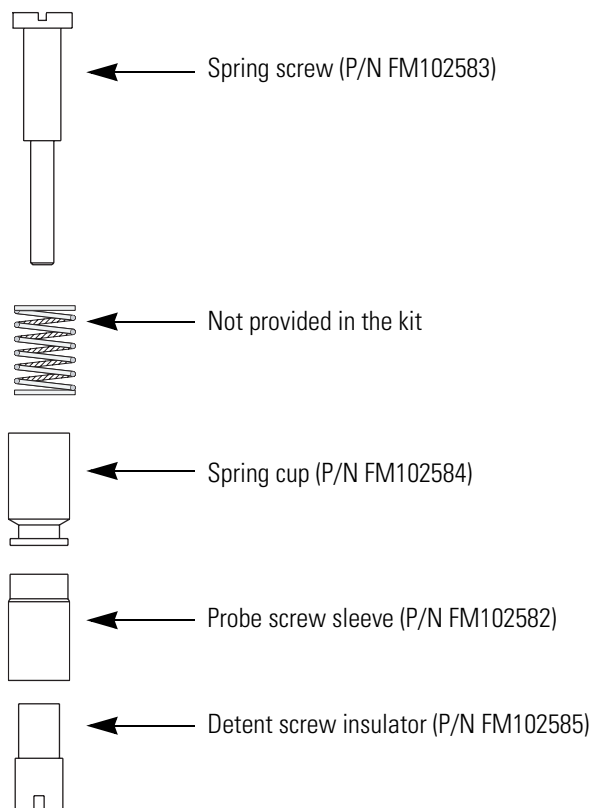


Figure 86. Components of the Probe Heater Repair Kit (P/N 60111-62010)



Vacuum Spares

Oil wick, Pfeiffer replacement	FM104398
Oil, Edwards pump, 1-L bottle	00301-15102
Seal, vacuum housing	FM101633
Tee, KF25	FM100203
Vent valve with 1 m cable assembly	FM104308
Hose, blue exhaust, 1-in. ID (order by the foot length, 10 = 10 ft.)	00301-08301

Gas Flow Spares and Nitrogen Generator

Flow controller, Legris	FM100205
Tubing, 6 mm OD, PTFE, for nitrogen line (order by the foot length)	00109-99-00004
Nitrogen valve assembly	60111-60019
PEAK nitrogen generator	OPTON-97104

Solvent Path and Calibrant Spares

Assembly, waste bottle	FM102770
Assembly, reference bottle	FM102771
Calibrant, Surveyor MSQ system (for Surveyor MSQ 1.4 and higher software)	60111-01001
Nut, PEEK, 1-piece.	FM100513
Kit, sensitivity test, Surveyor MSQ Plus system.	FM104284

Electronic Spares

Assembly, power supply, low-voltage, MSQ.	FM103098
PCB assembly, status.	60111-61090R
PCB, 2000 RF/Digital control	60111-61050S or EXFM102818
PCB, electrometer.	60111-61020
PCB, source	60111-61030
PCB, RF generator	60111-61040
PCB, digital	60111-61050
PCB, RF/dc prefilter.	60111-61080

Connection Kits

LC Devices supports liquid chromatographs manufactured by Agilent and Thermo Fisher Scientific.

To connect an Agilent liquid chromatograph to your Surveyor MSQ Plus MS detector, order the appropriate kit listed below:

Ethernet Communication Kit for Agilent 1100 LC system	OPTON-30012
JetDirect® Ethernet Control kit for Agilent 1100 LC system (contains Jet Direct card)	OPTON-30018

Manuals

The following manuals are on the instrument software CD:

- *Surveyor MSQ Plus Preinstallation Requirements Guide*
- *Surveyor MSQ Plus Getting Connected Guide*
- *Surveyor MSQ Plus Getting Started Guide*
- *Surveyor MSQ Plus Hardware Manual*

Optimizing the LC Conditions

This appendix includes helpful information for optimizing your LC system and your Cone Wash system.

Contents

- Flow Rates
- LC Solvents and Mobile Phase Additives
- Cone Wash System
- Flow Splitting
- PEEK Tubing

Flow Rates

In general, the LC column employed determines the choice of flow rate. Each column has an optimum flow rate. The guidelines in [Table 8](#) apply.

