

# ETD Module

## Hardware Manual

For Velos Pro and LTQ XL Mass Spectrometers  
(compatible with Orbitrap Velos Pro, LTQ Orbitrap Velos,  
Orbitrap Elite, and LTQ Orbitrap XL)

98000-97004 Revision D September 2015



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Release history: Rev A, March 2008; Rev B, April 2009; Rev C, June 2012; Rev D, Sept 2015

Software version: Microsoft Windows 7 Professional (32-bit and 64-bit) SP1—Thermo Foundation 2.0 and later, and Thermo Xcalibur 2.2 and later; Windows XP Workstation SP3—Foundation 1.0.2 SP2 or earlier, and Xcalibur 2.1 SP1 or earlier; Thermo LTQ Tune Plus 2.7.0 and later

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CISPR 11: 2009 + A1	EN 61000-3-3: 2013
CISPR 22: 2008	EN 61000-4-2: 2009
ICES-003: 2014	EN 61000-4-3: 2006 + A1+ A2
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EN 55011: 2009 + A1	EN 61000-4-5: 2006
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EN 55024: 2010

EN 61326-1: 2013

EN 61000-3-2: 2006 + A1+ A2

EN 61000-3-3: 2008

EN 61000-4-2: 2009

EN 61000-4-3: 2006 + A1+ A2

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EN 61000-4-5: 2006

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# Preface

The *ETD Module Hardware Manual* describes the modes of operation and hardware components for the Thermo Scientific™ Velos Pro™/ETD, LTQ™ Velos™/ETD, and LTQ XL™/ETD systems. It also provides the systems' cleaning and maintenance procedures.

For hardware information about the mass spectrometers, refer to the *LTQ Series Hardware Manual*. For information about the required gases, refer to the *LTQ Series Getting Connected Guide*.

**Note** For the LTQ Velos/ETD system, follow the Velos Pro/ETD system information, unless otherwise noted.

## Contents

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### ❖ To suggest changes to the documentation or to the Help

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## Related Documentation

The Velos Pro and LTQ XL mass spectrometers (MSs) include complete documentation. In addition to this guide, you can also access the following documents as PDF files from the data system computer:

- *LTQ Series Preinstallation Requirements Guide*
- *LTQ Series Getting Connected Guide*
- *LTQ Series Getting Started Guide*
- *LTQ Series Hardware Manual*
- *ETD Module Getting Started Guide*
- *Safety and Regulatory Guide*

The ETD module also ships with a printed copy of the *Safety and Regulatory Guide*. This guide contains important safety information about Thermo Scientific liquid chromatography (LC) and mass spectrometry (MS) systems. Make sure that all lab personnel have read and have access to this document.

### ❖ To view the product manuals

From the Microsoft™ Windows™ taskbar, choose **Start > All Programs > Thermo Instruments > Manuals > *model***, where *model* is your specific LTQ Series model, and then open the PDF file to view it.

**Note** For Thermo Xcalibur™ version 2.0.7 or earlier (Microsoft Windows XP operating system), choose **Start > All Programs > Xcalibur > Manuals > LTQ > *model***.

### ❖ To view the data system Help

- From the application window, choose **Help** from the menu bar.
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## Cautions and Special Notices

Make sure you follow the cautions and special notices presented in this guide. Cautions and special notices appear in boxes; those concerning safety or possible system damage also have corresponding caution symbols.

This guide uses the following types of cautions and special notices.



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





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**Note** Highlights information of general interest.

**Tip** Highlights helpful information that can make a task easier.

The *ETD Module Hardware Manual* contains the following caution-specific symbols (Table 1).

**Table 1.** Caution-specific symbols and their meanings

Symbol	Meaning
	<b>Chemical hazard:</b> Observe Good Laboratory Practices (GLP) when handling chemicals. Only work with volatile chemicals under a fume or exhaust hood. Wear gloves and other protective equipment, as appropriate, when handling toxic, carcinogenic, mutagenic, corrosive, or irritant chemicals. Use approved containers and proper procedures to dispose of waste oil and when handling wetted parts of the instrument.
	<b>Hot surface:</b> Allow heated components to cool before touching or servicing the instrument.
	<b>Risk of electric shock:</b> This instrument uses voltages that can cause electric shock and/or personal injury. Before servicing, shut down the instrument and disconnect it from line power. While operating the instrument, keep covers on.
	<b>Risk of eye injury:</b> Eye injury could occur from splattered chemicals or airborne particles. Wear safety glasses when handling chemicals or servicing the instrument.

## Safety Precautions

Observe the following safety precautions when you operate or perform service on the mass spectrometer.



**CAUTION Make sure you have sufficient nitrogen for the API source and reagent inlet.** Before beginning normal operation each day, make sure that you have sufficient nitrogen for your API source and reagent inlet. The presence of atmospheric oxygen in the API source and reagent inlet could be unsafe when the mass spectrometer and ETD module are turned on. (The MS/ETD system automatically turns the mass spectrometer off when you run out of nitrogen; however, it is best to take this added precaution.)



**CAUTION Place the MS/ETD system in Standby (or Off) mode before you open the atmospheric pressure ionization (API) source.** The presence of atmospheric oxygen in the API source when the mass spectrometer and ETD module are turned on could be unsafe. (The MS/ETD system automatically turns the mass spectrometer and ETD module off when you open the API source; however, it is best to take this added precaution.)





**CAUTION Shut down the mass spectrometer and ETD module and disconnect them from line power before servicing.** High voltages capable of causing personal injury are used in the instrument. Some maintenance procedures require that the mass spectrometer and ETD module be shut down and disconnected from line power before service is performed. Do not operate the mass spectrometer and ETD module with the top or side covers off. Do not remove protective covers from printed circuit boards (PCBs).



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



**CAUTION Respect heated zones.** Treat heated zones with respect. The ion transfer tube, the atmospheric pressure chemical ionization (APCI) vaporizer, and reagent ion source might be very hot and might cause severe burns if touched. Allow heated components to cool to room temperature before you service them.



**CAUTION Use care when changing the vacuum pump oil.** Treat drained vacuum pump oil and pump oil reservoirs with care. Hazardous compounds introduced into the system might have dissolved in the pump oil. Always use approved containers and procedures for disposing of waste oil. Whenever a pump has been operating on a system used for the analysis of toxic, carcinogenic, mutagenic, or corrosive/irritant chemicals, you must decontaminate the pump and certify that it is free of contamination before repairs or adjustments are made by a Thermo Fisher Scientific field service engineer or before it is sent back to the factory for service.



**CAUTION Provide an adequate fume exhaust system and contain waste streams.**

The API source can accommodate high flow rates. Therefore, you must make provisions to collect the waste solvent. Connect the API source housing drain to the solvent waste container provided with the instrument, and vent the waste container to a fume exhaust system. For more information, refer to Chapter 3, “Ion Max and Ion Max-S Ion Sources,” in the *LTQ Series Getting Connected Guide*.






You must provide an adequate fume exhaust system. Samples and solvents that are introduced into the ETD module are eventually exhausted from the forepump. Therefore, connect the forepump to a fume exhaust system. Consult local regulations for the proper method of exhausting the fumes from your system.



Equip your lab 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 forepumps connect to the same fume exhaust system. Route the exhaust tubing from the forepumps to a dedicated fume exhaust system.
- Do not vent the PVC drain tube (or any vent tubing connected to the waste container) to the same fume exhaust system that the forepumps connect to. Vent the waste container to a dedicated fume exhaust system. The exhaust system for the Ion Max™ or Ion Max-S™ API source must accommodate a flow rate of up to 30 L/min (64 ft<sup>3</sup>/h).

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## Introduction

The Velos Pro/ETD and LTQ XL/ETD (see [Figure 1](#)) systems are members of the Thermo Scientific family of mass spectrometers (MSs). The Velos Pro/ETD and LTQ XL/ETD systems are advanced analytical instruments that include a syringe pump, a divert/inject valve, a mass spectrometer, the ETD module, and the Xcalibur data system.

**Figure 1.** LTQ XL MS with the ETD module and NSI source



In liquid chromatography/mass spectroscopy (LC/MS) analysis, you first inject a sample into an LC column. In the LC column, the sample separates into its components. These components elute from the LC column and pass sequentially into the mass spectrometer for analysis.

In the case of direct infusion or flow injection, no LC is performed. The sample enters the mass spectrometer without being separated into its components.

With the ETD module, you can analyze the sample (direct infusion/flow injection) or its components (LC/MS) by electron transfer dissociation (ETD). The data system stores and processes the data from the mass spectrometer.

The Velos Pro/ETD and LTQ XL/ETD systems consist of an API source, ion optics, a mass analyzer, an ion detection system, and a reagent ion source (ETD module). A vacuum manifold encloses the ion optics, mass analyzer, ion detection system, reagent ion source, and part of the API source. Ionization of the analyte occurs in the API source. The specific method used to ionize the analyte is called the ionization technique. The ion optics transmit the analyte ions produced in the API source into the mass analyzer, where they are trapped in stable orbits by a time-varying electric field. The polarity of the potentials applied to the API source and ion optics determines whether positively charged analyte ions or negatively charged analyte ions transmit to the mass analyzer. You can configure the MS/ETD system to analyze positively or negatively charged ions (called the positive or negative ion polarity mode).

Negatively charged reagent ions (fluoranthene radical anions) from the reagent ion source (ETD module) can also fragment protein or peptide analyte ions in the mass analyzer. These negatively charged ions transfer electrons to protein or peptide analyte cations and cause them to dissociate and produce C and Z type product ions (versus the Y and B type product ions produced by collision-induced dissociation [CID]). The resulting analyte product ions provide another way of analyzing these molecules as compared to CID and pulsed Q collision-induced dissociation (PQD).

The lenses in the API source and ion optics act as a gate to start and stop the transmission of ions from the API source to the mass analyzer. The Automatic Gain Control™ (AGC) feature controls the function of these lenses by setting them to transmit the optimum number of ions to the mass analyzer.

The mass analyzer measures the mass-to-charge ratios ( $m/z$ ) of the ions produced in the API source. Selected ions, ejected from the mass analyzer, reach the ion detection system where they produce a signal. The signal produced is then amplified by the detection system electronics.

The Xcalibur data system analyzes the ion detection system signal and serves as the user interface to the mass spectrometer, autosampler, and LC. For information about the data system processing and instrument control application, refer to the Xcalibur application Help.

Each sequence of injecting the mass analyzer with ions followed by mass analysis of the ions is called a scan. The MS/ETD system can use several different scan modes, scan types, and dissociation techniques to inject, dissociate, and eject ions from the mass analyzer. The ability of this instrumentation to vary the scan mode, scan type, and dissociation technique, as well as the ionization and ion polarity modes, provides exceptional flexibility to solve complex analytical problems.

# Functional Description

This chapter describes the principal components of the ETD module and their functions.

**Note** For the LTQ Velos/ETD system, follow the Velos Pro/ETD system information, unless otherwise noted.

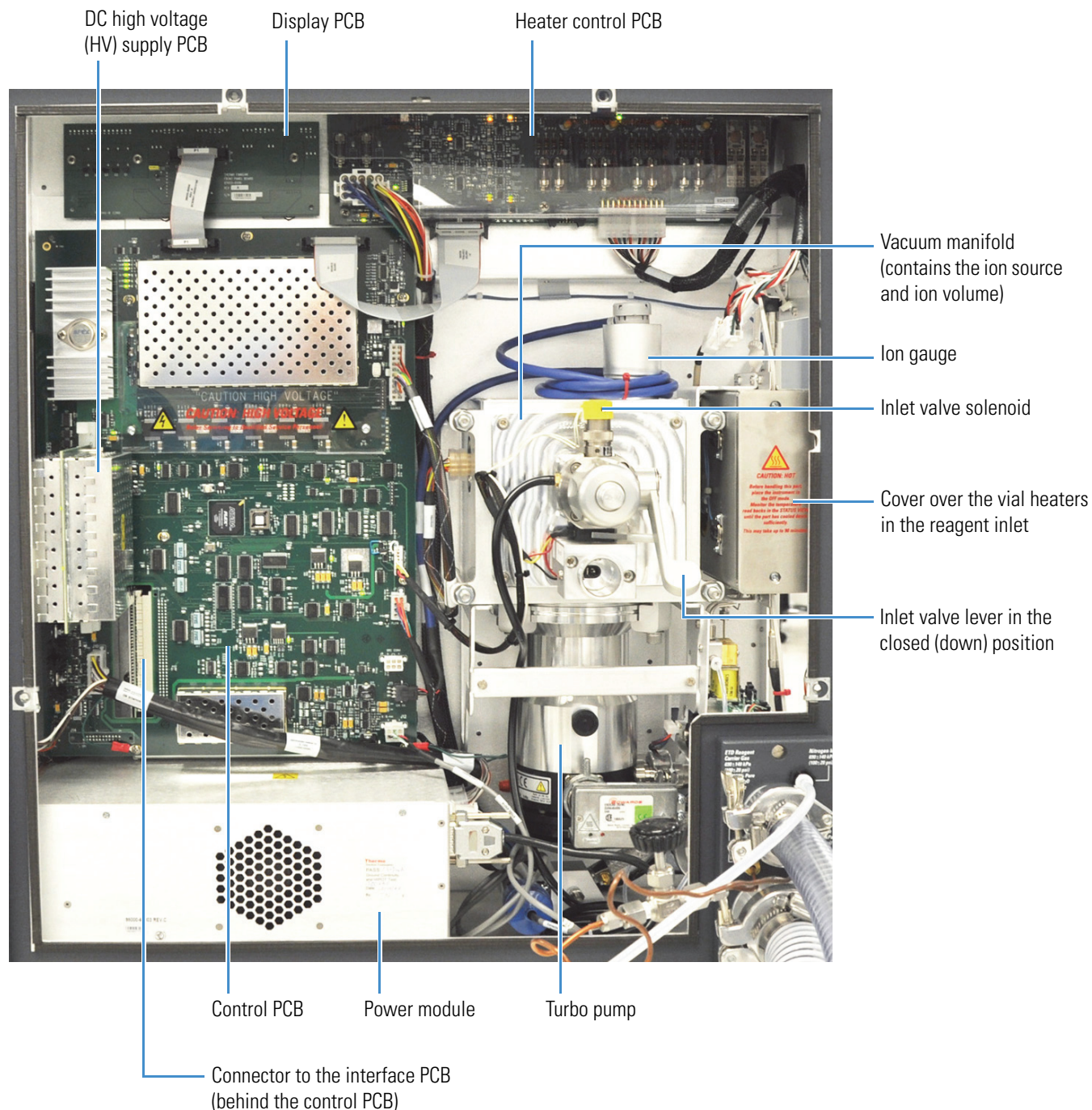
## Contents

- Power Panel
- Interface PCB
- ETD Control PCB
- Heater Control PCB
- LEDs
- Flow Control
- Reagent Heaters
- Ion Source

## 2 Functional Description

Figure 2 shows the inside of the ETD module, and the following sections describe the ETD module's major components.

**Figure 2.** Internal back view of the ETD module

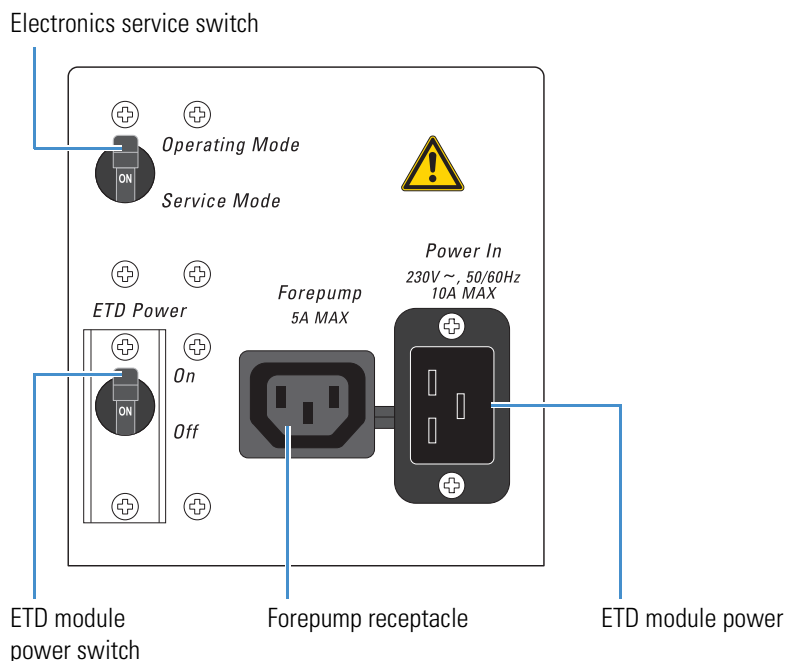




# Power Panel

Figure 3 shows the ETD module's right-side power panel.

**Figure 3.** ETD module power panel



The power panel for the ETD module contains a power switch (circuit breaker) and an electronics service switch. During normal operation, the ETD Power switch is in the On (up) position and the electronics service switch is in the Operating Mode (up) position. As a safety feature, both components of the MS/ETD system (the mass spectrometer and the ETD module) shut down with one set of switches—the mass spectrometer switches. Before performing maintenance on the ETD module as described in [Chapter 5, “Maintenance,”](#) you set the mass spectrometer’s electronics service switch to the Service Mode (down) position. The electronics service switch turns the power on or off to all ETD module components, except the turbo pump and forepump.

The ETD module power panel also contains a Power In receptacle that connects the module to line power and a Forepump receptacle to power the ETD module’s forepump. For more information about connecting the MS/ETD system, refer to the *LTQ Series Getting Connected Guide*.

## Interface PCB

The interface printed circuit board (PCB) provides an electronic connection between the ETD module and the mass spectrometer. This board also gives the mass spectrometer panel switches control over the mass spectrometer and ETD module.

- The mass spectrometer's power switch turns power on and off to all components in both the mass spectrometer and the ETD module.
- The mass spectrometer's electronics service switch turns power on and off to all mass spectrometer and ETD module components, except to the vacuum pumps connected to the mass spectrometer and the ETD module.

**Note** The ability to control the power from one location—the mass spectrometer's power panel switches—to all components of the MS/ETD system is a safety feature.

## ETD Control PCB

The ETD control PCB controls most of the ETD module functions:

- ETD module operating logic
- Heater temperature and readback logic (for the reagent heaters, transfer line heater, and restrictor oven heater)
- Ion gauge
- Ion source (filament, ion source heater, and lenses)
- Oven cooling gas control
- Convectron™ gauge
- Reagent gas flow

The DC HV supply PCB connects to the ETD control PCB.

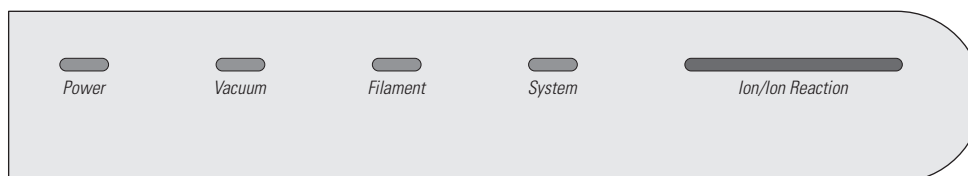
## Heater Control PCB

The heater control PCB contains the power source and temperature sensing circuitry for the four heaters in the reagent inlet, which are the transfer line heater, restrictor oven heater, and two vial heaters. The heater control PCB sends temperature information to the heater temperature and readback logic on the ETD control PCB. The heater temperature and readback logic control how the heater control PCB applies power to the ETD module heaters.

## LEDs

Figure 4 shows the ETD module status LEDs, which are described in Table 2. (The status LEDs are not provided for the Orbitrap™/ETD systems.)

**Figure 4.** ETD module status LEDs for the Velos Pro/ETD and LTQ XL/ETD systems



**Table 2.** ETD module status LED descriptions (Sheet 1 of 2)

LED	State	Description
Power	Green	The ETD module is receiving power.
	Off	The ETD module is not receiving power.
Vacuum	Green	The vacuum pressure is within the allowable operating range.
	Yellow	A vacuum sensor detects a fault and the ETD module has gone into standby mode.
	Off	High voltage is not being applied to the ETD module.
Filament	Green	The filament is on.
	Yellow	There is a filament fault, such as a burnt out filament.
	Off	The filament is off.
System	Flashing yellow	All of the electronics are on and the restrictor, transfer line, and ion source heaters are on but not at their target temperatures. The vial heaters are off.
	Solid yellow	The restrictor, transfer line, and ion source heaters have reached their target temperatures. The vial heaters remain off.
	Flashing green	The vial heaters are on but not at their target temperatures. The restrictor, transfer line, and ion source heaters are at their target temperatures.
	Solid green	The five heaters (restrictor, transfer line, ion source, and both vial heaters) are at their target temperatures.
	Off	Power is not being supplied to the ETD module.

**Table 2.** ETD module status LED descriptions (Sheet 2 of 2)

LED	State	Description
Ion/Ion Reaction	Blue, sequentially lighting up	An ETD activation (or ion/ion reaction) is being used during a scan.
	On	A scan has stopped. This is not a fault.
	Off	No scan is occurring.

For information about the mass spectrometer LEDs, refer to Chapter 2, “Functional Description,” in the *LTQ Series Hardware Manual*.

## Flow Control

The ETD module contains an electronic pressure regulator for flow control of the chemical ionization (CI) gas/reagent carrier gas. The ETD control PCB provides the control signal. This gas serves several functions in the ETD module:

- As a carrier gas, the nitrogen sweeps the reagent (fluoranthene) from the vial to the ion source where the reagent radical anions form.
- As a CI vehicle, the nitrogen undergoes collisions with 70 eV electrons from the filament in the ion volume. These 70 eV electrons knock electrons off of the nitrogen molecules (creating nitrogen ions). The secondary electrons resulting from these collisions have near thermal-kinetic energies. The fluoranthene captures these thermal electrons to form reagent radical anions that react with the analyte.

## Reagent Heaters

The reagent inlet contains the reagent heaters that heat the reagent (fluoranthene) to obtain a sufficient amount of reagent vapor in the carrier gas. The heater control PCB powers the reagent heaters. The ETD control PCB, in turn, controls the heater control PCB.

When the reagent ion source goes into standby mode, the two reagent heaters and the filament turn off. An audible rush (hissing noise) from the reagent ion source area in the back of the ETD module confirms the flow of the nitrogen cooling gas (if enabled).

