KNAUER

PLATINblue MSQ Plus

Hardware Guide

V6950A

UHPLC/HPLC

Mass spectrometry

Content

Preface

This *MSQ Plus Mass Detector Hardware Manual* describes the operational modes and principal hardware components of the MSQ™ Plus Mass Detector. It also provides step-by-step instructions for cleaning and maintaining the MSQ Plus Mass Detector.

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

Safety

Fuses

If the fuses blow repeatedly, consult with KNAUER Technical Support for replacements and help in identifying the cause.

Lab regulations

Observe the laboratory regulations

- ▶ Observe national and international regulations on laboratory work!
- Good Laboratory Practice (GLP) of the American Food & Drug Administration
- For development of methods and validation of modules: Protocol for the Adoption of Analytical Methods in the Clinical Chemistry Laboratory, American Journal of Medical Technology, 44, 1, pages 30–37 (1978)
- On the Internet: Accident prevention regulations published by the accident insurance companies for laboratory work

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.

LC Solvents

Water, acetonitrile, and methanol are the solvents that are the most compatible with the MSQ Plus Mass Detector. These common reverse-phase LC solvents are ideal for LC/MS. When you use high percentages of water, you usually need to raise the probe temperature to aid desolvation.

Less commonly used solvents include normal-phase solvents; alcohols such as isopropanol, 2-methoxyethanol, and ethanol; and 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.

Toxicity Organic solvents are toxic above a certain concentration. Ensure that work areas are always well-ventilated! Wear protective gloves and safety glasses when working on the device!

Combustibility Organic solvents are highly flammable. Since capillaries can detach from their screw fittings and allow solvent to escape, it is prohibited to have any open flames near the system

Flammability Organic solvents are highly flammable.
Since capillaries can detach from their screw fittings and allow solvent to escape, it is prohibited to have any open flames near the analytical system!

Leaks and clogged capillaries

- ▶ Regularly check for leaks and clogged capillaries – test back pressure without column!

Protective measures

You are only permitted to perform the maintenance tasks described in this manual. All other maintenance tasks are to be performed exclusively by KNAUER or a company authorized by KNAUER.

Without exception, the following applies to all maintenance tasks that can be performed by the user: Switch off the module; pull the power plug! Never open a module! The high-voltage components in the modules pose a lethal hazard!

Safety precautions

Observe the following safety precautions when you operate or perform service on the MSQ Plus Mass Detector:

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

Shut down the mass 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 mass detector be shut down and disconnected from line power before service is performed. Do not operate the mass detector with the top or side covers off. Do not remove protective covers from PCBs.

Do not interfere with the safety interlock. Interfering with the safety interlock will expose you to potentially lethal electrical hazards.

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 mass 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 mass detector is on could be unsafe. The mass detector automatically goes into Standby when you open the API source; however, take this added precaution for safety reasons.

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 mass detector is on could be unsafe. The mass detector automatically goes into Standby when you run out of nitrogen; however, take this added precaution for safety reasons.

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 mass detector and the other to vent the forepump exhaust. As described in the *MSQ Plus Mass Detector Connection 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 mass 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 that is connected to 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 MSQ Plus Mass 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 MSQ Plus Mass Detector can detect, use distilled water.

Contacting Us

There are several ways to contact KNAUER for the information you need.

To contact Technical Support or Customer Service for ordering information

Phone:	+49-(0)30-809727-0
Fax:	+49-(0)30-8015010
email:	info@knauer.net
Internet:	www.knauer.net

To suggest changes to documentation or to Help

▶ Send an email message to the Technical Support at info@knauer.net

Introduction

The MSQ Plus Mass Detector is an advanced analytical instrument that includes a mass detector, forepump, data system, and an optional cone wash pump. Integrated with an LC system, the MSQ Plus Mass Detector provides the separation capability of an HPLC and the detection capability of a single-quadrupole mass detector. See Figure 1.

Fig. 1 MSQ Plus Mass Detector and the UHPLC 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 MSQ Plus Mass Detector.

The MSQ Plus Mass 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.

Mass detectors can detect only ionized molecules. The MSQ Plus Mass Detector provides two ionization techniques: atmospheric pressure chemical ionization (APCI) and electrospray ionization (ESI). In APCI mode, molecules ionize in the gaseous phase as

they enter the API source. In ESI mode, molecules ionize in the liquid phase before they enter the ion source. For both ionization techniques, the mass 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 mass detector can switch the charge applied to the capillary. By repelling ions of like charge towards the entrance of the mass 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 mass 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 mass 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 mass 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 MSQ Plus Mass Detector data system.

Ion Polarity Modes

You can operate the MSQ Plus Mass Detector in the following ion polarity modes: positive, negative, or positive-negative switching. The application 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 ($R\text{-}NH_2 + H^+ \rightarrow R\text{-}NH_3$)—and respond best in the positive ion polarity mode. Other compounds have functional groups that readily lose a

proton—for example, carboxylic acids ($R\text{-}CO_2H$ --> $R\text{-}CO_2^-$)—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 MSQ Plus Mass Detector in both electrospray ionization (ESI) and atmospheric pressure chemical ionization (APCI) modes.

Electrospray Ionization (ESI)

The electrospray ionization (ESI) technique transfers ions in solution into the gas phase[1].

Ion Desolvation Mechanism

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 mass detector through the entrance cone. Figure 2 shows the steps in the formation of gas phase ions from highly charged droplets.

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.

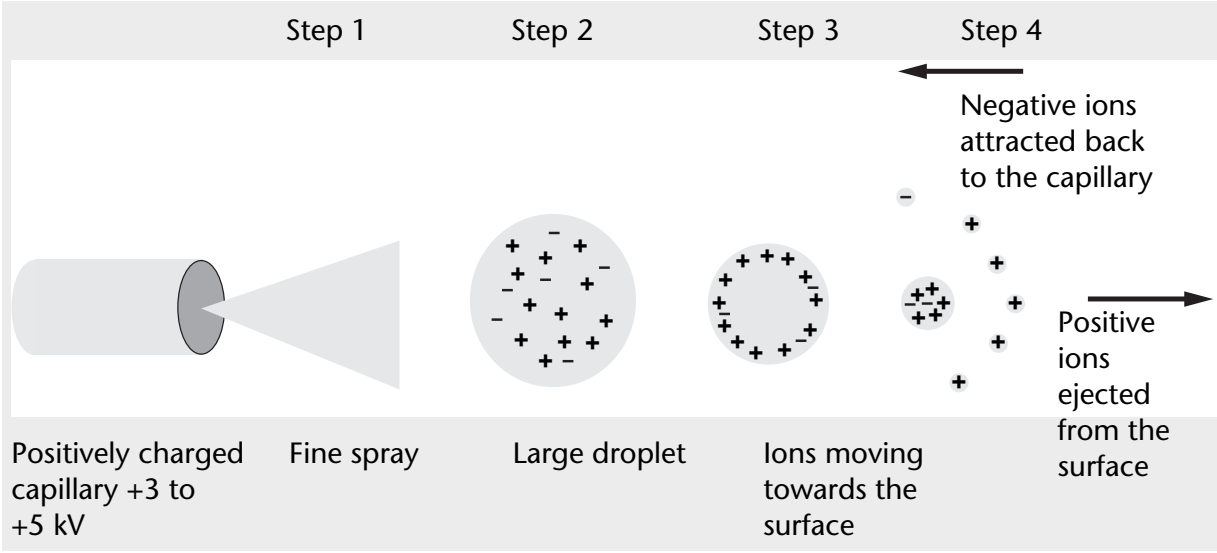


Fig. 2 Positive ion electrospray mechanism

Spectral Characteristics

In 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 100000 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 typically

consist 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 or 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.

Ion Generation Mechanism

The following sequence of events produces ions in APCI:

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 shows these four steps in APCI.

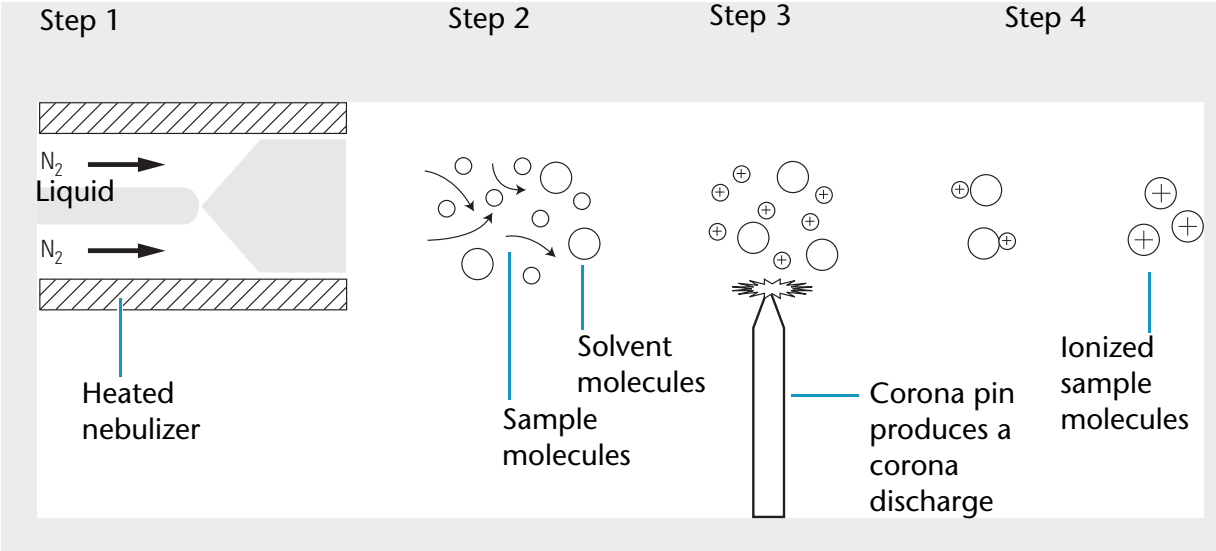


Fig. 3 Positive-ion APCI mechanism

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. Following are typical examples of primary, secondary, and adduct-ion formation.

Primary ion formation:

$e^- + N_2 \longrightarrow N_2^{+\bullet} + 2e^-$

Secondary ion formation:

$N_2^{+\bullet} + H_2O \longrightarrow N_2 + H_2O^{+\bullet}$

$H_2O^{+\bullet} + H_2O \longrightarrow H_3O^+ + HO\bullet$

Proton transfer:

$H_3O^+ + M \longrightarrow (M + H)^+ + H_2O$

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, especially 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, so it 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 MSQ Plus Mass Detector provides two scan types, full scan and selected ion monitoring (SIM).

Full Scan

A full scan provides a mass spectrum over a defined mass range. Because the mass 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. Use SIM if you need to detect small quantities of a target compound and 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. SIM monitors only specific ions. Therefore, any compound that produces those ions appears to be the target compound, resulting in false positives.

Data Types

The MSQ Plus Mass Detector provides profile, centroid, and MCA data types.

From the Xcalibur data system, you can acquire and display mass spectral data (intensity versus mass-to-charge ratio) in the profile or centroid data types (peak formats). 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, as shown in Figure 4.

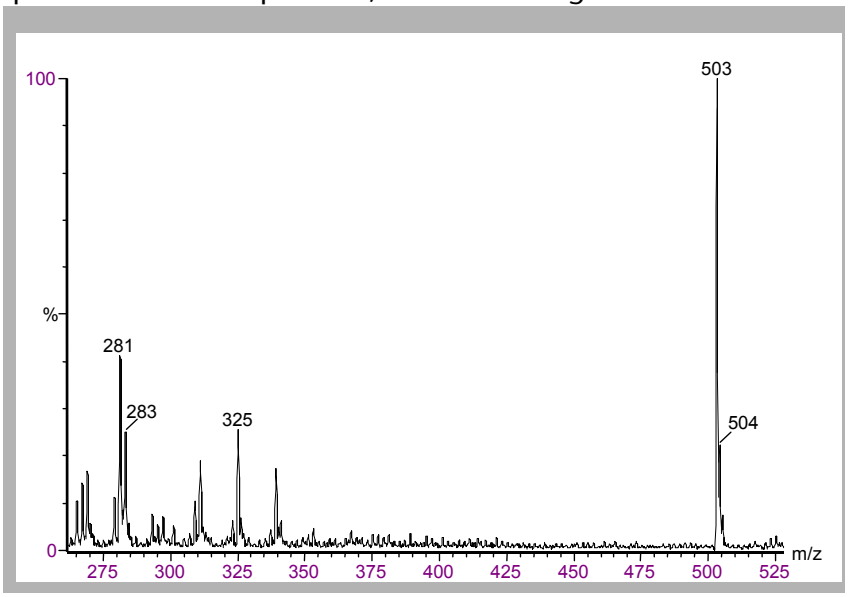


Fig. 4 Spectrum of D-raffinose shown in profile peak format in full scan

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 mass detector so that you can easily see and measure mass resolution.

Centroid Data Type

In the centroid data type, the mass spectrum appears as a bar graph, as shown in Figure 5. In this data type, the Xcalibur data system 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.

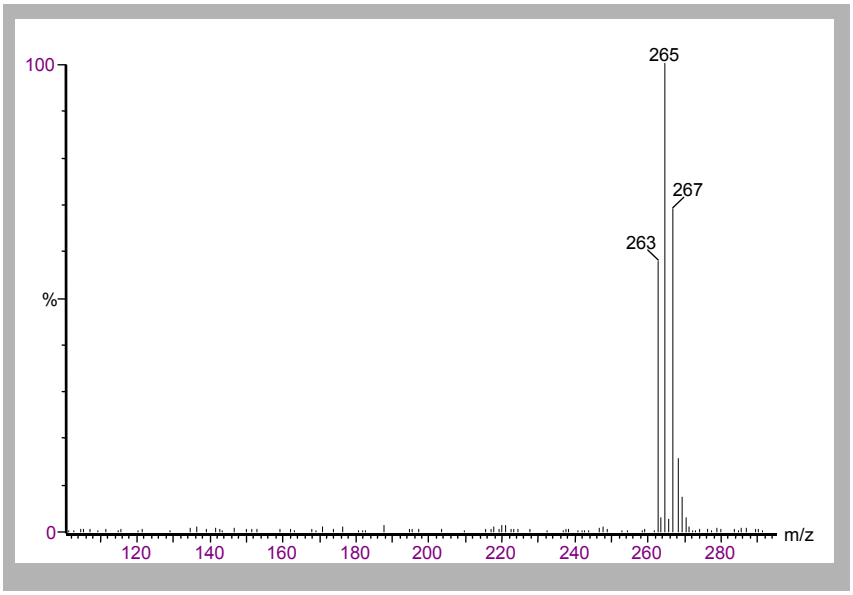


Fig. 5 Spectrum of pentachlorophenol shown as centroid peak type in full scan

MCA Data Type

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

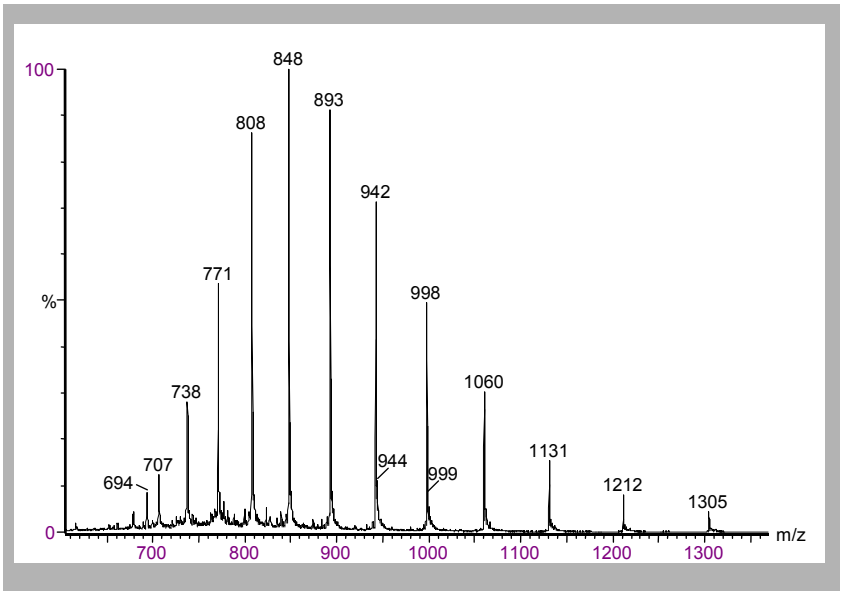


Fig. 6 Spectrum of horse heart myoglobin shown as MCA in full scan

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 the Xcalibur data system 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.

Functional Description

This chapter describes the principal components of the MSQ Plus Mass Detector and their functions.

A functional block diagram of the LC/MS integrated system with an UHPLC pump, UHPLC autosampler, and the MSQ Plus Mass Detector is shown in Figure 7.

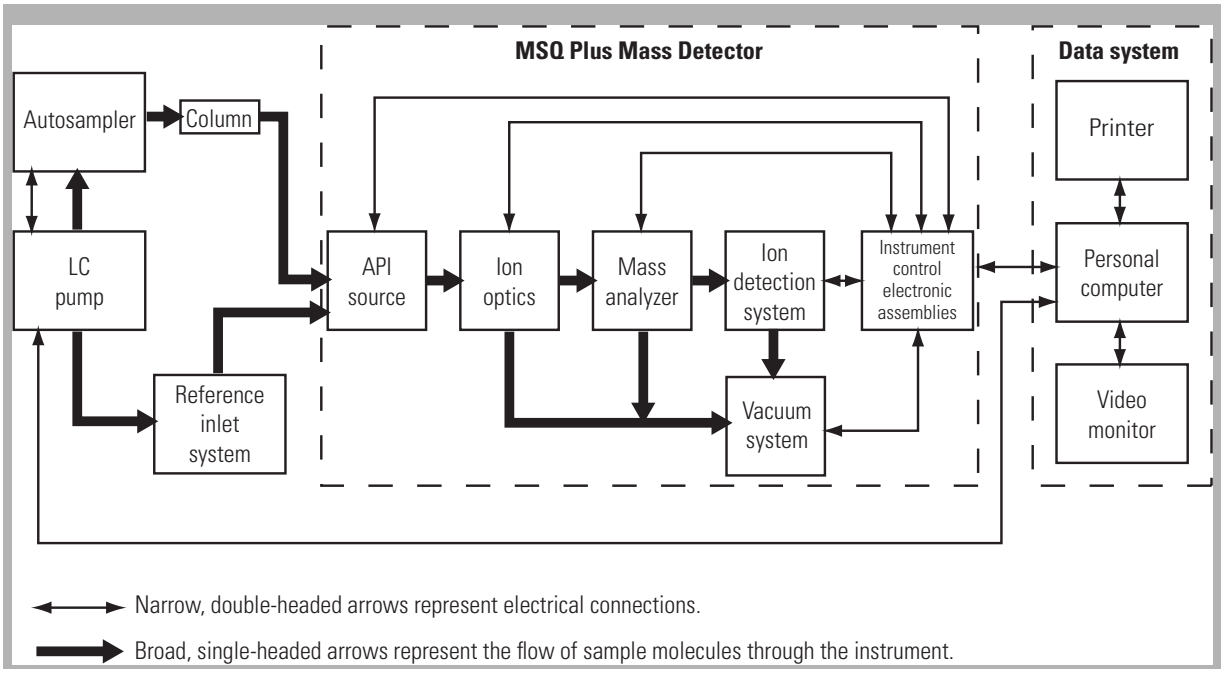


Fig. 7 Functional block diagram of the LC/MS system

A sample transfer line connects the UHPLC to the MSQ Plus Mass Detector. The UHPLC system is usually installed to the left of the MSQ Plus Mass Detector to minimize the length of tubing required to connect the outlet from the LC to the inlet of the mass spectrometer. You can also integrate liquid chromatography systems supplied by other manufacturers with the MSQ Plus Mass Detector.

An autosampler injects samples into the mobile phase stream provided by the LC pump. As the stream passes through the LC column, the sample mixture divides 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 or PDA detector, the mass detector, or both with a split flow tee. For instructions on connecting a split flow tee, see “Optimizing the LC Conditions” on page 129. When preformed sample ions enter the API source of the mass detector, they 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, an ion detection system detects them, producing an ion current signal. The system electronics receive the ion current signal, which is proportional to the number of ions in solution, and amplify it. Then they pass it on to the data system for further processing, storage, and display.

Liquid Chromatograph

The LC pump pumps the mobile phase through the LC column and into the API source. The autosampler introduces the sample into the mobile phase stream.

Contact closure provides autosampler start and stop signals to the MSQ Plus Mass Detector. See the *MSQ Plus Mass Detector Connection Guide* for information on connecting an autosampler to the MSQ Plus Mass Detector by contact closure.

Configure the Xcalibur data system for your LC devices with the Xcalibur Instrument Configuration application.

To open the Instrument Configuration application

▶ Double-click the **Instrument Configuration** icon,  , on the Windows desktop.

–or–

▶ For Xcalibur 2.0.7, choose *Start* > *Programs* > *Xcalibur* > *Instrument Configuration.*

The Instrument Configuration application appears.

–or–

For Xcalibur 2.1.*x*, choose *Start* > *Programs* >*Thermo Foundation 1.0* > *Instrument Configuration*.

The Instrument Configuration dialog box opens, as shown in Figure 8

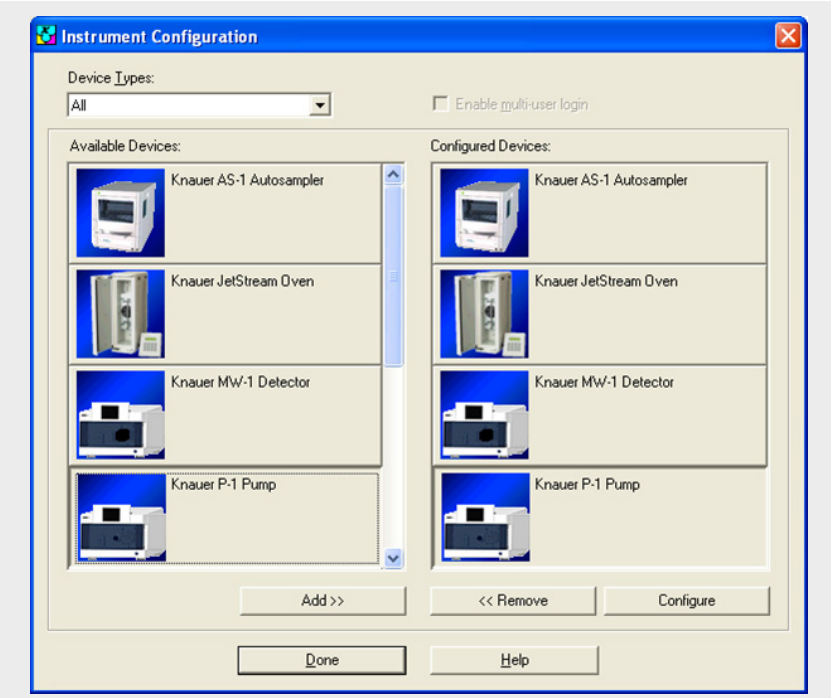


Fig. 8 Instrument Configuration dialog box

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 mass detectors.

For more information on configuring the software for LC devices, refer to the chapter in the *MSQ Plus Mass Detector Connection Guide* that pertains to LC devices or to the Help available from the Xcalibur Instrument Configuration window.

For information on controlling your LC devices from the Xcalibur data system, refer to the 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.

Reference Inlet System

Use the reference inlet system to introduce calibrant solution into the MSQ Plus Mass Detector to perform a full-system autotune or a mass-scale calibration, which is a subset of the full-system autotune procedure.

During an automated full-system autotune, the MSQ Plus Mass Detector and instrument control software perform these steps:

- The mass 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 MSQ Plus Mass Detector, a KNAUER service engineer performs a full-system autotune. You must repeat the procedure if you move the MSQ Plus Mass Detector to a new location, install or update the Xcalibur data system, or change the laboratory environment. If you notice a drift in the mass accuracy of your analyses, you should perform a mass-scale calibration.

Note: Perform a mass-scale 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 reservoir 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 µL sample loop, as shown in Figure 9. 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 10.

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.

Note: KNAUER recommends that you avoid using the reference inlet for sample introduction. This inlet is used exclusively for autotune.

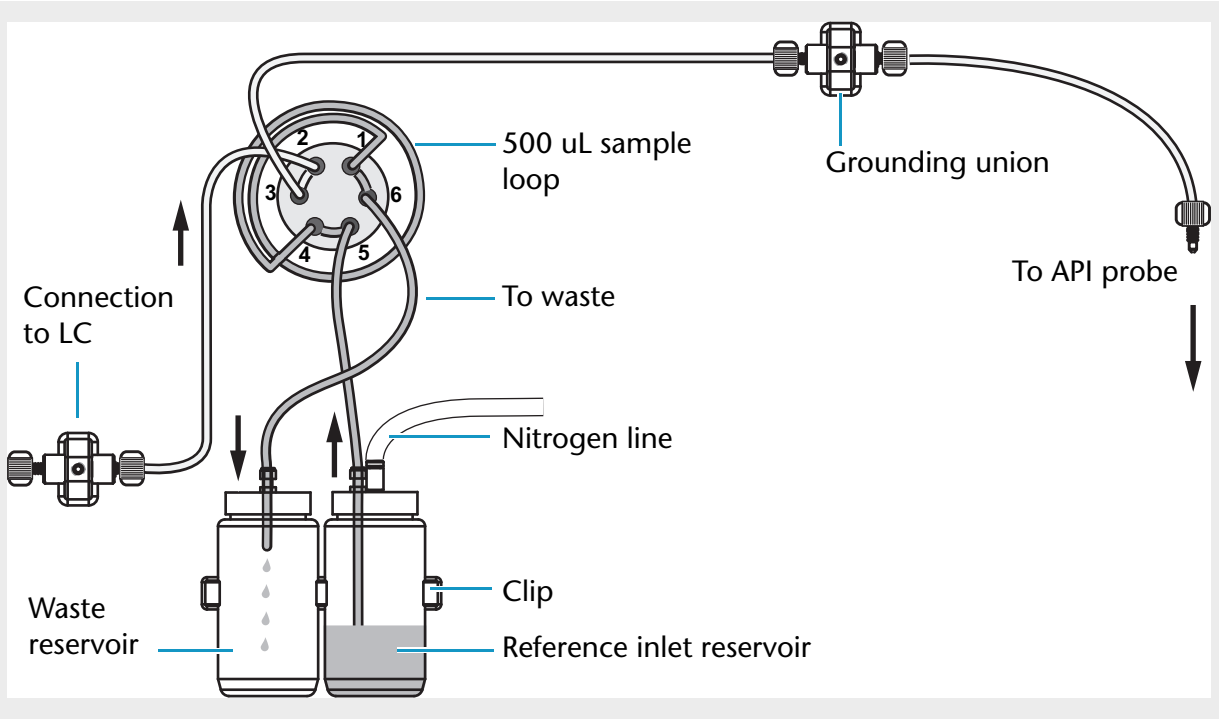


Fig. 9 Microinjection (switching) valve in the Load position

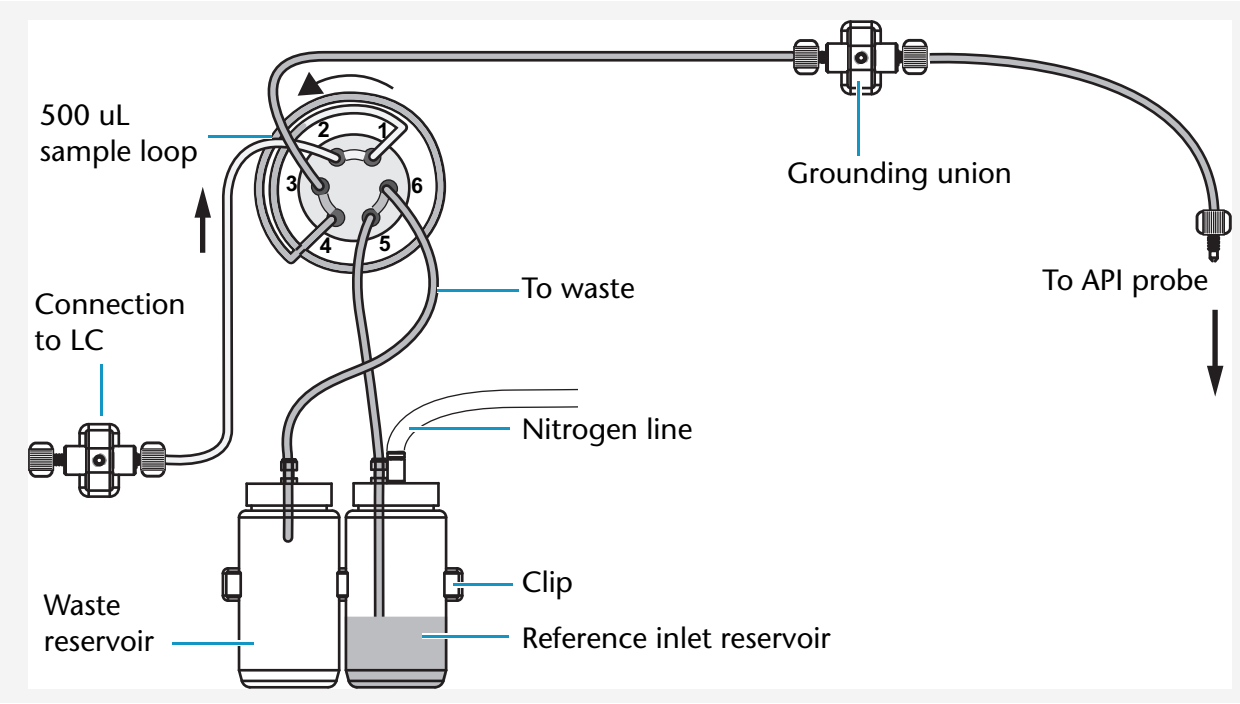


Fig. 10 Microinjection (switching) valve in the Inject position

Mass Detector

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

This section describes the following components of the MSQ Plus Mass Detector:

- Front Panel Status Indicator
- Rear Panel Controls and Connections
- Connection Between LC and Mass 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 MSQ Plus Mass Detector, as shown in Figure 11 and Table 1 lists the states of the status LED.

Fig. 11 Front view of the MSQ Plus Mass Detector

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 (MSQ Plus Mass Detector in use)	Green

Table 1. States of MSQ Plus Mass Detector Status LED

Initially, when a KNAUER service engineer installs the MSQ Plus Mass Detector and 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 two hours (for example, to perform maintenance on the source block), wait 15 minutes after pumping down the system to place the mass detector in 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 eight hours before operating the system.

