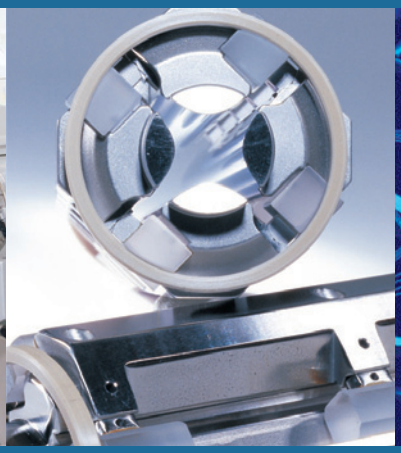
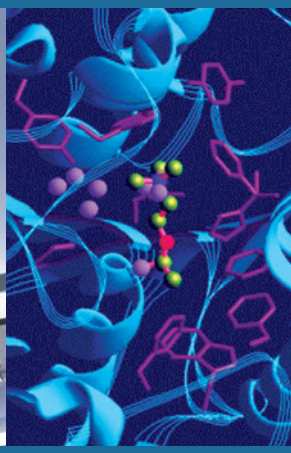
Thermo Fisher Scientific

# Exactive™
## Operating Manual

Revision C - 1249360

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Release History: Revision A released in January 2009.
Revision B released in July 2009.
Revision C released in April 2010.

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**EC Declaration of conformity** according to ISO/IEC 17050-1:2004

| | |
|---|---|
| **Name des Herstellers:**<br>manufacturers name | Thermo Fisher Scientific |
| **Adresse des Herstellers:**<br>manufacturers address | Hanna-Kunath-Strasse 11<br>28199 Bremen<br>Germany |

**Der Hersteller erklärt, dass das Produkt**
The manufacturer declares that the following product

| | |
|---|---|
| **Name des Produkts:**<br>product name | Mass Spectrometer |
| **Modell:**<br>model number | EXACTIVE |
| **Produktoptionen:**<br>product options | inkl. / incl. HCD |

**mit den folgenden EG Richtlinien und harmonisierten Standards übereinstimmt:**
is in conformity with the following EC Directives and harmonized standards

| | | |
|---|---|---|
| **EMV-Richtlinie**<br>EMC Directive<br>**2004/108/EG** | EN 55011 (11.2007)<br>EN 61000-3-2 (10.2006)<br>EN 61000-3-3 (06.2006)<br>EN 61000-4-2 (12.2001)<br>EN 61000-4-3 (12.2006)<br>EN 61000-4-4 (05.2005) | EN 61000-4-5 (06.2007)<br>EN 61000-4-6 (04.2008)<br>EN 61000-4-11 (02.2005)<br>EN 61326-1 (10.2006)<br>+ Corr. (06.2008) |
| **Niederspannungsrichtlinie**<br>Low Voltage Directive<br>**2006/95/EG** | EN 61010-1 (08.2002) | |
| **Ergänzende Informationen:**<br>Complementary information | - | |

Bremen, Germany, 03. November 2008

ThermoFisher
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Jörg Behrens
**Technischer Leiter**
Director of operations

ThermoFisher SCIENTIFIC

# FCC Compliance Statement

THIS DEVICE COMPLIES WITH PART 18 OF THE FCC RULES.

# Read This First

Welcome to the Thermo Scientific, Exactive™ system! Exactive is a member of the Thermo Scientific family of advanced mass spectrometer (MS) detectors.

## About This Guide

This *Exactive Operating Manual* describes the modes of operation and principle hardware components of your Exactive instrument. In addition, this manual provides step-by-step instructions for cleaning and maintaining your instrument.

### Who Uses This Guide

This *Exactive Operating Manual* is intended for all personnel that need a thorough understanding of the instrument (to perform maintenance or troubleshooting, for example). This manual should be kept near the instrument to be available for quick reference.

### Scope of This Guide

This manual includes the following chapters:

- Chapter 1: "Functional Description" describes the principal components of the Exactive.
- Chapter 2: "Daily Operation" outlines the checks and cleaning procedures of the Exactive system that should be performed every day to ensure the proper operation of your system.
- Chapter 3: "User Maintenance" outlines the maintenance procedures that you should perform on a regular basis to maintain optimum MS detector performance.
- Chapter 4: "System Shutdown, Startup, and Reset" provides procedures for shutting down and starting up the Exactive.
- Chapter 5: "Replaceable Parts" lists the replaceable parts for the MS detector and data system.
- Appendix A: "Calibration Solutions" provides information about preparing the calibration solutions for the Exactive.

- Appendix B: "Getting Connected" provides information on how to connect your Exactive mass spectrometer to external devices and describes the specifications for the peripheral control connections.

# Related Documentation

In addition to this guide, Thermo Fisher Scientific provides the following documents for Exactive:

- *Exactive Preinstallation Requirements Guide*
- *Exactive Software Manual*
- *Exactive QuickStart Guide*
- *Ion Max and Ion Max-S API Source Hardware Manual*

You can access PDF files of the documents listed above from the data system computer. The software also provides Help.

# Contacting Us

There are several ways to contact Thermo Fisher Scientific.

## Assistance

For technical support and ordering information, **visit us on the Web**:

www.thermo.com/advancedms

## Customer Information Service

cis.thermo-bremen.com is the Customer Information Service site aimed at providing instant access to

- latest software updates
- manuals, application reports, and brochures.

**Note** Thermo Fisher Scientific recommends that you register with the site as early as possible. ▲

To register, visit register.thermo-bremen.com/form/cis and fill in the registration form. Once your registration has been finalized, you will receive confirmation by e-mail.

## Changes to the Manual

❖ **To suggest changes to this manual**

- Please send your comments (in German or English) to:

  Editors, Technical Documentation
  Thermo Fisher Scientific (Bremen) GmbH
  Hanna-Kunath-Str. 11

  28199 Bremen

  Germany

- Send an e-mail message to the Technical Editor at

  documentation.bremen@thermofisher.com

You are encouraged to report errors or omissions in the text or index. Thank you.

# Typographical Conventions

This section describes typographical conventions that have been established for Thermo Fisher Scientific manuals.

## Data Input

Throughout this manual, the following conventions indicate data input and output via the computer:

- Messages displayed on the screen are represented by capitalizing the initial letter of each word and by italicizing each word.
- Input that you enter by keyboard is identified by quotation marks: single quotes for single characters, double quotes for strings.
- For brevity, expressions such as "choose **File > Directories**" are used rather than "pull down the File menu and choose Directories."
- Any command enclosed in angle brackets < > represents a single keystroke. For example, "press **<F1>**" means press the key labeled *F1*.
- Any command that requires pressing two or more keys simultaneously is shown with a plus sign connecting the keys. For example, "press **<Shift>** + **<F1>**" means press and hold the <Shift> key and then press the <F1> key.
- Any button that you click on the screen is represented in bold face letters. For example, "click **Close**".

## Topic Headings

The following headings are used to show the organization of topics within a chapter:

# Chapter 1 Chapter Name

## Second Level Topics

### Third Level Topics

#### Fourth Level Topics

# Safety and EMC Information

In accordance with our commitment to customer service and safety, this instrument has satisfied the requirements for the European CE Mark including the Low Voltage Directive.

Designed, manufactured and tested in an ISO9001 registered facility, this instrument has been shipped to you from our manufacturing facility in a safe condition.

This instrument must be used as described in this manual. Any use of this instrument in a manner other than described here may result in instrument damage and/or operator injury.

**Notice on Lifting and Handling of Thermo Scientific Instruments**

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

**Notice on the Proper Use of Thermo Scientific Instruments**

In compliance with international regulations: If this instrument is used in a manner not specified by Thermo Fisher Scientific, the protection provided by the instrument could be impaired.

**Notice on the Susceptibility to Electromagnetic Transmissions**

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

## Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear different from the main flow of text. Safety and special notices include the following:

**Warning** Warnings highlight hazards to human beings. Each Warning is accompanied by a Warning symbol. ▲

**Caution** Cautions highlight information necessary to protect your instrument from damage. ▲

**Note** Notes highlight information that can affect the quality of your data. In addition, notes often contain information that you might need if you are having trouble. ▲

## Identifying Safety Information

The *Exactive Operating Manual* contains precautionary statements that can prevent personal injury, instrument damage, and loss of data if properly followed. Warning symbols alert the user to check for hazardous conditions. These appear throughout the manual, where applicable. The most common warning symbols are:

**Warning General Hazard.** This general symbol indicates that a hazard is present that could result in injuries if it is not avoided. The source of danger is described in the accompanying text. ▲

**Warning Electric Shock Hazard.** High Voltages capable of causing personal injury are used in the instrument. The instrument must be shut down and disconnected from line power before service is performed. Do not operate the instrument with the top cover off. Do not remove protective covers from PCBs. ▲

**Warning Burn Hazard.** Treat heated zones with respect. Parts of the instrument might be very hot and might cause severe burns if touched. Allow hot components to cool before servicing them. ▲

**Warning Corrosive Material.** Wear gloves when handling toxic, carcinogenic, mutagenic, or corrosive/irritant chemicals. Use approved containers and procedures for disposal of waste solution. ▲

In addition to the above described, every instrument has specific hazards. So, be sure to read and comply with the precautions described in the subsequent chapters of this guide. They will help ensure the safe, long-term use of your system.

## General Safety Precautions

Observe the following safety precautions when you operate or perform service on your instrument:

- The system should be operated by trained personnel only. Read the manuals before starting the system and make sure that you are familiar to the warnings and safety precautions!
- Accurate results can be obtained only, if the system is in good condition and properly calibrated.

- Service by the customer should be performed by trained qualified personnel only and should be restricted to servicing mechanical parts! Service on electronical parts should be performed by Thermo Fisher Scientific Service Engineers only!
- Before plugging in any of the instrument modules or turning on the power, always make sure that the voltage and fuses are set appropriately for your local line voltage.
- Only use fuses of the type and current rating specified. Do not use repaired fuses and do not short-circuit the fuse holder.
- The supplied power cord must be inserted into a power outlet with a protective earth contact (ground). When using an extension cord, make sure that the cord also has an earth contact.
- Do not change the external or internal grounding connections. Tampering with or disconnecting these connections could endanger you and/or damage the system.
- The instrument is properly grounded in accordance with regulations when shipped. You do not need to make any changes to the electrical connections or to the instrument's chassis to ensure safe operation.
- Never run the system without the housing on. Permanent damage can occur. When leaving the system, make sure that all protective covers and doors are properly connected and closed, and that heated areas are separated and marked to protect for unqualified personnel!
- Do not turn the instrument on if you suspect that it has incurred any kind of electrical damage. Instead, disconnect the power cord and contact a Thermo Fisher Scientific Service Engineer for a product evaluation. Do not attempt to use the instrument until it has been evaluated. (Electrical damage may have occurred if the system shows visible signs of damage, or has been transported under severe stress.)
- Damage can also result if the instrument is stored for prolonged periods under unfavorable conditions (e.g., subjected to heat, water, etc.).
- Always disconnect the power cord before attempting any type of maintenance.
- Capacitors inside the instrument may still be charged even if the instrument is turned off.
- Never try to repair or replace any component of the system that is not described in this manual without the assistance of your Thermo Fisher Scientific Service Engineer.

- Do not place any objects – especially not containers with liquids – upon the instrument. Leaking liquids might get into contact with electronic components and cause a short circuit.

# Safety Advice for Possible Contamination

**Hazardous Material Might Contaminate Certain Parts of Your System During Analysis.**

In order to protect our employees, we ask you to adhere to special precautions when returning parts for exchange or repair.

If hazardous materials have contaminated mass spectrometer parts, Thermo Fisher Scientific can only accept these parts for repair if they have been properly decontaminated. Materials, which due to their structure and the applied concentration might be toxic or which in publications are reported to be toxic, are regarded as hazardous. Materials that will generate synergetic hazardous effects in combination with other present materials are also considered hazardous.

Your signature on the **Repair-Covering letter** confirms that the returned parts have been decontaminated and are free of hazardous materials.

The Repair-Covering letter can be ordered from your service engineer or downloaded from the **Customer Information Service** (**CIS**) site. Please register under http://register.thermo-bremen.com/form/cis.

Parts contaminated by radioisotopes are not subject to return to Thermo Fisher Scientific – either under warranty or the exchange part program. If parts of the system may be possibly contaminated by hazardous material, please make sure the Field engineer is informed before the engineer starts working on the system.

# Contents

# Figures

# Tables

# Procedures

# Chapter 1 Functional Description

This chapter provides an overview of the functional elements of the Exactive. It contains the following topics:

# General Description

Exactive is a stand-alone Orbitrap™ instrument with an API source for LCMS high throughput applications. The instrument is designed to be placed on a bench in the laboratory. Figure 1-1 shows a front view of the instrument. This chapter describes the principal components of the Exactive system and their respective functions.

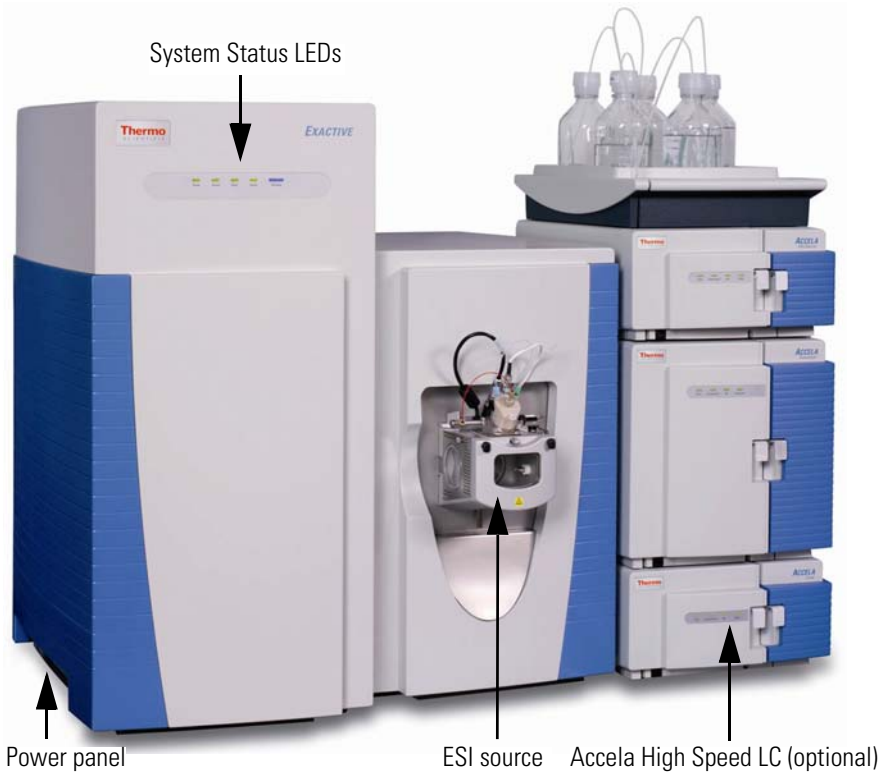



**Figure 1-1.** Exactive front view

## Instrument Layout

The Exactive consists of three main components, which are described in the following topics:

- An ion source.
- An intermediate storage device (C-Trap) that is required for short pulse injection.
- An Orbitrap analyzer for Fourier transform mass analysis.

A collision cell for performing HCD (Higher Energy Collisional Dissociation) experiments is available as option.

For a schematic view of the instrument layout, see Figure 1-10 on page 1-18.

## Operating Mode of the Instrument

Samples can be introduced into the API source at the front side by a variety of methods including direct infusion (via a syringe pump) or an U-HPLC system (Thermo Scientific Accela™). The source is similar to the commercial source of the Thermo Scientific TSQ Quantum Ultra™. As default, the instrument is delivered with the ESI probe. Probes for APCI, APPI, HESI, and NSI technique are available as options. See "API Source" on page 1-13 for details.

The ions are transferred into the C-Trap through four stages of differential pumping. See "Ion Optics" on page 1-18 for details. In the C-Trap, the ions are accumulated and their energy dampened using a bath gas (nitrogen). The ions are then injected through three further stages of differential pumping using a lens system (Z-lens) into the Orbitrap analyzer where mass spectra are acquired via image current detection. The vacuum inside the Orbitrap mass analyzer is maintained below $1 \times 10^{-9}$ mbar. See "Orbitrap Analyzer" on page 1-21 for details.

If the instrument is equipped with the optional HCD collision cell, it allows performing *All Ion Fragmentation* experiments by means of Higher Energy Collision Induced Dissociation (HCD). Ions are passed through the C-Trap into a multipole collision cell where they are fragmented. After that, the HCD cell voltages are ramped up and the ions are transferred back into the C-Trap from where they are injected into the Orbitrap for detection. See page 1-25 for a description of the HCD collision cell.

## Measurement Specifications

Table 1-1 lists the measuring properties of the Exactive.

**Table 1-1.** Measuring properties of the instrument

| | |
|---|---|
| **Mass Range** | *m/z* 50–4000 (Scan range: last mass <= 20 × first mass) |
| **Scan speed** | 1 to 10 scans/s (depends on resolution setting) |
| **Resolution** | 100000 at *m/z* 200 at a scan rate of 1 Hz, minimum resolution 10000 at *m/z* 200 at a scan rate of 10 Hz |
| **Mass Accuracy** | <5 ppm with external calibration<br><2 ppm using internal standard, lock masses |
| **Polarity Switching** | One full cycle in < 1 sec (one full scan positive mode and one full scan negative mode at resolution setting of 10000) |
| **Sensitivity** | A flow injection of 500 fg of Buspirone giving a S/N of better than 10:1 for the selected mass chromatogram of the $[M+H]^+$ ion (*m/z* = 386.2551) |
| **Dynamic Range** | >4000 within a single scan |

# Placing the Instrument

This section describes the conditions for an operating environment that will ensure continued high performance of your Exactive system.

## Instrument Dimensions

The instrument has maximum dimensions of *h* 94 cm (37 in.), *w* 91 cm (36 in.), *l* 83 cm (33 in.)[1]. Figure 1-2 shows a schematic view of the Exactive with important instrument dimensions.

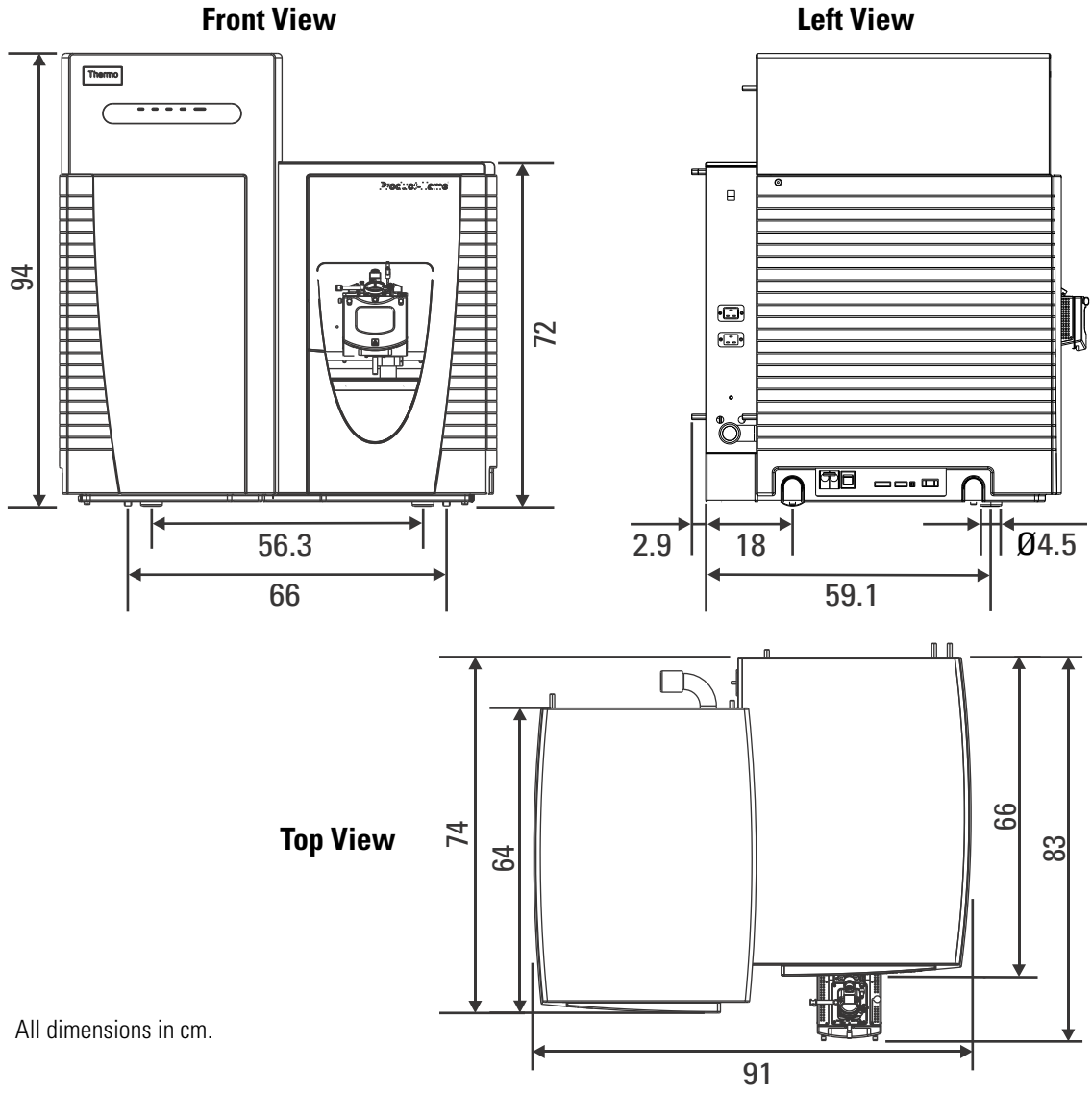



**Figure 1-2.** Instrument dimensions

[1]Length without source mounted.

## Moving the Instrument

The Exactive is provided with four retractable handles for carrying. Safety catches prevent the handles from inadvertently being retracted after they have been pulled out. Inspect the handles to verify that their safety catches are engaged before you start lifting the instrument. Push down the safety catch on a handle to slide it into the instrument.

The rear pair of the four support points for the instrument are wheels. Thus, only two persons are necessary for moving the instrument into its final position on a bench, while holding the two front handles. See Figure 1-2 on page 1-4 for information about the position of the support points.

**Warning Lifting Hazard.** This instrument is too heavy for one person alone to handle safely. Lifting and moving the instrument requires the effort of at least four persons.
When lifting the instrument, note that its center of gravity is the top of the front side: the instrument has a tendency to tilt forward. Support the top of the instrument and keep the tilt angle below 5°; never exceed 10°. ▲

## Workbench for Instrument

The workbench for the MS system must stand in a secure and level position. Note that only workbenches with four legs provide sufficient stability for the Exactive. The workbench top must be dry and clean (free of grease). Thermo Fisher Scientific recommends using a workbench with a skid proof top.

## Vibration

Floors must be free of vibration caused, for example, by equipment in adjoining locations.

**Caution** Because of the natural vibration of the forepump during operation, it must not have any mechanical contact to the Exactive with exception of the vacuum hose. Otherwise, the vibration might impede the performance of the instrument. Therefore, install the pump on the floor beneath the Exactive and not near the system on the workbench. ▲

## Minimum Clearance

Allow at least 15 cm (6 in.) of clear space behind the system for proper air circulation and for clearance of the gas lines and electrical connections. This also provides sufficient space for accessing the fan

filters on the rear side of the MS detector. In addition, allow at least 92 cm (36 in.) of vertical clearance between the top of the Exactive and any shelves above it.

To allow shutting off the MS detector in an emergency, free access to the power panel on the left side of the instrument must be possible at any time.

**Caution** Avoid blocking the ventilation slots at the rear of the instrument. Items may fall behind the instrument, inhibit airflow, and cause the system to overheat. ▲

## Laboratory Room Temperature

The Exactive is designed to operate at a laboratory room temperature between 15 and 26 °C (59 and 78 °F). The optimum temperature of operation is 18–21 °C (65–70 °F).

**Note** Do not put the Exactive instrument under an air duct, near windows, or near heating and cooling sources. Temperature fluctuations of 1°C or more over a 10 minutes period can affect performance. ▲

# Control Elements

The Exactive is mainly operated from the desktop computer (data system). LEDs at the front side of the instrument give general information about the system status. Switches and ports for peripheral devices are located at the power panel on the left side of the instrument. The mains inlet, a power outlet for the forepump, and the Ethernet port are located at the rear side of the instrument. This section describes the control elements for important system functions.

## System Status LEDs

Figure 1-3 shows the system status LEDs at the front side of the instrument. These five LEDs are controlled by the source board (See page 1-40.) and indicate main functions of the system. Table 1-2 explains the function of the LEDs.


**Figure 1-3.** System status LEDs

**Table 1-2.** System status LEDs of the Exactive

| LED | Status | Information |
|---|---|---|
| Power | Green | Main power circuit breaker switch on |
| | Off | Main power circuit breaker switch off |
| Vacuum | Green | Operating vacuum reached |
| | Yellow | Vacuum insufficient for measurements, RF can be switched on* |
| | Green/Yellow flashing | System bakeout in progress |
| | Off | Vacuum insufficient for measurements, RF cannot be switched on* |
| Status | Green | Instrument in operating mode |
| | Yellow | Minor error that does not prevent measurement* |
| | Off | Grave error that prevents measurement* |
| System | Green | System on |
| | Yellow | System in Standby mode |
| | Off | System off |
| Scanning | Blue flashing | Instrument scanning |
| | Off | Instrument not scanning |

* See the Exactive Tune software for detailed information about malfunctions of system components.

**Note** The system status LEDs give a quick overview of the general system status; they do not have any function for the safety status of the instrument. Before performing any maintenance on the instrument, make sure that the main power circuit breaker switch (labeled Main Power) is in the Off (O) position and that the power cord is disconnected. It is not sufficient that the Power LED is off because it might be defective. All system status LEDs are off, when the electronics service switch is in the Service Mode position. ▲

## Power Panel

Figure 1-4 shows the rear side of the instrument with the power panel. Also visible are the power column, the forevacuum port, and the ventilation slots.


**Figure 1-4.** Rear side view of the instrument

The *main power circuit breaker switch* (labeled Main Power) is located on the power panel at the lower right corner of the left side panel of the MS detector. See Figure 1-1 on page 1-2. In the Off (O) position, the circuit breaker removes all power to the MS detector, including the vacuum pumps. In the On (|) position, power is supplied to the MS detector. In the standard operational mode, the circuit breaker is kept in the On (|) position.

**Note** Power is to remain on. The Exactive system should remain on and pumping continuously for optimum performance. ▲

The *electronics service switch* is located on the power panel. See Figure 1-5. In the Service Mode position, the switch removes power to all components of the MS detector other than the vacuum system. In the Operating Mode position, power is supplied to all components of the MS detector.

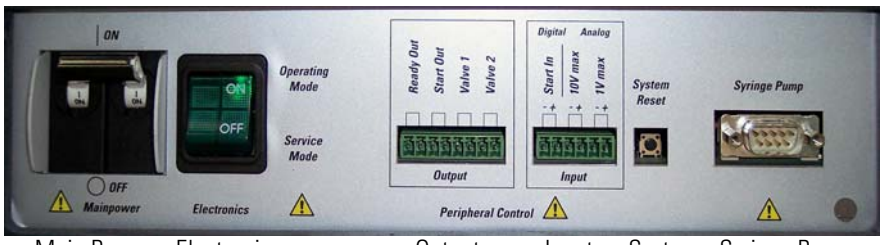



**Figure 1-5.** Power panel

**Caution** To shut Off all power to the MS detector in an emergency, place the main power circuit breaker switch (labeled Main Power) in the Off (O) position. Do not use the electronics service switch. ▲

**Note** To ensure that the instrument is free from all electric current, disconnect the power cord (before attempting any type of maintenance, for example). ▲

Both switches contain circuit breakers that open in case of a thermal overload to interrupt the power supply to the instrument. After cooling down (and removal of the overload), the circuit breakers close and both switches can be used again. If you cannot reset the instrument to the operating mode despite repeated attempts, the circuit breaker inside the switch is blown. In this case, call a Thermo Fisher Scientific Service Engineer to replace it.

**Note** To allow shutting off the MS detector in an emergency, free access to the power panel on the left side of the instrument must be possible at any time. ▲

## Peripheral Control

The *peripheral control output* connection allows transmitting *digital* contact closure signals from the Exactive to peripheral equipment:

- The *Ready Out* port allows Exactive to provide ready status through contact closure for an external receiving device.

- The *Start Out* port allows Exactive to provide a programmable contact closure output signal to the inputs of an external receiving device (a fraction collector, for example).
- The *Valve 1* port and the *Valve 2* port allow Exactive to provide contact closure signals for two switching valves. A suitable two-position switching valve is available from Thermo Fisher Scientific (Rheodyne MXT 715-000, P/N 1239650). See "Establishing Power Supply and Communication for the Switching Valve(s)" on page B-19 for instructions about connecting switching valves to the Exactive.

**Note** Any connected switching valve is controlled via the Exactive Tune software. See the *Exactive Software Manual* or the online Help for details. ▲

The contact closure signals are transmitted through a trigger cable that connects the respective port to the external device. A suitable plug connector (P/N 2087270) for the peripheral control output connection is provided with the Exactive Installation Kit. See "User I/O Connections" on page B-21 for specifications of the peripheral control output connection ports.

The *peripheral control input* connection allows transmitting signals from peripheral equipment to the Exactive:

- The *Start In* port allows Exactive to start data acquisition upon receiving a *digital* contact closure signal from an external device. (For example, see the *Accela LC System Getting Connected Guide* for instructions on installing a system interconnect cable between the autosampler of the Accela LC and the Exactive MS detector.)
- The *10 V max* port and the *1 V max* port allow Exactive to start data acquisition upon receiving an *analog* contact closure signal from an external device (for example, an autosampler). Use the *10 V max* port, if the output signal from the analog device is between 0 and +10 V; use the *1 V max* port, if the output signal from the analog device is between 0 and +1 V.

The contact closure signals are transmitted through a trigger cable that connects the respective port to the external device. A suitable plug connector (P/N 2098690) for the *peripheral control input* connection is provided with the Exactive Installation Kit. See Appendix B: "Getting Connected" for specifications of the peripheral control input connection ports.

The *reset button* is also located on the power panel. When you press the reset button, the Exactive software is reloaded from the data system. See "Resetting the System" on page 4-8 for information on resetting the MS detector.

The *syringe contact* allows controlling established syringe pumps by the instrument software by means of the RS232 serial interface. A suitable syringe pump is available from Thermo Fisher Scientific (Chemyx Fusion 100, P/N 1245740). Also supported is the Harvard Apparatus Model 11 Plus Advanced pump. See also "Syringe Pump" on page 1-26. See "Establishing Power Supply and Communication for the Syringe Pump" on page B-19 for instructions about connecting a syringe pump to the Exactive.

**Note** Any connected syringe pump is controlled via the Exactive Tune software. See the *Exactive Software Manual* or the online Help for details. ▲

## Power Column

Figure 1-6 shows the power column at the rear side of the instrument with the external connections for mains supply, gases, and Ethernet communication.


**Figure 1-6.** Power column

Located at the top is the Ethernet port for connecting the Exactive with the data system computer.

The power cord of the forepump is plugged into the outlet labeled Forepump on the power column. This outlet supplies power to the pump and is controlled by the main power circuit breaker switch and not by the electronics service switch.

**Note** Use wall outlets to provide electrical power for the data system or other peripherals. ▲

The power connector for the mains supply is located below the two power outlets. The Exactive instrument is designed to operate at a nominal voltage of 230 V ac, 50/60 Hz. Line voltages can vary between a minimum of 207 V ac and a maximum of 253 V ac. Connect the power cord of the instrument to a wall receptacle that is fused with 15 A or 16 A.

**Caution** Systems installed in areas with 208 V power experience voltage sags during high use periods that might place the line voltage below the operating parameters discussed in this section. In this case, you must protect your instrument by using a buck/boost transformer to ensure that power is within the specified parameters at all times. ▲

Above the gas ports, a ground bolt provides a protected earth connection for the ground wires of peripheral devices.

Two ports for nitrogen gas and HCD[1] collision gas allow connecting Teflon® hoses from the gas supplies of the laboratory to the instrument. (See also "Gas Supply" on page 1-37.) The required gas pressure for both ports is 690 ± 140 kPa (6.9 ± 1.4 bar, 100 ± 20 psi).
If you plan to use nitrogen as HCD collision gas, use the T-piece (P/N 1128140) that is included in the Exactive Installation Kit.

**Caution** Do not connect other gases than nitrogen or argon to the Exactive! The maximum pressure for both gas inlets is 830 kPa (8.3 bar, 120 psi). ▲

[1]HCD is an option for Exactive. The feature will be not available if the instrument is not equipped with this option.

# API Source

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 interface between the LC and the MS detector. As default, the API source of Exactive is delivered with a probe for electrospray ionization (ESI). See Figure 1-7.