**Note** When the reagent ion source is off or in standby mode, cooling gas (nitrogen) turns on. This is normal operation. For information about the cooling gas, refer to “Specifying the Reagent Ion Source for the Mass Spectrometer” in Chapter 2 of the *ETD Module Getting Started Guide*.



**CAUTION Hot surface.** When the reagent ion source is off, the restrictor oven, transfer line, and ion source remain at 160 °C (320 °F).

If enabled in Thermo Foundation™ Instrument Configuration, the cooling gas (nitrogen) turns off when the reagent heaters reach 70 °C (158 °F). To replace the reagent vials, follow the procedure “[Reagent Vials Maintenance](#).”

**Note** The rushing or hissing noise of the nitrogen coming from the back of the ETD module stops when the cooling gas (nitrogen) turns off.



**CAUTION Hot surface.** Do not attempt to handle the vials or vial holders when the cooling nitrogen turns off at 70 °C (158 °F). They are still too hot to handle. To replace the reagent vials, follow the procedure “[Reagent Vials Maintenance](#).”

When starting at room temperature, the reagent heaters and vials take at least 20 minutes to stabilize at the configured vial operating temperature (Thermo Fisher Scientific recommends the 90 °C setting). The ion source receives the reagent only after the temperatures stabilize. For information about setting the vial temperature, refer to Chapter 2, “Setting Up the Xcalibur Instrument Configuration,” in the *ETD Module Getting Started Guide*.

## Ion Source

The reagent molecules are converted to reagent ions in the ion source. [Figure 32](#) and [Figure 33](#) show the ion source assembly, which contains the filament, reagent ion volume, ion source heater, and so on. The filament is the source of 70 eV electrons that collide with the nitrogen molecules to form nitrogen cations and near-thermal-energy electrons, which the reagent molecules capture to yield reagent anions. The reagent ion volume is the space where this reaction takes place. The ETD Control PCB controls the ion source heater.

## 2 Functional Description

Ion Source

## Shutdown and Startup

As a safety feature, both components of the MS/ETD system (the mass spectrometer and the ETD module) shut down with one set of switches—the mass spectrometer switches. This safety feature is one of the functions of the ETD module interface board (see [page 6](#)).

For a description of the ETD module's power panel, see [page 5](#). For information about the cooling gas, refer to step 3 on page 11 of the *ETD Module Getting Started Guide*.

### Note

- If you are not going to use the system or ETD module for a short period of time, such as overnight or through the weekend, place the system in standby mode as described in [“Placing the ETD Module in Standby Mode.”](#)
- If you are not going to use the system for an extended period of time or if it must be shut down for maintenance or service, follow the procedure [“Shutting Down the MS/ETD System.”](#)

Refer to Chapter 3 in the *LTQ Series Hardware Manual* for the following:

- The mass spectrometer's shutdown and startup procedures
- A table showing the On/Off status of mass spectrometer components when the mass spectrometer is in standby or off mode

### Contents

- [Shutting Down the ETD Module in an Emergency](#)
- [Opening the Tune Plus Window](#)
- [Placing the ETD Module in Standby Mode](#)
- [Shutting Down the MS/ETD System](#)
- [Starting Up the MS/ETD System](#)

## Shutting Down the ETD Module in an Emergency

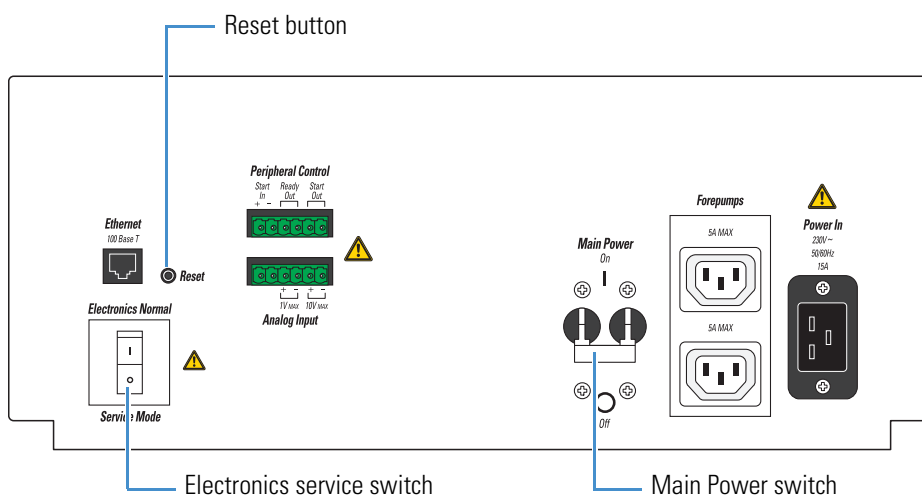


**CAUTION** If you must turn off the ETD module in an emergency, **turn off the mass spectrometer’s Main Power switch** located on its right-side power panel (Figure 5). This switch turns off all power to the mass spectrometer and the ETD module, including the forepumps, without harming components within the system. However, do not use this method as part of the standard shutdown procedure. Instead, refer to “Shutting Down the Mass Spectrometer Completely” in Chapter 3 of the *LTQ Series Hardware Manual*.

If you turn off the mass spectrometer’s Main Power switch and the ETD module remains on, turn off the ETD module’s main ETD Power switch located on its right-side power panel. Contact your local Thermo Fisher Scientific field service engineer regarding the connection malfunction.

To turn off the LC, autosampler, and data system computer in an emergency, use their respective on/off switch or button.

**Figure 5.** Power panel for the Velos Pro and LTQ XL mass spectrometers





## Opening the Tune Plus Window

There are several ways to open the Tune Plus window (Figure 6).

### ❖ To open the Tune Plus window

Do one of the following:

- On the Windows taskbar, choose **Start > All Programs > Thermo Instruments > LTQ > *model* Tune**, where *model* is your specific MS/ETD system.

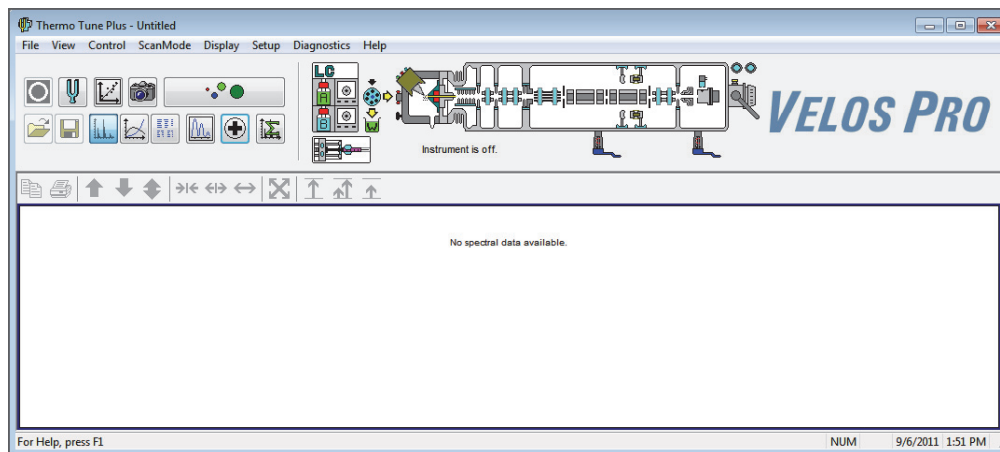
**Note** For LTQ Series version 2.5.0 or earlier, choose **Start > All Programs > Xcalibur > *model* Tune**.

- In the Xcalibur application, click the **Roadmap View** icon (under the menu bar), the **Instrument Setup** icon, the *model* MS icon, and then **Tune Plus**.

—or—

- In the Xcalibur application, click the **Roadmap View** icon (under the menu bar), the **Instrument Setup** icon, and the *model* MS icon. Then, from the main toolbar choose *model* > **Start Tune Plus**.

**Figure 6.** Tune Plus window for the Velos Pro/ETD system



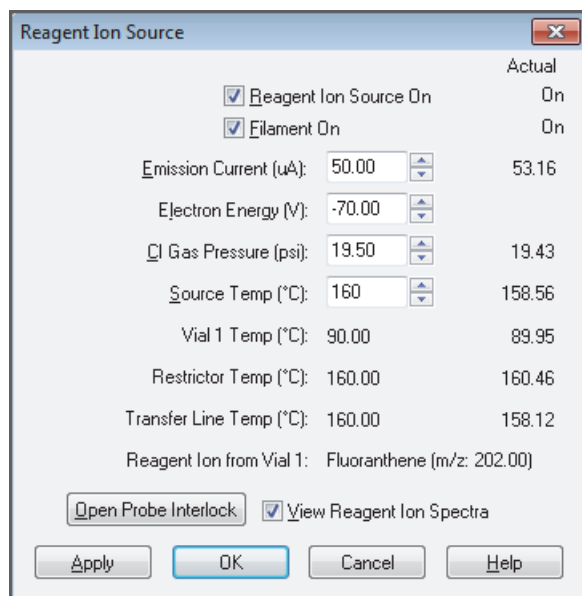
## Placing the ETD Module in Standby Mode

If you are not going to use the ETD module for a short period of time (see [page 11](#)), place the ETD module in standby mode.

### ❖ To place the ETD module in Standby mode

1. Complete all data acquisition, if any.
2. Open the Tune Plus window (see [page 13](#)).
3. Choose **Setup > Reagent Ion Source** to open the Reagent Ion Source dialog box ([Figure 7](#)).

**Figure 7.** Reagent Ion Source dialog box



4. Clear the **Filament On** check box.

When you clear this check box, the filament turns off and the ETD module's Filament LED turns off. The System LED switches from either solid green or flashing green to solid yellow. This change signifies that the vial heaters are off and that the restrictor, transfer line, and ion source heaters are on and at their target temperatures.

5. (Optional) If you are not going to use the ETD module for several days or more, clear the **Reagent Ion Source On** check box.

When you clear this check box, the reagent vial heaters turn off. If you selected the Use Cooling Gas check box for the reagent ion source configuration, the cooling gas turns on and stays on until the reagent vial heaters reach 70 °C. For information about the cooling gas option, refer to "Specifying the Reagent Ion Source for the Mass Spectrometer," in Chapter 2 of the *ETD Module Getting Started Guide*.

**Tip** If you place the MS/ETD system in standby mode, instead of only the ETD module, the reagent vial heaters turn off followed by a 1 hour delay until the cooling gas turns on (if enabled).



**CAUTION Hot surface.**

- Install or exchange the reagent vials by following the procedure “[Reagent Vials Maintenance](#).” Note that the reagent vials are too hot to touch when the cooling nitrogen turns off at 70 °C (158 °F). Verify that the reagent vials are cool to the touch before handling them.
- The restrictor, transfer line, and ion source heaters operate at 160 °C (320 °F). Do not attempt to touch them unless you have shut down the MS/ETD system ([Shutting Down the MS/ETD System](#)) and these heaters have cooled to room temperature.

For more information about the ETD module LEDs, see “[LEDs](#).” For information about the Filament On check box, refer to the Tune Plus application Help. For more information about turning the reagent heaters on and off, see “[Reagent Heaters](#).”

## Shutting Down the MS/ETD System

The mass spectrometer’s power switches control the power to the MS/ETD system (ETD module and mass spectrometer). The ETD module’s power switches control only the ETD module. When the system is fully operational (all systems on), the mass spectrometer’s Main Power switch is in the On position and its electronics service switch is in the Electronics Normal position.

Normally the ETD module’s power and electronics service switches are not used and remain on. To place the MS/ETD system in service mode, use the mass spectrometer’s electronics service switch. Turn the MS/ETD system on and off with the mass spectrometer’s power switch.

**IMPORTANT** If you accidentally turn off the ETD module’s electronics service switch, turn the ETD module switch back on, and then turn off and on the mass spectrometer’s electronics service switch (not the power switch).



**CAUTION Hot surface.** When the MS/ETD system is on, the flow restrictor, transfer line, and ion source heaters can reach 160 °C (320 °F). The vial heaters can be at 108 or 90 °C, depending on the reagent ion source configuration. (Thermo Fisher Scientific recommends the 90 °C setting.) Do not attempt to replace the reagent vials or service heated components until they have cooled to room temperature, which can take an hour or more.

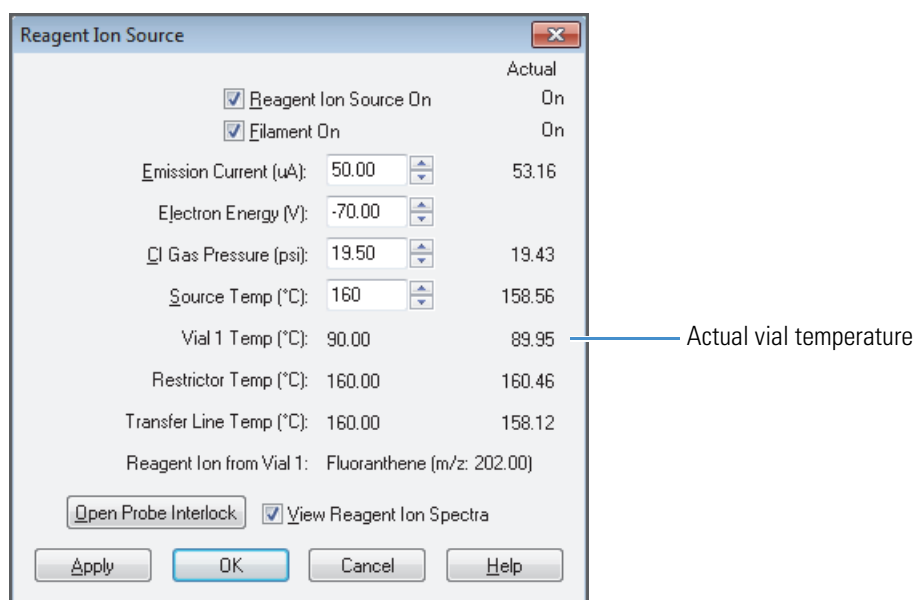
❖ **To shut down the MS/ETD system**

1. Complete all data acquisition, if any.
2. Turn off the flow of sample solution from the LC (or other sample introduction device).

**Note** For instructions about operating the LC from the front panel, refer to the LC manual.

3. Open the Tune Plus window (see [page 13](#)).
4. Choose **Setup > Reagent Ion Source** to open the Reagent Ion Source dialog box ([Figure 8](#)).

**Figure 8.** Reagent Ion Source dialog box



5. Clear the **Reagent Ion Source On** check box.
6. Observe the vial temperature in the Actual column on the right.  
If enabled, nitrogen cooling gas flows until the vial cools to 70 °C.
7. Click **OK**.

8. Choose **Control > Off** to turn off the MS/ETD system.



#### CAUTION

- Turning off the mass spectrometer also turns off the ion source sheath gas, auxiliary gas, high voltage, and all of the ETD module heaters. You must choose **Control > Off** to turn off all of the ETD module heaters.
- The ability to control the service mode for both the mass spectrometer and the ETD module at one location (at the mass spectrometer's electronics service switch) is a safety feature. Therefore, do not toggle the ETD module electronics service switch to the Service Mode position while the mass spectrometer switches are in their On (up) positions. This could cause communication problems between the mass spectrometer and the ETD module.

You can determine the status of the ETD module from its status LEDs on the front panel; see “LEDs.”

9. Turn off the mass spectrometer's Main Power switch.

## Starting Up the MS/ETD System

If you followed the procedures in the *LTQ Series Hardware Manual* to shut down the system completely, you must turn the LC and data systems back on before turning on the MS/ETD system, which does not operate until it receives instructions from the data system.

#### ❖ To start the MS/ETD system

1. Turn on the flows for the helium and nitrogen gases at their tanks, if they are off.
2. Turn on the mass spectrometer's Main Power switch.
3. Wait at least 1 hour to allow the mass spectrometer to pump down.
4. Make sure that the mass spectrometer's electronics service switch is in the Electronics Normal position.

The system starts up and goes into standby mode. The ion source heater, flow restrictor, and transfer line heaters start heating.

The ETD module's status LEDs indicate the MS/ETD system states as it powers up:

- The MS/ETD system's Power LED is solid green when the MS/ETD system power is on.
- The Vacuum LED is solid green when the MS/ETD system vacuum pressure is in the proper range as measured by both the ion and Convectron gauges.
- The Filament LED is off when the filament is off.

The ETD module's system LED changes to solid yellow when the restrictor, transfer line, and ion source heaters reach their target temperatures and the vial heaters are off.

The carrier/CI gas turns on.

The mass spectrometer LED flashes yellow when all of the following occur:

- The MS/ETD system electronics are on.
- The restrictor, transfer line, and ion source heaters are on but not at their target temperatures.
- The vial heaters are off.

For more information about the ETD module's status LEDs, see [“LEDs.”](#)

## Daily Operation

To ensure the proper operation of the ETD module, Thermo Fisher Scientific recommends that you perform daily preventive maintenance. This chapter specifies the items to check before operating the system and the cleaning procedures to perform after completing the analyses.

**Note** You do not need to tune and calibrate the ETD module as part of your daily routine. However, ensure that you perform ETD-related calibration at least every three months (on the Semi-Automatic page of the Calibrate dialog box, Electron Multiplier Gain for the negative ion mode and Reagent Ion Selection). For information about tuning and calibrating the ETD module, refer to the *ETD Module Getting Started Guide*.

**IMPORTANT** For LTQ version 2.7 or later, the Tune Plus application saves the ETD parameters (excluding the ETD reaction time) in an ETD-dedicated system file. This means you can switch to another ion source probe type without losing the current ETD parameters. In LTQ version 2.6 SP3 or earlier, the ETD parameters were saved in the tune file associated with a specific ion source probe.

### Contents

- [Before Operating the MS/ETD System](#)
- [Turning On the Reagent Ion Source](#)
- [After Operating the MS/ETD System](#)

## Before Operating the MS/ETD System

Follow these preventive maintenance procedures every day before beginning the first analysis.

**Note** Before performing these procedures for the MS/ETD system, perform the corresponding procedures for the mass spectrometer as described in Chapter 4, “Daily Operation,” in the *LTQ Series Hardware Manual*.

- [Checking the System Mode](#)
- [Checking the Vacuum Pressure Levels](#)
- [Checking the Gas Supplies](#)

### Checking the System Mode

Before beginning your daily work, turn on the mass spectrometer.

❖ **To turn on the mass spectrometer**

1. Open the Tune Plus window (see [page 13](#)).
2. Click the **On/Standby** button to select the **On** mode.



On



Standby



### Note

When on, the System LED on the mass spectrometer's front panel turns green and the high voltage to the electron multipliers turns on. (The status LEDs are not provided for the Orbitrap/ETD systems.)

The following conditions can cause the MS/ETD system to remain in standby mode even though you try to turn it on:

- Attempting to turn on the reagent ion source when the restrictor, source, and transfer line heaters are not at their target temperatures.
- When either the mass spectrometer or the ETD module goes into standby mode. If enabled, the reagent vial nitrogen cooling turns on if the vials are at an elevated temperature.

Exception: If you place the MS/ETD system in standby mode, the cooling nitrogen turns on after a 1-hour delay.

- Whenever the pressure in the mass spectrometer or the ETD module exceeds its protection limit. If enabled, the reagent vial nitrogen cooling turns on if the vials are at an elevated temperature.
- Whenever the flow of reagent ions becomes insufficient as determined by the AGC setting. When this occurs, the MS/ETD system completes the current Xcalibur sequence step before entering standby mode. This prevents the loss of analysis results that might be affected by the reduced reagent ion production.

## Checking the Vacuum Pressure Levels

Before beginning daily operation of the MS/ETD system, check the system's vacuum pressure levels and check for major air leaks. If there is a major air leak, the system does not pump down to sufficient levels to turn on the system.

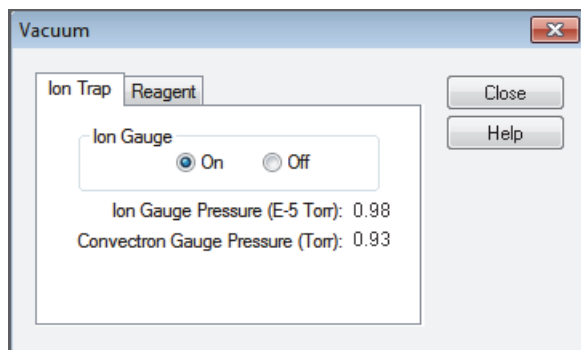


**CAUTION** For proper performance, operate the MS/ETD system at the proper vacuum levels. Operating the system with poor vacuum levels can cause reduced sensitivity, tuning problems, and reduced filament life.

❖ **To check the vacuum pressure**

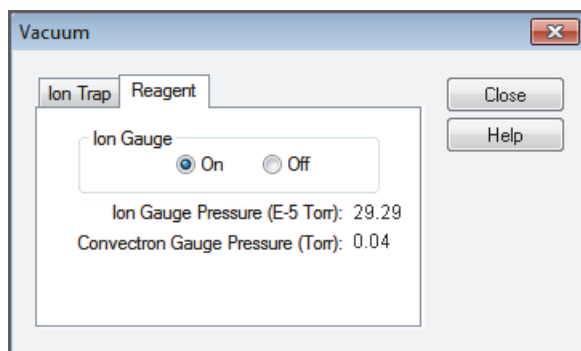
1. In the Tune Plus window, choose **Setup > Vacuum** to open the Vacuum dialog box (Figure 9).

**Figure 9.** Vacuum dialog box



2. Click the **Reagent** tab (Figure 10).

**Figure 10.** Reagent page of the Vacuum dialog box



3. Compare the current values of the pressures in the vacuum manifold with the following gauge values.

Condition	Ion gauge reading (reagent source region)	Convectron gauge reading (reagent foreline)
CI gas pressure set to 20 psi	15–35 × 10 <sup>-5</sup> Torr	0.01–0.05 Torr

**Note**

- If the current pressure values are higher than normal, there might be an air leak.
- If the pressure is above 40 × 10<sup>-5</sup> Torr in the reagent source region and the system restarted within the last 30 to 60 minutes, wait another 30 minutes and recheck the pressure. If the pressure decreases with time, check the pressure periodically until it is within the typical range for the ETD module.
- If the pressure remains high, the system might have an air leak.

❖ **To check the system for major air leaks**

Listen for a rush of air or a hissing sound coming from the instrument.