Rear Panel Controls and Connections

The rear panel of the MSQ Plus Mass Detector is shown in Figure 12. 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

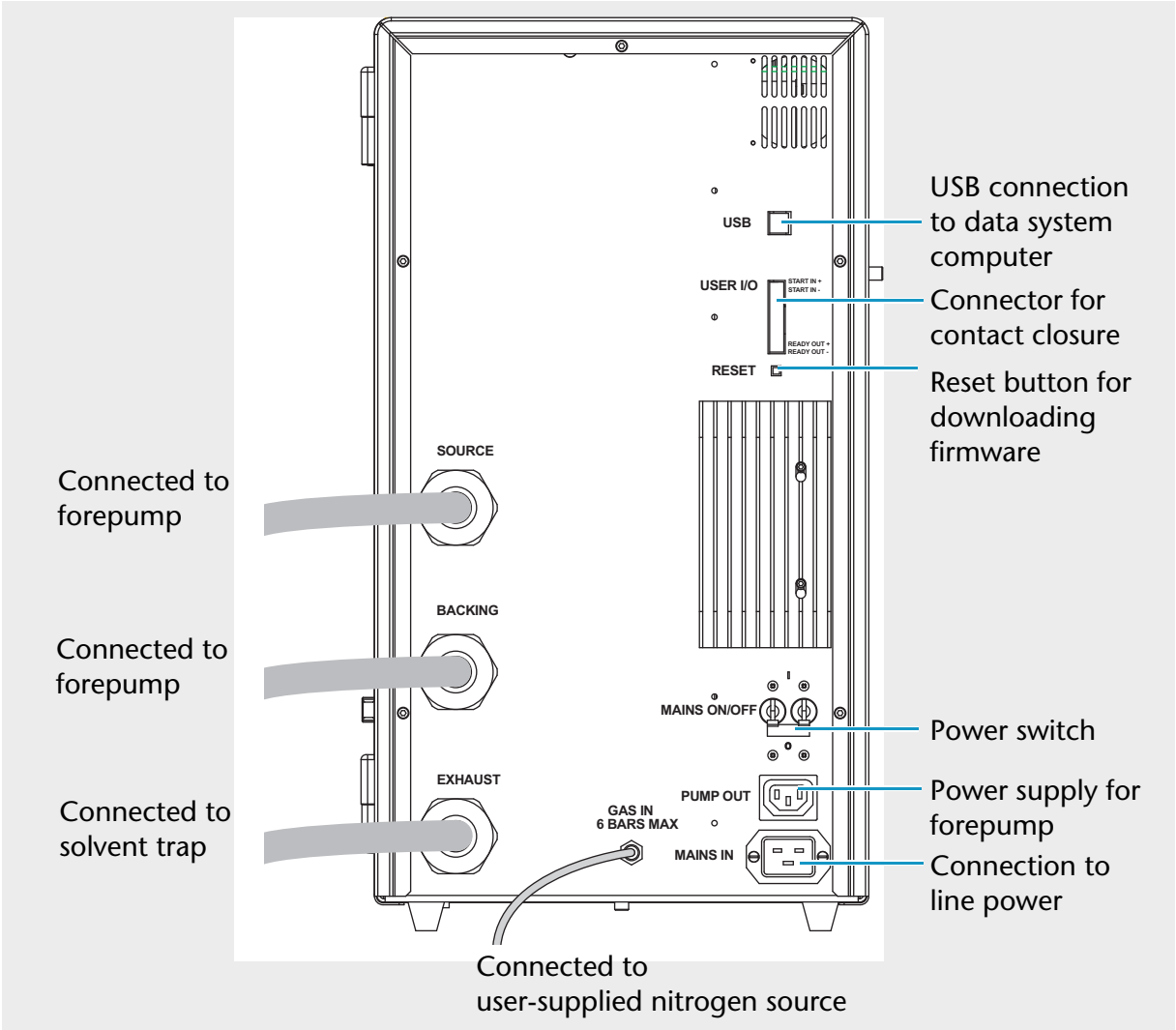


Fig. 12 Rear panel of the MSQ Plus Mass Detector

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 mass detector. In the Off (O) position, the circuit breaker removes all power to the mass detector, including the forepump (rotary vacuum pump). In the On (I) position, power is supplied to the mass 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 MSQ Plus Mass Detector are 230 Vac, regulated to ± 5% at 50 or 60 Hz. The application 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 MSQ Plus Mass 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 MSQ Plus Mass Detector from the data system and restores communications.

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 MSQ Plus Mass Detector is connected to the user-supplied nitrogen source with 6 mm OD PTFE tubing.

Connection Between LC and Mass Detector

The connection between the liquid chromatograph and the MSQ Plus Mass Detector is a PEEK union. This union is located behind the front door of the mass detector to the left of the source block cover. Figure 13 shows this connection.

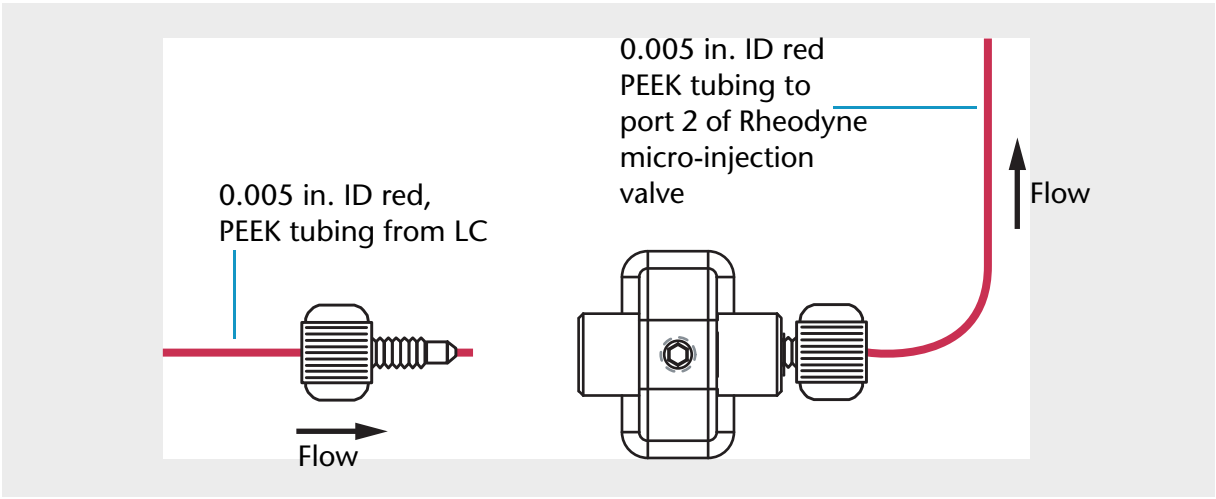


Fig. 13 Connection between LC and mass detector

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 mass 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 electrospray ionization (ESI) or 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 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.

Figure 14 shows the components used in electrospray ionization.

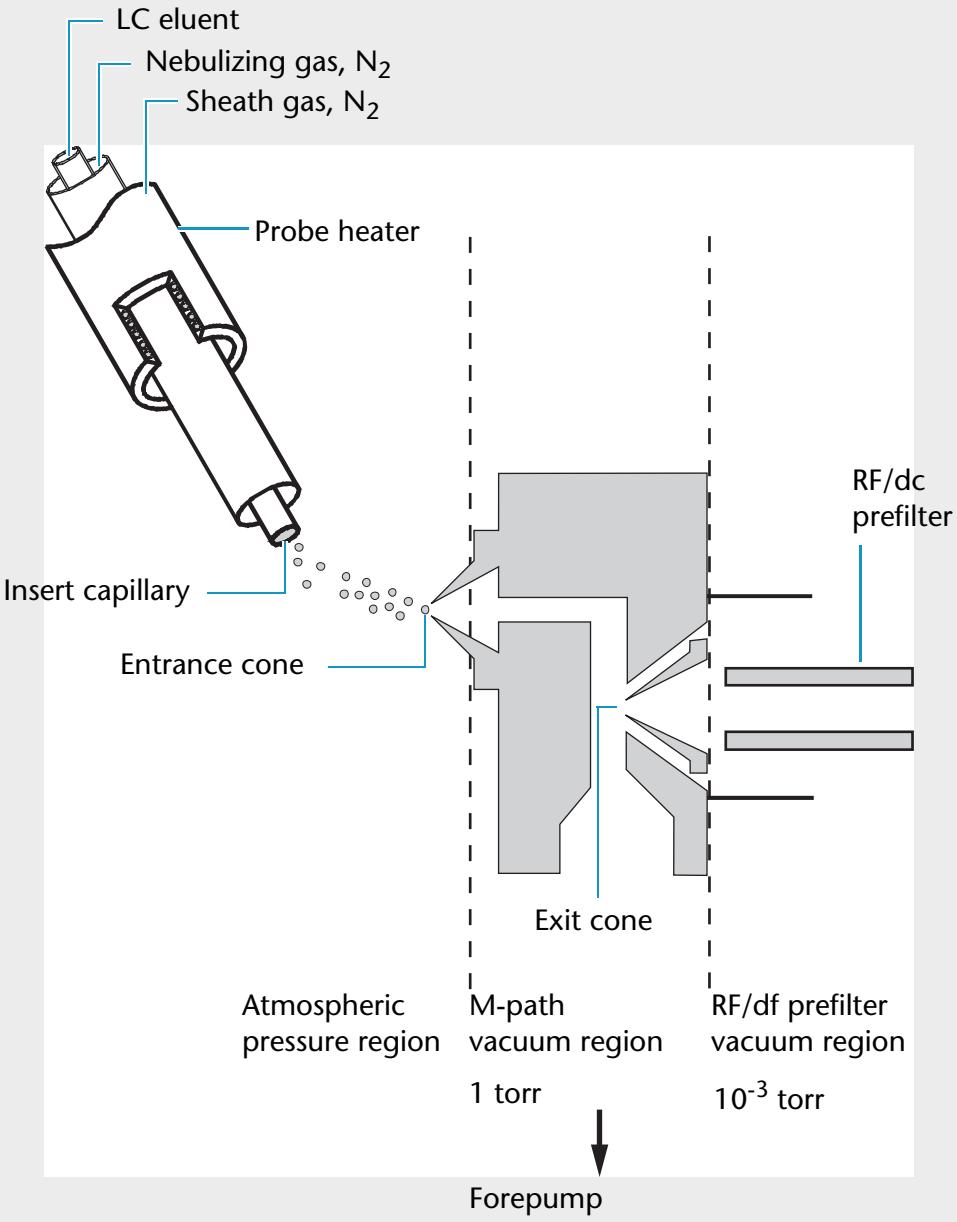


Fig. 14 Principal components and the pressure regions used by the ESI probe

Atmospheric Pressure Chemical Ionization (APCI)

In contrast to electrospray ionization, 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 15.

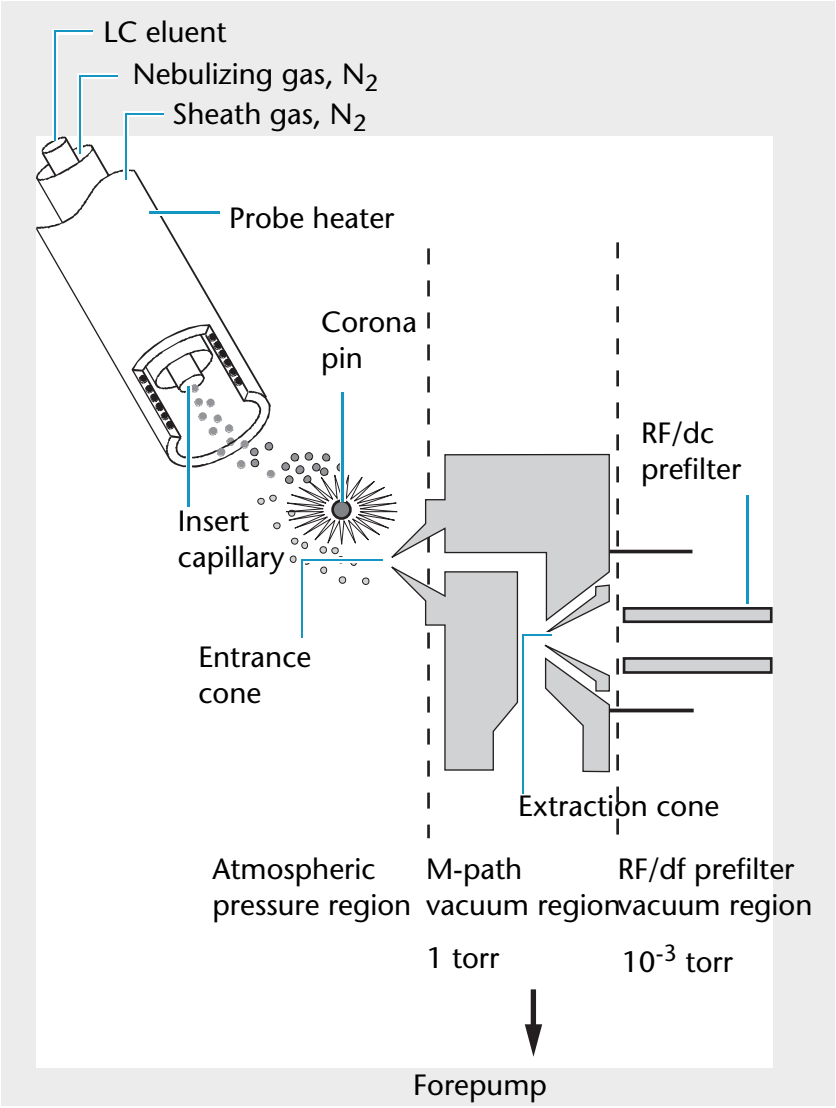


Fig. 15 Principal components and the pressure regions used by the APCI probe

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, as shown in Figure 16.

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 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.

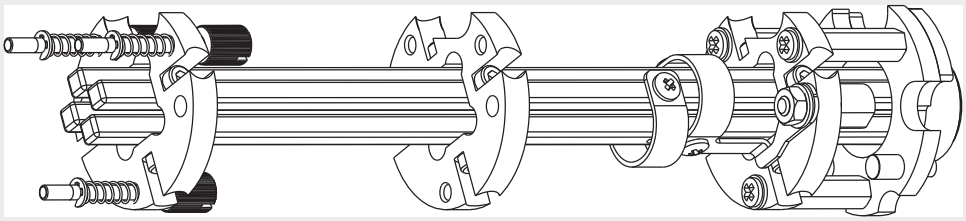


Fig. 16 RF/dc prefilter, square array of square-profile rods

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 MSQ Plus Mass Detector, the mass analyzer is a single quadrupole rod assembly.

RF and DC Fields Applied to the Quadrupoles

In a quadrupole rod assembly, rods diagonally opposite each other in the array are connected electrically, so 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 17.

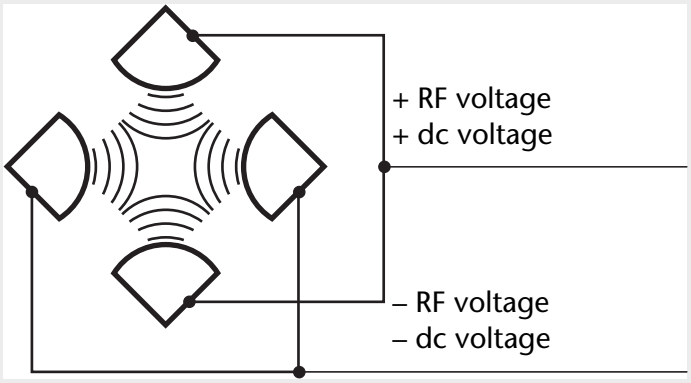


Fig. 17 Polarity of the RF and dc voltages applied to the rods of the mass analyzer

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 mass detector to distinguish between ions of different mass-to-charge ratios.

Mass Analysis

The mass analyzer in the MSQ Plus Mass 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 MSQ Plus Mass 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 18.

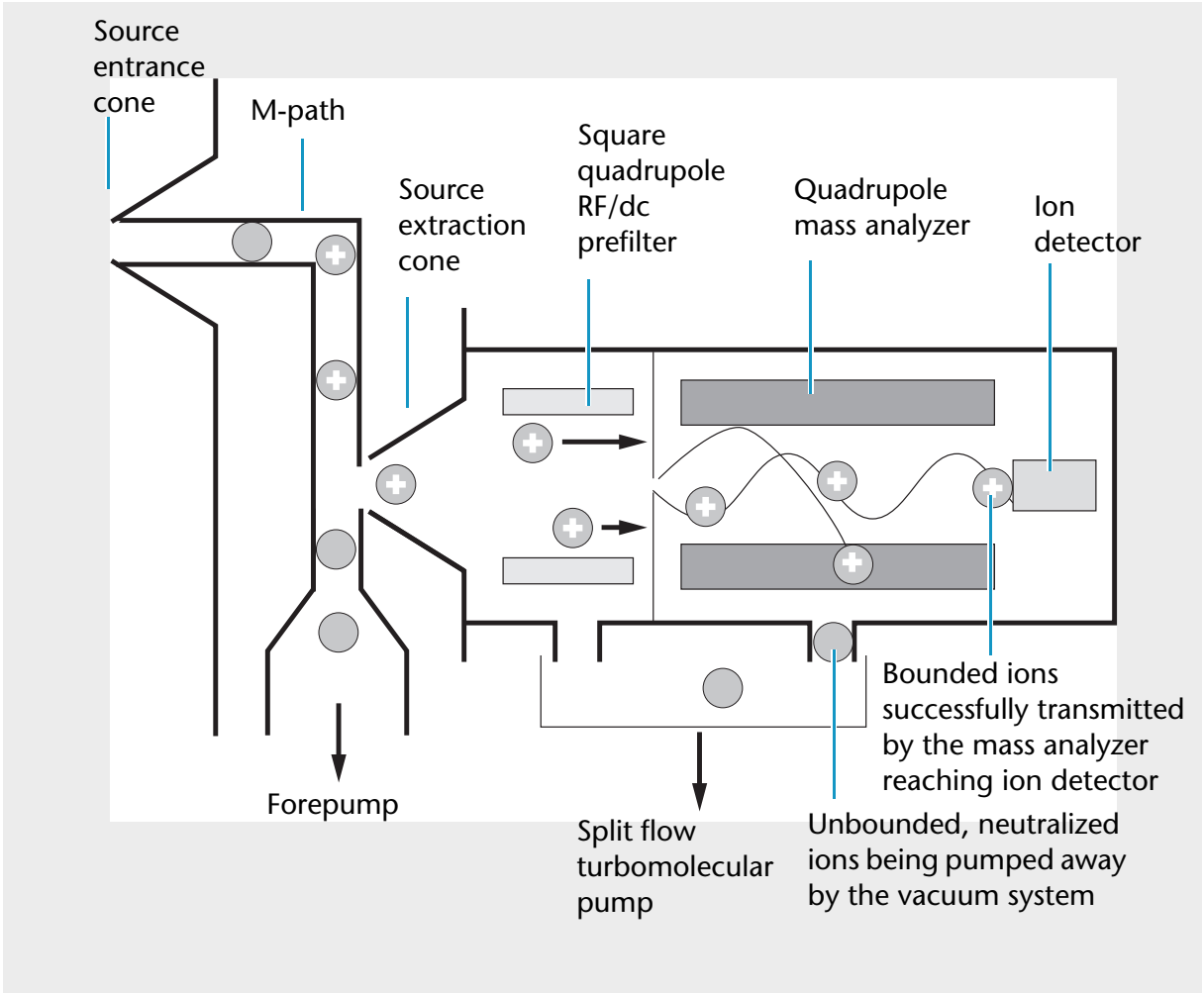


Fig. 18 Schematic of the RF/dc prefilter, mass analyzer, and ion detector

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 MSQ Plus Mass 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 seconds. 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.

Ion Detection System

The MSQ Plus Mass 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. Figure 19 shows a cross-sectional view of the ion detection system.

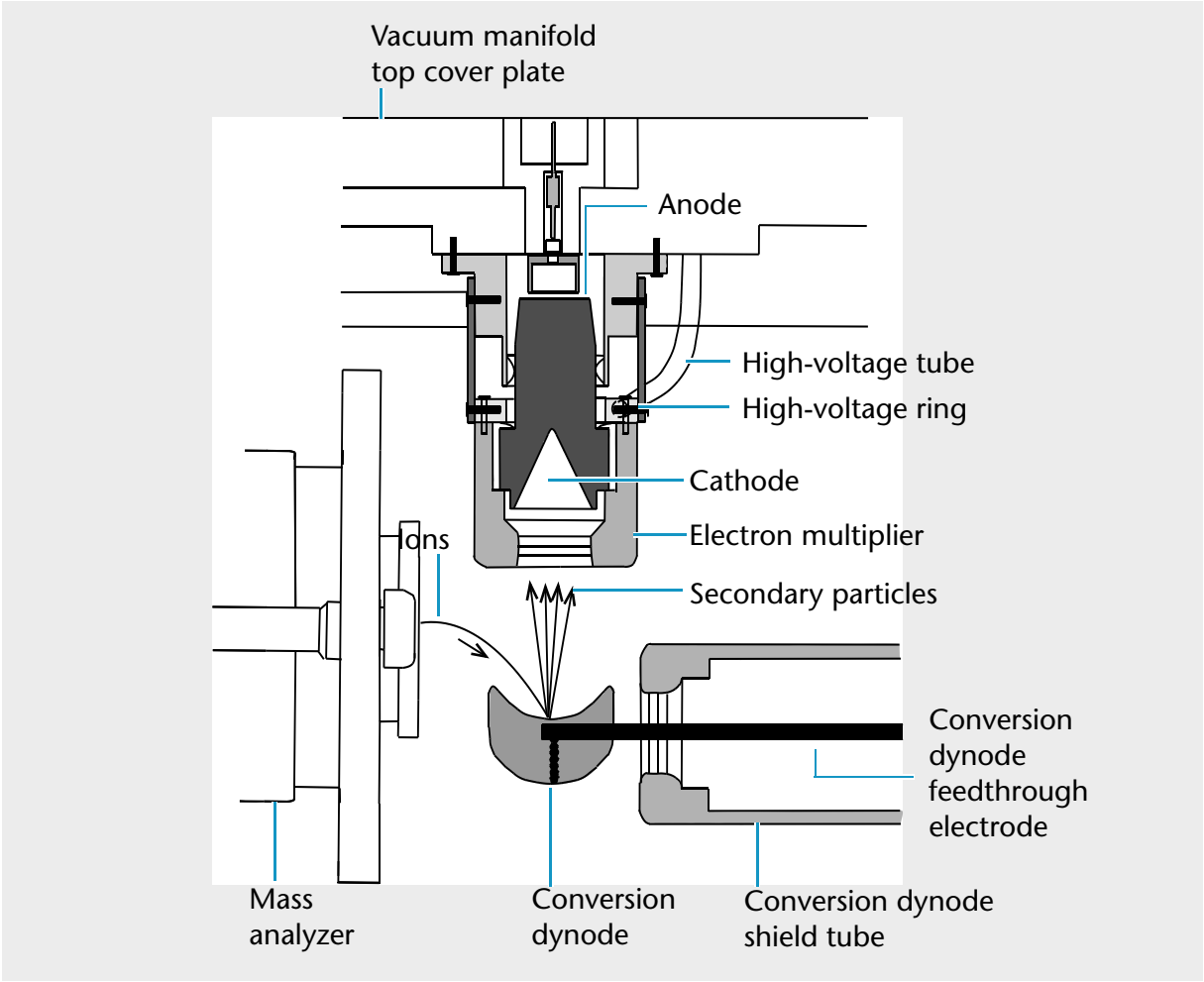


Fig. 19 Cross-sectional view of the ion detection system, showing the electron multiplier and the conversion diode

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. Because of 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. A cascade of electrons is therefore 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.

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 MSQ Plus Mass Detector increases the signal while maintaining a low noise level. 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.

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
- Turbomolecular Pump
- Pirani Gauge
- Vent Valve

A functional block diagram of the vacuum system is shown in Figure 20.

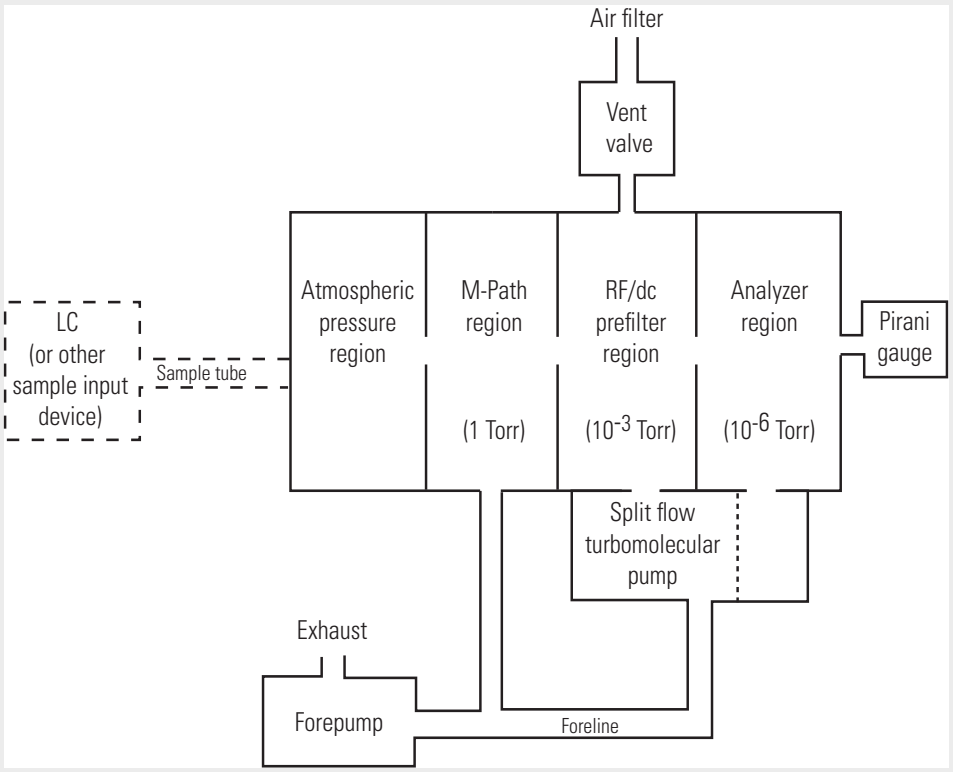


Fig. 20 Functional block diagram of the vacuum system

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 260-250 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 m^3/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 MSQ Plus Mass 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 MSQ Plus Mass 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 mass detector. Dry nitrogen [nominally set to 520 kPa (75 psi) or 450 kPa (45 psi), 99% purity] enters the MSQ Plus Mass Detector through a 6-mm port labeled GAS IN located on the rear panel of the mass detector. The inlet gas hardware then controls the flow of nitrogen gas to the API probes and the reference inlet reservoir. As Figure 21 shows, the inlet gas hardware consists of two regulators, three solenoid valves, and two restrictors.

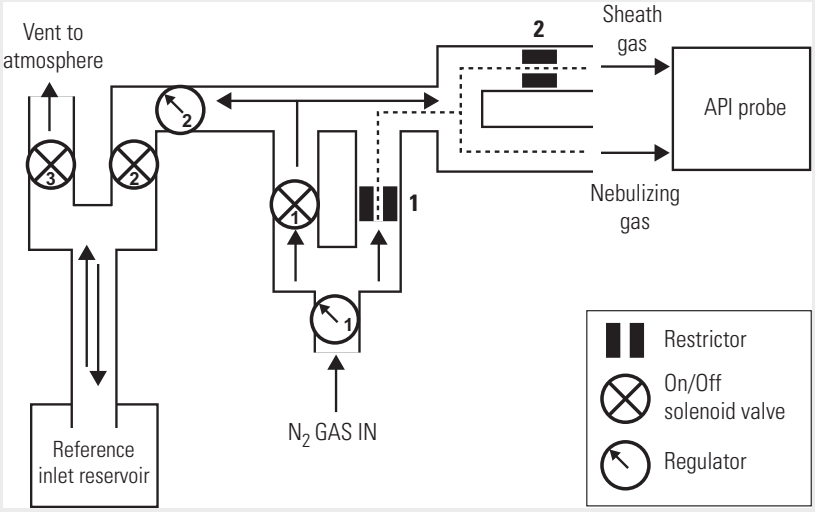


Fig. 21 Functional block diagram of the inlet gases hardware

The first regulator, which is located on the front of the mass detector below the source compartment, as shown in Figure 22, limits the flow of nitrogen gas to the API probe. You nominally set this regulator to 5.2 bar (75 psi) for ESI mode or 3.1 bar (45 psi) for APCI mode.

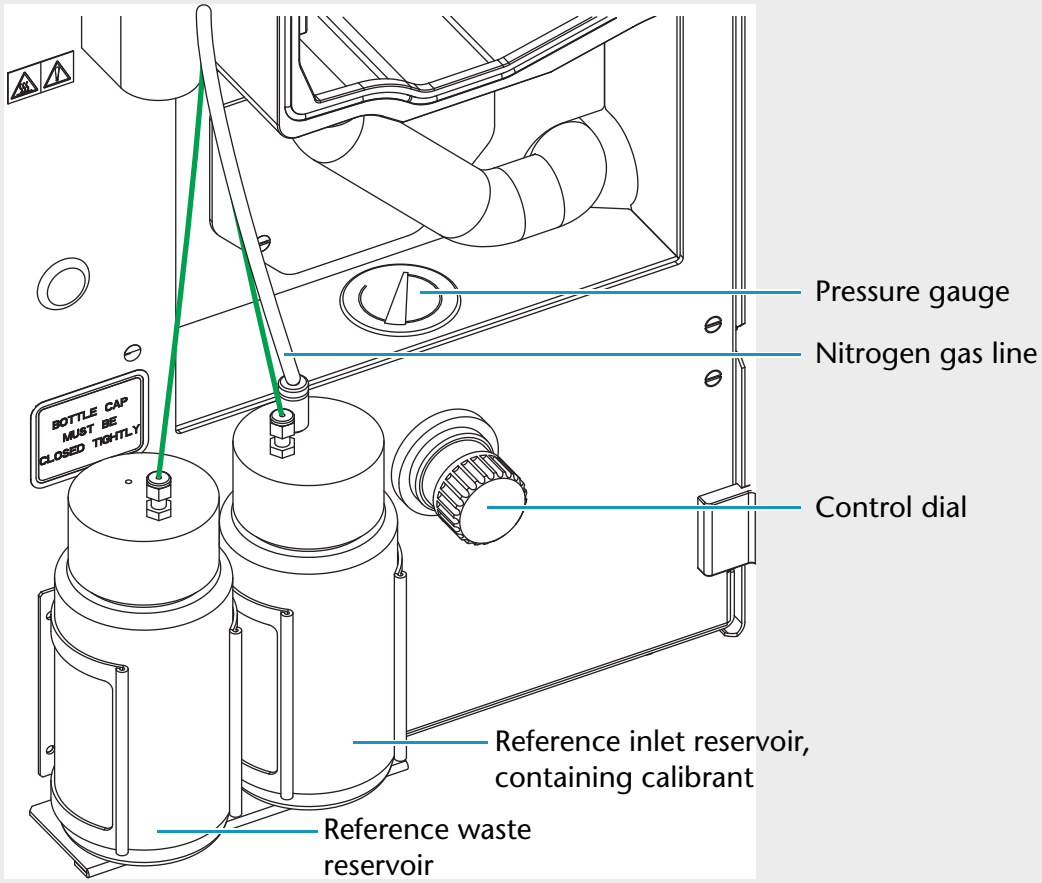


Fig. 22 User-controlled auxiliary nitrogen regulator

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

Note: Call a KNAUER service representative if you suspect that the nitrogen regulator inside the mass detector is malfunctioning.