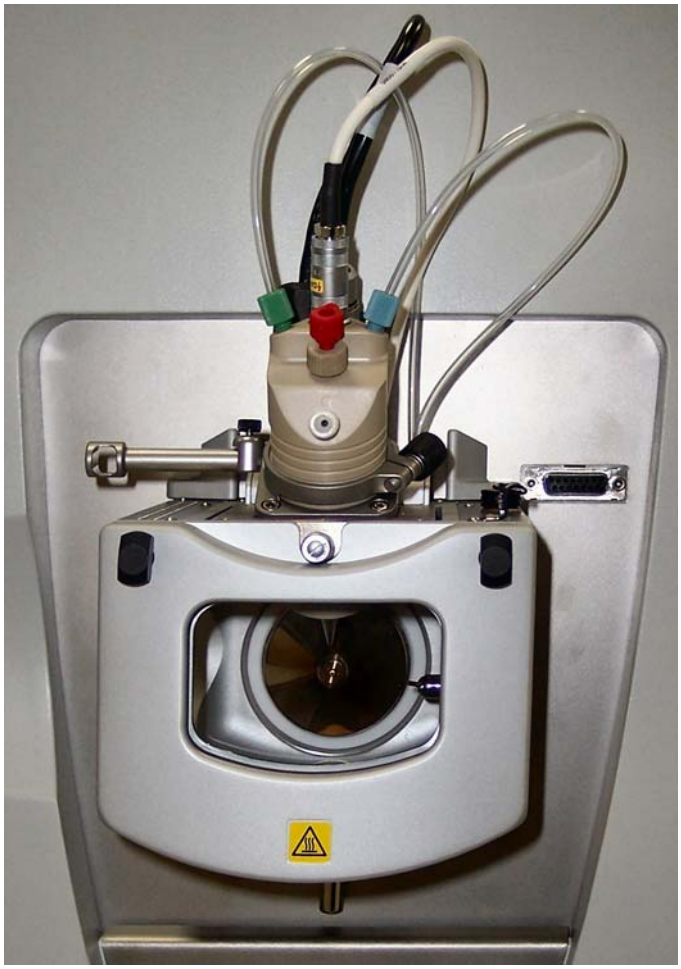


**Figure 1-7.** Ion Max ion source with ESI probe attached

Additionally, you can operate the API source with atmospheric pressure chemical ionization (APCI), atmospheric pressure photoionization (APPI), or nanospray ionization (NSI) technique. These probes are available as options.

The API source consists of the Ion Max™ ion source and the ion source interface, which are described in the following topics.

# Ion Max Ion Source

The Ion Max ion source is the part of the API source that is at atmospheric pressure. The Ion Max ion source can be configured to operate in any of several API modes, including electrospray ionization (ESI), atmospheric pressure chemical ionization (APCI), and atmospheric pressure photoionization (APPI). See Figure 1-7. A separate nanospray ionization (NSI) probe is necessary to operate in NSI mode. The ions produced in the API source are transmitted by the ion optics into the Orbitrap mass analyzer, where they are separated according to their mass-to-charge ratio.

## Ion Source Housing

The Ion Max ion source housing enables you to quickly switch between ionization modes without the need for specialized tools. A safety relay switches off the high voltage supply (8 kV) when the source is removed from the Exactive.

Ventilating the ion source housing keeps the housing cool and easy to handle. Pressure in the ion source housing is kept at atmospheric levels. This reduces the chemical noise that can be caused by nebulized gases when they are not properly evacuated from the ion source. The probe mounting angle is fixed at the optimum angle for signal intensity and ion source robustness. Minor adjustment of the probe position in the X, Y, and Z dimensions is allowed, with marked adjustments to allow for freedom in probe position during ionization optimization. View ports are placed at the front and the side of the ion source housing to permit viewing the probe position during ESI operation and the easy addition of accessories.

The Ion Max ion source is equipped with special features to ensure maximum lifetime. The drain size and angle prevents ion source corrosion by allowing eluants to flow directly from the probe into the drain when auxiliary gases are off. For liquids that do not enter the drain directly, the floor of the ion source interior is sloped to enable maximum drainage of collected eluants. Additionally, the zero dead volume LC grounding union that connects the LC flow to the ESI sample inlet is offset from the ion source. This is to prevent LC leaks from dripping directly on the ion source housing.

The Ion Max ion source has a universal mounting platform and interface for use with ESI, APCI, NSI, and APPI ionization sources. See below. For more information on the analysis of ions produced by the ion source, refer to the *Ion Max and Ion Max-S API Source Hardware Manual*.

### Solvent Drainage

Because the Ion Max API source can accommodate high flow rates, you must collect the waste solvent in a manner that avoids pressure buildup in the source. The Ion Max API source is fitted with a 25.4 mm (1.0 in.) OD outlet for solvent drainage. A 25.4 mm to 12.7 mm (1 in. to 0.5 in.) reducing fitting (P/N 00101-03-00001) connects to a waste container (P/N 00301-57020), both of which come with the system. To avoid pressure buildup in the source, make sure that the 1 inch diameter hose from the API source drain to the reducing fitting (P/N 00101-03-00001) is as long as possible. The 25.4 mm (1 in.) diameter Tygon™ tubing (P/N 00301-01-00020) that comes with the system is 1.52 m (5 ft) long. See "Connecting the Source Housing Drain to the Waste Container" on page 3-15 for additional information.

**Warning Injury Hazard.** The interior of the Ion Max API source housing contains parts that might be at high temperatures or high voltages. To prevent users from inadvertently touching such parts, always operate the Ion Max API source with the drain tubing assembly mounted to the source housing drain. ▲

**Caution** Do **not** vent the drain tubing (or any vent tubing connected to the waste container) to the same fume exhaust system to which you have connected the forepump. ▲

## Ion Source Mount

The ion source mount on the front side of the Exactive allows interchanging ESI, APCI, and APPI probes without using tools. The mount has high voltage electrical connections for the electrospray needle (ESI) and for the vaporizer and corona discharge needle (APCI). See Figure 1-8. A high voltage safety interlock switch turns off the following voltages when the ion source is removed:

- ESI spray voltage (or APCI corona discharge voltage)
- All API source and lens voltages, including the ion transfer capillary offset voltage
- The voltages on the ion guides

The above voltages are also turned off if the APCI vaporizer cable (APCI mode) is not plugged into the APCI vaporizer cable interlock connector on the source housing.

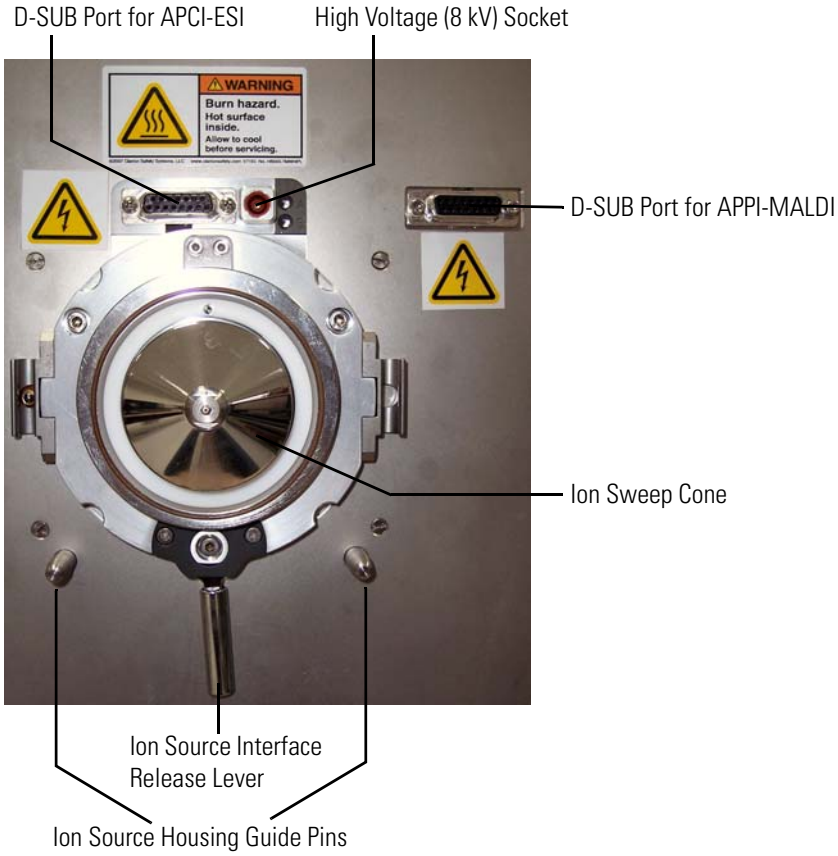



**Figure 1-8.** Ion source mount

## Ion Source Interface

The ion source interface consists of the components of the API source that are held under vacuum (except for the atmospheric pressure side of the ion sweep cone). The ion source interface includes an ion transfer capillary, two cartridge heaters, a heater block, a platinum probe sensor, a vent prevent ball, an ion sweep cone, a tube lens, and a skimmer. See Figure 1-9 on page 1-17.

The *ion transfer capillary* (heated capillary) assists in desolvating ions that are produced by ESI, APCI, NSI, or APPI. The capillary is an elongated, 4 inch (~10.2 cm) cylindrical tube made of metal that has a hole bored through the center of its long axis. Two heater cartridges are embedded in the heater block. The heater block surrounds the ion transfer capillary and heats it to temperatures up to 400 °C. A platinum probe sensor measures the temperature of the heater block. Typical temperatures of the ion transfer capillary are 270 °C for ESI and 250 °C for APCI, but they will vary with flow rate and mobile phase composition.

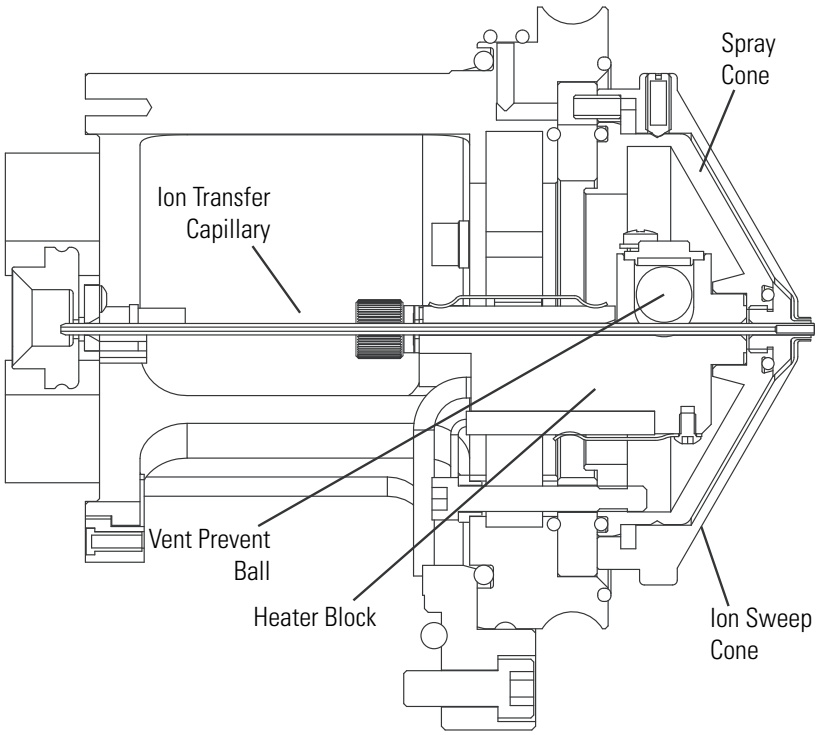



**Figure 1-9.** Cross sectional view of the ion source interface

Ions are drawn into the ion transfer capillary in the atmospheric pressure region and transported to the capillary-skimmer region of the vacuum manifold by a decreasing pressure gradient. A potential of typically ±35 V (positive for positive ions and negative for negative ions) assists in repelling ions from the ion transfer capillary to the skimmer. The *vent prevent ball* falls into the space occupied by the ion transfer capillary when the capillary is removed, thus preventing air from entering the vacuum manifold. The vent prevent ball allows removing the ion transfer capillary for cleaning without venting the system.

The *ion sweep cone* is a metallic cone over the capillary. The ion sweep cone channels the sweep gas towards the entrance of the tube.

The system electronics include a voltage monitor circuit and an overtemperature/undertemperature circuit to protect the heaters. The voltage monitoring circuit detects shorting failures. The overtemperature portion of the circuit works as a thermal limit switch to prevent the heater from turning on continuously above a preset temperature. The undertemperature feature identifies faults in the platinum probe sensor that would otherwise cause the heater to turn full on.

The ion source interface is enclosed in a vacuum chamber that is evacuated by the forepump to a pressure of approximately 2 mbar (1.5 Torr).

For instructions on the maintenance of the ion source interface, refer to section "API Source Maintenance" on page 3-11.

# Ion Optics

The ion optics focus the ions produced in the API source and transmit them to the mass analyzer. Figure 1-10 shows the schematic view of the Exactive. Voltages for the elements of the ion optics are provided by the ion optic supply dc board (See page 1-41.) and the ion optic supply RF board. (See page 1-41.)

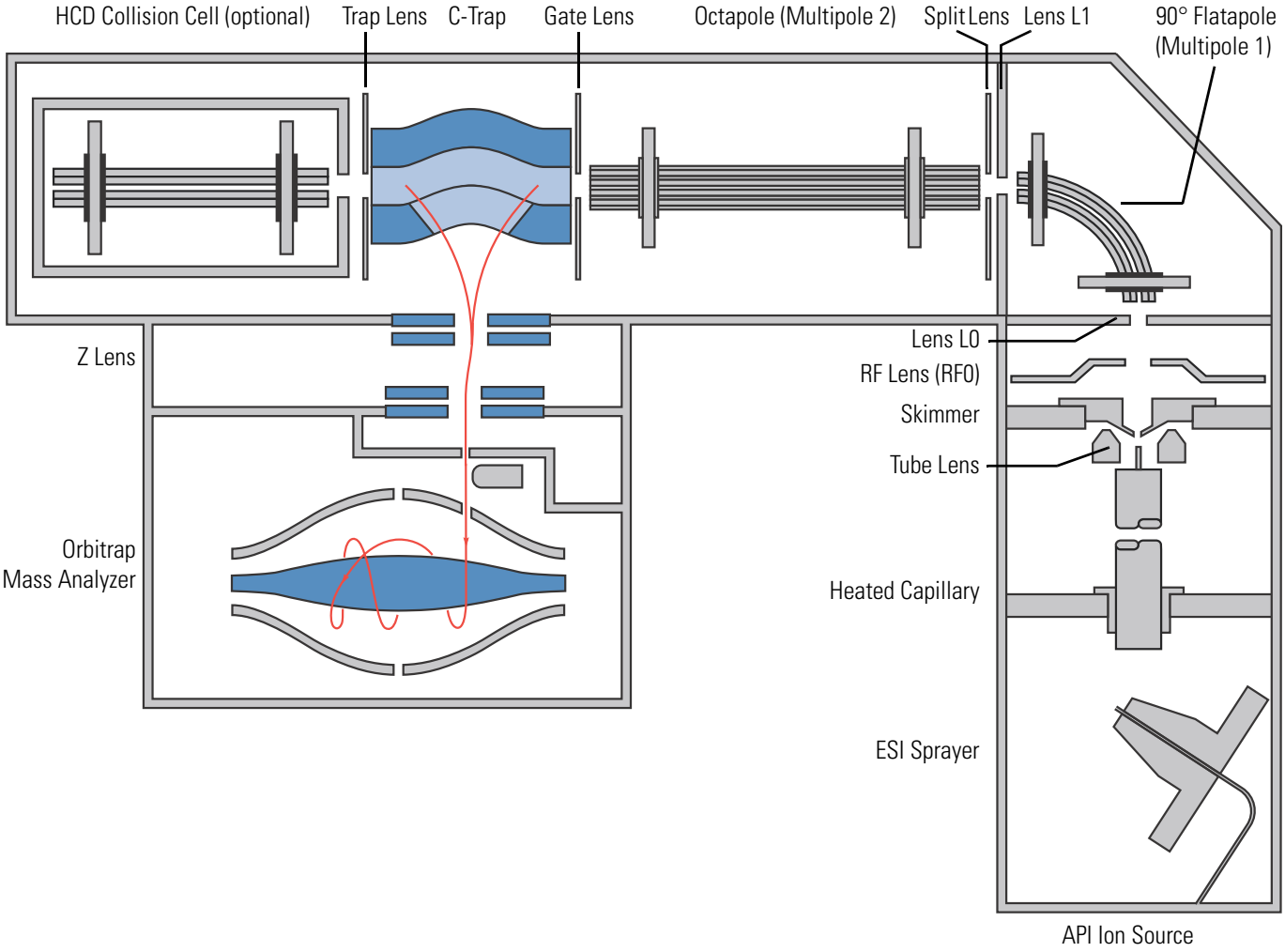



**Figure 1-10.** Schematic view of the Exactive

**Note** Capillary voltage, tube lens voltage, and in-source CID offset voltage are the only ion optics parameters that you can set in the Exactive Tune software. All other ion optics parameters are set automatically. See the *Exactive Software Manual* or the Exactive Tune online Help for details. ▲

## Source Ion Optics

The source ion optics are the ion optics that are located closest to the API source. They include the tube lens, skimmer, RF lens, and lens L0.

Ions from the heated capillary enter the *tube lens*. The tube lens has a mass dependent potential (tube lens voltage) applied to it to focus the ions towards the opening of the skimmer. When you tune the Exactive, you adjust the tube lens potential to maximize sensitivity by balancing desolvation with fragmentation.

Ions from the tube lens pass through the skimmer and move toward the RF Lens. The *skimmer* acts as a vacuum baffle between the ion source interface region (higher pressure) and the RF lens region (lower pressure) of the vacuum manifold. The aperture of the skimmer is offset with respect to the bore of the ion transfer capillary. This arrangement reduces the number of large, charged particles that pass through the skimmer and into the mass analyzer, thereby reducing detector noise.

The *RF lens* is a square array of thin metal elements that acts as an ion focusing device. An RF voltage that is applied to the elements gives rise to an electric field that focuses the ions along the axis of the lens.

In-source CID is accomplished by increasing the dc offset voltage of skimmer, tube lens, and capillary. You use the in-source CID parameter in the Exactive Tune software to adjust this offset voltage.

The *lens L0* is a metal aperture. A potential within ±50 V is applied to lens L0 by the ion optic supply dc board to aid in ion transmission. Lens L0 also acts as a vacuum baffle.

## Analyzer Ion Optics

The analyzer ion optics include multipole 1, lens L1, split lens, and multipole 2. Gate lens, trap lens, and Z-lens are described in the following sections.

The RF-only *multipole 1* always acts as an ion transmission device. The multipole rods are bent through a 90° arc. In addition to reducing the footprint of the instrument, this prevents the transmission of unwanted neutral species to the detector and lowers the noise level in the data.

A potential within ±25 V is applied to *lens L1* by the ion optic supply dc board. Lens L1 also acts as a vacuum baffle.

The *split lens* is used to start and stop the injection of ions into the mass analyzer. It provides a high deflection voltage most of time so that ions are deflected into a baffle except when they are to be allowed into the C-Trap. The fast switching of the ion beam ensures the precise determination of the ion injection time that is required for Automatic Gain Control (AGC).

After passing the RF-only *multipole 2* (Octapole), which acts as another transmission device, the ions enter the C-Trap through the gate lens.

# Orbitrap Analyzer

This section describes the basic principle of the Orbitrap™ mass analyzer. The heart of the system is an axially-symmetrical mass analyzer. It consists of a spindle-shape central electrode surrounded by a pair of bell-shaped outer electrodes. See Figure 1-11. The Orbitrap employs electric fields to capture and confine ions.

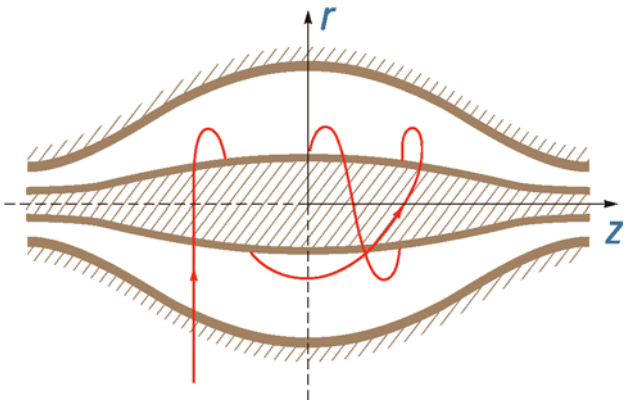



**Figure 1-11.** Schematic view of the Orbitrap cell and example of a stable ion trajectory

## Curved Linear Trap

On their way from the ion source to the Orbitrap, ions move through the gas-free RF octapole (Multipole 2) into the gas-filled curved linear trap (C-Trap). See Figure 2-8 on page 2-14. Ions in the C-Trap are returned by the trap electrode. Upon their passage, the ions loose enough kinetic energy to prevent them from leaving the C-Trap through the gate. The nitrogen collision gas (bath gas) is used for dissipating the kinetic energy of injected ions and for cooling them down to the axis of the curved linear trap.

Voltages on the end apertures of the C-Trap (trap and gate apertures) are elevated to provide a potential well along its axis. These voltages may be later ramped up to squeeze ions into a shorter thread along this axis. The RF to the C-Trap (“Main RF”) as well as the trap and gate dc voltages are provided by the CLT RF main board. (See page 1-43.) The RF voltages to the octapole are provided by the ion optic supply RF board. (See page 1-41.) High voltages to the lens system are provided by the central electrode high voltage power supply board. (See page 1-43.)

## Extraction of Ion Packets

For ion extraction, the RF on the rods of the C-Trap is switched off and extracting voltage pulses are applied to the electrodes, pushing ions orthogonally to the curved axis through a slot in the inner hyperbolic electrode. Because of the initial curvature of the curved trap and the subsequent lenses, the ion beam converges on the entrance into the Orbitrap. The lenses that follow the C-Trap (Z-lens) form also differential pumping slots and cause spatial focusing of the ion beam into the entrance of the Orbitrap. Ions are electrostatically deflected away from the gas jet, thereby eliminating gas carryover into the Orbitrap.

Owing to the fast pulsing of ions from the curved trap, ions of each mass-to-charge ratio arrive at the entrance of the Orbitrap as short packets only a few millimeters long. For each mass-to-charge population, this corresponds to a spread of flight times of only a few hundred nanoseconds for mass-to-charge ratios of a few hundred Daltons per charge. Such durations are considerably shorter than a half-period of axial ion oscillation in the trap. When ions are injected into the Orbitrap at a position offset from its equator (See Figure 1-12.), these packets start coherent axial oscillations without the need for any additional excitation cycle.

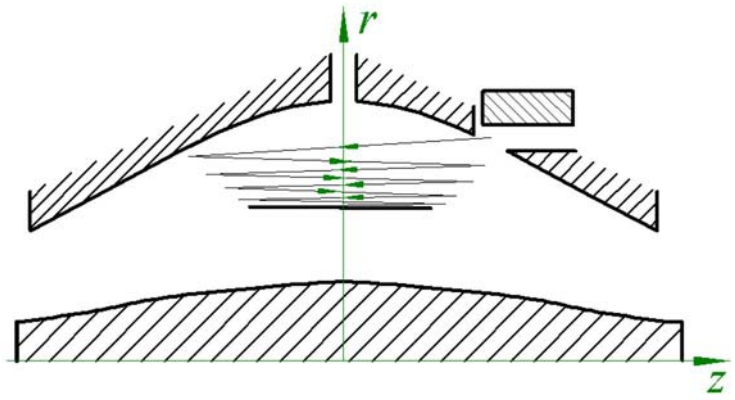



**Figure 1-12.** Principle of electrodynamic squeezing of ions in the Orbitrap as the field strength is increased

The evolution of an ion packet during the increase of the electric field is shown schematically on Figure 1-12. When the injected ions approach the opposite electrode for the first time, the increased electric field (owing to the change of the voltage on the central electrode) contracts the radius of the ion cloud by a few percent. The applied voltages are adjusted to prevent collision of the ions with the electrode. A further increase of the field continues to squeeze the trajectory closer to the axis, meanwhile allowing for newly arriving ions (normally, with higher *m/q*) to enter the trap as well. After ions of all *m/q* have entered the Orbitrap

and moved far enough from the outer electrodes, the voltage on the central electrode is kept constant and image current detection takes place.

## Measuring Principle

In the mass analyzer shown in Figure 1-11 on page 1-21, stable ion trajectories combine rotation around an axial central electrode with harmonic oscillations along it. The frequency $\omega$ of these harmonic oscillations along the z-axis depends only on the ion mass-to-charge ratio $m/q$ and the instrumental constant $k$:

$$w = \sqrt{\frac{q}{m} \times k}$$

Two split halves of the outer electrode of the Orbitrap detect the image current produced by the oscillating ions. By Fast Fourier Transformation (FFT) of the amplified image current, the instrument obtains the frequencies of these axial oscillations and therefore the mass-to-charge ratios of the ions.

## Ion Detection

During ion detection, both the central electrode and deflector are maintained at very stable voltages so that no mass drift can take place. The outer electrode is split in half at $z$=0, allowing the ion image current in the axial direction to be collected. The image current on each of half of the outer electrode is differentially amplified and then undergoes analog-to-digital conversion before processing using the fast Fourier transform algorithm.

As mentioned above, stable ion trajectories within the Orbitrap combine axial oscillations along the z-axis with rotation around the central electrode and vibrations in the radial direction. (See Figure 1-11 on page 1-21.) For any given $m/q$, only the frequency of axial oscillations is completely independent of initial ion parameters, whereas rotational and radial frequencies exhibit strong dependence on initial radius and energy. Therefore, ions of the same mass-to-charge ratio continue to oscillate along $z$ together, remaining in-phase for many thousands of oscillations.

In contrast to the axial oscillations, the frequencies of radial and rotational motion will vary for ions with slightly different initial parameters. This means that in the radial direction, ions dephase orders of magnitude faster than in the axial direction, and the process occurs in a period of only 50–100 oscillations. After this, the ion packet of a given $m/q$ assumes the shape of a thin ring, with ions uniformly distributed along its circumference. See Figure 1-13 on page 1-24. Because of this

angular and radial smearing, radial and rotational frequencies cannot appear in the measured spectrum. Meanwhile, axial oscillations will persist, with axial thickness of the ion ring remaining small compared with the axial amplitude. Moving from one half outer electrode to the other, this ring will induce opposite currents on these halves, thus creating a signal to be detected by differential amplification.

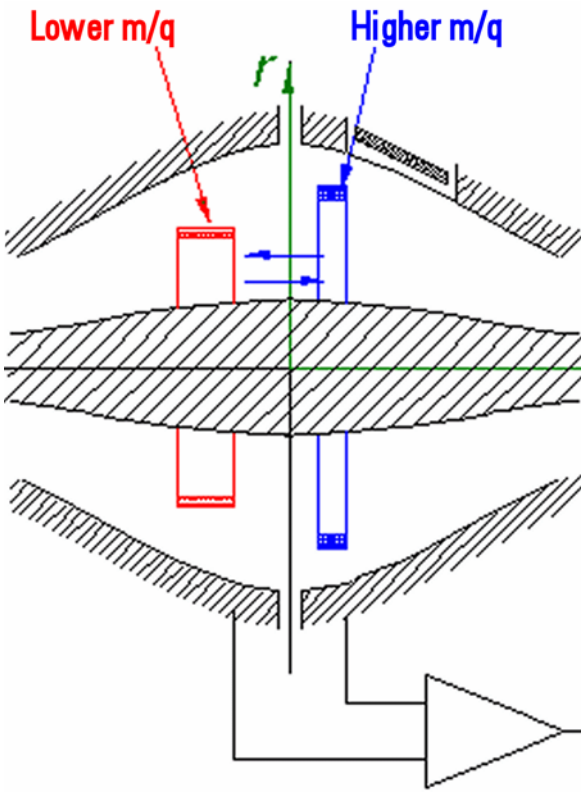



**Figure 1-13.** Approximate shape of ion packets of different m/q after stabilization of voltages

# HCD Collision Cell

The optional HCD collision cell consists of a straight multipole mounted inside a metal tube, which is connected in direct line-of-sight to the C-Trap. It is supplied with a collision gas through the open split interface, providing increased gas pressure inside the multipole. The choice of collision gas is independent from the gas in the C-Trap. See "Gas Supply" on page 1-37 for details. The front of the tube is equipped with a lens for tuning transmission and ejection to/from the C-Trap. The ion optic supply dc board provides the dc voltages for the entrance lens and the reflection lens of the collision cell. (See page 1-41.)

For HCD (Higher Energy Collisional Dissociation), ions are passed through the C-Trap into the collision cell. The offset between the C-Trap and HCD is used to accelerate the parents into the gas-filled collision cell. A potential gradient is applied to the collision cell to provide fast extraction of ions, such that it transmits ions at a reliable rate.

The fragment spectra generated in the collision cell and detected in the Orbitrap show a fragmentation pattern comparable to the pattern of typical triple quadrupole spectra. See the *Exactive QuickStart Guide* for more information.

# Additional Hardware

This section describes additional equipment that can be used for operating the Exactive.

## Syringe Pump

The Exactive requires a syringe pump for delivering sample solution and/or sheath liquid from a syringe into the ESI ion source. A suitable syringe pump (Chemyx Fusion 100) is available from Thermo Fisher Scientific (P/N 1245740). See Figure 1-14.

**Figure 1-14.** Exactive with Chemyx Fusion 100 syringe pump

An RS232 serial interface allows controlling established syringe pumps by the instrument software. The external connection for the communication between MS detector and syringe pump is located on the power panel. See Figure 1-5 on page 1-9. Because the Exactive does not provide electric power for the syringe pump, connect the power cord of the pump to a wall outlet instead. "Connecting the Inlet Plumbing" on page B-3 provides instructions on connecting the syringe pump to the MS detector.

Syringe pump parameters can be set via the Exactive Tune software software. The Exactive Tune software allows switching on/off the syringe pump by an instrument method or manually. See the *Exactive Software Manual* or the Exactive Tune online Help for details.

## Switching Valves

The Exactive allows connecting up to two external switching valves. Suitable switching valves (Rheodyne MX Series II™) are available from Thermo Fisher Scientific (P/N 1239650). The valves are controlled by the instrument software by means of contact closures. The external connection for the valves is located on the power panel. See Figure 1-5 on page 1-9. Plug the power cord of a switching valve into a properly grounded wall outlet. The Universal Power Supply of the valve can be operated from inputs of 100–240 V ac, 50-60 Hz.

You can configure (plumb) a switching valve as a loop injector (for flow injection analysis) or as a divert valve. Procedures for plumbing the valve in the loop injector or divert valve configuration are given in Appendix B: “Getting Connected”.

You can control the switching valves from the data system. Refer to the online Help for instructions on operating the switching valve from the data system.

You can also use the switching valve button to divert the LC flow between the mass spectrometer and waste when the valve is in the divert valve configuration, or switch between load and inject modes when the valve is in the loop injector configuration.

# Vacuum System

The vacuum manifold encloses the ion source interface, ion guides, C-Trap, and the Orbitrap mass analyzer. The vacuum manifold consists of thick-walled aluminum chambers with machined flanges on the front, sides, and bottom, and various electrical feedthroughs and gas inlets.

The vacuum manifold is divided into three chambers. The region inside the first chamber, called the capillary-skimmer region, is evacuated by a rotary-vane pump (forepump). The region inside the second chamber, called the source chamber, is evacuated by a turbomolecular pump (source TMP). The region inside the third chamber, called the analyzer chamber, is evacuated by another turbomolecular pump (analyzer TMP). Figure 1-15 shows a schematic overview of the vacuum system, Table 1-3 shows the vacuum regions of the Exactive.

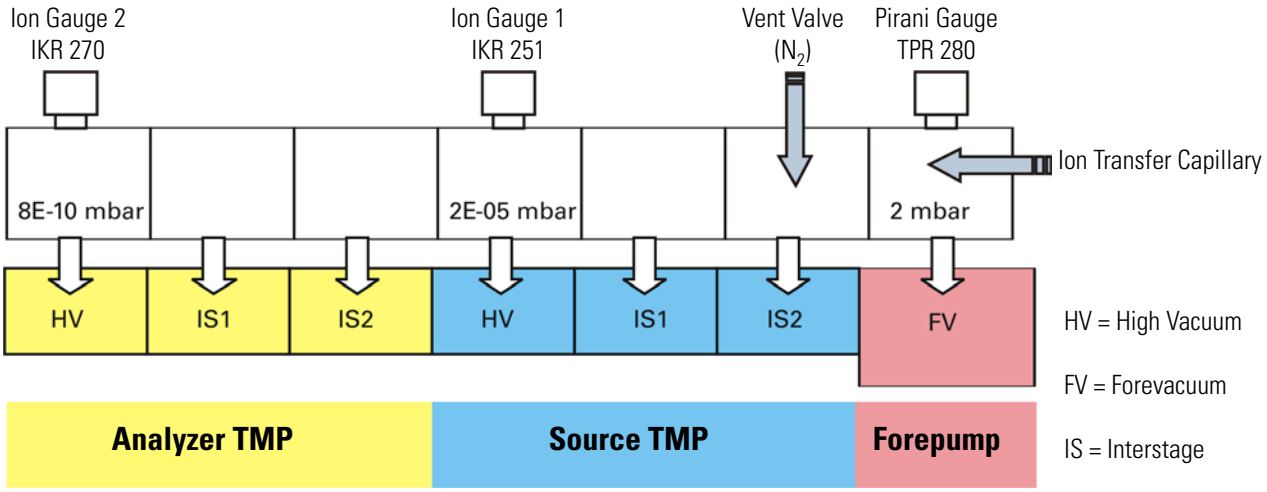



**Figure 1-15.** Schematic view of vacuum system

**Table 1-3.** Vacuum regions overview

| Region in instrument | Vacuum [mbar] | applied by |
|---|---|---|
| Ion Max housing | 1E+03 | Atmosphere |
| Capillary-skimmer region | 2 | Forepump |
| RF lens | | Source TMP IS2 |
| Multipole 1 | | Source TMP IS1 |
| Multipole 2 / C-Trap | 1E-05 to 3E-05 | Source TMP HV |
| Z-lens | | Analyzer TMP IS2 |
| Region between Z-lens and Orbitrap | | Analyzer TMP IS1 |
| Orbitrap | < 8E-10 | Analyzer TMP HV |

See page 1-33 for information about the vacuum gauges in the Exactive.