Possible causes of a major leak might be a loose or disconnected fitting, an improperly positioned O-ring, or an open valve.

❖ **To fix an air leak**

1. Shut down the system. See “[Shutting Down the MS/ETD System.](#)”
2. Make a visual inspection of the vacuum system and vacuum lines for leaks.
3. 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 changed recently or to fittings subjected to heating and cooling.

4. Make sure that the cover plate for the vacuum manifold ([Figure 2](#)) is properly positioned.

**Note** If the air leak persists, contact your local Thermo Fisher Scientific field service engineer for assistance.

## Checking the Gas Supplies

Before beginning daily operation of the MS/ETD system, check the gas supplies. The mass spectrometer uses helium as a damping gas and collision activation partner in the mass analyzer, and nitrogen as the API sheath gas, auxiliary gas, and sweep gas. The ETD module requires an additional ultra-high-purity reagent carrier gas mixture.

❖ **To check the gas supplies**

1. Check that the gas lines connect properly to the back of the ETD module.
2. Check that gases are flowing and that the pressure for each gas is as follows:
  - Helium: 275 ±70 kPa (40 ±10 psi)
  - Nitrogen: 689 ±138 kPa (100 ±20 psi)
  - Reagent carrier gas: 689 ±138 kPa (100 ±20 psi)
3. Make sure that you have sufficient gas to complete your analysis. If necessary, replace the helium, nitrogen, or reagent carrier gas tank.

**Note** Typical daily nitrogen gas consumption (when nitrogen is on 24 hours per day) is 2832 L (100 ft<sup>3</sup>).

For more information about the gas requirements, refer to the *LTQ Series Preinstallation Requirements Guide*.

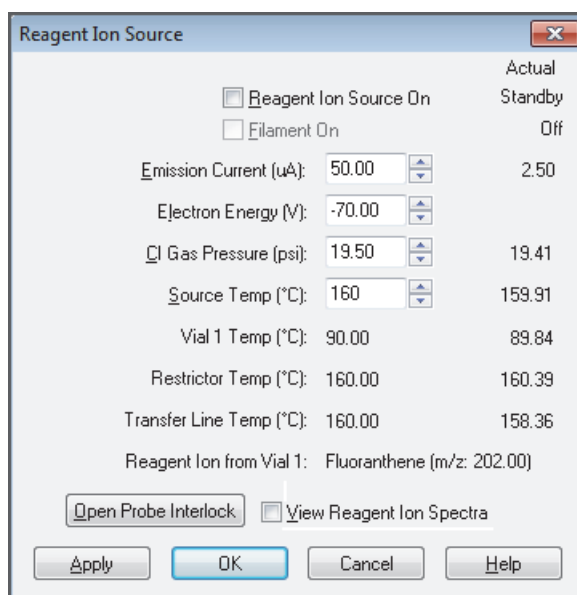
## Turning On the Reagent Ion Source

Even when the ETD module is on, the reagent ion source within it stays off until you turn it on.

### ❖ To turn on the reagent ion source

1. Open the Tune Plus window (see [page 13](#)).
2. Set the reagent ion source as follows:
  - a. Choose **Setup > Reagent Ion Source** to open the Reagent Ion Source dialog box ([Figure 11](#)).

**Figure 11.** Reagent Ion Source dialog box



- b. Select the following check boxes:

- **Reagent Ion Source On**
- **Filament On**
- **View Reagent Ion Spectra**

For tuning, you must manually select the Filament On check box.

- c. Click **OK**.

The reagent ion peaks appear in the Tune Plus spectrum view ([Figure 12](#)).

**Note**

If the reagent vials are not at their target temperature, a message appears:

Reagent Vial NOT At Temperature! Please wait ...

The System LED on the ETD module flashes green to indicate that the reagent vial heaters are turned on but not at their target temperatures (the other heaters are at their target temperatures).

When the reagent vial heaters reach their target temperatures, which can take 20 minutes or more, the System LED illuminates solid green.

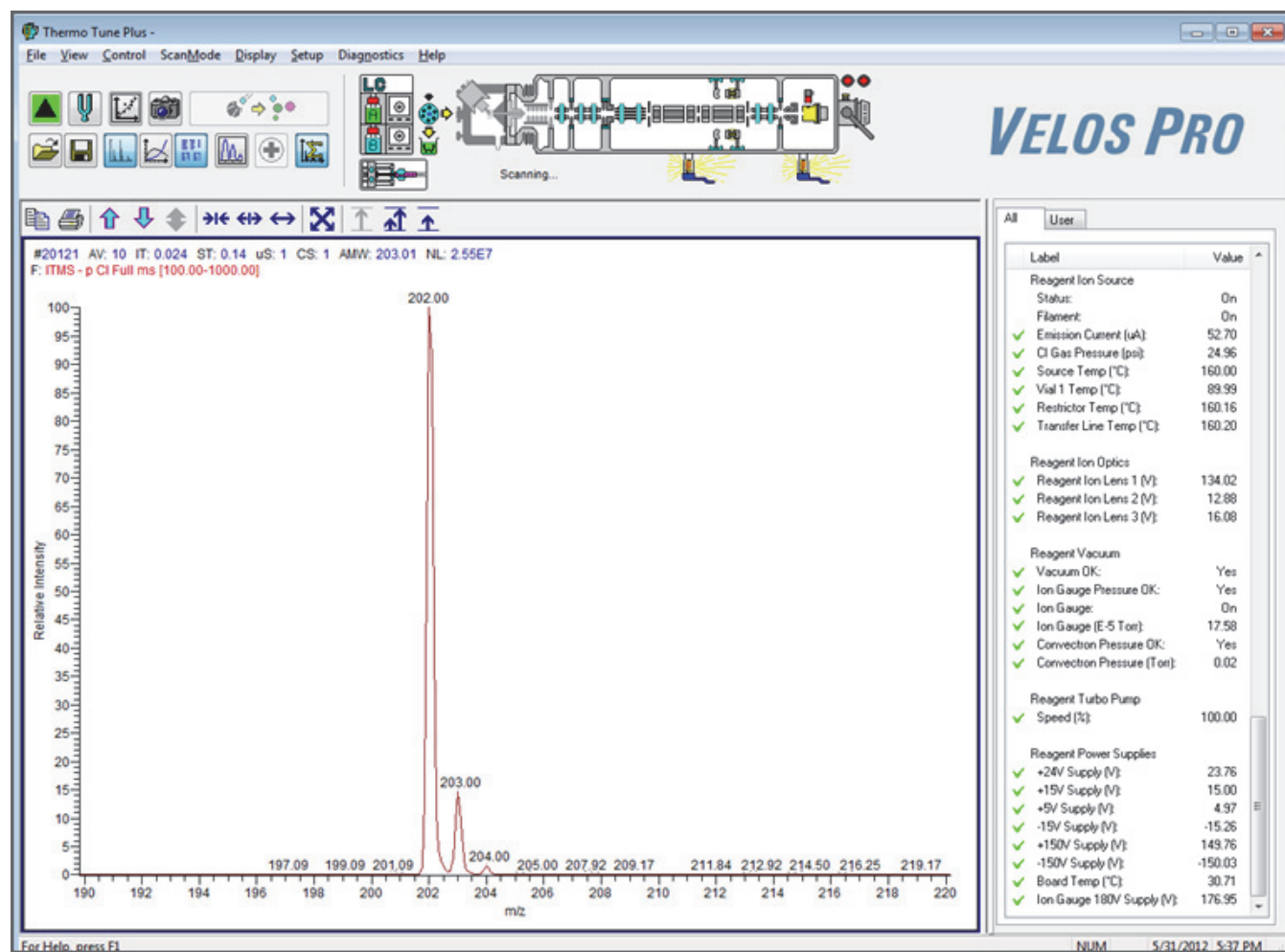
When the reagent vials reach their target temperature, voltage is applied to the ETD module ion optics, and when the check box is selected, the filament turns on and the Filament LED illuminates solid green.

(The status LEDs are not provided for the Orbitrap/ETD systems.)

## 4 Daily Operation

### Turning On the Reagent Ion Source

**Figure 12.** Mass spectrum for the reagent ion displayed in Tune Plus for the Velos Pro/ETD system



3. Check the mass spectrum and do one of the following:
  - If  $m/z$  202 has satisfactory abundance and the reagent ion isolation is suppressing the background anions (for example,  $m/z$  216, and so on), proceed to [step 4](#).
  - If the intensity is not satisfactory, do the following:
    - Make sure that you have a recent calibration of the Electron Multiplier Gain for the negative ion mode, which is on the Semi-Automatic page of the Calibrate dialog box. If the calibration is old, check the calibration status on the Check page, and then recalibrate if necessary. For information, refer to the *LTQ Series Getting Started Guide*.
    - Tune the reagent ion optics as described in Chapter 3, “Tuning,” in the *ETD Module Getting Started Guide*.
  - If the reagent ion isolation is not suppressing the background anions, perform the Reagent Ion Selection calibration on the Semi-Automatic page of the Calibrate dialog box, Electron Multiplier Gain for the negative ion mode.
4. In the Reagent Ion Source dialog box, clear the **View Reagent Ion Spectra** check box.  
This action clears the Tune Plus spectrum view from the ETD module.

## After Operating the MS/ETD System

Follow these preventive maintenance procedures every day after completing your daily work:

- [Placing the MS/ETD System in Standby Mode](#)
- [Purging the Oil in the Forepump](#)

### Placing the MS/ETD System in Standby Mode

#### ❖ To place the MS/ETD system in standby mode

1. Follow the procedure “[Placing the ETD Module in Standby Mode](#).”
2. In the Tune Plus window, click the **On/Standby** button to select the **Standby** mode.



If the reagent ion source is on when you place the mass spectrometer in standby mode, the filament turns off immediately but the vial heaters stay on for 60 minutes before they turn off and the cooling gas begins. Therefore, you can perform quick, minor maintenance procedures on the MS/ETD system without cooling the reagent vials.

## **4 Daily Operation**

After Operating the MS/ETD System

### **Purging the Oil in the Forepump**

Purge (decontaminate) the oil in the forepump daily to remove water and other dissolved chemicals, which can cause corrosion and decrease the lifetime of the forepump. The best time to purge the oil is at the end of the working day.

For instructions, refer to the forepump's documentation. Remember to close the purge valve before continuing normal operation.



# Maintenance

Perform maintenance on the ETD module on an as-needed basis or when the ion signal deteriorates.

## Contents

- [Maintenance Schedule](#)
- [Work Area Preparations](#)
- [Guidelines](#)
- [Tools and Supplies](#)
- [Cleaning Parts](#)
- [Ion Volume Maintenance](#)
- [Inlet Valve Maintenance](#)
- [Removing the Access Panels](#)
- [Ion Source Maintenance](#)
- [Reagent Vials Maintenance](#)
- [Forepump Maintenance](#)
- [Fan Filter Maintenance](#)

## Maintenance Schedule

Over time, the ion signal from the ETD module decreases as the internal components become dirty and therefore need to be cleaned. Before performing any procedure in this chapter, run the diagnostics as described in [Chapter 6, “Diagnostics,”](#) to determine if there are any problems with the major electronic circuit boards.

The first and easiest procedure to perform is to clean or replace the ion volume as described in “[Ion Volume Maintenance](#).” You can perform this procedure without shutting down and venting the system. If the performance of the ETD module does not improve, you must shut down and vent the system to perform the procedures on the ion source components and reagent vials.

How often you clean the reagent ion source depends on the amount of time that the source is operating. In general, the closer a component is to where the reagent ions are generated, the more quickly it becomes dirty. For example, you must clean the ion volume and ion volume holder more often than the lenses. Therefore, you typically need to clean the ion source block with every third (or less) lens cleaning.

[Table 3](#) lists the maintenance procedures and their recommended frequency.

**Table 3.** ETD module maintenance procedures and frequency

Component	Procedure	Recommended frequency	Location
Cooling fans	Clean the fan filter.	Every 4 months	<a href="#">page 74</a>
Forepump	Purge (decontaminate) the oil and check for leaks.	Daily	<a href="#">page 73</a>
	Change the oil.	Every 3 months, or if the oil is cloudy or discolored	See also the manufacturer’s documentation.
	Add oil.	As needed, based on the oil level	
Inlet valve components	Replace the inlet valve components.	As the components wear out, which could be after several years	<a href="#">page 47</a>
Ion source block	Clean the ion source block.	Every 6 months, depending on the usage	<a href="#">page 66</a>
Ion source filament	Inspect or replace the filament.	As needed, depending on analytical conditions, source usage, and reagent ion abundance	<a href="#">page 53</a>
Ion source lenses	Clean the ion source lenses.	As needed, depending on usage	<a href="#">page 64</a>
Ion volume	Clean or replace the ion volume.	As needed, depending on usage	<a href="#">page 37</a>
Reagent vials	Replace the reagent vials.	Annually to eliminate the possibility that the septum on the vials might start to leak with age	<a href="#">page 68</a>

For instructions about maintaining the mass spectrometer, refer to the *LTQ Series Hardware Manual*. For instructions about maintaining the LC modules, refer to that instrument’s manual.

## Work Area Preparations

### ❖ To prepare the work area

Do the following:

- Cover the area with clean, lint-free paper or a large sheet of clean aluminum foil.
- Have nearby the necessary tools, supplies, and replacement parts (when applicable).

## Guidelines

For optimal results, follow these guidelines when performing the procedures in this chapter.

- Proceed methodically.
- Always wear protective eye wear when cleaning parts or changing the reagent vials.
- Always wear a new pair of lint- and powder-free gloves when handling the internal source components or clean parts, or when changing the reagent vials. Never reuse gloves after you remove them because the surface contaminants on them recontaminate clean parts.
- Always place the components on a clean, lint-free work surface.
- Never overtighten a screw or use excessive force.

## Tools and Supplies

You can remove and disassemble many parts by hand. Make sure you have all the necessary tools before starting a procedure. [Table 4](#) lists the tools, equipment, and chemicals you need to provide for maintenance of the reagent ion source. Have a set of tools to use exclusively for maintaining the reagent ion source and that are free of grease and other residues.



**CAUTION** Make sure to maintain the cleanliness of the parts when you handle them. If you are not returning the parts directly to the instrument after cleaning, place them on clean, unused aluminum foil.

**Table 4.** Tools, equipment, and chemicals (Sheet 1 of 3)

Description	Part number
<b>Tools</b>	
Forceps <sup>a</sup>	76360-0400
Hex or hex ball drivers: 2 mm, 2.5 mm, 3 mm, 4 mm, 1/16 in., 5/64 in., 7/64 in., 9/64 in., and 5/32 in.	–
Ion volume insertion and removal tool <sup>b</sup>	98000-60028

**Table 4.** Tools, equipment, and chemicals (Sheet 2 of 3)

Description	Part number
Pliers	–
Screwdriver, Phillips, #2	–
Screwdriver, slotted, small	–
Tweezers, large	–
Wrench, adjustable	–
Wrench, socket, 1/2 in.	–
Wrenches, open-ended: 1/4 in., 5/16 in., 7/16 in., 1/2 in., and 9/16 in.	–
(Optional, recommended) Dremel™ rotary tool or equivalent, cordless	–
<b>Equipment</b>	
Aluminum foil, heavy gauge <sup>c</sup>	Fisher Scientific 01-213-104
Aluminum oxide abrasive, number 600 <sup>a</sup>	32000-60340
Beakers, glass: 100 mL and 500 mL	–
Cotton-tipped applicators	Fisher Scientific A030102000
Gloves, lint-free and powder-free	Fisher Scientific 19-120-2947 <sup>d</sup>
	<b>Unity Lab Services:</b> <ul style="list-style-type: none"> <li>• 23827-0008 (size medium)</li> <li>• 23827-0009 (size large)</li> </ul>
Lamp, small, high-intensity	–
Protective eye wear	–
Sonicator	–
(Optional) Oven	–
<b>Chemicals</b>	
Acetone (or other suitable polar solvent)	
HPLC grade	Fisher Scientific A949
GC Resolv	Fisher Scientific A928-4 (amber glass, 4L)
Clean, dry gas (air or nitrogen)	–
Detergent (Alconox™, Liquinox™, Micro-90™, or equivalent)	–
Glycerol, reagent grade	–

**Table 4.** Tools, equipment, and chemicals (Sheet 3 of 3)

Description	Part number
Water, deionized	—
Water, tap	—

<sup>a</sup> Provided in the ETD Accessory Kit.

<sup>b</sup> Provided with your ETD module order.

<sup>c</sup> Rinse each sheet with acetone before use.

<sup>d</sup> Multiple sizes are available.



### CAUTION Avoid exposure to potentially harmful materials.

By law, producers and suppliers of chemical compounds are required to provide their customers with the most current health and safety information in the form of Material Safety Data Sheets (MSDSs) or Safety Data Sheets (SDSs). The MSDSs and SDSs must be freely available to lab personnel to examine at any time. These data sheets describe the chemicals and summarize information on the hazard and toxicity of specific chemical compounds. They also provide information on the proper handling of compounds, first aid for accidental exposure, and procedures to remedy spills or leaks.

Read the MSDS or SDS for each chemical you use. Store and handle all chemicals in accordance with standard safety procedures. Always wear protective gloves and safety glasses when you use solvents or corrosives. Also, contain waste streams, use proper ventilation, and dispose of all laboratory reagents according to the directions in the MSDS or SDS.

## Cleaning Parts

To clean parts, follow one of these procedures:

- [Cleaning Stainless Steel Parts](#)
- [Cleaning Nonstainless Steel Parts](#)

### IMPORTANT

- Prepare a clean work surface by covering the area with aluminum foil, dull side up.
- Put on a new pair of lint- and powder-free gloves before starting each of these cleaning procedures.

## Cleaning Stainless Steel Parts

The reagent ion source, ion volume assembly, ion source block, and lenses are made from stainless steel. To clean these parts, follow the procedure in this section.



**CAUTION** Use caution, because exposure to liquids can damage some components.

You need the following tools and supplies (see also [Table 4](#)).

Tools	Supplies
Forceps	Acetone, reagent grade (or other suitable polar solvent)
Sonicator	Aluminum foil
(Optional) Dremel rotary tool or equivalent, cordless	Aluminum oxide abrasive, number 600
(Optional) Oven	Clean, dry gas (air or nitrogen)
–	Cotton-tipped applicators
–	Detergent (Alconox, Micro-90, or equivalent)
–	Glass beakers: 100 mL and 500 mL
–	Glycerol, reagent grade
–	Water, deionized
–	Water, tap



**CAUTION** Do not use this procedure to clean ceramic, aluminum, or gold-plated parts. For those types of parts, see [“Cleaning Nonstainless Steel Parts.”](#)

### ❖ To clean stainless steel parts

1. Clean the parts with an aluminum oxide slurry as follows:
  - a. In a 100 mL glass beaker, make a slurry of number 600 aluminum oxide and glycerol.
  - b. Dip a clean, cotton-tipped applicator into the slurry and clean the parts thoroughly.

Contamination often appears as dark or discolored areas, but might not be visible. The heaviest contamination is usually found around apertures, such as the electron entrance hole on the reagent ion source.



**Note** To speed up the cleaning process and make it more efficient, you can use a Dremel tool—be sure to wear protective eye wear when using this tool. Cut the wood handle of a cotton-tipped applicator to 5 cm (2 in.) long and secure it in the chuck of a Dremel tool. Dip the cotton tip into the slurry. Set the Dremel tool to its lowest speed and clean the parts.



**CAUTION** To prevent personal injury, use a cordless Dremel tool or, if you use a corded tool, be sure to keep the tool away from possible hazards, such as standing water or flammable solvents.

- c. Cut the wooden end of a cotton-tipped applicator at an angle to clean the inside corners.
- d. Rinse the parts with clean tap water.
- e. Remove any remaining slurry by using a clean, cotton-tipped applicator.

**Tip** Do not let the slurry dry on the metal because dried aluminum oxide is difficult to remove.

2. Clean the parts in a detergent solution as follows:
  - a. In a 500 mL glass beaker, make a solution of detergent and warm tap water.
  - b. Use forceps to place the parts in the beaker containing the warm detergent solution.
  - c. Place the beaker and its contents in the sonicator for 5 min.
  - d. Rinse the parts with clean tap water.
3. Clean the parts in deionized water as follows:
  - a. Use forceps to place the parts in a 500 mL glass beaker containing deionized water.
  - b. Place the beaker and its contents in the sonicator for 5 min.
  - c. If the water is cloudy after sonicating, pour off the water, add fresh water, and place the beaker and its contents in the sonicator again for 5 min. Repeat until the water is clear.
4. Clean the parts in fresh acetone as follows:
  - a. Use forceps to place the parts in a 500 mL glass beaker containing fresh acetone.
  - b. Place the beaker and its contents in the sonicator for 5 min.
5. Blow-dry the parts immediately by using clean, dry gas (air or nitrogen).

6. Complete the drying process, as follows:

- Use forceps to place the parts in a 500 mL glass beaker, cover it with aluminum foil, and then put it in an oven set at 100 °C (212 °F) for 30 min.

–or–

- Lay the parts on clean aluminum foil (dull side facing up) and let them dry for 30 min.

7. Wait until the parts are cool before reassembling them.

## Cleaning Nonstainless Steel Parts

The reagent ion source's block heater ring, filament spacer, lens holder, and spacers are nonstainless steel parts made from aluminum or ceramic. To clean these parts, follow the procedure in this section.

You need the following tools and supplies (see also [Table 4](#)):

- Acetone, reagent grade (or other suitable polar solvent)
- Clean, dry gas (air or nitrogen)
- Cotton-tipped applicators
- Detergent (Alconox, Micro-90, or equivalent)
- Forceps
- Glass beakers, 500 mL
- Water, deionized
- Water, tap



**CAUTION** Do not use this procedure to clean stainless steel parts—instead, see “[To clean stainless steel parts](#).” Do not allow the aluminum oxide slurry to contact the aluminum, ceramic, or gold-plated portions of nonstainless steel parts.

### ❖ To clean nonstainless steel parts

1. Clean the parts in a detergent solution as follows:

- a. In a 500 mL glass beaker, make a solution of detergent and warm tap water.
- b. Dip a clean, cotton-tipped applicator in the detergent mixture and scrub the parts.