The first solenoid valve is the On/Off control for the flow of nitrogen gas into the MSQ Plus Mass Detector. To turn this valve on or off, click the Nitrogen Gas On/Off toggle button in the Per Method Parameters table of the Tune window, shown in Figure 23. After you turn on the nitrogen gas, you can hear the jet of nitrogen gas flowing into the API probe.

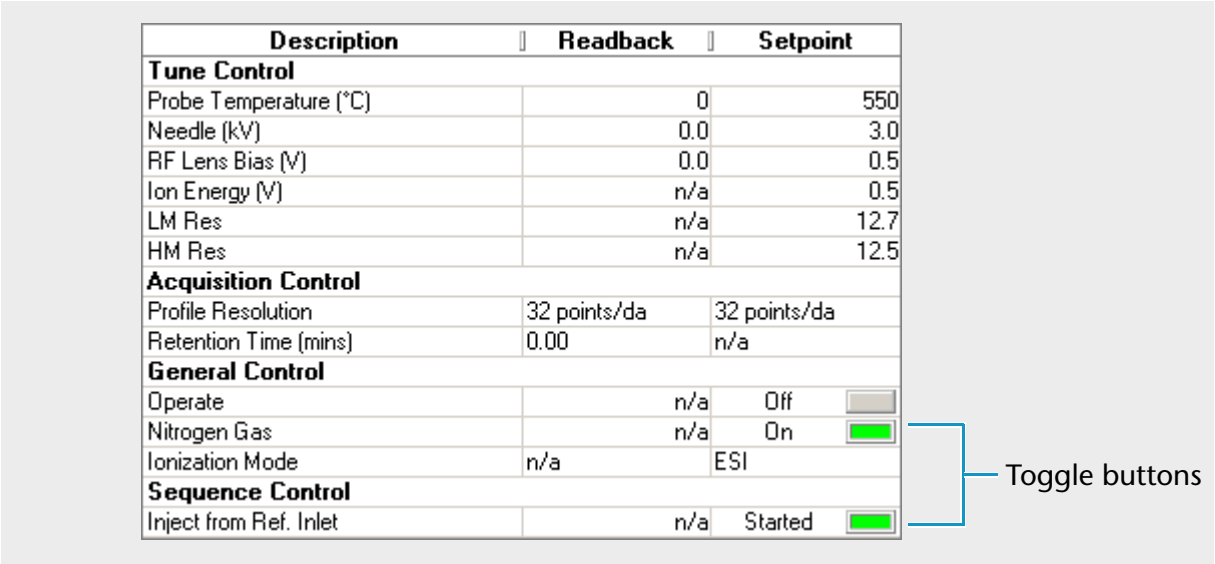


Fig. 23 Per Method Parameters table

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 µL sample loop attached to the microinjection valve of the mass 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 the 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 21. The second restrictor shown in Figure 21 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 mass 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:

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

Cone Wash System

The API source on the MSQ Plus Mass Detector includes a self-cleaning solvent delivery system called the cone wash system. Figure shows an example of this system. It 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 24. 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.

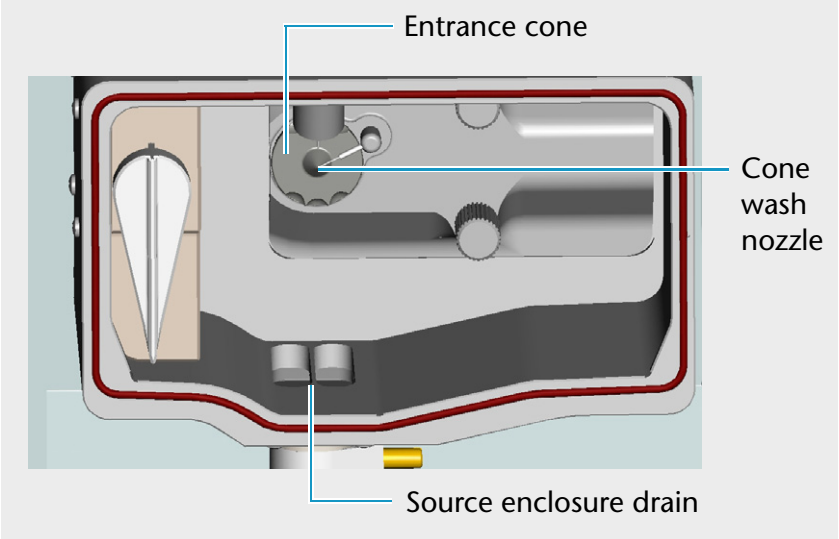


Fig. 24 Source enclosure

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 MSQ Plus Mass Detector. Inside the MSQ Plus Mass 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 24. 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.

Data System

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

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 mass 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 MSQ Plus Mass Detector and a variety of liquid chromatography devices. The Xcalibur software package for the MSQ Plus Mass Detector includes the application programs listed in Table 2 on page 47. Xcalibur Home Page version 2.0 or 2.1 is required for the MSQ 2.0 version of the MSQ Plus Mass Detector software. The firmware versions of the LC devices controlled by the Xcalibur data system are listed in the *MSQ Plus Mass Detector Connection Guide.*

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

Table 2. Xcalibur software application programs

Note: The Signal-to-Noise Calculator program (SigNoise.exe) is only installed for Xcalibur 2.0 data systems. It is not installed for Xcalibur 2.0.*x* data systems.

When you start the Xcalibur data system from the Windows desktop by clicking its application icon, the Xcalibur Roadmap view, shown in Figure 25, opens to show a 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. In addition, the Xcalibur data system runs the MSQ Plus Mass Detector server.

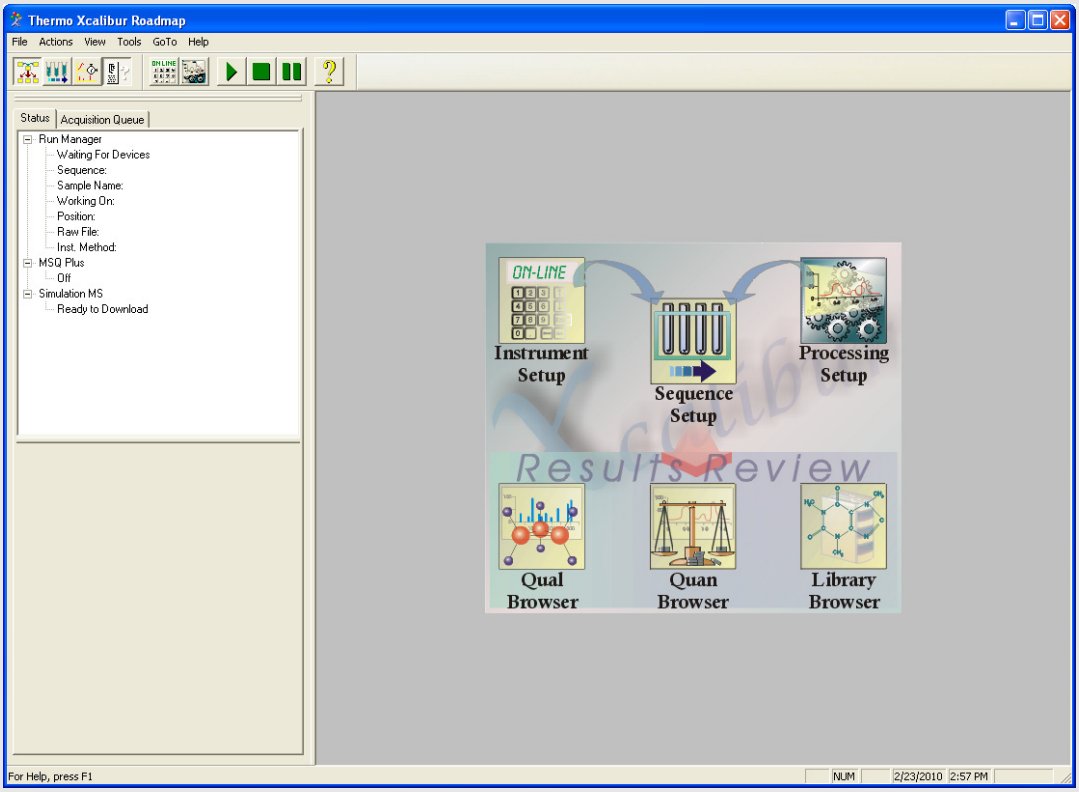


Fig. 25 Xcalibur Roadmap view

MSQ Plus Mass Detector Server

The server is the software that handles all communication between the MSQ Plus Mass Detector and its controlling PC. When you activate the server by running either the Xcalibur data system or Tune, the Server LED icon is displayed in the system tray of the Windows taskbar, just to the left of the time display.

Fig. 26 View of taskbar, showing the Server icon

The Server icon provides you with information about the status of the MSQ Plus Mass Detector and mimics a tristate 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 Operate mode
- Steady green when the instrument is pumped down and is in Operate mode

Right-clicking the Server icon opens the shortcut menu shown in Figure 27.

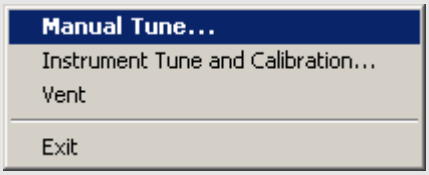


Fig. 27 Server shortcut menu

This menu allows you to do the following:

- Choose **Manual Tune** to open the Tune window, where you can optimize the performance of your MSQ Plus Mass 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 mass detector, set the On/Off switch on the forepump to the Off position.
- Choose **Exit** to close the server.

Tune Window

The MSQ Plus Mass Detector Tune window allows you to optimize the mass detector parameters (manually tune) for your analytes and acquire data to a raw (.raw) file. When you finish tuning your mass detector for a compound of interest, you save the current values of the tuning parameters in a tune (.tune) file. 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.

Practical tip! There are three ways to open the Tune window:

1. Start the Xcalibur data system. Then double-click the Server icon that appears in the system tray of the Windows taskbar.
2. 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.
3. From the Windows XP taskbar, choose *Start* > *Programs* > *Thermo 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 the Xcalibur data system.

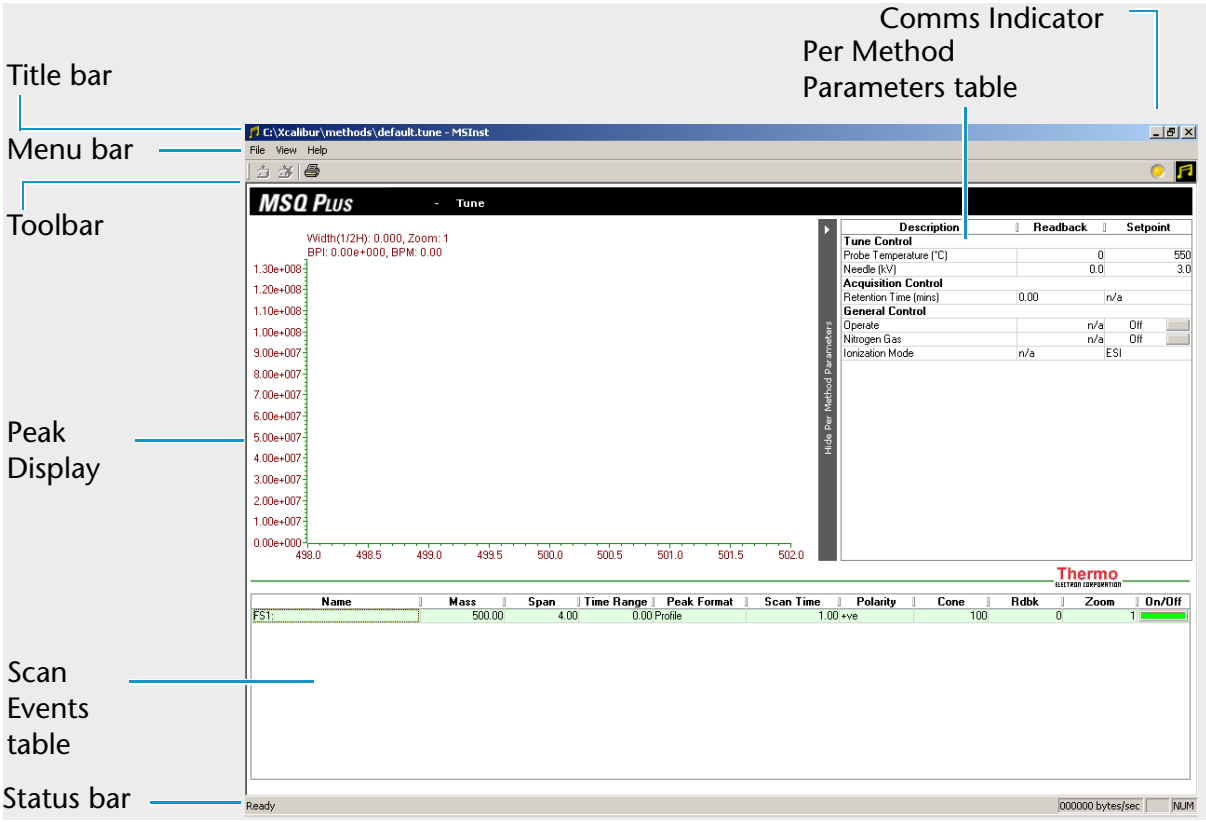


Fig. 28 Features of the Tune window

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 MSQ Plus Mass Detector default tuning file.

Menu Bar

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

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 there is proper communication between the Tune software and the MSQ Plus Mass 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 working communication is occurring.

The Tune icon,  , 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 communication between the MSQ Plus Mass Detector and the PC is established. For example, whenever the Tune software uploads or downloads a command, the icon spins to indicate that communication is working correctly.

The Acquisition icon,  , 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 MSQ Plus Mass 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. Use the Peak Display to monitor the tuning peaks for both peak form and intensity, particularly when optimizing the MSQ Plus Mass Detector during a manual tune. Using the parameters available in the Scan Events table, you can change the number of peaks displayed in the Peak Display and their appearance.

Status Bar

The status bar at the bottom of the Tune window displays information on the current status of the MSQ Plus Mass Detector.

Printer

KNAUER does not ship a printer with the MSQ Plus Mass Detector. If you want 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.

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 MSQ Plus Mass Detector, you must perform various routine operations both before and after you operate the system.

Before Operating the Mass Detector

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 15800 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 Roadmap view (which is available by choosing *Start* > *All Programs* > *Xcalibur* > *Xcalibur* from the Windows taskbar), choose *Actions* > *Check Disk Space*.

The Disk Space dialog box opens. It lists the following:

- Current drive and directory, for example, C:\Xcalibur\system\programs
- Number of megabytes 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 and 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, as shown in Figure 29, 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 55 for instructions on draining the oil mist filter.

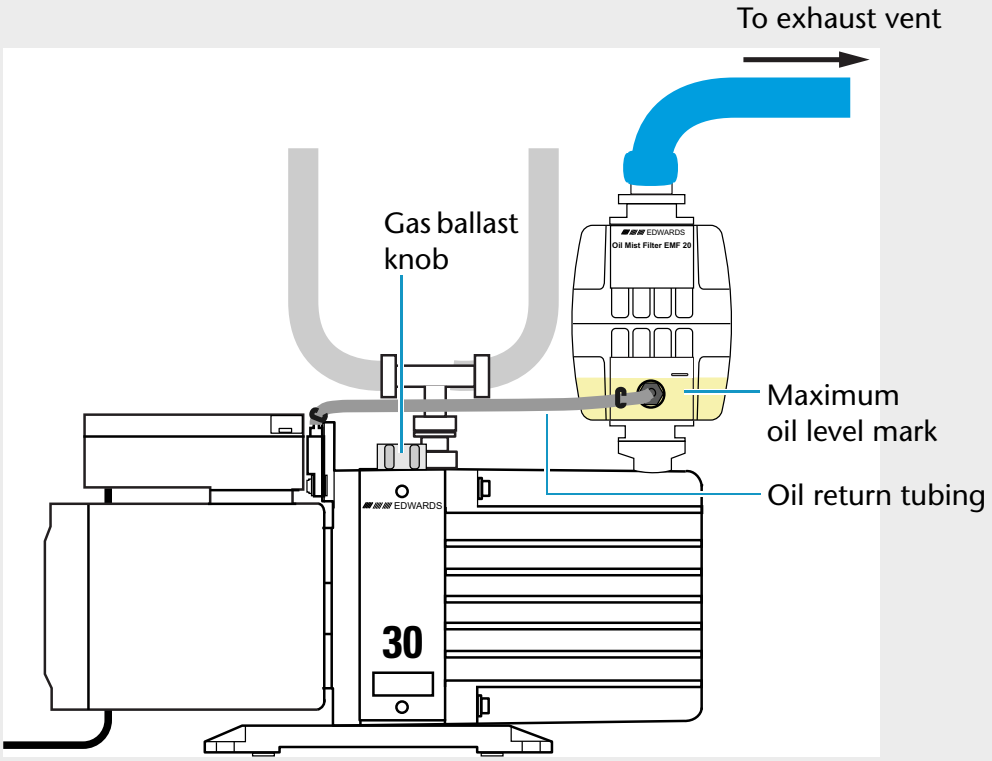


Fig. 29 View of Edwards forepump with oil mist filter, showing the maximum oil level mark

After Operating the Mass 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 MSQ Plus Mass 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. 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 MSQ Plus Mass 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.

For instructions on turning the system to the Off mode, see “Placing the System in the Off Mode” on page 107.

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, as shown in Figure 29, 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 Status page of the Information view in the Xcalibur data system, right-click the **MSQ Plus** 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 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 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 MSQ Plus Mass Detector and the LC system. In the MSQ Plus Mass 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 UHPLC autosampler, perform a flush operation after each injection. The flush solution drains to a solvent waste bottle. Dispose of the solvent waste in accordance with local and federal regulations.

Switching Probes

This chapter describes how to connect an atmospheric pressure ionization (API) probe to the MSQ Plus Mass Detector.

Switching from ESI to APCI

Follow these steps to connect an APCI probe to the MSQ Plus Mass Detector.

To switch from ESI mode to 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 Status page in the Information view in the Xcalibur data system, right-click the **MSQ Plus** listing to display a shortcut menu, and choose *Turn Device Off* from the menu.

–or–

▶ Open the Per Method Parameters table in the Tune window. Take the system out of 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 MSQ Plus Mass 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 30. Then, turn the locking plate counter-clockwise to the closed position.

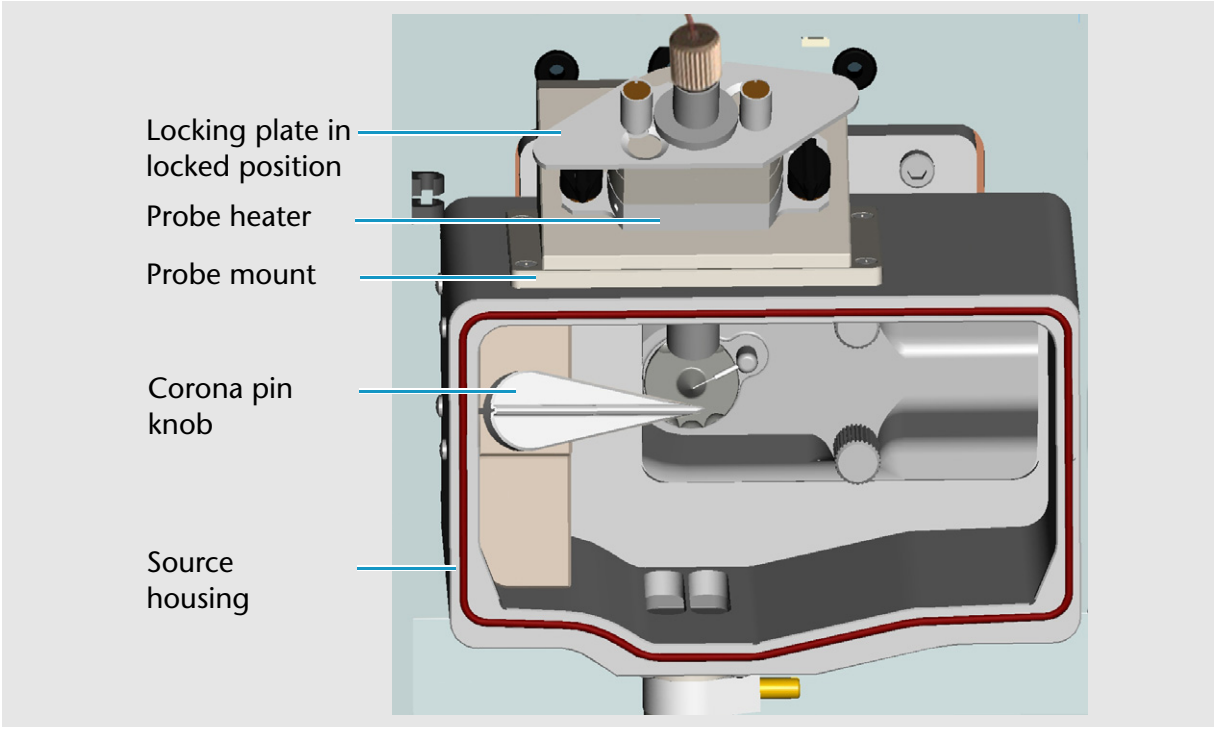


Fig. 30 MSQ Plus Mass Detector setup for the APCI mode

Figure 30 shows the MSQ Plus Mass Detector setup for the APCI mode. Figure 31 shows the corona pin in the operational position for the APCI mode.

Fig. 31 View of 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).

Switching from APCI to ESI

Follow these steps to connect an ESI probe to the MSQ Plus Mass Detector.

To switch from APCI mode to 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 Status page in the Information view in the Xcalibur data system, right-click the **MSQ Plus** listing to display a shortcut menu, and choose *Turn Device Off* from the menu.

–or–

▶ Open the Per Method Parameters table in the Tune window. Take the system out of 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 mass detector.
7. Remove the ESI probe from the holder located in the door of the MSQ Plus Mass 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.

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 32. Then, turn the locking plate counterclockwise to the closed position.

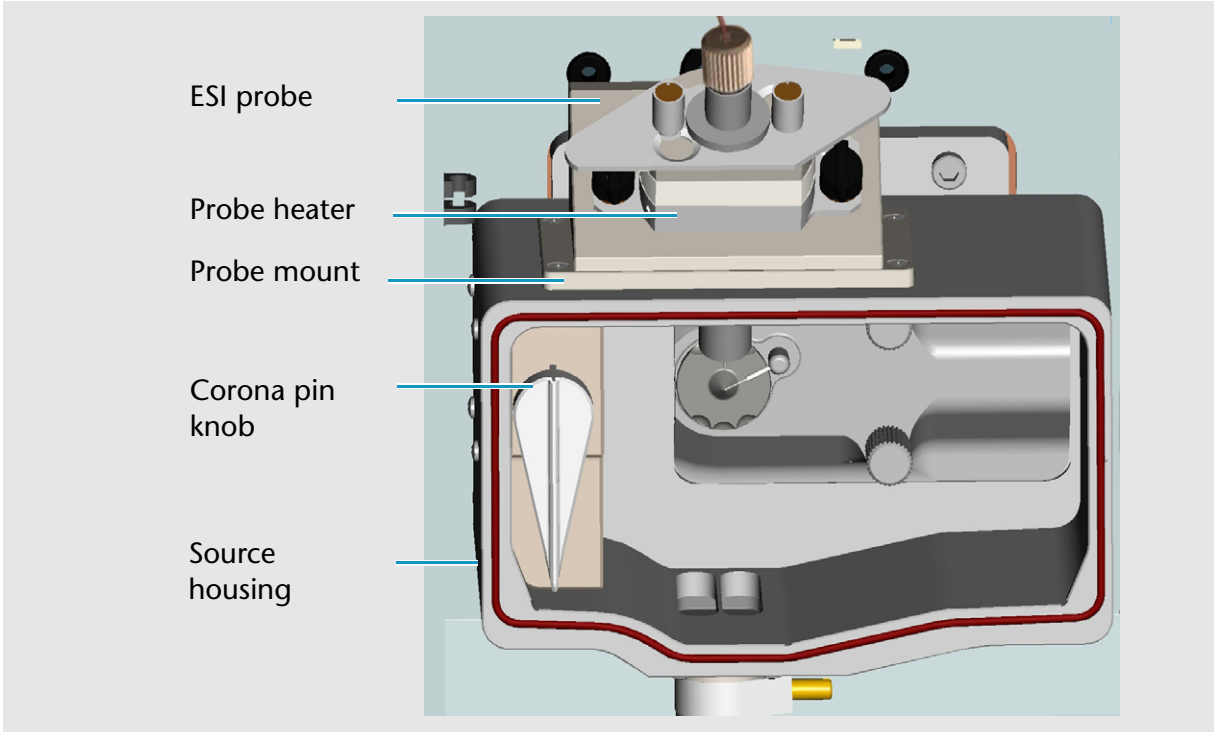


Fig. 32 MSQ Plus Mass Detector setup for ESI mode

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

See Figure 32, which shows the MSQ Plus Mass Detector setup for the ESI mode.

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

Routine and Preventive Maintenance

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

The MSQ Plus Mass 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.

Maintenance Schedule

Table 3 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 that 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.

Frequency	Action
As needed	Clean the source if you see a drop in sensitivity during analyses.
	Flush the capillaries after running an analysis that required buffered solvents. Refer to “Flushing the API Probes” on page 54.
	Replace the capillary if it becomes clogged. Refer to “Maintaining the ESI Probe” on page 61 or “Maintaining the APCI Probe” on page 72.
	Clean the probe if it becomes contaminated. Refer to “Maintaining the ESI Probe” on page 61 or “Maintaining the APCI Probe” on page 72.
	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 55.
Monthly	Clean the probe heater. Refer to “Maintaining the Probe Heater” on page 79.
	Check the oil level and color in the rotary pump and add oil if necessary. Refer to “Maintaining the Forepump” on page 101. 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 94. Perform maintenance of the turbo pump every two years.

Table 3. Maintenance schedule

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. The inner diameter of the ESI probe is 127 +/- 30 uM, and the expected backpressure at 1 ml/mn is about 150 psi.

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

Removing the ESI Probe

Follow these steps to remove the ESI probe from the probe heater.

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 Status page of the Information view in the Xcalibur data system, right-click the **MSQ Plus** 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 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 mass detector to cool.
4. Unscrew and remove the PEEK fingertight fitting, shown in Figure 33, from the ESI probe.

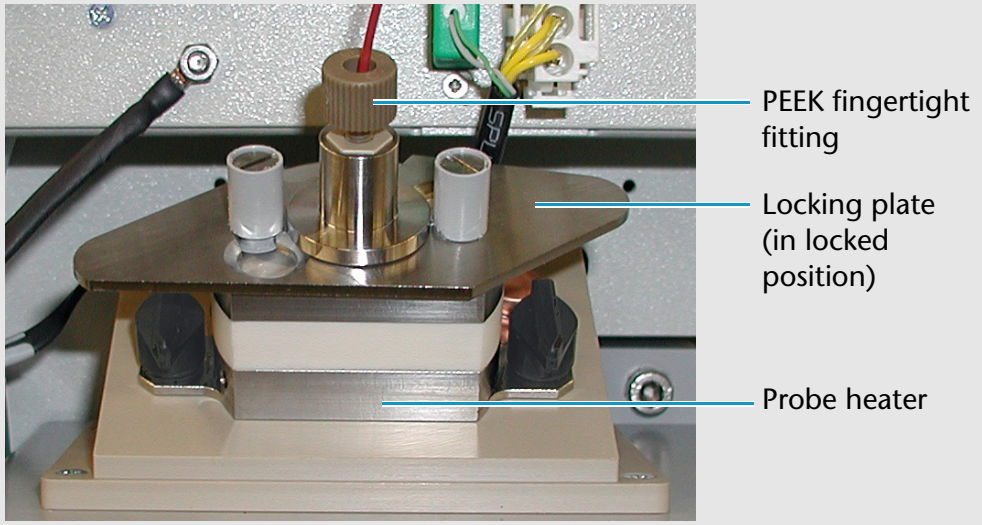


Fig. 33 ESI probe installed in the probe heater

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 34.

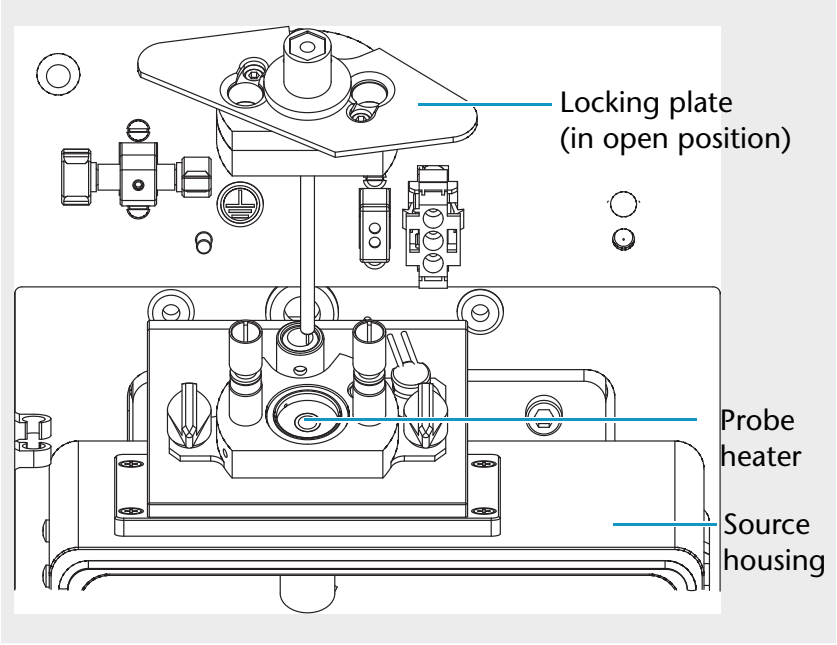


Fig. 34 Removing the ESI probe from the probe heater

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 for the part numbers of the ESI probe components.

To remove the capillary from the ESI probe

1. Remove the ESI probe from the mass detector, as described in “Removing the ESI Probe” on page 62.
2. 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, as shown in Figure 35.