## Forepump

A single stage rotary vane pump (SOGEVAC® SV 40 BI, manufacturer: Oerlikon Leybold Vacuum) is used as forepump. It establishes the vacuum necessary for the proper operation of the source TMP. The forepump also evacuates the capillary-skimmer region of the vacuum manifold. The pump has a rated pumping speed of about 42 $m^3$/h at 50 Hz (50 $m^3$/h at 60 Hz).

The forepump is placed on a drip pan under the workbench immediately behind the Exactive. See Figure 1-16. A section of 4.5 cm (1.8 in.) ID reinforced PVC tubing connects it to the vacuum port at the rear side of the Exactive. See Figure 1-4 on page 1-8. A noise reduction cover for the forepump is delivered with the instrument.

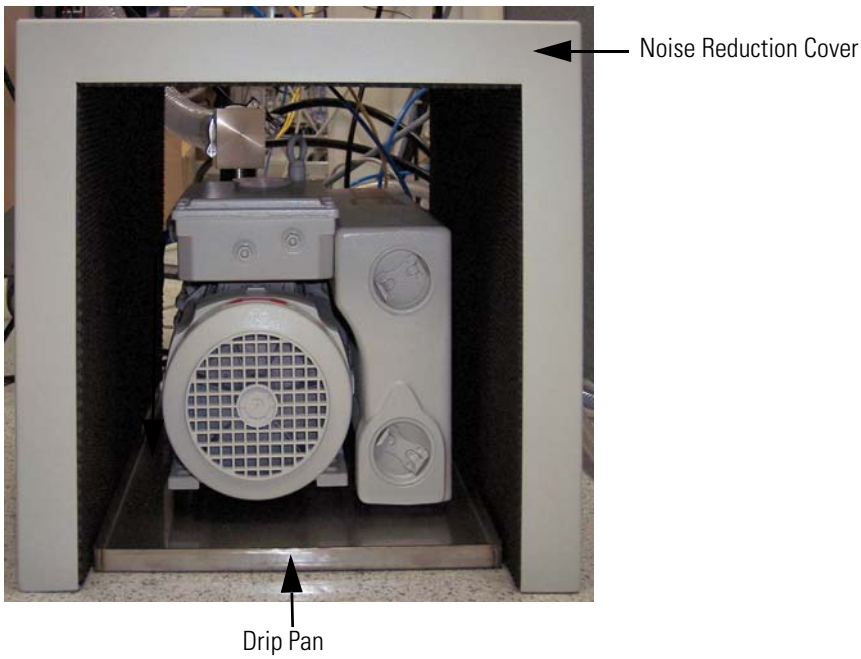



**Figure 1-16.** Forepump with noise reduction cover and drip pan

**Warning** **Risk of burn by touching.** Pump in function is hot and some surfaces could reach a temperature higher than 80 °C (176 °F). Take note of the warning labels on the pump. ▲

An exhaust hose connects the forepump to the exhaust system in the laboratory.

**Warning** **Hazardous materials may be present in the effluent of the forepump.** The connection to an adequate exhaust system is mandatory! ▲

The power cord of the forepump is plugged into the outlet labeled Forepump on the power column. (See Figure 1-6 on page 1-11.) This outlet supplies power to the pump and is controlled by the main power circuit breaker switch and not by the electronics service switch.

Two models of the SOGEVAC SV 40 BI pump are used with Exactive instruments. The model with On/Off switch has an overload breaker integrated in the motor connection box and can be directly connected to the instrument. See Figure 1-17.


**Figure 1-17.** Forepump with On/Off switch

The model without On/Off switch must be used with an external overload protection between the pump and the Exactive instrument. See Figure 1-18.

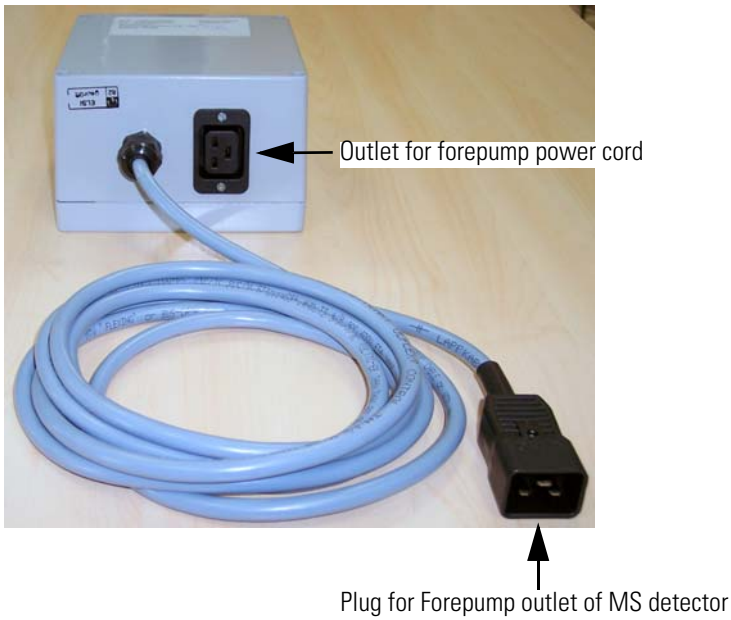



**Figure 1-18.** Overload protection for forepump without On/Off switch

**Caution** Always plug the forepump power cord into the outlet labeled Forepump on the rear side of the MS detector. Never plug it into a wall outlet. If the pump is not plugged into the Exactive rear, it will not shut down when the instrument is powered off. As a result, the pump will continue to run and the Exactive will not be able to vent to atmosphere. ▲

Before using the forepump, make sure that the following conditions are met:

- If the forepump is equipped with an On/Off switch, the switch is set to the On (I) position.
- The forepump is filled with oil.
- It is connected to the power supply, and
- The gas ballast is shut.

For a detailed description of the forepump and for instructions on user maintenance, refer to the handbook of the manufacturer. See also "Maintenance of the Forepump" on page 3-5.

## Turbomolecular Pumps

Two turbomolecular pumps (TMPs) provide the vacuum for the Exactive. The TMPs are controlled by individual controllers and powered by +24 V dc (480 W) power supplies. Power for the TMPs is turned off by the main power circuit breaker switch, but not by the electronics service switch. The TMPs are air cooled by fans that are mounted in fan inserts on the bottom of the instrument.

Pump controllers provide power to and control of the TMPs. The status of each TMP (such as temperature or rotational speed) is sent from the TMP controller to the vacuum control board.

### Source TMP

The vacuum in the source chamber is created by a three-stage split flow TMP (SplitFlow 310, manufacturer: Pfeiffer Vacuum). The TMP mounts onto the bottom of the vacuum manifold. The source TMP has three pumping inlets (see Figure 1-15 on page 1-28):

- A 200 L/s high-vacuum (HV) inlet at the top of the rotor stack, which evacuates the multipole 2 / C-Trap region.
- An 155 L/s interstage inlet (IS1) about half way down the rotor stack, which evacuates the multipole 1 region.

- A 30 L/s interstage inlet (IS2) in the molecular drag section of the pump, which evacuates the RF lens region.

The forepump provides the forevacuum for the source TMP.

For a detailed description of the source TMP and for instructions on user maintenance, refer to the handbook of the manufacturer. See also "Maintenance of the Source TMP" on page 3-6.

### Analyzer TMP

The vacuum in the analyzer chamber is created by a second three-stage split flow TMP (TURBOVAC TW 290/20/20-UHV, manufacturer: Oerlikon Leybold Vacuum). The pump mounts onto the bottom of the vacuum manifold. The analyzer TMP has three pumping inlets (see Figure 1-15 on page 1-28):

- A 250 L/s high-vacuum (HV) inlet at the top of the rotor stack, which evacuates the UHV (Orbitrap) chamber.
- An 20 L/s interstage inlet (IS1) about half way down the rotor stack, which evacuates the region between Z-lens and Orbitrap.
- A 20 L/s interstage inlet (IS2) in the molecular drag section of the pump, which evacuates the Z-lens region.

The interstage inlet IS1 of the source TMP provides the forevacuum for the analyzer TMP.

For a detailed description of the analyzer TMP and for instructions on user maintenance, refer to the handbook of the manufacturer. See also "Maintenance of the Analyzer TMP" on page 3-6.

## Vacuum System Controls

An interface for RS485 data connects the TMPs to the vacuum control board. The vacuum gauges of the Exactive are controlled by the same board. (See "Vacuum Control Board" on page 1-42.)

## Vacuum Gauges

As shown in Table 1-4, vacuum in the Exactive is measured in three regions.

**Table 1-4.** Vacuum measurement regions in the Exactive

| Region | Gauge type | Name in Exactive Tune | Typical values |
|---|---|---|---|
| Capillary-skimmer region | Pirani | Forevacuum | about 2 mbar (depends on temperature of heated capillary) |
| Multipole 2 region | Cold cathode | High Vacuum | from about 1E-05 mbar (without HCD) to about 3E-05 mbar (with HCD) |
| Orbitrap analyzer | Cold cathode | Ultra High Vacuum | < 8E-10 mbar |

The vacuum is monitored by three vacuum gauges:

- The vacuum in the capillary-skimmer region is monitored by an Active Pirani gauge (TPR 280, manufacturer: Pfeiffer Vacuum) connected to the Exactive forevacuum line.
- The vacuum in the multipole 2 region is monitored by a Compact Cold Cathode Gauge (IKR 251, manufacturer: Pfeiffer Vacuum).
- The vacuum in the Orbitrap chamber of the Exactive is monitored by a Compact Cold Gauge (IKR 270, manufacturer: Pfeiffer Vacuum).

## Switching on the Vacuum System

When the vacuum system is switched on, the following occurs:

1. After the main switch is switched On, the pumps of the Exactive are run up. To monitor the vacuum readings in the Exactive Tune software, it is necessary to switch on the electronics switch, too. The Pirani gauge (see above) controls the pressure at the forepump. Within a short time, a significant pressure decrease must be observed. The goodness of the vacuum can be estimated by means of the rotation speed of the TMPs (e.g. 80% after 15 minutes).
2. If the working pressure is not reached after the preset time, the complete system is switched off.

**Note** The vacuum control board triggers an alert in the Exactive Tune software when a vacuum failure has occurred. ▲

3. The Ion Gauge 1 (IKR 251) is switched on only after the source TMP has exceeded 90% of its maximum rotation speed for five minutes. To extend its life time, this ion gauge is switched off automatically after 30 minutes.

**Note** For diagnostic purposes, the ion gauge can be switched on manually in the instrument status view of the Exactive Tune software. ▲

If the pressure exceeds 1E-4 mbar for more than 10 seconds , the ion gauge is switched off. After five minutes, the ion gauge is switched on again. After three failed attempts it is only possible to switch on Ion Gauge 1 manually in the Exactive Tune software.

4. The Ion Gauge 2 (IKR 270) is switched on only after both TMPs have exceeded 90% of their maximum rotation speed for five minutes. It is then used to monitor the vacuum in the Orbitrap region.

   If the pressure exceeds 1E-4 mbar for more than 10 seconds, the ion gauge is switched off. After five minutes, the ion gauge is switched on again. After three failed attempts it is only possible to switch on Ion Gauge 2 manually in the Exactive Tune software.

5. The Vacuum LED on the system panel is illuminated green when all the following conditions are met:

   - In the Exactive Tune software, all LEDs are green. (The Ion Gauge 1 is allowed to be off).
   - Analyzer temperature is below 45°C.
   - Both TMP frequencies have exceeded 90% of their maximum rotation speed.

6. When the vacuum measured by the Ion Gauge 2 is better than 1E-8 mbar, the power supplies of the high voltage electronics and the capillary heater are switched on.

**Note** Only if both ion gauges are defect, it is not possible to switch on the RF and the high voltages. ▲

The Exactive is now ready for measurements.

## Vent Valve

The vent valve is a solenoid-operated valve that allows the vacuum manifold to be vented. The vent valve is closed when the solenoid is energized. The vacuum control board switches the vent valve.

The vacuum manifold is vented when external power is removed from the instrument. (Power is removed from the instrument by a power failure or by placing the main power circuit breaker in the Off (O) position.) Power is provided to the vent valve for a short time after the external power is removed. If external power is not restored to the instrument within this time, power to the vent valve solenoid is shut off.

When power to the vent valve solenoid is shut off, the vent valve opens and the manifold is vented with nitrogen. See Figure 1-15 on page 1-28 and Figure 1-19 on page 1-37. The vent valve closes after power is restored to the instrument.

## System Bakeout

After the system has been open to the atmosphere (e.g. for maintenance work), the vacuum deteriorates due to contaminations of the inner parts of the vacuum system caused by moisture or a power outage. These contaminations must be removed by heating the vacuum system: a system bakeout. See "Baking Out the System" on page 3-7 for instructions on performing a system bakeout.

### Bakeout Devices

To provide the high temperatures required for performing a system bakeout, the Exactive is equipped with two halogen bulbs inside the vacuum chamber. Furthermore, two heating cartridges are built into the block that serves as housing for the analyzer TMP.

### Bakeout Control

For a system bakeout, the instrument software simultaneously switches all heaters by relays. The vacuum control board controls the bakeout procedure. For example, the heaters are switched off in case of a failure of the fan below the analyzer TMP.

**Note** Individual printed circuit boards have also devices for checking the actual temperature. ▲

# Cooling Fans

Six fans provide cooling for the Exactive. On the right instrument side, a fan insert with four fans cools the source TMP and the electronic boards. On the left instrument side, a second insert with two fans cools the analyzer chamber and the analyzer TMP. From the rear of the MS detector, air is drawn in through fan filters. The exhaust air is expelled from the ventilation slots on the sides of the MS detector. For maintenance instructions for the fan filters, see "Maintenance of the Fan Filters" on page 3-9.

**Caution** To ensure safety and proper cooling, always operate the Exactive with its covers in place! ▲

**Caution** Avoid blocking the ventilation slots at the rear of the instrument. Items might fall behind the instrument, inhibit airflow, and cause the system to overheat. ▲

The vacuum control board monitors all fans within the instrument. During a system bakeout it switches off one of the two fans below the analyzer TMP. In case of a fan failure, the board triggers an alert in the Exactive Tune software. In case of a failure of the fans below the analyzer TMP, no bakeout procedure is possible.

**Note** In addition to the fans described in this topic, various printed circuit boards are equipped with individual fans. ▲

# Gas Supply

The Exactive requires high-purity (99%) nitrogen for the API source gases (sheath/auxiliary/sweep gas) and for the collision gas (bath gas) of the curved linear trap. Nitrogen is also used to vent the system via the vent valve. Furthermore, nitrogen serves as collision gas for the optional HCD collision cell.[1] If argon is used as HCD collision gas, it should be of high purity (99.99%). For all gases, the required pressure is 690 ±140 kPa (6.9 ±1.4 bar, 100 ±20 psi). Both gas lines are protected against overpressure by pressure relief valves. Figure 1-19 shows a schematical view of the gas supply in the instrument.

**Caution** Do not connect other gases than nitrogen or argon to the Exactive! The maximum pressure for both gas inlets is 830 kPa (8.3 bar, 120 psi). ▲

Checking the gas supplies in the laboratory is one of the daily routines to perform before you can start operating the Exactive. See page 2-2.

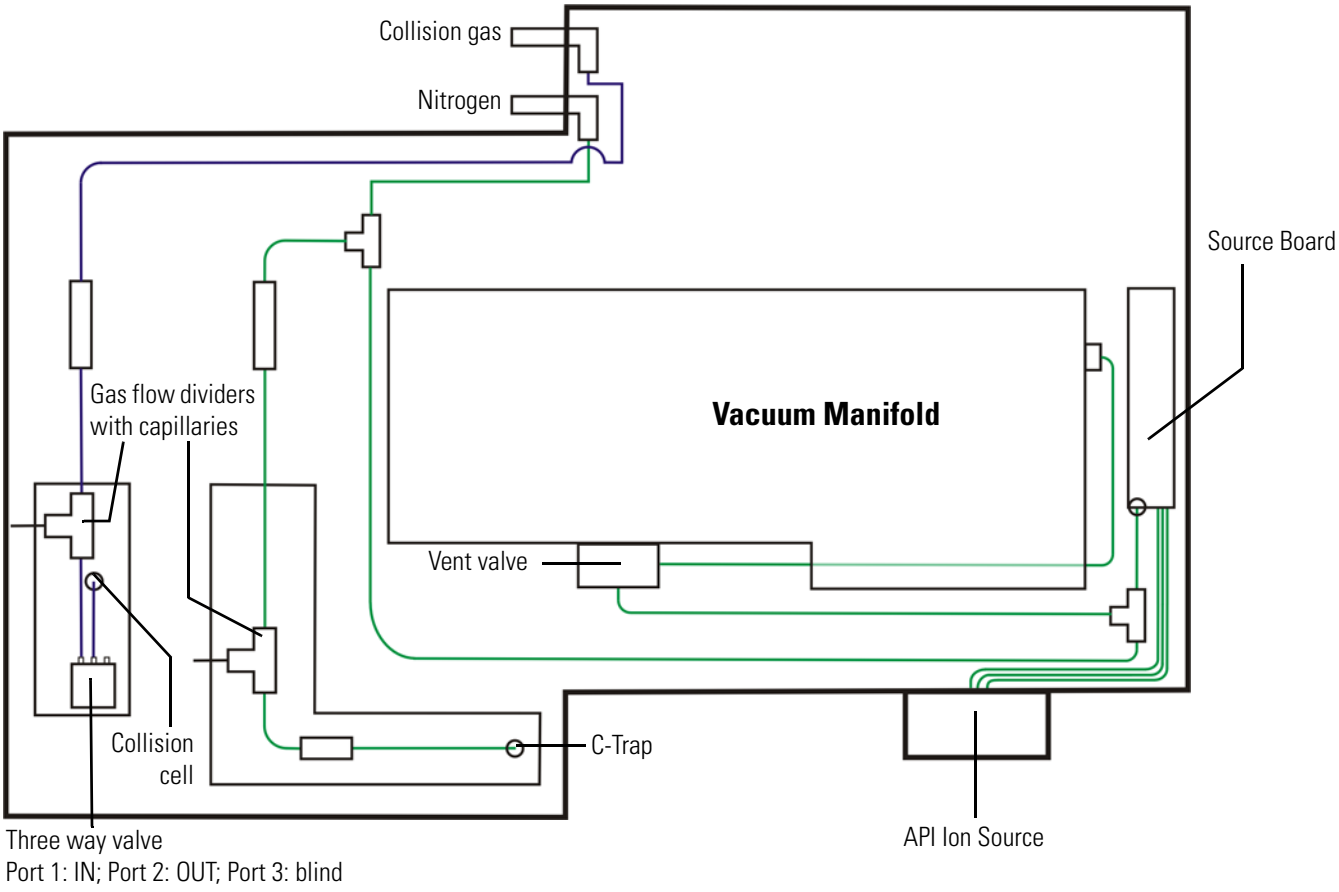



**Figure 1-19.** Schematic view of the gas supply

[1]HCD is an option for Exactive. The feature will be not available if the instrument is not equipped with this option.

## Nitrogen Gas Distribution

Connect an appropriate length of Teflon™ tubing to the nitrogen source in the laboratory. The Exactive Installation Kit contains 10 m (33 ft) of suitable Teflon tubing (OD 6 mm, P/N 0690280). The connection for the Teflon hose to the nitrogen gas supply is not provided in the kit; you have to supply this part. Connect the opposite end of the Teflon tubing to the press-in fitting labeled Nitrogen, which is located at the rear side of the instrument. See Figure 1-6 on page 1-11. To connect the tubing, align the Teflon tubing with the opening in the fitting and firmly push the tubing into the fitting until the tubing is secure.

**Warning Danger of asphyxiation.** Accumulation of nitrogen gas could displace sufficient oxygen to suffocate personnel in the laboratory. Ensure that the laboratory is well ventilated. ▲

Part of the gas flow from the nitrogen port is directed through Teflon tubing to the vent valve (See page 1-34 for further information.) and the source board. On the board, a valve terminal divides the nitrogen flow into three streams to the API source (sheath/auxiliary/sweep gas). Another part of the nitrogen flow is directed through Teflon tubing to the C-Trap. Nitrogen gas pressure to the C-Trap is kept constant by using an "open-split" interface (gas flow divider). It contains a capillary line from the nitrogen line of the instrument to atmosphere (flow rate: ~20 mL/min), with another capillary leading from the point of atmospheric pressure into the C-Trap (flow rate: ~0.2 mL/min). For the nitrogen gas to the C-Trap, *black* PEEKSil™ tubing is used (75 µm ID silica capillary in 1/16 in. PEEK tubing).

## Collision Gas Distribution

If your instrument is equipped with the optional HCD collision cell, connect the collision gas source to the press-in fitting labeled Collision Gas. See Figure 1-6 on page 1-11. If you intend to use nitrogen as HCD collision gas, connect the tubing coming from the nitrogen supply to the T-piece (P/N 1128140) that is also included in the Exactive Installation Kit. Connect the other ports of the T-piece with short lengths of Teflon tubing to the collision gas inlet and the nitrogen inlet. If you intend to use argon as HCD collision gas, proceed in an analogous manner as described for nitrogen in "Nitrogen Gas Distribution" above.

From the collision gas inlet, the HCD collision gas is led to a gas flow divider. Part of the gas is led through a capillary line to the atmosphere (flow rate: ~20 mL/min). The other part of the gas (flow rate: ~0.5 mL/min) enters the IN port of a three way valve. The gas leaves the valve through the OUT port and is led to the collision cell. The third

port of the valve is closed. For the HCD collision gas, *red* PEEKSil™ tubing is used (100 µm ID silica capillary in 1/16 in. PEEK tubing).

The three way valve is switched by the software via the vacuum control board. (See page 1-42.)

# Printed Circuit Boards

The Exactive is controlled by a PC running the Xcalibur™ software suite. The software controls all aspects of the instrument. The main software elements are the control of ion detection and the control of the Orbitrap mass analyzer. The following pages contain a short overview of the various electronic boards of the Exactive.

The electronics of the Exactive contains complicated and numerous circuits. Therefore, only qualified and skilled electronics engineers should perform servicing.

A Thermo Fisher Scientific Service Engineer should be called if servicing is required. It is further recommended to use Thermo Fisher Scientific spare parts only. Before calling a Service Engineer, please try to localize the defect via errors indicated in the Exactive Tune software. A precise description of the defect will ease the repair and reduce the costs.

**Warning Danger of electric shock.** Parts of the printed circuit boards are at high voltage. Opening the electronics cabinet is only allowed for maintenance purposes by qualified personal. ▲

## Preamplifier

The preamplifier board contains a broadband preamplifier with differential high-impedance inputs. It serves as a detection amplifier and impedance converter for the image current created by the oscillating ions. The output current is transferred to the data acquisition board. By means of a relay, the data acquisition board provides the electric power for the preamplifier.

## Source Board

The source board controls the temperature of the ion source. Each source type is identified by its specific resistance. The board also controls the flow rates of the gases in use. The board controls the 8 kV power supply for the ion sources. By means of a safety relay, it switches off the power supply when the source is removed from the Exactive. The source board controls the heaters for the heated capillary and the APCI heater. It also provides support for the APPI lamp. Communication between the source board and external ion sources is established by an RS232 connection (Serial Source Interface) to the FT adapter board.

The source board distributes power to the ion gauges, the switching valves, the syringe pump, and the nitrogen gas valves – including the vent valve. Two connections to the I/O board allow the source board to communicate with peripheral devices.

The system status LEDs on the front side of the instrument (See Figure 1-3 on page 1-7.) are controlled by the source board. The displayed status information partially comes from other boards. The board communicates via the SPI bus.

## 8 kV Power Supply

The 8 kV power supply delivers voltage to either the ESI needle in the ESI mode, or the corona discharge needle in the APCI mode. Typical operating voltages range from ±3 to ±6 kV. In the ESI mode the voltage is regulated, whereas in the APCI mode the current is regulated. This power supply is controlled by the source board.

## Ion Optic Supply DC Board

The ion optic supply dc board provides the dc voltages for heated capillary, tube lens, skimmer, lens L0, and lens L1. If the board detects an HCD collision cell, it provides the dc voltages for the entrance lens and the reflection lens.

## Ion Optic Supply RF Board

The ion optic supply RF board provides all RF voltages for the complete ion optics from the heated capillary to the multipole 2. If the board detects an HCD collision cell, it also provides the RF voltage for it. It has an RF detector for the RF output control. Furthermore, the board provides the dc voltages for the split lens.

## FT Adapter Board

The FT adapter board links the internal computer to the various system components. Its main function is the control of scan events within the instrument. The board contains a micro controller, an FPGA (Field Programmable Gate Array), and serial port connectors.

Via the USB interface, the internal computer transmits set commands for system components to the FT adapter board. By the same interface, the internal computer requests readback values. The FT adapter board converts the set commands during the scan and sends the readback data via USB to the internal computer. The master interface of the Extended Orbitrap SPI bus of the FT adapter board serves for transmitting the set commands and readbacks.

Communication between the FT adapter board and external ion sources is established by an RS232 connection (Serial Source Interface) to the source board. The FT adapter board communicates with the syringe pump via the I/O board.

### SPI Bus

Various boards communicate system parameters and monitor readbacks over the Serial Peripheral Interface (SPI) bus. The master interface of the SPI bus is located on the FT adapter board.

## Data Acquisition Board (DAQ Board)

The data acquisition board converts detected ion signals coming from the preamplifier to digital form. Then it sends the digital signals via an USB connection to the mainboard of the internal computer. The SPI bus is used for transmitting set commands and readbacks. A trigger input is used for synchronizing the DAQ board with the FT adapter board. By means of a relay, the DAQ board provides the electric power for the preamplifier.

## Internal Computer (Data Acquisition Unit)

The internal computer performs the Fast Fourier Transformation (FFT) of the image current after it has been converted by the DAQ board.

The internal computer communicates with the data system computer and LC modules via a 10/100 base-T Ethernet switch. USB connections connect it to the vacuum control board, the data acquisition board, and the FT adapter board.

## Vacuum Control Board

The vacuum control board controls the vacuum system. Depending on the quality of the vacuum and the status of the TMPs, it switches the vacuum gauges, the pumps, and the 230 V relays. When the vacuum measured by the Ion Gauge 2 is better than 1E-8 mbar, the vacuum control board switches on the power supplies of the high voltage electronics and the capillary heater.

On the vacuum control board, analog signals from vacuum gauges are converted to digital signals and passed to the internal computer via an USB connection. TMPs are attached to a serial port connector and this is connected via the signal lines to the vacuum control board. (See "Vacuum System" on page 2-19.)

The board controls external relays with 24 V dc connections. In addition to switching the vent valve, the board also switches the valve that controls the flow of the HCD collision gas. The vacuum control board controls the bakeout heaters. It also monitors six fans within the instrument; it switches off the two fans below the analyzer TMP during a system bakeout. In case of a fan failure, the board triggers an alert in the Exactive Tune software.

## CLT-RF Board

The CLT RF board operates the C-Trap with four phases RF voltage (“Main RF”) and three pulsed dc voltages (PUSH, PULL, and OFFSET). It allows switching off the RF and simultaneous pulsing of each CLT electrode. See “Orbitrap Analyzer” on page 2-13 for further information. The board also provides the trap voltage and the gate voltage to the C-Trap. The board communicates with the other boards via the SPI bus.

## CE HV Supply Board

The central electrode high voltage supply board provides five voltages for the ion optics of the Exactive:

- Two high voltages power lenses that follow the C-Trap.
- Two dc voltages and one ac voltage are applied to the RF CLT main board to be used as focusing potentials for the curved linear trap.

Furthermore, the board supplies six dc voltages to the Orbitrap:

- Four central electrode (CE) voltages: CE POS, CE NEG, CE LOW+, and CE LOW-.
- Two deflector electrode (DE) voltages: DE HIGH and DE LOW.

For positive ions, the CE voltages are negative and the DE voltages are positive. The maximum CE voltage is 5 kV and the maximum DE voltage is 1.25 kV. See “Orbitrap Analyzer” on page 2-13 for further information.

Also, the AD conversion of the PT-100 signal takes place on this board.

The board communicates via the SPI bus. A Peltier element on the rear side of the board serves as means of dissipation.

## CE Pulser Board

**Warning Danger of electric shock.** The CE high voltage supply board creates voltages up to 5 kV! ▲

The central electrode pulser board switches the injection and measurement voltages for the central electrode and the detection electrodes of the Orbitrap. Resistor-capacitor circuits on the board convert the switching pulse into a smooth transition between the voltages. Furthermore, the board switches the polarity of the central Orbitrap electrode.

The temperature of the Orbitrap analyzer is measured by the PT-100 temperature probe. The signal of the probe is converted and analyzed on the CE pulser board. Then it is sent to the CE HV supply board where AD conversion takes place.

## Power Supply DAQ Board

The power supply DAQ board provides the voltages that are required by the data acquisition board. The board is equipped with a fan for heat dissipation.

## I/O Board

The I/O board provides the interfaces for data exchange with peripheral devices of the Exactive. It contains a reset switch, four relay outputs, a digital inlet port, two analog inlet ports, and a serial interface for controlling an external syringe pump. (See "Peripheral Control" on page 1-9.) It is connected to the FT adapter board and to the source board.

# Chapter 2 Daily Operation

This chapter outlines the checks and cleaning procedures of the Exactive system that should be performed every day to ensure the proper operation of your system. It contains the following sections:

- Things to Do Before Operating the Exactive System
- Things to Do After Operating the Exactive System

Additional information for basic system operations are described in Chapter 4: "System Shutdown, Startup, and Reset". Maintenance procedures for the Exactive are described in Chapter 3: "User Maintenance".

# Things to Do Before Operating the Exactive System

Every day before starting analyses, verify that the instrument is ready for operation by doing the following:

- Checking the Argon and Nitrogen Supplies
- Checking the ESI Fused-Silica Sample Tube for Elongation
- Checking the System Vacuum Levels
- Checking the Disk Space on the Data System
- Checking the Mass Accuracy of the Instrument

## Checking the Argon and Nitrogen Supplies

If you use argon as HCD collision gas, check the argon supply on the regulator of the gas tank. Make sure that you have sufficient gas for your analysis. If necessary, install a new tank of argon. Verify that the pressure of argon reaching the mass spectrometer is between 690 ± 140 kPa (6.9 ± 1.4 bar, 100 ± 20 psi). If necessary, adjust the pressure with the tank pressure regulator.

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. Based on 24 hour per day operation, typical nitrogen consumption is 5560 L (200 $ft^3$) per day. If necessary, replace the tank. Verify that the pressure of nitrogen reaching the mass spectrometer is between 690 ± 140 kPa (6.9 ± 1.4 bar, 100 ±20 psi). If necessary, adjust the pressure with the tank pressure regulator.

**Warning** **The presence of oxygen in the ion source when the mass spectrometer is On could be unsafe.** Before you begin normal operation each day, ensure that you have sufficient nitrogen for your API source. The Exactive system displays a message if the nitrogen pressure is too low. ▲

Go to the next topic: “Checking the ESI Fused-Silica Sample Tube for Elongation” .

## Checking the ESI Fused-Silica Sample Tube for Elongation

Using acetonitrile in the mobile phase can cause elongation of the polyimide coating on the fused-silica sample tube. Elongation of the polyimide coating can degrade signal intensity and signal stability over time.

If you are running in ESI mode with a fused-silica sample tube, verify that the sample tube is not elongated past the tip of the ESI spray needle. Cut and reposition the end of the sample tube 1 mm inside the end of the ESI needle.

❖ **To cut and reposition the end of the sample tube**

1. Remove the ESI probe from the Ion Max ion source by following the procedure described in the *Ion Max and Ion Max-S API Source Hardware Manual*.
2. Loosen the sample inlet fitting.
3. Gently pull back on the sample tube to free it from the fitting.
4. Push the sample tube forward so that it extends beyond the end of the electrospray needle.
5. Use a fused-silica cutting tool to cut off a small length of the sample tube. Ensure that you cut the end of the sample tube squarely.
6. Pull the sample tube backwards until the exit end of the sample tube is recessed just inside the ESI needle by approximately 1 mm.
7. Tighten the sample inlet fitting securely to hold the sample tube in place.

**Note** The sample tube might move forward when you tighten the sample inlet fitting. Ensure that the sample tube is retracted into the ESI needle approximately 1 mm. If necessary, loosen the fitting and reposition the sample tube. ▲

8. Reinstall the ESI probe as described in the *Ion Max and Ion Max-S API Source Hardware Manual*.

Go to the next topic: "Checking the System Vacuum Levels" .