**Note** Do not soak or sonicate the parts in detergent.

2. Using forceps, rinse the parts thoroughly with tap water.



**CAUTION** Do not leave aluminum parts, such as the block heater ring, in the detergent. Basic solutions, such as detergent, discolor aluminum.



- Using forceps, rinse the parts in a 500 mL glass beaker containing deionized water.

Change the water if it becomes cloudy.

**Note** Do not soak or sonicate the parts in water.

- Using forceps, rinse the parts in a 500 mL glass beaker containing acetone.

Change the acetone if it becomes cloudy.

**Note** Do not soak or sonicate the parts in acetone.

- Blow-dry the parts immediately using clean, dry gas (air or nitrogen).

## Ion Volume Maintenance

The reagent ion source consists of an ion volume, a filament, and ion source lenses. To restore system performance, always clean the ion volume first as described in the following procedure. Reagent molecules interact with energetic electrons to form ions in the ion volume. In general, the closer a component is to where the reagent ions are generated, the more quickly it becomes dirty. For example, the ion volume needs to be cleaned more often than other parts. With the ion volume tool, you can access the ion volume by entering the probe interlock through the inlet valve without shutting down and venting the system.

If cleaning or replacing the ion volume does not improve performance, clean the ion source lenses (see [Cleaning the Ion Source Lenses](#)). If cleaning either of these components does not restore system performance, clean the ion source block (see [Cleaning the Ion Source Block](#)).

To maintain the ion volume, follow these procedures:

- [To insert the ion volume tool into the inlet valve](#)
- [To open the probe interlock](#)
- [To remove the ion volume](#)
- [To reinstall the ion volume](#)

In addition to the appropriate cleaning tools and supplies, you need the following items (see also [Table 4](#)):

- Aluminum foil
- Gloves, lint- and powder-free
- Ion volume insertion and removal tool
- Protective eye wear

**IMPORTANT**

- Prepare a clean work surface by covering the area with aluminum foil, dull side up.
- Put on a new pair of lint- and powder-free gloves before starting each of these removal, cleaning, and reinstallation procedures.

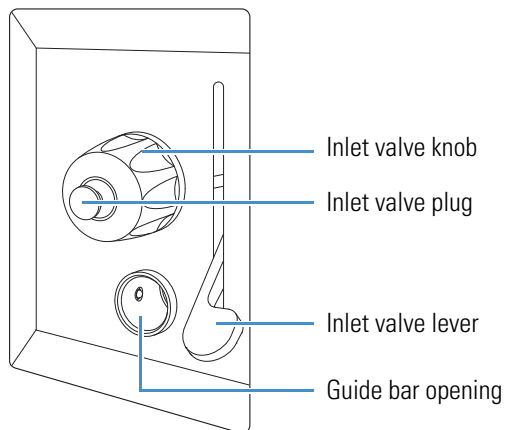
For field service use only	Part number
Ion volume	98000-20052
Ion volume holder	70001-20532

❖ **To insert the ion volume tool into the inlet valve**

1. Put on a pair of new, lint- and powder-free gloves and protective eye wear.

Before proceeding, familiarize yourself with the location of the inlet valve components on the back of the ETD module ([Figure 13](#)).

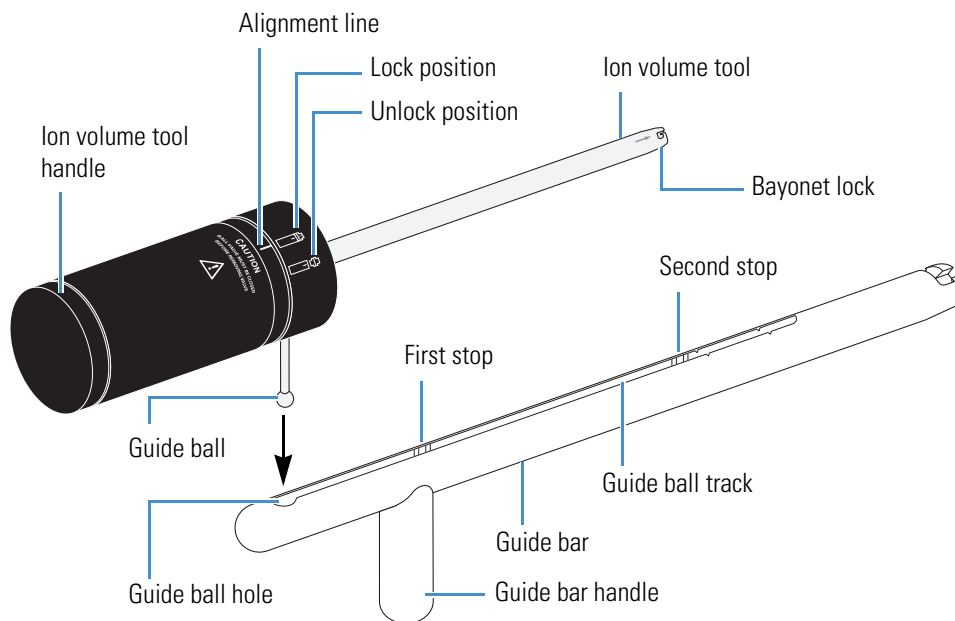
**Figure 13.** Inlet valve components (back of the ETD module)



2. Make sure the inlet valve lever is in the down (closed) position ([Figure 13](#)).

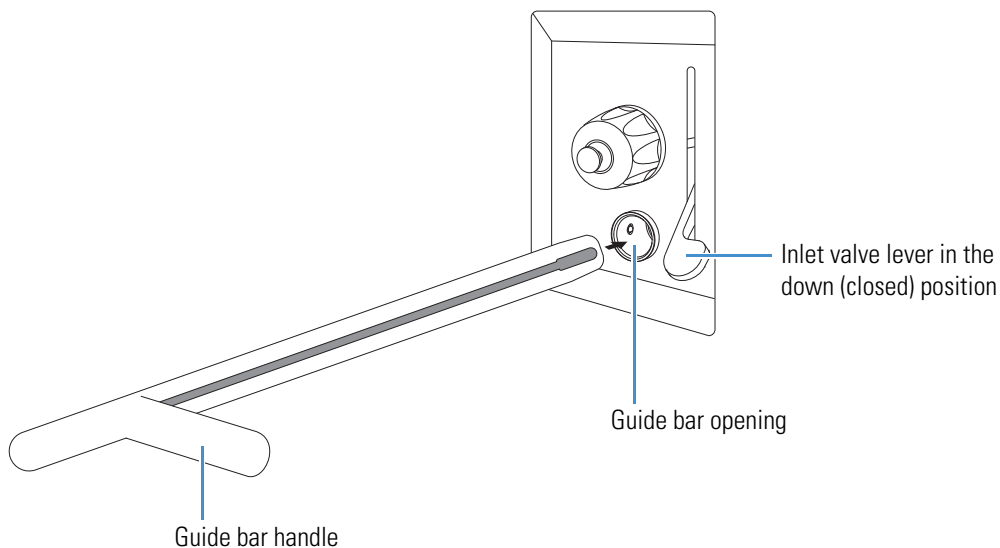
Also, note the components of the ion volume insertion and removal tool ([Figure 14](#)). The guide bar has first and second stops and first and second marks ([Figure 21](#) shows a close-up view).

**Figure 14.** Ion volume insertion and removal tool components



3. Hold the guide bar with the handle horizontal, pointing toward the right (Figure 15).

**Figure 15.** Guide bar positioned for insertion



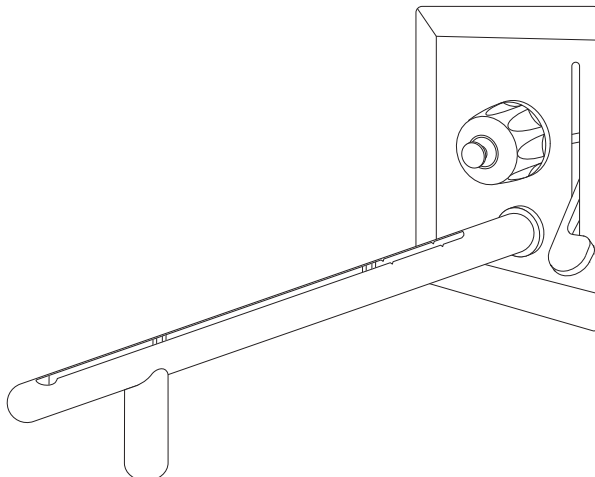
4. Insert the guide bar into the guide bar opening in the back of the ETD module, and push it in as far as it will go.

**IMPORTANT** The use of the guide bar is required. It has a solenoid actuated stop position that prevents you from moving the ion volume tool too far, which could cause catastrophic venting of the system. Therefore, by using the guide bar you prevent damage to the system, including the turbo pump.

5. Rotate the guide bar 90-degrees clockwise to lock it in position (Figure 16).

The guide bar handle now points down.

**Figure 16.** Guide bar insertion complete



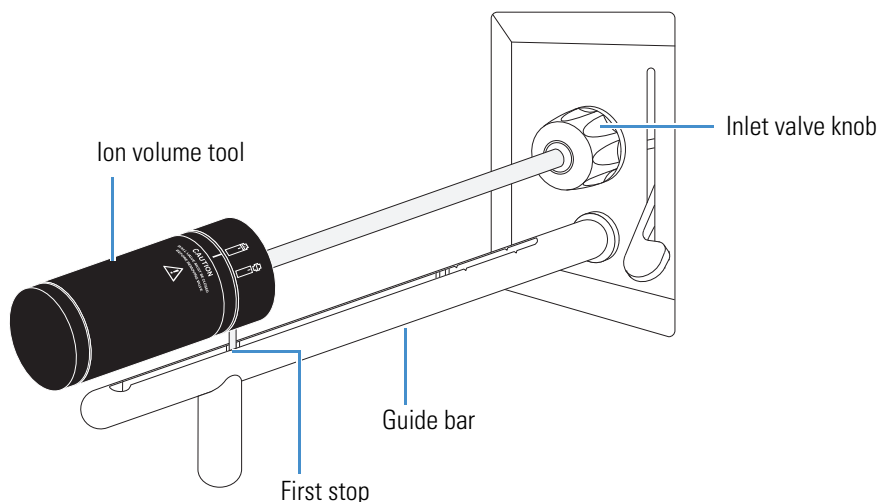
6. Rotate the inlet valve knob (Figure 13) counterclockwise and remove the inlet valve plug.

The inlet valve knob has a stainless steel ferrule inside. Keep the inlet valve knob and ferrule together.

**Note** The inlet valve plug prevents air from entering the vacuum manifold in case the inlet valve is inadvertently opened.

7. Insert the ion volume tool's guide ball into the guide bar's guide ball hole (Figure 17).

**Figure 17.** First stop of the ion volume tool guide bar



- Slide the ion volume tool forward in the guide bar track until it is at the first stop on the guide bar (Figure 17).

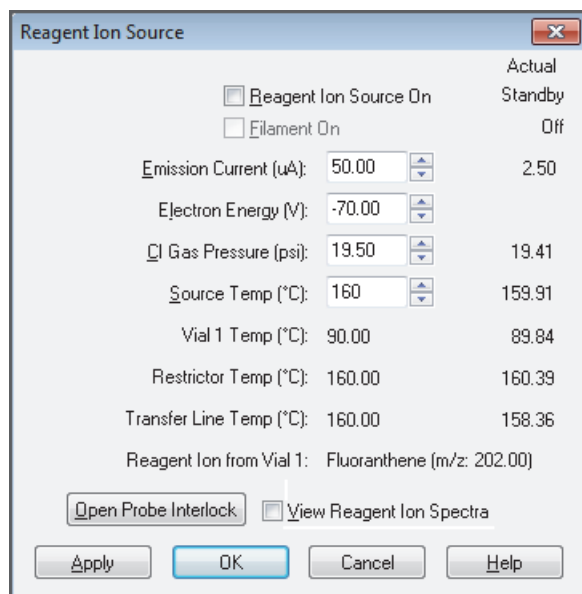
This location prevents the probe from being pulled forward when the inlet valve is evacuated.

- Use your fingers to tighten the inlet valve knob to ensure a leak-tight seal (Figure 17).

❖ **To open the probe interlock**

- Open the Tune Plus window (see page 13).
- Click the **On/Standby** button to select the **Standby** mode.
- Choose **Setup > Reagent Ion Source** to open the Reagent Ion Source dialog box (Figure 18).

**Figure 18.** Reagent Ion Source dialog box



- Click **Open Probe Interlock**.

When the probe interlock has pumped down to less than 0.1 mTorr, a message appears:

The Ball Valve can now be opened.

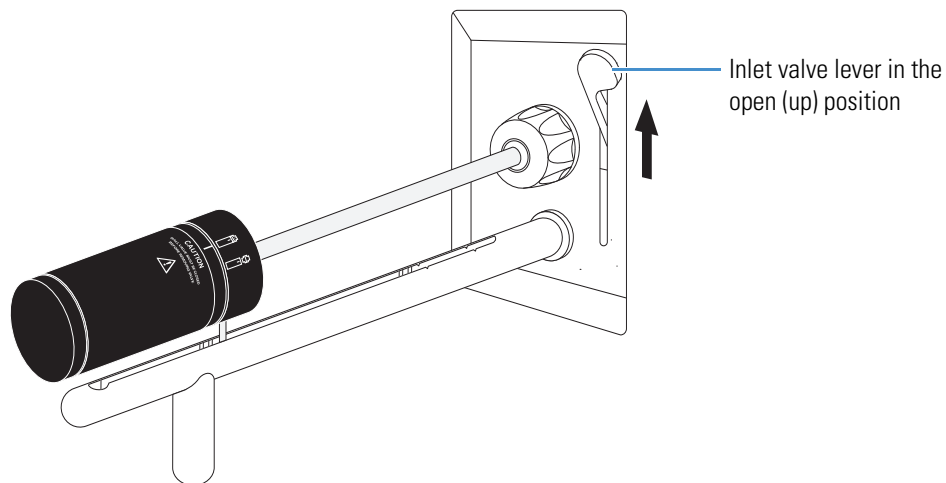
If a pressure of 0.1 mTorr or less is not reached, check to ensure that the inlet valve knob is tight. If the inlet valve knob is tight but the pressure remains high, replace the inlet valve seal as described in “[Inlet Valve Maintenance](#).”

Do not close the Reagent Ion Source dialog box.

❖ **To remove the ion volume**

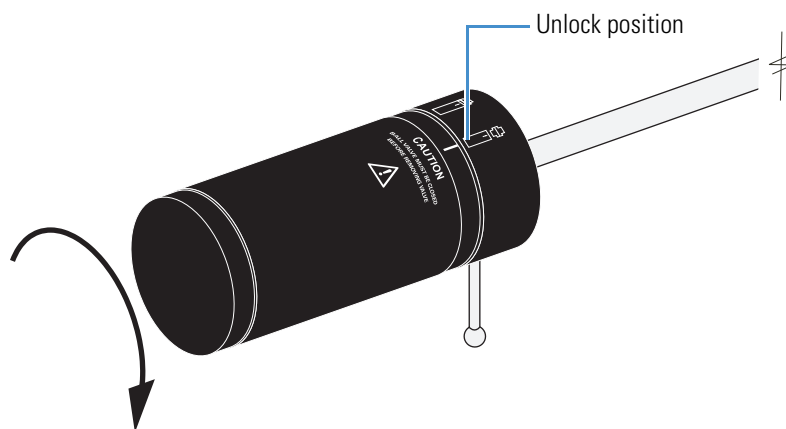
1. Attach the ion volume tool to the ion volume inside the ion source as follows:
  - a. Push the inlet valve lever up to open the inlet valve (Figure 19).

**Figure 19.** Inlet valve lever in the open position



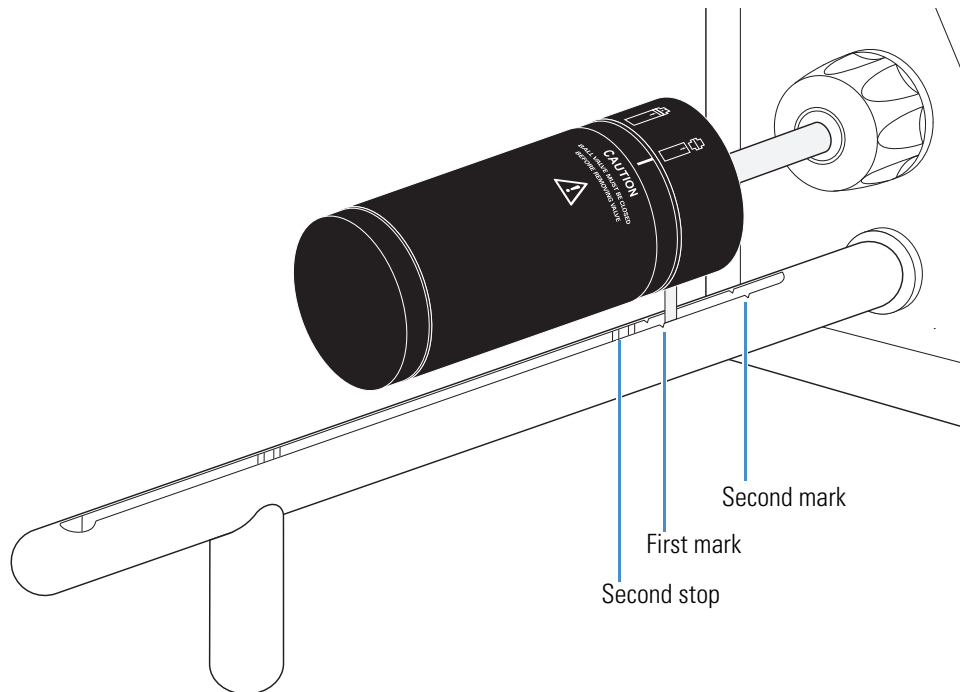
- b. Turn the ion volume tool handle to the unlock position (Figure 20), which indicates that the ion volume tool is in position to accept the ion volume.

**Figure 20.** Ion volume tool handle in the unlock position



- c. Slide the ion volume tool into the inlet valve opening until the guide ball is located just past the first mark on the guide bar (Figure 21).

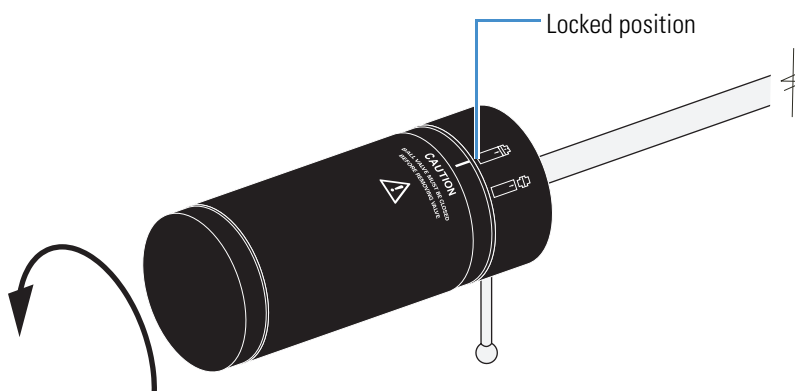
**Figure 21.** Ion volume tool fully inserted into the inlet valve showing the guide ball just past the first mark



- d. Turn the ion volume tool handle to the locked position (Figure 22).

Listen for a click, indicating that the handle is fully engaged in the locked position and is holding the ion volume.

**Figure 22.** Ion volume tool handle in the locked position



2. Remove the ion volume from the ion source as follows:

- a. Withdraw the ion volume tool (the ion volume is attached) until the guide ball reaches the first stop (see [Figure 17](#) for the first stop position).



**CAUTION** Do not withdraw the ion volume tool beyond the point where the guide ball reaches the first stop in the guide bar. Close the inlet valve before withdrawing the ion volume tool past the first stop. Otherwise, the system vents to the atmosphere, which might require cleaning the components that are under vacuum.

- b. Close the inlet valve by pushing the inlet valve lever down.

The probe interlock dialog box displays a message indicating that the valve is closed.

- c. Loosen the inlet valve knob.
- d. Withdraw the ion volume tool completely from the inlet valve by pulling the ion volume tool in the guide bar to the guide ball hole.
- e. Remove the ion volume tool from the guide bar.

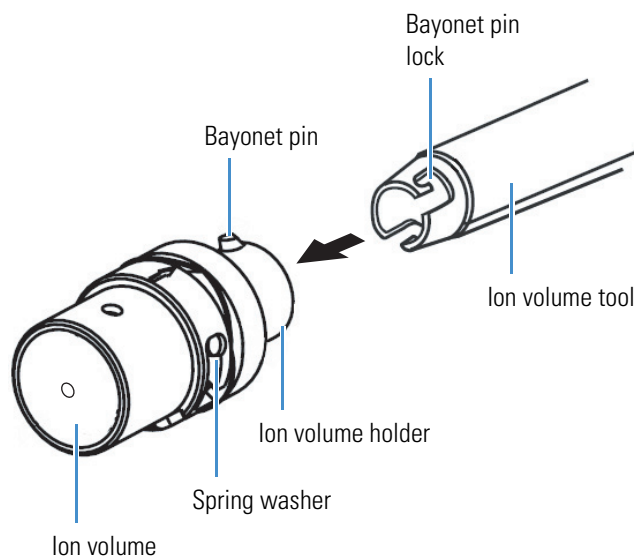


**CAUTION Hot surface.** The ion volume might be too hot to touch. Let it cool to room temperature before handling it.

3. Remove the ion volume from the ion volume tool as follows:

- a. Turn the ion volume tool handle to the unlock position ([Figure 20](#)).
- b. After the part has cooled, press the ion volume into the tip of the ion volume tool and rotate it to disconnect the bayonet pin on the ion volume holder from the bayonet pin lock on the ion volume tool ([Figure 23](#)).

**Figure 23.** Ion volume assembly





4. Press the ion volume into the ion volume holder, and then rotate and remove it from the ion volume holder.

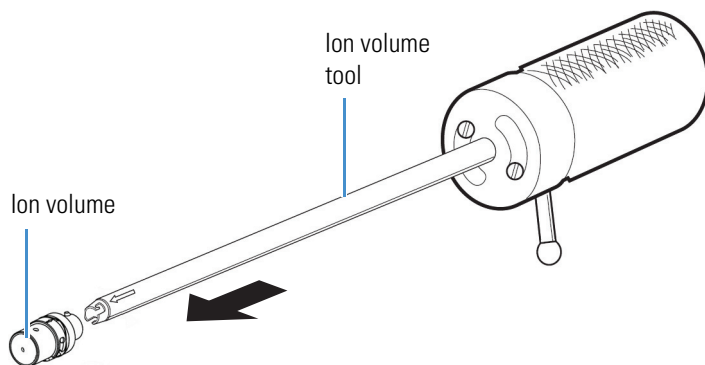
Rotate the ion volume holder and ion volume in opposite directions to separate the two components.