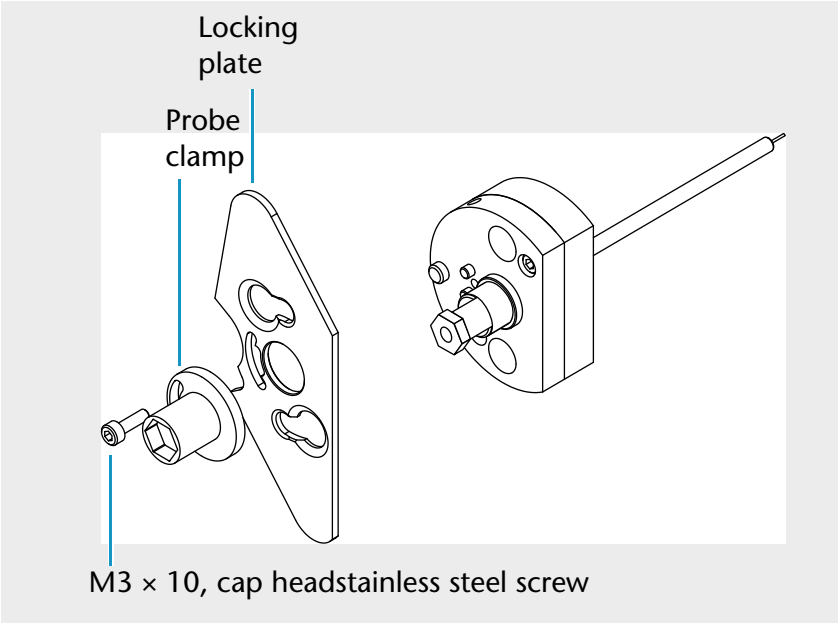


Fig. 35 Removing the probe clamp and locking plate

3. Using the front end of the probe clamp, unscrew the capillary retaining nut (adaptor) and remove it from the ESI probe. See Figure 36.

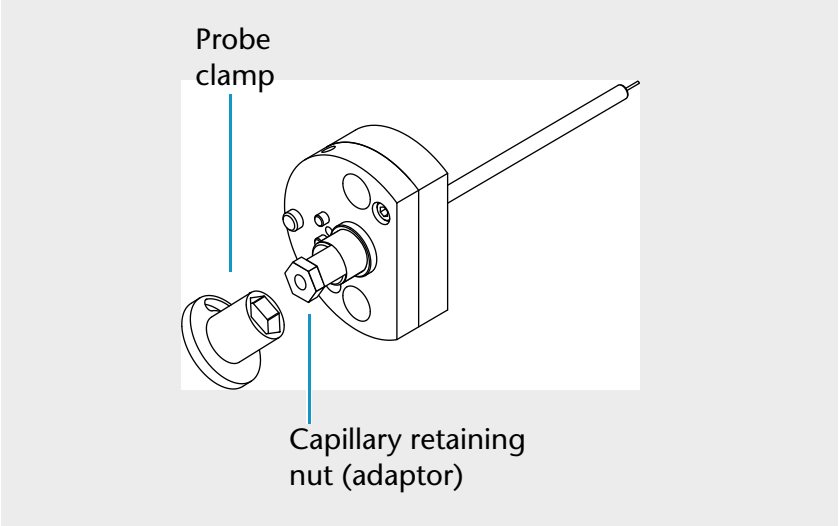


Fig. 36 Removing the capillary retaining nut (adaptor)

4. Place a lint-free cloth on the workbench, and then gently shake the ESI capillary (part number A66542), graphite ferrule (part number A66538), and PEEK tube insert (part number A66540) assembly out of the ESI probe onto the cloth, as shown in Figure 37.

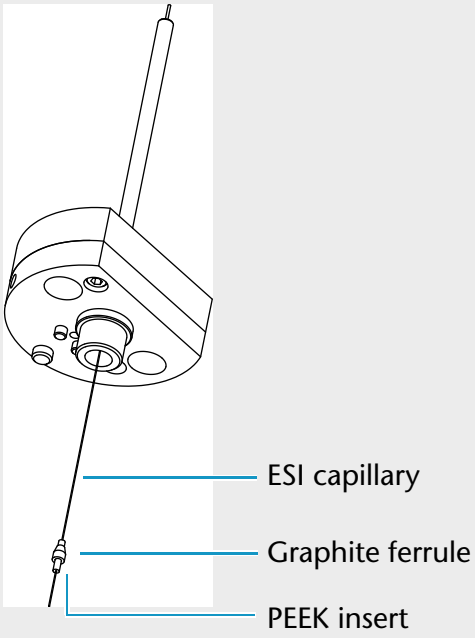


Fig. 37 Removing the capillary, graphite ferrule, and PEEK insert assembly from the probe

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

Follow this procedure to clean or replace 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 62.
2. Remove the ESI capillary from the probe, as described in “Removing the ESI Capillary” on page 63.
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 68.

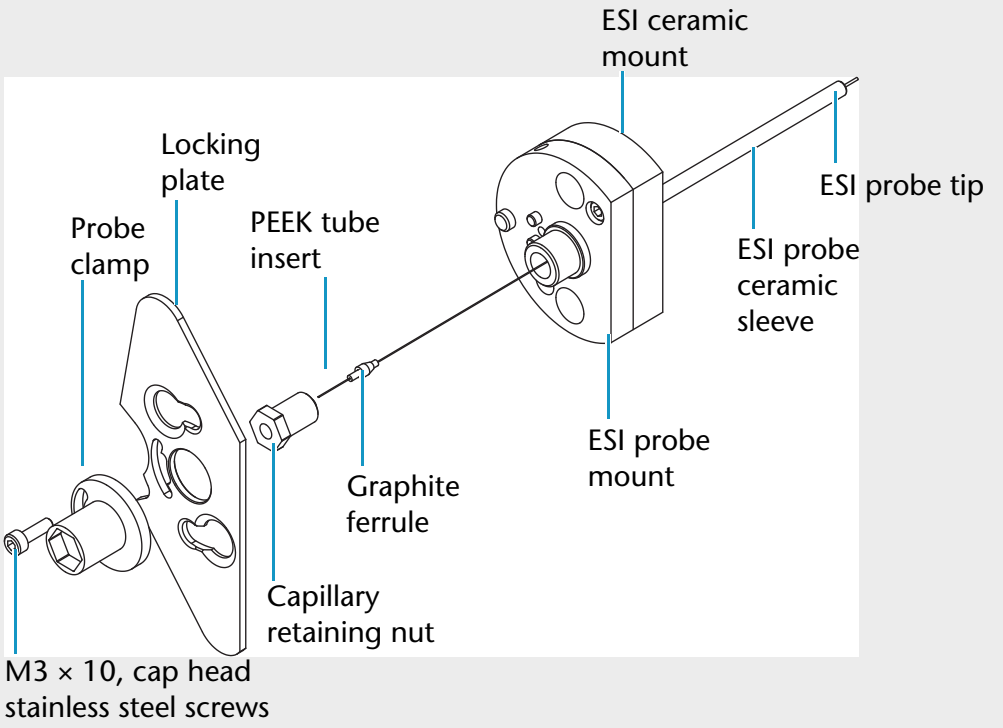


Fig. 38 Partially disassembled ESI probe with capillary removed

Replacing the Ceramic Sleeve of the ESI Probe

If you break the ceramic sleeve of the ESI probe, replace it. See Figure 85 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 mass detector as described in “Removing the ESI Probe” on page 62.
2. Disassemble the ESI probe and remove the capillary as described in “Removing the ESI Capillary” on page 63.
3. Use the 2.5 mm Allen key to unscrew the two M3 × 8 cap head stainless steel screws from the ESI probe mount. See Figure 38.
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 39 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.

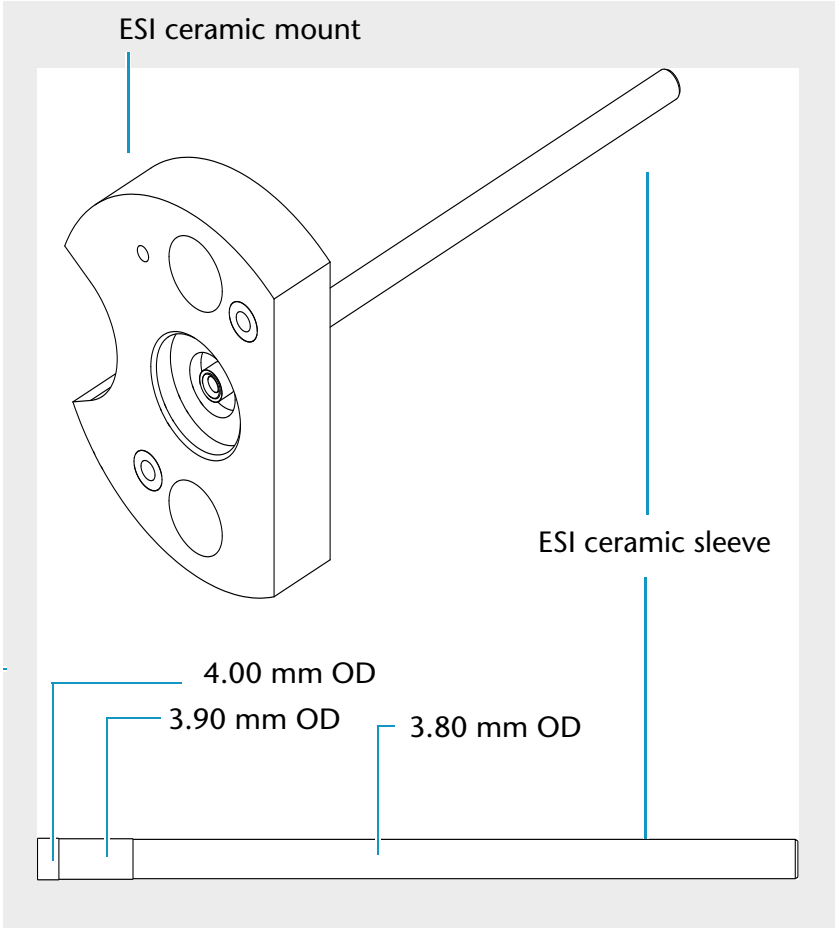


Fig. 39 ESI ceramic mount and ceramic sleeve

6. Insert a new ESI ceramic sleeve (part number A66543) into the ESI ceramic mount. See Figure 40.
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 68.

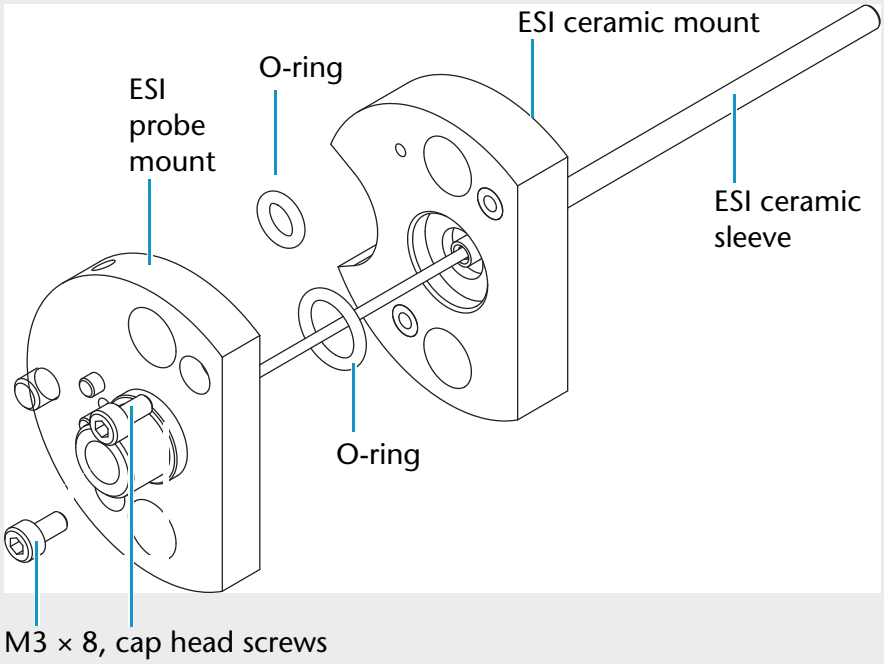


Fig. 40 Pulling the ESI probe mount out of the ESI ceramic mount

Installing the ESI Capillary

The ESI capillary is presized 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 so that 2 mm of the PEEK tube insert is visible on the back end of the assembly. See Figure 41.

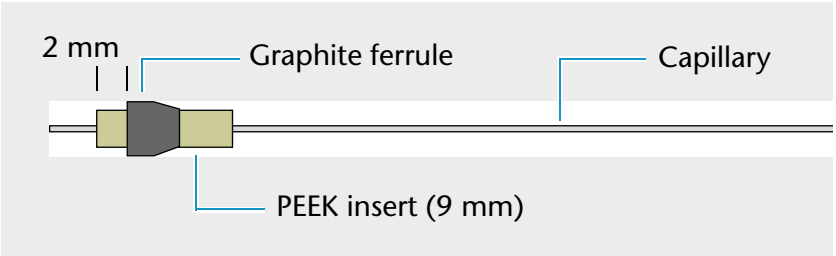


Fig. 41 Capillary, PEEK tube, and graphite ferrule assembly

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 42.

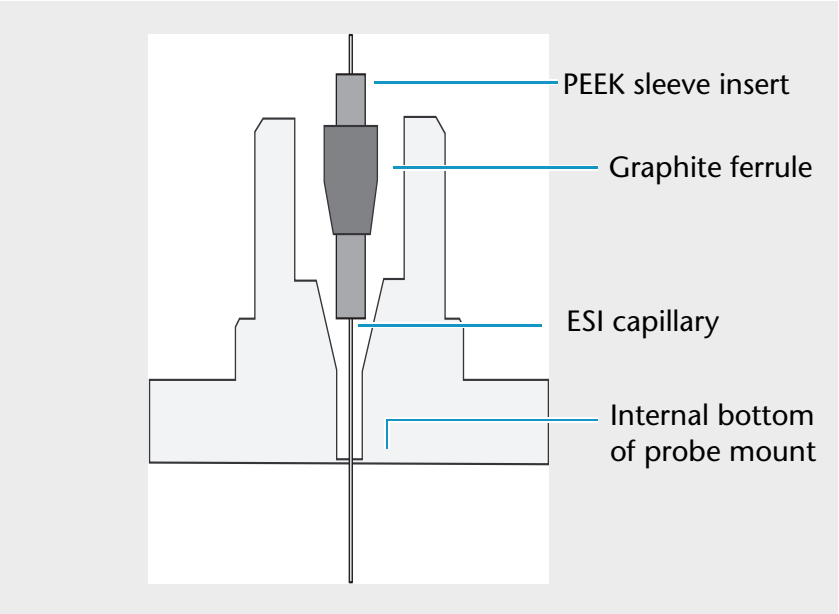


Fig. 42 View of ESI probe mount, showing the insertion of the capillary

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. Tap the body of the ESI probe until it falls past the obstruction at the end of the steel sleeve.

▶ 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 43 shows, the capillary should protrude from the tip of the probe.

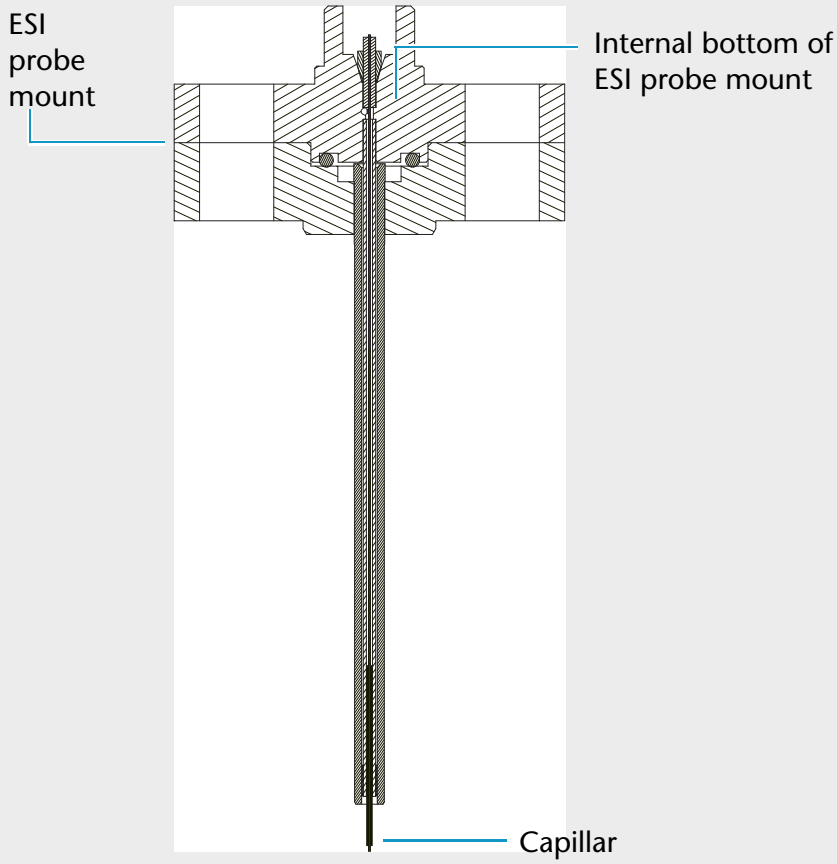


Fig. 43 Capillary inserted into the ESI probe mount

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.

–or–

▶ 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 visually verify that the capillary protrudes from the probe tip.

Figure 44 shows the capillary protruding from the probe tip.

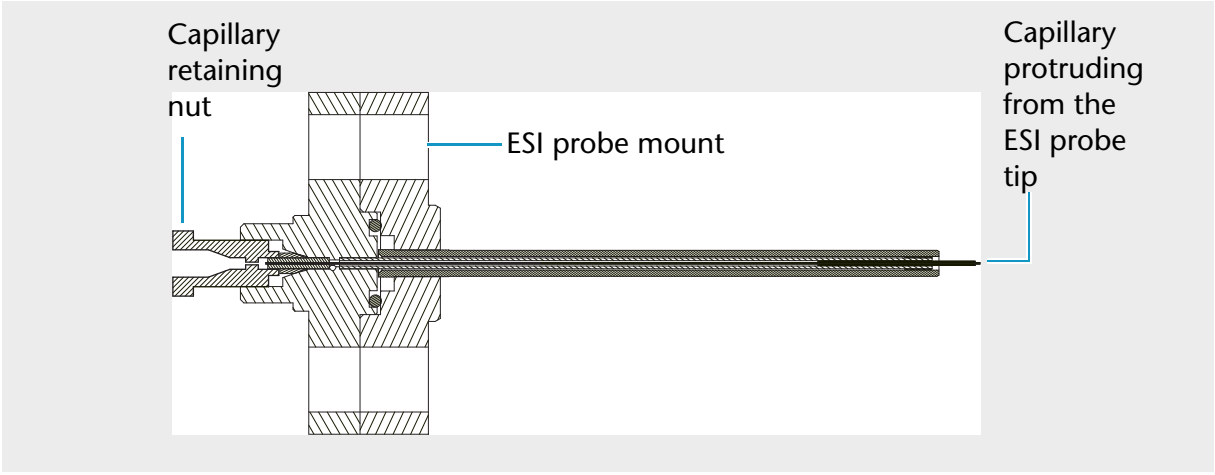


Fig. 44 ESI probe with the capillary retaining nut screwed in finger tight

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, and it may collapse the ESI capillary.
7. Finish reassembling the ESI probe:

▶ Place the locking plate and probe clamp in position.

▶ Insert the M3 × 10 cap head stainless steel screw (part number A66552) 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 45 shows the ESI spacer being used to check the protrusion depth of the capillary.

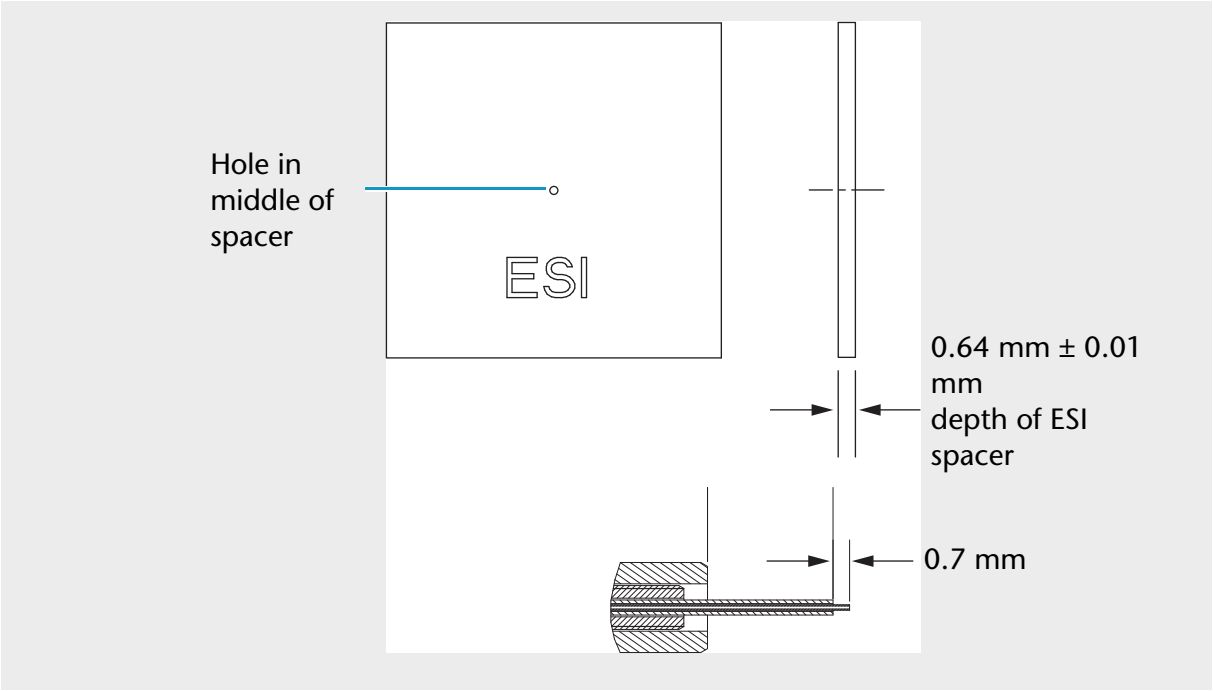


Fig. 45 ESI probe tip with the capillary protruding by 0.7 mm

Installing the ESI Probe

Follow this procedure to install the ESI probe.

To install the ESI probe into the mass 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 33.
4. Connect the PEEK fingertight fitting to the ESI probe. See Figure 33.

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 (part number A66541) 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.

Removing the APCI Probe

Follow these steps to remove the APCI probe from the probe heater.

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 in the Information view of the Xcalibur data system, right-click the **MSQ Plus** 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 Operate mode by clicking the Operate toggle button, and turn off the nitrogen gas by clicking the Nitrogen Gas toggle button. See Figure 23.

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 46.

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

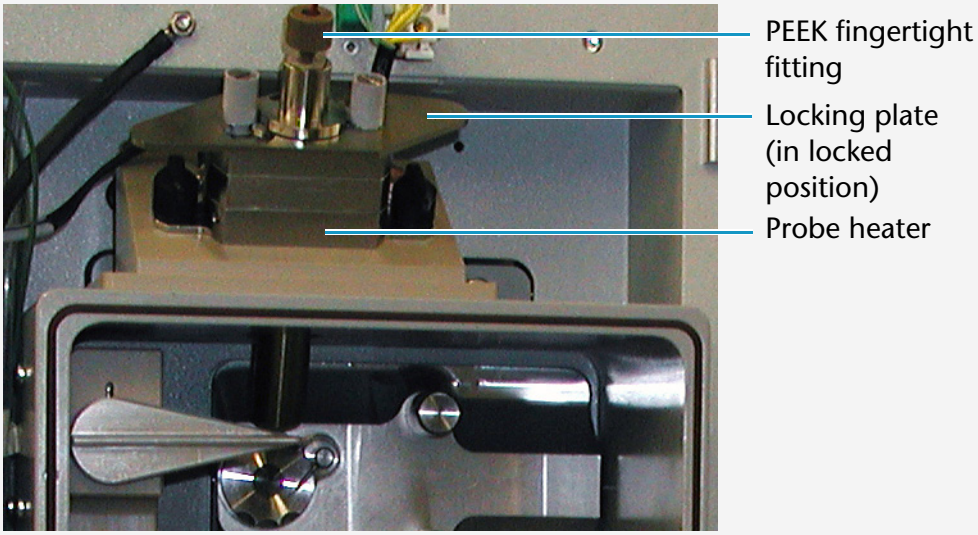


Fig. 46 MSQ Plus Mass Detector set up for the APCI mode

5. Turn the locking plate clockwise to the open position and remove the APCI probe from the probe heater. See Figure 47.

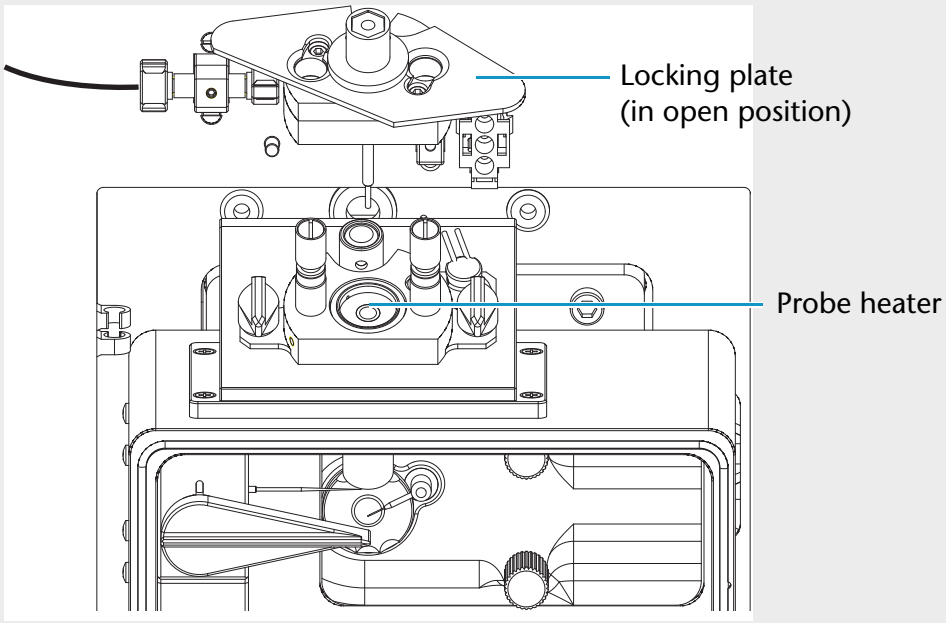


Fig. 47 Remove the APCI probe from the probe heater

Removing the APCI Capillary

Follow these steps to remove the capillary from the APCI probe.

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 (part number A66552) from the probe clamp. See Figure 48.

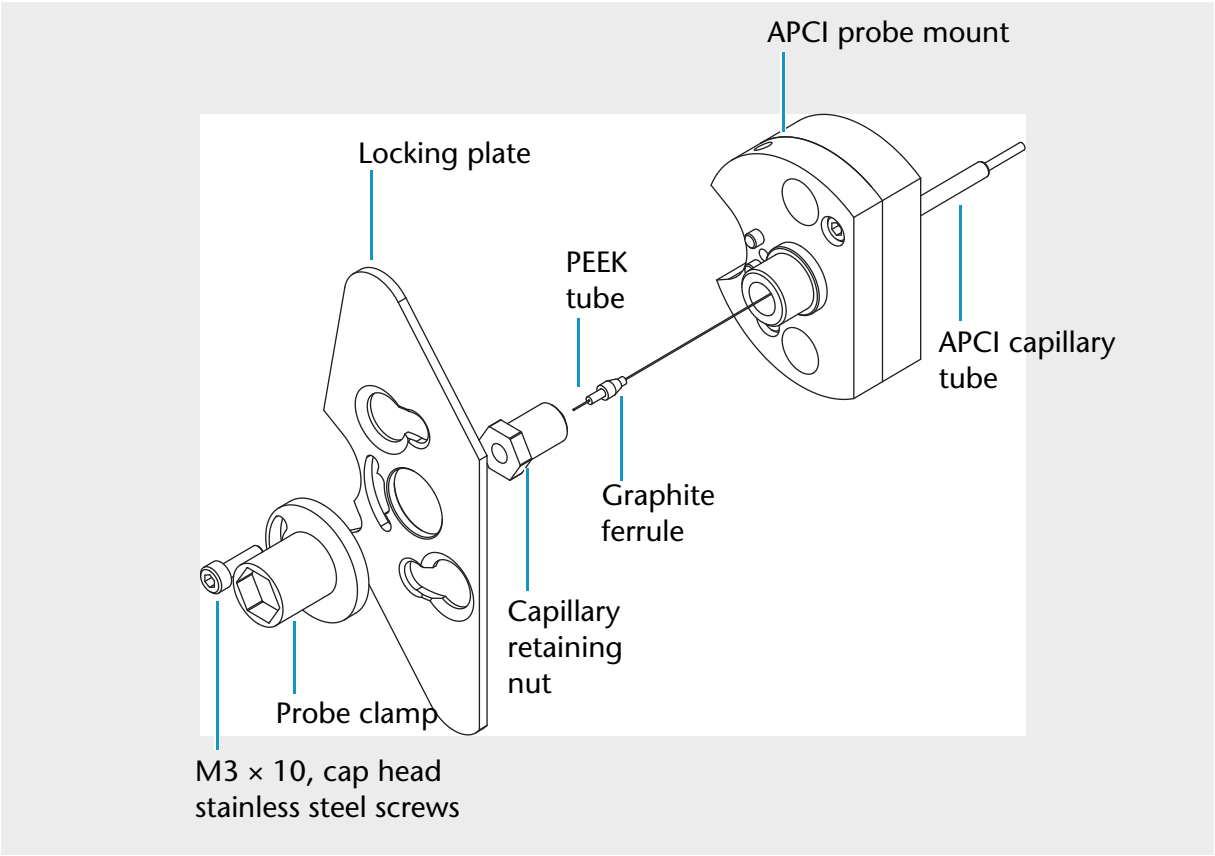


Fig. 48 View of partially disassembled APCI probe with capillary removed

2. Remove the probe clamp and the locking plate.
3. Using the probe clamp, unscrew the capillary retaining nut.
4. Carefully shake the graphite Vespel ferrule (part number A66538), PEEK tube insert (part number A66540), and APCI probe capillary (part number A66541) out of the probe.

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

5. 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.

–or–

▶ If the capillary is reusable, clean its surface with [50:50] methanol/water.

Installing the APCI Capillary

This procedure describes how to install the APCI capillary in the APCI probe.

To install the APCI capillary in 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 49 and Figure 50.