Table 8. LC columns and flow rates

Column ID	Flow Rate
4.6 mm	1.0 mL/min
3.9 mm	0.5 mL/min
2.1 mm	0.2 mL/min
1.0 mm	40-50 μ L/min
Capillary	<10 μ L/min

The different ionization modes require different flow rates and column IDs. The following guidelines apply when using the Surveyor MSQ Plus MS detector:

- Electrospray can operate at all the flow rates described in [Table 8](#).
- APCI cannot operate at flow rates below 0.2 mL/min. Therefore, suitable column IDs are 2.1 mm, 3.9 mm and 4.6 mm.

LC Solvents and Mobile Phase Additives

The choice of solvents for LC is dictated primarily by the separation requirements, but there are some guidelines that need to be followed when performing LC/MS analyses.

This section contains the following topics:

- [LC Solvents](#)
- [Mobile Phase Additives](#)

LC Solvents

The solvents that are the most compatible with the Surveyor MS detector are:

- Water
- Acetonitrile
- Methanol

These common reverse phase LC solvents are ideal for LC/MS. When using high percentages of water, the probe temperature usually needs to be raised to aid desolvation.

Solvents that are less commonly used include:

- Normal phase solvents
- Alcohols such as isopropanol, 2-methoxyethanol, ethanol, and so on
- Dimethyl sulfoxide (DMSO)

Normal phase solvents such as dichloromethane, hexane and toluene are most suitable for use in APCI. Alcohols have all been used with LC/MS but their use tends to be application-specific. DMSO is commonly used by synthetic chemists for primary dilutions.

Mobile Phase Additives

Additives can be divided into three categories:

- [Commonly Used Compatible Additives](#)
- [Less Commonly Used Additives](#)
- [Unsuitable Additives](#)

[Table 9](#) contains a summary of suitable additives. [Table 10](#) contains a summary of additives to avoid.

Table 9. Summary of suitable additives

Ion Polarity Mode	Additive
Positive ion	<ul style="list-style-type: none"> Acetic acid Formic acid Ammonium acetate (<0.1M)
Negative ion	<ul style="list-style-type: none"> Triethylamine (TEA) Ammonium hydroxide (ammonia solution) Ammonium acetate (<0.1M)

Table 10. Summary of additives to avoid

Ion Polarity Mode	Additive
Positive ion	<ul style="list-style-type: none"> Surfactants Trifluoroacetic acid (TFA) (>0.1% v/v)
Negative ion	<ul style="list-style-type: none"> Surfactants Organic acids such as acetic acid, formic acid, trifluoroacetic acid (TFA).

Commonly Used Compatible Additives

The additives that are the most compatible with the Surveyor MS detector are:

- Acetic acid or formic acid
- Ammonium hydroxide
- Ammonium acetate or ammonium formate
- Non-volatile salts
- Ion pairing agents

LC separations can be enhanced by reducing the pH of the mobile phase. Suitable additives for this are acetic acid or formic acid. (Formic acid is stronger than acetic acid and therefore less needs to be added to reach a required pH.) Addition of acids can suppress ionization in negative ion analysis and weakly acidic compounds might not form $[M-H]^-$ ions in acidic conditions.

Ammonium hydroxide (ammonia solution) is suitable for increasing the pH of the mobile phase, which can enhance LC separations. When analyzing weakly acidic compounds, in negative ion mode, it is unlikely that there will be any suppression of ionization.

Volatile salts such as ammonium acetate or ammonium formate are often used to buffer mobile phases. Use as little ammonium acetate or ammonium formate as possible, keeping the concentration below 100 mM. Ensure that the cone wash is running when using high concentrations.

When using non-volatile salts, ensure that the cone wash is running as they can crystallize in the source, block the entrance cone, and prevent the mass spectrometer from functioning. The most common non-volatile salts used are phosphates.

Ensure that the cone wash is running when using ion pairing agents (for example, sodium octanesulfonic acid). Many ion pairing agents suppress electrospray ionization.