5. Clean the ion volume and ion volume holder, as described in “[Cleaning Stainless Steel Parts.](#)” Make sure you remove the ion burn from the inside of the holes in the ion volume.

❖ **To reinstall the ion volume**

1. Attach the ion volume to the ion volume tool as follows:
  - a. Press the ion volume into the ion volume holder and rotate the ion volume to secure it to the ion volume holder.
  - b. Align the arrow on the ion volume holder with the arrow on the ion volume tool ([Figure 24](#)).

**Figure 24.** Placement of the ion volume on the ion volume tool



**CAUTION** To avoid damage to the ion source, make sure that the arrows on the ion volume tool and ion volume are aligned.

- c. Press the ion volume holder into the tip of the ion volume tool and rotate it counterclockwise to lock the bayonet pin.
  - d. Turn the ion volume tool handle to the lock position ([Figure 22](#)).
2. Insert the ion volume into the ion source as follows:
    - a. Insert the guide ball on the ion volume tool into the guide ball hole and slide it forward until the guide ball is at the first stop ([Figure 17](#)).
    - b. Tighten the inlet valve knob to ensure a leak-tight seal.
    - c. Follow the procedure “[To open the probe interlock.](#)”
    - d. After evacuation is complete, push the inlet valve lever up to open the inlet valve ([Figure 19](#)).

- e. Slide the ion volume tool into the inlet valve opening (Figure 21).
- f. Listen for a click indicating a connection between the ion volume and the ion source block.
- g. The guide ball is now slightly beyond the second stop (and first mark) on the guide bar (Figure 21).
- h. Turn the ion volume tool handle to the unlock position (Figure 20).
- i. Pull the ion volume tool away from the ion volume to the first stop position, and then turn the ion volume tool handle to the locked position (Figure 22).



**CAUTION** Do not withdraw the ion volume tool beyond the point where the guide ball reaches the first stop in the guide bar. Close the inlet valve before withdrawing the ion volume tool past the first stop. Otherwise, the system vents to the atmosphere, which might require cleaning the components that are under vacuum.

3. Remove the ion volume tool as follows:
  - a. Close the inlet valve by pushing down on the inlet valve lever (Figure 17).
  - b. Loosen the inlet valve knob.
  - c. Continue withdrawing the ion volume tool completely from the inlet valve by sliding the ion volume tool through the guide ball track in the guide bar to the guide ball hole.
  - d. Lift the ion volume tool out of the ion volume guide bar.
4. (Optional) Remove the guide bar by rotating it 90-degrees counterclockwise and sliding it out of the guide bar opening.
5. Replace the inlet valve plug and tighten the inlet valve knob (Figure 13).

A Tune Plus message box opens.
6. Click **Close** to close the message box.
7. Retune the mass spectrometer.

## Inlet Valve Maintenance

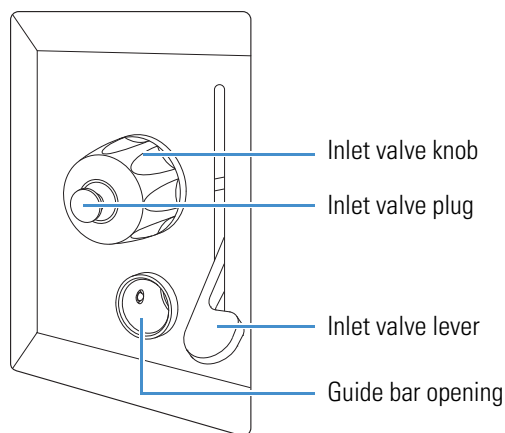
This section describes how to replace the worn out inlet valve components (inlet valve seal or inlet valve). In addition to the appropriate cleaning tools and supplies, you need the inlet valve seal and lint- and power-free gloves (see also [Table 4](#)).

For field service use only	Part number
Inlet Valve Seal Kit	119265-0003
Inlet valve seal removal tool	119283-0001
0.530 in. ID O-rings (2)	3814-6530

### ❖ To replace the inlet valve components

1. Make sure the inlet valve lever is in the down (closed) position ([Figure 25](#)).

**Figure 25.** Inlet valve components (back of the ETD module)



2. Rotate the inlet valve knob counterclockwise and remove the inlet valve plug.

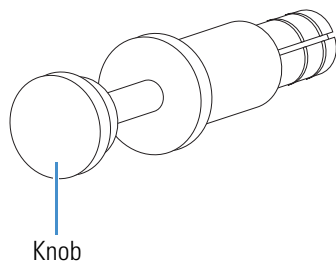
The inlet valve knob has a stainless steel ferrule inside. Keep the inlet valve knob and ferrule together.

**Note** The inlet valve plug prevents air from entering the vacuum manifold in case the inlet valve is inadvertently opened.

3. Remove the inlet valve seal as follows:

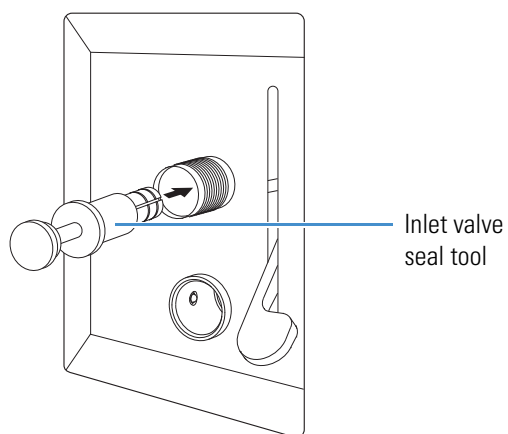
- a. Pull out the knob on the inlet valve seal tool (Figure 26).

**Figure 26.** Inlet valve seal tool



- b. Insert the tool straight into the inlet valve.

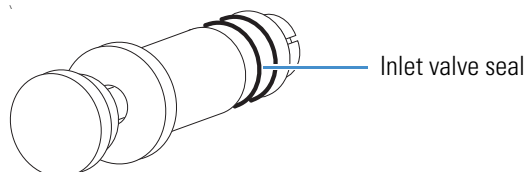
**Figure 27.** Inlet valve seal tool inserted into the inlet valve



- c. Press the knob on the tool until it stops.
- d. Remove the tool.

The inlet valve seal should be on the tool (Figure 28).

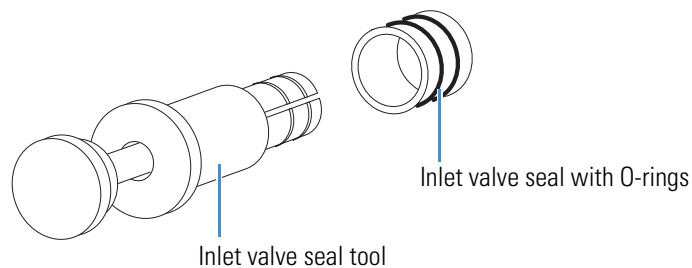
**Figure 28.** Inlet valve seal on the inlet valve seal tool



**Note** Use only the inlet valve seal tool to remove or install the inlet valve seal.

- Loosen the knob to disengage the seal ([Figure 29](#)).

**Figure 29.** Inlet valve seal disengaged from the tool



- Discard the seal and its O-rings.
- Place the two new O-rings into the slots on the new inlet valve seal ([Figure 29](#)).
- Insert the new inlet valve seal as follows:
  - Place the new inlet valve seal onto the inlet valve seal tool.
  - Insert the inlet valve seal tool into the inlet valve until it stops.
  - Remove the tool.

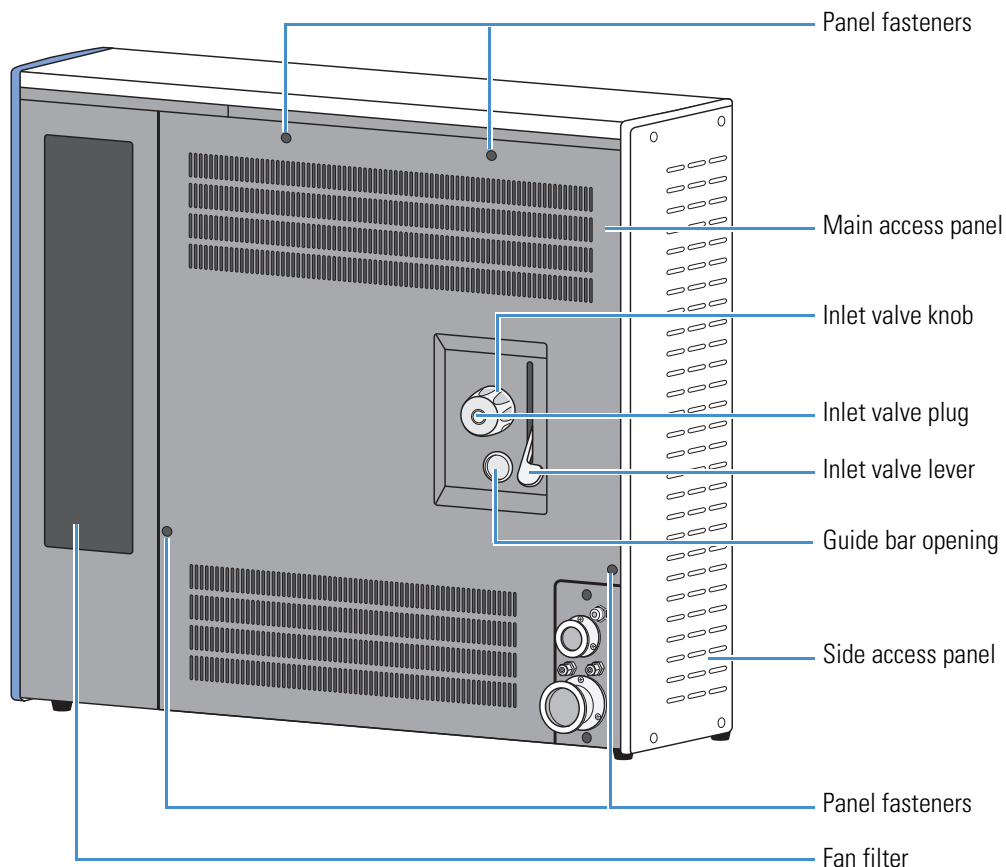
The O-rings secure the inlet valve seal in the opening.

- Reinstall the ferrule, knob, and plug into the inlet valve opening.

## Removing the Access Panels

Some of the ETD module maintenance procedures require that you remove the ETD main access panel, the side access panel, or both panels (Figure 30). To remove these panels, follow the procedures in this section.

**Figure 30.** ETD module (back view)



In addition to the appropriate cleaning tools and supplies, you need a 7/64 in. hex or hex ball driver and a Phillips #2 screwdriver (see also Table 4).

### IMPORTANT

- Prepare a clean work surface by covering the area with aluminum foil, dull side up.
- Put on a new pair of lint- and powder-free gloves before starting each of these removal, cleaning, and reinstallation procedures.

❖ **To remove the ETD main access panel**

1. Place the mass spectrometer's electronics service switch in the Service Mode position.



**CAUTION** In Service mode all power to the MS/ETD system electronics turns off. There are no user accessible components that carry a voltage in this mode. However, the vacuum pumps continue to operate.



**CAUTION Hot surface.** When the MS/ETD system is on, the flow restrictor, transfer line, and ion source heaters can reach 160 °C (320 °F). The vial heaters can be at 108 or 90 °C, depending on the reagent ion source configuration. (Thermo Fisher Scientific recommends the 90 °C vial setting.) Do not attempt to replace the reagent vials or service heated components until they have cooled to room temperature, which can take an hour or more.

2. Remove the inlet valve lever (Figure 30) by pulling it down and away from the back panel. Do not rotate the lever up. It must remain in its down (closed) position to avoid catastrophic venting of the system.



**CAUTION** To prevent a catastrophic venting of the system, the inlet valve lever must be in place and in the down position.

3. Use your fingers to unscrew the inlet valve knob and remove the inlet valve plug, inlet valve knob, and internal ferrule.
4. Using the Phillips #2 screwdriver, loosen the captive screws on the back panel.
5. Tilt the top of the panel toward you and lift it up and away from the ETD module.

❖ **To remove the ETD side access panel**

1. Place the mass spectrometer's electronics service switch in the Service Mode position.



**CAUTION** In Service mode all power to the MS/ETD system electronics turns off. There are no user accessible components that carry a voltage in this mode. However, the vacuum pumps continue to operate.



**CAUTION Hot surface.** When the MS/ETD system is on, the flow restrictor, transfer line, and ion source heaters can reach 160 °C (320 °F). The vial heaters can be at 108 or 90 °C, depending on the reagent ion source configuration. Do not attempt to replace the reagent vials or service heated components until they have cooled to room temperature, which can take an hour or more.

2. Using the 7/64 in. hex driver, loosen the captive hex socket-head screws securing the external side panel, and then remove the panel.

- Using the #2 Phillips screwdriver, loosen the captive screws securing the internal metal side panel, and then remove the panel.



**CAUTION Hot surface.** The reagent vial heaters, ion source heater, flow restrictor, and transfer lines are heated components that are accessible under the side access panel. Verify that they are safe to touch before handling them.

To replace the panels, follow the above steps in reverse order and reverse the instructions in each step.

## Ion Source Maintenance

Maintaining the ion source requires that you shut down and vent the MS/ETD system. The procedures in this section are arranged in order according to how likely the maintenance would need to be done to restore the system to complete functionality. However, Thermo Fisher Scientific recommends that you clean all the elements of the ion source while you have it disassembled.

To maintain the ion source, follow these procedures:

- [Inspecting or Replacing the Ion Source Filament](#)
- [Cleaning the Ion Source Lenses](#)
- [Cleaning the Ion Source Block](#)

In addition to the appropriate cleaning tools and supplies, you need the following items (see also [Table 4](#)):

- Aluminum foil
- Gloves, lint-free and powder-free
- Hex or hex ball driver, 9/64 in.
- Lamp, small, high-intensity
- Magnification device
- Pliers
- Protective eye wear
- Screwdriver, Phillips, #2
- Tweezers, large



### IMPORTANT

- Prepare a clean work surface by covering the area with aluminum foil, dull side up.
- Put on a new pair of lint- and powder-free gloves before starting each of these removal, cleaning, and reinstallation procedures.

#### For field service use only

#### Part number

Filament Assembly DSQ II

120320-0030

## Inspecting or Replacing the Ion Source Filament

The number of ions produced in the ion source is approximately proportional to the filament emission current. Low ion production, for example, can indicate filament problems. If the instrument indicates a filament fault or a failure to regulate, or if the reagent ion signal intensity decreases over time, the filament might have sagged, failed, or is failing and must be replaced.

### ❖ To inspect or replace the ion source filament

1. Prepare a clean work space by covering the area with aluminum foil, dull side up.
2. Shut down and vent the MS/ETD system. See “[Shutting Down the MS/ETD System.](#)”
3. Unplug the mass spectrometer and ETD module power supply cords.



**CAUTION** The ion source might be too hot to touch even if the cooling nitrogen has completed its cycle. Make sure that the ion source has cooled to room temperature (at least 1 hour after venting) before handling it.

4. Remove the ETD module’s main (back) access panel (see [page 50](#)).

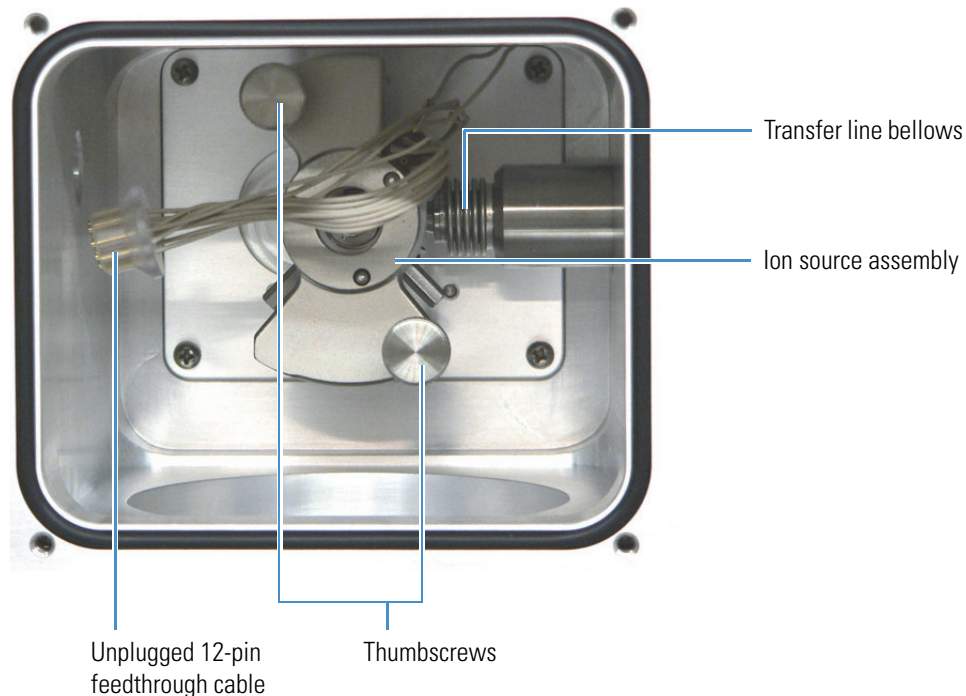


**CAUTION** Keep the inlet valve lever in the down (closed) position whenever it is not explicitly required to be in the up position (open), even if the vacuum manifold is at atmospheric pressure. This is consistent with maintenance procedures that rely on the inlet valve lever being closed at the appropriate step to prevent the accidental loss of vacuum. If the vacuum is accidentally lost, the system might be damaged. At a minimum, the components that were under vacuum may need cleaning.

5. Put on a pair of new, lint- and powder-free gloves.
6. Using the 9/64 in. hex driver, remove the four hex socket-head screws that secure both the EMI cover and the probe plate to the vacuum manifold ([Figure 2](#)).

7. Remove the ion source assembly as follows:
  - a. Unplug the 12-pin feedthrough cable (Figure 31).

**Figure 31.** Interior of vacuum manifold



- b. Loosen the thumbscrews (Figure 31).
  - c. Remove the ion source assembly from the vacuum manifold.

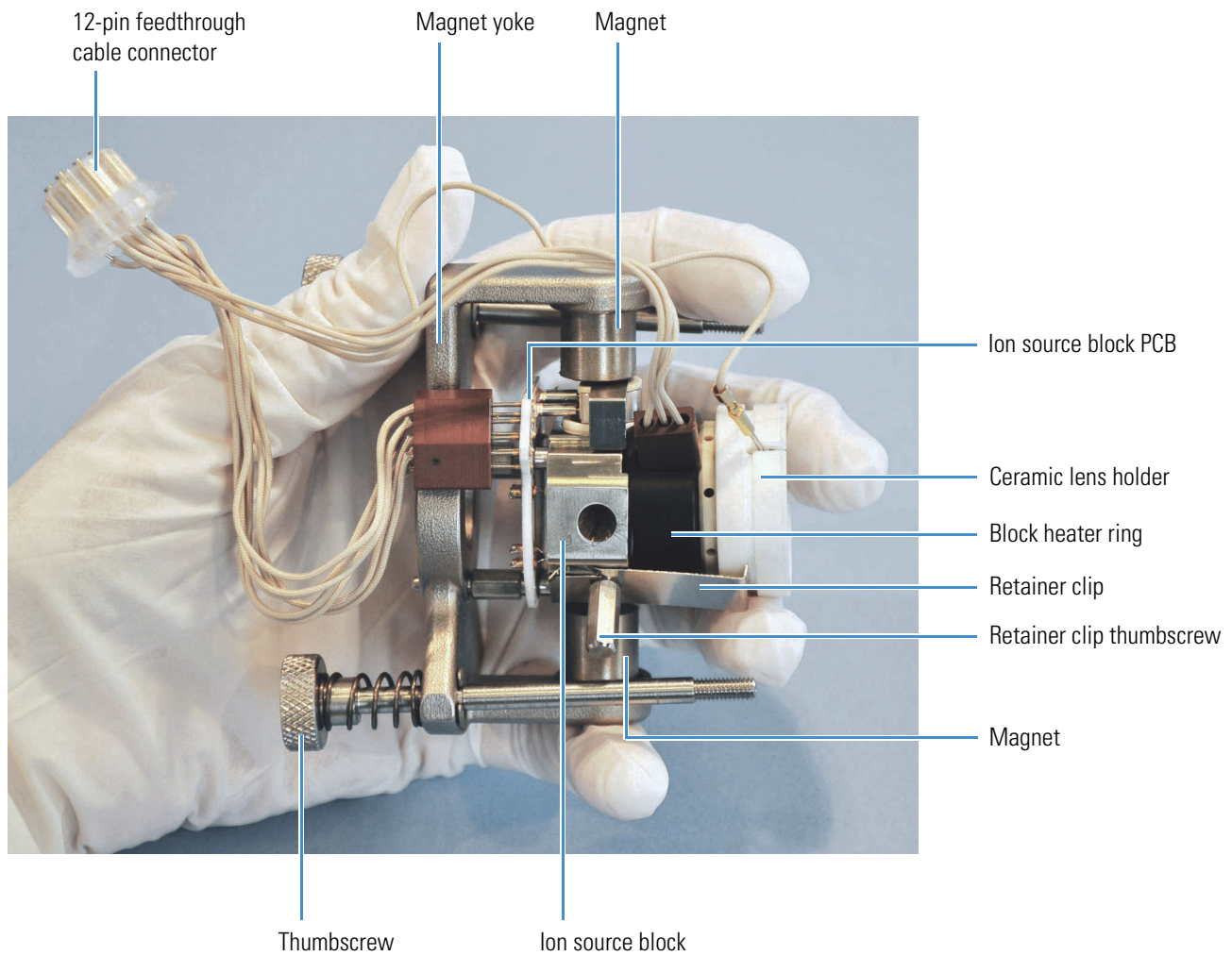


**CAUTION** To prevent unsecured components inside the lens holder from falling out, keep the tips of your gloved fingers on both the front edge of the lens holder and the back of the magnet yoke when handling the ion source assembly. See Figure 32.