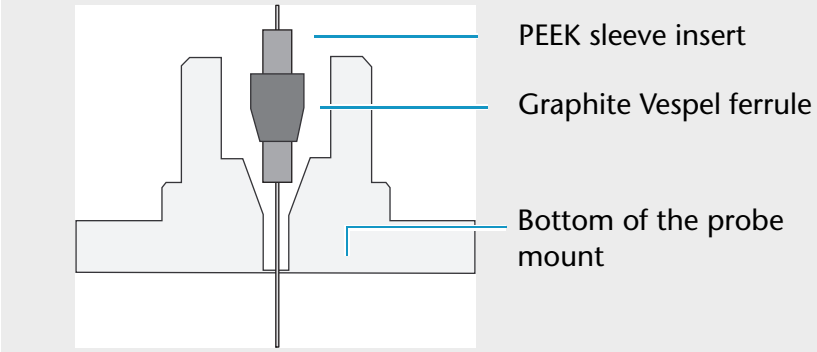


Fig. 49 Inserting the capillary, PEEK sleeve, and graphite ferrule into the APCI probe

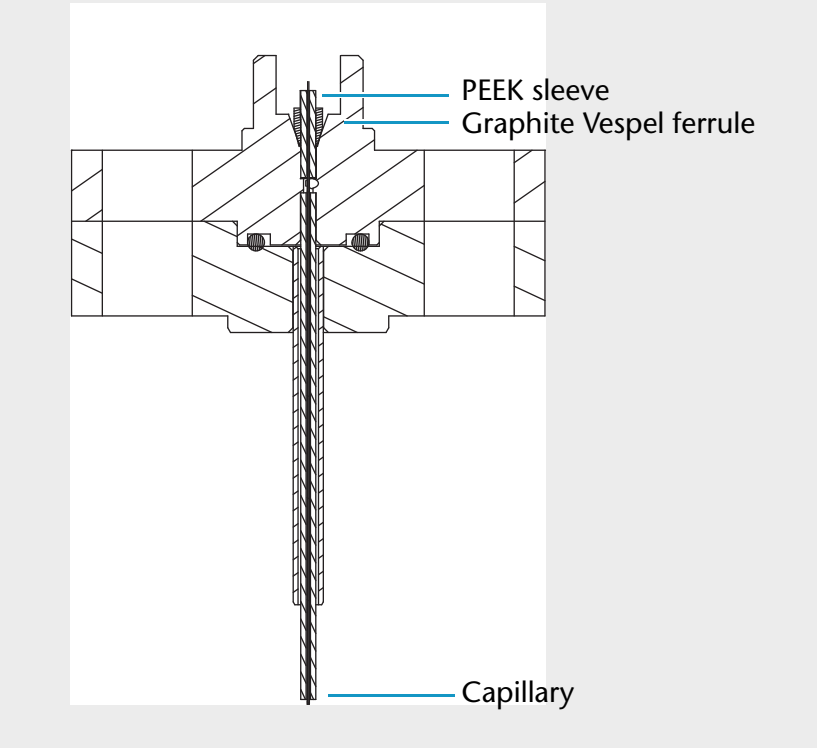


Fig. 50 APCI capillary, PEEK sleeve, and graphite ferrule inserted into probe

3. Adjust the position of the APCI capillary:

- Holding the APCI probe in the vertical position, carefully shake the PEEK tube insert into the probe until it meets resistance. Tap the probe assembly.
- Using tweezers, push the capillary into the probe until it is flush with the PEEK sleeve.
- Visually verify that the capillary is protruding from the tip of the 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.

–or–

- 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 5 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 51.

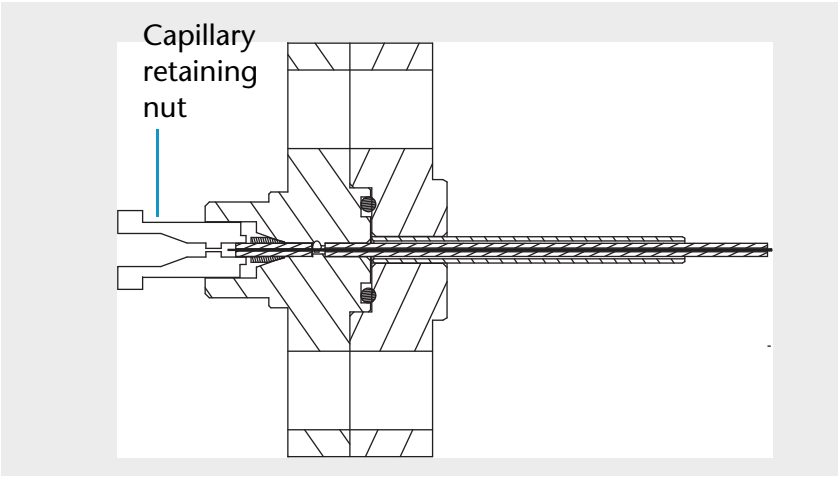


Fig. 51 APCI probe with the capillary retaining nut screwed in finger tight

6. Using the probe clamp, tighten the capillary retaining nut by one-half turn. Do not overtighten the screw.
7. Finish reassembling the probe:

- Place the locking plate and probe clamp in position.
- Insert the M3 × 10 cap head stainless steel screw (part number A66552) 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 MSQ Plus Mass Detector 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 space plate, the capillary protrudes from the APCI probe tip by more than 0.41 mm. See Figure 52.

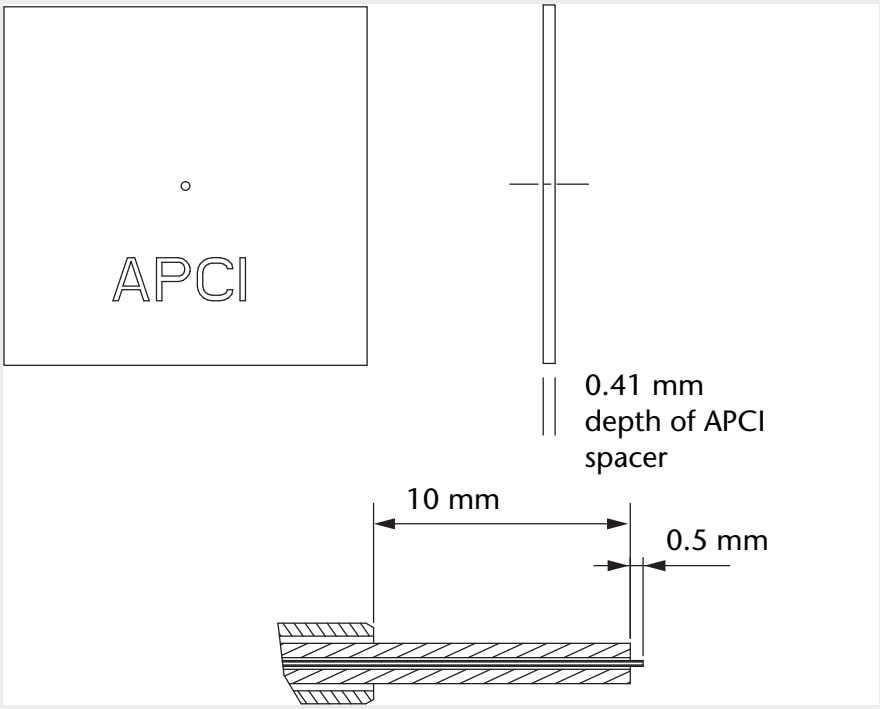


Fig. 52 Enlarged view of the tip of the APCI probe

Installing the APCI Probe

Follow these steps to insert the APCI probe into the probe heater.

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.
2. Turn the locking plate counterclockwise to the closed position to lock the probe in place. See Figure 33.

3. Connect the PEEK fingertight fitting to the APCI probe. See Figure 33.

Maintaining the Probe Heater

If you use the instrument primarily in the ESI mode, you can 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 (part number A66545) to repair the probe. Filing down the edges of the latching plate might compromise the ability of the plate to lock the probe down, so KNAUER recommends that you avoid filing these edges unless pin breakage becomes a common problem.

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

- Removing the Probe Heater
- Cleaning the Probe Heater or Replacing the Detent Screw Insulator
- Installing the Probe Heater

Removing the Probe Heater

Follow these instructions to remove the probe heater from the probe mount.

To remove the probe heater from the probe mount

1. Remove the ESI or APCI probe. Refer to “Removing the ESI Probe” on page 62 or “Removing the APCI Probe” on page 73.

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 53 on page 82 and Figure 54 on page 82.

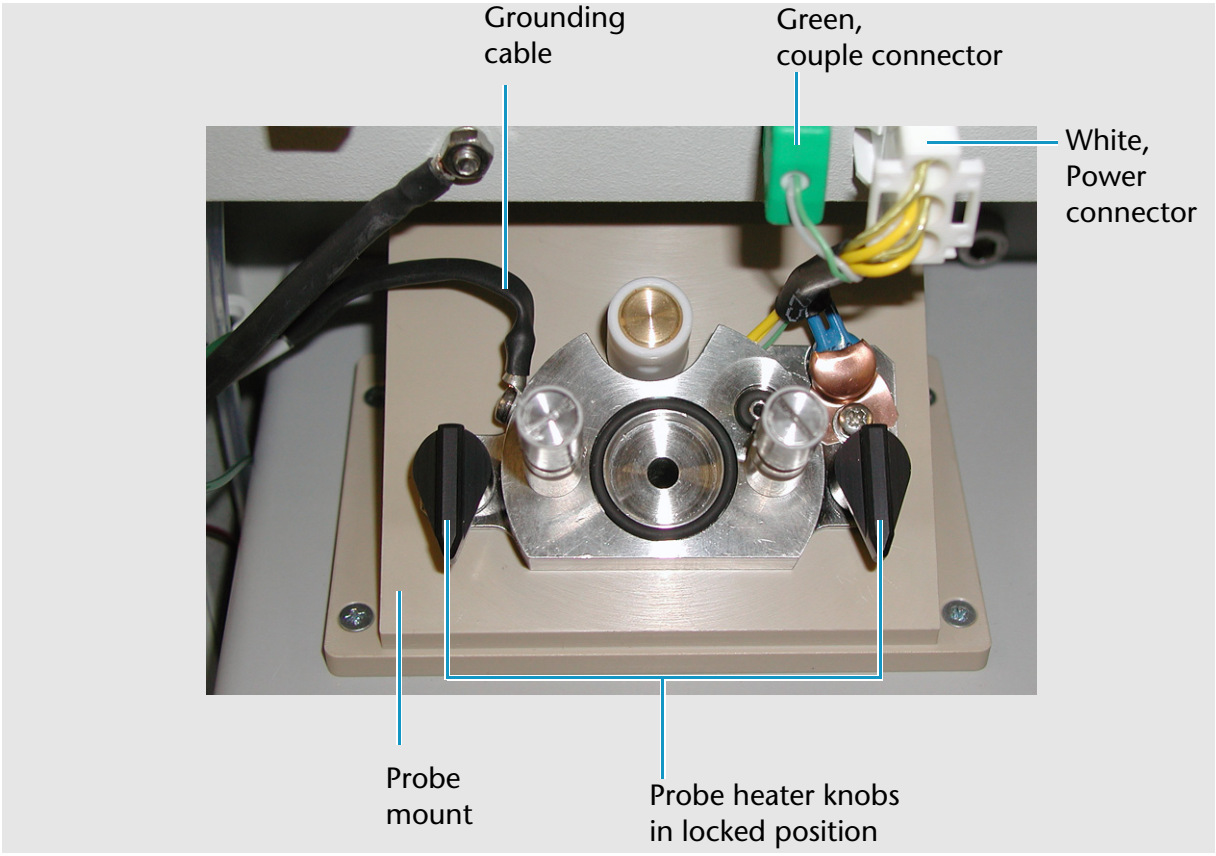


Fig. 53 Probe heater and its connections to the front panel of the mass detector

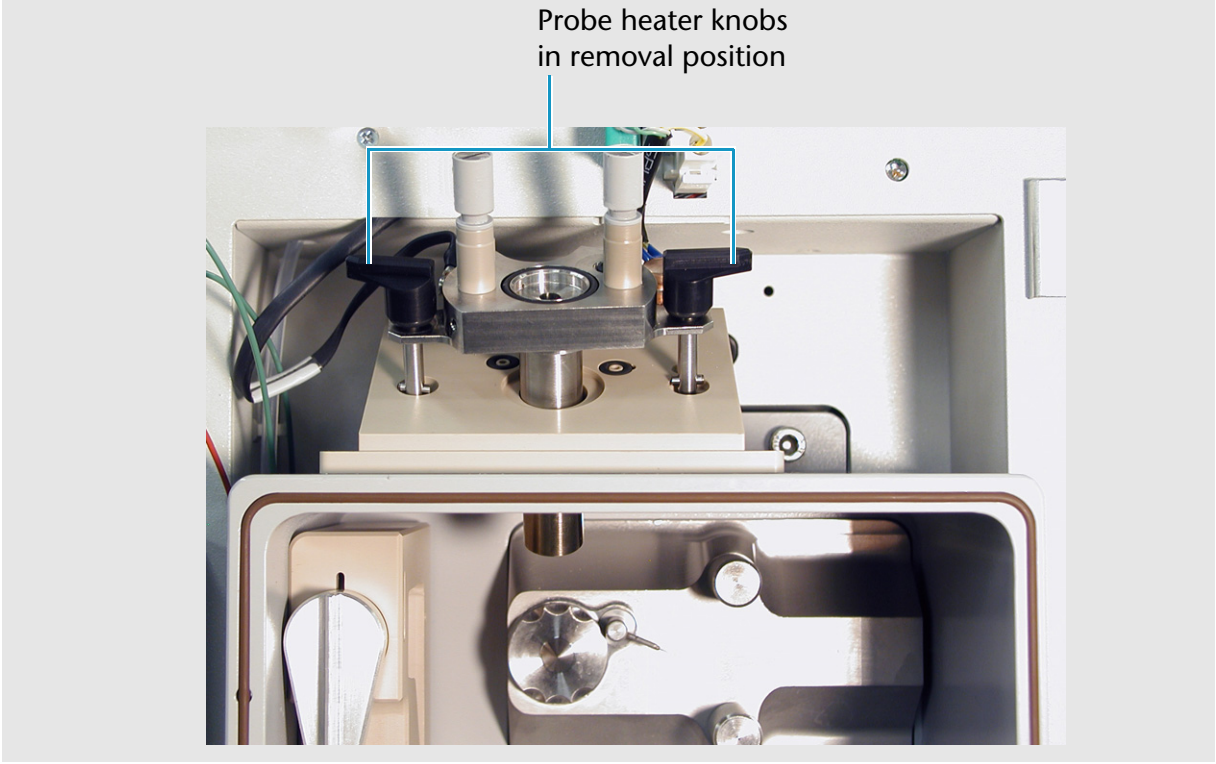


Fig. 54 Probe heater knobs in the removal position

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.

3. Pull the probe heater out of the probe mount, and then disconnect the green connector and the white connector from the mass detector. Be careful not to lose the O-rings. See Figure 55.

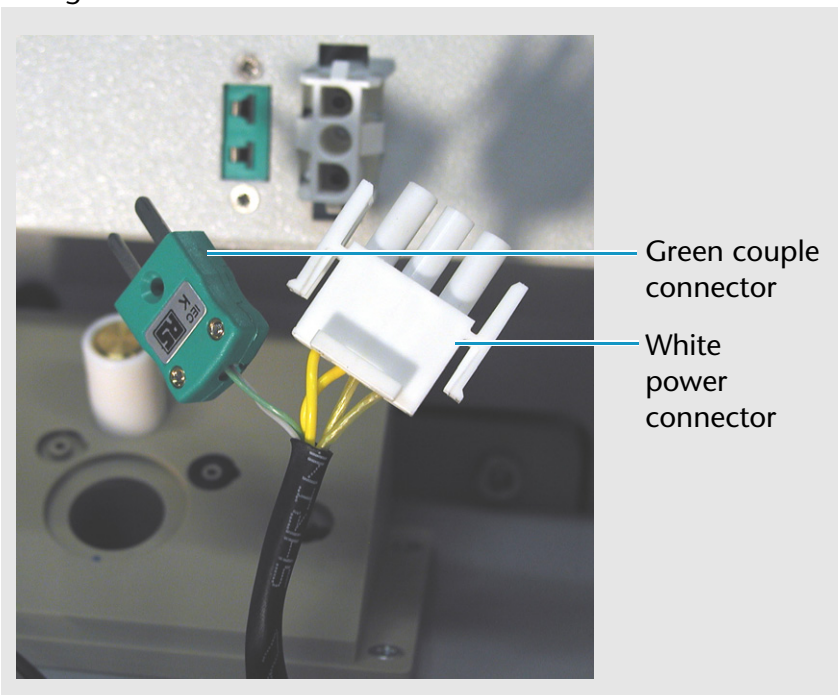


Fig. 55 Power connector and couple connector

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.

The probe heater is attached to the mass detector by the grounding strap, but it is not necessary to remove the grounding strap during the cleaning procedure. See Figure 56.

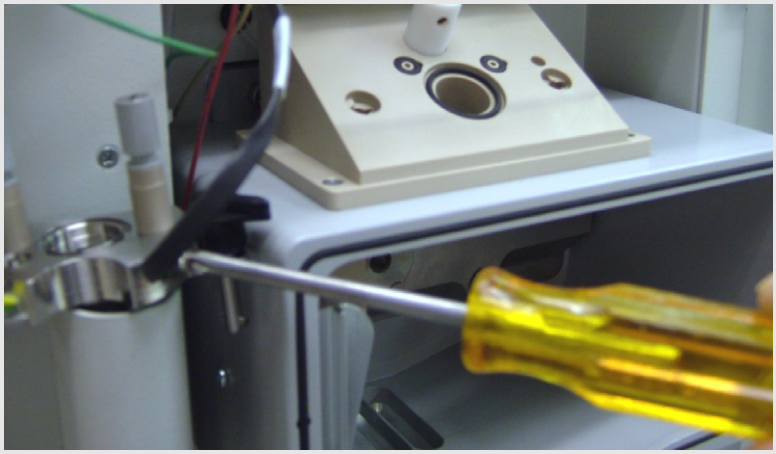


Fig. 56 Probe heater with the operator detaching the grounding strap

Note: The 18-gauge wires in the probe heater may darken with time.

Cleaning the Probe Heater

Be sure to clean the probe heater periodically.

To clean the probe heater

Clean the inside of the heater tube with a cotton swab soaked in [50:50] methanol/water. See Figure 59 on page 86.

Replacing the Detent Screw Insulator

Use the parts supplied in the Probe Heater Repair Kit (part number A66545) to repair the probe heater. See Figure 87 on page 132.

To replace a broken detent screw insulator

1. Disassemble the detent screw assembly:

▶ Using a flat-blade screwdriver, loosen and remove the spring screw.

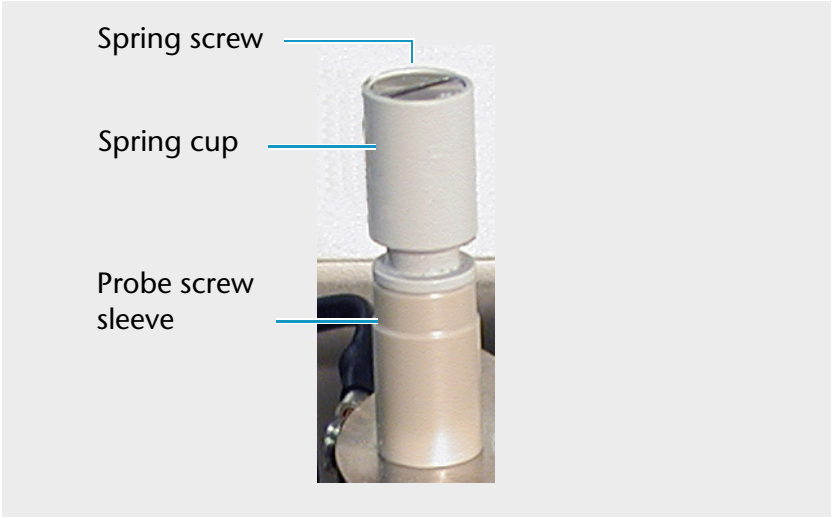


Fig. 57 Slot in the top of the spring screw

▶ 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.

- ▶ Pull the probe screw sleeve off the detent screw insulator.
- ▶ Pull the detent screw insulator out the underside of the probe heater body. See Figure 58.

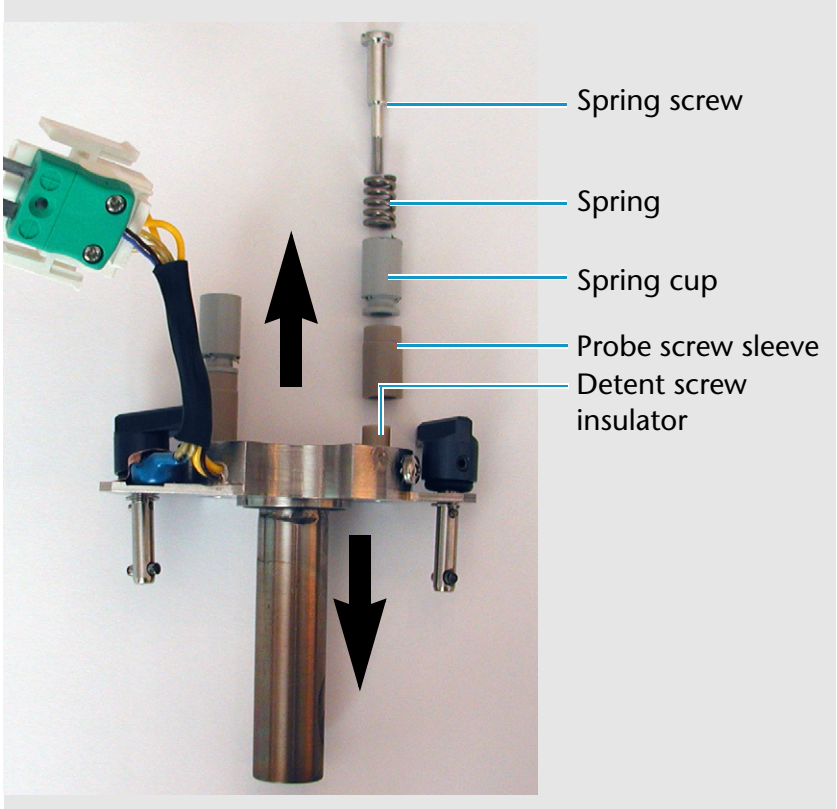


Fig. 58 Partially disassembled probe heater

2. Using the parts supplied in the repair kit, rebuild the assembly:

- ▶ Insert a new detent screw insulator into the underside of the probe heater body.
- ▶ Slide the probe screw sleeve over the top of the detent screw insulator.
- ▶ Insert the spring that you removed from the broken assembly into the spring cup.
- ▶ Align the bottom of the spring cup with the probe screw sleeve.
- ▶ Insert the spring screw into the spring cup and tighten the assembly with a flat-blade screwdriver.

Installing the Probe Heater

This section describes how to install 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 59.

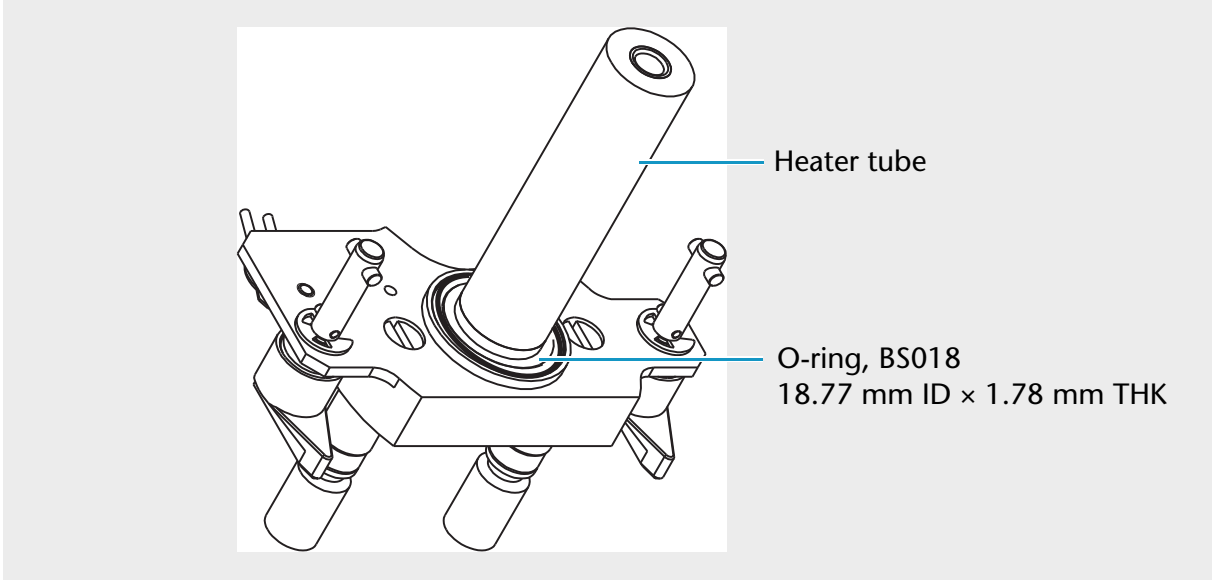


Fig. 59 View of O-ring installed in the underside of the probe heater base

2. If you disconnected the grounding strap from the probe heater, reconnect it to the probe heater. See Figure 56 on page 83.

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 54 on page 82.
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 53 on page 82.
6. Plug the green connector and the white connector into their respective receptacles located above the probe mount. See Figure 53.

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 60 on page 90.

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 mass detector and disassemble it.

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

- Preparing the LC/MS System for Maintenance
- Clearing Access to the Source Block Assembly
- Removing the Entrance Cone and the Cone Wash Nozzle
- Cleaning the Entrance Cone and the Cone Wash Nozzle
- Removing the Source Block Assembly
- Cleaning the RF/dc Prefilter
- Cleaning the Extraction Cone and the Source Block
- Repairing the Entrance Cone
- Assembling the Source Block Assembly
- 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

The first step in maintaining the source block assembly is to prepare 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 Status page of the Information view in the Xcalibur data system. Right-click the **MSQ Plus** listing to open a shortcut menu, and choose *Turn Device Off* from the shortcut menu.

4. If the MSQ Plus Mass Detector is under vacuum, vent it. Right-click the **Server** icon and choose *Vent* from the shortcut menu.

Venting the MSQ Plus Mass Detector turns off the turbomolecular pump. It does not turn off the Edwards forepump.

5. Wait two or more minutes before proceeding so that the system has time to vent.

CAUTION! It takes approximately two 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 MSQ Plus Mass Detector by placing its MAINS ON/OFF switch to the Off position.

–or–

▶ 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

It is important to clear access to the source block assembly to prevent damage to its parts.

To clear access to the source block assembly

1. To open the front door of the mass 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:

▶ Remove the PEEK fingertight fitting from the probe.

▶ 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.
4. Store the probe heater in the holder to the left of the source compartment.
5. If your MSQ Plus Mass 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

Follow these steps to remove 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 mass detector, as described in “Preparing the LC/MS System for Maintenance” on page 87, and clear the access to the source block, as described in “Clearing Access to the Source Block Assembly” on page 88.

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 60.

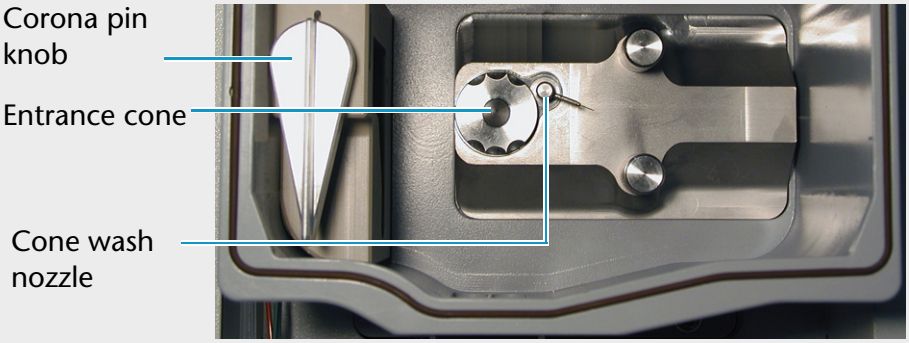


Fig. 60 View of the front side of the source block with the cone wash nozzle turned away from the entrance cone

2. Being careful to handle the cone wash nozzle by its base, rotate the cone wash nozzle away from the entrance cone. See Figure 60.

CAUTION! The tip of the cone wash nozzle is very fragile, so take care to handle the cone wash nozzle by its base.

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

Practical 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 prevents 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, turn it clockwise. To install it, turn it counter-clockwise.

CAUTION! Take care when handling the entrance cone. Always store the entrance cone with its cone facing upwards, as shown in Figure 61.

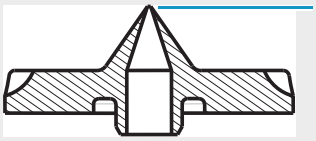


Fig. 61 Entrance cone with cone facing upwards

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, “Removing the Entrance Cone and the Cone Wash Nozzle.”
- For instructions on cleaning the internal components of the source block assembly, go to “Removing the Source Block Assembly” on page 93.

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. Figure 62 shows the location of the O-ring.

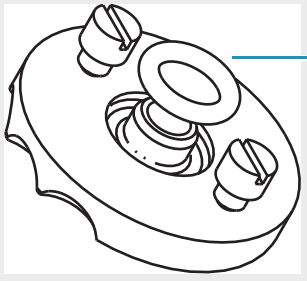


Fig. 62 Rear of the entrance cone, showing its O-ring (BS010)

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. Also inspect the outside of the cone to verify that the opening is circular and has a sharp edge. See “Repairing the Entrance Cone” on page 98 for information on temporarily repairing the cone. See Figure 63.

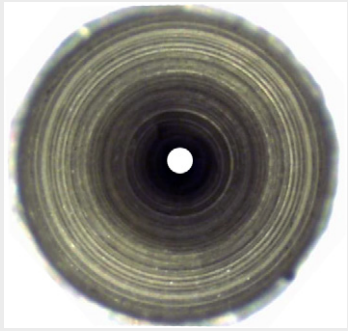


Fig. 63 View of the inside rear of the entrance cone

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.

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 nozzle in a 10% v/v aqueous solution of formic acid, rinse with distilled water, and then rinse with 100% methanol.

Figure 64 shows the cone wash nozzle.