## Checking the System Vacuum Levels

For proper performance, your Exactive system must operate at the proper vacuum levels. Operation of the system with poor vacuum levels can cause reduced sensitivity and tuning problems. Check your system for air leaks by checking the system vacuum levels before you begin your first acquisition.

You can check the current pressure values in the Vacuum / Bakeout window of the Exactive Tune software window. To display the Exactive Tune software window, choose **Start > Programs > Thermo Exactive >**

**Exactive Tune**. The Instrument status LED in the toolbar (see image in margin) is illuminated green when the vacuum levels of the instrument are sufficient for operating your Exactive system.

Compare the current values of the pressures in the vacuum manifold with the values listed in Table 2-1. If the current values are higher than normal, you might have an air leak.

**Table 2-1.** Typical pressure readings

| Gauge type | Name in Exactive Tune | Typical values |
|---|---|---|
| Pirani | Forevacuum | about 2 mbar (depends on temperature of heated capillary) |
| Cold cathode | High Vacuum | about 1E-05 mbar (without HCD)<br>about 3E-05 mbar (with HCD) |
| Cold cathode | Ultra High Vacuum | < 8E-10 mbar |

If the pressure is high (above $1 \times 10^{-8}$ mbar in the analyzer region), and you have restarted the system within the last 30 to 60 minutes, wait an additional 30 minutes and recheck the pressure. If the pressure is decreasing with time, check the pressure periodically until it is within the typical pressure range of the MS detector.

**Note** Major air leaks are often identifiable merely by listening for a rush of air or a hissing sound somewhere on the instrument. A major leak might be caused, for example, by a loose or disconnected fitting, by an O-ring that is not properly seated, or by an open valve. ▲

If the pressure remains high, your system may have an air leak. If you suspect an air leak, shut down the system as described in "Shutting Down the System" on page 4-5. Make a visual inspection of the vacuum system and vacuum lines for leaks. Check each fitting and flange on the system for tightness, and tighten the fittings or flanges that are loose. Do not tighten fittings indiscriminately. Pay particular attention to fittings that have been changed recently or to fittings that have been subjected to heating and cooling.

Go to the next topic: "Checking the Disk Space on the Data System" .

## Checking the Disk Space on the Data System

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

❖ **To determine the amount of available disk space**

1. From the Home Page window (which is available by choosing **Start > Programs > Thermo Xcalibur > Xcalibur**), choose **Actions > Check Disk Space** to open the Disk Space dialog box. The Disk Space dialog box lists the following:
   - Current drive and directory (for example, C:\Xcalibur\system\programs)
   - Number of Mb 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 on **Directory**.
3. When you have completed this procedure, choose **OK** to close the dialog box.

If necessary, you can 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. After you have copied the files, you can delete them from the hard disk.

Go to the next topic: "Checking the Mass Accuracy of the Instrument" .

## Checking the Mass Accuracy of the Instrument

Thermo Fisher Scientific recommends checking the mass accuracy before you start working. Follow the procedure described in the *Exactive QuickStart Guide*.

If the instrument indicates that the tune parameters and calibration parameters are not in their optimum, Thermo Fisher Scientific recommends tuning and calibrating the Exactive. Follow the procedure described in the *Exactive QuickStart Guide*.

**Note** *Calibration parameters* are instrument parameters that affect the mass accuracy and resolution. *Tune parameters* are instrument parameters that affect the intensity of the ion signal. ▲

You need to optimize the tune parameters (or change the Tune Method) whenever you change the type of experiment. Refer to the *Exactive QuickStart Guide* for a procedure for optimizing the tune parameters for your ESI or APCI experiment.

# Things to Do After Operating the Exactive System

After operating the Exactive, perform the following steps in sequence:

1. Flushing Sample Transfer Line, Sample Tube, and API Probe
2. Flushing Ion Sweep Cone and Ion Transfer Capillary
3. Purging the Oil in the Forepump
4. Empty the Solvent Waste Container

**Note** Thermo Fisher Scientific recommends leaving Exactive in Standby overnight to provide the best mass accuracy next day. ▲

## Flushing Sample Transfer Line, Sample Tube, and API Probe

Flush sample transfer line, sample tube, and API probe at the end of each working day (or more often if you suspect they are contaminated) by flowing a 50:50 methanol:distilled water solution from the LC through the API source.

❖ **To flush sample transfer line, sample tube, and API probe**

1. Wait until data acquisition, if any, is complete.
2. Make sure that the door to the API chamber in closed and secured.
3. Choose **Start > Programs > Thermo Exactive > Exactive Tune** to open the Exactive Tune software window.

4. From the Exactive Tune software window, click the **On/Standby** button to toggle it from Standby to On. The voltages and gas flows to the API source are turned on.
5. Set up the ESI source as follows:
   a. In the Exactive Tune software window, display the ESI Source window.
   b. In the ESI Source window, enter 30 in the Sheath Gas Flow Rate text box.
   c. In the ESI Source window, enter 5 in the Aux Gas Flow Rate text box.
   d. In the ESI Source window, enter 0 in the Sweep Gas Flow Rate text box.
   e. In the ESI Source window, enter 0 in the Spray Voltage text box.
   f. Click **Apply**.

6. Set up and start a flow of 50:50 methanol:water solution from the LC to the API source:
   - Set the Flow Rate to a value that is typical for your experiments.
   - Set the solvent proportions to 50% methanol and water.
7. Let the solution flow through sample transfer line, sample tube, and API probe for 15 minutes. After 15 minutes, turn off the flow of liquid from the LC to the API source. Leave the API source (including APCI vaporizer, sheath gas, and auxiliary gas) on for additional 5 minutes. Stop the LC pump.

8. After 5 minutes, place the system in Standby condition as described in "Placing the System in Standby Condition" on page 4-4.

Go to the next topic: "Flushing Ion Sweep Cone and Ion Transfer Capillary" .

## Flushing Ion Sweep Cone and Ion Transfer Capillary

Clean the ion sweep cone (or spray cone) and the ion transfer capillary on a regular basis to prevent corrosion and to maintain optimum performance of your API source. A good practice is to flush the ion sweep cone and ion transfer capillary at the end of each operating day – after you have flushed the sample transfer line, sample tube, and API probe with a 50:50 methanol:water solution from the LC. (See "Flushing Sample Transfer Line, Sample Tube, and API Probe" on page 2-7.) If you are operating the system with nonvolatile buffers in your solvent system or high concentrations of sample, you might need to clean the ion sweep cone and ion transfer capillary more often. It is not necessary to vent the system to flush the ion sweep cone and ion transfer capillary.

❖ **To clean ion sweep cone and ion transfer capillary**

1. Turn off the flow of liquid from the LC (or other sample introduction device) to the API source.

2. From the Exactive Tune software window, click the **On/Standby** button to put the MS detector in Standby.
3. Open the door of the Ion Max ion source.

**Warning** **Danger of Burns.** At operating temperatures, the APCI vaporizer and ion transfer capillary can severely burn you! The APCI vaporizer typically operates at 350 to 500 °C and the ion transfer capillary typically operates at 100 to 300 °C. To cool the ion transfer capillary, set the capillary temperature to 25 °C or place the electronics service switch in the Service Mode position. Allow vaporizer and ion transfer capillary to cool to room temperature for approximately 20 minutes before you touch or remove either component. ▲

4. Fill a spray bottle with a 50:50 solution of HPLC-grade methanol:distilled water. Spray approximately 5 mL of the solution at the opening of the ion transfer capillary. Do not touch the ion transfer capillary with the tip of the spray bottle.

5. Use the spray bottle filled with the 50:50 solution of methanol:water to flush contaminants from the accessible surfaces of the ion source chamber and the spray cone or ion sweep cone (if it is installed).

6. Ensure that you have removed any salt or other contaminants that may have been deposited on the ion sweep cone or spray cone. If necessary, remove the Ion Max ion source and clean the ion sweep cone or spray cone as follows:

    a. Remove the Ion Max ion source from the front of the MS detector as described in "Removing the Ion Max Ion Source Housing" on page 3-12.

    b. Remove the ion sweep cone (if it is installed) as follows:

        i. Put on a pair of talc-free gloves.

        ii. Grasp the outer ridges of the ion sweep cone and pull the cone straight off of the API cone seal. You might need to loosen the set screws on the ion sweep cone in order to remove it.

**Note** This is a good point to remove and clean the ion transfer capillary. You remove the ion transfer capillary by unscrewing it counter clockwise with the custom removal tool. See "Removing and Cleaning the Ion Transfer Capillary" on page 3-17. ▲

    c. Clean the ion sweep cone as follows:

        i. Place the ion sweep cone and the ion capillary tube in a beaker of 50:50 methanol/water.

        ii. Sonicate it for 15 minutes.

        iii. Dry the ion sweep cone.

d. Clean the spray cone with a lint-free tissue soaked in methanol.

e. Reinstall the ion sweep cone as follows:

i. Note the location of the sweep gas supply port in the API cone seal. The gas inlet on the ion sweep cone is placed in this port. See Figure 2-1 and Figure 2-2.

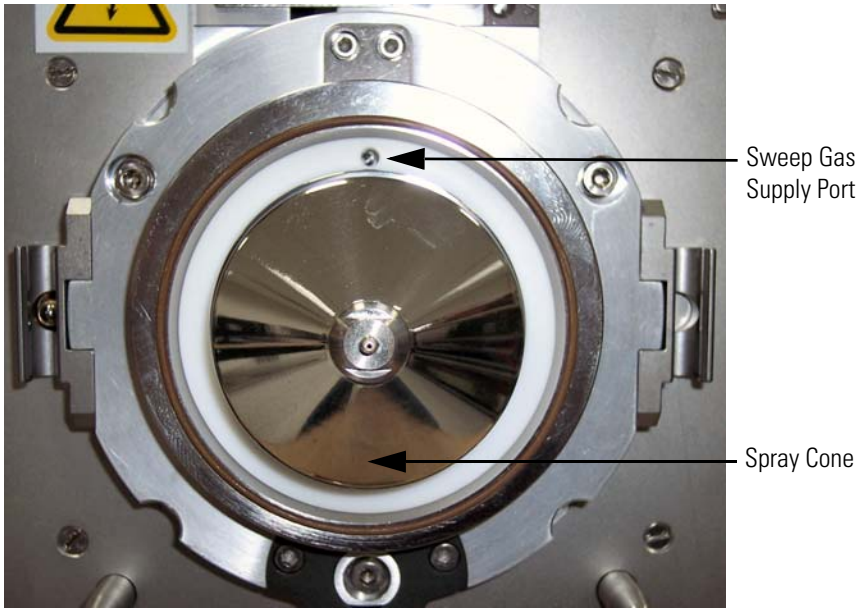



**Figure 2-1.** Sweep gas supply port in the API cone seal

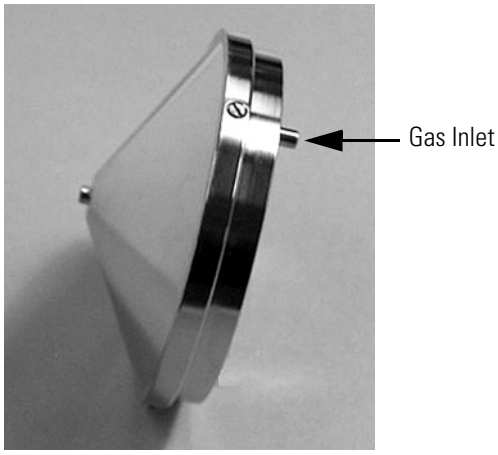



**Figure 2-2.** Ion sweep cone, showing the gas inlet

ii. Carefully align the gas inlet on the ion sweep cone with the sweep gas supply port in the API cone seal. Firmly press the ion sweep cone into position.

iii. If necessary to achieve a proper ion sweep cone installation, adjust the set screws around the perimeter of the ion sweep cone.

f. Reinstall the Ion Max ion source as described in "Installing the Ion Max Ion Source Housing" on page 3-13.

Go to the next topic: "Purging the Oil in the Forepump" .

## Purging the Oil in the Forepump

Purge (decontaminate) the oil in the forepump on a regular basis to remove water and other dissolved chemicals from the pump oil. Water and other chemicals in the forepump can cause corrosion and decrease the lifetime of the pump. A good time to purge the oil is at the end of the working day after you flush API probe, ion sweep cone, and ion transfer capillary.

❖ **To purge the oil in the forepump**

1. Turn off the flow of liquid from the LC (or other sample introduction device) to the API source.

2. From the Exactive Tune software window, click the **On/Standby** button to put the MS detector in Standby. (See image in margin.) Ensure that a septum seals the entrance to the ion transfer capillary.

3. Open the gas ballast valve on the forepump by turning it to position |. Refer to the manual that came with the pump for the location of the gas ballast valve.

4. Allow the pump to run for 30 minutes with the gas ballast valve open.

5. After 30 minutes, close the gas ballast valve by turning it to position —.

Go to the next topic: "Empty the Solvent Waste Container" .

## Empty the Solvent Waste Container

Check the solvent level in the solvent waste container on a daily basis. Empty the solvent waste container if necessary. Dispose of the solvent waste in accordance with national and local regulations.

# Chapter 3 User Maintenance

This chapter describes routine maintenance procedures that must be performed to ensure optimum performance of the Exactive.

**It is the user's responsibility to maintain the system properly by performing the system maintenance procedures on a regular basis.**

The following topics are described in this chapter:



**Note** For instructions on maintaining LCs or autosamplers, refer to the manual that comes with the LC or autosampler. ▲

# General Remarks on Maintenance

Preventive maintenance must commence with installation, and must continue during the warranty period to maintain the warranty. Thermo Fisher Scientific offers maintenance and service contracts. Contact your local Thermo Fisher Scientific office for more information. Routine and infrequent maintenance procedures are listed in Table 3-1.

**Table 3-1.** User maintenance procedures

| MS Detector Component | Procedure | Frequency | Procedure Location |
|---|---|---|---|
| Instrument | System bakeout | If necessary (e.g. after performing maintenance work on the vacuum system) | page 3-7 |
| | Check warning labels on instrument | Annually | page 3-4 |
| | Leak check gas lines | Annually | page 3-4 |
| Cooling fans | Check fan filters | Every 4 weeks | page 3-9 |
| | Clean fan filters | If necessary | |
| Forepump | Purge (decontaminate) oil | Daily | page 2-11, manufacturer's documentation |
| | Check oil level | Daily | page 3-5, manufacturer's documentation |
| | Check oil condition | Depends on process | |
| | Add oil | If oil level is low | |
| | Check gas ballast valve | Monthly | |
| | Clean dirt trap | Monthly | |
| | Change oil | Every 8000 h (~ one year) of operation | |
| | Replace exhaust filter | If oil mist appears at exhaust or annually | |
| | Check anti-suckback valve | Annually | |
| | Clean fan guard | Annually | |
| API source | Flush (clean) sample transfer line, sample tube, and API probe | Daily | page 2-7 |
| | Remove and clean ion transfer capillary | Weekly, or if ion transfer capillary bore is contaminated or obstructed | page 3-17 |
| | Remove and clean ion sweep cone | As needed* | page 3-17 |
| | Replace ion transfer capillary | If ion transfer capillary bore is corroded | page 3-17 |
| Ion optics | Clean skimmer and tube lens | As needed* | page 3-23 |

* The frequency of cleaning the components of the mass spectrometer depends on the types and amounts of samples and solvents that are introduced into the instrument.

To successfully carry out the procedures listed in this chapter, observe the following rules:

- Proceed methodically
- Always wear clean, talc-free, and lint-free gloves when handling the components of the API source, ion optics, mass analyzer, and ion detection system.
- Always place the components on a clean, lint-free surface.
- Always cover the opening in the top of the vacuum manifold with a large, lint-free tissue whenever you remove the top cover plate of the vacuum manifold.
- Never overtighten a screw or use excessive force.
- Dirty tools can contaminate your system. Keep the tools clean and use them exclusively for maintenance and service work at the Exactive.
- Never insert a test probe (for example, an oscilloscope probe) into the sockets of female cable connectors on PCBs.

**Note** To ensure that the instrument is free from all electric current, always disconnect the power cord before attempting any type of maintenance. ▲

## Returning Parts

In order to protect our employees, we ask you for some special precautions when returning parts for exchange or repair to the factory. Your signature on the Repair Covering Letter confirms that the returned parts have been de-contaminated and are free of hazardous materials. See ”Safety Advice for Possible Contamination” on page x for further information.

## Cleaning the Surface of the Instrument

Clean the outside of the instrument with a dry cloth. For removing stains or fingerprints on the surface of the instrument (panels, for example), slightly dampen the cloth (preferably made of microfiber) with distilled water.

**Caution** Prevent any liquids from entering the inside of the instrument. ▲

## Checking Warning Labels

In addition to the safety instructions that can be found throughout this guide, various warning labels on the instrument inform the user about possible hazards (for example, caused by hot surfaces or high voltage). To protect all personnel coming next to the instrument, check regularly whether all warning labels on the instrument are still present. Warning labels can be found at the power panel (See Figure 1-5 on page 1-9.), the power column (See Figure 1-6 on page 1-11.), and the ion source mount (See Figure 1-8 on page 1-16). Also check the warning labels on the forepump and the peripherals. See the manuals that came with these devices for information about possible hazards.

## Leak-Checking Gas Lines

Regularly leak check each gas line from the gas supply in the laboratory to the instrument.

❖ **To perform a leak check for a gas line**

1. After closing all valves in the instrument, monitor the manometer of the gas regulator for some minutes.
2. If the pressure falls significantly (for example, the nitrogen pressure falls by more than 10 psi / 690 mbar within two minutes), you should search for leaks in the gas line.
3. Search for leaks in the gas line (for example, by using a conventional thermal conductivity-based leak detector, such as is widely used to check leaks in gas chromatography equipment).
4. If you detect a leak (which is usually at a connection), verify the tightness of the connection. In case of doubt, replace it.
5. When you cannot find a leak in the gas line, we recommend calling a Thermo Fisher Scientific Service Engineer to check for gas leaks within the instrument.

# Maintaining the Vacuum System

This section describes maintenance procedures for the vacuum pumps and the vacuum manifold of the Exactive.

## Pumps Maintenance

This section only outlines the maintenance procedures for the forepump and the TMPs of the Exactive. The manuals of the pump manufacturers give detailed advice regarding safety, operation, maintenance, and installation. Please, note the warnings and precautions contained in these manuals!

### Maintenance of the Forepump

The forepump requires several maintenance procedures to be performed by the user. Table 3-2 outlines the maintenance schedule for the forepump as recommended by the pump manufacturer. To simplify the maintenance work, the pump manufacturer recommends combining several jobs. For maintenance instructions, see the manual that came with the forepump.

**Table 3-2.** Maintenance schedule of the forepump

| Maintenance job | Frequency |
|---|---|
| Check oil level | Daily |
| Check oil condition | Depends on process |
| Check gas ballast valve | Monthly |
| Clean dirt trap | Monthly |
| Change oil | Every 8000 h (~ one year) of operation |
| Replace exhaust filter | If oil mist appears at exhaust or annually |
| Check anti-suckback valve | Annually |
| Clean fan guard | Annually |

**Warning Risk of burn by touching.** Pump in function is hot and some surfaces could reach a temperature higher than 80 °C (176 °F). Also be careful when handling hot pump oil. Take note of warning labels on the pump. ▲

Forepump oil (P/N 1249170) and exhaust filters (P/N 1249350) are available from Thermo Fisher Scientific. If you want to order other spare parts, contact the pump manufacturer (Oerlikon Leybold Vacuum).

**Note** When disposing of used oil, please observe the relevant environmental regulations! For instructions about proper handling, refer to the Material Safety Data Sheet (MSDS) for the forepump oil. ▲

## Maintenance of the Turbomolecular Pumps

This topic outlines the maintenance work for the TMPs. These procedures require removing the housing of the instrument and partially disassembling it. Therefore, a Thermo Fisher Scientific Service Engineer should be called if servicing is required.

### Maintenance of the Source TMP

The pump manufacturer (Pfeiffer Vacuum) recommends changing the operating fluid reservoir every three years at the latest. Depending on the operating conditions, changing in two years intervals might be necessary. Changing of the TMP bearing is recommended every four years, at least.

### Maintenance of the Analyzer TMP

The pump manufacturer (Oerlikon Leybold Vacuum) recommends changing the standard bearing every 15000 operating hours at the latest, and exchanging the rotor assembly every 45000 operating hours.

# Baking Out the System

This section provides information and help concerning the system bakeout of the Exactive. The bakeout procedure removes unwanted gases or molecules (collected or remaining) from the high vacuum region of the instrument. Ions can collide with those gases or molecules resulting in lower overall sensitivity. Therefore, we recommend to bake out the instrument if the high vacuum decreases noticeable during routine operation.

**Note** Bakeout is mandatory after maintenance or service work is performed in the analyzer region where the system is vented. You should bake out an instrument that has been vented for at least twelve hours before you can start using it again. ▲

In case the system has been vented during a power failure, it is necessary to bake out the system to obtain the operating vacuum. See "Behavior of the System in Case of a Main Failure" on page 4-2.

❖ **To perform a system bakeout**

1. Place the system in Standby condition as described in "Placing the System in Standby Condition" on page 4-4. (See image in margin.)
2. In the Tasks panel of the Exactive Tune software window, click on 

to display the Vacuum / Bakeout window. See Figure 3-1.

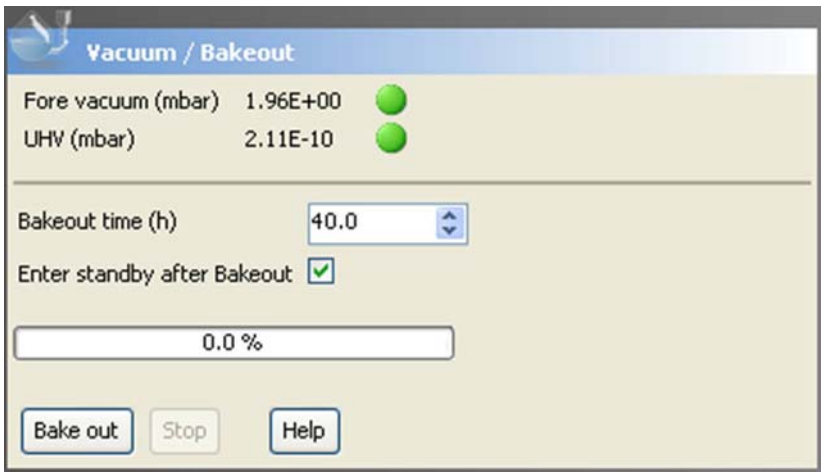



**Figure 3-1.** Vacuum / Bakeout window

3. Enter the desired baking duration (in hours) into the spin box. The range is 4 to 100 hours.

4. Click **Bake out**. A dialog box shows the duration of the baking procedure. See Figure 3-2. Click on **Yes** to confirm the message.

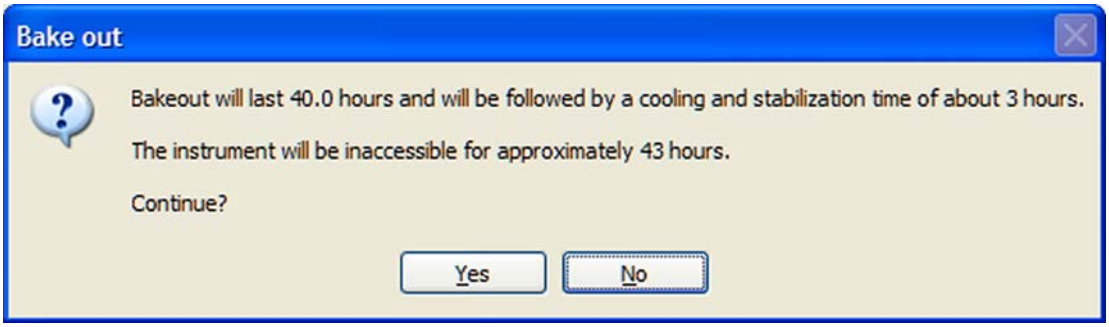



**Figure 3-2.** Bakeout dialog box

5. The message box disappears and the baking procedure starts. The instrument indicates the active bakeout procedure by a flashing Vacuum LED. (See Figure 1-3 on page 1-7.) Additionally, the Exactive Tune software displays a corresponding message box. See Figure 3-3.

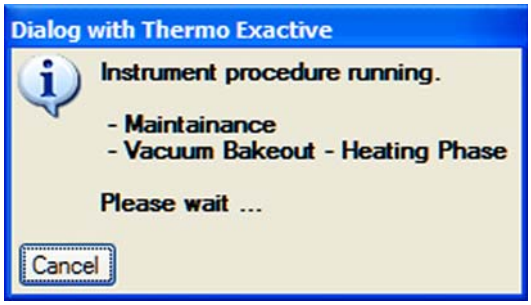



**Figure 3-3.** Message box: Vacuum Bakeout active

6. The baking of the instrument stops after the preset duration. The Vacuum LED keeps flashing until the cooling and stabilization time (of about 3 hours) is finished.

   Click on **Stop** in the Vacuum / Bakeout window to abort the baking routine before the preset time.

**Warning** **Burn Hazard.** If you abort a system bakeout, parts of the instrument can be hot. Allow the instrument to cool for at least three hours before you start operating it again. ▲

# Maintenance of the Fan Filters

Each of the two ventilation slots at the rear side of the Exactive is equipped with a fan filter. See Figure 3-4.

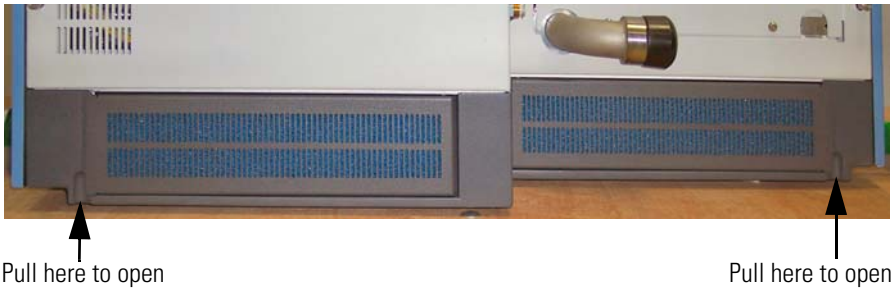



**Figure 3-4.** Ventilation slots at the rear side of the instrument

**Caution** Avoid blocking the ventilation slots at the rear of the instrument. Items may fall behind the instrument, inhibit airflow, and cause the system to overheat. ▲

## Checking the Fan Filters

Check the fan filters every four weeks.

❖ **To check the fan filters**

1. Each fan filter bracket is mounted on hinges. Insert a finger into the recess in the instrument frame and pull at the fan filter bracket to open it. See Figure 3-4 and Figure 3-5.
2. Remove each fan filter from the rear of the Exactive by pulling it out of the filter bracket. See Figure 3-5.

**Figure 3-5.** Removing a fan filter from the filter bracket

If the fan filters are covered with dust, proceed as described in "Cleaning the Fan Filters" below. Replacements for the fan filters are available from Thermo Fisher Scientific (P/N 1234880).

## Cleaning the Fan Filters

❖ **To clean the fan filters**

1. Remove the fan filters as described in "Checking the Fan Filters" above.
2. Wash the fan filters in a solution of soap and water.
3. Rinse the fan filters with tap water.
4. Squeeze the water from the fan filters and allow them to air dry.
5. Reinstall the fan filters in the fan filter brackets.

# API Source Maintenance

This section describes routine maintenance procedures that must be performed to ensure optimum performance of the API source. Most of the procedures involve cleaning. For example, procedures are provided for cleaning the ion transfer capillary, the ion sweep cone, the skimmer, and the tube lens. Procedures are also presented for replacing the ion transfer capillary and the capillary heater assembly.

See also the *Ion Max and Ion Max-S API Source Hardware Manual* for information about maintenance procedures for the API source.

## Frequency of Cleaning

The frequency of cleaning the components of the API source depends on the types and amounts of samples and solvents that are introduced into the instrument. In general, for a given sample and ionization technique, the closer a mass spectrometer component is to the source of the ions, the more rapidly it becomes dirty.

- Sample transfer line, sample tube, and API probe should be cleaned at the end of each operating day to remove any residual salts from buffered mobile phases or other contamination that might have accumulated during normal operation. See "Flushing Sample Transfer Line, Sample Tube, and API Probe" on page 2-7.
- Ion transfer capillary and ion sweep cone of the API source need to be removed and cleaned periodically. See "Ion Sweep Cone and Ion Transfer Capillary Maintenance" on page 3-16.
- Tube lens and skimmer of the ion optics become dirty at a slower rate than the API probe, ion sweep cone, and ion transfer capillary. See "Cleaning Tube Lens and Skimmer" on page 3-23.

When the performance of your system decreases significantly due to contamination, clean the components of the mass spectrometer in the following order:

1. Clean API probe, ion sweep cone, and ion transfer capillary.
2. Clean tube lens and skimmer.

**Note** Generally, you should not open the vacuum manifold. Cleaning of the components therein is rarely (if ever) required. If you believe that cleaning of these components is required, you should contact Thermo Fisher Scientific and schedule a visit by a Thermo Fisher Scientific Field Service Engineer. ▲

# Removing and Reinstalling the Ion Max Ion Source Housing

You need to remove the Ion Max ion source housing before performing maintenance on the ion optics and the ion source interface.

This section contains the following sections:



## Removing the Ion Max Ion Source Housing

You need to remove the Ion Max ion source housing to access the ion sweep cone.

**Note** If an ion source probe is still installed in the ion source housing, the external liquid lines should first be disconnected before removing the ion source housing. ▲

### ❖ To remove the ion source housing

1. Remove the drain tube from the ion source housing drain. See Figure 3-6.

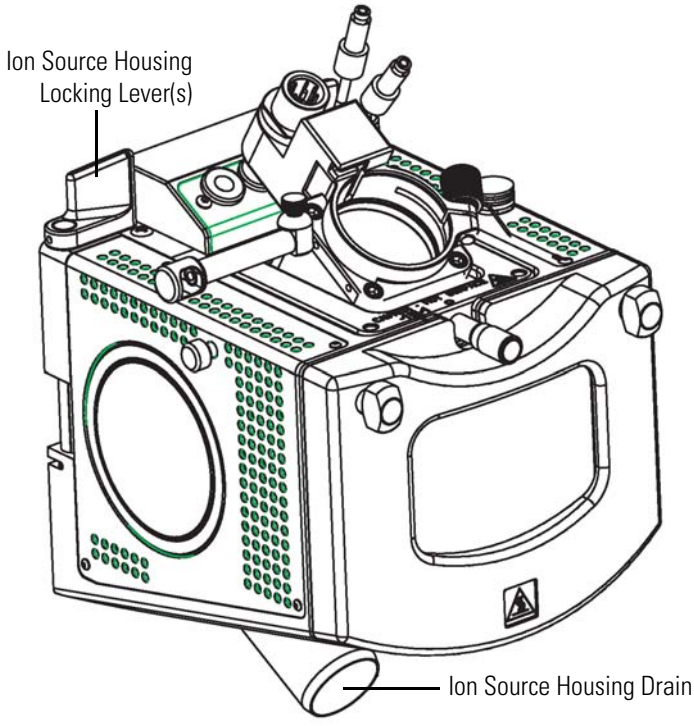



**Figure 3-6.** Ion Max source housing, showing detail of components

2. Rotate the ion source housing locking levers 90 degrees to release the ion source housing from the ion source mount assembly.

3. Remove the ion source housing by pulling it straight off the ion source mount assembly, and place the housing in a safe location for temporary storage.

## Installing the Ion Max Ion Source Housing

❖ **To reinstall the Ion Max ion source housing**

1. Carefully align the two guide pin holes on the rear of the ion source housing with the ion source housing guide pins on the mass spectrometer. Carefully press the ion source housing onto the ion source mount. See Figure 3-7 and Figure 3-8 on page 3-14.

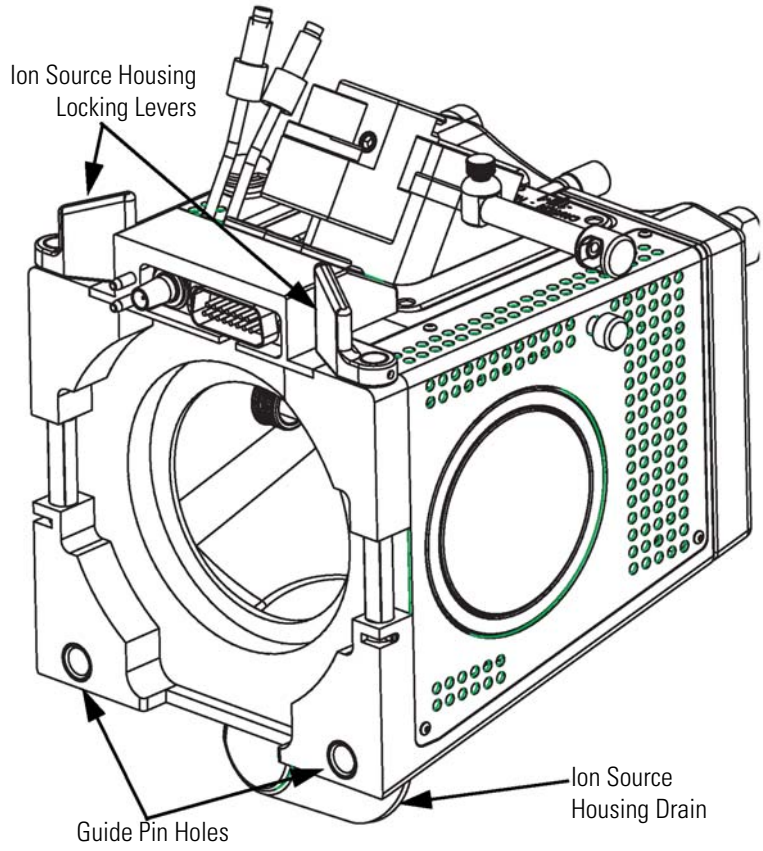



**Figure 3-7.** Rear view of the Ion Max ion source housing

2. Rotate the ion source housing locking levers 90 degrees to lock the ion source housing onto the ion source mount assembly.