A clip holds the ion source assembly together. As you remove the assembly, gently shift it to the left before and while pulling it out. This allows the assembly to disengage from the transfer line bellows (Figure 31) as you remove it.

Or, gently depress the transfer line bellows to disengage it from the ion source assembly.

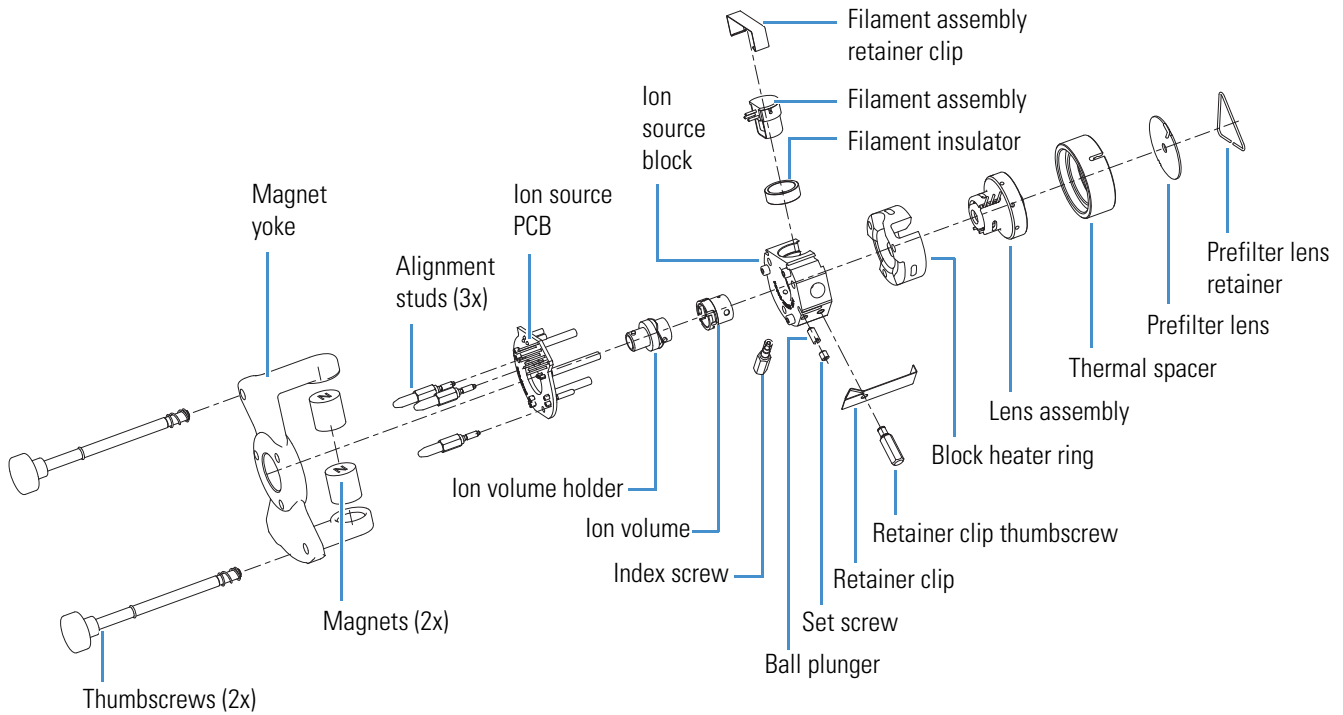
Figure 32. Ion source assembly<sup>1</sup>



<sup>1</sup> Your block heater ring might look different from that shown in these drawings and photographs.

Figure 33 shows an exploded view of the ion source assembly.

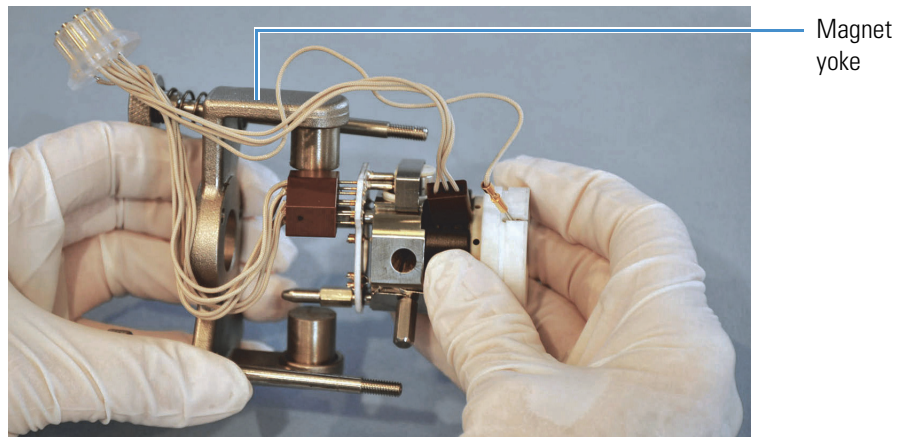
Figure 33. Ion source assembly (exploded view)



8. Remove the filament assembly as follows:

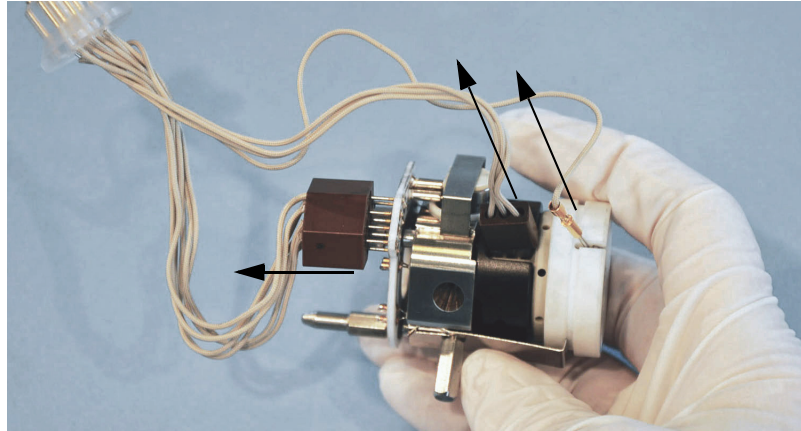
- a. Remove the magnet yoke from the rest of the assembly (Figure 34).

Figure 34. Removing the magnet yoke from the assembly



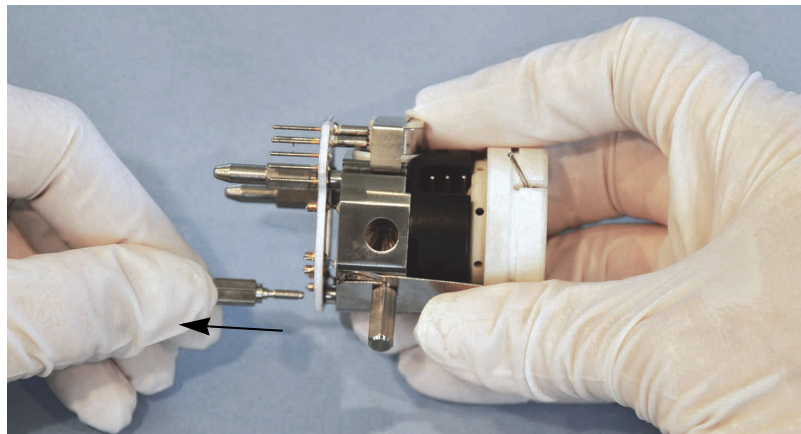
- b. Disconnect the 12-pin feedthrough cable connectors from the ion source PCB, block heater ring, and lens assembly (Figure 35).

**Figure 35.** Removing the feedthrough cable connectors



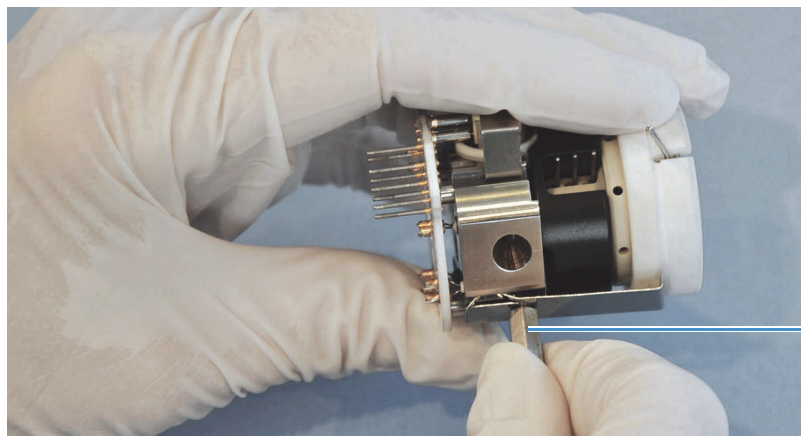
- c. Remove the three alignment studs (Figure 36).

**Figure 36.** Removing the alignment studs



- d. Loosen the retainer clip thumbscrew (Figure 37).

**Figure 37.** Retainer clip thumbscrew



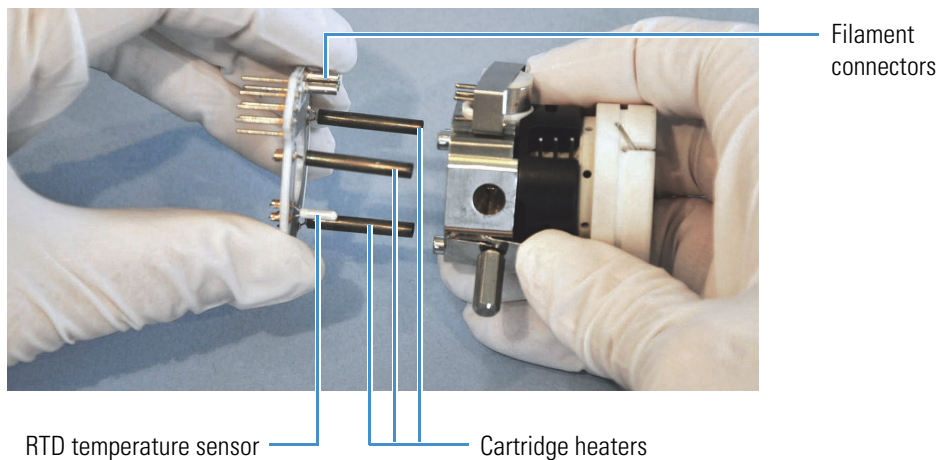
Retainer  
clip  
thumbscrew

- e. Gently pull the ion source PCB away from the assembly, disconnecting the filament connectors, RTD temperature sensor, and three cartridge heaters (Figure 38).



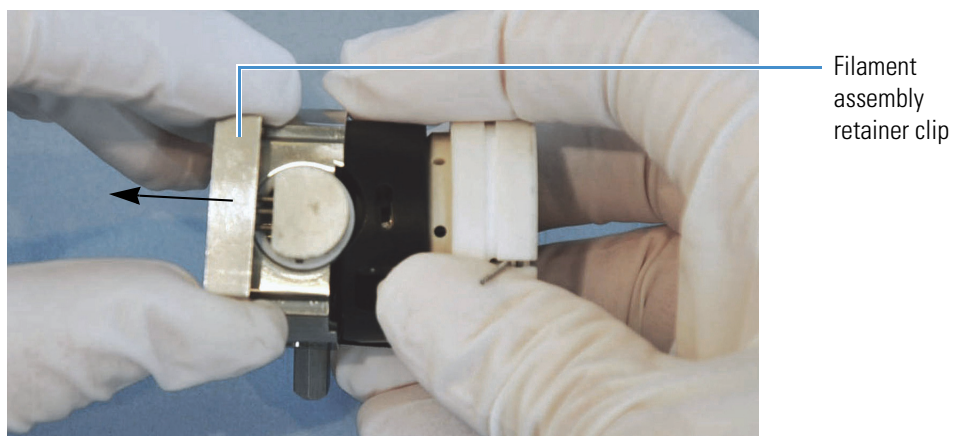
**CAUTION** Do not bend or twist the cartridge heaters or RTD temperature sensor.

**Figure 38.** Detaching the ion source PCB from the assembly



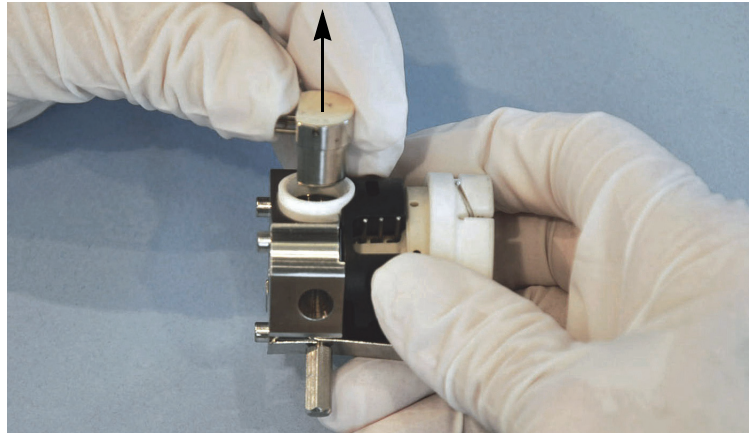
- f. Slide off the filament assembly retainer clip (Figure 39).

**Figure 39.** Removing the filament assembly retainer clip



- g. Remove the filament assembly (Figure 40).

**Figure 40.** Removing the filament assembly

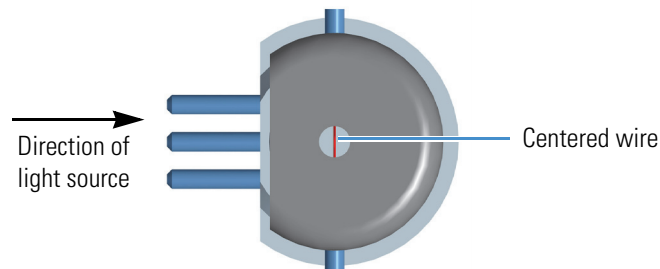


**Tip** Now is a good time to clean the ion source lenses. See “Cleaning the Ion Source Lenses.”

- 9. Turn the filament assembly over and, using a strong light and a magnification device, verify that the filament wire is centered in the electron lens hole.

Figure 41 shows the centered filament wire as seen from the bottom of the filament through the electron lens hole. If the filament wire is sagging, not centered, or otherwise damaged, you must replace the filament assembly.

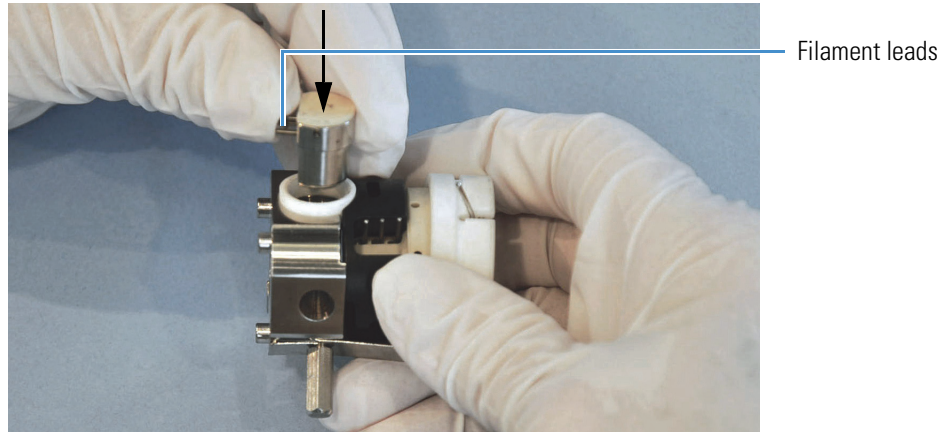
**Figure 41.** Filament wire visible through the bottom electron lens hole



10. Install the filament assembly as follows:

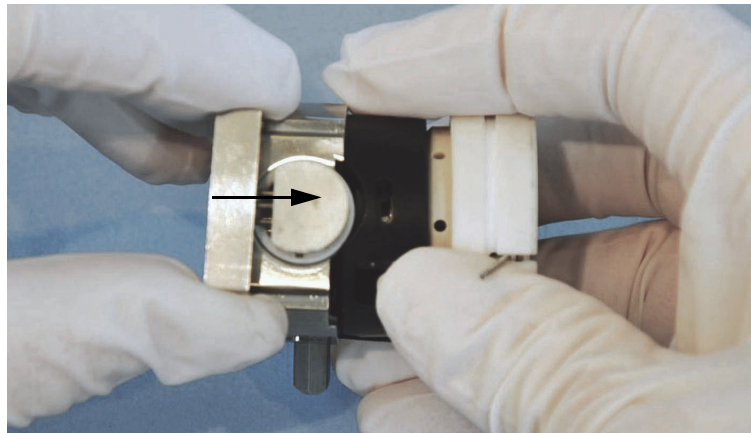
- a. Put the existing or new filament assembly into the ion source block, aligning the filament leads as shown in [Figure 42](#).

**Figure 42.** Filament assembly ready to be installed



- b. Slide the filament assembly retainer clip back over the filament assembly ([Figure 43](#)).

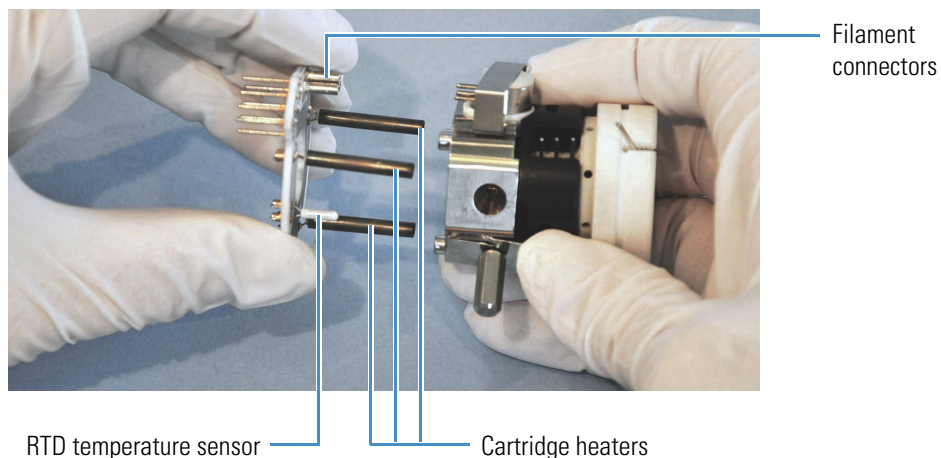
**Figure 43.** Filament assembly retainer clip ready to be installed





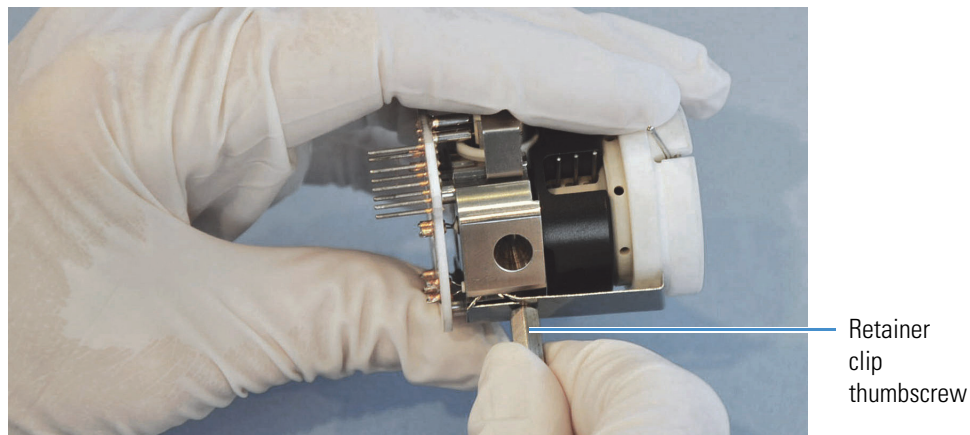
- c. Align the cartridge heaters on the ion source PCB with the holes in the ion source block, and the filament leads with the ion source PCB connectors (Figure 44).

**Figure 44.** Cartridge heaters aligned with the ion source block



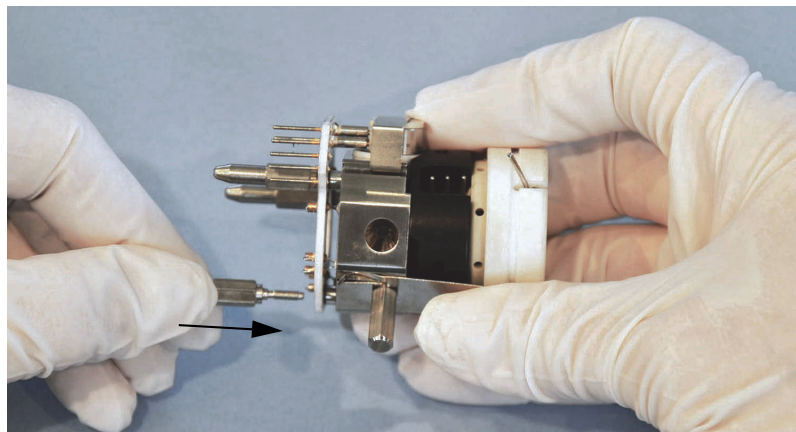
- d. Gently press the ion source PCB onto the ion source block, positioning the RTD temperature sensor under the retainer clip (Figure 44), and make sure that the PCB fits snugly against the ion source block.
- e. Tighten the retainer clip thumbscrew (Figure 45).

**Figure 45.** Retainer clip thumbscrew



- f. Reinstall the three alignment studs (Figure 46) and hand-tighten only.