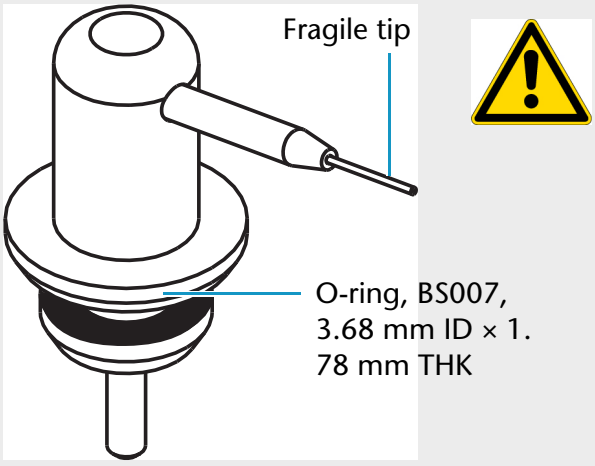


Fig. 64 Cone wash nozzle assembly (part number A66539)

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

This O-ring is difficult to remove and put back. Lubricating the O-ring with methanol might help. It is usually not necessary to remove the O-rings when you sonicate the metal parts.

Practical 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. To determine whether these components need cleaning, look for evidence of ion burn or sample deposit behind the entrance cone.

▶ If the internal components of the source block assembly do not need cleaning, go to step 7.

–or–

▶ 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 60 on page 90.
8. Insert the entrance cone into the source block, and then turn the entrance cone counter-clockwise until it locks in place. See Figure 60.
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.
11. Lock the probe heater in place by turning its black knobs forward 90 degrees.
12. Reinsert the API probe into the probe heater and reattach the PEEK fingertight fitting to the probe.

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 mass 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 87.

▶ Remove the probe and the probe heater, as described in “Clearing Access to the Source Block Assembly” on page 88.

▶ Remove the entrance cone and the cone wash nozzle, as described in “Removing the Entrance Cone and the Cone Wash Nozzle” on page 89.

2. Loosen the thumbscrews on the source block and pull the source block assembly out of the mass detector. See Figure 65.

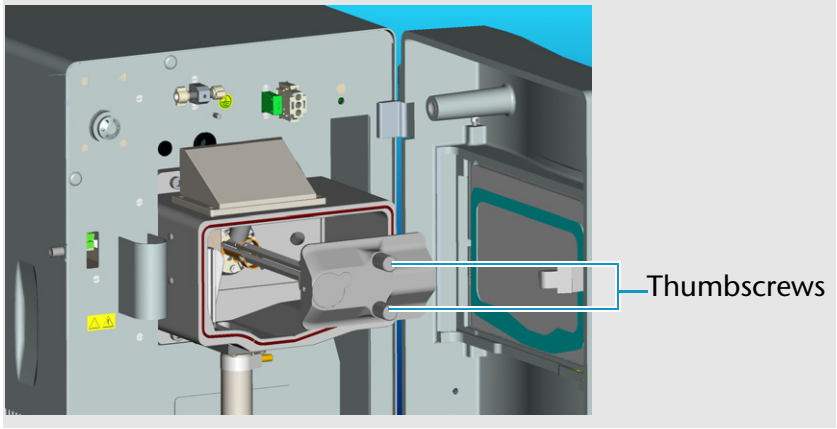


Fig. 65 Source block assembly being removed from the mass detector

Cleaning the RF/dc Prefilter

Refer to Table 4 to determine the frequency at which you need to clean the 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

Table 4. Cleaning schedule for RF/dc pefilter

To clean the RF/dc prefilter

CAUTION! 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. Tools are not required to remove the quadrupole. See Figure 66.

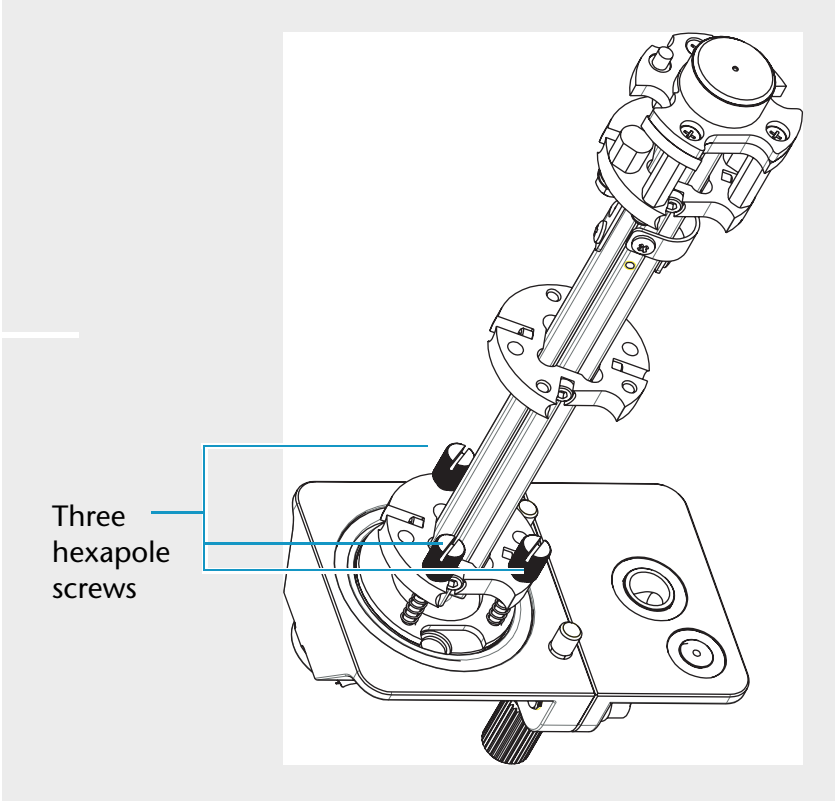


Fig. 66 Source block assembly with the three screws connecting the RF/dc prefilter to the source block

2. Carefully slide the RF/dc prefilter into a 500 mL graduated glass cylinder containing 100% methanol. See Figure 67.

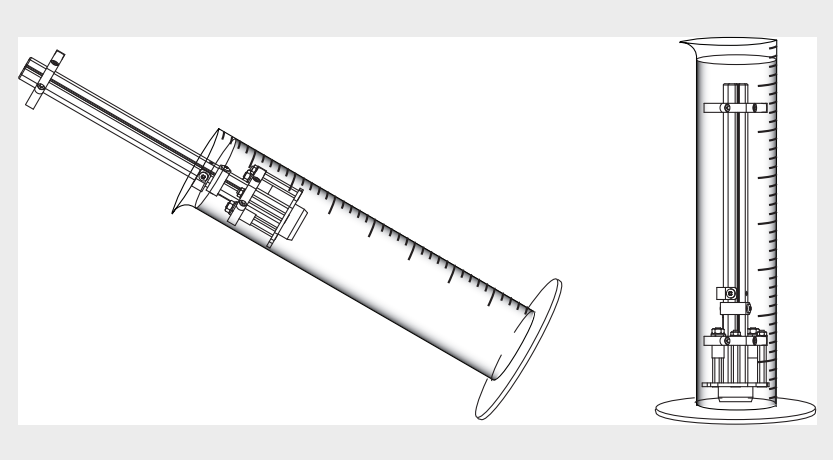


Fig. 67 Cleaning the RF/dc prefilter

3. Sonicate the cylinder for 15 minutes.
4. Carefully remove the RF/dc prefilter from the graduated cylinder. Then dry the prefilter with a gentle stream of nitrogen gas.
5. Reassemble the source block assembly and reinstall it into the mass detector. Pay careful attention to the orientation of the Teflon insulator, which has an indent on one side that should face *up* when assembling the transfer lens. Improper placement of this insulator interrupts the delivery of voltage to the entrance cone, leading to charging of the source block and a decrease in sensitivity.
6. Pump down the system and determine if the RF/dc prefilter is still dirty.

To make this determination, increase the voltage on the lens to see if beam intensity improves. If the prefilter is dirty, the default voltages result in lower-than-expected sensitivity, especially for ions below 200 m/z.

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 the following procedure.

7. 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

1. Immerse the RF/dc prefilter in a graduated cylinder containing 100% distilled water. Sonicate for 15 minutes.
2. Decant the water and fill the graduated cylinder with 100% methanol. Sonicate for 15 minutes.

3. Decant the methanol and fill the graduated cylinder with 100% acetone. Sonicate for 15 minutes.
4. Carefully remove the RF/dc prefilter from the cylinder. Rinse the RF/dc prefilter with methanol.
5. Dry the prefilter with a gentle stream of nitrogen gas.
6. If the differential aperture plate is contaminated with ion burn, clean it:

▶ Unscrew the three screws that connect the differential aperture plate to the quadrupole. See Figure 68.

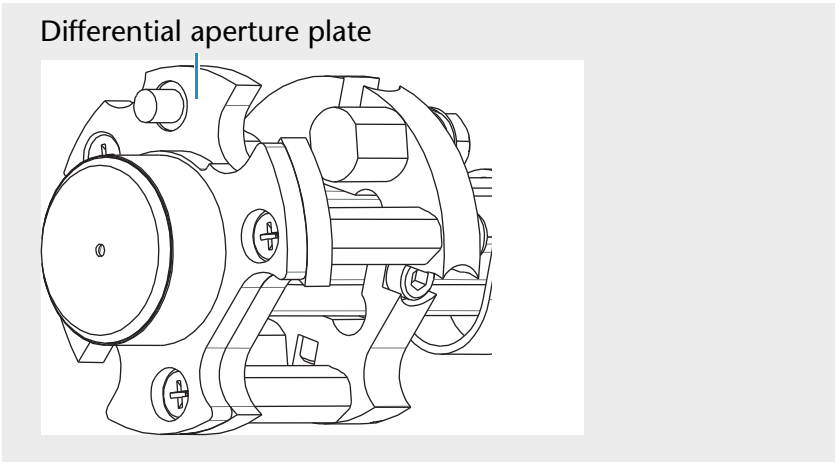


Fig. 68 View of differential aperture plate

▶ Wipe the inside of the differential aperture plate with a cotton swab soaked in [50:50] methanol/water.

▶ Reconnect the differential aperture plate to the quadrupole.

▶ 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 99.

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:

▶ Remove the hexapole screw insulator, extraction cone, and extraction cone insulator. See Figure 69.

Note: Check the source block screws to ensure that they are tight, but do not remove them.

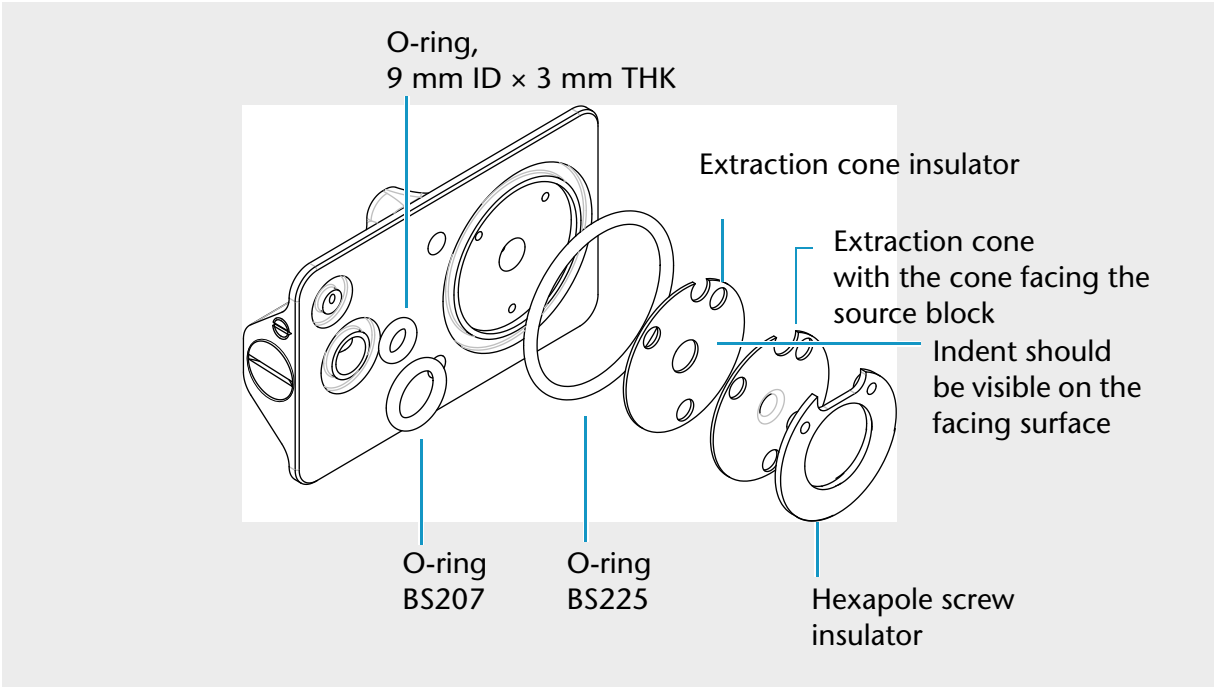


Fig. 69 Exploded view of the source block and its components

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

▶ Remove the three O-rings:

- Top-left O-ring, 9.12 mm ID × 3.53 mm THK, viton black part of **MSQ Plus Hardware and O-ring Kit** (A66548). This O-ring seals the exit cone wash.
- Bottom-left O-ring, BS207, viton black part of **MSQ Plus Hardware and O-ring Kit** (A66548).
- Right O-ring, BS225, viton black part of **MSQ Plus Hardware and O-ring Kit** (A66548).

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

2. To clean the source block, sequentially sonicate it in the following solvents:

▶ 1% v/v solution of formic acid/distilled water

▶ 100% distilled water

▶ 100% methanol

3. To clean the extraction cone:

▶ 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.

▶ Fill the beaker with 10% v/v solution of formic acid, and then sonicate for approximately 15 minutes.

▶ Decant the formic acid, fill the beaker with methanol, and then sonicate again for approximately 15 minutes.

▶ Examine the entrance cone under magnification to be certain that the entrance is circular with sharp edges. If the entrance cone is damaged, you might be able to repair the damage, using the technique in “Repairing the Entrance Cone.”

Repairing the Entrance Cone

You can temporarily repair the MSQ Plus Mass Detector entrance cone (part number A66357) pending replacement of the entrance cone.

To temporarily repair the entrance cone

1. Remove the entrance cone and clean according to standard procedures: sonicate in water and then methanol, and blow dry with compressed air or nitrogen.
2. Using a microscope set to 30x magnification, examine the cone.

The tip must be circular with a sharp edge. The inner diameter specification is 300 +/- 10 μM.

3. If the cone has been damaged and is no longer circular, you can use the corona pin needle (part number A66515) by gently inserting the needle from the rear of the cone until it makes contact. Slowly roll the needle back and forth, applying light pressure. Re-examine the cone under magnification.

The steel pin should be able to recircularize the titanium cone, but it might leave a ragged and somewhat larger opening. You can polish the ragged edge of the cone to a sharp circular rim by using 12 uM grit sandpaper or similar abrasive.

4. Clean the repaired entrance cone and attach it to the MSQ Plus Mass Detector system.

The cone inner diameter is likely to be somewhat larger than the specification.

5. Test the cone for sensitivity, using erythromycin or another test compound with known performance specifications.

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 69 on page 98):

- Top left O-ring, 9.12 mm ID × 3.53 mm THK, viton black part of **MSQ Plus Hardware and O-ring Kit** (A66548)
- Bottom left O-ring, BS207, viton black part of **MSQ Plus Hardware and O-ring Kit** (A66548)
- Right O-ring, BS225, viton black part of **MSQ Plus Hardware and O-ring Kit** (A66548)

2. Install the extraction cone insulator (part number A66556).
3. Verify proper orientation by checking for the indent on the facing surface.
4. Install the extraction cone (part number A66553) with the cone facing the source block.
5. Install the hexapole screw insulator (part number A66557). Be certain that this part is free from contamination, particularly from metal filings that might result from the action of the hexapole screws on the source block. See Figure 69 on page 98.
6. Ensure that the semicircular cutouts in these three components line up, as shown in Figure 70.

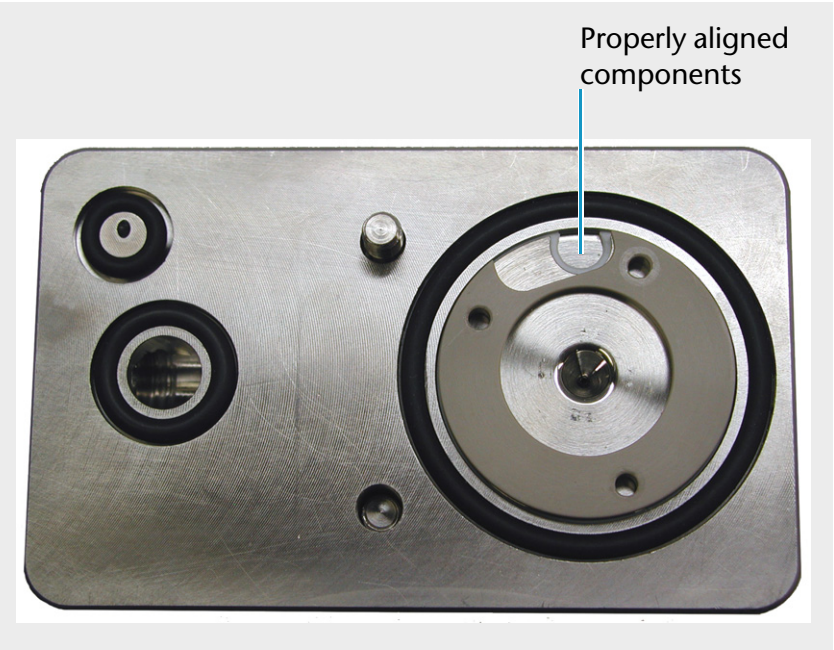


Fig. 70 Source block with the proper alignment of the extraction cone insulator, extraction cone, and hexapole insulator

7. Align the three spring screws at the base of the RF/dc prefilter to the three holes on the rear 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. It might help to remove the thumbscrews from the source block, allowing the block to sit upright on a flat surface. See Figure 66 on page 95.

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 71.

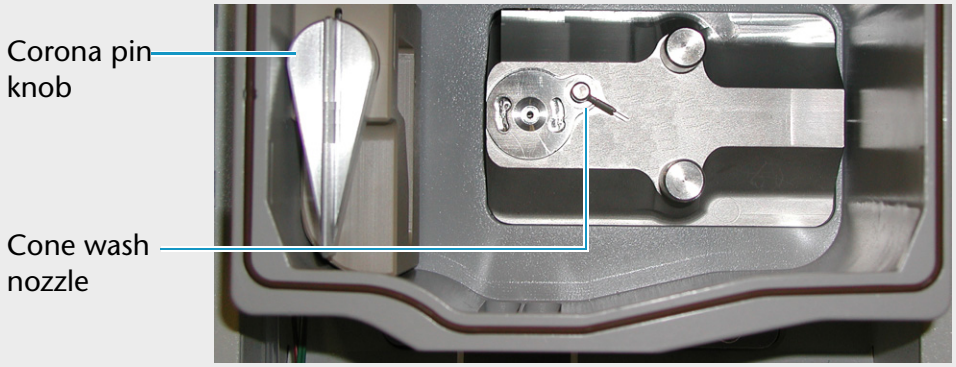


Fig. 71 Cone wash nozzle turned to the right in the source block

To install the source block assembly into the mass detector

1. Insert the source block assembly (part number A66512) into the source block enclosure, and tighten the thumbscrews on the source block.

The source block is self-aligning when the thumbscrews are fully secure. Finger-tighten only. See Figure 65 on 93.

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 (part number A66539) into the source block, and carefully rotate the nozzle tip to the right. Lubricating the O-ring with a little methanol helps the insertion process. See Figure 71.
3. Remove the lint-free cloth from the bottom of the source enclosure.
4. Insert the entrance cone into the source block, and turn it counter-clockwise. See page 90.

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

5. Install the probe heater:

▶ Remove the probe heater from its holder, and rotate the black knobs of the probe heater so that they face away from each other. See Figure 54 on page 82.

▶ Insert the tube portion of the probe heater into the probe mount. Be certain that the two small O-rings are still in place on the probe mount.

▶ Rotate the black knobs of the probe heater forward to their locking position. See Figure 52 on page 82.

6. Install the API probe:

▶ Turn the locking plate clockwise to the open position, and carefully insert the ESI probe (part number A66514) (see Figure 34 on page 65) or the APCI probe (part number A66513) (see Figure 46 on page 76) into the probe heater.

Note: 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

▶ Turn the locking plate counterclockwise into the closed position.

▶ Screw the PEEK fingertight fitting into the ESI probe assembly (part number A66514) or APCI probe assembly (part number A66513).

7. Pump down the mass detector:

▶ Right-click the *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.

▶ Choose *Pump* from the shortcut menu.

▶ Wait for the MSQ Plus Mass 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 MSQ Plus Mass 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 MSQ Plus Mass 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 72. 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.

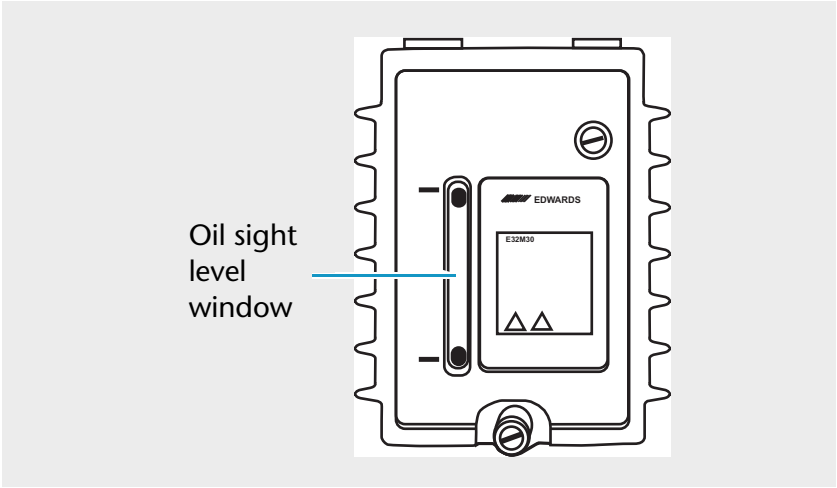


Fig. 72 View of Edwards forepump that shows the oil sight level window

- 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 55.

Maintaining the Turbomolecular Pump

A turbomolecular pump creates the vacuum for the mass detector. The lubricant reservoir of this pump may occasionally need to be replaced to keep the pump operating optimally.

Removing the Turbomolecular Pump Lubricant Reservoir

Follow the steps in this section to remove the existing lubricant reservoir before replacing it. Figure 73 shows the parts and tools involved in replacing the lubricant reservoir of the turbomolecular pump.

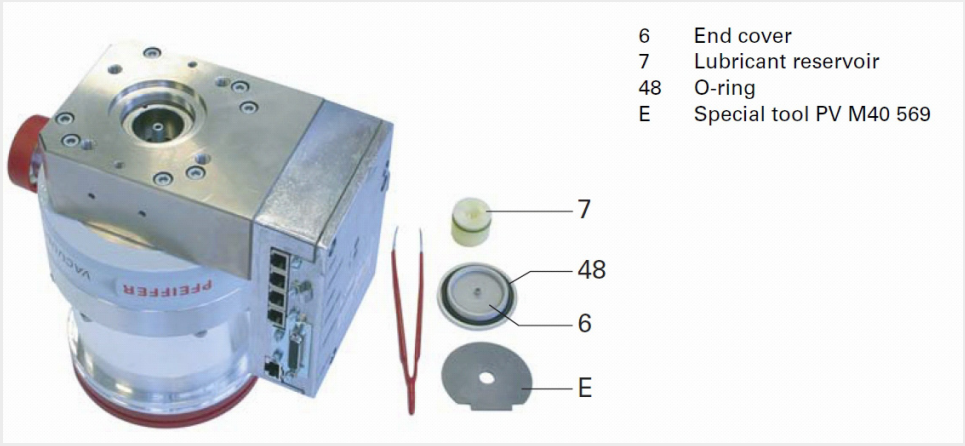


Fig. 73 Parts needed to replace the lubricant reservoir of the turbomolecular pump[1]

To remove the turbomolecular pump lubricant reservoir

CAUTION! Always wear protective gloves. Contaminants in the oil are extremely hazardous.

1. Leave the system pumping for at least a half an hour before starting the removal procedure.
2. Vent the mass detector.
3. Using a No. 1 point Posidrive™ screwdriver, remove the pump-access plate-securing screws.
4. Remove the access plate.
5. Using the supplied locking cap removal tool, shown in Figure 74, unscrew the locking cap by turning the tool and cap counter-clockwise.

1. Image of the turbomolecular pump from the *Compact Turbo™ TurboDrag Pump TMH/TMU 261 Manual* PM 0470 BE/O (0709) by Pfeiffer Vacuum.

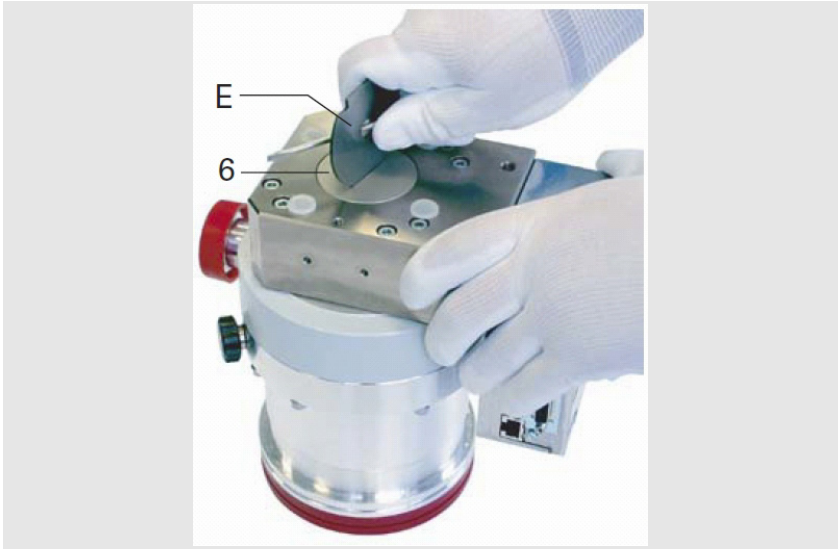


Fig. 74 Removing the locking cap[1]

6. Remove the cap, including the O-ring, and wipe with a clean, lint-free cloth. Place in a secure location.
7. Using a small flat-blade screwdriver or tweezers, gently pry out the lubricant reservoir, including the O-ring, as shown in Figure 75.

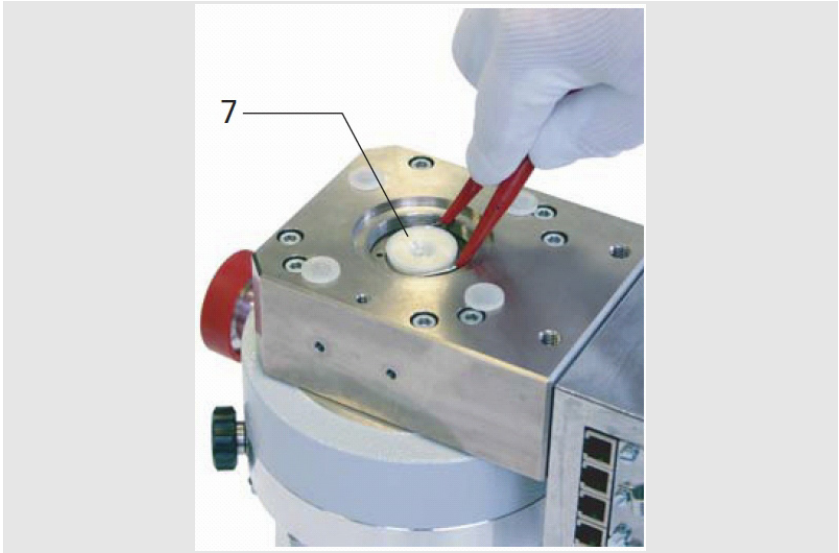


Fig. 75 Removing the lubricant reservoir[2]

8. Lift the reservoir and dispose of it safely.
9. Using a clean, lint-free cloth, remove any dirt from the opening.

---

1. Image of the turbomolecular pump from the *Compact Turbo™ TurboDrag Pump TMH/TMU 261 Manual* PM 0470 BE/O (0709) by Pfeiffer Vacuum.
2. Image of the turbomolecular pump from the *Compact Turbo™ TurboDrag Pump TMH/TMU 261 Manual* PM 0470 BE/O (0709) by Pfeiffer Vacuum.

Replacing the Turbomolecular Pump Lubricant Reservoir

This procedure explains how to replace the lubricant reservoir of the turbomolecular pump.

To replace the turbomolecular pump lubricant reservoir

1. Insert the new reservoir (part number A66536), into the opening.
2. Replace the O-ring and cap. Use a new O-ring, if necessary.
3. Tighten the cap, using the supplied locking cap removal tool, turning it clockwise. Do not over-tighten the cap.
4. Replace the access plate:

- Insert the left-hand side lug first.
- Ensure that the PEEK tubing is located in the appropriate grooves.
- Insert the bottom lug,.
- Insert the right-hand side lug.

System Shutdown

When you are not performing analyses, you can temporarily turn off the nitrogen gas or set the MSQ Plus Mass 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 <Hypertext>”Routine and Preventive Maintenance,” 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, KNAUER recommends that you perform a full-system autotune.

Shutting Down the System in an Emergency

If you need to turn off the mass detector in an emergency, place the main power circuit breaker switch in the Off (O) position, as shown in Figure 76. The main power switch, which is labeled MAINS ON/OFF, is located on the rear panel of the mass detector in the lower-right quadrant. Turning the main power switch to the Off position turns off all power to the mass detector, including the forepump. Although removing power abruptly does not harm any component within the system, it is not the recommended shutdown procedure to follow. Refer to “Shutting the System Down for Non-Routine Maintenance” on page 111 for the recommended procedure.