Less Commonly Used Additives

Additives that are less commonly used include:

- Trifluoroacetic acid (TFA)
- Triethylamine (TEA)
- Tetrahydrofuran (THF)
- Inorganic acids

Trifluoroacetic acid (TFA) is frequently used for peptide and protein analysis. High levels, >0.1% v/v, can cause suppression of sensitivity in positive ion mode. TFA might completely suppress ionization in negative ion mode.

Triethylamine (TEA) can suppress the ionization of less basic compounds in positive ion mode (as it also is readily ionized to give a $[M+H]^+$ ion at m/z 102). TEA enhances ionization of other compounds in negative ion mode because it is basic. This is a particularly useful additive for the analysis of nucleic acids.

In ESI, use of THF can reduce sensitivity. This effect can be counteracted by post-column addition of ammonium acetate. It has no effect in APCI.



CAUTION Do not use a concentration of THF greater than 5% with PEEK tubing. THF causes swelling in the PEEK tubing and consequently presents a risk of the LC tubing bursting.

Inorganic acids (for example, sulfuric acid or phosphoric acid) can be used. Check the suitability of the LC column to low pHs.



CAUTION After using phosphoric acid, thoroughly clean the source, source enclosure and hexapole RF lens to minimize the physical damage.

Unsuitable Additives

Unsuitable additives include:

- Surface-active agents
- Detergents

Surface-active agents and detergents can suppress the ionization of other compounds. Detergents, by their very nature are concentrated at the surface of a liquid. This causes problems with electrospray as the ionization relies on the evaporation of ions from the surface of a droplet. The detergent therefore suppresses the evaporation of other ions. Use surfactants only when they are being analyzed themselves, not as additives to HPLC mobile phases.

Cone Wash System

Historically, LC/MS has only been compatible with volatile buffer systems using modifiers such as trifluoroacetic acid, formic acid and acetic acid. Phosphate buffers, although extensively used in LC separations, were not suited to LC/MS due to the rapid blocking of the ion sampling region caused by the deposition of non-volatile phosphate salts. The self-cleaning API source (provided by the cone wash system) of the Surveyor MSQ Plus MS detector allows routine LC/MS with chromatographic buffers such as phosphates or ion pairing agents and samples in dirty matrices.

The cone wash system consists of a cone wash nozzle, internal tubing, and a cone wash pump. Refer to your *Surveyor MSQ Plus Getting Connected* manual for instructions on connecting the cone wash pump to the Surveyor MSQ Plus MS detector. The recommended flow rate for the cone wash solvent is 200 µl/min and the recommended cone wash solvent is [50:50] methanol / water (v/v).

Note It is necessary to use the cone wash only for dirty matrices or with non-volatile buffers. Choose the cone wash solvent to give the most effective solubility for the expected contaminants.

❖ To optimize the position of the cone wash nozzle:

1. Turn the cone wash nozzle counterclockwise until the tip of the nozzle is just above the top of the entrance cone.
2. Turn the cone wash pump on by turning its On/Off switch to On.
3. Adjust the nozzle so that the drops of solvent just touch the tip of the entrance cone as they fall to the drain at the bottom of the Source chamber.



CAUTION Do not leave the cone wash running when the source heater is turned off as this can lead to cone wash solvents condensing on the RF/dc prefilter.

Flow Splitting

Because the Surveyor MSQ Plus MS detector can handle flow rates up to 2 mL/min, flow splitting of the LC eluent is not usually required. However, if hyphenated detection (using both a UV detector and an MS detector) is required, flow splitting can be achieved by using a zero dead volume Tee fitting. Eliminating the flowcell of the UV detector from the solvent path to the MS detector minimizes the peak broadening for the chromatograms produced by the MS detector.

The split ratio between the flow going to the UV detector and the flow going to the MS detector is determined by the relative backpressure in the two lines. If the backpressure exerted by the connection to the API source probe is greater than the backpressure exerted by the connection to the UV detector, the flow to the API source probe is lower than the flow to the UV detector.

PEEK Tubing

PEEK (Poly-Ether-Ether-Ketone) tubing is a widely used alternative to stainless steel tubing in the high-pressure parts of the system. It is compatible with most LC solvents except THF (tetrahydrofuran), methylene chloride, and concentrated nitric acid. It works well to a reasonably high pressure, is easy to cut and route, and is less expensive than stainless steel.