**Figure 46.** Reinstalling the alignment studs

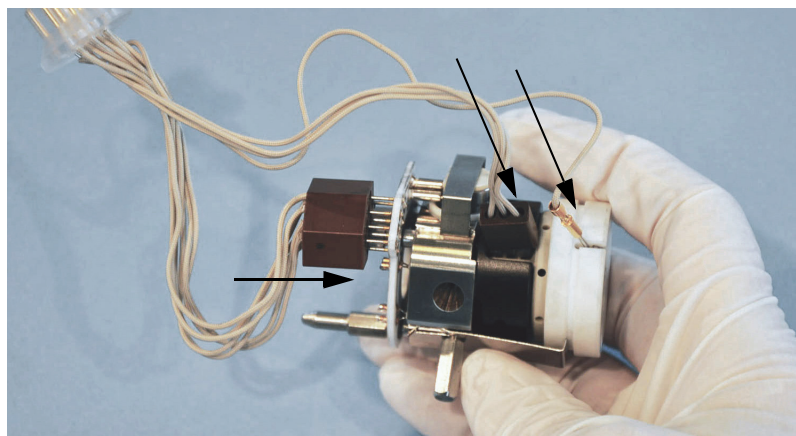


- g. Reattach the leads for the 12-pin feedthrough cable to the ion source PCB, block heater ring, and lens assembly (Figure 47).

To ensure the correct installation of the triple-lead connector to the lenses, position the beveled edge of the connector against the thermal spacer.

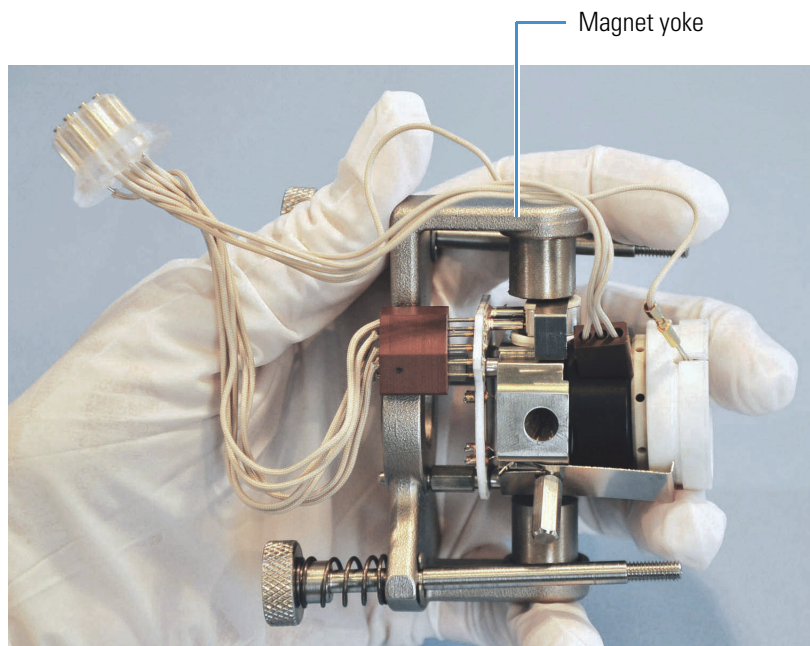
**Tip** The lens lead only makes contact with the top of the pins. It does not go down into the slot.

**Figure 47.** Reattaching the feedthrough cable connectors



- h. Reposition the magnet yoke around the ion source assembly (Figure 48).

**Figure 48.** Ion source assembly repositioned in the magnet yoke



11. Reinstall the ion source assembly as follows:
  - a. Place the ion source assembly in the vacuum manifold and hand-tighten the thumbscrews all the way in.
  - b. Reconnect the feedthrough connector (notch at the top) to the vacuum manifold.
12. Using the 7/64 in. hex driver and hex socket-head screws, reinstall the vacuum manifold probe plate and EMI cover.
13. If you do not need to clean any parts, reinstall the main access panel, inlet valve knob, and inlet valve plug.
14. Plug in the mass spectrometer and ETD module power supply cords.
15. Restore the MS/ETD system to operational status. See [Chapter 3, “Shutdown and Startup.”](#)

## Cleaning the Ion Source Lenses

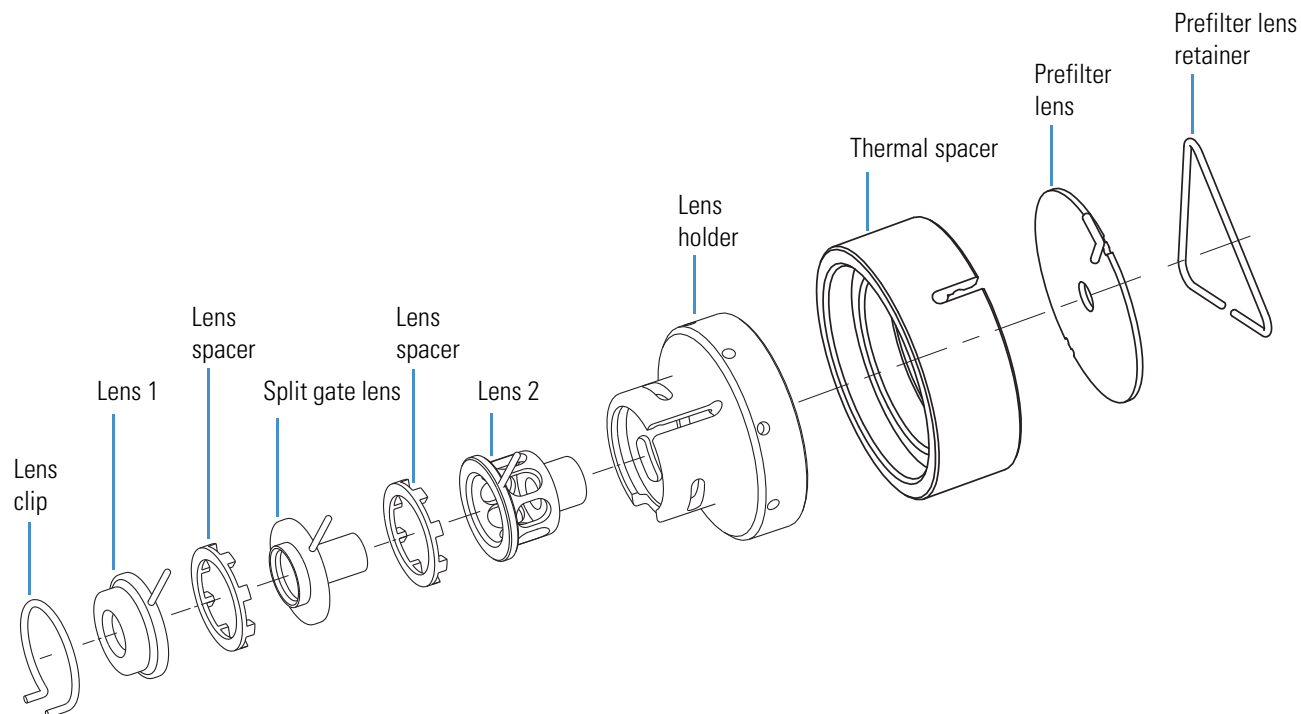
If you cleaned the ion volume and did not find any problems with the filament, try cleaning the ion source lenses. The ion source lenses are in direct contact with reagent ions introduced into the ETD module and must be cleaned periodically, though not as often as the ion volume.

❖ **To clean the ion source lenses**

1. Remove and disassemble the ion source lens assembly as follows:
  - a. See [step 3](#) through [step 8g](#) in “[Inspecting or Replacing the Ion Source Filament.](#)”

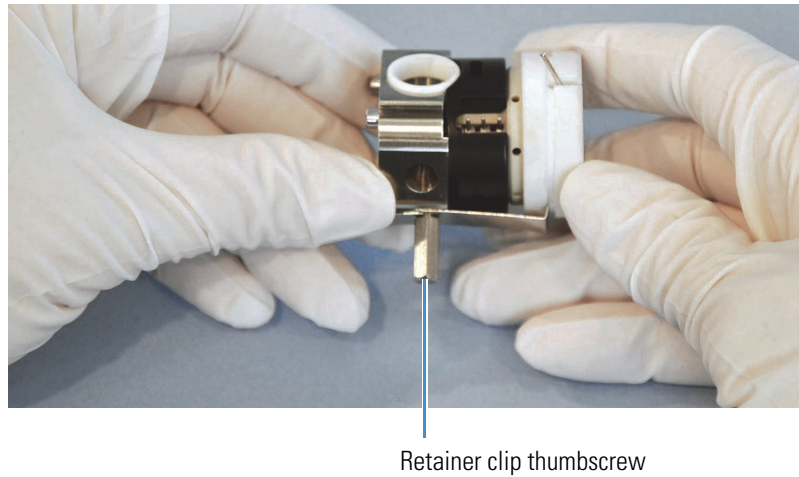
[Figure 49](#) shows an exploded view of the ion source lens assembly.

**Figure 49.** Ion source lens assembly (exploded view)



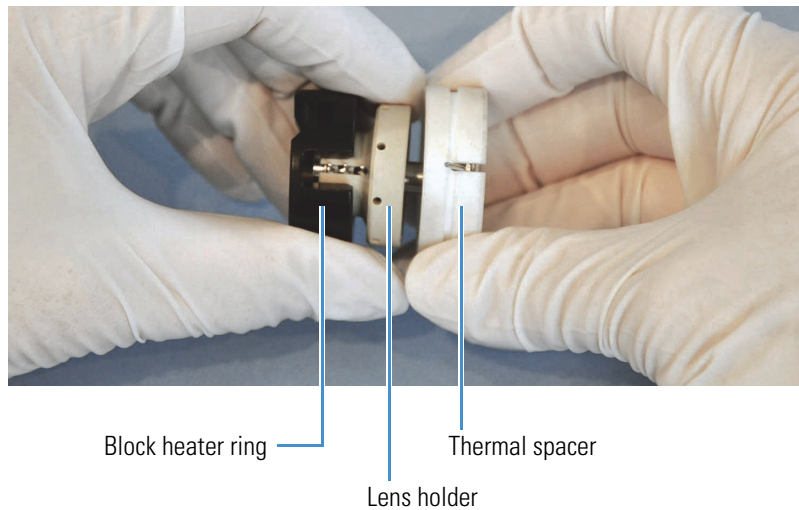
- b. Loosen the retainer clip thumbscrew, and then remove the retainer clip (Figure 50).

**Figure 50.** Location of retainer clip thumbscrew



- c. Separate the block heater ring, lens holder, and thermal spacer (Figure 51).

**Figure 51.** Block heater ring, lens holder, and thermal spacer



- d. Using the clean tweezers, remove the lens clip, lenses, and spacers from the lens holder (Figure 49) and place them on the aluminum foil.



**CAUTION** Do not chip the lens holder.

- e. Pinch the prefilter lens retainer with your fingers or the pliers, and then remove the prefilter lens from the thermal spacer (Figure 49).
2. Clean the lenses as described in [Cleaning Stainless Steel Parts](#) and the lens spacers and lens holder as described in [Cleaning Nonstainless Steel Parts](#).

3. Reassemble the ion source assembly as follows:
  - a. Insert the components of the lens assembly into the lens holder (Figure 50).
  - b. Using the clean tweezers, reinstall the lens clip.
  - c. Place the prefilter lens in the thermal spacer.
  - d. Pinch the prefilter lens retainer with your fingers or the pliers and reinstall it to secure the prefilter lens.
4. Reassemble the ion source assembly and reinstall it into the vacuum manifold as described in step 10a through step 12 in “Inspecting or Replacing the Ion Source Filament.”
5. Using the 9/64 in. hex driver, reinstall the probe plate and the EMI cover to the vacuum manifold (Figure 2).
6. Plug in the mass spectrometer and ETD module power supply cord.
7. Restore the MS/ETD system to operational status. See Chapter 3, “Shutdown and Startup.”

## Cleaning the Ion Source Block

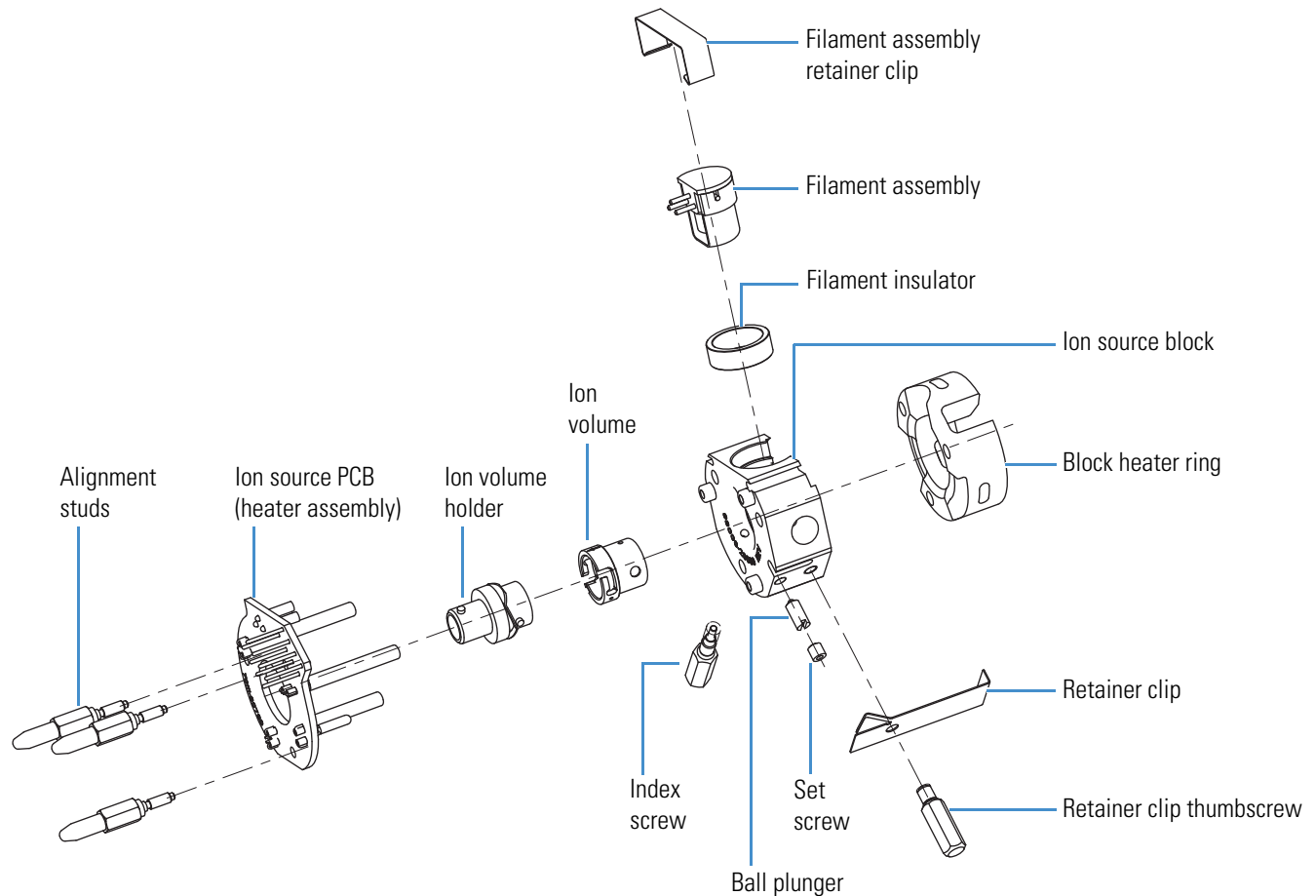
If you change or check the filament and you observe significant ion burn or carbon buildup around the electron entrance hole of the ion source block, Thermo Fisher Scientific recommends that you clean the ion source block. Generally, you must clean the ion source block once every six months, depending on the usage.

### ❖ To clean the ion source block

1. Remove the ion source lens assembly (Figure 49). See step 3 through step 8g in “Inspecting or Replacing the Ion Source Filament.”
2. Remove the lenses and spacers. See step 1b through step 1e in “Cleaning the Ion Source Lenses.”

See Figure 52 to locate the following components that must be removed from the ion source block before cleaning: ion source block, ion volume, and ion volume holder.

Figure 52. Ion source components (exploded view)

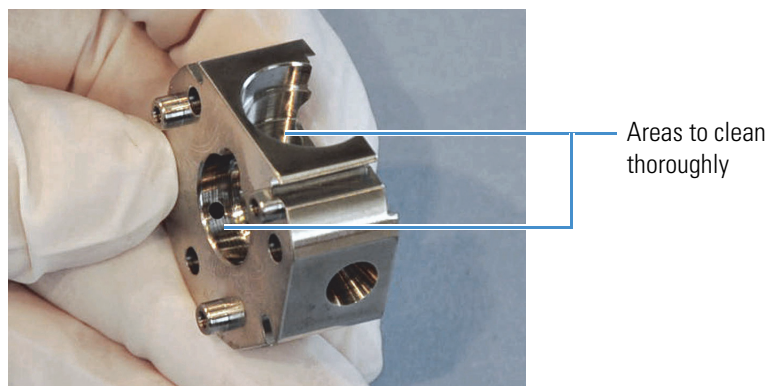


3. Remove the filament insulator (ring) from the ion source block.
4. Remove the retainer clip thumbscrew and retainer clip.
5. Clean the ion source block (Figure 53) as described in “Cleaning Stainless Steel Parts.”

**Note** For the ion source block, ion burn accumulates in the hole that is opposite from the filament. Spend extra time cleaning this area.

**IMPORTANT** Because it is difficult to properly adjust the retention of the ion volume, Thermo Fisher Scientific recommends that you do NOT remove the set screw and ball plunger from the ion source block.

**Figure 53.** Ion source block



6. Clean the ion volume and ion volume holder as described in [“Cleaning Nonstainless Steel Parts.”](#)
7. Reassemble the ion source block, and then the ion source assembly.
8. Reinstall the ion source assembly into the vacuum manifold by following in reverse order the procedure in [“Cleaning the Ion Source Lenses.”](#)
9. Restore the MS/ETD system to operational status. See [Chapter 3, “Shutdown and Startup.”](#)

## Reagent Vials Maintenance

Replacing the reagent vials requires that the MS/ETD system be placed in Service mode after the vials have cooled, which can take at least 1 hour.

To change the reagent vials, follow these procedures:

1. [Shutting Down the MS/ETD System](#)
2. [Replacing the Reagent Vials](#)
3. [Starting Up the MS/ETD System](#)

In addition to the appropriate cleaning tools and supplies, you need the following items (see also [Table 4](#)):

- Gloves, lint-free and powder-free
- Protective eye wear
- Screwdriver, Phillips, #2



### IMPORTANT

- Prepare a clean work surface by covering the area with aluminum foil, dull side up.
- Put on a new pair of lint- and powder-free gloves before starting each of these removal, cleaning, and reinstallation procedures.

#### For field service use only

#### Part number

ETD Reagent Kit

98000-62008



**CAUTION** Ensure that the crimping cap and membrane (septum) for both vials in the ETD Reagent Kit are dark blue. The septum prevents the reagent from escaping into the lab environment. To preserve the integrity of the reagent vial septums, remove and replace the reagent vials annually or only when they are empty. Never reinstall used vials because the multiple, unfilled needle openings in a septum could potentially cause leaks.



#### **CAUTION Avoid exposure to potentially harmful materials.**

By law, producers and suppliers of chemical compounds are required to provide their customers with the most current health and safety information in the form of Material Safety Data Sheets (MSDSs) or Safety Data Sheets (SDSs). The MSDSs and SDSs must be freely available to lab personnel to examine at any time. These data sheets describe the chemicals and summarize information on the hazard and toxicity of specific chemical compounds. They also provide information on the proper handling of compounds, first aid for accidental exposure, and procedures to remedy spills or leaks.



Read the MSDS or SDS for each chemical you use. Store and handle all chemicals in accordance with standard safety procedures. Always wear protective gloves and safety glasses when you use solvents or corrosives. Also, contain waste streams, use proper ventilation, and dispose of all laboratory reagents according to the directions in the MSDS or SDS.

For safety information about fluoranthene, see [Chapter 7, “Reagent Kit.”](#)

## Shutting Down the MS/ETD System

### ❖ To shut down the system to replace the reagent vials

1. Follow [step 1](#) through [step 4](#) in “Shutting Down the MS/ETD System.”

**Note** If the vial temperature does not decrease after 5 minutes, check that the reagent ion source configuration has the Use Cooling Gas option selected. For information about this option, refer to “Specifying the Reagent Ion Source for the Mass Spectrometer,” in Chapter 2 of the *ETD Module Getting Started Guide*. The vials eventually cool down without the nitrogen but much more slowly.

**Tip** If your system does not use cooling gas, when in standby mode choose from Tune Plus **Diagnostics > Diagnostics**, select **Set Device** from the Tools list, and then set the values for the **Capillary Heater** and **Source Heater** to 0 °C.

2. When the vial reaches a temperature that is safe for handling (about 30 °C [86 °F]), place the mass spectrometer’s electronics service switch in the Service Mode position.

Service mode turns off all components except the turbo pump and all forepumps.



**CAUTION Hot surface.** Do not place the system in Service mode until the vials reach a safe temperature (about 30 °C [86 °F]). System temperature monitoring stops when the system is in Service mode. Do not attempt to handle the any components until they reach a safe temperature.



**CAUTION** Do not toggle the ETD module’s electronics service switch to the Service Mode position while the mass spectrometer’s switches are in their On positions. This could cause communication problems between the mass spectrometer and ETD module. The ability to control the Service mode for both the mass spectrometer and the ETD module at one location (the mass spectrometer’s electronics service switch) is a safety feature.

The MS/ETD system is now in Service mode and the vials are at a safe temperature for handling. You can now install or replace the reagent vials.

## Replacing the Reagent Vials

After the reagent vial heaters have cooled to room temperature, the reagent vials are ready to be replaced.