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

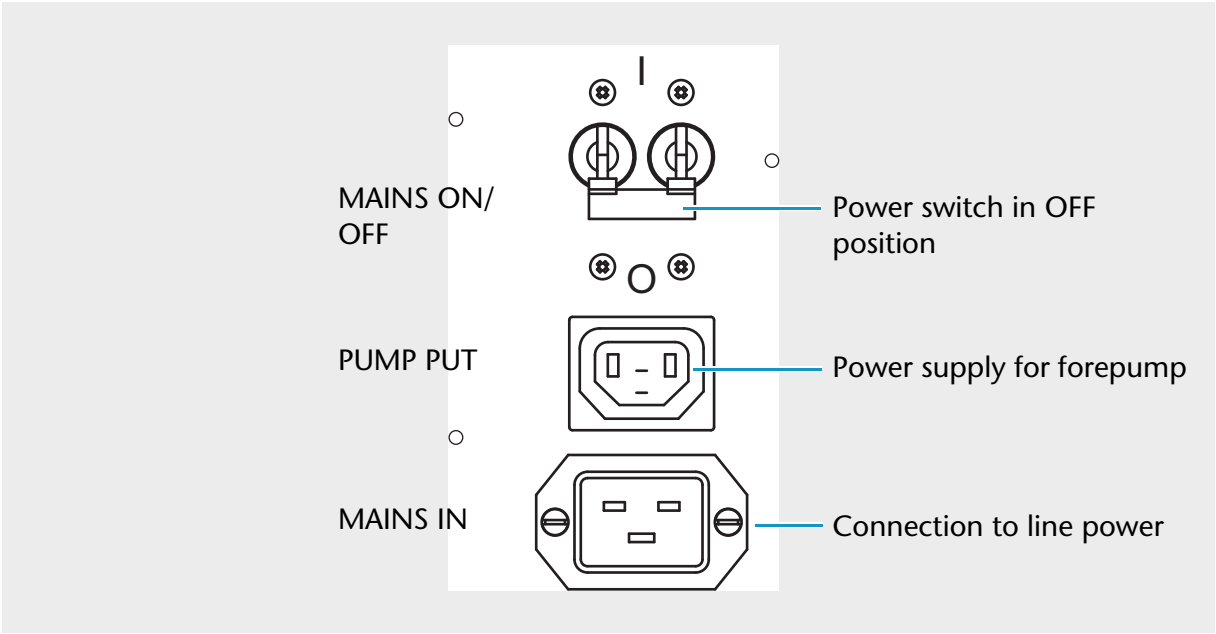


Fig. 76 MAINS ON/OFF circuit breaker switch

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 the external cone wash pump switch to the Off position.
2. Turn off the flow from your LC pump:

▶ 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 Status page of the Information view of the Xcalibur data system.

▶ 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:

▶ Double-click the **Tune** icon,  , on the Windows desktop.

The Server icon now appears in the System tray (to the left of the clock) of the Windows taskbar.

▶ 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 gases from back-streaming from the waste solvent bottle.

Placing the System in the Off Mode

Place the MSQ Plus Mass 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 MSQ Plus Mass 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 MSQ Plus Mass 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 MSQ Plus Mass Detector in the Off mode, turn off the flow from the LC pump and the flow from the optional cone wash pump.

Turn the mass detector off from the Status view in the Xcalibur data system or from the Tune window.

Turning Off the Mass Detector from the Xcalibur Data System

One way to turn off the MSQ Plus Mass Detector is to use the Status page of the Information view of the Xcalibur data system.

To turn off the MSQ Plus Mass Detector from the Xcalibur Status page

1. If you are using the optional cone wash pump, turn it off by turning its power switch to the Off position.
2. If the Xcalibur data system is not open, open it by double-clicking the **Xcalibur** icon,  , on the Windows desktop.
3. If the Information 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 and choose *Turn Device Into Standby* from the shortcut menu shown in Figure 77.

–or–

▶ If your LC pump is not controlled from the Xcalibur data system, turn it off from its control keypad.

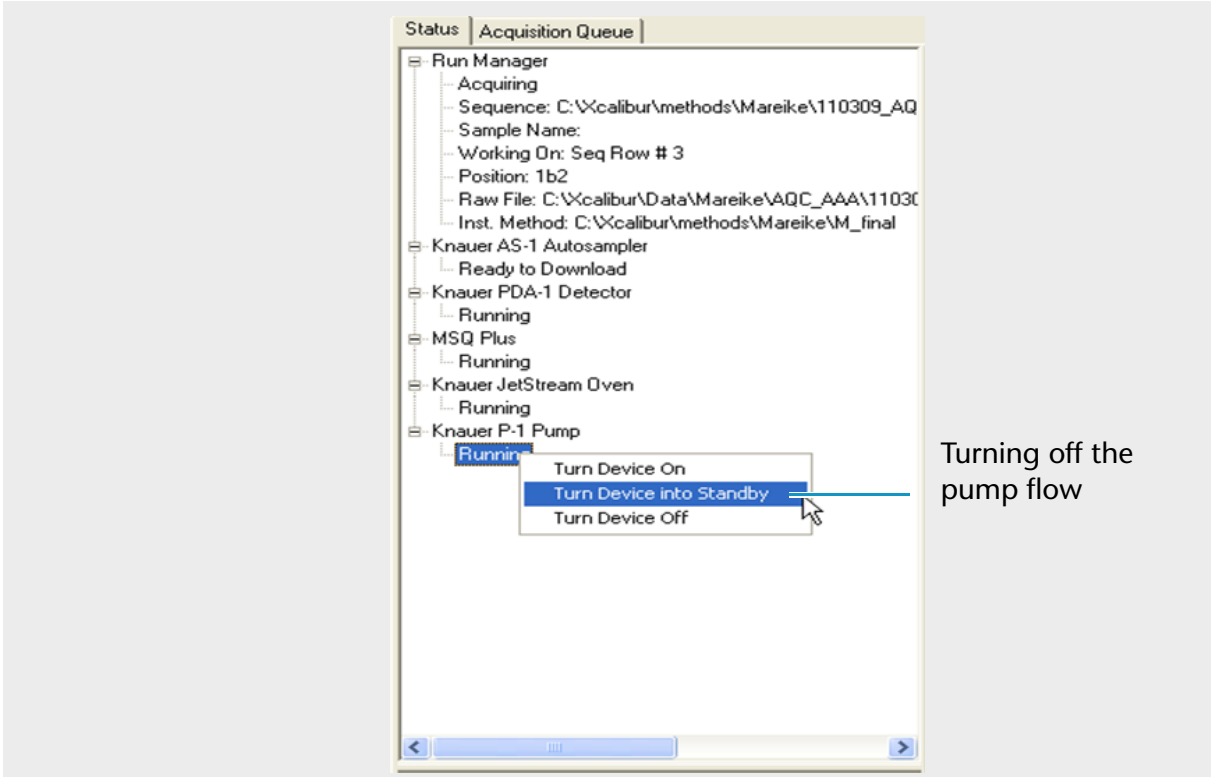


Fig. 77 Status of the UHPLC MS pump and its shortcut menu shown on the Status page of the Information view

5. Right-click the **MSQ Plus** listing on the Status page, and choose *Turn Device Off* from the shortcut menu to place the mass detector in the Off mode, as shown in Figure 78.

The status LED on the front of the MSQ Plus Mass Detector turns yellow.

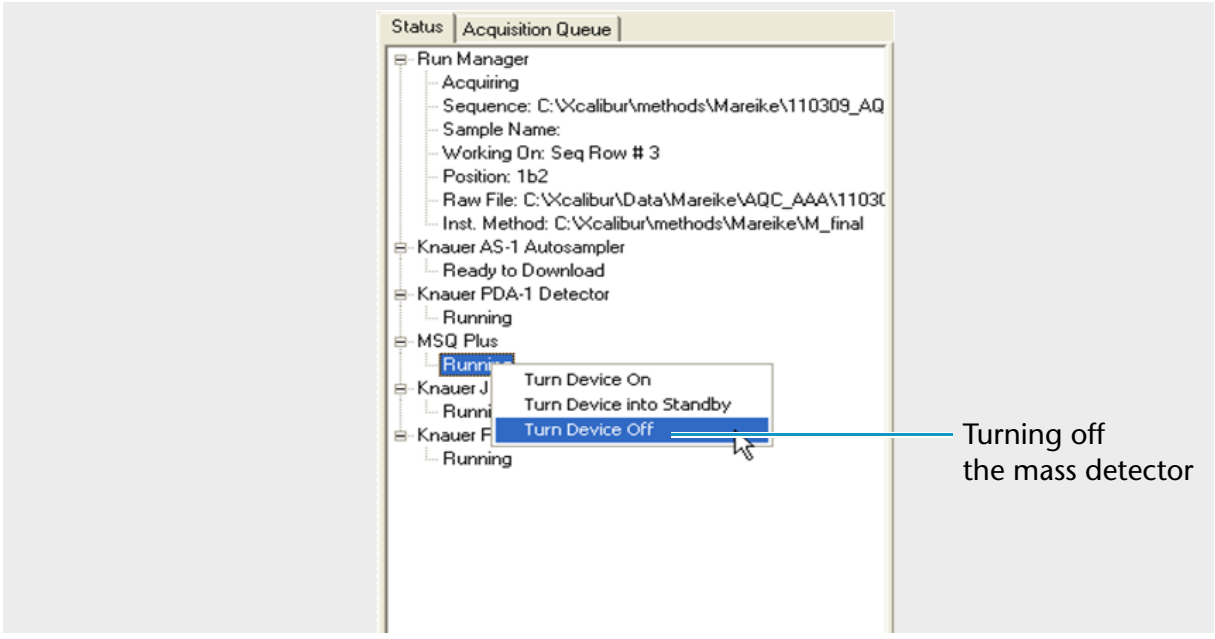


Fig. 78 Shortcut menu for the MSQ Plus Mass Detector on the Status page of the Information view

Turning Off the Mass Detector from the Tune Window

Another way to turn off the MSQ Plus Mass Detector is to use the Tune window.

To turn off the MSQ Plus Mass 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 the LC pump:

▶ 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 Status page of the Information view.

–or–

▶ 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:

▶ Double-click the **Tune** icon,  , on the Windows desktop.

The Server icon appears in the Windows taskbar.

▶ 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 MSQ Plus Mass 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

You might need to shut down the MSQ Plus Mass Detector for a non-routine maintenance procedure or to relocate the instrument.

To shut down the MSQ Plus Mass Detector

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* > *Programs* > *Thermo Xcalibur* > *Xcalibur* from the Windows desktop.
3. If the Information view is not displayed, choose *View* > *Info View* to display it.
4. Turn off the flow from the LC pump:

- 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.
- If your LC pump is not controlled from the Xcalibur data system, turn it off from its keypad control panel.

5. Right-click the **MSQ Plus** listing on the Status page, and choose *Turn Device Off* from the shortcut menu.
6. Right-click the **Server** icon in the system tray of the Windows taskbar, and 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, as shown in Figure 79.

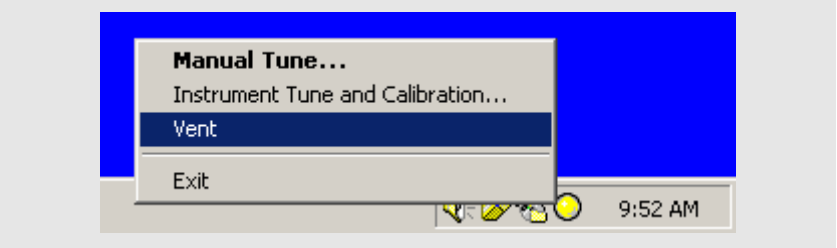


Fig. 79 View of taskbar showing the Server icon and the shortcut menu

7. Exit the Xcalibur data system, and close the server.
8. Wait for approximately two minutes, and turn off the MSQ Plus Mass Detector by setting the MAINS IN switch to the Off position.

Turning off the power to the MSQ Plus Mass 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 mass 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 5, and then follow the system start-up procedure.

After you complete these procedures, the system is ready for a full-system autotune. Refer to the *MSQ Plus Mass Detector Startup Guide* for details.

Checking the System Connections

Before you switch on the system after an extended shutdown period, a major overhaul, or instrument relocation, perform the visual checks on the system listed in table 5.

Items	Check
Power Connections	
The MSQ Plus Mass 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 MSQ Plus Mass Detector.	
Communication Connections	
MSQ Plus Mass 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	
The GAS IN port on the rear panel of the MSQ Plus Mass Detector is connected to a nitrogen supply, and the auxiliary regulator is set to 75 psi (5.2 bar).	
Any gas connections required for the LC system have been made.	
Vacuum Connections	
The source manifold on the rear panel of the MSQ Plus Mass Detector is connected to the forepump.	
The backing manifold on the rear panel of the MSQ Plus Mass Detector is connected to the forepump.	
Exhaust Connections	

Table 5. Pre-switch on checklist (Sheet 1 of 2)

Items	Check
The Exhaust manifold on the rear panel of the MSQ Plus Mass 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	
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 MSQ Plus Mass Detector.	
For the UHPLC, 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.	

Table 5. Pre-switch on checklist (Sheet 2 of 2)

Restarting the MSQ Plus Mass Detector

Follow these steps to start the MSQ Plus Mass Detector.

To start the MSQ Plus Mass Detector

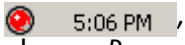
1. Turn on the power for your system:

▶ Turn on the power to the MSQ Plus Mass Detector by toggling the **MAINS IN** switch to the On position.

▶ Turn on the power to your LC devices. Wait for the LC devices to complete their initialization before proceeding.

▶ 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:

▶ Right-click the **Server** icon, 

, in the system tray of the Windows taskbar, and choose *Pump* from the shortcut menu.

- ▶ Wait for the MSQ Plus Mass 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.

If the MSQ Plus Mass Detector has not reached vacuum after 30 minutes, the server light might still be red or flashing amber. Refer to Table 1 on page 28 to 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, as shown in Figure 80.

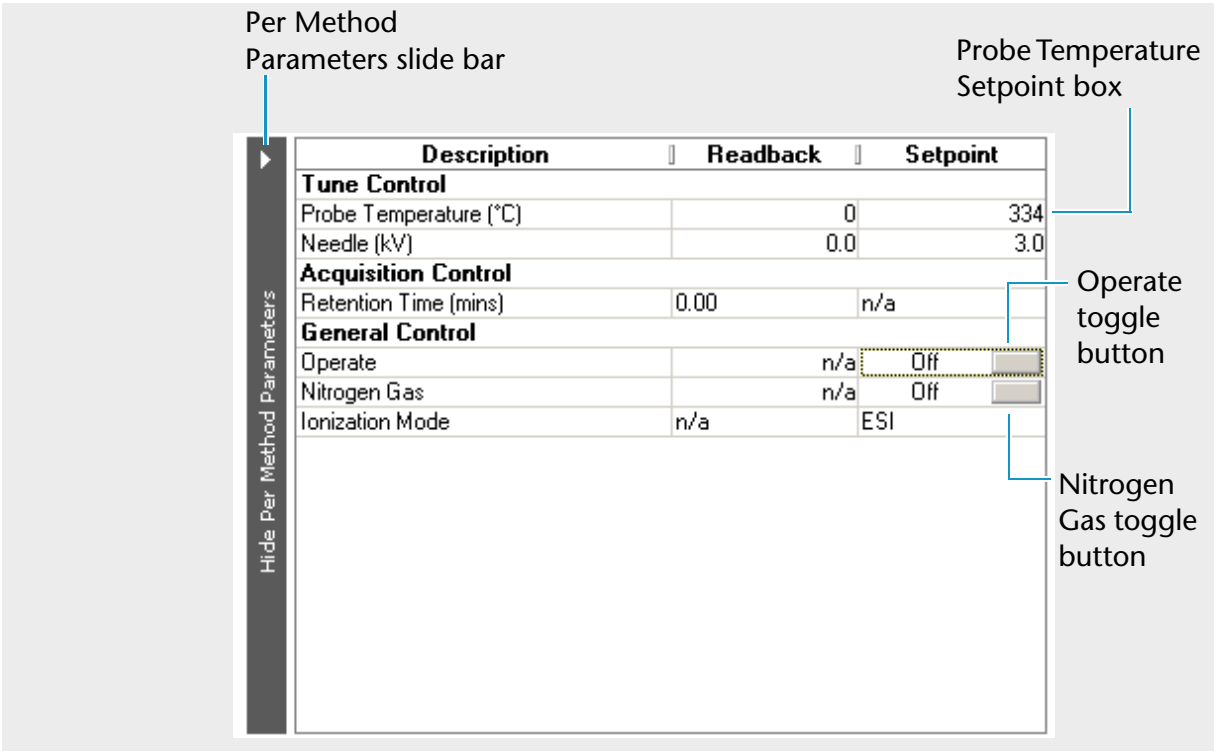


Fig. 80 Per Method Parameters table

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, shown in Figure 80.

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 shown in Figure 80.

The button turns from gray to green, and the text changes from Off to On.

8. To set the probe temperature, click the Probe Temperature Setpoint box shown in Figure 80, and type an appropriate value for your application.

The MSQ Plus Mass Detector is ready to use as soon as the probe temperature readback value approaches that in the setpoint box, although for most stable operation KNAUER recommends that you wait approximately 10 minutes for the source to equilibrate. There is an allowable 2-5% tolerance on the readback.

9. Turn on the flow from the LC pump.

Resetting the MSQ Plus Mass Detector

If communication between the mass detector and data system computer is lost, it might be necessary to reset the mass detector by using the Reset button on the power panel. Pressing the Reset button creates an interrupt on the CPU PCB of the embedded computer, causing the embedded computer to restart into a known (default) state. You might hear a high or low tone that confirms the reset when you open the Tune window.

The procedure given here assumes that the mass detector and data system computer are both powered on and operational. If the mass detector, data system computer, or both are off, refer to “Restarting the MSQ Plus Mass Detector” on page 113.

To reset the mass detector, press the **Reset** button located on the mass detector’s rear panel, as shown in Figure 81.

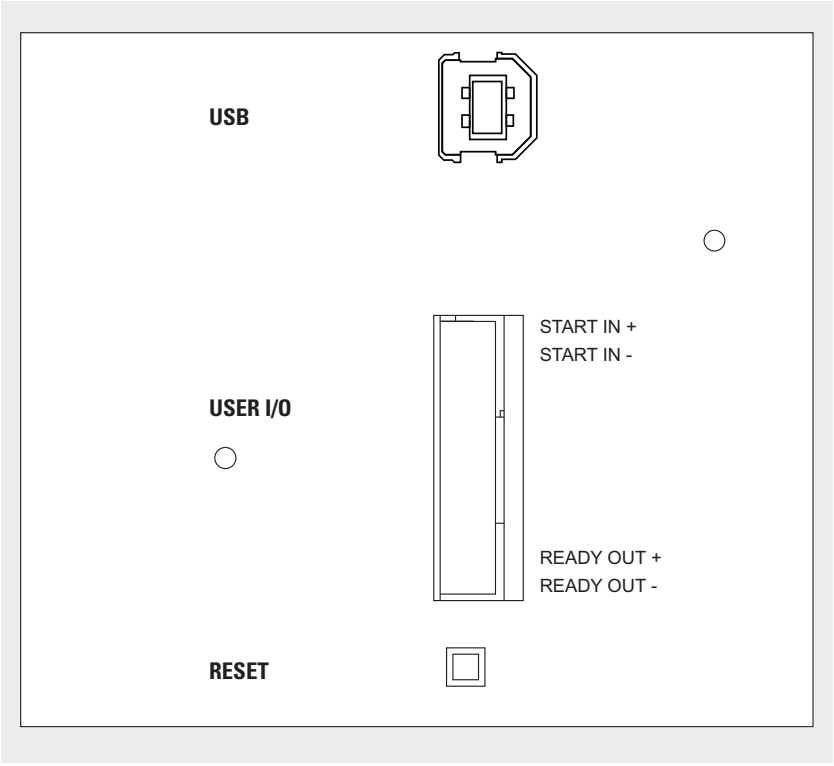


Fig. 81 View of the upper right-hand corner of the mass detector’s rear panel

Replaceable Parts

This chapter lists the parts most commonly used in the course of working with and maintaining your MSQ Plus Mass Detector.

The parts are categorized as follows

- Consumables. Keep a stock of each of these parts, because you might need to replace them frequently.
- Spares. You can order these parts as required.
- Communication Kit. Use these interface kit to connect an LC system to your mass detector.

The manuals for the MSQ Plus Mass Detector are provided on the software CD.

Consumables

The MSQ Plus Annual Maintenance kit (part number A66535) contains all the consumables required for the upkeep of your MSQ Plus Mass Detector. The parts contained in this kit are listed in Table 6 on page 116.

Item	Part number
MSQ Plus Annual Maintenance kit	A66535
Adaptor (capillary retainer nut)	A66546
APCI probe capillary (3 each)	A66541
Aqua nozzle assembly (cone wash nozzle) (1 each)	A66539
Entrance skimmer assembly, titanium (1 each)	A66537
ESI probe capillary (3 each)	A66542
ESI ceramic sleeve (1 each)	A66543
Exit cone (extraction cone) (1 each)	A66553
Ferrule, SGE 1/16 graphite vespel (10 per package)	A66538
Kit, hardware and O-ring, MSQ Plus	A66548
Kit, heater repair	A66545
Oil reservoir 190/240/260 for turbomolecular pump	A66536
Turbomolecular pump oil change tool	A66547
Tube insert for API probe, PEEK (1 each)	A66540

Table 6. Parts in the MSQ Plus Annual Maintenance Kit (Sheet 1 of 2)

Item	Part number
Heater Repair Kit	A66545
Detent screw insulator	
Screw sleeve	
Spring screw	
Spring cup	
MSQ Plus Hardware and O-ring Kit	A66548
Sealing plug (2 each)	
Ferrule, SGE 1/16 graphite vespel (10 each)	
Spring, E-type clip, stainless steel, MSQ only (3 each)	
Spring, compression, 4.6 mm OD, 0.45 N/mm, 30 mm length	
O-ring, 13.87 mm ID × 3.53 mm THK, BS207[a], BLK VITON (1 each)	
O-ring, 3.68 mm ID × 1.78 mm THK, BS007, BLK VITON (2 each)	
O-ring, 3.0 mm ID × 1.0 mm THK, BLK VITON (2 each)	
O-ring, 9.25 mm ID × 1.78 mm THK, BS012, VITON, for entrance cone(2 each)	
O-ring, 12.42 mm ID × 1.78 mm THK, BS014, BLK VITON (1 each)	
O-ring, 9.0 mm ID × 3.0 mm THK, BLK VITON (1 each)	
O-ring, 3.30 mm ID × 2.4 mm THK, BLK VITON (2 each)	
O-ring, 47.22 mm ID × 3.53 mm THK, BS225, BLK VITON (1 each)	
O-ring, 6.07 mm ID × 1.78 mm THK, BS010, BLK VITON (2 each)	
O-ring, 18.77 mm ID × 1.78 mm THK, BS018, BLK VITON (2 each)	
O-ring, 2.90 mm ID × 1.78 mm THK, BS5006, BLK VITON (2 each)	
O-ring, 5.28 mm ID × 1.78 mm THK, BS009, BLK VITON (2 each)	

Table 6. Parts in the MSQ Plus Annual Maintenance Kit (Sheet 2 of 2)

a. British Standard

Figure 82 shows the location of the O-rings.

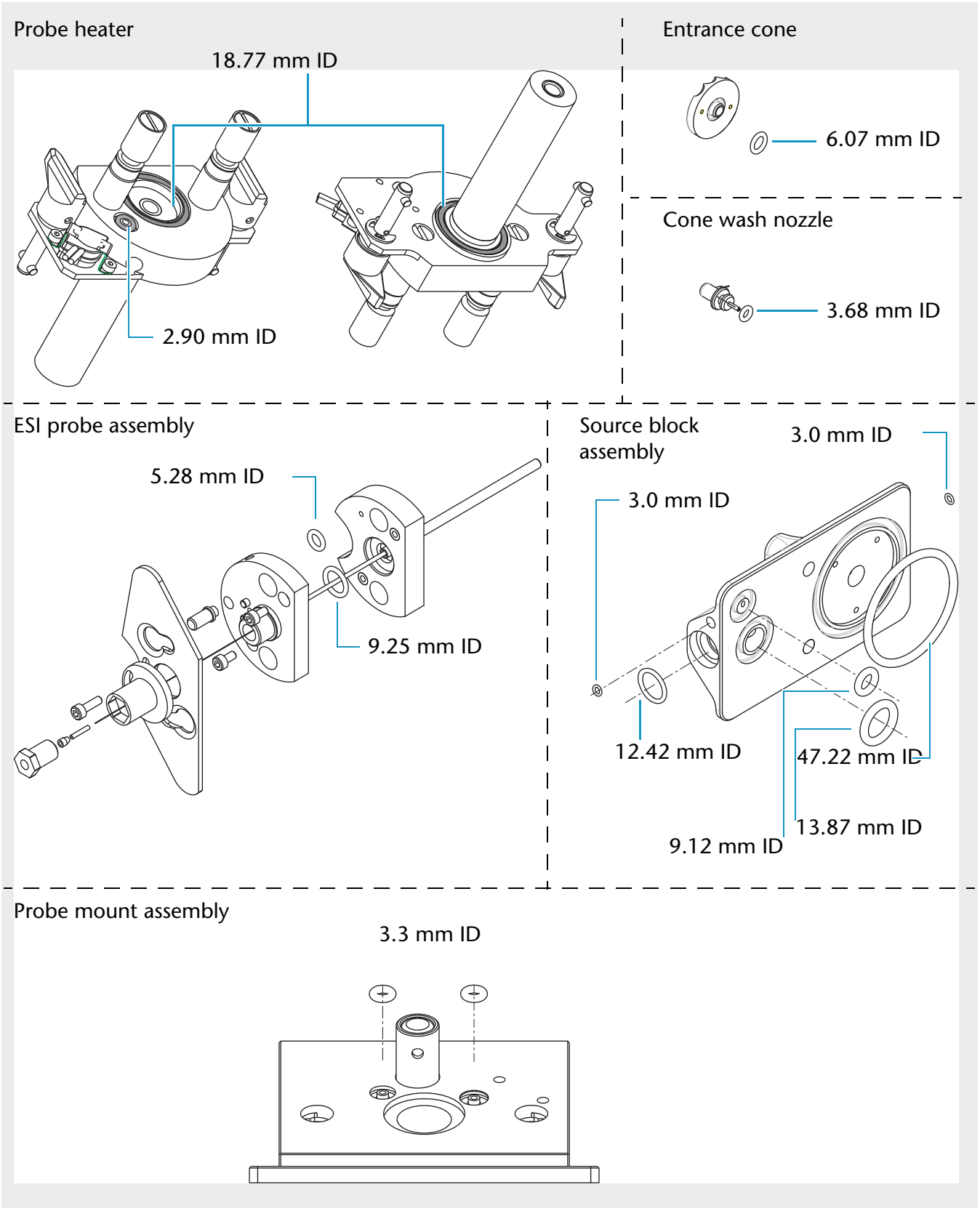


Fig. 82 Location of O-rings

Spares

Order the spare parts shown in table 7 and kits as required.

Item	Part number
Assembly, probe heater	A66517
Cable, USB, A to B (2 m)	A66526
Electron multiplier	A66511
Digital board	A66503
Entrance, cone, titanium (with O-ring and bayonet pins)	A66357
Fitting, Swagelok tube fitting, stainless steel female ISO tapered thread connector for 1/4 in. OD tubing	A66530
Fitting, pipe, 6 mm × 1/4 in. NPT	A66532
Hexapole screw insulator	A66557
Needle, corona, MSQ Plus (for APCI mode)	A66515
Source board, sub-assembly standard with bracket	A66504
Hexapole screws (Nitronic 60 alloy)	
Tubing, 6 mm OD, PTFE, for nitrogen line, (order by the foot length)	A66521

Table 7. General spare parts

Kits

Table 8 lists the kits available.

Item	Part number
MSQ engineer tool kit	A66518
MSQ Plus installation kit	A66519
MSQ sensitivity test kit	A66523

Table 8. Kits

Source Block Assembly

Table 9 lists the parts in the source block assembly.

Item	Part number
Assembly, source block and transfer lens	A66512
Assembly, titanium entrance cone (with O-ring and bayonet pins)	A66357
Assembly, cone wash nozzle (includes O-ring)	A66539
CAP kit	A66555
Exit cone (extraction skimmer)	A66553
Exit cone insulator (extraction cone insulator)	A66556
Hexapole screw insulator	A665567
O-ring, 6.07 mm ID × 1.78 mm THK, BS010, BLK VITON (for entrance cone)	part of MSQ Plus Hardware and O-ring kit (A66548)
O-ring, 3.0 mm ID × 1.0 mm THK, BLK VITON (for sealing plug)	part of MSQ Plus Hardware and O-ring kit (A66548)
O-ring, 13.87 mm ID × 3.53 mm THK, BS207, BLK VITON	part of MSQ Plus Hardware and O-ring kit (A66548)
O-ring, 12.42 mm ID × 1.78 mm THK, BS014, BLK VITON	part of MSQ Plus Hardware and O-ring kit (A66548)
O-ring, 9.0 mm ID × 3.0 mm THK, BLK VITON	part of MSQ Plus Hardware and O-ring kit (A66548)
O-ring, 47.22 mm ID × 3.53 mm THK, BS225, BLK VITON	part of MSQ Plus Hardware and O-ring kit (A66548)
O-ring, 3.68 mm ID × 1.78 mm THK, BS007, BLK VITON (for cone wash nozzle)	part of MSQ Plus Hardware and O-ring kit (A66548)
Plug, source block sealing	A66544
Screw, M3 × 10, cap head, stainless steel	A66552
Screw, hexapole	A66559
Spring, transfer lens special	A66558

Table 9. Source block assembly parts

Figure 83 shows an exploded view of the source block assembly with component descriptions and part numbers.

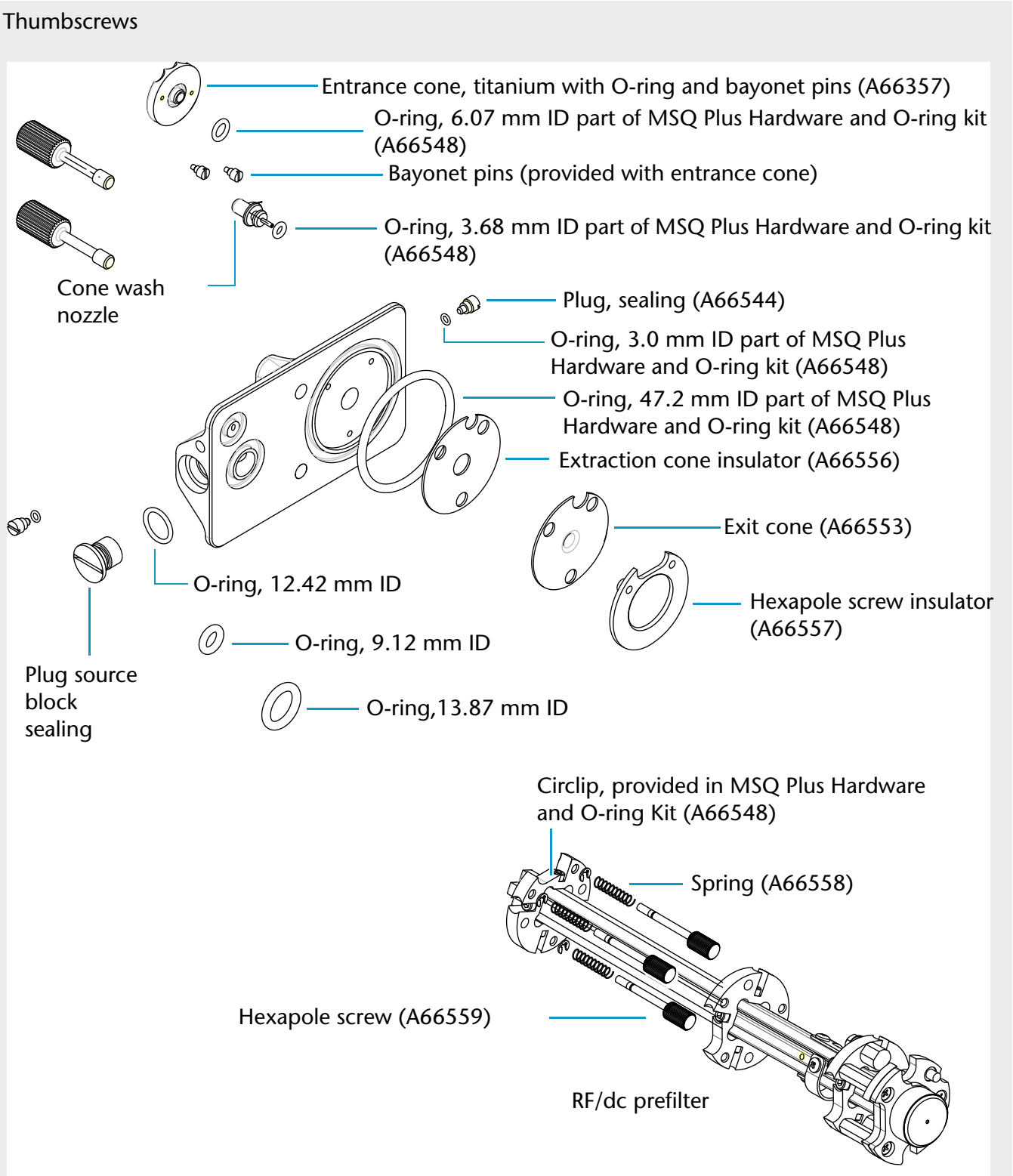


Fig. 83 Exploded view of the source block assembly

ESI Probe Assembly

Table 10 lists the parts in the ESI probe assembly.