PEEK tubing comes in eight different internal diameters that are color-coded. The tubing comes in solid colors or in natural with a color-coded stripe on its external surface. The inner diameters and internal volume of five of the most commonly used colors are listed in [Table 11](#).

Table 11. 1/16-in. OD PEEK tubing color coding

Color	Inner Diameter (in.)	(mm)	Internal Volume (μL/in.)
Green	0.030	0.75	11.577
Orange	0.020	0.50	5.146
Blue	0.010	0.25	1.288
Yellow	0.007	0.18	0.632
Red	0.005	0.13	0.323

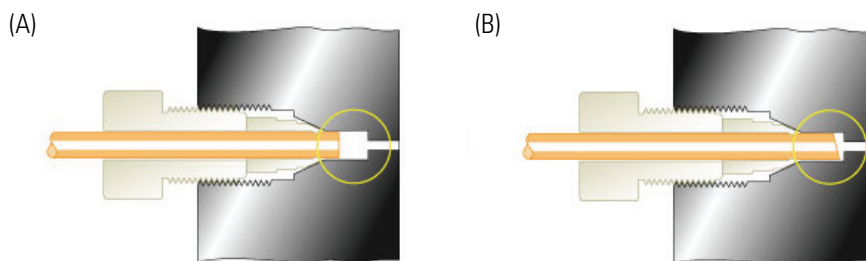
❖ **To plumb your system with PEEK tubing:**

1. Cut PEEK tubing with a polymeric tubing cutter to ensure a square cut, to prevent distortion of the tubing, and to avoid creating burrs that will constrict flow. Thermo Fisher Scientific recommends a polymeric tubing cutter that is engineered with guide holes for 1/16-in. and 1/8-in. OD tubing. The following instructions apply to the Upchurch Scientific Model A18 Polymeric Tubing Cutter and are provided courtesy of Upchurch Scientific:
 - a. Squeeze the tabs at the back of the cutter together to raise the blade.
 - b. Insert your tubing through the appropriate guide hole.
 - c. Release the tabs, allowing the blade of the cutter to rest on the tubing.
 - d. Holding the tubing, spin the cutter around the tubing to begin the cut. For PEEK tubing, spin the cutter two to three times, then remove the tubing and snap the tubing at the cut.
2. Slip a fitting over the end of the tubing.
3. As you insert PEEK tubing into a port, ensure that the end of the tubing makes contact with the bottom of the port. Then, tighten the fitting finger-tight. See [Figure 87](#).

Note

1. Tubing that is not properly seated can add dead volume to a chromatographic system.
2. Never over-tighten PEEK fittings as this can cause leaks.

Figure 87. Poor connections result if tubing is not bottomed in the port (A) or is not cut square (B)



Courtesy of Rheodyne website

Index

A

acetic acid 121
 acetonitrile 120
 acidic compounds 4
 ammonium acetate 121
 ammonium formate 121
 ammonium hydroxide 121
 analyzer region vacuum manifold description 32
 anode electron multiplier description 29
 atmospheric pressure chemical ionization (APCI)
 described 6
 probe removal 67
 atmospheric pressure ionization source (API) 23
 autosamplers 15

B

backing manifold 21
 backing pump, See forepump 32
 Balzers-Pfeiffer turbomolecular pump 32
 basic compounds 4
 buffers for mobile phase solvents 121

C

calibrant bottle 16
 capillaries
 ESI insert capillary 23
 flushing 48
 flushing (Note) 48
 replacing 56, 67
 carboxylic acid 3
 cathode electron multiplier description 29
 CAUTIONS

 APCI probe removal 67
 cleaning the entrance cone 80, 85
 disassembling the source block assembly 88
 ESI probe removal 56
 forepump power cord 33
 removing the heater 73

 removing the source block 79
 Cautions
 phosphoric acid 122
 tetrahydrofuran, THF 122
 changing ionization mode
 APCI to ESI 53
 ESI to APCI 51
 cleaning
 entrance cone 80
 flushing capillaries 48
 communications icon 43
 computer features 38
 cone wash
 described 36
 setup 123
 solvent (Note) 123
 configuration of the Xcalibur data system 15
 consumables 108
 contact closure 21
 conversion dynode voltages 28