**Caution** Prevent solvent waste from backing up into the ion source and mass spectrometer. Always ensure that liquid in the drain tube is able to drain to a waste container. ▲

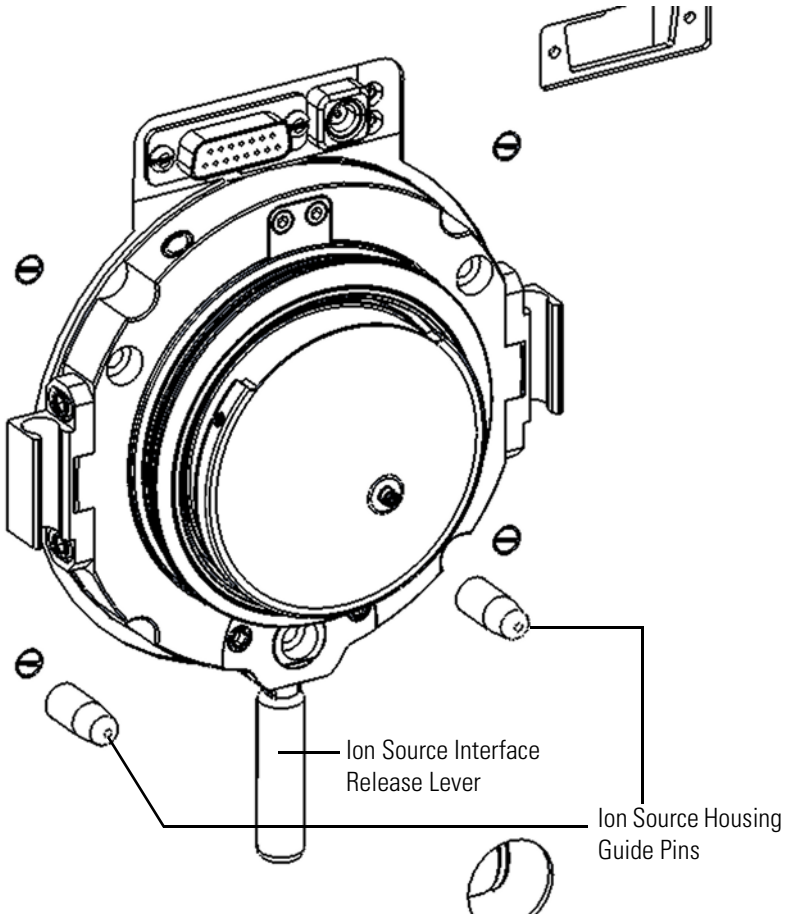



**Figure 3-8.** Ion source mount showing ion source housing guide pins and ion source interface release lever

3. Reinstall the ion source housing drain tube. See "Connecting the Source Housing Drain to the Waste Container" below for advice.

The Ion Max ion source is now properly installed on the mass spectrometer.

## Connecting the Source Housing Drain to the Waste Container

When you reinstall the Ion Max API source, reconnect the drain at the bottom of the source housing to the solvent waste container (see Figure 3-9).

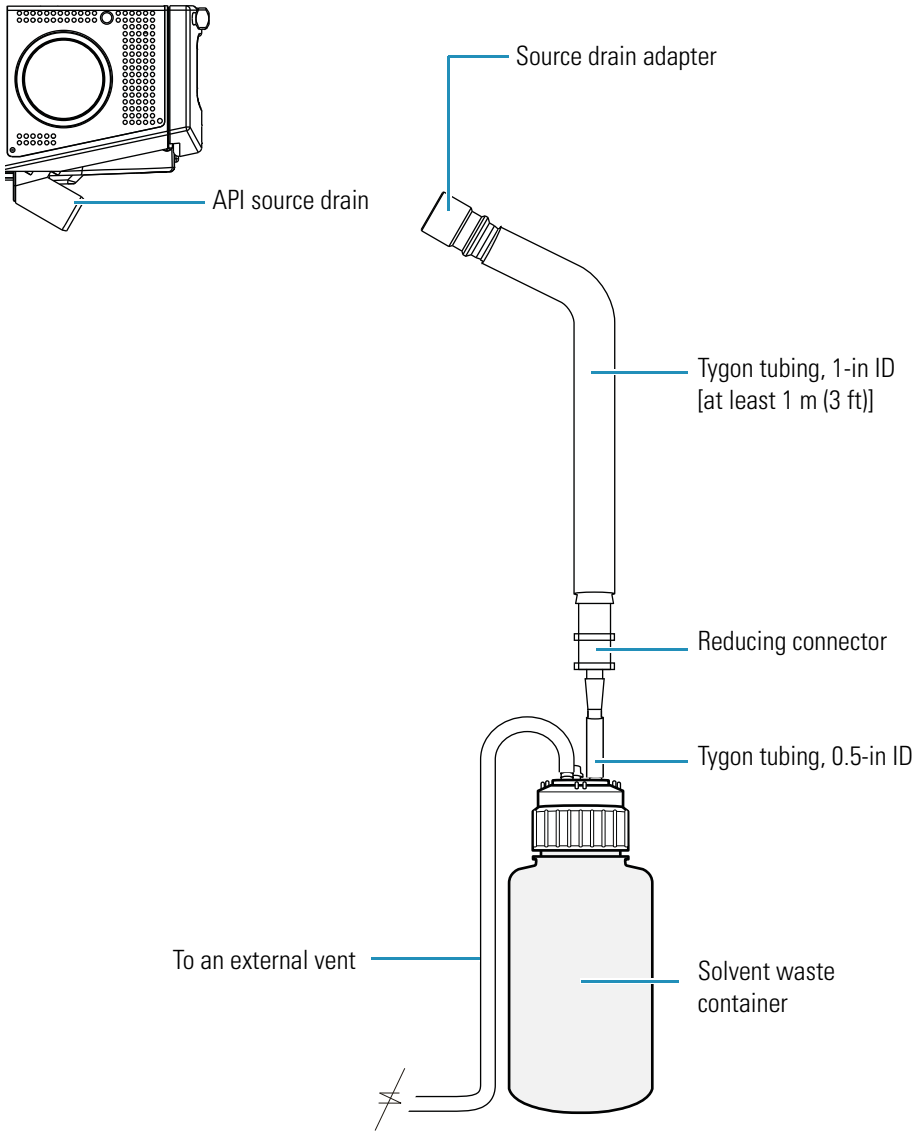



**Figure 3-9.** View of the Ion Max API source and the drainage system

When you reconnect the drain tubing to the drain at the bottom of the Ion Max API source, ensure that you connect the Teflon source drain adapter, which can withstand the high temperatures produced by the H-ESI source or the APCI source, to the source drain. In addition, ensure that the tubing assembly includes at least 1 m (3 ft) of 1 inch ID Tygon tubing.

**Caution** Do not connect Tygon tubing directly to the source drain. At high temperatures, Tygon releases volatile contaminates. ▲

**Caution** Do not connect silicone tubing to the API source outlet drain. If silicone tubing is connected to the outlet drain, you might observe background ions at *m/z* 536, 610, and 684. Use the silicone tubing that is provided with the filling/venting cap to connect the waste container to a fume exhaust system. ▲

**Caution** Prevent solvent waste from backing up into the API source and mass spectrometer. Always ensure that the drain tubing is above the level of liquid in the waste container. ▲

**Caution** Your laboratory must be equipped with at least two fume exhaust systems:
The analyzer optics can become contaminated if the API source drain tube and the exhaust tubing from the forepump are connected to the same fume exhaust system. Route the exhaust tubing from the forepump to a dedicated fume exhaust system.
Do not vent the drain tube (or any vent tubing connected to the waste container) to the same fume exhaust system that you have connected the forepump to. Vent the waste container to a dedicated fume exhaust system. ▲

Table 3-3 lists the components of the solvent waste system. They are contained in the Exactive Installation Kit. During the initial installation of the mass spectrometer, a Thermo Fisher Scientific Field Service Engineer installs the solvent waste system.

**Table 3-3.** Solvent waste system parts

| Part description | Part number |
|---|---|
| DRAIN, ION MAX 2 ,LTQ | 97055-20488 |
| Source drain adapter, Teflon | 70111-20971 |
| Reducing connector, single barbed fitting, 1-in × 0.5-in | 00101-03-00001 |
| Tube, 1-in ID × 1.3/8-in OD, Tygon 2001 | 00301-01-00020 |
| Tubing, Tygon, 0.5-in ID × 0.75-in OD | 00301-22920 |
| Cap, filling/venting | 00301-57022 |
| Heavy-duty, 4 L Nalgene® bottle | 00301-57020 |

## Ion Sweep Cone and Ion Transfer Capillary Maintenance

You need to remove and clean the ion sweep cone and the ion transfer capillary on a regular basis to prevent corrosion and to maintain optimum performance of your API source.

## Removing and Cleaning the Ion Transfer Capillary

The bore of the ion transfer capillary can become blocked by buffer salts or high concentrations of sample. The ion transfer capillary can be easily removed for cleaning. You do not have to vent the system to remove the ion transfer capillary.

If the pressure in the capillary-skimmer region (as measured by the Pirani gauge) drops considerably below 1 mbar, you should suspect a blocked ion transfer capillary. You can check the Pirani gauge pressure in the Exactive Tune software (Vacuum / Bakeout window > Fore vacuum).

Table 3-3 lists the parts that are required when performing maintenance of the ion transfer capillary. They are contained in the Exactive Installation Kit.

**Table 3-4.** Parts for maintenance of the ion transfer capillary

| Part description | Part number |
|---|---|
| CAPILLARY-580 MICRON COAXIAL | 97055-20199 |
| TOOL, CAPILLARY REMOVAL, QUANTUM | 70111-20258 |
| SEAL, RING, GRAPHITE VESPEL | 97055-20442 |

❖ **To remove and clean the ion transfer capillary**

1. Turn off the flow of liquid from the LC (or other sample introduction device) to the API source.
2. Place the electronics service switch (located on the left side of the MS detector) in the Service Mode position to remove power to all components of the MS detector with exception of the vacuum system. Wait for at least 20 minutes to allow hot components to cool down.

**Warning Danger of electric shock.** Make sure that the Exactive electronics service switch is in the Service Mode position before proceeding. ▲

**Warning Danger of burns.** The ion transfer capillary typically operates at 250 to 400 °C. Allow the ion transfer capillary and ion sweep cone to cool before you remove them. ▲

3. Remove the Ion Max ion source housing from the front of the mass spectrometer as described in "Removing the Ion Max Ion Source Housing" on page 3-12.
4. Remove the ion sweep cone by pulling it off the ion source interface.

5. Remove the ion transfer capillary by turning it counterclockwise with the custom removal tool (P/N 70111-20258, in the Exactive Installation Kit) until you can pull it free from the ion source interface. See Figure 3-10.

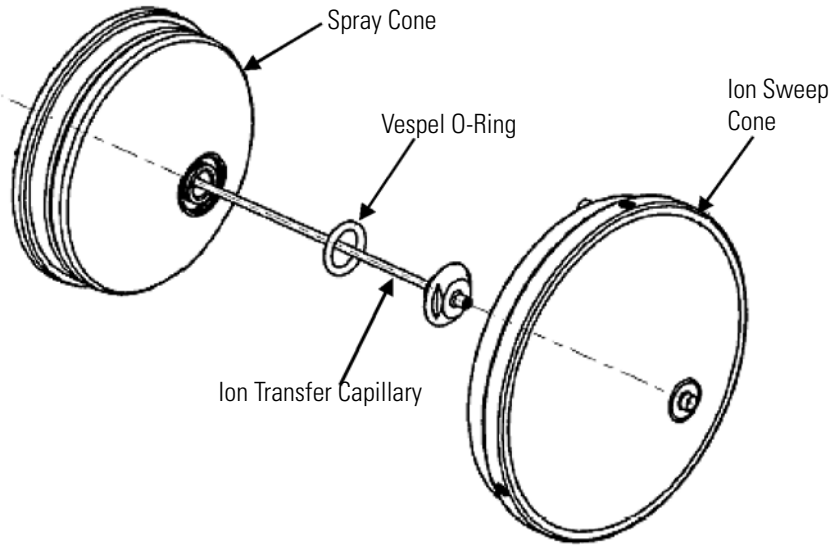



**Figure 3-10.** Spray cone, O-ring, ion transfer capillary, and ion sweep cone of the ion source interface

6. Soak the ion transfer capillary in a dilute solution of nitric acid to remove contaminants.
7. Sonicate the ion transfer capillary in distilled water.
8. Clean the ion sweep cone by wiping the inside and outside with methanol and a lint-free tissue.
9. Remove, clean with methanol, and inspect the Vespel® O-ring that seats in the spray cone under the entrance end of the ion transfer capillary. Replace it if necessary (P/N 97055-20442).
10. Reinsert the O-ring in the spray cone.

**Caution** Use caution when reinstalling the ion transfer capillary:

- Ensure that everything is properly aligned to prevent stripping the threads on the ion transfer capillary.
- Be careful not to bend the ion transfer capillary. Rotate the capillary as you insert it. ▲

11. Insert the ion transfer capillary into the heater block. Rotate the capillary as you insert it. After it is inserted, turn the capillary clockwise until it is finger tight.

12. Align the gas inlet on the ion sweep cone with the sweep gas supply port on the ion source mount. Firmly press the ion sweep cone into ion source mount. See Figure 3-11 and Figure 3-12.

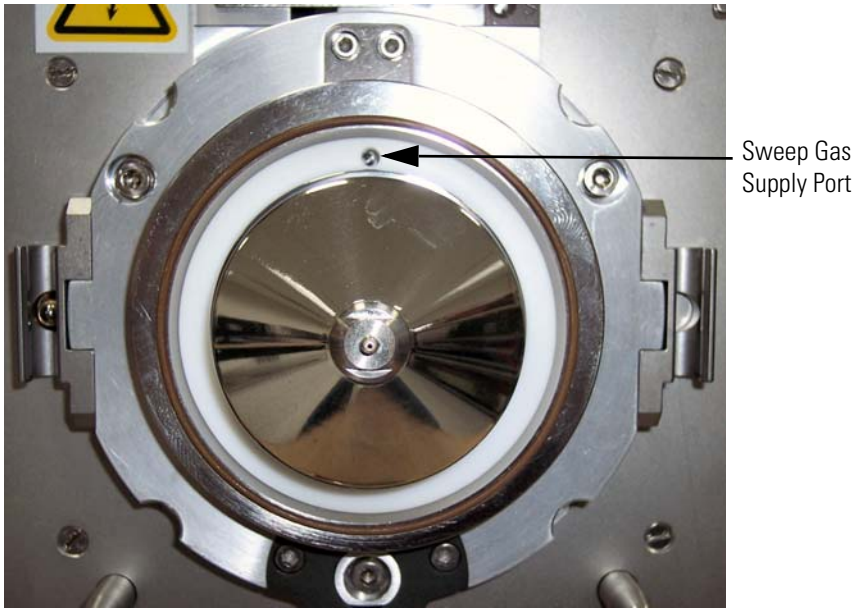



**Figure 3-11.** Ion source interface, showing the sweep gas supply port in the API cone seal

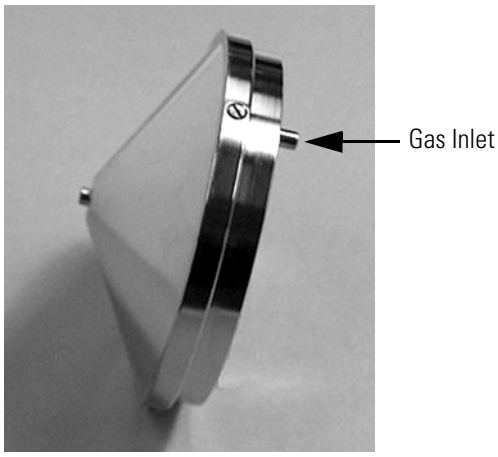



**Figure 3-12.** Ion sweep cone, showing the gas inlet

13. Reinstall the Ion Max source housing on the mass spectrometer as described in "Installing the Ion Max Ion Source Housing" on page 3-13.

**Note** If you have unblocked the ion transfer capillary, the Pirani gauge pressure should increase to a normal value (approximately 2 mbar). If you cannot clear the ion transfer capillary by this method, replace the ion transfer capillary (P/N 97055-20199). ▲

14. Place the electronics service switch in the Operating Mode position to supply power to all components of the MS detector.

## Maintaining the API Probes

The API probes of the Ion Max ion source require a minimum of maintenance. The sample tube needs to be replaced when it becomes obstructed with salt precipitates or is broken. The API probe may need to be disassembled so you can clean it or replace a part.

Refer to the *Ion Max and Ion Max-S API Source Hardware Manual* for procedures for maintaining the API probes.

**Note** You should flush the API probe at the end of each working day by flowing a 50:50 HPCL-grade methanol:distilled water solution from the LC through the APCI probe. See "Flushing Sample Transfer Line, Sample Tube, and API Probe" on page 2-7. ▲

## Maintaining the Ion Source Interface

The ion source interface assembly includes ion sweep cone, ion transfer capillary, capillary heater assembly, tube lens, and skimmer. The ion transfer capillary has a finite lifetime. You need to replace it if its bore becomes corroded.

It is a good practice to flush the ion sweep cone and the bore of the ion transfer capillary at the end of each working day with a 50:50 methanol:water solution. See "Flushing Ion Sweep Cone and Ion Transfer Capillary" on page 2-8.

For cleaning or replacement, you can remove the ion sweep cone and ion transfer capillary without venting the system. See "Ion Sweep Cone and Ion Transfer Capillary Maintenance" on page 3-16.

### Sequence of Steps

To replace ion source interface assembly components other than the ion sweep cone and ion transfer capillary, proceed in the following order:

1. Shut down and vent the system
2. Remove the Ion Max source housing
3. Remove the ion source interface assembly
4. Remove tube lens and skimmer
5. Clean tube lens and skimmer

6. Replace the capillary heater (optional)
7. Reinstall tube lens and skimmer
8. Reinstall the ion source interface assembly
9. Reinstall the Ion Max source housing
10. Start up the system

The above steps are described in the following topics.

### Shutting Down and Venting the System

Shut down and vent the system as described in "Shutting Down the System" on page 4-5.

Go to the next topic: "Removing the Ion Max Ion Source Housing".

### Removing the Ion Max Ion Source Housing

You need to remove the Ion Max ion source housing to access the ion source interface assembly. Remove the Ion Max ion source housing as described in "Removing the Ion Max Ion Source Housing" on page 3-12.

Go to the next topic: "Removing the Ion Source Interface".

### Removing the Ion Source Interface

You need to remove the ion source interface to access skimmer, tube lens, and capillary heater assembly.

**Warning Danger of burns.** Wait for the ion source interface to cool to ambient temperature before you remove it. ▲

❖ **To remove the ion source interface**

1. Put on clean, talc-free gloves.
2. Using a right-to-left motion, move the ion source interface release lever several times to loosen the ion source interface from the ion optics cage assembly. See Figure 3-13 on page 3-22.
3. Grab the exposed outer grooves of the ion source interface and firmly pull the assembly straight out.

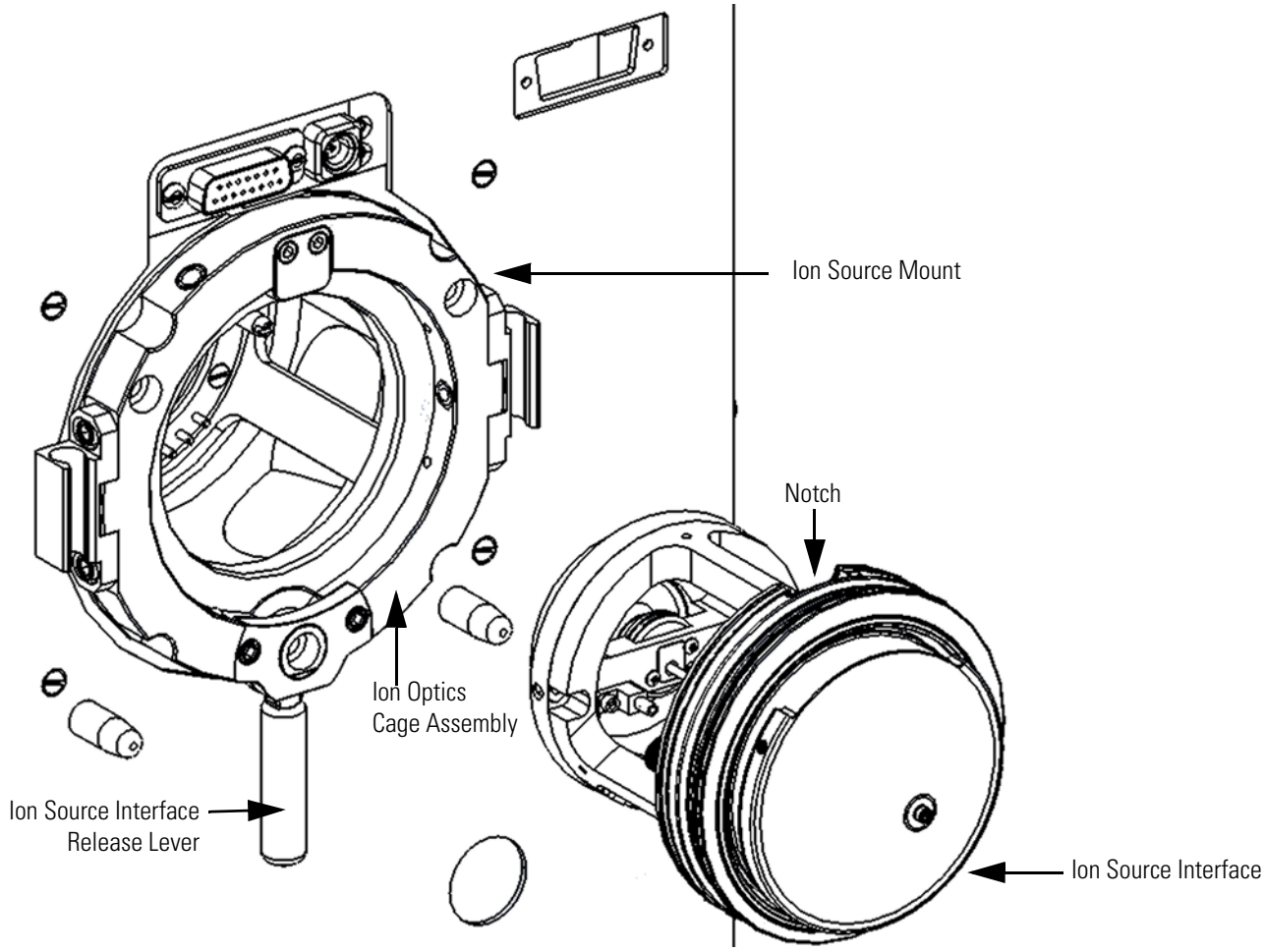



**Figure 3-13.** Removing the ion source interface

4. Place the assembly on a clean, lint-free surface.

Go to the next topic: "Removing Tube Lens and Skimmer".

## Removing Tube Lens and Skimmer

Tube lens and skimmer must be removed from the ion source interface assembly before they can be cleaned.

**Note** Wear clean, talc-free gloves when you handle tube lens and skimmer. ▲

**Caution** Take care not to scratch or nick the skimmer cone. ▲

❖ **To remove tube lens and skimmer from the ion source interface assembly**

1. Reaching behind the skimmer with your fingers, gently press the skimmer out of the contact ring support. If necessary, loosen the set screws. See Figure 3-14 on page 3-25.
2. Place the skimmer on a clean, lint-free surface.
3. Grab the exposed lip of the tube lens and pull the tube lens out of the contact ring support.
4. Place the tube lens on a clean, lint-free surface.

Go to the next topic: "Cleaning Tube Lens and Skimmer".

## Cleaning Tube Lens and Skimmer

An accumulation of chemicals on the surfaces of tube lens and skimmer forms an insulating layer that can modify the electrical fields that control ion transmission. Tube lens and skimmer require cleaning less often than the ion sweep cone and the ion transfer capillary.

**Caution** Do not clean tube lens or skimmer with detergents, acidic or caustic substances, or abrasives. ▲

**Caution** The sharp edge of the skimmer cone is fragile and any deformation may result in a loss of efficient ion transmission. Do not drop the skimmer while cleaning it. Sonicate the skimmer alone with the cone side up. ▲

Sonicate tube lens and skimmer alone in organic or aqueous solution.

**Note** For most cleaning applications, HPLC grade methanol is the solvent of choice. However, use of buffers or salt solutions might require that you use an aqueous solution. If you need to use a solvent other than methanol, after cleaning the component, flush the component with distilled water and then flush it with methanol as a final wash. Tube lens and skimmer can be air dried or blown dry with nitrogen gas. In all cases, ensure that all solvent has evaporated from the component(s) before reassembly. ▲

If you want to replace the capillary heater assembly, go to the next topic: "Replacing the Capillary Heater Assembly". Otherwise, go to the topic "Reinstalling Tube Lens and Skimmer" on page 3-26.

## Replacing the Capillary Heater Assembly

**Note** Wear clean, talc-free gloves when you handle the ion source interface components. ▲

The capillary heater assembly must be replaced as a unit.

❖ **To remove the capillary heater assembly**

1. Remove the ion transfer capillary by turning it counterclockwise until you can pull it free from the API source cone. See Figure 3-14 on page 3-25.

**Note** Make careful note of the position of all of the wires routed to the contact ring. ▲

2. Remove the screw that fastens the capillary voltage lead to the nose cone insulator bushing on the capillary heater mount cage.
3. Remove the socket clamp that fastens the tube lens lead to the tube lens contact.
4. Remove the two knurled nuts that hold the capillary heater assembly to the capillary heater mount cage.
5. Remove the screws that fasten the contact ring support to the capillary heater mount cage.
6. Carefully remove the capillary heater assembly.
7. Ensure that all the wires to the contact ring stay in their proper orientation while you remove the contact ring from the contact ring support.
8. To install a new capillary heater assembly (P/N 70111-60175), follow steps 1 through 7 in reverse order.

Go to the next topic: "Reinstalling Tube Lens and Skimmer".

**Note** When reinstalling the capillary heater assembly, ensure that the assembly is properly aligned and inserted completely so that the heater block is flush against the graphite seal that is pressed into the API source cone. ▲

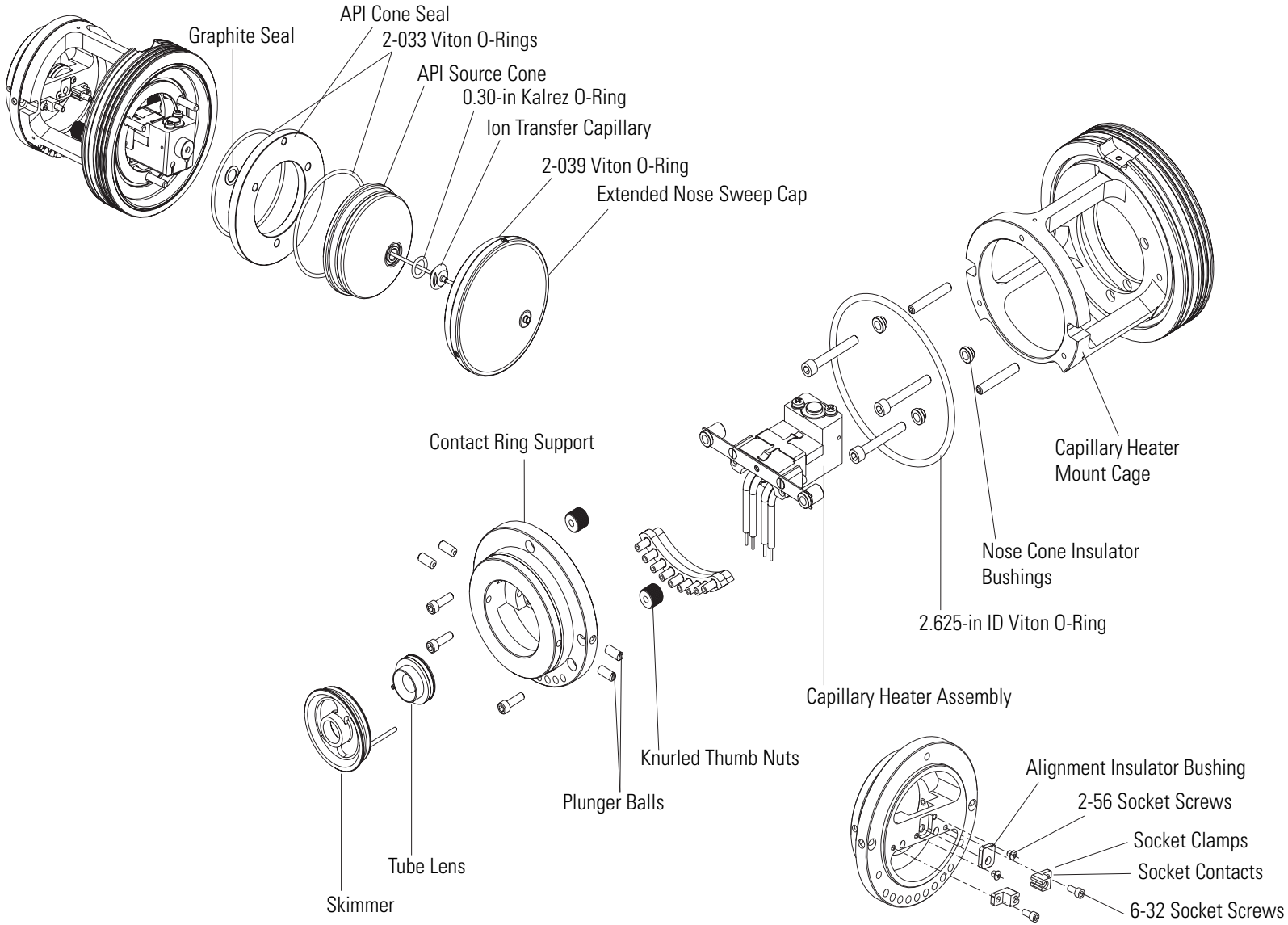



**Figure 3-14.** Exploded view of ion source interface assembly

## Reinstalling Tube Lens and Skimmer

❖ **To reinstall tube lens and skimmer**

1. Reinstall the tube lens into the ion source interface assembly:
   a. Orient the tube lens such that the lead pin points toward the socket on the tube lens connection wire in the contact ring support. See Figure 3-14 on page 3-25.
   b. Insert the lead pin into the socket and firmly press the tube lens into the contact ring support.

**Caution** Ensure that you do not scratch or nick the skimmer cone. ▲

2. Reinstall the skimmer into the ion source interface assembly:
   a. Orient the skimmer such that the lead pin points toward the socket on the tube lens connection wire in the contact ring support.
   b. Insert the lead pin into the socket and firmly press the skimmer into the contact ring support.

Go to the next topic: "Reinstalling the Ion Source Interface Assembly".

## Reinstalling the Ion Source Interface Assembly

❖ **To reinstall the ion source interface assembly**

1. Orient the ion source interface assembly such that the sweep gas inlet notch on the ion source interface is aligned with the sweep gas supply block on the ion source mount.
2. Carefully slide the ion source interface assembly into the ion optics cage assembly.

Go to the next topic: "Reinstalling the Ion Max Ion Source Housing".

## Reinstalling the Ion Max Ion Source Housing

Reinstall the Ion Max ion source housing as described in "Installing the Ion Max Ion Source Housing" on page 3-13.

You may also need to reinstall an ion source probe and LC liquid lines, as appropriate.

Go to the next topic: "Starting Up the System" on page 3-27.

## Starting Up the System

Start up the system as described in the "Starting Up the System after a Shutdown" on page 4-6.

# Chapter 4 System Shutdown, Startup, and Reset

Many maintenance procedures for the Exactive system require that the MS detector be shut down. In addition, the Exactive system can be placed in Standby condition if the system is not to be used for 12 hours or more.

The following topics are discussed in this chapter:



See also Chapter 2: "Daily Operation" for information about checks and cleaning procedures of the Exactive system to be performed every day. Maintenance procedures for the Exactive are described in Chapter 3: "User Maintenance".

# Shutting Down the System in an Emergency

**Note** To allow shutting off the MS detector in an emergency, free access to the power panel on the left side of the instrument must be possible at any time. ▲

If you need to turn off the MS detector in an emergency, place the main power circuit breaker switch, located on the power panel on the left side panel of the MS detector (see Figure 4-1), in the Off (O) position. This turns off all power to the MS detector, including the vacuum pumps.