Item	Part number
Assembly, ESI probe	**A66514**
ESI probe capillary	A66542
ESI ceramic sleeve	A66543
Ferrule, GVF/16, graphite vespel	A66538
O-ring, 9.25 mm ID × 1.78 mm THK, BS012, BLK VITON	part of MSQ Plus Hardware and O-ring kit (A66548)
O-ring, 5.28 mm ID × 1.78 mm THK, BS009, BLK VITON	part of MSQ Plus Hardware and O-ring kit (A66548)
PEEK tube insert	A66540
Screw, M3 × 8, cap head, stainless steel	N/A
Screw, M3 × 10, cap head, stainless steel	A66552
Capillary retaining nut	A66546

Table 10.ESI probe assembly parts

Figure 84 shows an exploded view of the ESI probe assembly.

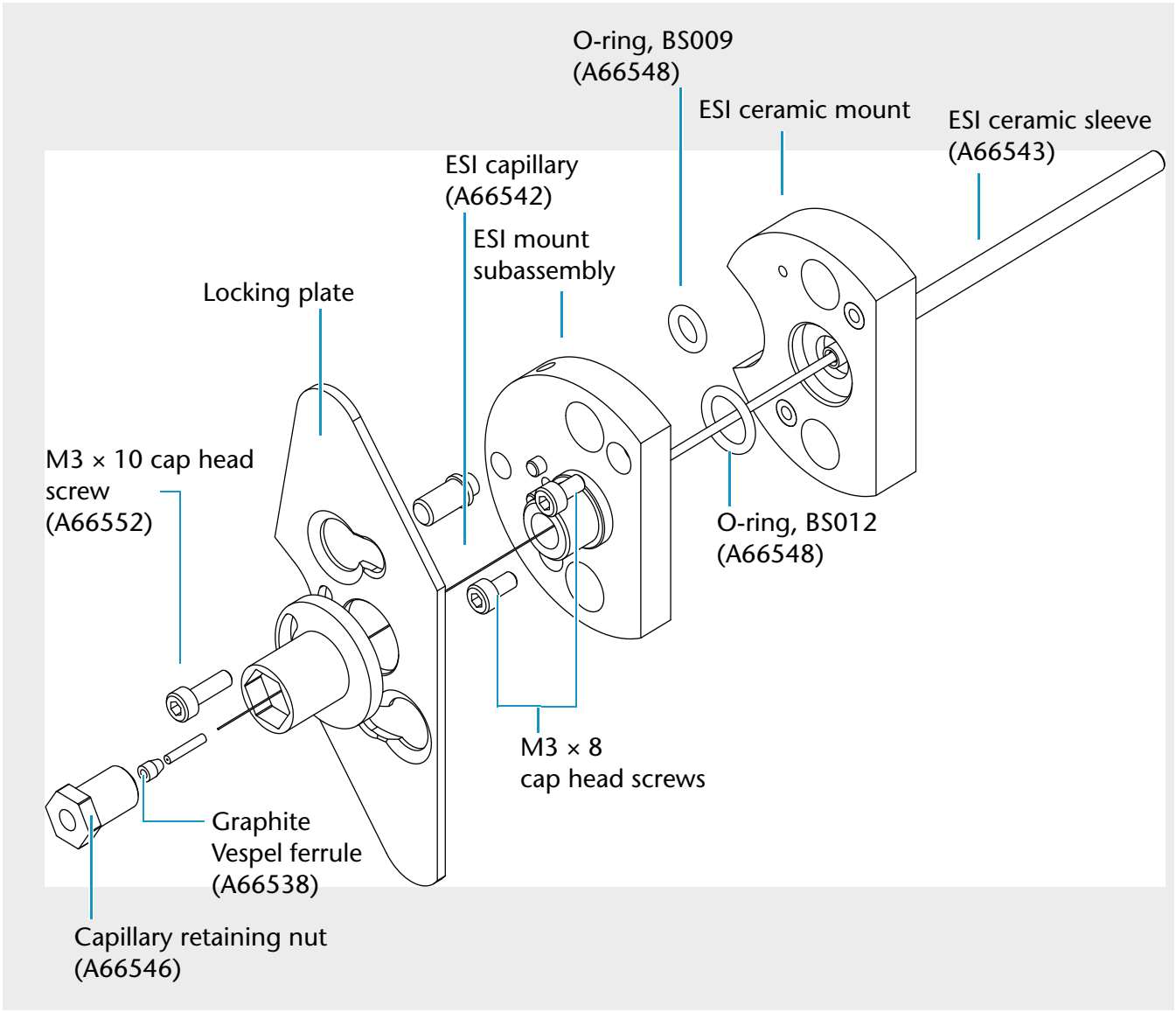


Fig. 84 Exploded view of ESI probe assembly

APCI Probe Assembly

Table 11 lists the parts in the APCI probe assembly.

Item	Part number
Assembly, APCI probe	**A66513**
APCI capillary tube (6 each)	A66541
Ferrule, GVF/16, graphite vespel	A66538
O-ring, 9.25 mm ID × 1.78 mm THK, BS012, BLK VITON	part of MSQ Plus Hardware and O-ring kit (A66548)
O-ring, 5.28 mm ID × 1.78 mm THK, BS009, BLK VITON	part of MSQ Plus Hardware and O-ring kit (A66548)
PEEK tube insert (12 each)	A66540
Screw, M3 × 8, cap head, stainless steel	N/A
Screw, M3 × 10, cap head, stainless steel	A66552
Capillary retaining nut	A66546

Table 11.APCI probe assembly parts

Figure 85 shows an exploded view of the APCI probe assembly.

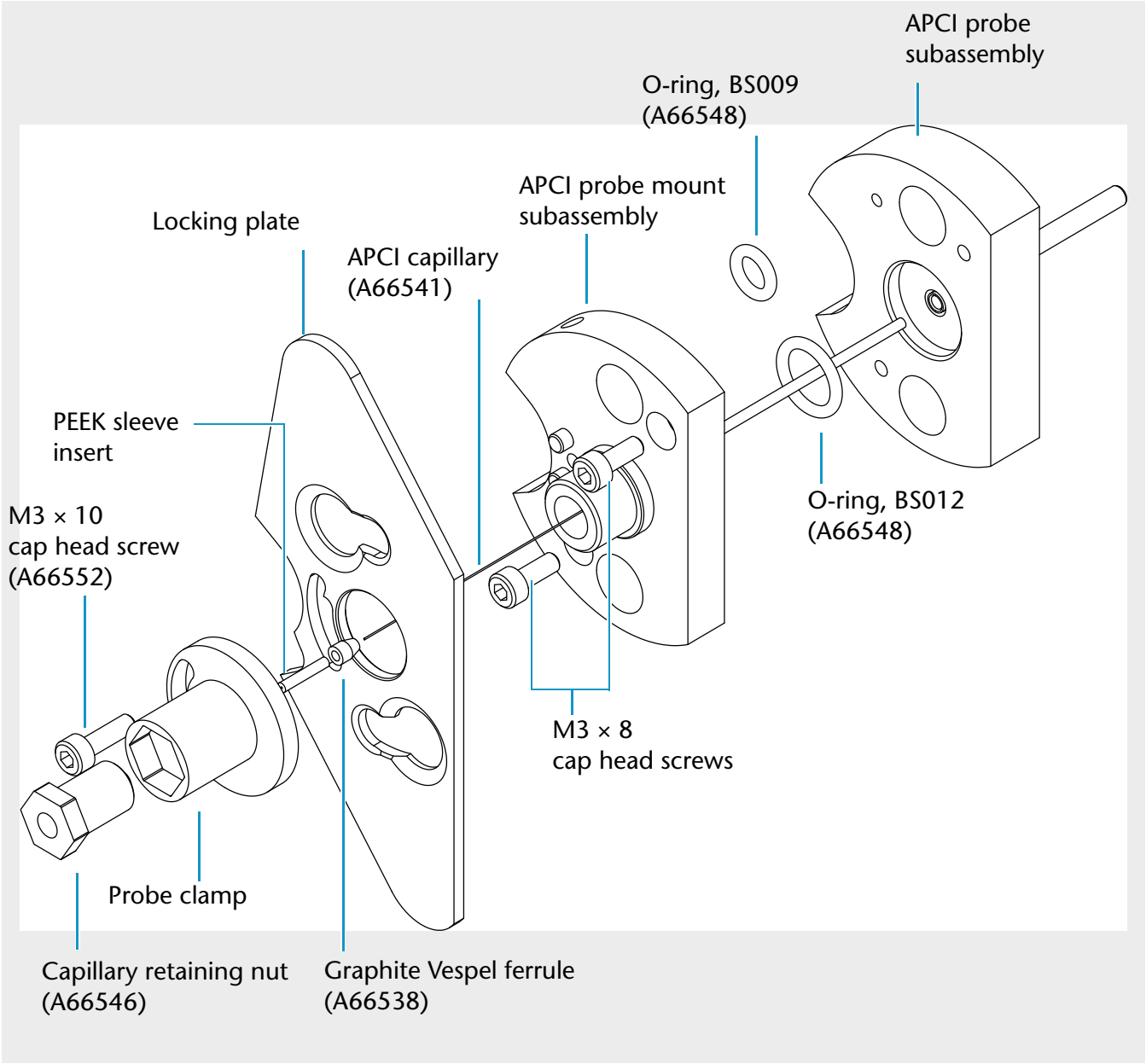


Fig. 85 Exploded view of APCI probe assembly

Probe Heater Assembly

Table 12 lists the parts in the probe heater assembly.

Item	Part number
Probe heater assembly	**A66517**
Detent screw insulator	part of Probe Heater Repair Kit (A66545)
Spring screw	part of Probe Heater Repair Kit (A66545)
Spring cup	part of Probe Heater Repair Kit (A66545)
Probe screw sleeve	part of Probe Heater Repair Kit (A66545)
O-ring, 18.77 mm ID × 1.78 mm THK, BS018, BLK VITON	part of MSQ Plus Hardware and O-ring kit (A66548)

Table 12.Probe heater assembly parts

Figure 86 shows the probe heater assembly. Figure 87 shows the components of the Probe Heater Repair Kit (part number A66545).

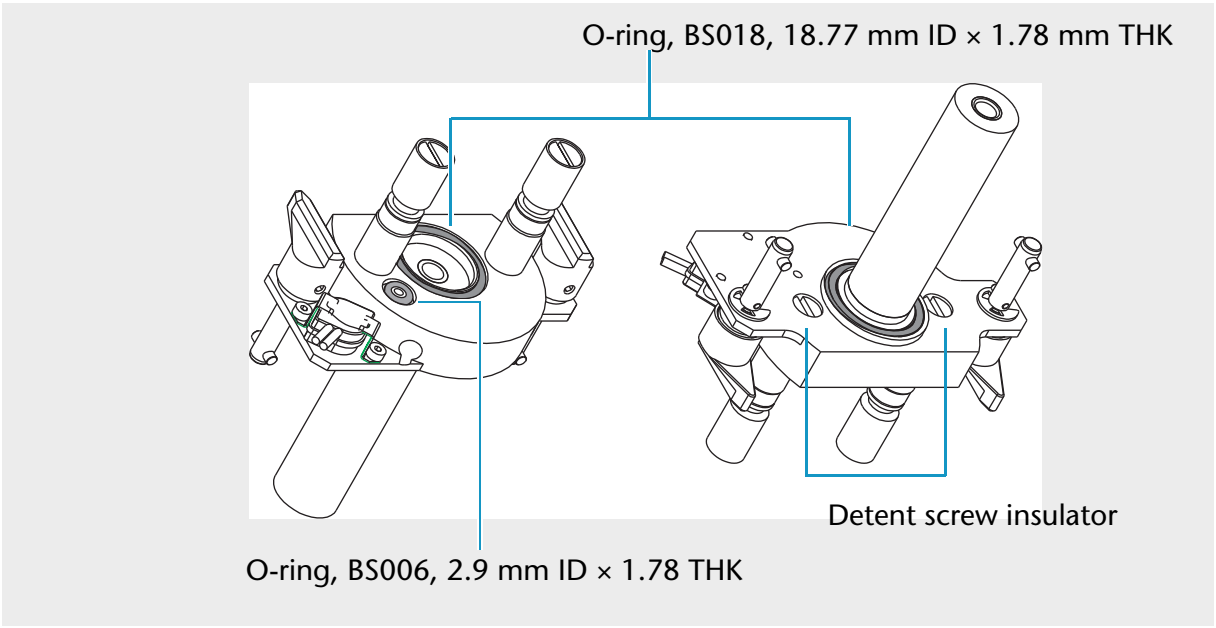


Fig. 86 Probe heater assembly with a view of the O-rings

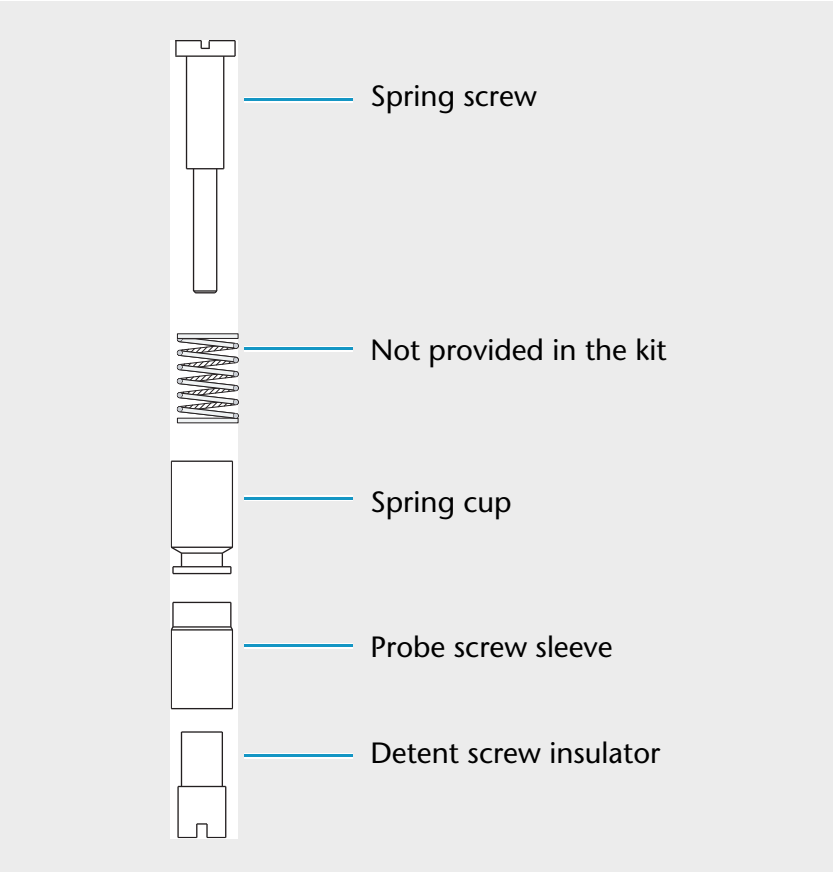


Fig. 87 Components of the Probe Heater Repair Kit (part number A66545)

Vacuum Spares

Table 13 lists the vacuum spare parts.

Item	Part number
Tee, KF25	A66534
Hose, blue exhaust, 1 in. ID	A66529

Table 13.Vacuum spare parts

Gas Flow Spares and Nitrogen Generator

Table 14 lists the gas flow spare parts and the nitrogen generator parts.

Item	Part number
Tubing, 6 mm OD, PTFE, for nitrogen line (order by the foot length)	A66521
PEAK nitrogen generator	A66501

Table 14.Gas flow spare parts and nitrogen generator parts

Solvent Path and Calibrant Spares

Table 15 lists the spare parts for the solvent path and the calibrant.

Item	Part number
MSQ calibration solution kit (for MSQ software version 1.4 and higher)	A66516
MSQ Plus Mass Detector sensitivity test kit. This kit must be shipped as hazardous material.	A66523

Table 15.Solvent path and calibrant spare parts

Electronic Spares

Table 16 lists the electronic spare parts.

Item	Part number
Assembly, power supply, low-voltage, MSQ	A66507
PCB, 2000 RF/Digital control	A66503
PCB, electrometer	A66508
PCB, source	A66504
PCB, RF generator	A66506
PCB, RF/dc prefilter. Replacement requires “redipping” the transfer lens coil. Call field service.	A66505

Table 16.Electronic spare parts

Communication Kit

For information on the communication kit available, refer to the manuals stored on the LC devices CD that is part of the media kit.

LC communication kit	A66610

Optimizing the LC Conditions

This appendix includes information that you might find helpful for optimizing the LC system and the cone wash system.

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 17 apply.

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 μL/min
Capillary	<10 μL/min

Table 17.LC columns and flow rates

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

- Electrospray can operate at all the flow rates described in Table 17.
- 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.

LC Solvents

Water, acetonitrile, and methanol are the solvents that are the most compatible with the MSQ Plus Mass Detector. These common reverse-phase LC solvents are ideal for LC/MS. When you use high percentages of water, you usually need to raise the probe temperature to aid desolvation.

Less commonly used solvents include normal-phase solvents; alcohols such as isopropanol, 2-methoxyethanol, and ethanol; and 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

Ion Polarity Mode	Additive
Positive ion	Acetic acid Formic acid Ammonium acetate (<0.1M)
Negative ion	Triethylamine (TEA) Ammonium hydroxide (ammonia solution) Ammonium acetate (<0.1M)

Table 18.Summary of suitable additives

Ion Polarity Mode	Additive
Positive ion	Surfactants Trifluoroacetic acid (TFA) (>0.1% v/v)
Negative ion	Surfactants Organic acids such as acetic acid, formic acid, trifluoroacetic acid (TFA).

Table 19.Summary of additives to avoid

Commonly Used Compatible Additives

The following additives are the most compatible with the MSQ Plus Mass Detector:

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

You can enhance LC separations 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 you analyze 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, because 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

The following additives are less commonly used:

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

Trifluoroacetic acid (TFA) is frequently used for peptide and protein analysis. High levels greater than 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 (because it is also 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. It is a particularly useful additive for the analysis of nucleic acids.

In ESI, using THF can reduce sensitivity. You can counteract this effect by the 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 and 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. They can cause problems with electrospray because 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 because of 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 MSQ Plus Mass 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. 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. The cone wash can be used for a short duration at the beginning of the LC analysis and turned off after the void volume of the LC column is cleared.

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, because this can lead to cone wash solvents condensing on the RF/dc prefilter.

Flow Splitting

Because the MSQ Plus Mass 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 a mass detector is required, you can split the flow by using a zero dead volume tee fitting. Eliminating the flowcell of the UV detector from the solvent path to the mass detector minimizes the peak broadening for the chromatograms produced by the mass detector.

The split ratio between the flow going to the UV detector and the flow going to the mass 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 is manufactured by SGE International Pty, Ltd.

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. Table 20 lists the inner diameters and internal volume of five of the most commonly used colors.

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

Table 20.1/16 in. OD PEEK tubing color coding

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. KNAUER 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:

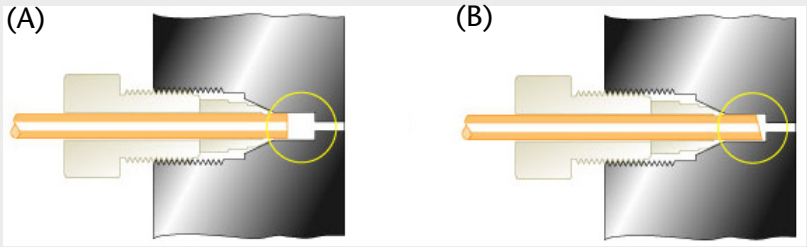
- ▶ Squeeze the tabs at the back of the cutter together to raise the blade.
- ▶ Insert your tubing through the appropriate guide hole.
- ▶ Release the tabs, allowing the blade of the cutter to rest on the tubing.
- ▶ Holding the tubing, spin the cutter around the tubing to begin the cut. For PEEK tubing, spin the cutter two to three times, 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 88.

Note:

1. Tubing that is not properly seated can add dead volume to a chromatographic system.

Never over-tighten PEEK fittings, because this can cause leaks.



Courtesy of Rheodyne website

Fig. 88 Poor connections result if tubing is not bottomed in the port (A) or is not cut square (B)

Chemical Kit

To prepare the MSQ Plus Mass Detector calibration solution for autotune and mass calibration, you can either prepare it yourself using the instructions in the "Calibrant Solution" section of the *MSQ Plus Mass Detector Startup Guide*, or you can use the MSQ Plus Mass Detector Chemical Kit, shown in Figure 89. This kit contains calibration, qualification, and sensitivity chemicals for the MSQ Plus Mass Detector. Specifically, it contains the following:

- MSQ Plus Mass Detector Calmix Kit (part number A66516), shown in Figure 90, which includes the following items:
  - *MSQ Plus Mass Detector Calmix Kit Preparation Guide*. This document contains the same information as the "Preparing the MSQ Plus Mass Detector Calibration Solution" section in the *MSQ Plus Mass Detector Startup Guide*.
  - Certificate of Analysis, which verifies that the Calmix solution actually contains the specified ingredients.
  - Material Safety Data Sheet, which provides health, safety and handling information in compliance with ISO requirements.
- Sensitivity Kit (part number A66523), which contains the two test chemicals used to perform the sensitivity specification tests during MSQ performance qualification.
- Functionality Test Standard (part number A66554), which is a 20-ppm caffeine standard that is used to check performance of the LC-MS system as a complete system. The Sensitivity Kit (part number A66523) only tests the MS portion. The LC system includes its own chemical test kit.

The chemical kit is shipped separately from the MSQ Plus Mass Detector.

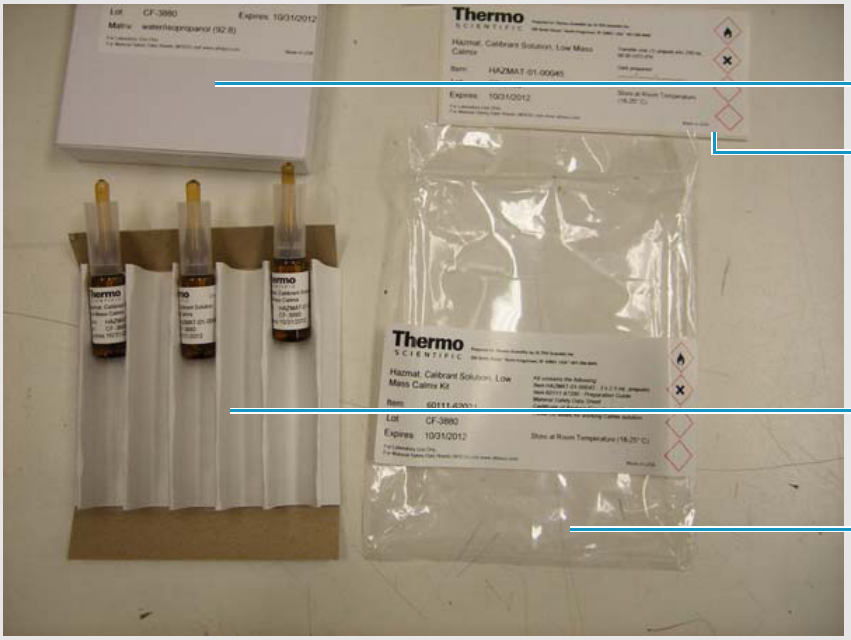
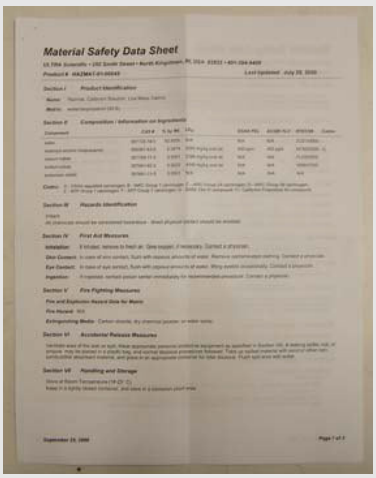


Fig. 89 Contents of Chemical Kit


Material Safety Data Sheet


Certificate of Analysis

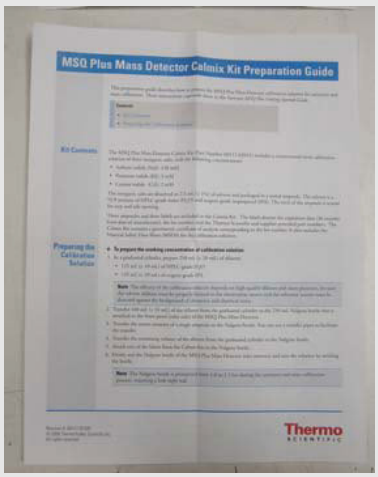

MSQ Plus Mass Detector Calmix Kit Preparation Guide

Fig. 90 Contents of MSQ Plus Mass Detector CALMIX Kit (part number A66516)

Decontamination

Contamination of devices with toxic, infectious or radio-active substances poses a hazard for all persons during operation, repair, sale and disposal of a device.

CAUTION! Danger caused by toxic, infectious or radio-active substances! A contaminated device must never be submitted for repairs, sold or disposed of! Contract a specialist company to decontaminate the device or perform the decontamination yourself if you have the required expertise!

All contaminated devices must be properly decontaminated by a specialist company or the operating company before they can be recommissioned, repaired, sold or disposed of.
All materials or fluids used for decontamination must be collected separately and disposed of properly.

Environmental protection

Disposal

Old devices of the manufacturer can be dropped off at the local municipal waste facilities, where it will be disposed of properly, or can be returned if desired.

KNAUER Eluent Savings Handbook

Reducing the consumption of solvents

If UHPLC can be used in place of an HPLC method in the laboratory, one of the main benefits is lower solvent costs.

Example: Compared to the optimized HPLC method, an 80% reduction in acetonitrile consumption can be achieved when separating paracetamol with the UHPLC method.

More information can be found in KNAUER's Solvent Savings Handbook.

Legal information

Trademarks

This documentation contains references to the following products from other manufacturers:

- PEEK is a trademark of Victrex plc.
- Windows is a trademark of Microsoft Cooperation
- Xcalibur is a trademark of Thermo Fisher Scientific Inc.

Warranty conditions

The factory warranty for the device is valid for 12 months after the date of installation. All warranty claims shall expire in the event that any unauthorized changes are made to the device.

During the warranty period, any components with material or design-related defects will be replaced or repaired by the manufacturer free of charge.

This warranty excludes the following

1. Accidental or willful damage
2. Consumables, parts and accessories expendable in the normal operation of the instrument
3. Improper or inadequate operation, car, maintenance, adjustment or calibration by the user
4. User-induced chemical action (such as precipitation of samples, sample matrix, buffers, ion-pairing reagents, etc.), contamination or leaks and other customer competence issues
5. Any loss, damage and/or instrument malfunction resulting from use of user supplied software, hardware, interfaces or consumables other than those specified by the manufacturer
6. Packaging and transport damage

In the event of device malfunctions, contact:

Manufacturer

Wissenschaftliche Gerätebau
Dr. Ing. Herbert KNAUER GmbH
Hegauer Weg 38
14163 Berlin, Germany
Phone: +49–(0)30–809727–0
Fax: +49–(0)30–8015010
E-Mail: info@knauer.net
Internet: www.knauer.net

The packaging of our devices provides the best possible protection against transport damage. However, immediately inspect each delivery for signs of transport damage. If the shipment is incomplete or damaged, inform the manufacturer within three workdays. Also inform the freight carrier about transport damage.

Declaration of conformity

Manufacturer name and address

Wissenschaftliche Gerätebau
Dr. Ing. Herbert KNAUER GmbH
Hegauer Weg 38
14163 Berlin, Germany

MSQ Plus Mass Detector

Order numbers A66500

comply with the following requirements and product specifications:

- DIN EN 55011 (May 2010) Industrial, scientific and medical equipment – Radio frequency disturbance characteristics
- DIN EN 60799 (June 1999) Electrical accessories – Cord sets and interconnection cord sets
- DIN EN 61000-3-2 (October 2006) Electromagnetic compatibility (EMC) Part 3-2
  - EMC directive (2004/108/EC)
- DIN EN 61000-3-3 (June 2009) Electromagnetic compatibility (EMC) Part 3-3 – Limitation of voltage changes
- DIN EN 61000-4-2 (December 2009) Electromagnetic compatibility (EMC) Part 4-2 – Testing and measurement techniques
- DIN EN 61010-1 (August 2002) Safety requirements for electrical equipment for measurement, control and laboratory use
  - Low voltage directive (2006/95/EC)
- DIN EN 61326-1 (October 2006) Electrical equipment for measurement, control and laboratory use - EMC requirements
- Directives for an environmentally sound use of electrical and electronic equipment
  - RoHS directive 2002/95/EC (February 2003) on the restriction of the use of certain hazardous substances in electrical and electronic equipment
  - WEEE directive 2002/96/EC (February 2003) on waste electrical and electronic equipment

Date

The product was tested with a typical configuration.

Berlin, 2011-01-31

Signature

Dr. Alexander Bünz (Managing Director)

The mark of conformity has been applied to the rear panel of the module.

CE

Index

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