D

daily system checks
 disk space 46
 nitrogen supply 45
 oil level in oil mist filter 46
 solvent waste from autosampler 49
 solvent waste from MS detector 49
 data 38
 data system
 chromatography 38
 Xcalibur 38
 data types 8
 dead volume 125
 devices controlled by Xcalibur 2.0 15
 dimethyl sulphoxide 120
 disassembling the RF/dc prefilter 88
 disk space, checking 46
 dynode 28

E

- electron multiplier
 - anode, description 29
 - description 29
 - voltages 29
- electrospray ionization (ESI)
 - described 3
 - probe removal 57
 - probe removal, hot parts (CAUTION) 56
 - rules for good electrospray 5
- emergency shutdown procedure 95
- entrance cone, cleaning 80
- exhaust fumes, trapping 46, 48
- exhaust manifold 21

F

- Figures
 - APCI capillary 70
 - APCI mechanism 6
 - APCI probe 25
 - block diagram of LC/MS system 14
 - cleaning the transfer lens 87
 - connection between MS and LC 23
 - desktop setup of LC/MS system 13
 - ESI capillary 64
 - ESI mechanism 4
 - ESI probe 24
 - exploded view of APCI probe 69, 115
 - exploded view of ESI probe 61, 114
 - exploded view of source block assembly 113
 - Info view - Status page 98
 - inlet gas hardware 33
 - ion detector 30
 - oil level in forepump 93
 - on/off switch for MS detector 96
 - poor tubing connections 125
 - Pump Out connection to forepump 96
 - rear panel of MS detector 22
 - reference inlet system
 - in inject position and load position 18
 - Reset button 105
 - Server icon 39
 - source block assembly 86
 - transfer lens 26
 - Tune window 42
 - USB connection 105
 - User I/O 105
 - vacuum system 31
 - Xcalibur - Home Page - Roadmap view 39
- firmware
 - downloading the Surveyor MSQ firmware 21
 - versions for LC devices controlled by Xcalibur 38

- flow rates
 - columns 119
 - ESI mode 23
- flow splitting procedure 124
- forepump 32
 - checking the oil color 93
 - checking the oil level in the forepump 93
 - description 32
 - maintenance 93
 - maintenance (Note) 93
 - power cord (caution) 33
 - purging the oil of volatile contaminants 49
- formic acid 121
- full scan type, defined 8
- Full System Autotune 17
- functional description
 - autosampler 15
 - MSQ MS detector 13, 19

G

- Gas In port 22
- gases, sheath and nebulizing 23

H

- hard drive, checking disk space 46
- heater removal, hot parts (Warning) 73
- Home Page 38
- HPLC
 - devices controlled by Xcalibur 15
 - flow rates 119
 - use of solvents and additives with MS detector 120

I

- inlet gasses hardware
 - description 33
 - sheath gas valve 33
- inorganic acids 122
- ion detection system
 - conversion dynode 28
 - electron multiplier gain 30
 - electron multiplier, description 29
- ion pairing agents 121
- ion polarity modes
 - choosing positive or negative 5, 7
- ionization mode, changing 51
- ionization techniques
 - atmospheric pressure chemical ionization (APCI) 6
 - electrospray ionization (ESI) 3