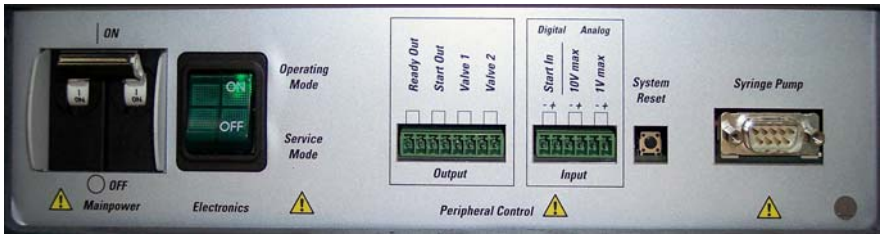



**Figure 4-1.** Power panel, showing main power circuit breaker switch and reset button

The instrument is automatically vented by the vent valve. The vent valve vents the system 30 seconds after power is switched off.

Although removing power abruptly will not harm any component within the system, this is not the recommended shutdown procedure to follow. See "Shutting Down the System" on page 4-5 for the recommended procedure.

**Note** To ensure that the instrument is free from all electric current, disconnect the power cord. ▲

**Note** To separately turn off the computer in an emergency, use the On/Off switch on the computer. ▲

## Behavior of the System in Case of a Main Failure

A main power failure has the same consequence as switching off via the main power circuit breaker switch. If the power is available again, the system is started up automatically i.e. the pumps are switched on and the vacuum is created. If the system has been vented during the mains failure, it is necessary to bake out the system to obtain the operating vacuum. See "Baking Out the System" on page 3-7.

It is not possible to check whether the system was vented. The log file of the data system indicates a reboot of the system. In case of frequent but short power failures we recommend installing an uninterruptible power supply (UPS). If main power failures occur frequently while the system is not attended (e.g. in the night), we recommend installing a power fail detector.

# Placing the System in Standby Condition

The Exactive system should not be shut down completely if you are not going to use it for a short period of time, such as overnight or over the weekend. When you are not going to operate the system for 12 hours or more, you can leave the system in Standby condition.

**Note** Thermo Fisher Scientific recommends leaving Exactive in Standby overnight to provide the best mass accuracy next day. ▲

❖ **To place the Exactive system in the Standby condition**

1. Wait until data acquisition, if any, is complete.
2. Turn off the flow of sample solution from the LC (or other sample introduction device).

**Note** For instructions on how to operate the LC from the front panel, refer to the manual that came with the LC. ▲

3. In the Exactive Tune software window, click the **On/Standby** button to put the instrument in Standby condition. The System LED on the front panel of the Exactive is illuminated yellow when the system is in Standby condition.
4. Leave the LC power On.
5. Leave the autosampler power On.
6. Leave the data system power On.
7. Leave the Exactive main power circuit breaker switch in the On position.

# Shutting Down the System

The Exactive system does not need to be shut down completely if you are not going to use it for a short period of time, such as overnight or over a weekend. See "Placing the System in Standby Condition" on page 4-4. This section describes how to shut down the system for a maintenance or service procedure.

❖ **To shut down the Exactive system**

1. Wait until data acquisition, if any, is complete.
2. Turn off the flow of sample solution from the LC (or other sample introduction device).

**Note** For instructions on how to operate the LC from the front panel, refer to the manual that came with the LC. ▲

3. In the Exactive Tune software window, click the **On/Standby** button to put the instrument in Off condition. (See image in margin.) All high voltages are shut off, as are the sheath and auxiliary gas.
4. Place the electronics service switch, located on the power panel (See Figure 1-5 on page 1-9.), in the Service Mode position.
5. Put the main power circuit breaker switch of the Exactive in the Off position.

**Note** To ensure that the instrument is free from all electric current, always disconnect the power cord before attempting any type of maintenance. ▲

**Warning Burn Hazard.** Allow heated components to cool before you service them (the ion transfer capillary at about 300 °C, for example). ▲

**Note** If you are planning to perform routine or preventive system maintenance on the Exactive only, you do not need to turn off the LC, autosampler, or data system. In this case, the shutdown procedure is completed. However, if you do not plan to operate your system for an extended period of time, you might want to turn off the LC, autosampler, and data system. ▲

# Starting Up the System after a Shutdown

To start up the Exactive after it has been shut down (and vented), you need to do the following:

1. Start up the instrument
2. Set up conditions for operation

## Starting Up the Instrument

**Note** The data system must be running before you start up the instrument. The instrument will not operate until software is received from the data system. ▲

❖ **To start up the Exactive**

1. Start up the (optional) LC and autosampler as is described in the manual that came with the LC and autosampler.
2. Start up the data system.
3. Turn on the nitrogen flow at the tank, if it is off.
4. Make sure that the main power circuit breaker switch is in the Off (O) position and that the electronics service switch is in the Service Mode position.
5. If the forepump is equipped with an On/Off switch (See Figure 1-17 on page 1-30.), make sure that the switch is set to the On (I) position.
6. Place the main power circuit breaker switch in the On (|) position. When you place the main power circuit breaker switch in the On (|) position, the forepump and the turbomolecular pumps are started. All LEDs on the MS detector front panel are off.
7. Allow the Exactive to pump down for 5 minutes.
8. Place the electronics service switch in the Operating Mode position. See Figure 1-5 on page 1-9. When you place the electronics service switch to the Operating Mode position, the following occurs:
   a. Power is provided to all electronic boards. (The 8 kV power to the API source, main RF voltage, and octapole RF voltage remain off.)
   b. The internal computer reboots. After several seconds, the Status LED on the front panel is illuminated yellow to indicate that the data system has started to establish a communication link.

c. After several more seconds, the Status LED is illuminated green to indicate that the data system has established a communication link. Software for the operation of the instrument is then transferred from the data system to the instrument.

d. After 3 minutes, the System LED is illuminated yellow to indicate that the software transfer from the data system is complete and that the instrument is in Standby condition.

If you have an LC or autosampler, start it as is described in the manual that came with the LC or autosampler. If you do not have either, go to topic "Setting Up Conditions for Operation" below.

## Setting Up Conditions for Operation

❖ **To set up your Exactive for operation**

1. Operation of the system with excessive air and water in the vacuum manifold can cause reduced sensitivity and tuning problems. Before you begin data acquisition with your Exactive system, you need to bake out the system for at least twelve hours plus a cooling period of three hours. See "Baking Out the System" on page 3-7.

2. Ensure that the gas pressures are within the operational limits:

   - Nitrogen: 690 ± 140 kPa (6.9 ± 1.4 bar, 100 ± 20 psi)
   - Argon: 690 ± 140 kPa (6.9 ± 1.4 bar, 100 ± 20 psi)[1]

3. In the Exactive Tune software window, check the Vacuum / Bakeout window to see whether the pressure measured by the ion gauge is $\leq 1 \times 10^{-8}$ mbar, and the pressure measured by the Pirani gauge is around 2 mbar. Compare the values of the other parameters in the instrument status window with values that you recorded previously.

4. Continue to set up for ESI or APCI operation as described in *Exactive QuickStart Guide*.

See also "Things to Do Before Operating the Exactive System" on page 2-2 for additional information.

[1]If your system is equipped with the HCD collision cell and argon is used as collision gas.

# Resetting the System

If communication between the Exactive and data system computer is lost, it may be necessary to reset the system using the reset button of the Exactive.

The procedure given here assumes that the Exactive and data system computer are both powered on and are operational. If the instrument, data system computer, or both are off, see "Starting Up the System after a Shutdown" on page 4-6.

To reset the MS detector, press the reset button located on the power panel. See Figure 4-1 on page 4-2. Make sure the Status LED is extinguished before releasing the reset button. When you press the reset button, the following occurs:

1. An interruption of the embedded computer causes the CPU to reboot. All LEDs on the front panel are off except the Power LED.
2. After several seconds, the Status LED is illuminated yellow to indicate that the data system and the instrument are starting to establish a communication link.
3. After several more seconds, the Status LED is illuminated green to indicate that the data system and the instrument have established a communication link. Software for the operation of the instrument is then transferred from the data system to the instrument.
4. After 3 minutes, the software transfer is complete. The System LED is illuminated either green to indicate that the instrument is functional and the high voltages are on, or yellow to indicate that the instrument is functional and it is in Standby condition.

# Chapter 5 Replaceable Parts

This chapter contains part numbers for replaceable and consumable parts for the MS detector, data system, and kits. To ensure proper results in servicing the Exactive system, order only the parts listed or their equivalent.

For information on how to order parts, see "Contacting Us" in the front section of this guide.

## Source Accessory Parts

Stainless steel needle kit, 34 gauge . . . . . OPTON-30004
ESI Probe . . . . . OPTON-20011
Ion Max Housing . . . . . 70005-60176
Syringe 500 µL . . . . . 1248730
Syringe Adaptor Kit . . . . . 70005-62011

See also the *Ion Max and Ion Max-S API Source Hardware Manual* for more lists of replaceable parts for the Ion Max API source and available API probes.

## Heated Ion Transfer Capillary Parts

Ion transfer capillary, 580 micron . . . . . 97055-20199
Ion transfer capillary removal tool . . . . . 70111-20258
Graphite vespel seal ring . . . . . 97055-20442

## Capillary Heater Assembly Exchange Parts

Skimmer . . . . . 70111-20659
Tube lens . . . . . 97055-20038
Complete capillary heater assembly . . . . . 70111-60175

## Source Drain Parts

API source drain . . . . . 97055-20488
Source drain adaptor, Teflon . . . . . 70111-20971
Reducing connector, 1-in × 0.5-in . . . . . 00101-03-00001
Tygon tubing 1-in ID . . . . . 00301-01-00020
Tygon tubing, 0.5-in ID . . . . . 00301-22920
Solvent waste container . . . . . 00301-57020
Filling/venting cap . . . . . 00301-57022

## Gas Supply Parts

Plug In T-piece, 3 × 6 mm . . . . . 1128140
Teflon hose 4 × 1 . . . . . 0690280

## Forevacuum Parts

Pump Sogevac SV 40 BI . . . . . 1238750
Forepump oil GS495 (1L) . . . . . 1249170
Exhaust filter . . . . . 1249350
Noise reduction cover . . . . . 1245190
Drip pan . . . . . 1245210
Exhaust hose, 13 × 3.5, PVC . . . . . 0690720
Forevacuum hose with steel helix; ID=45 mm, 1.6 m . . . . . 1184330
Hose nipple, DN 40, ISO-KF-45 . . . . . 1159230
Centering ring; NW 40, Viton/stainless steel. . . . . 1168170
KF Clamping chain; synthetic, DN 40 . . . . . 1258190
KF Elbow; stainless steel, DN 40. . . . . 1258200
KF Adapter; stainless steel, DN 40, 125 mm long . . . . . 1258210
Hose clamp, NW 40 . . . . . 1181320
Elbow, DN 40, KF, aluminum. . . . . 1181310
Tube clamp, 12-20 mm, W4 . . . . . 1005970

## Ethernet Switch and Cables

Switch . . . . . 2108640
Patch cord, RJ45 SFTP, 5 m . . . . . 2066750
Patch cord, RJ45 SFTP, 1m . . . . . 2064990

## Plug Connectors (to Connect Peripherals)

COMBICON_MC1.5/8ST-3.81 . . . . . 2087270
COMBICON_MC1.5/6ST-3.81 . . . . . 2098690

## Power Cord

Power cord; 16A/230V, C19, 5M UL . . . . . 2112490

## Fan Filter

Fan Filter . . . . . 1234880

## Options

Syringe pump Chemyx Fusion 100 . . . . . 1245740
SWITCHING VALVE MXT 715-000 . . . . . 1239650

# Appendix A Calibration Solutions

This appendix describes how to obtain ready-to-use calibration solutions and how to prepare calibration solutions from the chemicals.

The *positive ion mode calibration solution* allows calibrating the Exactive MS detector and LTQ based Thermo Scientific hybrid instruments (LTQ FT, LTQ FT Ultra, and LTQ Orbitrap series) with ESI source in positive ion mode. It covers a mass range from *m/z* 138 to *m/z* 1822 and is therefore usable for calibrations between *m/z* 50 and 2000.

The *negative ion mode calibration solution* allows calibrating the Exactive MS detector and LTQ based Thermo Scientific hybrid instruments (LTQ FT, LTQ FT Ultra, and LTQ Orbitrap series) with ESI source in negative ion mode. It covers a mass range from *m/z* 265 to *m/z* 1880 and is therefore usable for calibrations between *m/z* 50 and 2000.

This appendix contains the following topics:

# Obtaining Ready-to-Use Calibration Solutions

To free you from time-consuming mixing and dilution steps and to allow you to focus on data acquisition, Thermo Fisher Scientific has arranged with Sigma-Aldrich® that you can order ready-to-use calibration solutions from their website (www.sigma-aldrich.com). Table A-1 shows the available calibration solution packages. 1 EA is a 10 mL Teflon® bottle.

**Table A-1.** Available calibration solution packages

| Calibration Solution | Designation | Product Number |
|---|---|---|
| Positive ion mode | ProteoMass™ LTQ/FT-Hybrid ESI Pos. Mode CalMix | MSCAL5-1EA (1 EA)<br>MSCAL5-10EA (10 EA) |
| Negative ion mode | ProteoMass™ LTQ/FT-Hybrid ESI Neg. Mode CalMix | MSCAL6-1EA (1 EA)<br>MSCAL6-10EA (10 EA) |

**Note** If you have not ordered from Sigma-Aldrich in the past, fill out an on-line My Profile form on the Sigma-Aldrich web site (www.sigma-aldrich.com) to request access to the site. ▲

**Caution** The prepared calibration solutions are light and temperature sensitive. They are shipped under dry ice. Store them in darkness and at -20 °C (-4 °F). ▲

# Calibration- and Test-Chemicals

For preparing the calibration solutions, you need the chemicals listed in Table A-2. They are delivered as part of the Exactive Preinstallation Kit.

**Table A-2.** Exactive calibration- and test-chemicals

| Description | Quantity | Supplier Product Number |
|---|---|---|
| **Supplier: Sigma Chemical Company, see below.** | | |
| Sodium Dodecyl Sulfate | 10 g | L4509-10G |
| Sodium Taurocholate Hydrate | 250 mg | T4009-250MG |
| Caffeine Methanol Solution | 1 mL | C6035-1ML |
| Met-Arg-Phe-Ala acetate salt | 1 mg | M1170-1MG |
| **Supplier: ABCR GmbH & Co. KG, see below.** | | |
| Ultramark® 1621 Mass Spec. Standard | 250 mg | AB172435 |

To order more of these compounds, contact:

Sigma Chemical Company
P. O. Box 14508
St. Louis, Missouri, USA 63178-9916
Phone (800) 325-3010 (in the USA or Canada)
(314) 771-3750 (outside the USA or Canada)

Web site www.sigma-aldrich.com

or

ABCR GmbH & Co. KG
Im Schlehert 10
D-76187 Karlsruhe, Germany
Phone +49 (0)721 950 61-0
Fax +49 (0)721 950 61-80
Email info@abcr.de

Web site www.abcr.de/english.htm

# Preparing the Positive Ion Mode Calibration Solution

The positive ion mode calibration solution consists of caffeine, MRFA, and Ultramark 1621 in an acetonitrile:methanol:water solution containing 1% acetic acid.

Vials of caffeine, MRFA, and Ultramark 1621 are included in the Exactive Calibration- and Test-Chemicals Kit. See Table A-2 on page A-3.

**Caution** **Avoid exposure to potentially harmful materials.** Always wear protective gloves and safety glasses when you handle solvents or corrosives. Also contain waste streams and use proper ventilation. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling of a particular solvent. ▲

### ❖ To prepare the positive ion mode calibration solution

1. Stock Solution: MRFA

   Prepare a 1.5 mL stock solution of 166.7 pmol/µL MRFA in 50:50 methanol:water as follows:

   a. Obtain the vial of L-methionyl-arginyl-phenylalanyl-alanine acetate × $H_2O$ (MRFA). In this form, the MRFA sample has an average molecular weight of 607.7 u.

   b. Dissolve the MRFA sample (1.0 mg) in a total volume of 333 µL of 50:50 methanol:water. Mix the solution (5.0 nmol/µL) thoroughly.

   c. Transfer 50 µL of the 5 nmol/µL solution into a clean polypropylene tube.

   d. Add 1.45 mL of 50:50 methanol:water to the tube. Mix this solution (166.7 pmol/µL) thoroughly.

   e. Label the tube *MRFA stock solution* and store it in a freezer until it is needed.

2. Ultramark 1621 stock solution

   Prepare a 10 mL stock solution of 0.1% Ultramark 1621 in acetonitrile as follows:

   a. Obtain the vial of Ultramark 1621.

   b. Using a syringe, measure out 10 µL of Ultramark 1621, and dissolve it in 10 mL of acetonitrile. Mix the solution thoroughly.

   c. Label the vial *Ultramark 1621 stock solution* and store it in a freezer until it is needed.

3. Positive ion mode calibration solution

   Prepare 10 mL of the positive ion mode calibration solution as follows:

   a. Obtain the 1 mg/mL stock solution of caffeine in 100% methanol.

   b. Pipet 200 µL of the caffeine stock solution into a light-protected, clean, dry 10 mL volumetric flask.

   c. Pipet 100 µL of the MRFA stock solution into the flask.

   d. Pipet 100 µL of the Ultramark 1621 stock solution into the flask.

   e. Pipet 100 µL of glacial acetic acid into the flask.

**Note** Using plastic pipet tips causes contamination of acid stock solutions that can introduce contaminants into the calibration solution. ▲

   f. Pipet 5 mL of acetonitrile into the flask.

   g. Bring the volume of the solution up to the 10 mL-mark on the flask with 50:50 methanol:water.

   h. Mix the calibration solution thoroughly.

   i. Optional: Transfer the solution to a light-protected, clean, dry vial.

   j. Label the flask/vial *Positive Ion Mode Calibration Solution* and store it in a freezer until it is needed.

# Preparing the Negative Ion Mode Calibration Solution

The negative ion mode calibration solution consists of sodium dodecyl sulfate, sodium taurocholate, and Ultramark 1621 in an acetonitrile:methanol:water solution containing 1% acetic acid.

Vials of sodium dodecyl sulfate and sodium taurocholate are included in the Exactive Calibration- and Test-Chemicals Kit. See Table A-2 on page A-3.

**Caution** **Avoid exposure to potentially harmful materials.** Always wear protective gloves and safety glasses when you handle solvents or corrosives. Also contain waste streams and use proper ventilation. Refer to your supplier's Material Safety Data Sheet (MSDS) for proper handling of a particular solvent. ▲

### ❖ To prepare the negative ion mode calibration solution

1. Stock Solution: Sodium Dodecyl Sulfate

    a. Obtain the vial of sodium dodecyl sulfate. In this form, the sample has an average molecular weight of 288.4 u.

    b. Prepare the stock solution of sodium dodecyl sulfate by dissolving 2.88 mg in 10 mL of 50:50 methanol:water.

    c. Mix the solution (1.0 nmol/µL) thoroughly.

    d. Label the vial *Sodium Dodecyl Sulfate stock solution (1 nmol/µL)*.

2. Stock Solution: Sodium Taurocholate

    a. Obtain the vial of sodium taurocholate. In this form, the sample has an average molecular weight of 537.7 u.

    b. Prepare the stock solution of sodium taurocholate by dissolving 5.38 mg in 10 mL of 50:50 methanol:water.

    c. Mix the solution (1.0 nmol/µL) thoroughly.

    d. Label the vial *Sodium Taurocholate stock solution (1 nmol/µL)*.

3. Stock Solution: Ultramark 1621

    To prepare the Ultramark 1621 stock solution (0.1% Ultramark 1621 in acetonitrile), see step 2 of "Preparing the Positive Ion Mode Calibration Solution" above.

4. Negative Ion Mode Calibration Solution

   Prepare 10 mL of the negative ion mode calibration solution, as follows:

   a. Pipet 100 µL of the sodium dodecyl sulfate stock solution into a light-protected, clean, dry 10 mL volumetric flask.

   b. Pipet 100 µL of the sodium taurocholate stock solution into the flask.

   c. Pipet 100 µL of the Ultramark 1621 stock solution into the flask.

   d. Pipet 100 µL of glacial acetic acid into the flask.

**Note** Using plastic pipet tips causes contamination of acid stock solutions that can introduce contaminants into the calibration solution. ▲

   e. Pipet 5 mL of acetonitrile into the flask.

   f. Bring the volume of the solution up to the 10 mL-mark on the flask with 50:50 methanol:water.

   g. Mix the solution thoroughly.

   h. Optional: Transfer the solution to a light-protected, clean, dry vial.

   i. Label the flask/vial *Negative Ion Mode Calibration Solution* and store it in a freezer until it is needed.

# Appendix B Getting Connected

This appendix provides information on how to connect your Exactive mass spectrometer to external devices that are frequently used with the instrument. It also describes the specifications for the peripheral control connections.

This chapter contains the following topics:

# Connecting the API Probe to the MS Detector

❖ **To connect liquid lines to the API probe**

1. Install the Ion Max source housing and the API probe onto the Exactive MS detector as described in the *Ion Max and Ion Max-S API Source Hardware Manual*.
2. Install liquid lines between the switching valve, the LC system, the syringe pump, and the grounding union, as appropriate for your application. For more information, see "Connecting the Inlet Plumbing" on page B-3.
3. Connect the 1 inch ID Tygon™ tubing (P/N 00301-01-00020) to the source housing drain.
4. Insert the other end of the tubing into a waste container, and vent the waste container to a fume exhaust system.

**Caution** Prevent solvent waste from backing up into the API source and mass spectrometer. Always ensure that the drain tubing is above the level of liquid in the waste container.
Do not vent the drain tube connected to the waste container to the same fume exhaust system to which you have connected the forepump. Connecting the API source drain tube and the forepump exhaust to the same fume exhaust system is likely to contaminate the analyzer optics. Equip your laboratory with at least two independent fume exhaust systems. Route the exhaust tubing from the forepump to a dedicated fume exhaust system. Route the drain tube from the API source to the waste container. Vent the waste container to a dedicated fume exhaust system that is independent from that used for exhausting the forepump. ▲

# Connecting the Inlet Plumbing

This section describes how to make the appropriate plumbing connections to introduce sample into the Ion Max API source of the MS detector.

**Note** This guide describes only making plumbing connections for ESI/MS and APCI/MS. For information about making plumbing connections for other probe types (H-ESI probe for example), please refer to the manuals that came with these probes. ▲

## Sample Introduction

With the Exactive MS detector, which allows using a switching valve or a syringe pump, you can introduce sample into the API source as follows:

- The syringe pump is often used to introduce calibration solution for automatic tuning and calibrating in ESI mode. You can also use this technique to introduce a solution of pure analyte at a steady rate in ESI mode, for example, for determining the structure of an unknown compound.

  For direct infusion, connect the syringe pump directly to the Ion Max API source. To push sample into the ion source, set the rate at which the syringe pump depresses the plunger of the syringe.

- You can also use a union Tee to direct samples from the syringe pump into an LC flow (without a column), which then enters the MS detector. This technique is used to introduce sample at a steady rate and at higher solvent flow rates; it is used especially for tuning in ESI or APCI on an analyte of interest. You can also use this technique to introduce a solution of pure analyte at a steady rate in ESI or APCI.

  For high-flow infusion, connect the syringe pump and the outlet of an LC pump to two legs of a union Tee, and then connect the third leg of the union Tee to the ion source. To introduce sample into the ion source, set the rate at which the syringe pump depresses the plunger of the syringe and the flow rate of the solvent stream produced by the LC pump.

- You can introduce samples from a syringe into the loop of the injector valve. You can then use the switching valve to introduce the sample into an LC flow, which then enters the MS detector. This technique is used in ESI or APCI to introduce pure analytes into the MS detector in a slug. It is useful when you have a limited quantity of pure analyte.

For loop injection (flow injection analyses), connect the solvent flow from an LC pump to port 2 of the switching valve, a sample loop to ports 1 and 4 of the valve, and a loop filler to port 5 of the valve. To introduce sample into the ion source, load sample into the sample loop through the loop filler, and then switch the position of the injection valve, allowing the solvent stream to backflush the contents of the sample loop into the ion source.

- You can also use an LC autosampler to introduce samples into an LC flow. This technique is also used in ESI or APCI to introduce a slug of *pure analyte* into the LC flow and then into the MS detector. Furthermore, you can perform LC/MS experiments by using an LC autosampler to introduce a *mixture* onto an LC column. This technique is used with ESI or APCI to separate the analytes before they are introduced sequentially into the MS detector.

  For automated injections using an autosampler, connect the outlet from a liquid chromatography system that contains an autosampler to port 2 of the switching valve. Then set up an autosampler to make automated injections into the solvent flow produced by the LC pump.

Table B-1 summarizes the sample introduction and analytical techniques for ESI/MS and APCI/MS.

**Table B-1.** Sample introduction and analytical techniques for ESI/MS and APCI/MS

| Sample introduction into the MS detector | ESI analytical technique | APCI analytical technique | Procedure for connecting the plumbing |
|---|---|---|---|
| Direct infusion | Analysis of a pure analyte<br>Automatic calibration and tuning | | "Setting Up the Inlet for Direct Infusion" on page B-6 |
| High-flow infusion (syringe pump injection into LC solvent flow) | Analysis of a pure analyte | Analysis of a pure analyte | "Setting Up the Inlet for High-Flow Infusion" on page B-9 |
| Loop injection into LC solvent flow | Analysis of a pure analyte<br>Automatic optimization of tuning using an analyte | Analysis of a pure analyte<br>Automatic optimization of tuning using an analyte | "Setting Up the Inlet for Loop Injections (Flow Injection Analyses)" on page B-14 |
| LC system with autosampler (without chromatographic separation) | Analysis of one or more pure analytes | Analysis of one or more pure analytes | "Setting Up the Inlet for an LC/MS System with an Autosampler" on page B-17 |
| LC system with autosampler (with LC column for chromatographic separations) | Analysis of a mixture | Analysis of a mixture | "Setting Up the Inlet for an LC/MS System with an Autosampler" on page B-17 |

## Fittings, Tubing, Unions, and Sample Loops

The Exactive allows connecting up to two external switching valves (Rheodyne MX Series II™, for example). These valves are 6-port, two-position injection valves. The six ports use standard 10-32 fittings

for high-pressure 1/16 inch OD tubing. The LC union and the union Tee that you use to connect the syringe pump to the ion source also use standard 10-32 fittings for 1/16 inch OD tubing.

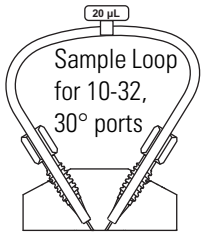
**Note** When cutting PEEK tubing, ensure that you make square cuts. Thermo Fisher Scientific recommends that you use a tubing cutter to cut the PEEK tubing used to make the inlet plumbing connections. ▲

Table B-2 lists the frequently used parts for making plumbing connections for ESI/MS and APCI/MS.

**Table B-2.** Frequently used parts for making plumbing connections for ESI/MS and APCI/MS

| Part | Part Description | Part Number |
|---|---|---|
| | Tubing, fused-silica, 0.1-mm ID × 0.4-mm OD (infusion line) | 00106-10504 |
| | Tubing, fused-silica, 0.1-mm ID × 0.190-mm OD (fused-silica sample tube and fused-silica capillary tube) | 00106-10499 |
| | Tubing, PEEK, 0.005-in ID × 1/16-in OD (red) | 00301-22912 |
| | Tube, Teflon, 0.03-in ID × 1/16-in OD (for use with syringe needle and LC union) | 00301-22915 |
| | Tubing, PVC, unreinforced, 3/8-in ID (clear) (API probe drain tube) | 00301-22895 |
|  | Fitting, adapter, Kel-F™, Upchurch Scientific ™ (connects directly to ESI probe inlet) | 00101-18080 |
|  | Fitting, fingertight, Upchurch Scientific (natural) (used with (red) PEEK tubing) | 00101-18081 |
|  | Ferrule, Kel-F, 0.008-in ID, Upchurch Scientific (clear) (used with fused-silica tubing and the blunt-tip, 34-gauge stainless steel needle included in Metal Needle Kit) | 00101-18114 |
|  | Ferrule, Kel-F, 0.012-in ID, Upchurch Scientific (clear) (used with blunt-tip, 32-gauge stainless steel needle included in Metal Needle Kit) | 00101-18116 |
|  | Ferrule, 0.016-in ID, PEEK, Upchurch Scientific (natural) (for use with fused-silica infusion line) | 00101-18120 |
|  | Ferrule, LC, 1/16-in, stainless steel (used to connect tubing and the sample loop to the switching valve) | 2522-3830 |
|  | Fitting, grounding union, 1/16-in orifice, stainless steel | 00101-18182 |
|  | Fitting, fingertight, Upchurch Scientific (used with red PEEK tubing) | 00101-18195 |

**Table B-2.** Frequently used parts for making plumbing connections for ESI/MS and APCI/MS, continued

| Part | Part Description | Part Number |
|---|---|---|
|  | Ferrule, Fingertight 2, Upchurch Scientific (natural) (used with the Teflon tubing and red PEEK tubing) | 00101-18196 |
|  | Fitting, LC union, 0.010-in orifice, PEEK (black) | 00101-18202 |
|  | Fitting, union Tee, 0.020-in orifice, PEEK (black) | 00101-18204 |
|  | Fitting, adapter union, PEEK, Upchurch Scientific (natural) (used with blunt-tip 32- or 34-gauge stainless steel needle, included in Metal Needle Kit) | 00101-18206 |
|  | Nut, LC for 1/16-in stainless steel, Rheodyne | 2522-0066 |
|  | 5 µL sample loop, stainless steel, Rheodyne | 00110-22026 |
| | 10 µL sample loop, stainless steel, Rheodyne | 00110-22012 |
| | 20 µL sample loop, stainless steel, Rheodyne | 00110-22028 |
| | 100 µL sample loop, stainless steel, Rheodyne | 00110-22018 |

## Setting Up the Inlet for Direct Infusion

To tune and calibrate the Exactive MS detector, use the optional syringe pump to infuse a sample solution into the ion source that is set up for the ESI mode. A suitable syringe pump is available from Thermo Fisher Scientific (Chemyx Fusion 100, P/N 1245740). Also supported is the Harvard Apparatus Model 11 Plus Advanced pump. For instructions about establishing power supply and communication between syringe pump and MS detector, see page B-19.

To introduce sample solution with the syringe pump, you must connect an infusion line between the syringe pump and the grounding union that is held by the grounding bar of the Ion Max API source. See Figure B-1.

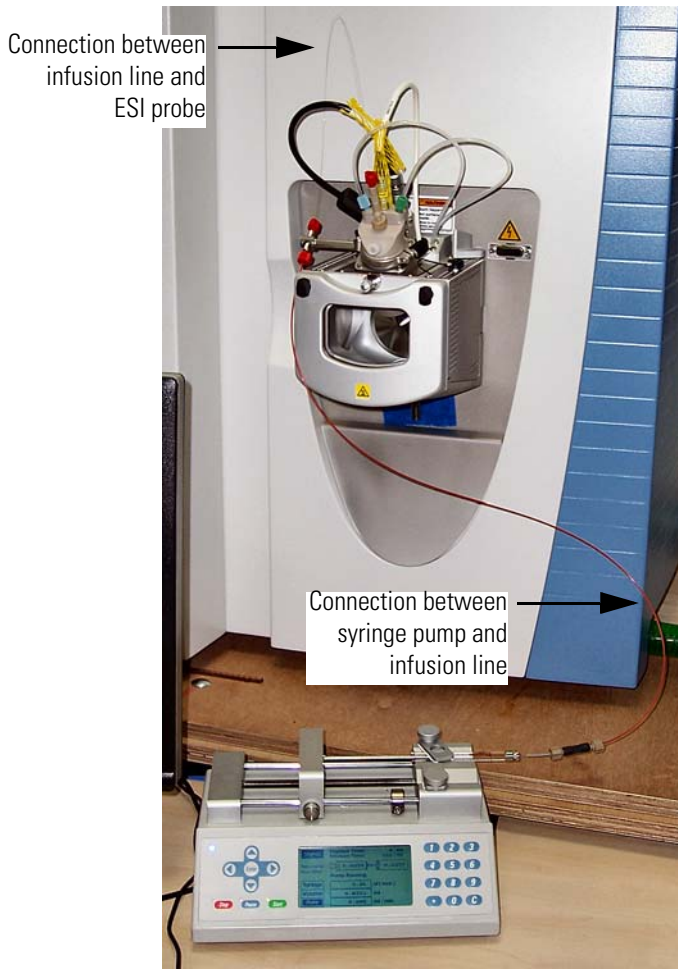



**Figure B-1.** Connection between ESI probe and syringe pump

To connect the syringe to the grounding union, follow these procedures:

1. Setting Up the Syringe
2. Connecting an Infusion Line to the Grounding Union

## Setting Up the Syringe

❖ **To fill the syringe, connect it to the LC union, and insert it into the syringe pump**

1. Fill a clean, 500-µL Unimetrics syringe with your sample solution.
2. Connect a 4-cm (1.5-in) length of Teflon tubing (0.03-in ID × 1/16-in OD) with a fingertight fitting (for a 10-32 receiving port and 1/16-in OD tubing) and a ferrule to the LC union. See Figure B-2.
3. Insert the needle of the syringe into the segment of Teflon tube. Check that the needle tip of the syringe fits readily into the opening

in the free end of the Teflon tubing. If necessary, you can enlarge the opening in the end of the tubing slightly.