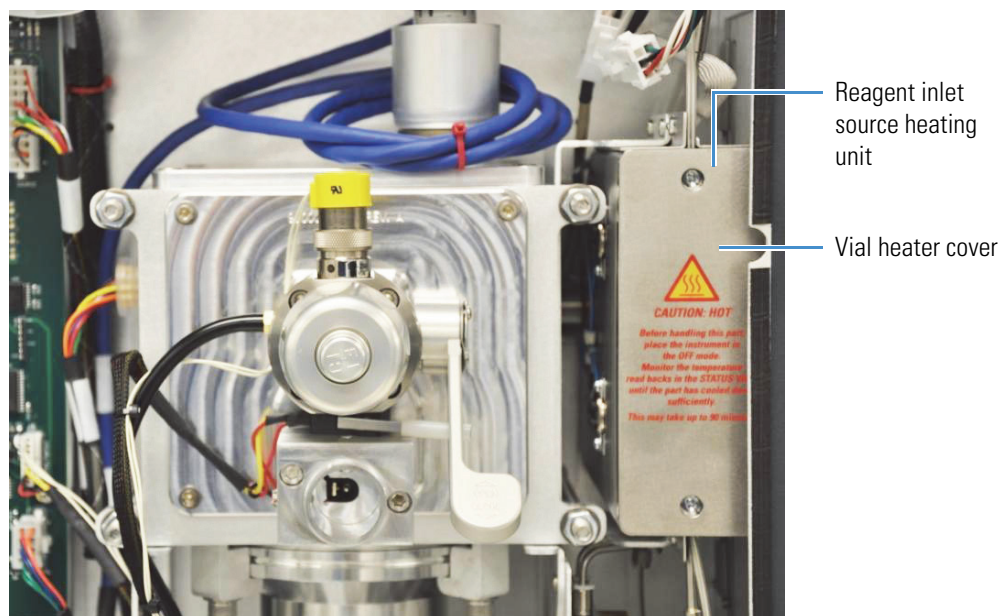
**CAUTION Hot surface.** Before removing the ETD module's back panel, follow the preceding procedure “[Shutting Down the MS/ETD System.](#)” Removing the main access panel before the system is in Service mode triggers the panel's safety interlock switches and stops all system activity including temperature monitoring. In the absence of temperature monitoring, you might attempt to handle the vials before it is safe to do so.

### ❖ To replace the reagent vials

1. Remove the ETD module's main access (back) panel. See “[Removing the Access Panels.](#)”

This exposes the reagent inlet source heating unit and the vial heater cover ([Figure 54](#)).

**Figure 54.** Reagent inlet source heating unit (EMI cover not shown)



2. Make sure that the vial heater cover is cool to the touch.

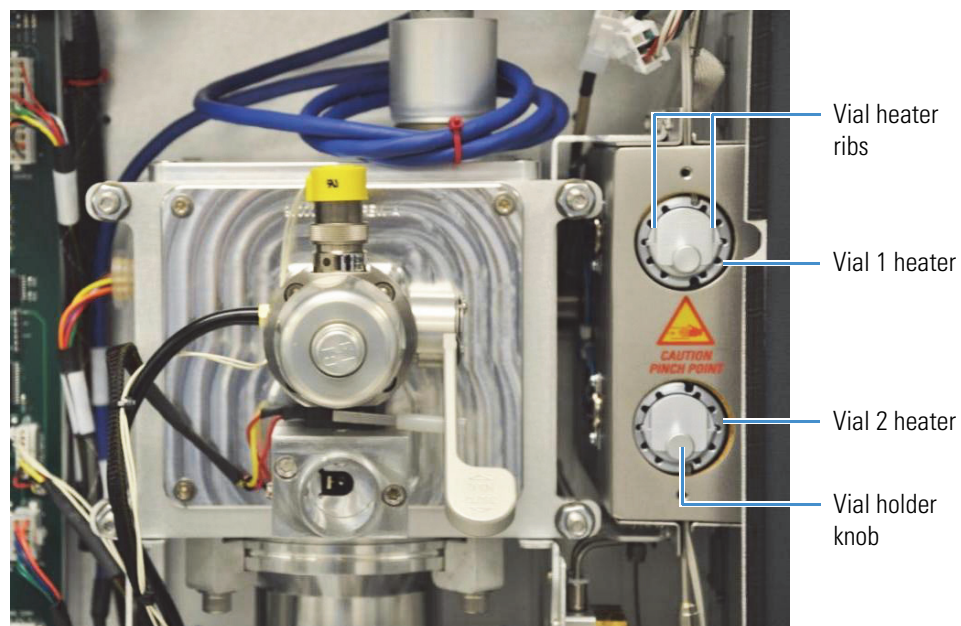


**CAUTION Hot surface.** Verify that the vial heater cover is cool to the touch before removing it. Allow sufficient time for the vials to cool (up to 90 minutes) and then place the system in Service mode. Verify that the vial heater cover is safe to handle before attempting to remove the vial holders and reagent vials.

3. Using the 7/64 in. hex driver, remove the hex socket-head screws in the vial heater cover.
4. Replace the reagent vial as follows:
  - a. Remove the reagent vial holder, which is typically the Vial 1 holder, by gently pulling it out of the vial heater.

The vial holder is a cylindrical tube with a handling knob at one end and ribs along its length. These ribs prevent the vial holder from rotating after you place it into the vial heater (Figure 55).

**Figure 55.** Reagent inlet source heating unit with the vial heater cover removed



- b. Remove the empty reagent vial.
- c. Dispose of the empty fluoranthene vial in accordance with its MSDS.  
See the cautionary statement on [page 69](#).
- d. Take the vial containing the ETD reagent (fluoranthene) from its box and remove the aluminum tab from the top of the vial's crimp seal.
- e. Place the reagent vial into the vial holder.
- f. Reinstall the Vial 1 holder into the Vial 1 heater (top vial heater).

**Note** If you place the reagent vial in the Vial 2 heater, remember to change the location on the Reagent Ion Source page of the Instrument Configuration window.

5. If an empty vial is not already installed, follow this step:
  - a. Take the empty vial from its box and remove the aluminum tab from the top of the vial's crimp seal.
  - b. Install the vial into the Vial 2 heater (bottom vial heater).



**CAUTION** The empty vial installed in one of the heaters is an integral part of the carrier/CI gas system. You must install both vials (reagent and empty) to keep the carrier/CI gas system closed off from the laboratory. If you do not install the empty vial, the following occurs:

- The carrier/CI gas containing the reagent might escape to the laboratory causing a safety problem.
- The ETD module does not operate correctly and the filament burns out.

6. Reinstall the vial heater cover.
7. Reinstall the ETD module's main access panel. See [“Removing the Access Panels.”](#)

## Starting Up the MS/ETD System

### ❖ To start up the MS/ETD system

Place the mass spectrometer's electronics service switch in the Normal Service position.

The system starts up and goes into standby mode. The ion source heater, flow restrictor, and transfer line heaters start heating.

## Forepump Maintenance

Maintaining the ETD module forepump requires inspecting, adding, purging, and changing the pump oil.

Check the forepump oil often. It is a translucent light amber color. During normal operation, oil must always be visible in the oil level sight glass between the MIN and MAX marks. If the oil level is below the MIN mark, add oil. If the oil is cloudy or discolored, purge the oil to decontaminate dissolved solvents. If the pump oil is still discolored, change it. Plan to change the pump oil after every 3000 hours (or about every 4 months) of operation.

Because the optional oil mist filter connects to the ballast port, be aware that opening the valve also drains the oil mist filter. While Thermo Fisher Scientific recommends that you periodically open the gas ballast valve (on the top of the pump) to purge the accumulated solvents, opening the valve might allow a large volume of volatile solvent waste to enter the fume exhaust system. Make sure that your exhaust system can accommodate the periodic purging of these solvents. The frequency of the purging depends on the throughput of the system—never operate a pump continuously with the gas ballast valve open.

Refer to the manufacturer's documentation for procedures to purge, add, and change the forepump oil.

## Fan Filter Maintenance

Clean the fan filter every 4 months, or sooner if it is dirty. The fan filter is located on the back on the ETD module.

### ❖ To clean the fan filter

1. Remove the fan filter from the back of the ETD module by pulling it out of the bracket.
2. Wash the filter in a solution of soap and water.
3. Rinse the filter with tap water.
4. Squeeze the water from the filter and allow it to air dry.
5. Reinstall the filter in the bracket.

# Diagnostics

The diagnostics test many of the mass spectrometer and ETD module components. If the diagnostics indicate that PCBs or other electrical components within the ETD module need replacement, contact Thermo Fisher Scientific Technical Support or your local Thermo Fisher Scientific field service engineer.

## Contents

- [Running the ETD Module Diagnostics](#)
- [Replacing a Fuse in the ETD Module](#)
- [Replacing Power Supplies](#)
- [Replacing PCBs](#)

## Running the ETD Module Diagnostics

Use the ETD module diagnostics, which are completely integrated into the mass spectrometer diagnostics, to test the major electronic circuits within the instrument. Refer to the Tune Plus application Help and Chapter 6, “Diagnostics,” in the *LTQ Series Hardware Manual*.

**IMPORTANT** Thermo Fisher Scientific recommends that you optimize the reagent CI gas pressure once a week.

### ❖ To optimize the reagent CI gas pressure

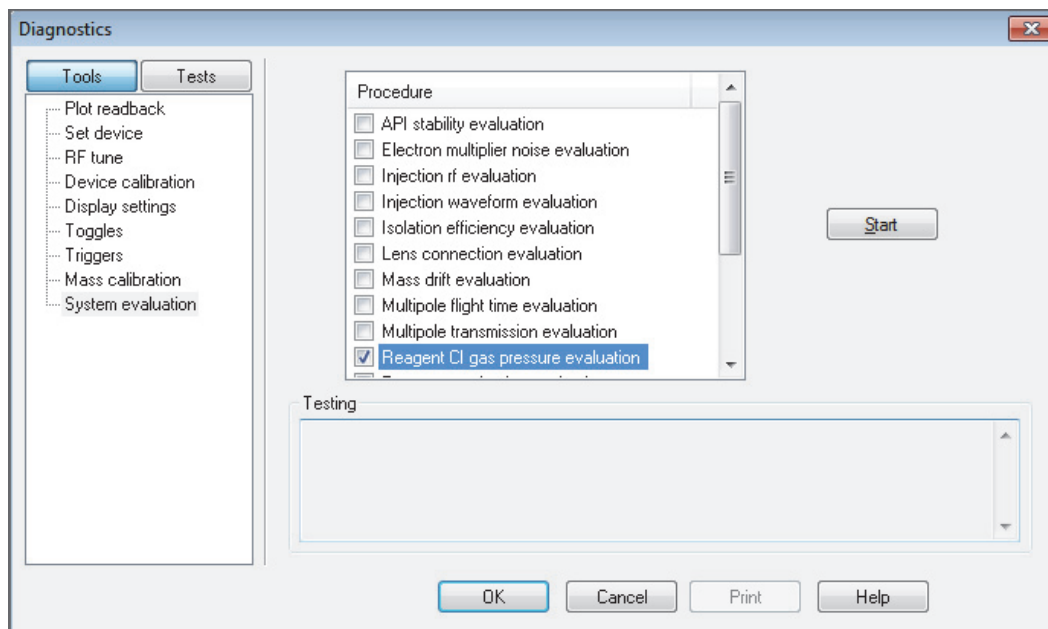
1. Open the Tune Plus window (see [page 13](#)).
2. Choose **Setup > Reagent Ion Source** to open the Reagent Ion Source dialog box.
3. Select the **View Reagent Ion Spectra** check box, and then click **Apply**.
4. Choose **Diagnostics > Diagnostics** to open the Diagnostics dialog box.

## 6 Diagnostics

### Running the ETD Module Diagnostics

5. In the Tools list, click **System Evaluation** to open the System Evaluation page (Figure 56).

**Figure 56.** Diagnostics dialog box showing the System Evaluation page (Velos Pro/ETD)



6. Select the **Reagent CI Gas Pressure Evaluation** check box.
7. Click **Start**.

This diagnostic evaluation takes approximately 15 minutes. After the evaluation finishes, the following message appears in the Testing area:

Est. required CI Gas Pressure for Max Reagent Ion Signal (psi): *number*

8. In the Reagent Ion Source dialog box, in the CI Gas Pressure (psi) box, enter the estimated gas pressure number determined from the reagent CI gas pressure evaluation, and then click **OK**.

You must manually enter the new reagent CI gas pressure.

9. In the Diagnostics dialog box, click **OK**.
10. Click **File > Save**.



## Replacing a Fuse in the ETD Module

Fuses protect various electrical circuits whenever an overcurrent condition occurs. The ETD module's replaceable fuses are located on the ETD heater control PCB and the 150 Vac power supply PCB.

Check fuses when power is lost to a fused subsystem.



**CAUTION** Always shut down the system and disconnect the power cord before you replace fuses.

**Table 5.** Fuses for the ETD heater control PCB (P/N 98000-61031R)

Location	Circuit	Description	Part number
F1, F2	220 Vac	6.3 A, 250 Vac, slow blow, 5 × 20 mm	00006-11450
F3–F10	220 Vac Heater	2.5 A, 250 Vac, fast blow, 5 × 20 mm	00006-11202

**Table 6.** Fuses for the ±150 Vac power supply PCB (P/N 98000-61040)

Location	Circuit	Description	Part number
F1	24V input	2 A, 250 Vac, fast blow, 5 × 20 mm	00006-02-00006



**CAUTION** Use only the fuses specified in [Table 5](#) and [Table 6](#). Never replace a fuse with a fuse of a different type, voltage, or current rating.

## Replacing Power Supplies

Due to the complexity of removing and reinstalling the ETD module power supplies, a Thermo Fisher Scientific field service engineer must replace these components.

## Replacing PCBs

Due to the complexity of removing and reinstalling many of the ETD module PCBs, a Thermo Fisher Scientific field service engineer must replace these boards.

## **6** Diagnostics

### Replacing PCBs

## Reagent Kit

Table 7 lists the items in the Reagent Kit (P/N 98000-62008) for the Velos Pro/ETD and LTQ XL/ETD systems.

**Table 7.** Reagent Kit contents

Description	Part number
Angiotensin I, 1 mg	00301-15517
Fluoranthene, 0.15 g	HAZMAT-00013



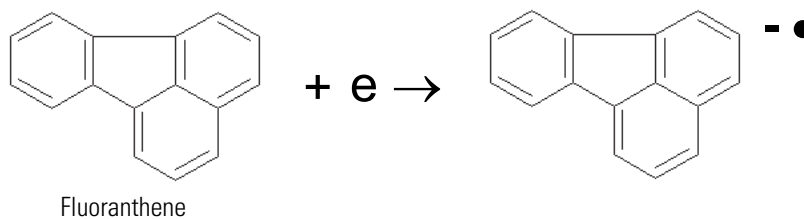
### CAUTION Avoid exposure to potentially harmful materials.

By law, producers and suppliers of chemical compounds are required to provide their customers with the most current health and safety information in the form of Material Safety Data Sheets (MSDSs) or Safety Data Sheets (SDSs). The MSDSs and SDSs must be freely available to lab personnel to examine at any time. These data sheets describe the chemicals and summarize information on the hazard and toxicity of specific chemical compounds. They also provide information on the proper handling of compounds, first aid for accidental exposure, and procedures to remedy spills or leaks.

Read the MSDS or SDS for each chemical you use. Store and handle all chemicals in accordance with standard safety procedures. Always wear protective gloves and safety glasses when you use solvents or corrosives. Also, contain waste streams, use proper ventilation, and dispose of all laboratory reagents according to the directions in the MSDS or SDS.

Fluoranthene is used as the ETD reagent in the ETD module. Fluoranthene is potentially hazardous. Use it in accordance with its MSDS.

The fluoranthene radical anion is generated according to the reaction shown in [Figure 57](#).

**Figure 57.** ETD reagent (fluoranthene radical anion) generation from fluoranthene

Thermo Fisher Scientific supplies fluoranthene as a two-vial kit. One vial contains 0.15 g of fluoranthene and the other is the required empty vial. The fluoranthene vial in the Velos Pro/ETD or LTQ XL/ETD system Reagent Kit is Sigma-Aldrich™ Supelco™ #48535. To obtain another fluoranthene vial, click the MSDS link from the Sigma-Aldrich website:

[www.sigmaaldrich.com/catalog/search/ProductDetail/SUPELCO/48535](http://www.sigmaaldrich.com/catalog/search/ProductDetail/SUPELCO/48535)

Angiotensin I (human acetate hydrate) is potentially hazardous. Use it in accordance with its MSDS.

The angiotensin I vial in the Velos Pro/ETD or LTQ XL/ETD system Reagent Kit is Sigma-Aldrich Sigma #A9650. To obtain another angiotensin I vial, click the MSDS link:

[www.sigmaaldrich.com/catalog/search/ProductDetail/SIGMA/A9650](http://www.sigmaaldrich.com/catalog/search/ProductDetail/SIGMA/A9650)

## Replaceable Parts

This chapter provides the part numbers for the chemical and accessory kits that come with the ETD module as part of the Velos Pro/ETD or LTQ XL/ETD system. It also lists the replaceable and consumable parts and their corresponding part numbers.

**Note** Accessory kits for the Velos Pro and LTQ XL mass spectrometers are listed in the *LTQ Series Hardware Manual*.

### Contents

- ETD Ship Kit
- ETD Accessory Kit
- ETD Reagent Kit
- ETD Nitrogen Scrubber (Gas Filter) Kit
- Inlet Valve Seal Kit
- Reagent Inlet Module Installation Kit
- Miscellaneous Parts

### ETD Ship Kit

<b>ETD Ship Kit</b> .....	<b>98000-62004</b>
Exhaust hose, blue, 1.0 in. ID, 1.8 m (6 ft) long .....	00301-08301
Fitting, barbed tee, 1.0 in. OD .....	00102-10120
Power supply cord, international, 230 Vac, 10 A .....	00302-03069
Power supply cord, international, 230 Vac, 15 A .....	96000-98036
Power supply cord, North American, 230 Vac, 16 A .....	96000-98035

### ETD Accessory Kit

<b>ETD Accessory Kit</b> .....	<b>98000-62002</b>
Aluminum oxide sample .....	32000-60340
Centering ring, aluminum/neoprene, DN 16 ISO-KF .....	00108-02-00014
Centering ring with O-ring, nitrile/aluminum, NW40 .....	00108-02-00005

## 8 Replaceable Parts

### ETD Reagent Kit

Centering ring with O-ring, Viton™/aluminum, NW25	00108-02011
Clamp, swing, aluminum, DN 10-16 ISO-KF	00108-02-00015
Clamp, swing, aluminum, NW10/25	00108-02-00018
Clamp, swing, aluminum, NW40	00108-02-00004
Clip, hose, stainless steel, 21.8–27 mm (0.85–1.06 in.) ID	00201-99-00109
Ferrule, two-piece set, brass, back, 1/8 in. ID	00101-02500
Ferrule, two-piece set, brass, front, 1/8 in. ID	00101-08500
Ferrule, column, graphite/Vespel™, 0.4 mm ID, 1/16 in. OD	00101-18100
Filament assembly	120320-0030
Fitting, straight tube, 10-32 thread, 1/8 in. <i>l</i>	00103-10990
Fitting, tube, bulkhead union, 1/4 in. OD	00103-01-00001
Fitting, tube, bulkhead union, 1/8 in. OD	00101-04-00002
Forceps, specimen, stainless steel, 10 in.	76360-0400
Fuse, fast-blow, 2 A, 250 Vac, 5 × 20 mm	00006-02-00006
Fuse, fast-blow, 2.5 A, 250 Vac, 5 × 20 mm	00006-11202
Fuse, slow-blow, 6.3 A, 250 Vac, 5 × 20 mm	00006-11450
O-ring, Viton, 2-005, 0.101 in. ID, 0.70 in. <i>w</i>	00107-02456
O-ring, Viton, 2-116, 0.737 in. ID, 3/32 in. <i>w</i>	00107-10056
O-ring, Viton, 2-254, 5.484 in. ID, 0.139 in. <i>w</i>	00107-01-00054
O-ring, Viton, 2-258, 5.984 in. ID, 0.139 in. <i>w</i>	00107-01-00055
O-ring, Viton, 1.237 in. ID, 0.103 in. <i>w</i>	00107-10404
Screw, thumb, knurled, 8-32 thread, 3/8 in. <i>l</i>	00430-01-00004
Swagelok™-type nut, brass, 1/8 in. ID	00101-15500
Tool, lens alignment	120271-0001
Tool, Torx™ star-bit driver, T-10	00725-01-00022
Tool, Torx star-bit driver, T-27	00725-01-00006
Kit, ETD Nitrogen Scrubber	98000-62011
Kit, Inlet Valve Seal Replacement	119265-0003
Kit, Reagent Inlet Module Installation	98000-62006

## ETD Reagent Kit

<b>ETD Reagent Kit</b>	<b>98000-62008</b>
Chemical, angiotensin I, 1 mg vial	HAZMAT-15517
Chemical, fluoranthene, 0.15 g vial	HAZMAT-00013

## ETD Nitrogen Scrubber (Gas Filter) Kit

<b>ETD Nitrogen Scrubber Kit</b>	<b>98000-62011</b>
Ferrule, two-piece set, brass, back, 1/8 in. ID	00101-02500
Ferrule, two-piece set, brass, front, 1/8 in. ID	00101-08500
Ferrule, two-piece set, stainless steel, 1/8 in. ID	00101-08-00009
Hydrocarbon trap	A0950-01600
Swagelok-type nut, brass, 1/8 in. ID	00101-15500
Swagelok-type nut, stainless steel, 1/8 in. ID	00101-12900
Tubing, copper, precleaned, 1/8 in. OD, 3 m (10 ft) long	00301-22701

## Inlet Valve Seal Kit

<b>Inlet Valve Seal Kit</b> .....	<b>119265-0003</b>
O-ring, Viton, 0.530 in. ID, 0.082 in. <i>w</i> .....	3814-6530
Spool, inlet valve seal .....	119683-0100
Tool, inlet valve seal removal .....	119283-0001

## Reagent Inlet Module Installation Kit

<b>Reagent Inlet Module Installation Kit</b> .....	<b>98000-62006</b>
Ferrule, back, PTFE, for Swagelok 1/16 in. tube .....	00101-08-00008
Ferrule, front, PTFE, for Swagelok 1/16 in. tube .....	00101-08-00007
Ferrule, Vespel/graphite, 1-hole, 1/16 in. OD, 0.4 mm ID .....	00101-08-00005
Ferrule, Vespel/graphite, 2-holes, 1/16 in. OD, 0.4 mm ID .....	00101-08-00006
Tubing, fused silica, 0.363 in. OD, 0.1 mm ID, 100 mm long .....	98000-20060
Tubing, PEEKsil™, 1/16 in. OD, 0.05 mm ID, 100 mm long .....	00109-02-00021

## Miscellaneous Parts

### Forepump

Forepump oil, 1 L .....	00301-15101
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### Ion Volume Parts

Ion volume .....	98000-20052
Ion volume holder .....	70001-20532
Ion volume insertion and removal tool .....	98000-60028

### Ion Source Filament Parts

Filament Assembly DSQ II .....	120320-0030
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## **8 Replaceable Parts**

Miscellaneous Parts





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