K

kits, consumables 108

L

LC devices controlled by Xcalibur 2.0 15

LC/MS

connection between LC and MS detector 23

flow rates 119

leaks, causes of 125

M

maintenance schedule, description 55

manifolds on rear panel 21

manual tuning for specific analytes 40

mechanical pump, See forepump 32

methanol 120

mobile phase additives 120

modes, ion polarity 2

molecules

deprotonated 4

protonated 4

M-path region, description 32

MSQ MS detector

API source 23

emergency shutdown 95

functional description 13, 19

inlet gasses hardware 33

overview 1

resetting 104

scan data types 8, 8

turbomolecular pump 32

vacuum manifold 32

vacuum system 31

vent valve 33

musical note icon 43

N

nebulizing gas 23

Nitrogen Gas button 97

nitrogen regulator, auxiliary 34

nitrogen supply

checking 45

pressure 45

rate of consumption 45

uses 25

non-volatile salts 121

normal phase solvents 120

Notes

cone wash solvent 123

entering the needle voltage or corona current into a method 41

flushing the capillaries 48

forepump pump maintenance 93

O

oil

checking oil level in the forepump 93

checking the color of the oil in the forepump 93

purging volatile contaminants from the forepump oil 49

oil mist filter, checking the oil level 46

Operate button 100

Operate, status 39

P

part categories 107

parts kit, consumables 108

parts lists 107

PEEK tubing

chemical compatibility 124

color coding 124

connections for cone wash system 36

cutting 125

making connections 125

personal computer features 38

phosphates 121

phosphoric acid, use (Caution) 122

polymeric tubing cutter 125

power cord of forepump (caution) 33

printer, setting up and connecting 44

probe disassembly

APCI 68

ESI 58

probe removal

APCI 67

ESI 57

profile scan, defined 9

Pump Out connection to forepump 21

pumps

cone wash 36

forepump 32

LC pumps 15

turbomolecular pump 32

purity requirements of solvents xv

Q

quadrupoles

RF and dc fields 26

RF/dc prefilter 26

R

raw files 40
 reference inlet system 16
 restarting the system 101
 reverse phase solvents 120
 RF/dc prefilter
 assembling 90
 description 26
 disassembly 88
 rod assemblies for quadrupoles 26
 roughing pump, See forepump 32

S

Safety Precautions *xiv*
 disconnect MS detector from line power *xiv*
 discussion *xiv*
 fume exhaust and waste streams *xv*
 MS detector in standby before opening API source *xiv*
 nitrogen for API source *xiv*
 no unauthorized servicing *xiv*
 respect heated zones *xiv*
 shut down MS detector *xiv*
 sample 19
 sample loop attached to microinjection valve 17
 scan data types
 defined 8
 profile scan 9
 scan types
 defined 8
 full scan 8
 selected ion monitoring (SIM) 8
 schedule, maintenance 55
 selected ion monitoring (SIM) 8
 Server
 defined 39
 Operate status 39
 vacuum status 39
 sheath gas
 defined 23, 25
 pressure 33
 shutdown emergency procedure 95
 shutting down the system 100
 singly charged ions 4, 7
 software, Xcalibur 38
 solvent waste
 from autosampler 49
 from MS detector 49
 solvents
 compatibility with PEEK tubing 124
 for liquid chromatography 120
 solvents, purity requirements *xv*

source block
 allow to cool (CAUTION) 88
 removing from the MS detector 85
 replacing 91
 source manifold 21
 spares 111
 spinning musical note icon 43
 sulfuric acid 122
 system shutdown emergency procedure 95

T

Tables
 devices controlled by Xcalibur 1.4 15
 PEEK tubing 124
 Status LED states 20
 Xcalibur software application programs 38
 technique 3
 tetrahydrofuran, THF, use (Caution) 122
 triethylamine, TEA 122
 trifluoroacetic acid, TFA 122
 tubing
 cutting PEEK 125
 PEEK color coding (table) 124
 poor connections 125
 seating in port 125
 tune files 40
 Tune window
 described 41
 figure 42
 launching 41, 96
 turning off the MS detector from 99
 turbomolecular pump, description 32

U

USB ports
 data system computer 44
 on rear panel of MS detector 21
 User I/O 21

V

vacuum manifold, description 32
 vacuum pumps
 forepump 32
 turbomolecular pump 32
 vacuum status 39
 vacuum system
 description 31
 forepump 32
 turbomolecular pump 32
 vent valve 33

vent valve description [33](#)
venting the MS detector [100](#)
volatile contaminants, purging from forepump oil [49](#)
voltages
 conversion dynode [28](#)
 electron multiplier (cathode) [29](#)

W

waste bottle for reference inlet system [17](#)
waste solvent, disposing [49](#)
water [120](#)
water vapor, purging from forepump oil [49](#)

X

Xcalibur
 devices controlled by [15](#)
 Home Page [38](#)
 Instrument Configuration dialog box [15](#)
 introduction [38](#)
 launching [38](#)
 Server [39](#)
 turning off the MS detector from [98](#)