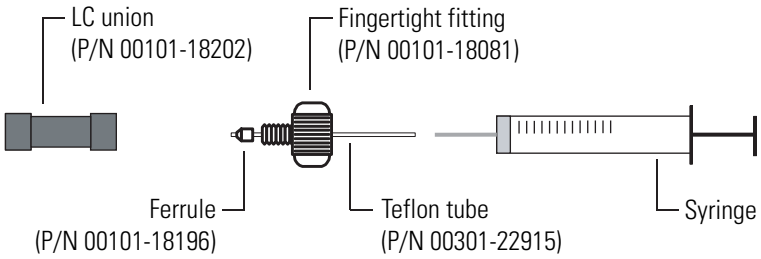



**Figure B-2.** Connecting the syringe and the LC union

4. Place the syringe into the syringe holder of the syringe pump.
5. While squeezing the release buttons on the syringe pump handle, push the handle forward until it just contacts the syringe plunger.

## Connecting an Infusion Line to the Grounding Union

❖ **To connect an infusion line between the LC union and the grounding union**

1. Connect a section of red PEEK tubing (infusion line) with a fingertight fitting and ferrule (for a 10-32 conical receiving port and 1/16-in OD tubing) to the free end of the LC union.
2. Connect the other end of the infusion line with a fingertight fitting (for a 10-32 port and 1/16-in OD tubing) and a ferrule to the grounding union.

   Figure B-3 shows the connection between the grounding union and the LC union made with red PEEK tubing and fingertight fittings.

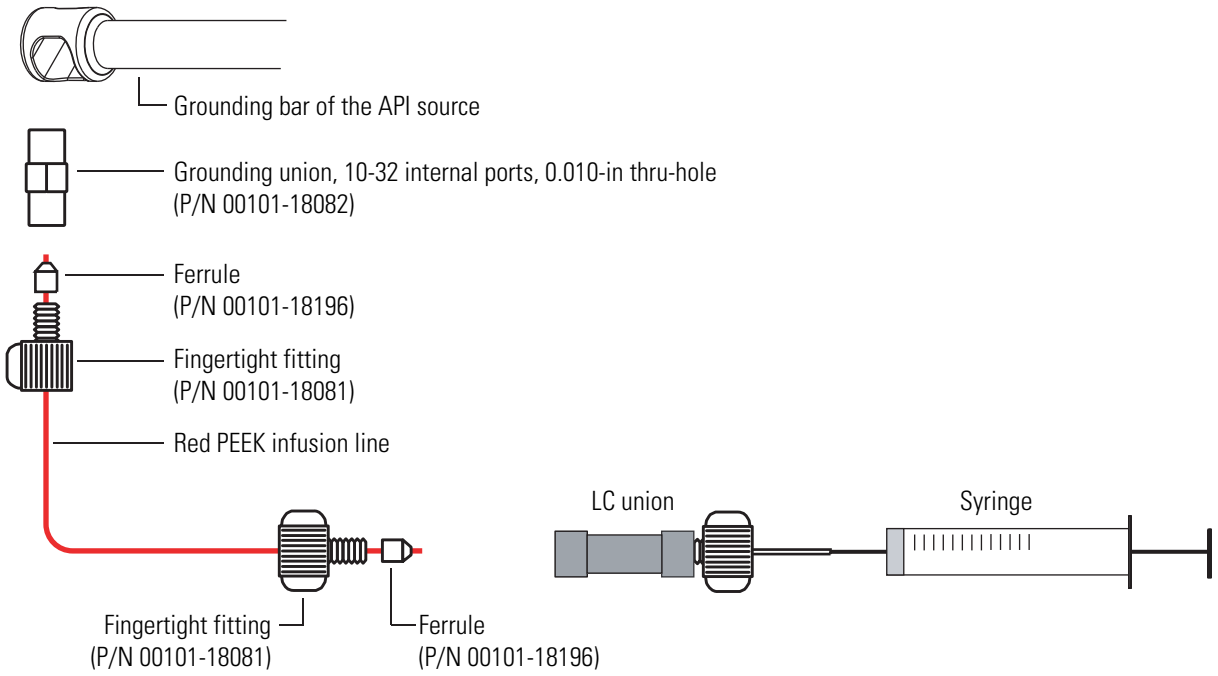



**Figure B-3.** Connecting the infusion line to the LC union and the grounding union

## Setting Up the Inlet for High-Flow Infusion

You can configure (plumb) a switching valve as a loop injector (for flow injection analysis) or as a divert valve. You can also use the switching valve to divert the LC flow between the mass spectrometer and waste when the valve is in the divert valve configuration, or switch between load and inject modes when the valve is in the loop injector configuration.

The Exactive allows connecting up to two external switching valves (Rheodyne MX Series II™, for example). The valves are controlled by the instrument software by means of contact closures. For instructions about establishing power supply and communication between switching valve(s) and MS detector, see page B-19.

For high-flow infusion analyses, connect the syringe pump and the outlet of an LC pump to two legs of a union Tee. Connect the third leg of the union Tee to the ion source.

To make the plumbing connections for sample introduction from the syringe pump into solvent flow from an LC pump, perform these procedures in any order:

- Connecting the Syringe to the Union Tee
- Connecting the Union Tee to the Switching Valve
- Connecting the LC Pump to the Switching Valve

- Connecting the Switching Valve to a Waste Container
- Connecting the Union Tee to the Ion Source

## Connecting the Syringe to the Union Tee

Use red PEEK tubing and fingertight fittings with ferrules to connect the syringe to the LC union Tee.

❖ **To connect the syringe to the LC union Tee**

1. Set up the syringe as described in "Setting Up the Syringe" on page B-7.
2. Using a fingertight fitting and a ferrule, connect a red PEEK infusion line to the free end of the LC union that is connected to the syringe.
3. Using a fingertight fitting and a ferrule, connect the other end of the red PEEK infusion line to the union Tee.

Figure B-4 shows the fittings required to connect the LC union to the union Tee.

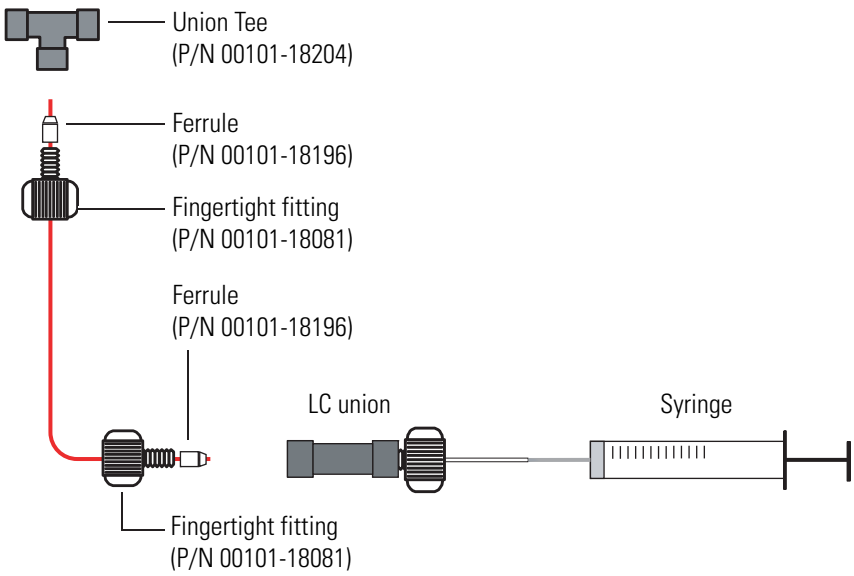



**Figure B-4.** Connecting the LC union to the union Tee

## Connecting the Union Tee to the Switching Valve

❖ **To connect the union Tee to the switching valve**

1. Using a fingertight fitting and a ferrule, connect a length of red PEEK tubing to port 3 of the switching valve. Or, use a stainless steel nut and ferrule to connect the tubing to the switching valve.

2. Using a fingertight fitting and a ferrule, connect the other end of the tubing to the free end of the union Tee. See Figure B-5 and Figure B-6.

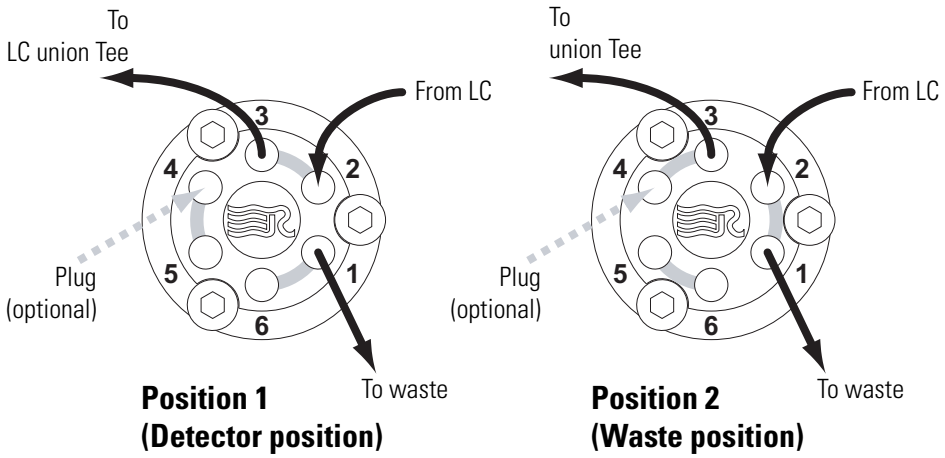



**Figure B-5.** Six-port switching valve connections

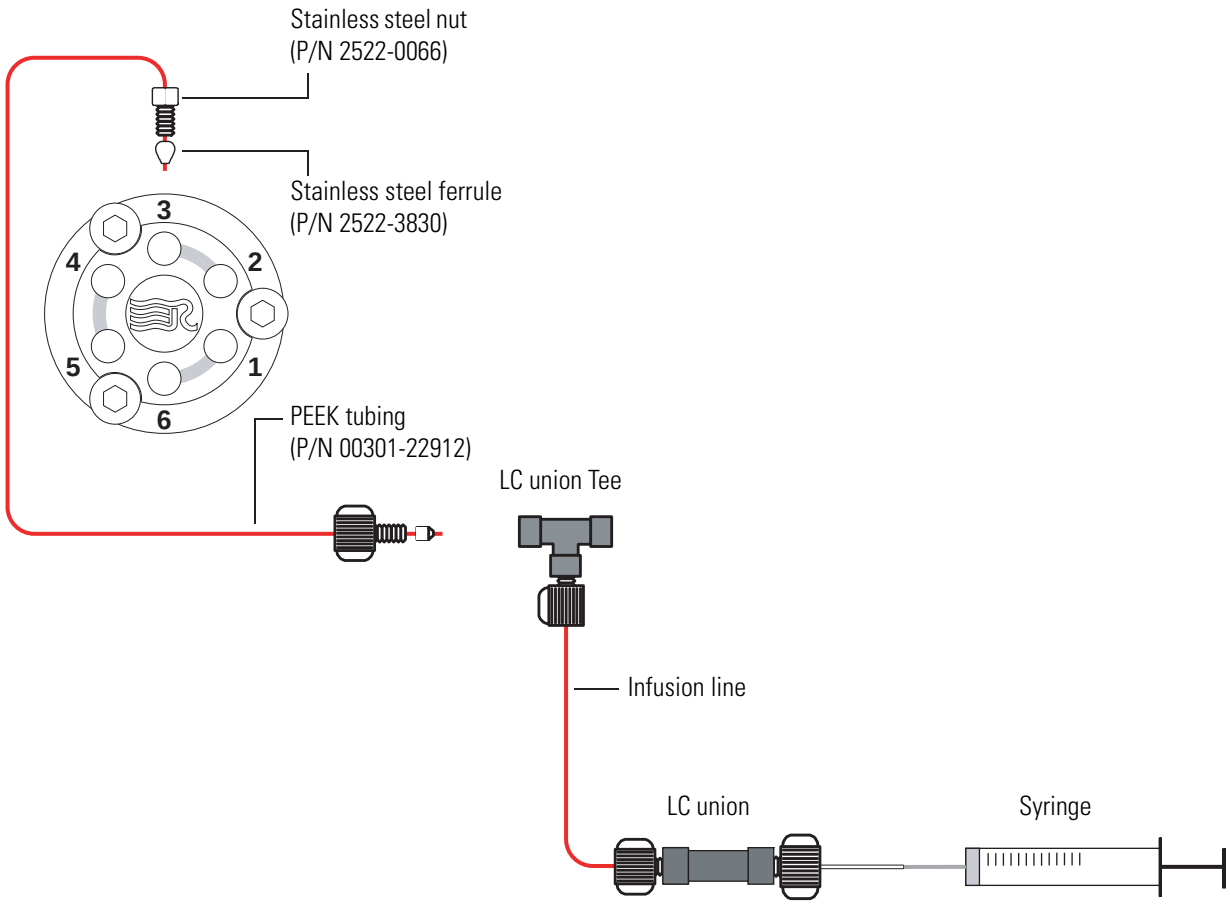



**Figure B-6.** Connecting the LC union to the union Tee

## Connecting the LC Pump to the Switching Valve

❖ **To connect the LC pump to the switching valve**

1. Using a fingertight fitting and a ferrule, connect a length of PEEK tubing to port 2 of the switching valve. Figure B-5 on page B-11 shows the ports of the switching valve.
2. Using an appropriate fitting and ferrule, connect the other end of the tubing to the outlet of the LC.

## Connecting the Switching Valve to a Waste Container

❖ **To connect the switching valve to a waste container**

1. Using a fingertight fitting and a ferrule, connect a length of red PEEK tubing to port 1 of the switching valve. Figure B-5 on page B-11 shows the ports of the switching valve.
2. Insert the other end of the tubing into a suitable waste container.

## Connecting the Union Tee to the Ion Source

❖ **To connect the union Tee to the ion source**

1. Using a fingertight fitting and a ferrule, connect one end of a length of red PEEK tubing to the union Tee. Figure B-7 shows the connections to the union Tee.
2. Depending on whether you are using the ESI probe or the APCI probe, do one of the following:
   - For the APCI probe, use a fingertight fitting and a ferrule to connect the other end of the tubing directly to the sample inlet of the APCI probe. Figure B-7 shows the connection between the union Tee and the sample inlet of the APCI probe.

**Note** Do not use the grounding bar of the Ion Max API source for the APCI probe. A knurled nut secures the grounding bar to the Ion Max ion source. You do not need to remove the grounding bar to run the system in the APCI mode. ▲

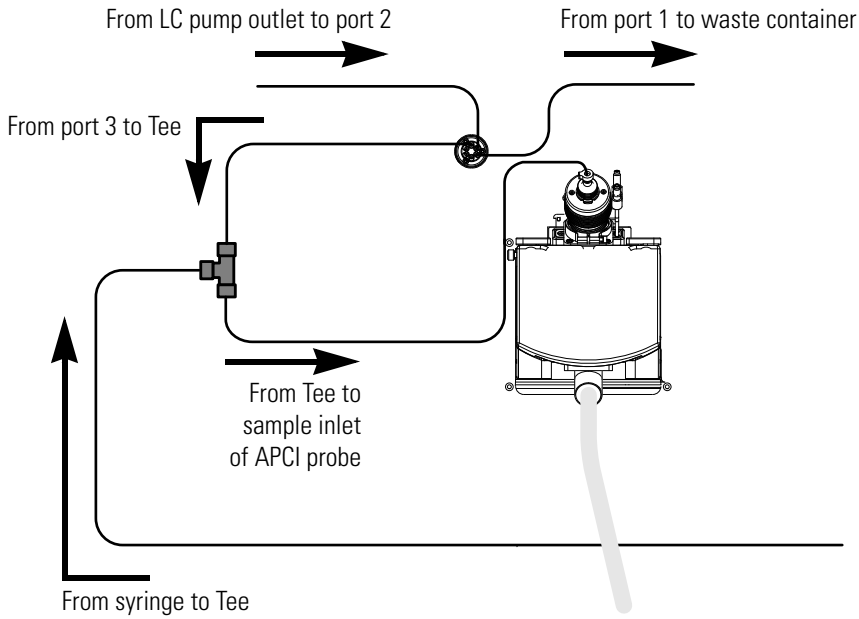



**Figure B-7.** Plumbing diagram showing APCI/MS sample introduction with high-flow infusion

- For the ESI probe, use a fingertight fitting and a ferrule to connect the other end of the tubing to the grounding union that is held by the grounding bar of the Ion Max API source. See Figure B-8 and Figure B-9.

  The grounding union slides into the grounding bar on the Ion Max API source as shown in Figure B-13 on page B-18. For instructions on connecting the grounding union to the ESI probe sample inlet, refer to the *Ion Max and Ion Max-S API Source Hardware Manual*.

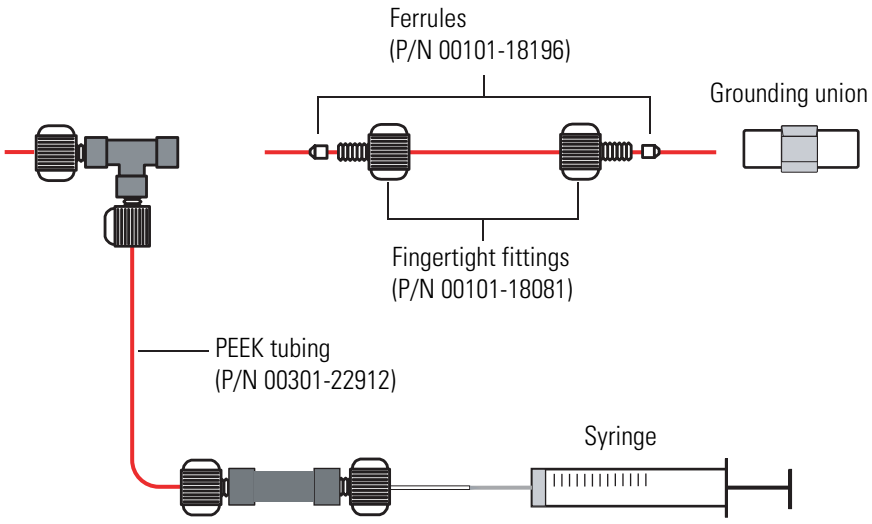



**Figure B-8.** Connecting the union Tee to the grounding union used for the ESI probe

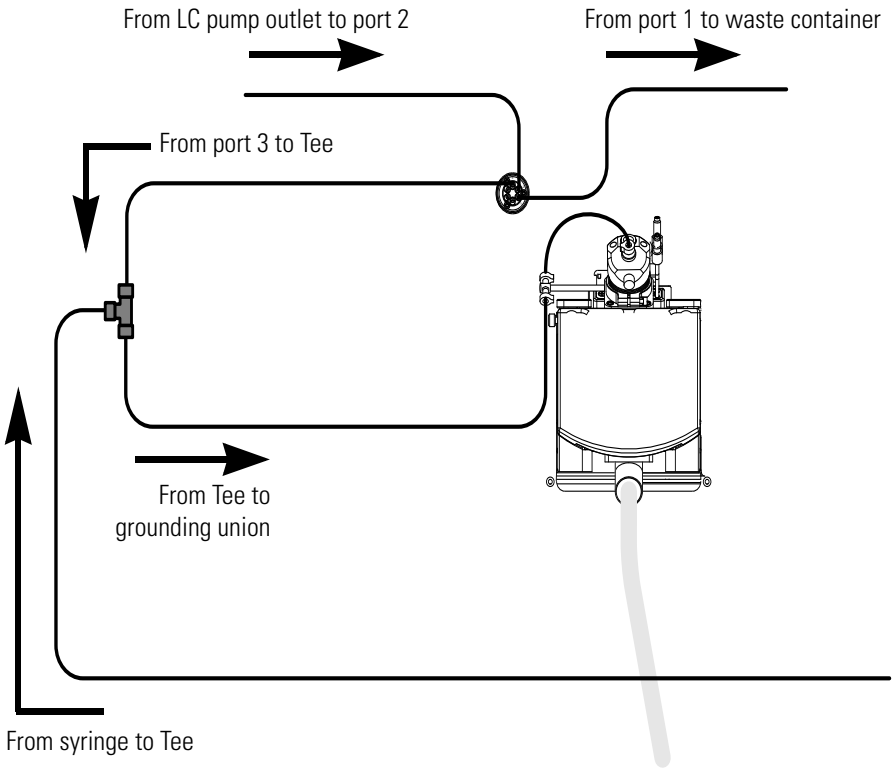



**Figure B-9.** Plumbing diagram showing ESI/MS sample introduction with high-flow infusion

## Setting Up the Inlet for Loop Injections (Flow Injection Analyses)

❖ **To set up the inlet for loop injections**

1. Connect a loop filler to port 5 of the switching valve. See Figure B-10.

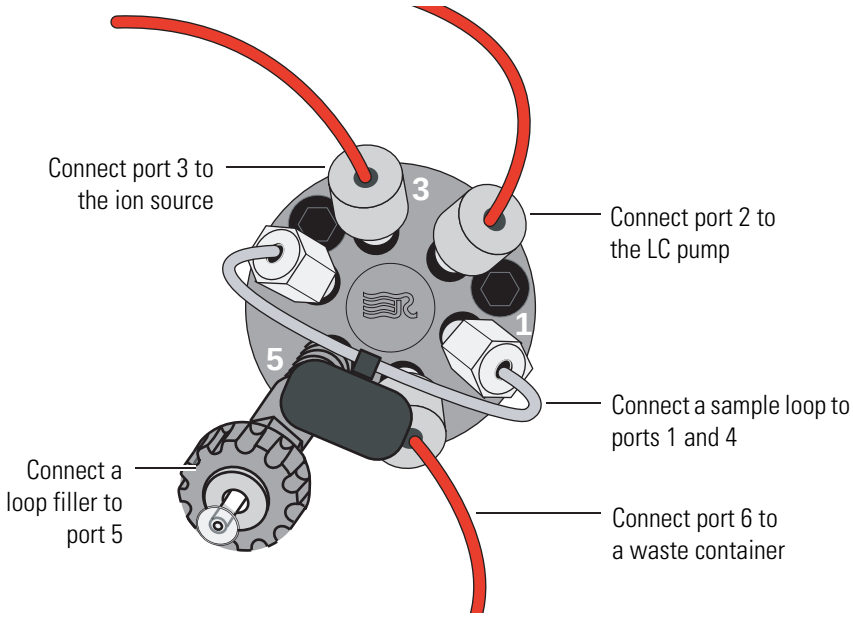



**Figure B-10.** Switching valve set up for loop injections

2. Connect a sample loop to ports 1 and 4.
3. To connect the LC pump to port 2 of the switching valve:
   a. Using an appropriate fitting and ferrule, connect one end of a length of red PEEK tubing to the outlet of the LC pump.

      To produce a stable solvent flow, the Accela Pump requires a minimum backpressure of 40 bar (580 psi). To connect the Accela Pump, use a length of 0.005 inch ID PEEK tubing sufficient to exert a backpressure of 40 bar (580 psi), or connect an in-line back pressure regulator between the LC pump outlet and the switching valve. See the *Accela Pump Hardware Manual* for details.

   b. Using a fingertight fitting and a ferrule, connect the other end of the tubing to port 2 of the switching valve.
4. Depending on whether you are using the APCI probe or the ESI probe, do one of the following to connect port 3 of the switching valve to the ion source:
   - For the APCI probe, use two fingertight fittings and two ferrules to connect a length of red PEEK tubing between port 3 of the switching valve and the sample inlet of the APCI probe. See Figure B-11.

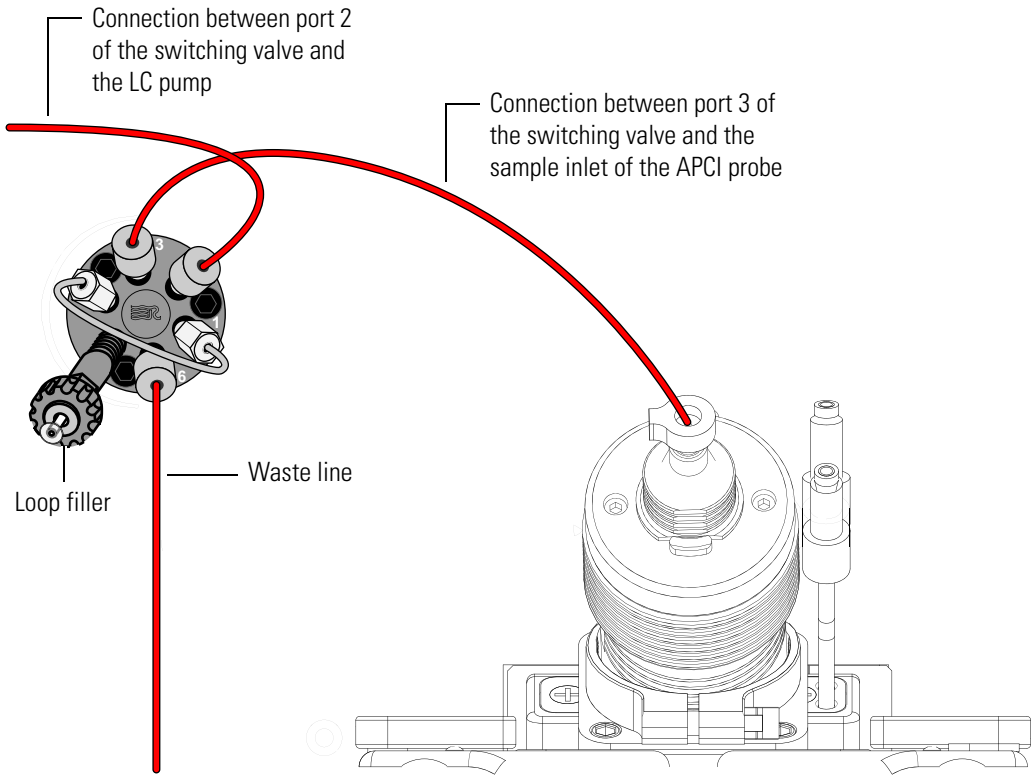



**Figure B-11.** Plumbing diagram for loop injection into the solvent flow from an LC into an APCI probe

- For the ESI probe, use two fingertight fittings and two ferrules to connect a length of red PEEK tubing between port 3 of the switching valve and the grounding union. See Figure B-12. To connect the other end of the grounding union to the ESI probe sample inlet, follow the instructions in the *Ion Max and Ion Max-S API Source Hardware Manual*.

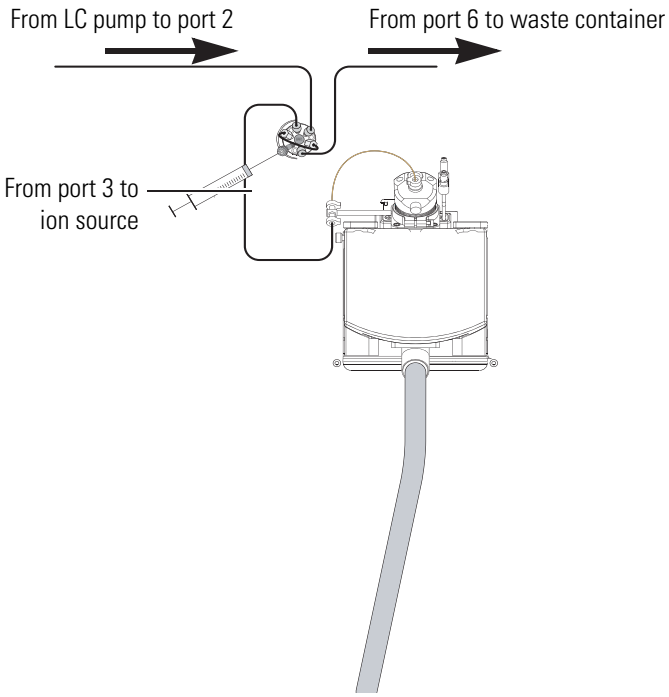



**Figure B-12.** Plumbing diagram for loop injection into the solvent flow from an LC into an ESI probe

5. To connect the switching valve to a waste container:

   a. Use a fingertight fitting and a ferrule to connect one end of a length of red PEEK tubing to port 6 of the switching valve.

   b. Place the other end of the tubing to an appropriate waste container.

## Setting Up the Inlet for an LC/MS System with an Autosampler

❖ **To connect the inlet plumbing for an LC/MS system with an autosampler**

1. Using an appropriate fitting and ferrule, connect one end of a length of red PEEK tubing to the outlet of your LC system.

2. Using a fingertight fitting and a ferrule, connect the other end of the tubing to port 2 of the switching valve.

3. To connect port 3 of the switching valve to the ion source, depending on the probe type, do one of the following:

   - For the ESI probe, use two fingertight fittings and two ferrules to connect a length of red PEEK tubing between port 3 of the switching valve and the grounding union. To connect the other end of the grounding union to the ESI probe sample inlet, follow the instructions in the *Ion Max and Ion Max-S API Source Hardware Manual*.

   - For the APCI probe, use two fingertight fittings and two ferrules to connect a length of red PEEK tubing between port 3 of the switching valve and the APCI probe sample inlet.

4. To connect the switching valve to a waste container:

   a. Using a fingertight fitting and a ferrule, connect one end of a length of red PEEK tubing to port 1 of the switching valve.

   b. Place the other end of the tubing to an appropriate waste container.

## Connecting the Grounding Union to the ESI Probe Sample Inlet

For instruction on connecting the PEEK safety sleeve and fused-silica sample tube from the grounding union to the ESI probe sample inlet, refer to the *Ion Max and Ion Max-S API Source Hardware Manual*.

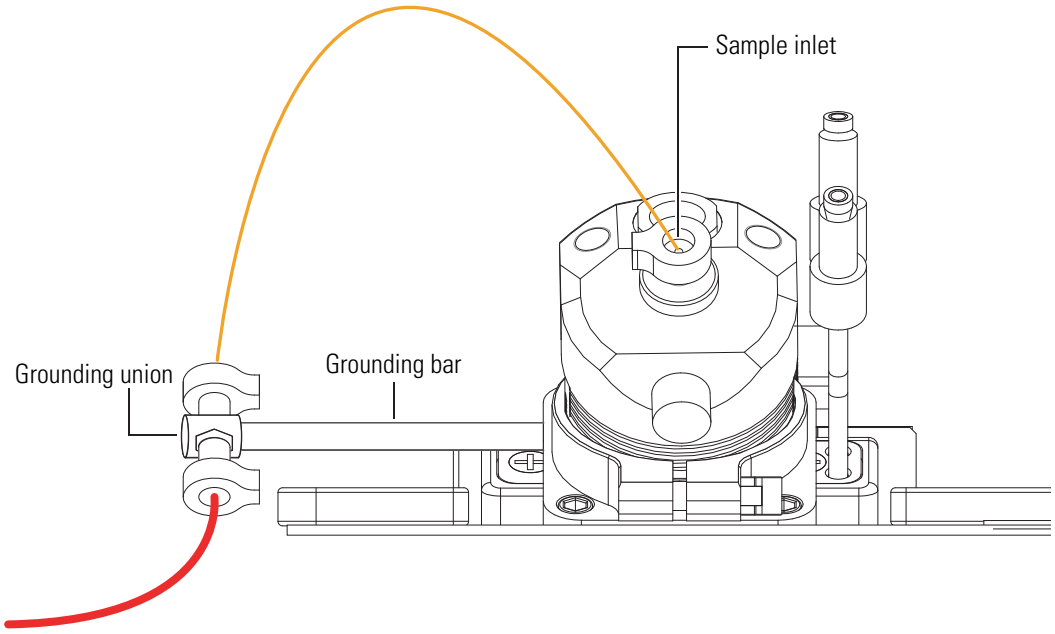


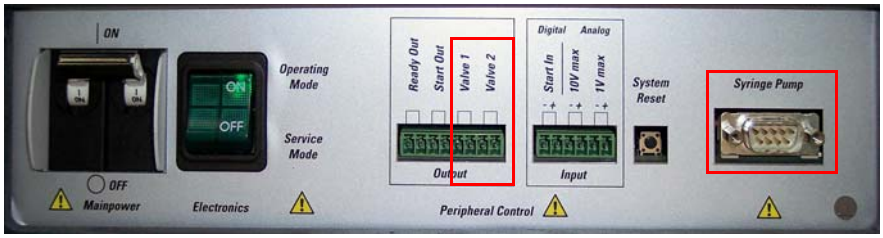

**Figure B-13.** Connecting the grounding union to the sample inlet of the ESI probe

## Establishing Power Supply and Communication for the Syringe Pump

❖ **To establish power supply and communication between syringe pump and MS detector**

1. Use the RS232 interface cable to connect the syringe contact on the power panel of the Exactive MS detector to the RS232 serial port at the rear side of the syringe pump. See Figure B-14. The syringe contact allows controlling the syringe pump by the instrument software.

2. Exactive does not provide electric power for the syringe pump. Connect the power cord of the syringe pump to a wall outlet. To switch on the syringe pump, use the power switch at the rear side of the syringe pump.




Valve 1 port, Valve 2 port  Syringe Pump

**Figure B-14.** Power panel of the Exactive MS

See the *Exactive Software Manual* or the online Help for details about controlling the syringe pump via the Exactive Tune software. See also the manual that came with the syringe pump for further information.

## Establishing Power Supply and Communication for the Switching Valve(s)

❖ **To establish power supply and communication between switching valve(s) and MS detector**

1. Contact closure signals from the Exactive MS detector to a switching valve are transmitted by an interface cable.

   a. Connect the interface cable to the mating connector on the back of the MX Series II Module. The Valve 1 port and the Valve 2 port of the MS detector are located on the power panel of the Exactive. See Figure B-14.

   b. Connect the interface cable of each switching valve to a port: connect wire #1 of the interface cable to one terminal and wire #6 to the other terminal. Exactive does not differentiate between the terminals. A suitable plug connector (P/N 2087270) for the peripheral control output connection is provided with the Exactive Installation Kit.

2. Exactive does not provide electric power for the switching valve(s).

    a. Plug the Universal Power Supply male barrel connector into the MX Module female port at the rear side of the switching valve.

    b. Plug the female connector of the Power Cord into the Universal Power Supply.

    c. Plug the opposite end of the Power Cord into a properly grounded wall outlet. The Universal Power Supply of the valve can be operated from inputs of 100–240 V ac, 50-60 Hz.

See the *Exactive Software Manual* or the online Help for details about controlling a switching valve via the Exactive Tune software. See also the manual that came with the switching valve for further information.

# User I/O Connections

This section describes the specifications for the peripheral control connections. It contains the following topics:



Location and function of the peripheral control connections are described in "Peripheral Control" on page 1-9.

## Output Specifications

Exactive outputs correspond to the status functions listed below. The outputs are potential-free relay contacts, which are closed when the status indicated by the name is true.

The following outputs are available:

- Ready Out (Pins 1 and 2)
- Start Out (Pins 3 and 4)
- Valve 1 (Pins 5 and 6)
- Valve 2 (Pins 7 and 8)

Location and function of the peripheral control outputs are described on page 1-9. Figure B-15 shows the circuit diagram and Table B-3 lists the specifications of the peripheral control output.

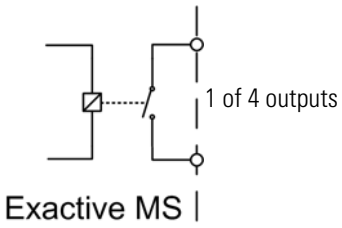



**Figure B-15.** Output equivalent schematic

**Table B-3.** Output circuit specifications

| | |
|---|---|
| Contact closed | $R_{on} < 0.2\ \Omega$ |
| Contact open | $R_{off} > 1\ G\Omega$ |
| Current | $I_{max} = 0.5\ A$ |
| Voltage | $V_{max} = 30\ V$ |
| Power | $P_{max} = 10\ W$ |

# Input Specifications

Exactive provides one digital and two analog inputs to connect external devices.

The following inputs are available:

- Start In port (Pins 1 and 2)
- Analog Input ports (10 V: Pins 3 and 4), (1 V: Pins 5 and 6)

## Start In Input Specification

The Start In input is an input with internal pull-up resistor for connecting external relays contacts or open collector transistors. Start is triggered with the falling edge of input voltage.

Location and function of the Start In input are described on page 1-10. Figure B-16 shows the circuit diagram and Table B-4 lists the specifications of the peripheral control output.

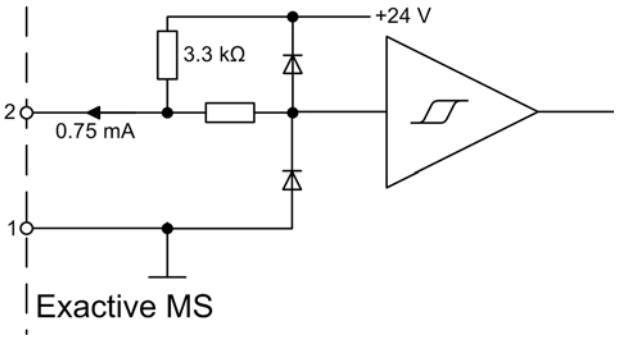



**Figure B-16.** Start In input equivalent schematic

**Table B-4.** Start In input circuit specifications

| | |
|---|---|
| Low level input voltage | $U_{in}$ < 0.8 V @ 0.75 mA |
| High level input voltage | $U_{in}$ > 2.0 V |

## Analog Inputs Specifications

The analog inputs are differential inputs without isolation for connecting external analog voltages. Both inputs reference to the same analog ground.

Location and function of the analog inputs are described on page 1-10. Figure B-17 on page B-23 shows the circuit diagram and Table B-5 on page B-23 lists the specifications of the peripheral control output.

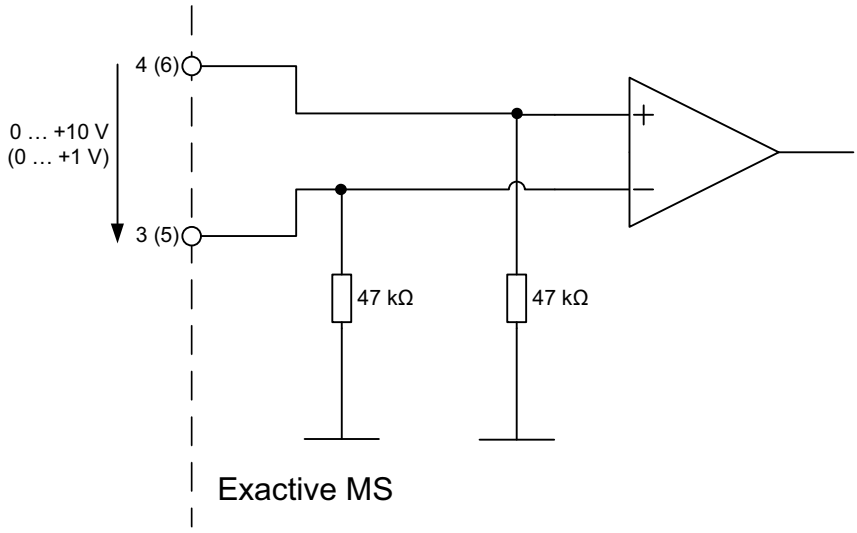



**Figure B-17.** Analog inputs equivalent schematic

**Table B-5.** Analog inputs circuit specifications

| | | |
|---|---|---|
| Input Voltage | $U_{in}$ | 0 …+10 V / 0 … +1 V |
| Input Resistor | $R_{in}$ | 47 kΩ to Ground (connected to PE) |

# Glossary

This section lists and defines terms used in this manual. It also includes acronyms, metric prefixes, symbols, and abbreviations.



## A

**A** ampere

**ac** alternating current

**ADC** analog-to-digital converter; a device that converts data from analog to digital form.

**adduct ion** An ion formed by the joining together of two species, usually an ion and a molecule, and often within the ion source, to form an ion containing all the constituent atoms of both species.

**AGC™** See Automatic Gain Control™ (AGC).

**APCI** See atmospheric pressure chemical ionization (APCI).

**APCI corona discharge current** The ion current carried by the charged particles in the APCI source. The voltage on the APCI corona discharge needle supplies the potential required to ionize the particles. The APCI corona discharge current is set; the APCI corona discharge voltage varies, as required, to maintain the set discharge current.

See also corona discharge and APCI corona discharge voltage.

**APCI corona discharge voltage** The high voltage that is applied to the corona discharge needle in the APCI source to produce the APCI corona discharge. The corona discharge voltage varies, as required, to maintain the set APCI spray current.

See also APCI spray current.

**APCI manifold** The manifold that houses the APCI sample tube and nozzle, and contains the plumbing for the sheath and auxiliary gas.

**APCI needle, corona discharge** A needle to which a sufficiently high voltage (typically ±3 to ±5 kV) is applied to produce a chemical ionization plasma by the corona discharge mechanism.

See also chemical ionization (CI), chemical ionization (CI) plasma, atmospheric pressure chemical ionization (APCI), and corona discharge.

**APCI nozzle** The nozzle in the APCI probe that sprays the sample solution into a fine mist.

See also atmospheric pressure chemical ionization (APCI).

**APCI sample tube** A fused silica tube that delivers sample solution to the APCI nozzle. The APCI sample tube extends from the sample inlet to the APCI nozzle.

See also atmospheric pressure chemical ionization (APCI), and API stack.

**APCI source** Contains the APCI probe assembly, APCI manifold, and API stack.

See also atmospheric pressure chemical ionization (APCI), APCI manifold, and API stack.

**APCI spray current** The ion current carried by the charged particles in the APCI source. The APCI corona discharge voltage varies, as required, to maintain the set spray current.

**APCI vaporizer** A heated tube that vaporizes the sample solution as the solution exits the sample tube and enters the atmospheric pressure region of the APCI source.

See also atmospheric pressure chemical ionization (APCI).

**API** See atmospheric pressure ionization (API).

**API atmospheric pressure region** The first of two chambers in the API source. Also referred to as the spray chamber.

**API capillary-skimmer region** The area between the capillary and the skimmer, which is surrounded by the tube lens. It is also the area of first-stage evacuation in the API source.

**API heated capillary** A tube assembly that assists in desolvating ions that are produced by the ESI or APCI probe.

See also API heated capillary voltage.

**API heated capillary voltage** The dc voltage applied to the heated capillary. The voltage is positive for positive ions and negative for negative ions.

See also API source and API heated capillary.

**API ion transfer capillary** A tube assembly that assists in desolvating ions that are produced by the ESI, NSI, or APCI probe.

See also API ion transfer capillary offset voltage and API ion transfer capillary temperature.

**API ion transfer capillary offset voltage** A dc voltage applied to the ion transfer capillary. The voltage is positive for positive ions and negative for negative ions.

See also API source and API ion transfer capillary.

**API ion transfer capillary temperature** The temperature of the ion transfer capillary, which should be adjusted for different flow rates.

See also API source and API ion transfer capillary.

**API source** The sample interface between the LC and the mass spectrometer. It consists of the API probe (ESI or APCI) and API stack.

See also atmospheric pressure ionization (API), ESI source, APCI source, ESI probe, and API stack.

**API spray chamber** The first of two chambers in the API source. In this chamber the sample liquid exits the probe and is sprayed into a fine mist (ESI or NSI) or is vaporized (APCI) as it is transported to the entrance end of the ion transfer capillary.

**API spray shield** A stainless steel, cylindrical vessel that, in combination with the ESI or APCI flange, forms the atmospheric pressure region of the API source.

See also atmospheric pressure ionization (API).

**API stack** Consists of the components of the API source that are held under vacuum and includes the API spray shield, API ion transfer capillary, API tube lens, skimmer, the ion transfer capillary mount, and the tube lens and skimmer mount.

See also atmospheric pressure ionization (API) and API source.

**API tube lens** A lens in the API source that separates ions from neutral particles as they leave the ion transfer capillary. A potential applied to the tube lens focuses the ions toward the opening of the skimmer and helps to dissociate adduct ions.

See also API tube lens offset voltage, API source, API ion transfer capillary, and adduct ion.

**API tube lens and skimmer mount** A mount that attaches to the heated capillary mount. The tube lens and skimmer attach to the tube lens and skimmer mount.

**API tube lens offset voltage** A DC voltage applied to the tube lens. The value is normally tuned for a specific compound.

See also API tube lens, adduct ion, and source CID.

**APPI** See Atmospheric Pressure Photoionization (APPI).

**ASCII** American Standard Code for Information Interchange

**atmospheric pressure chemical ionization (APCI)** A soft ionization technique done in an ion source operating at atmospheric pressure. Electrons from a corona discharge initiate the process by ionizing the mobile phase vapor molecules. A reagent gas forms, which efficiently produces positive and negative ions of the analyte through a complex series of chemical reactions.

See also electrospray ionization (ESI).

**atmospheric pressure ionization (API)** Ionization performed at atmospheric pressure by using atmospheric pressure chemical ionization (APCI), electrospray ionization (ESI), or nanospray ionization (NSI).

**Atmospheric Pressure Photoionization (APPI)** A soft ionization technique in which an ion is generated from a molecule when it interacts with a photon from a light source.

**atomic mass unit** Atomic Mass Unit (u) defined by taking the mass of one atom of carbon12 as being 12u; unit of mass for expressing masses of atoms or molecules.

**Automatic Gain Control™ (AGC)** Sets the ion injection time to maintain the optimum quantity of ions for each scan. With AGC on, the scan function consists of a prescan and an analytical scan.

**auxiliary gas** The outer-coaxial gas (nitrogen) that assists the sheath (inner-coaxial) gas in dispersing and/or evaporating sample solution as the sample solution exits the APCI, ESI, or H-ESI nozzle.

**auxiliary gas flow rate** The relative rate of flow of auxiliary gas (nitrogen) into the API source reported in arbitrary units.

**auxiliary gas inlet** An inlet in the API probe where auxiliary gas is introduced into the probe.

See also auxiliary gas and atmospheric pressure ionization (API).

**auxiliary gas plumbing** The gas plumbing that delivers outer coaxial nitrogen gas to the ESI or APCI nozzle.

**auxiliary gas valve** A valve that controls the flow of auxiliary gas into the API source.

## B

**b** bit

**B** byte (8 b)

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

**BTU** British thermal unit, a unit of energy

## C

**°C** degrees Celsius

**CE** central electrode (of the Orbitrap)

**cfm** cubic feet per minute

**chemical ionization (CI)** The formation of new ionized species when gaseous molecules interact with ions. The process can involve transfer of an electron, proton, or other charged species between the reactants.

**chemical ionization (CI) plasma** The collection of ions, electrons, and neutral species formed in the ion source during chemical ionization.

See also chemical ionization (CI).

**CI** See chemical ionization (CI).

**CID** See collision-induced dissociation (CID).

**CLT** curved linear trap

**cm** centimeter

**$cm^3$** cubic centimeter

**collision gas** A neutral gas used to undergo collisions with ions.

**collision-induced dissociation (CID)** An ion/neutral process in which an ion is dissociated as a result of interaction with a neutral target species.

**consecutive reaction monitoring (CRM) scan type** A scan type with three or more stages of mass analysis and in which a particular multi-step reaction path is monitored.

**Convectron™ gauge** A thermocouple bridge gauge that is sensitive to the pressure as well as the thermal conductivity of the gas used to measure pressures between X and Y.

**corona discharge** In the APCI source, an electrical discharge in the region around the corona discharge needle that ionizes gas molecules to form a chemical ionization (CI) plasma, which contains CI reagent ions.

See also chemical ionization (CI) plasma and atmospheric pressure chemical ionization (APCI).

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

**CRM** See consecutive reaction monitoring (CRM) scan type.

**C-Trap** curved linear trap

**<Ctrl>** control key on the terminal keyboard

## D

***d*** depth

**Da** dalton

**DAC** digital-to-analog converter; a device that converts data from digital to analog form.

**damping gas** Helium gas introduced into the ion trap mass analyzer that slows the motion of ions entering the mass analyzer so that the ions can be trapped by the RF voltage fields in the mass analyzer.

**data-dependent scan** A scan mode that uses specified criteria to select one or more ions of interest on which to perform subsequent scans, such as MS/MS or ZoomScan.

**dc** direct current

**divert/inject valve** A valve on the mass spectrometer that can be plumbed as a divert valve or as a loop injector.

**DS** data system

**DSP** digital signal processor

## E

**ECD** See electron capture dissociation (ECD).

**EI** electron ionization

**electron capture dissociation (ECD)** A method of fragmenting gas phase ions for tandem mass spectrometric analysis. ECD involves the direct introduction of low energy electrons to trapped gas phase ions.

See also electron transfer dissociation (ETD) and infrared multiphoton dissociation (IRMPD).

**electron multiplier** A device used for current amplification through the secondary emission of electrons. Electron multipliers can have a discrete dynode or a continuous dynode.

**electron transfer dissociation (ETD)** A method of fragmenting peptides and proteins. In electron transfer dissociation (ETD), singly charged reagent anions transfer an electron to multiply protonated peptides within the ion trap mass analyzer. This leads to a rich ladder of sequence ions derived from cleavage at the amide groups along the peptide backbone. Amino acid side chains and important modifications such as phosphorylation are left intact.

See also fluoranthene.

**electrospray ionization (ESI)** A type of atmospheric pressure ionization that is currently the softest ionization technique available to transform ions in solution into ions in the gas phase.

**EMBL** European Molecular Biology Laboratory

**<Enter>** Enter key on the terminal keyboard

**ESD** ElectroStatic Discharge. Discharge of stored static electricity that can damage electronic equipment and impair electrical circuitry, resulting in complete or intermittent failures.

**ESI** See electrospray ionization (ESI).

**ESI flange** A flange that holds the ESI probe in position next to the entrance of the heated capillary, which is part of the API stack. The ESI flange also seals the atmospheric pressure region of the API source and, when it is in the engaged position against the spray shield, compresses the high-voltage safety-interlock switch.

**ESI probe** A probe that produces charged aerosol droplets that contain sample ions. The ESI probe is typically operated at liquid flows of 1 µL/min to 1 mL/min without splitting. The ESI probe includes the ESI manifold, sample tube, nozzle, and needle.

**ESI source** Contains the ESI probe and the API stack.

See also electrospray ionization (ESI), ESI probe, and API stack.

**ESI spray current** The flow of charged particles in the ESI source. The voltage on the ESI spray needle supplies the potential required to ionize the particles.

**ESI spray voltage** The high voltage that is applied to the spray needle in the ESI source to produce the ESI spray current. In ESI, the voltage is applied to the spray liquid as it emerges from the nozzle.

See also ESI spray current.

**ETD** See electron transfer dissociation (ETD).

**eV** Electron Volt. The energy gained by an electron that accelerates through a potential difference of one volt.

**external lock mass** A lock that is analyzed in a separate MS experiment from your sample. If you need to run a large number of samples, or if accurate mass samples will be intermingled with standard samples, you might want to use external lock masses. These allow more rapid data acquisition by eliminating the need to scan lock masses during each scan.

See also internal lock mass.

## F

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

**°F** degrees Fahrenheit

***.fasta* file** extension of a SEQUEST® search database file

**ft** foot

**Fast Fourier Transform (FFT)** An algorithm that performs a Fourier transformation on data. A Fourier transform is the set of mathematical formulae by which a time function is converted into a frequency-domain function and the converse.

**FFT** See Fast Fourier Transform (FFT).

**fluoranthene** A reagent anion that is used in an electron transfer dissociation (ETD) experiment.

**firmware** Software routines stored in read-only memory. Startup routines and low-level input/output instructions are stored in firmware.

**forepump** The pump that evacuates the foreline. A rotary-vane pump is a type of forepump.

**Fourier Transform - Ion Cyclotron Resonance Mass Spectrometry** A technique that determines the mass-to-charge ratio of an ion by measuring its cyclotron frequency in a strong magnetic field.

**fragment ion** A charged dissociation product of an ionic fragmentation. Such an ion can dissociate further to form other charged molecular or atomic species of successively lower formula weights.

**fragmentation** The dissociation of a molecule or ion to form fragments, either ionic or neutral. When a molecule or ion interacts with a particle (electron, ion, or neutral species) the molecule or ion absorbs energy and can subsequently fall apart into a series of charged or neutral fragments. The mass spectrum of the fragment ions is unique for the molecule or ion.

**FT** Fourier Transformation

**FT-ICR MS** See Fourier Transform - Ion Cyclotron Resonance Mass Spectrometry.

**FTMS** Fourier Transformation Mass Spectroscopy

**full-scan type** Provides a full mass spectrum of each analyte or parent ion. With the full-scan type, the mass analyzer is scanned from the first mass to the last mass without interruption. Also known as single-stage full-scan type.

**FWHM** Full Width at Half Maximum

## G

**g** gram

**G** Gauss; giga ($10^{9}$)

**GC** gas chromatograph; gas chromatography

**GC/MS** gas chromatography / mass spectrometer

**GUI** graphical user interface

## H

**h** hour

***h*** height

**handshake** A signal that acknowledges that communication can take place.

**HCD** Higher Energy Collision Induced Dissociation

**header information** Data stored in each data file that summarizes the information contained in the file.

**H-ESI source** Heated-electrospray ionization (H-ESI) converts ions in solution into ions in the gas phase by using electrospray ionization (ESI) in combination with heated auxiliary gas.

**high performance liquid chromatography (HPLC)**

Liquid chromatography in which the liquid is driven through the column at high pressure. Also known as high pressure liquid chromatography.

**HPLC** See high performance liquid chromatography (HPLC).

**HV** high voltage

**Hz** hertz (cycles per second)

## I

**ICR** ion cyclotron resonance

**ID** inside diameter

**IEC** International Electrotechnical Commission

**IEEE** Institute of Electrical and Electronics Engineers

**in.** inch

**infrared multiphoton dissociation (IRMPD)** In infrared multiphoton dissociation (IRMPD), multiply charged ions consecutively absorb photons emitted by a infrared laser until the vibrational excitation is sufficient for their fragmentation. The fragments continue to pick up energy from the laser pulse and fall apart further to ions of lower mass.

See also electron capture dissociation (ECD).

**instrument method** A set of experiment parameters that define Xcalibur operating settings for the autosampler, liquid chromatograph (LC), mass spectrometer, divert valve, syringe pump, and so on. Instrument methods are saved as file type .meth.

**internal lock mass** A lock that is analyzed during the same MS experiment as your sample and is contained within the sample solution or infused into the LC flow during the experiment. Internal lock masses provide the most accurate corrections to the data.

See also external lock mass.

**I/O** input/output

**ion gauge** Measures the pressure in the mass analyzer region (high vacuum region) of the vacuum manifold.

**ion optics** Focuses and transmits ions from the API source to the mass analyzer.

**ion source** A device that converts samples to gas-phase ions.

**IRMPD** See infrared multiphoton dissociation (IRMPD).

## K

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

**K** kilo ($2^{10}$, 1024)

**KEGG** Kyoto Encyclopedia of Genes and Genomes

**kg** kilogram

## L

***l*** length

**L** liter

**LAN** local area network

**lb** pound

**LC** See liquid chromatography (LC).

**LC/MS** See liquid chromatography / mass spectrometry (LC/MS).

**LED** light-emitting diode

**LHe** liquid helium

**liquid chromatography (LC)** A form of elution chromatography in which a sample partitions between a stationary phase of large surface area and a liquid mobile phase that percolates over the stationary phase.

**liquid chromatography / mass spectrometry (LC/MS)** An analytical technique in which a high-performance liquid chromatograph (LC) and a mass spectrometer (MS) are combined.

**LN2** liquid nitrogen

**lock mass** A known reference mass in the sample that is used to correct the mass spectral data in an accurate mass experiment and used to perform a real-time secondary mass calibration that corrects the masses of other peaks in a scan. Lock masses with well-defined, symmetrical peaks work best. You can choose to use internal lock mass or external lock mass.

**log file** A text file, with a .log file extension, that is used to store lists of information.

## M

**µ** micro ($10^{-6}$)

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

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

**$M^+$** molecular ion

**MALDI** See matrix-assisted laser desorption/ionization (MALDI).

**matrix-assisted laser desorption/ionization (MALDI)** Ionization by effect of illumination with a beam of laser generated light onto a matrix containing a small proportion of analyte. A mass spectrometric technique that is used for the analysis of large biomolecules.

**MB** Megabyte (1048576 bytes)

**$MH^+$** protonated molecular ion

**min** minute

**mL** milliliter

**mm** millimeter

**MRFA** A peptide with the amino acid sequence methionine–arginine–phenylalanine–alanine.

**MS** mass spectrometer; mass spectrometry

**MS** $MS^n$ power: where n = 1

**MS scan modes** Scan modes in which only one stage of mass analysis is performed. The scan types used with the MS scan modes are full-scan type and selected ion monitoring (SIM) scan type.

**MSDS** Material Safety Data Sheet

**MS/MS** Mass spectrometry/mass spectrometry, or tandem mass spectrometry is an analytical technique that involves two stages of mass analysis. In the first stage, ions formed in the ion source are analyzed by an initial analyzer. In the second stage, the mass-selected ions are fragmented and the resultant ionic fragments are mass analyzed.

**MSn scan mode** The scan power equal to 1 to 10, where the scan power is the power *n* in the expression MSn. MSn is the most general expression for the scan mode, which can include the following:

- The scan mode corresponding to the one stage of mass analysis in a single-stage full-scan experiment or a selected ion monitoring (SIM) experiment
- The scan mode corresponding to the two stages of mass analysis in a two-stage full-scan experiment or a selected reaction monitoring (SRM) experiment
- The scan mode corresponding to the three to ten stages of mass analysis ($n$ = 3 to $n$ = 10) in a multi-stage full-scan experiment or a consecutive reaction monitoring (CRM) experiment.

See also MS scan modes and MS/MS.

**multipole** A symmetrical, parallel array of (usually) four, six, or eight cylindrical rods that acts as an ion transmission device. An RF voltage and dc offset voltage are applied to the rods to create an electrostatic field that efficiently transmits ions along the axis of the multipole rods.

***m/z*** Mass-to-charge ratio. An abbreviation used to denote the quantity formed by dividing the mass of an ion (in u) by the number of charges carried by the ion. For example, for the ion $C_7H_7^{2+}$, $m/z$=45.5.

## N

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

**nanospray ionization (NSI)** A type of electrospray ionization (ESI) that accommodates very low flow rates of sample and solvent on the order of 1 to 20 nL/min (for static nanospray) or 100 to 1000 nL/min (for dynamic nanospray).

**NCBI** National Center for Biotechnology Information (USA)

**NIST** National Institute of Standards and Technology (USA)

**NMR** Normal Mass Range

**NSI** See nanospray ionization (NSI).

**octapole** An octagonal array of cylindrical rods that acts as an ion transmission device. An RF voltage and dc offset voltage applied to the rods create an electrostatic field that transmits the ions along the axis of the octapole rods.

## O

**OD** outside diameter

**OT** Orbitrap

**OVC** outer vacuum case

**Ω** ohm

## P

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

**Pa** pascal

**PCB** printed circuit board

**PDA detector** Photodiode Array detector is a linear array of discrete photodiodes on an integrated circuit chip. It is placed at the image plane of a spectrometer to allow a range of wavelengths to be detected simultaneously.

**PE** protective earth

**PID** proportional / integral / differential

**P/N** part number

**p-p** peak-to-peak voltage

**ppm** parts per million

**PQD** pulsed-Q dissociation

**psig** pounds per square inch, gauge

**PTM** posttranslational modification

## Q

**quadrupole** A symmetrical, parallel array of four hyperbolic rods that acts as a mass analyzer or an ion transmission device. As a mass analyzer, one pair of opposing rods has an oscillating radio frequency (RF) voltage superimposed on a positive direct current (dc) voltage. The other pair has a negative dc voltage and an RF voltage that is 180 degrees out of phase with the first pair of rods. This creates an electrical field (the quadrupole field) that efficiently transmits ions of selected mass-to-charge ratios along the axis of the quadrupole rods.

## R

**RAM** random access memory

**raw data** Uncorrected liquid chromatograph and mass spectrometer data obtained during an acquisition. Xcalibur and Xcalibur-based software store this data in a file that has a .raw file extension.

**resolution** The ability to distinguish between two points on the wavelength or mass axis.

**retention time (RT)** The time after injection at which a compound elutes. The total time that the compound is retained on the chromatograph column.

**RF** radio frequency

**RF lens** A multipole rod assembly that is operated with only radio frequency (RF) voltage on the rods. In this type of device, virtually all ions have stable trajectories and pass through the assembly.

**RF voltage** An ac voltage of constant frequency and variable amplitude that is applied to the ring electrode or endcaps of the mass analyzer or to the rods of a multipole. Because the frequency of this ac voltage is in the radio frequency (RF) range, it is referred to as RF voltage.

**RMS** root mean square

**ROM** read-only memory

**rotary-vane pump** A mechanical vacuum pump that establishes the vacuum necessary for the proper operation of the turbomolecular pump. (Also called a roughing pump or forepump.)

**RS-232** An accepted industry standard for serial communication connections. This Recommended Standard (RS) defines the specific lines and signal characteristics used by serial communications controllers to standardize the transmission of serial data between devices.

**RT** An abbreviated form of the phrase *retention time (RT)*. This shortened form is used to save space when the retention time (in minutes) is displayed in a header, for example, RT: 0.00-3.75.

## S

**s** second

**selected ion monitoring (SIM) scan type** A scan type in which the mass spectrometer acquires and records ion current at only one or a few selected mass-to-charge ratio values.

See also selected reaction monitoring (SRM) scan type.

**selected reaction monitoring (SRM) scan type** A scan type with two stages of mass analysis and in which a particular reaction or set of reactions, such as the fragmentation of an ion or the loss of a neutral moiety, is monitored. In SRM a limited number of product ions is monitored.

**SEM** secondary electron multiplier

**Serial Peripheral Interface (SPI)** hardware and firmware communications protocol

**serial port** An input/output location (channel) for serial data transmission.

**sheath gas** The inner coaxial gas (nitrogen), which is used in the API source to help nebulize the sample solution into a fine mist as the sample solution exits the ESI or APCI nozzle.

**sheath gas flow rate** The rate of flow of sheath gas into the API source. A measurement of the relative flow rate (in arbitrary units) that needs to be provided at the sheath gas inlet to provide the required flow of sheath gas to the ESI or APCI nozzle.

**sheath gas inlet** An inlet in the API probe where sheath gas is introduced into the probe.

**sheath gas plumbing** The gas plumbing that delivers sheath gas to the ESI or APCI nozzle.

**sheath gas pressure** The rate of flow of sheath gas (nitrogen) into the API source. A measurement of the relative flow rate (in arbitrary units) that needs to be provided at the sheath gas inlet to provide the required flow of inner coaxial nitrogen gas to the ESI or APCI nozzle. A software-controlled proportional valve regulates the flow rate.

See also sheath gas.

**sheath gas valve** A valve that controls the flow of sheath gas into the API source. The sheath gas valve is controlled by the data system.

**signal-to-noise ratio (S/N)** The ratio of the signal height (S) to the noise height (N). The signal height is the baseline corrected peak height. The noise height is the peak-to-peak height of the baseline noise.

**SIM** See selected ion monitoring (SIM) scan type.

**skimmer** A vacuum baffle between the higher pressure capillary-skimmer region and the lower pressure region. The aperture of the skimmer is offset with respect to the bore of the ion transfer capillary.

**source CID** A technique for fragmenting ions in an atmospheric pressure ionization (API) source. Collisions occur between the ion and the background gas, which increase the internal energy of the ion and stimulate its dissociation.

**SPI** See Serial Peripheral Interface (SPI).

**SRM** See selected reaction monitoring (SRM) scan type.

**sweep gas** Nitrogen gas that flows out from behind the sweep cone in the API source. Sweep gas aids in solvent declustering and adduct reduction.

See also sweep gas flow rate.

**sweep gas flow rate** The rate of flow of sweep gas into the API source. A measurement of the relative flow rate (in arbitrary units) to provide the required flow of nitrogen gas to the sweep cone of the API source.

See also sweep gas.

**syringe pump** A device that delivers a solution from a syringe at a specified rate.

## T

**T** Tesla

**target compound** A compound that you want to identify or quantitate or that a specific protocol (for example, an EPA method) requires that you look for. Target compounds are also called analytes, or target analytes.

**TIC** See total ion current (TIC).

**TMP** See turbomolecular pump.

**Torr** A unit of pressure, equal to 1 mm of mercury and 133.32 Pa.

**total ion current (TIC)** The sum of the ion current intensities across the scan range in a mass spectrum.

**tube lens offset** The voltage offset from ground that is applied to the tube lens to focus ions toward the opening of the skimmer.

See also source CID.

**Tune Method** A defined set of mass spectrometer tune parameters for the ion source and mass analyzer. Tune methods are defined by using the Exactive Tune, Tune Plus (LCQ Series, LXQ, and LTQ), or Tune Master (TSQ Quantum) window and saved as the file type .mstune, .LCQTune, .LTQTune, or .TSQTune, respectively.

A tune method stores tune parameters only. (Calibration parameters are stored separately, not with the tune method.)

**tune parameters** Instrument parameters whose values vary with the type of experiment.

**turbomolecular pump** A vacuum pump that provides a high vacuum for the mass spectrometer and detector system.

**TWA** time weighted average

## U

**u** atomic mass unit

**UHV** ultra high vacuum

**Ultramark 1621** A mixture of perfluoroalkoxycyclotriphosphazenes used for ion trap calibration and tuning. It provides ESI singly charged peaks at *m/z* 1022.0, 1122.0, 1222.0, 1322.0, 1422.0, 1522.0, 1622.0, 1722.0, 1822.0, and 1921.9.

**UMR** Universal Mass Range

## V

**V** volt

**V ac** volts alternating current

**V dc** volts direct current

**vacuum manifold** A thick-walled, aluminum chamber with machined flanges on the front and sides and various electrical feedthroughs and gas inlets that encloses the API stack, ion optics, mass analyzer, and ion detection system.

**vacuum system** Components associated with lowering the pressure within the mass spectrometer. A vacuum system includes the vacuum manifold, pumps, pressure gauges, and associated electronics.

**vent valve** A valve that allows the vacuum manifold to be vented to air or other gases. A solenoid-operated valve.

**vol** volume

## W

***w*** width

**W** watt

**WEEE** European Union Waste Electrical and Electronic Equipment Directive. Provides guidelines for disposal of electronic waste.

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